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## George MERKL

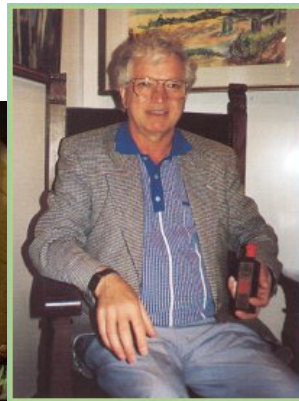
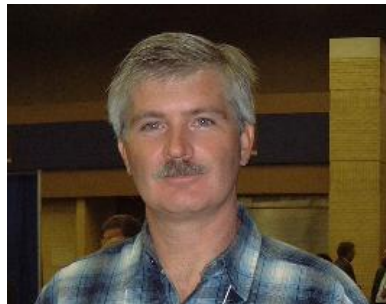
### Chondriana & Life Crystals

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### Dr. George Merkl



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[http://www.zoominfo.com/people/merkl\\_george\\_56848950.aspx](http://www.zoominfo.com/people/merkl_george_56848950.aspx)

George Merkl is a nuclear physicist and president of Life Crystal Incorporated in El Paso, Texas. He has conducted research for the past 30 years in his own research laboratories, and holds more than 40 U.S., and hundreds of international patents on his discoveries and inventions. Dr. Merkl is listed in "Who's Who Among the Pioneers in Science Today". His latest discovery is the present discovery particle biochemistry and the secret of life. He is the author of "Inner Space (Unified Theory)", and he has discovered a new treatment which can cure AIDS, cancer, and many other degenerative diseases.

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<http://www.consumerhealth.org/tothealth/tot01/29.html>

GEORGE MERKL, PhD (El Paso, Texas) -- George Merkl is a nuclear physicist and president of Life Crystals Canada of Oliver, BC, where his crystal-based health products are made. He has conducted research for the past 38 years in his own research laboratories, and holds more than 40 U.S., and hundreds of international patents on his discoveries and inventions. Dr. Merkl is listed in Who's Who Among the Pioneers in Science Today. His latest discoveries deal with chlorophyll crystals, crystals of consciousness and fractal perception. He is the author of *Inner Space* (Unified Theory) and *Whispers of the Cosmos Begin to Blow*.

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#### Life Crystals Canada Inc.

(250) 498-2228

Ray De Blasi, Vice President

Products based on discoveries by Dr. George Merkl about a new form of life that destroys cancer and repairs organs

### **Preface to the Genesis Factor**

In mid-summer of 1990, Warren York informed us of a most interesting man who had developed almost a new form of science single-handedly. His name is George Merkl.

After speaking with George on the phone several times, we were so ecstatic that a trip was planned over a weekend to visit with him.

Three of us made the trip by air and what we found exceeded all our expectations. George proved to be everything we hoped for and we learned much new information. We also took some of our papers and video tapes so he could see where we were coming from.

The primary discovery George had made years ago was a principle which he termed SCROLL WAVES. These waves are extremely small and can be likened to Aether or Zero-Point-Energy moving in continuous spiraling patterns.

To quote from George's privately published book "Inner Space":

"The photon is not a constant but is energy based on DECAYING spinning wave resonance. Now when an energy exchange takes place between two electrons (or particles), the electron may move with a velocity.

When the particle has absorbed or attained enough energy, it may get rid of that energy by emitting it in a full or partially MUTILATED wave form.

But a second particle or electron may REABSORB THE SAME ENERGY, whereas BOTH will SPIN and RECOIL IN OPPOSITE DIRECTIONS.

The direction will be determined by the recoiling photons. The photon transfer will be responsible for the force set up between the pair of electrons. The photon is energy in wave form which acts as a force field.

This moving wave form can convert to current as the force field is being produced when the photon RECOILS WHILE IT IS SPINNING.

This force is related to the inverse square law for the force of attraction and repulsion between two interacting particles through energy exchange."

George has many patents in several fields, with the majority on new chemical processes.

### **THE GENESIS FACTOR**

#### **Introduction by Warren York**

Up to this point I have covered the unifold of scalar technology covering up to and including relativity. I have talked about the relations of scalar technology with gravity, 3D time and space, and the phenomena spectrum.

I had planned to talk about scalar technology and free energy this issue. In the first issue I had stated scalar technology would open doors to new frightening and fantastic areas never before attempted in depth all under the same roof.

Well, thanks to Dr. George Merkl who is a part of our loose net group or team we have crossed over the fantastic into the unbelievable. I knew after I had talked about free energy and scalar technology I would have to explain it's relationship to what man calls the life element [Genesis Factor] or life itself.

This is a touchy area and one that should be approached with caution. George -- who does not like to be called DR -- has broken the code of life itself. Unlike Dr. Frankenstein, George has achieved it in an acceptable way. I cannot go into great detail at this time for the work must be checked and double checked to make sure what we are observing is indeed taking place.

We are in the process of recording the documentation for future release. We have reached the point that the test must be brought under control instead of uncontrolled as lack of proper equipment and lab conditions are forcing us to try and narrow down very exciting data that is slipping through our fingers at the moment.

If anybody out there can locate such needed equipment or proper funding to run a full-blown research lab and facility please contact me at the address below. We are putting on videotape what uncontrolled data we have learned but even that is hampered by lack of proper equipment.

We will make do until better is available and I am sure when you see the video does you will find it hard to believe. Dr. Henry C. Monteith who is also a part of our research group had been down here last weekend to see what we have and was very impressed on what George had discovered.

George's discovery alone can turn the world conditions around and we can once again have a clean and healthy planet to live on and offer our future generations. This new learned info can also help man as he ventures out into space to set up new homes and work places.

This research lab I am trying very hard to make a reality will benefit all mankind. A think tank for mankind [ not just a corp. ] which will generate new fields and jobs. Solve problems not yet thought of and bring together some of the best free thinking minds in the world to do something about tomorrow today.

Dr. Monteith alone wished to develop a new concept in microscopes. His project is known as the Litraonics Microscope and can show the structure of an actual atom unlike any resolution done by MIT or IBM to date. I will have photos taken of an atom with this equipment and more information on his progress about the device in future issues.

Also Mike Windell who is a part of our group has developed our first prototype of scalar communications. It is known as an advanced telecommunication project. The prototype is being developed as a demo to be presented to METS out of Florida for possible future funding for

future development.

The unit will transmit a signal through any kind of shielding. We are working with ranges up to one mile so far without any problems that so ever. The units are portable and mobile. Mike has discovered a very important element in any scalar field generation. In order to have true scalar generation one must generate a " Shear Effect ".

I have not gone into this in any of my past articles for this is new info. Also the design of the scalar coil has changed drastically since the original release here.

As I said all data is being put on video and sure there will always be people who say we faked it but we have witness to backup the tapings.

I'll let George tell you in his own words now about the Genesis Factor or what life actually is itself and how it is generated.

Keep in mind George is a nuclear physicist with over 400 patents to his name. George will talk about scroll waves. Scalar is a process where scroll waves is the actual Aether in motion. This motion is known as free energy or life energy itself. It is all related to what we have talked about in the pasted articles.

I wish to remind anybody who needs to contact any person in our active group please contact me through our New mailing address listed below. At this point we are in need of the use of an electron microscope to gain needed data on a new life form we can't match up to any known so far. They are still in the embryo stage at the printing of this paper.

On a microscopic scale they have rib cages, internal organs, eyes, mouth, digestive track and what looks like a hand.

We also have on videotape crystal clones [ that growing crystals but not alive ] on a micro scale of what looks like a complete pine tree branch or fern, insect wings and other fantastic events.

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[http://totse2.net/totse/en/fringe/fringe\\_science/genesis1.html](http://totse2.net/totse/en/fringe/fringe_science/genesis1.html)

## SYNTROPHIC TRANSDUCER

by

**George Merkl**

Discovery of the atomic wrench by myself, George Merkl, underlines my earlier work of transducing scroll waves (free energy braiding into photons) to a higher energy level to control chemical bonding.

Scroll resonant energy leaves its signature on everything throughout the whole universe including all forms of life.

### RESIDUAL OUTCOME FROM THE THIRD COLD FUSION PROCESS:

According to the law of quantum mechanics the electron between two carbon fluctuates between positive and negative carbon. To assure the stability between two carbons in the diamond lattice, I proved that the carbon in the diamond is indeed in the positive and negative ionic state, and is also responsible for the hardness.

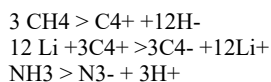
Since polarity is resonating back and forth, it seems non-polar.

The fact that I was able to make diamonds of both type one and two at atmospheric pressure by pairing homopolar carbon ions also proves that diamond is homopolar!

My real interest was to introduce single ionic nitrogen to form carbon bonds by an exchange reaction in the lattice substituting carbon isomorphously.

These carbon nitrogen bonds form paramagnetic centers, and convert ortho hydrogen to parahydrogen with paramagnetic properties simply by transducing an energy level via resonance from scrolls to reverse the spin of the proton!

I used the following reaction for this purpose:



The above CN bond spacing was 10% over carbon to carbon bond.

According to quantum mechanics the probability of nitrogen oscillation frequency in the molecule will be the function of the superimposed frequency which is generated by the oscillator crystal excited by the scrolls.

The nitrogen is stationary and once excited by its transition frequency in a giant molecule of carbon, the energy is transferred and the carbon bonds have to move or oscillate.

Therefore, the oscillation of the crystal transplants its energy to form chemical bonds, or transduces energy from scrolls sufficient to reverse the spin of a proton to form para proton. That shall be the base of a clean industrial revolution paving the way to save this planet.

[FIG 1]

Let's make a detour from this major technological breakthrough! Recently I discovered that the powerhouse I developed to transduce energy naturally had it all along.

Such a photo transducer power house is common to all living organisms including plants, microbes, and human cells (including brain cells).

As we know it as the chlorophyll family --- all are dependent upon scrolls, absorbing it from our atmosphere and utilizing it as we know is done in photosynthesis.

[FIG 2]

After thirty years of fruitful research, and research of plant life in the last five years, I do have ample proof that scrolls are responsible for the basic functions of life.

If we drastically change our atmospheric condition by changing conductivity, plants will be deprived of energy and will die!

Scrolls are responsible for pumping the fluid into the capillaries. This is directly related to moisture content, static charge, and conductivity of the atmosphere.

Life first evolved from the sea because the depths are a soup of scrolls where water is saturated under pressure.

This is the reason for the spiral or vortex fingerprint on seashells, "The very form of the free energy" scrolls. In search for a more nature-made phototransducer, I found many links to nature's chemistry by prediction.

For example, in a heteroautotrophic system, sugar was reduced to alcohol, and  $\text{CO}_2 + \text{H}_2\text{O}$  were synthesized to sugar, thus completing the cycle.

In addition to chlorophyll the first two photo transducers I tested were coffee and theobroma oil. I chose to study the latter over cocaine because I used them in phototransducing in the fermentation process. I did not want contamination by the nature of cocaine.

I found that both products are capable of phototransducing energy -- the maximum results having been achieved between twelve AM and three PM.

It was not hard to determine how drugs speed up biological processes and burn out the cells including that of the brain! The energy they transduce not only depletes the scrolls from the blood stream but overloads the cell circuits.

A chlorophyll pair absorbs the photon quantum at 670 nm. My atomic wrench output is at a shorter wavelength and a higher frequency of oscillation.

The following are some of the nature-made energy transducers:

PROCAIN, QUININE, HEROIN, METHADONE, CODEINE, COCAINE, CAFFEINE, NITROUS OXIDE (N-O-N EXCEPTION), ACTIVE VITAMIN B TWELVE, REACTIVE CYANOCARBON ETC.

They all carry the carbon nitrogen fingerprint!

Utilizing the phototransducers, I was able to raise the alcohol level of wine produced by solar fermentation to 80 proof containing only natural pure solar distilled grapejuice.

I would like to add here that during the drought in June 1990 in El Paso, Texas, the humidity level dropped to 6% which drastically reduced the conductivity of the air as well as the static charge of the air!

As a result most plants went into dormancy; the phototransducers in the chlorophyll stalled due to the lack of conductivity; all fruits stopped growing in spite of extensive irrigation.

Except on plants whose surroundings were altered by raising local conductivity artificially, I also found out that the capillaries of the Persian Lilac grown, were fewer but many times larger in diameter, due to lower flux density produced by the weak fields.

This phenomenon is directly related to the conductivity and static charge. These comparisons clearly showed the opposite effect on the same root section during the wet season here in 1989 when conductivity and static charge was above average.

[ FIG 3 ] THE POWERHOUSE TRANSDUCER IN THE CYTOCHROME [ evolution of life ]. WITHOUT THIS ENGINE, LIFE COULD NOT SURVIVE. IT'S FUEL IS THE SCROLL.

Now we have seen the syntrophic phototransducer are similar to autotrophic and heterotrophic transducers which nature built within the living organism, these are of course, unquestionably prerequisites to life formation and existence.

The leaders of the world with total ignorance, miscalculating the catastrophic consequences while tampering with our atmosphere, which their advisors know nothing about.

This planet could re-cycle too soon if the world continues to ignore the facts!

We have seen how the C-N-Bond, semi-absorbed, promotes paramagnetic sights where ortho to para conversion takes place. By upsetting the ortho-para ratio the hydrogen will lift ortho delta positive link to the delta positive atoms such as halo delta positive, CH delta positive, HWC delta positive, and so on.

This opens the way to "Transport Reactions", a new branch of science even the greatest person could never dream of. (MAJIC) (Transporting suboxides coupled to para hydrogen, vapor phase at low temp).

I utilized this new technology to build alternate rows of nitrogen-doped carbon atoms where the layers of 111 oriented surfaces formed a superlattice. (All 111-100 and 110 faces were constructed from the bent carbon rings of graphite).

By using the paraproton transport reaction technology, I pioneered new forms of metallic carbons, diamonds etc., with different D spacings, but I synthesized hundreds of exotic new materials which are perhaps a hundred years ahead of time.

The important facts are that some of these materials could solve many of the world's problems today, for instance, a pollution-free world; or harnessing of the sun's clean energy, by capturing the free energy (Scrolls).

My syntrophic transducer is not only able to stimulate solar fermentation, but could also produce electric current and free hydrogen through transducing the lower level energy.

Chlorophylls have different geometries of antenna. This produces different patterns of isobars, as the resonant molecule absorbs or emits its transduced photon energy.

The three dimensional field so produced follows the scroll pattern sideways, which is similar to my three dimensional field produced in my first cold fusion process in the sixties.

Electrons excited by the scrolls are different from electrons in the normal state as verified by their fingerprints on the EPR. spectrum. Through this, energy couples with the nuclei orientation of the nuclei differ with respect to an external field.

The larger the number of excited electrons, and their spectrum proportional to their number, "The higher, the narrower the band will be".

Scrolls determine the size of the photon, that is absorbed by the spin-aligned electron or proton, this alters their position correspondingly.

Thus, their energy level is transduced to the absorption frequency to match the absorption frequency of the host electron or proton.

Changing the direction of spin on a proton depends on the strength of the external field, and can also change the absorption spectrum due to spin polarization.

"A strong North pole of the proton (Blackhole) can suck in countless scrolls by their South poles and reverse the spin. By forcing the position of the proton to the opposite attracting position via scrolls and by reversing the orientation of the proton, ortho proton can be converted to para proton.

In the atomic wrench, the nitrogen and electrons result in hyperfine interaction. The hyper-excited carbon nitrogen transducer can lase immense photon flux and subsequently transduce enough upshift of the frequency to reverse the proton spin.

"FREE ENERGY" THE SEED OF LIGHT":

"A three dimensional vortex, performing three dimensional motion, inverting thru its poles back and forth and changing signs constantly."

The scrolls form strands with matching frequency of oscillation and equal wavelength. These strands of scrolls braid into photon vortexes. 918 pairs of photons (Magic number) feed the vortex of an electron.

Again a 917 pair (5 pairs off) electron vortex copies the genetic trait to form the proton, and 918 electrons form the neutron. (See: Inner Space 1968, balanced).

Every time an atomic vortex inverts through a blackhole the scrolls form a coherent field followed by pole splitting which then reverts through a white hole followed by an incoherent field.

Each absorbed scroll adds up to increase the mass of the host.

However due to the increased mass, the spin will change on the host, followed by a change in wavelength and frequency of oscillation.

[ FIG 4 ] FREE ENERGY [ SCROLLS ] AT WORK!: The plasma membrane of the living cell is permeable to ions that are driven by the self-induced field generated by free (scroll) energy.

The net result is an electrical potential between the inside of the cell (-) and the outside of the cell (+). Sodium and chlorine ions are responsible for the conductivity in the plasma medium. (Reference material concerning physics. See Patent: USP 3,993,595).

[ George Merkl -- USP # 3,993,595 -- Activated aluminum and method of preparation thereof (November 23, 1976 )

Abstract -- Reactive aluminum consisting essentially of aluminum having a purity by weight of at least about 99.99% permeated by a liquid metal selected from mercury, gallium and gallium/indium alloys. The permeation can be accomplished at room temperature and atmospheric conditions. The reactive aluminum exhibits greatly altered physical and chemical properties as compared to the aluminum before permeation. These new properties include a uniform alignment of atomic grain boundaries and domains and a multitude of channels defined in the reactive aluminum. ]

The cell membrane along with its plasma, acts like a condenser, and is negatively-charged inside with respect to the outside which is positive.

The condenser membrane is charged by the scrolls (static charge of the air). The potential difference varies between 40 to 80 mv depending on the scroll energy density.

Such free energy density will vary with the incoming cosmic rays, and also with atmospheric conditions such as pressure, moisture content, static charge, etc.

"THE GTP AND ATP ARE THE PRIMARY ENGINE OF THE LIVING CELL"

In the case of G.T.P. the Guanosine is the energy transducer and emitter while the Triphosphate is the storage battery. The free energy charging the cell membrane (from static charge like a condenser) on discharge self-induces its field to which the ions are attached and flow.

When the cyclic ring enzymatically cleaves onto the G.T.P., the battery releases energy and generates a reverse field. This action counters the self-induced field on the cell membrane, and becomes the driving force of the ion flow across the cell membrane.

This mechanism controls the ionic flow by switching it on and off.

On each cycle the electrical properties of the membrane change (depending on the nature of the signal) and give rise to nerve impulses.

The increase or decrease of the cell permeability comparable to the variation of domain cross-section in the above given reference patent and is of course directly proportional to the free energy and also to the ENVIRONMENTAL CONDITIONS.

"In general, all biological activity strictly follows the weather pattern", and it's being dependent on the unmolested atmospheric condition.

I hope you all realize the scope of this discovery, underscoring your general feeling or existence which is of course influenced by tampering with your environment.

As the cyclic G.M.P. opens during hydrolysis a water molecule inserts into the ring, being triggered by a light beam from below my incubator.

Reacting yeast and plant cells in separate experiments (My solgraft process) in a G.M.P. and A.T.P. soup, the experiment turned out as predicted. -- The G.M.P. depolarized the cell membranes by field.

The self-induced field on the cell membrane generated by the static charge of the air was cancelled by the G.M.P.'s gravitational field induced by sunlight!

The resulting decrease in ion flow across the cell membrane retarded the self-induced field and the potential difference, thus opening the way to communicate in and out of the cell's genetic material!

By (Solgraft) assembling, disassembling, grafting, transplanting within or outside of the cell and by architecture of R.N.A. and D.N.A., I began grafting G.M.P. and A.T.P. to proteins which led me to grafting alternate transducers to acceptor sights.

When we are talking light-induced hyperpolarization we should also talk about spin control which is controlled by the external field strength.

This in turn, controls the ion flow. I extracted sub-units of RNA-G.M.P. (by isol process).

After purification I hydrolyzed the crystals in distilled water, and I observed that the crystal strongly attracted to the polyethylene wall charged by static.

The mono crystal disintegrated into countless macro crystals, always forming hollow spherulets in water!

These spherulets possessed strong magnetic force! In other words, crystals coupled like tiny magnets to form what one might call "Supercrystals".

"I also observed and experienced levitation". I placed a one inch diameter incubator with 1 ml genetic mix sitting on a glass plate and passed a beam light thru it from below.

As I added the G.M.P., the incubator levitated a few mm above the glass plate. I moved the incubator away, at least fifteen times, and it kept on moving back and lined up with the beam of light.

By sliding the incubator it felt like moving a repelling magnetic pole. This gave me the clue that there are anti-gravity interactions within the living cell, a phenomenon I have known long since it has existed with plants.

I placed my synthetic soup of RNA and DNA into my incubator, I observed that within twenty-four hours it organized into its normal hair-like structure.

It appeared that the sub-units of the RNA and DNA had their own field and assembled accordingly.

I called my first synplant cofina because I derived its genetic part from coffee.

Cofina consists of a single capillary plant whereby the nutrients are delivered to the top where the budding developed via.

The size of the Mono capillary (length) is determined by the self-induced field generated by the free energy! Once the capillary reached its height (the peak of the fluxlines) the genetic soup oozed out, and all the G.M.P. moved to the surface forming a unisphere.

(I observed the clear transparent sphere of genetic soup). Within twelve hours the brown bundles branched out forming the flower that carried the genetic code for the next generation!

I harvested the abundant crops and germinated them in water and nutrients. The thousands of macro seeds or clones (Units) began to germinate, growing first their roots.

Within thirty-two hours they were grown to the mirror image of their parent. The seeds are in the sub-micron range and carry their genetic code. I want you to understand that this is not putting a patch on the double helix!

"THE MAIN THRUST HERE OF COURSE IS TO HARNESS SOLAR POWER"

I already engineered microbes and phototransducers that liberate hydrogen from water; however most importantly, I recently extracted genetic materials from pollen and I cured my hyper-allergy that tortured me for thirty years!

With this solgraft process it is feasible to cross-graft genetic materials between microbes and plants, replacing the transducers on the RNA or DNA with synthetic ones.

It is perhaps tempting to call Cafina and Tina inorganic life because it contains organic materials and so closely resembles grain and domain alignments.

In conclusion genetic materials can be easily produced, reconstructed, or transplanted and grafted at ease by the solgraft process.

[FIG 6]

[FIG 7] LIGHTHOLES -- gencrystals form a hollow unisphere due to their magnetic properties. The surface crystal layers of the unispheres absorb light and turn black, except at their poles, as light passing through them creates a vortex of light.

The absorbed light by the surface crystals is then transduced to higher energy level.

[FIG 8] Static field pumps the ion flow in opposite directions across the cell membrane and produces a self-induced field. This is comparable to the scroll fields of the roots of the plants, whereby driving the fluids into the capillaries is directly proportional to the static charge and the conductivity of the air.

By realigning the grain of an ultra-pure aluminum, the magnetic moments will couple, forming linear domain capillaries. The aluminum will acquire magnetic properties.

The free (static) energy will pump ions in the opposite direction by micro capillaries. This self-induces a strong magnetic field which is comparable to life process.

[FIG 9] (Contains genetic soup, held in spherical form via magnetic fields generated by genetic sub-units)

[FIG 10]

[FIG 11] An inorganic product that grows like crops and resembles living plants. Its growth rate is directly proportional to the free energy (Static charge of the air), and to the conductivity of the air -- the exact same criteria that are prerequisites for ALL FORMS OF LIFE!

[FIG 12] [SHEET A] SYNTHESIS OF LIFE BY GEORGE MERKL

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<http://sam.vmicrobial.info/>

Vinny Pinto

Excerpt from : **Syntropic Antioxidative Microbes (SAM) -- a Unique Class of Probiotic Beneficial Microbes**

## **Dr. George Merkl and his Solar-Fermented Life Crystals and Sumerian Elixir Products**

by Vinny Pinto

I first heard of Dr. George Merkl and his exotic nutritional supplements -- which were then marketed under the names Life Crystals and Sumerian Elixir, and later under numerous other names as well -- in the mid-1980s, when a small mail-order nutritional supplement vendor in northern Wisconsin from which I occasionally purchased products started listing his two products in their catalog and also started distributing marketing pamphlets making some very wild claims for Dr. Merkl's two products. I purchased his strange nutritional supplement products occasionally throughout the mid and late 1980s and more frequently throughout the 1990s. Eventually, by the mid-1990s, I became acquainted with two persons who had worked closely with George Merkl for many years in developing and producing his exotic nutritional supplements, and, over time, I heard a number of fascinating tales from them about Dr. Merkl and his products, which, by then were being marketed under a variety of brand names, including Life Crystals, Sumerian Elixir, Sumer-Tech, Sumerian Drink, Renaissance Drink, Renaissance Beverage, Vida Force, Vida Source, Vida Lyze, Gaiandriana and Aquagaia. It has been estimated by observers of the fringe nutritional supplement marketplace that at the height of their popularity -- which would be from roughly 1982 to 2003 -- many tens of millions of dollars worth of Dr. Merkl's products were sold in the US and Canada and also in over 48 other countries, often accompanied by claims that the products would, in short order, cure everything from diabetes and heart disease to cancer. These extreme and blatant -- and almost entirely undocumented and unverified -- health claims for a product that had never been approved for therapeutic drug use, along with Merkl's uncanny ability to manage to continually antagonize and run afoul of regulatory agencies -- led Dr. Merkl and some of his distributors into repeated troubles over the years with the FDA and Health Canada, and with several other government regulatory authorities across the world.

Dr. George Merkl was born and raised in the Soviet Union in the early 20th century, where he earned a PhD in physics. In the wake of some serious difficulties with the Soviet authorities in the 1940s just after the end of World War II, he emigrated to the USA in the 1950s, settling primarily in the El Paso, Texas area. He reportedly worked briefly for the US Naval Weapons Laboratory in the field of nuclear weapons research and then went into partnership with a few other PhD-level colleagues in forming a private R&D laboratory which primarily performed contractual R&D services for the US Naval Weapons Laboratory and for other defense-related sectors of the US government; much of his research work was still apparently in the realms of nuclear weapons development and the design of nuclear reactors for use on submarines. He often claimed in later years -- when he had shifted his focus to the field of esoteric nutritional supplements -- that he had been awarded over 400 patents between 1955 and 1980, and that many of them had been immediately classified as top secret by the NSA due to their applicability in defense-critical areas.

Dr. Merkl was badly injured in at least two accidental laboratory explosions during the 1960s and 1970s, and one of them also reportedly left him with severe plutonium poisoning due to inhalation of airborne plutonium particles. Concomitantly, he also developed several chronic and severe health problems commonly associated with aging, including heart disease, and it is safe to assume that his earlier injuries and the plutonium poisoning only greatly exacerbated these chronic illnesses. In the mid-1970s, Dr. Merkl suffered several severe heart attacks, and in the wake of his last one, apparently in early springtime of 1977, he was essentially sent home from the hospital by his doctors to die; they had reportedly advised him that his heart disease was beyond help and that he likely had only weeks to live; he also reportedly had rather severe bowel cancer at the time, with at least one large cancerous mass in his lower bowel. Now, this is where the story gets weird and where the fun begins....

Merkl reported in later years that he had several mystical experiences in those weeks after he had been sent home to die in 1977, and most of these experiences seemed to happen at night, during sleep. He later claimed that angelic beings had come to him during these mystical experiences, and had told him that it was not necessary for him to die from his illnesses, and they offered to guide him along the steps to a speedy recovery. Merkl reported that he accepted their offer, and that he was, in short order, instructed to go out to the field his lower backyard -- it happened to be early springtime -- and to carefully cut the pre-blossom rosebuds off the wild rose vines growing there, and was advised to collect the liquid sap that would emerge from the cut tips of the plant stems over the next few days. He was instructed to process this small quantity of liquid in a modest and simple alchemy-like process, and was then guided to drink it. He was, within a few weeks time, cured of his heart disease and of the cancer, and of a number of other illnesses from which he had also been suffering.

By mid-1977, once he had been returned to a state of good health, Dr. Merkl was then reportedly told by his inner angelic advisors that the process that he had been given to prepare the springtime rose vine sap elixir, while it had been quite useful for him on a one-time emergency basis, was not at all practical to adapt to large-scale production of a healing elixir, and so they offered to teach him an alternate method to create larger volumes of what they claimed with be a very much similar nutritional supplement elixir which would exhibit very powerful healing properties. The process that his angelic guides taught him required him to procure certain organic fruit juice concentrates (including



grape, blueberry, black cherry, raspberry and currant) from specific sources and to also procure several dried organic or wild-crafted teas (primarily chamomile, jasmine and oolong) that had been grown only in volcanic soils in certain specified regions of the world (primarily parts of western Central America and South America), along with two or three proprietary ingredients, and to ferment them for several months in a strange rooftop glass-enclosed apparatus -- which he eventually came to call a solar reactor or a solar still -- based upon a design that they had given to him, and his guides further specified that the rooftop solar reactor had to be positioned to allow maximal sun exposure. Merkl later, in both lectures and in some of his writings, often referred to his solar fermenters as syntrophic transducers, for he felt that they, and the process of fermentation therein, lent powerful syntropic properties to his products. He once referred, in a lecture, to his solar fermenter or solar reactor in the following fashion "My syntrophic transducer is not only able to stimulate solar fermentation, but could also produce electric current and free hydrogen through transducing the lower level energy. ..." In a related 4-page article that he wrote in the late 1980s or early 1990s, entitled Syntrophic Transducer, Merkl also wrote the his solar fermenter, aka syntrophic transducer, functioned by "....transducing scroll waves (free energy braiding into photons) to a higher energy level to control chemical bonding....Scroll resonant energy leaves its signature on everything throughout the whole universe including all forms of life...". Scroll resonant energy leaves its signature on everything throughout the whole universe including all forms of life. This concept, and terminology are, of course, very much similar to, and in fact, almost identical to, that employed by Dr. Terugo Higa (see the following section), the developer of a SAM Type 4 consortia culture called EM, in describing the syntropic or negentropic exotic energy effects, which he called "wave energy" or "gravitational wave energy" of his EM product between 1992 and 1998.

The result of this process was a mildly acidic reddish-brown liquid with a not-unpleasant taste somewhat akin to that of the old patent cough syrups of the early and mid-1900s. Merkl eventually, by the early 1980s (if not earlier) started to market this product as Sumerian Elixir and later as Renaissance Drink, and, much as referenced earlier, over the longer term Merkl and his associates and distributors marketed various versions of this liquid under a number of other trade names as well; a large reason for the never-ending parade of trade names was the simple fact that Dr. Merkl seemed to be constitutionally unable to simply market his product as a useful and nourishing nutritional supplement, and instead, he apparently simply could not refrain from making extreme health claims for his product, claiming that it exhibited drug-like effects in healing a wide variety of diseases. And, since he and his associates had never put his product through clinical testing and clinical trials in order to gain approval from the FDA and Health Canada to market it as a drug, his bizarre and ultimately self-defeating tactic ensured that he was constantly on the radar screens of the FDA and Health Canada, and that he was continually running afoul of these agencies and other regulatory agencies. Interestingly, most of the Merkl products disappeared from the marketplace by 2003, and the very last of the Merkl-inspired products seem to have disappeared from the marketplace by the year 2006. As of today (it is now early 2010), I am not aware of any products using Merkl's Sumerian Elixir (aka Life Crystals, Renaissance Drink, etc.) technology available on the market anywhere in the Western world.

A few years ago, in 2006, after I had developed some useful and rapid non-culturing-type proprietary assay methods for determining the presence of PNSB in liquid samples, I carefully searched my storage cabinets and managed to find some old bottles -- dating back to the 1980s and 1990s -- of Merkl's liquid product that had been marketed under each of several brand names, and I subjected the liquid samples to some tests in my lab. These tests showed in short order that the liquid exhibited a pH below 3.9 and that the acidity was largely due to lactic acid from fermentation, and my proprietary PNSB assays (mentioned earlier) quickly showed that the products contained very significant quantities of PNSB and their metabolites, thus confirming my hunch that the microbial culture that was found in Merkl's nutritional supplement product was a SAM Type 4 consortium. (With this information, along with some intuitive guidance from my own inner guides and angels, I was then quickly able to determine the likely proprietary ingredient that Merkl had used in creating his fermented elixir, and was able, in short order, to brew several fermented products which were very much like Sumerian Elixir (and, of course, Renaissance Drink.)

Since I know a good part of the process that Merkl used in brewing his primary product, which was the Life Crystals Drink (aka Renaissance Drink), and also based upon some of the characteristics (to be discussed below) of this product, it is obvious that Merkl's fermented product did not employ commercial cultures purchased from microbial type culture collection vendors, and rather, the exact makeup of the SAM Type 4 microbial consortia present in each of his batches of fermented product was determined solely by the naturally-occurring microbes present in and on the surfaces of the various natural ingredients that he used, along with the unique array of nutrients and micronutrients available in the organically-grown ingredients used in the formulation. However, this led to some interesting twists and challenges when it came to the marketing of his products, particularly the Life Crystals Drink and the Sumerian Elixir, for each of these nutritional supplement products contained a significant amount of alcohol due to ongoing fermentation by wild naturally-occurring yeast during the active brewing process.

While FDA rules allow nutritional supplements and foodstuffs to contain up to 1% alcohol content and still be classified as nutritional supplements or foods, in general, if any such product contains over one percent alcohol content due to natural fermentation processes, then, with few exceptions, the product no longer falls under the purview of the FDA, and rather, it is subject to the much stricter and harsher rules for the brewing and sale of alcoholic beverages formulated by and enforced by the Bureau of Alcohol, Tobacco and Firearms (BATF). Since most batches of Merkl's basic primary products -- the Life Crystals Drink and the Sumerian Elixir -- contained well over 1% alcohol, and most often contained from 2% to 3.5% alcohol, this fact eventually came to the attention of first the FDA and eventually BATF, and his US production facility in El Paso, TX was eventually classified by BATF as an alcoholic beverage brewery, and his products were likewise eventually classified as alcoholic beverages that could be sold only in liquor stores.

Had Merkl employed tighter control over the brewing process, including use of commercially-available cultures available from microbial type culture collection vendors, along with certain other measures, he could easily have ensured that the amount of alcohol present in these two products would have remained well below one percent, most likely on the order of 0.2% to 0.5%, and he could have avoided much of his eventual hassles with the FDA and BATF.

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[http://www.arthritistrust.org/Articles/Life%20%20Crystals%20&%20Chondrianas/pg\\_0001.htm](http://www.arthritistrust.org/Articles/Life%20%20Crystals%20&%20Chondrianas/pg_0001.htm)  
<http://www.consumerhealth.org/articles/display.cfm?id=19990303163417>  
[http://www.garynull.org/documents/arthritis/life\\_crystals\\_and\\_chondrianas.htm](http://www.garynull.org/documents/arthritis/life_crystals_and_chondrianas.htm)  
<http://www.friendsoffreedom.com/links01.html>

## **Life Crystals and Chondrianas**

by

**Perry A. Chapdelaine, Sr.**



*Published on: 4/1/1992*

"Medical data is for informational purposes only. You should always consult your family physician or one of our referral physicians prior to treatment" -- The Arthritis Trust of America

Article provided by: The Arthritis Trust of America. This is a final report

The Roger Wyburn-Mason and Jack M. Blount Foundation for the Eradication of Rheumatoid Disease, AKA The Arthritis Trust of America, 7111 Sweetgum Road, Suite A, Fairview, TN 37062-9384

### ***Summary as of December 20, 1993***

The Rheumatoid Disease Foundation board voted to open-mindedly investigate George Merkle's claim that he had discovered a new form of life that destroyed cancer and repaired damaged organs. He called this form of life Chondrianas. Apparently a mixture of adenosine triphosphate(ATP) and guanosine triphosphate(GTP), ribose, nucleotides, glucose, fructose and glucose phosphates, under the right conditions, enticed this organism from where it was hidden, in the human (or any life form), for an unknown number of millions of years.

If the claims are true, then mankind, through George Merkl, has apparently discovered its fountain of youth.

After reviewing (1) The Life Crystal Manual, (2) video tape New Frontiers in Molecular Biology: Life Crystals Capturing the Sun, and (3) Discovery, Part II, all purchased from Life Crystals, Inc., I visited George Merkl at his home, in El Paso, TX, finding him to be a sincere, engaging personality.

Several thousand dollars were sent to Life Crystals, Inc. for both the Life Crystals mixture and Chondriana. The Chondriana we received proved to be live gram-negative bacteria.

The Rheumatoid Disease Foundation then learned that physicians in all parts of the U.S. and Canada had had a similar experience in receiving live gram negative bacteria with their orders, and not receiving Chondriana. Altogether thousands of dollars have been spent for gram-negative bacteria for injections into the human body, rather than the alleged beneficent Chondriana.

Dr. Merkle's response to our laboratory findings of live gram negative bacteria in what was supposed to be his Chondriana order was his vehement denial that he had sent any Chondriana to anyone in the United States. As The Rheumatoid Disease Foundation, as well as other physicians, took pains to insure that money to purchase Chondrianas went directly to George Merkle (Life Crystals), then, either of two conditions seems to exist: (1) There are no Chondrianas, but rather live gram-negative bacteria have been sold as Chondriana, or (2) one or more of his suppliers are substituting gram-negative bacteria for Merkle's Chondriana.

To clear up this mystery, I asked George Merkle if he would send a sample of Chondriana, without further charge, to Lida Mattman, Ph.D., a specialist in pleomorphic organisms, for scientific study.

George Merkle agreed to do so, but no Chondriana has been received more than a year later, despite several faxes and letters reminding Dr. Merkle of his commitment.

Dr. Merkle may very well have what he claims, but he has not seen fit to share these results with the medical or biological community, so far as we know, to this date.

The major portion of this report was sent to Dr. Merkle, requesting correction of any false data prior to submission for publication.

### ***Life Crystals and Chondriana According to George Merkl***

Which choice does one make when a treatment promises everything and simply sounds too good to be true? How would one discriminate between the genius who has seen through to the heart of truth, and the con artist or self-dupe, who has merely obscured further the nature of reality?

John W. Campbell, Jr., the editor and writer who twitted the scientific establishment for their pompous airs and over-serious pronouncements, and also the same person who almost single-handedly formed the modern field of science fiction entertainment, advocated a simple criteria; namely, ask the question, "Does it work?"

This criteria, of course, is not always easy to apply, because it involves the willingness to divest oneself of the need to follow authoritarian leaders, to invest time and energy in self-search and appraisal, and to think for one's self. This latter criteria is not something we're exactly taught to do in modern society, even when the course is labeled "Creative Thinking," or even "Creative Science."

True geniuses are always outside the bounds of social restrictions, at least in the paths visited by their thoughts!

Dr. George Merkl may very well be one of those unsung geniuses quietly busy revolutionizing health, the world's food supply and cost of energy.

He says, "Nourish the body; it will thrive for a time. If you nourish the genes, it may thrive 100, 150 or 200 years.

His findings which he's titled "Life Crystals" and "Chondrianas" are most establishment-shaking, if true.

Dr. Merkl and his team "Produced gem grade diamonds and made them at various D spacing with brilliant luster and variations in colors. They researched and made fundamental discoveries related to cold fusion, transmuting aluminum-26 plus hydrogen to aluminum-27 1/2 isotope." In their "second cold fusion reaction, . . . [they achieved the ultimate by transmuting sodium, silicon, and nitrogen, to titanium, iron, and manganese, while releasing enormous amounts of energy [on a microscale]." Such experiments led to development of Life Crystals, which, in turn, led to discovery of microscopic, virtually intelligent "Chondrianas." "In their third cold fusion experiment, the researchers were able to advance far ahead of current technology, and actually reversed the spin of the proton," a new ability to control atomic particles which allows control of the bonding energies in chemistry. This work set the stage for creation of "synthetic transducers, and later to the natural energy transducers, those chemical structures that use" a newly discovered form of energy called "scroll" energy which is "free" and promotes the life process.

In some respects, this scroll energy can be described as "the breath of creation."

If these discoveries are true -- regarding the function and value of Life Crystals and Chondrianas -- and their interpretation of them even close to being correct, no one can afford to ignore their implications and use. Like the fantasy-figure, Superman, who, at a single bound, overtakes the fastest engine of destruction and neutralizes it, these researchers have seemed to abolish the need for pharmaceutical industries, the complex of interacting agri-industry giants and the constant search for cheap and clean fuel.

Their alleged astounding discoveries are based on application of nuclear physics to the biology of life and, like every other finding on the basic nature of reality, has seemed to cut through the Gordian knot of health, food supply and energy.

Do you believe this?

Well, it's too good to be true, and so you must either be the pessimist or the optimist. Possibly only by personal experimentation will you be able to decide that it therefore could not be true, or that it therefore must be true.

The reported discoveries are of such an order of magnitude that one simply cannot afford not to investigate Dr. Merkl's research, to try their developments and to make a decision oneself, especially at the relatively low costs for doing so.

Why?

Well, let's follow The Life Crystal Manual available, along with two video tapes, from Life Crystals, Inc., 5732 Kingsfield Dr., El Paso, TX 79912, copyrighted 1992. All materials reprinted from that manual are used with permission of its author.

### ***Life Crystals***

All life depends upon adenosine triphosphate (ATP) and guanosine triphosphate (GTP) which produces energy in conjunction with the citric acid cycle.

The energy reactions depend upon normalized enzyme activity. Therefore "all life is dependent upon properly formed and concentrated enzymes which in turn are dependent upon healthy deoxyribose nucleic acid (DNA) and ribonucleic acid (RNA) found within the nuclei of our cells.

"By providing readily available ATP and GTP, it is possible to ensure optimum energy levels necessary for cellular growth, repair, and function."

Life Crystals provide concentrated ATP and GTP in the purest form, with "the penta carbon sugars providing the four basic building blocks of life in pure form."

When light passes through these Life Crystals, they emerge polarized, and it is this polarized light that sets the stage for the evolution of life!

There are two forms of Life Crystals: one form (consisting, according to label, of ribose and nucleotides, 2% phosphates, 0.37 mg/ml including adeno tri phosphate and guano phosphate, fructose 15%, glucose 15%, including fructose and glucose phosphates, all solar extracts, natural products, no chemicals or preservatives added) can be used sub-lingually, placing two or three teaspoons (or tablespoons) per day under the tongue, leaving it there for two or three minutes and then swallowing it. It is also perfectly acceptable to drink several ounces per day. This form is available for purchase in the United States, as well as most of the rest of the world.

The other form is as an injectable, and in this form is available in the rest of the world, not the United States.

The advantage of the injectable is that the body will respond faster for cellular and genetic growth, repair and functioning.

While it is said that there are more than 4,000 patients who've used either the Life Crystals or Chondrianas or both, little clinical data seems to be easily available, at least so far to this writer. A small number of anecdotal medical case histories supplied to The Rheumatoid Disease Foundation by George Merkl, in Spanish, on partial translation did not seem to fit the exacting standards necessary for unequivocal findings. However, the alleged improvement (or cure) factor seems to be so huge and should be so obvious that perhaps clinical data will be useful only for those who must have such data -- or to satisfy FDA requirements.

As claimed from other than medical case histories, one person, George Merkl himself, claims that he had an incurable mixture of carcinoma and sarcoma in his hip, which is wholly cured from Life Crystals and Chondriana. He also had a serious heart problem, which is now wholly healed. His hair is turning dark from gray, his skin lacks the usual aged cross-linking signs, and Leydig Cells have been restored and he and his wife, beyond the normal age of childbirth, have had a child.

"If one could repair and normalize DNA and RNA function quantitatively as well as qualitatively, then it would be theoretically possible to postpone or even to some degree reverse the aging process by preserving cellular respiratory function. . . . it may now be possible to nourish DNA and RNA structures by providing the building blocks of these structures to the body. . . Nourish the body and it will thrive for a time, nourish the genes and it may thrive for multiples of that time. . . .With properly functioning enzymes, the body takes defensive measures against toxins and repairs any former damage. A disorder of any kind can be rectified by restoring enzymes under suppression. Properly functioning enzymes not only brings recovery but a measure of rejuvenation as well. More importantly, an ancient, prehistoric life form has been brought to life and replicated with a more concentrated form of the Life Crystal Solution. . . ." manifesting "the ancestors of our cell's nuclei and mitochondria." The process involved "forward and reverse synthesis of human RNA. . . ." The synthesized organisms were named Chondriana, a name related to the mitochondria found inside the cell, and the source of each cell's energy. Chondriana are our resurrected ancestors.

With the exception of the United States, it is said that many medical clinics around the world are "using the Chondriana in treating almost every known human disease with astonishing results."

While the Life Crystals by themselves will enable the body to function more normally, cleaning out arteries and capillaries, and producing optimum cell functioning, the Chondrianas are even more amazing, it is said, in their functioning inside the human body.

This is illustrated by placing one drop of human insulin in a vial of Chondrianas with Life Crystals "within 48 hours one has pancreatic beta cells producing insulin. . . . The insulin contains enough of the genetic information to generate the pancreatic beta cells from whence it came."

Diabetics, it is said, have been cured in three months!

A tremendous variety of other diseases, it is said, have also been cured!

### ***Chondrianas***

With the aid of life crystals, Chondrianas were discovered embedded as though frozen in time in the human gene.

Remember that ontogeny recapitulates phylogeny, meaning that our embryonic developmental forms pass through our ancestral forms (phenotypes), demonstrating our evolutionary predecessors.

"Now imagine the researchers taking apart sequenced T-RNA (Transfer RNA) chains that may hold primitive expressions of our ancestral heritage. Through the triplet nucleotides as a genetic remanent, imagine those genetic codes expressing themselves in the energy milieu of the life crystal solution, a solution that matches the energy of the ocean when the sun was young. Now picture these microscopic mobile creatures emerging from our genetic past, from the remanents of the three nucleotides: intelligent, protective, a reproducing ambitious enzyme manufacturing micro-culture dedicated to preserving human life at the colloidal life level. Does this discovery represent the bionic immunity of our ancestors of millions of years ago? Is this the Methuselah gene, uncovered? Or are these creatures the ancestors of human cells? Or of us? Indeed, they are! Other minds in future times will tell us, but relish now with me in not the why but in the reality, in the fact, that the researchers may have made the discovery of all time, the discovery of the Chondriana. A fact so spectacular that in spite of its reality, it should [or could] be placed more into the realm of science fiction."

Chondrianas are microbial, and appear to be "intelligent, sophisticated and highly organized . . . recalled to life from the triple nucleotides of human genes."

They are "precursors to our immune and cellular organelles."

They have male and female gender, undergoing courting and sexual reproduction.

The male chondrion enzymatically devours bacteria, viruses, and cancer cells, "or any microorganism that may jeopardize any human tissue," or endangers the female of its species, the chondriana.

The chondrion sequences and produces killer cells (chondrianettes), which are the precursors of the lymphocytes and T cells. "The killer cells can sequence DNA production of specific protein digesting enzymes that break up the cell membranes of foreign invaders, bacteria, viruses, and cancer cells, and thus make available the digested parts of their dead bodies as food for the chondriana. Although the life crystals are the primary energy source, the foreign invaders stimulate a fast reproductive cycle within the chondriana by giving them additional food. Life crystals supply the fuel for primary conversion via cyclic ATP complexing with magnesium ion. Once in the body, they convert foreign microbes to food for reproductive energy."

"The male chondrion resembles the mitochondria of our cells displaying a double layered membrane and producing energy. Thus the male, once placed in a concentrate of ATP solution, becomes activated, partially from ATP and partly from another source of energy . . . known as 'free' energy scrolls."

"Think of scrolls as energy packets, quanta, which are received from cosmic radiation which bathes the earth and universe night and day." Patent 399395, it is said, describes scrolls as "a three-dimensional energy vortex in motion. Nine hundred and eighteen pairs of such scrolls braid into one photon . . . the basic unit of light energy;" 918 photons into one electron; 918 electrons into one proton.

The DNA and RNA "hold the engines of life which actually absorb these scrolls. The various subunits of DNA are known as nucleotides. These in turn are made of ATP-GTP, ribose, and deoxyribose where the engines of life lie in the center of the ATP and GTP molecules with the remarkable ability, like the engine in the molecule, of absorbing and utilizing the 'free' scroll energy."

"The female has a single-layered membrane and is very inactive until she is 'charged' by the male. The female therefore resembles the nucleus of our cells, containing DNA, the blueprint of life. She is very full of this genetic material and has the ability to exchange part of it with the male during mating while the male passes on RNA to the female at the same time."

The female on being fertilized by the male produces three male chondrions, and then in succession, three female chondrianas.

The activities of the female and male chondrianas and chondrions have been photographed as they court, charge, bounce, mate and also exhibit various phases of orbital motions including the final moment of birth of male and female types.

Apparently these creatures were "independent life forms billions of years ago that have come back together, uniting to form a higher order of cellular life." Perhaps the output energy of our sun was greater millions of years ago. As the energy level decreased through time, creatures, such as the dinosaur, also decreased in size. Chondrianas can be observed to change size with changing energy levels, to a point of dormancy with lack of oxygen.

Energy deficient chondrianas revert to (and disperse into) small units that are virtually indestructible; and they reassemble again with the addition of the proper milieu.

When chondrianas are mixed with a five percent dextrose solution, and injected into the vein, one hour later almost to the minute the patient will begin shaking, experiencing a so-called T-Cell reaction. This reaction is usually followed by a Herxheimer reaction (die-off effect). Being dose-dependent, only small quantities at first are recommended until most foreign microbial invaders are slain and consumed by the chondrianas. Chondrions are enormously active in destroying foreign invaders, and as these invaders die, arteries are cleared and cancer cells are devoured, then the body goes through a healing crisis, it is claimed.

"Chondriana can survive and even thrive in human blood as part of the colloidal life of the plasma." They, themselves, are "colloidal life in crystallized form, supported by live particle biophysics. Their membrane structures are energy transducers using, to some degree, scroll 'free' energy."

"Oxygen is necessary to keep the chondriana active. If they are deprived [of oxygen] the colloidal energy generator will stall and no longer polarize their charge. At this point the chondrion will burst and colloidal chondriana will disperse. Upon reintroducing the oxygen and the life crystal solution, colloidal life will reassemble and resurrect from spilled genetic material."

In addition to producing insulin beta cells from appropriate genetic materials, chondrianas have produced all other parts of the human body, including brain, lymph node, kidney, heart, spleen and so on. The factory that produces faulty genes is often replaced by a factory that produces good genes. What has occurred in vitro has also occurred in vivo, producing genuine cures, it is said! The same person (George Merkl) who reportedly had the carcinoma/sarcoma mixture also had severe heart problems, and now appears to have a new heart, according to Dr. Merkl, himself.

"The sophistication of the AIDS virus is primitive compared to that of the chondriana." It has been reported that from wherever the T-cells count starts, with Chondrianas, the count doubles during a given time period, then doubles again and again.

"Researchers transferred [chondrianas] into ten different colonies and each colony acquired a defense system that nothing can match."

Rather than correct genetically defective cells, the chondriana replace the factory producing the genetic defects.

### ***Speculative Correlations***

It's easy to cast shadows against another man's dream, and sometimes harder to find supporting, concrete underpinnings for that same dream. I often try the latter first:

L. Ron Hubbard had the unusual capacity to be aware of his genetic evolutionary path, the efforts and counter-efforts that successively constructed different life forms from the earliest of genetic existence to that of man today. He perceived and published in 1952 *A History of Man*, a book designed to help Dianetic auditors to know the kinds of incidents that people would present during auditing. (This book can be purchased at any Church of Scientology location.)

One of the earliest precursors on Hubbard's genetic evolutionary linkage is the "photon convertor."

George Merkl speculates that millions of years ago, the sun produced more net energy for life-forms on earth. Perhaps that is the reason some plants and animals, such as the dinosaur, grew so large. According to Science News, "Speedy Spin Kept Early Earth From Freezing," (June 12, 1993), "calculations suggest the sun had only 70 percent of its current strength early in Earth's history, around 4 billion years ago." However, simulated computer calculations show that a then faster spinning earth had 20% less cloud cover, and with a 14 hour-long day, more sunlight reached the earth. Also, because there was more water surface, which absorbs sunlight, the temperature would have been 50 to 60 higher.

Life Crystal's earliest identification along the evolutionary pathway is a combination of a photon storage and power source, perhaps similar to ATP/GTP, and an organism that appears to be similar in form to Cyano Bacteria whose fossil remnants have been found in Australian rocks 3-1/2 billion years old. Together the ATP/GTP and resurrected chondriana appears to be best described as a "photon convertor," although "scroll" energy apparently also precedes "photon" energy.

Antoine Bechamp, a brilliant scientist of Louis Pasteur's day, probably outshining Pasteur as a scientist by light-years, described a microscopic organism called the Microzým which was pleomorphic, but reduced itself to small almost indestructible units on death of the body, and swept by the winds across the earth thereafter, to be recycled again in living tissue. (The Blood and It's Third Anatomical Element, by A. Bechamp, translated by Montague R. Leverson, M.D., 1911.) He concluded that disease was caused by the environment surrounding microorganisms, and that a one-to-one correspondence between unique organisms and specific disease was not universally true. The argument between Pasteur and Bechamp raged into political arenas, where Bechamp lost, and even though Pasteur recanted on his deathbed, stating that "Bechamp was right, the milieu is all," modern medicine chased after the one-to-one correspondence philosophy, thus helping to create the giant drug monopolies that constantly chase after a patented magic bullet.

Lida Mattman, Ph.D., in her second book on Cell Wall Deficient Forms (Chemical Rubber Company, 1993), identifies numerous pleomorphic organisms found from different disease states. Also, as Dr. Mattman reports, Ernst B. Almquist, Gerald J. Domingue, F.E. Haag, Koch, G. Koraen, Virginia Livingston-Wheeler, M.D., A. Maffucci, Gaston Naessen, P.G. Olsson, Royal Rife, E.J. Roukavischnikoff, Jorgan U. Schlegel, Gerda Troili-Petersson, Willibald Winkler, M.D., Hannah B. Woody, W. Zopf, and many others, have followed up wholly or in part, or rediscovered, Antoine Bechamp's work.

Alleged chondriana discoveries demonstrate that the very earliest organism had a form dependent not only upon its milieu, but also on the duties that it had to perform. This is nowhere clearer than in its reported production of T-cells and lymphocytes according to need, as well as other forms important to the construction, repair and defense of the female chondriana and its host partner, the human body. On reducing energy levels, chondrianas become sluggish, and then eventually disperse into very tiny "almost indestructible units" to be resurrected again on being provided with a photon/scroll energy source.

### ***How Used***

The life crystals sub-lingual solution is purchased in quart quantities at retail or discount prices. Although the sub-lingual dosage can vary considerably, according to physical condition, it was recommended that one teaspoon three times a day be placed sub-lingually for about 3 or 4 minutes, and then the solution swallowed.

As these life crystals pervade the system, the body should be enabled to clean out fine capillaries and arteries, and otherwise prepare the body for its own repair.

So long as Life Crystals makes no health claim for this product, it is easily purchased in the United States and elsewhere.

The injectable life crystals is preferred for severe illness and quick physical response, it is said.

There are apparently any one of several ways that these injections can be taken. One physician takes a small quantity alternatively with the Chondrianas twice a week. Probably the preferred method is to use the life crystals first, setting the body up for the Chondrianas later. The two products have different functions, but the Chondrianas rely on the life crystals, and later photon/scroll energy absorbed through the skin, for their anti-microbial and human tissue and organ reconstruction functions.

Life Crystals injectables are not available in the United States, but are available in nearly every other country in the world.

Presuming that the choice is to take the injectable life crystals first, the next step is to inject the Chondrianas into the vein. With Chondrianas two effects must be carefully observed: T-cell "chills" reactions and the Herxheimer reaction from release of toxins when killing off massive quantities of foreign micro-organisms.

It's important that the individual be capable of detoxifying these effects, and so in a very sick person, one must be extremely careful not to introduce large quantities of Chondrianas into the veins in one injection.

As one investigator said, who was also using these products on himself, "I've plenty of time. The body needs time to respond and repair, and I'm willing to give it that time." He started by injecting 0.05 cc, and very, very slowly increased the dosage, taking alternatively either life crystals or Chondrianas twice a week. He then took 0.2 cc Chondrianas and 0.3 cc life crystals, alternatively. Eventually he experienced an extreme chill from the T-cell reactions, feeling great the next day. He also reported that he could walk better now than he had since his spinal injury many years ago. On observation some months later, this author could not view any improvement, and it is believed that the investigator change his mind about his own improvement.

Another physician took at first treatment .10 cc and experienced a very heavy Herxheimer, but also felt great the next day.

The Chondrianas are also not available in the United States, but available nearly everywhere else.

It shouldn't take more than about 40 days to complete the total treatment of life crystals and chondrianas, unless one is exceedingly sick, in which case, perhaps more of both products would be required, it was said, and certainly it will take longer for the body to be repaired.

Chondrianas gain energy through the skin by exposing the skin to sunlight, and they will multiply and reproduce if placed in the sunshine, as on the windowsill.

### **FDA**

According to personal conversation with a Compliance Officer of the Food and Drug Administration, neither the Life Crystals injectable or the Chondrianas are being stopped at the border provided the patient has a letter from his/her doctor stating that the patient is authorized to use the products for their personal use, and that it is not for resale.

### **Anecdotal History**

A name and telephone number was given to me by George Merkl, at my request, because of their presumed "arthritis."

Two people who had heard of life crystals and Chondrianas two years ago, a mother and son, both pianists, had had crippling Rheumatoid Arthritis. The mother, 68, was so badly disabled that she had to use the palms of her two hands to pick up a bottle. Immediately on the first injection of life crystals arthritic pain disappeared and neither mother or son have had the pain during continuing treatments, or since, the son reported. The mother also suffered from nephritis, congestive heart failure and removal of one-half a lung but now has drastically improved health. She can pick up a bottle with her fingers, he said.

The 41 year old son stated that within 24 hours of injections, Rheumatoid Arthritis swelling had gone down 50%. During the past two years he has used a maintenance dosage of sub-lingual life crystals of 15 to 20 drops per day. He's had no serious flu or viral sickness for more than one and a half years. He can play the piano again, he said.

The son's mother-in-law in Korea has diabetes and high blood pressure. The diabetes is vastly improved and the blood pressure now normalized, he said.

I found it easy to reach the son on the first call and I was told, by him, that I would be able to pass through his secretaries at any time I wished to talk further. I was never again able to reach this person for follow-up questions.

The treatment described herein, and laid out by Life Crystals, Inc., appears to be superior to every known treatment, and applicable to all human conditions. It promises to eradicate virtually all known diseases!

We don't know that this statement is true!

But there was sufficient "probable cause" so that many physicians in foreign countries are, reportedly, trying it on themselves or their patients and there are some anecdotal reports -- chiefly from George Merkl -- of positive results, so that a number of U.S. physicians, including a number of The Arthritis Fund/The Rheumatoid Disease Foundation's referral physicians, have also investigated.

The Foundation mailed out the preceding to all of its referral physicians, and some of its board members and friends began taking the Life Crystals and alleged Chondrianas, as described, ordering either directly from Life Crystals, Inc., or through intermediary physicians who also sent International Money Orders directly to Life Crystals, Inc. or to George Merkl, Ph.D. Dr. Merkl stated that he would accept only International Money Orders from outside-the-U.S. for Chondrianas.

Information Learned and Received on Life Crystals and Chondrianas as of the last date shown: December 21, 1993

1. One doctor reported good results with several cancer patients. No data on the number or kind of effects, and the report is a secondhand source without clinical data.
2. One doctor reported zero success on two cancer patients. One non-success with diabetes.
3. One doctor had written the following: "I am very concerned about the information you sent out on July 8, 1993 with regard to life crystals and chondrianas. I have had personal experience with these products, not only on patients, but myself.

When I first took an injection of so-called chondrianas, I suffered a 'shake and bake' reaction followed by a severe headache that in my mind could only be compared to what one experiences in an endotoxin reaction from bacteria. Practicing holistic medicine, it had been years since I had seen such a reaction, but having experienced it myself I was convinced that is what it was.

I witnessed the use of chondriana on several patients and saw absolutely no results. In fact, one patient with a lung cancer proceeded to have his life wasted away while he was taking chondriana injections.

One of my patients was interested in chondriana even after I advised him my office performed a culture on a fresh tube and found at least 4 different varieties of live gram negative organisms in an unopened vial. He received a vial directly from Dr. Merkl and I received yet another one. At the same time, Dr. \_\_\_\_\_ in New York, was evaluating the material also. Cultures were performed simultaneously on 3 separate vials

received by Dr. Merkl and all three grew out at least 4 different gram negative organisms and no tube had the same organism. Dr. Merkl was confronted with this information and came up with various stories and explanations that wouldn't hold water even with my 11 year old child.

Usually anything that sounds too good to be true isn't true and I feel this is another case in point. I believe that treatment with so called chondrianas is nothing more than treating patients with a form of treatment known years ago as Coley's Endotoxins, which did have a substantial cure rate for certain cancers. However, using live bacteria could put the patient at a far greater risk.

"I would appreciate it if you would keep this information confidential and check out the product yourself. If you do, please let me know what you find."

The preceding letter was also furnished to Dr. George Merkl with a request for explanation and also a request that he furnish, free gratis, a sample of Chondrianas to a well-known scientist who has specialized in pleomorphic organisms, Lida Mattman, Ph.D.. There has been no response from Dr. Merkl on this request as of December 21, 1993. Contrary to the normal free-flow of communication, Dr. Merkl has not answered any further telephone calls from me after informing him of the above letter.

4. Another doctor has furnished this office with a copy of Richard B. Prior's Clinical Applications of the Limulus Amoebocyte Lysate Test (CRC Press, 2000 Corporate Blvd., N.W., Boca Raton, FL 33431, 1990). In particular, attention was directed to chapters 1 and 5. As the test described in this book is exceedingly sensitive and accurate for endotoxins, samples of Chondrianas will be tested when the test kit arrives.

When injecting small quantities (0.05-0.10cc) of Chondrianas, as purchased from Dr. Merkl, within 50 to 60 minutes an extreme chill will probably set in. This chill lasts for 1-2 hours, after which a light Herxheimer occurs.

The above-described phenomena fits well the symptoms of endotoxemia as described in Prior's work. Endotoxins in small amounts affect macrophages and monocytes, B-lymphocytes and Complement.

Macrophages activate, increasing interleukin-1 and tumor necrosis factor (TNF). These in turn create an acute protein phase and fever.

B-lymphocytes also activate, increasing antibody synthesis.

As does Complement by an alternative pathway, thereby producing inflammation.

Endotoxins in large amounts produce all of the above, plus possible shock via the macrophage/monocyte chain; and Hageman factor platelets which release clotting and other release factors resulting in disseminated intravascular coagulation.

The production of Tumor Necrosis Factor (TNF) could explain some of the good results obtained with some cancers.

Lipopolysaccharides (LPS) do not directly activate T cells but have been found to stimulate T-cell differentiation. This could explain why T-cells appear to increase in AIDS victims.

Effects considered to be beneficial by the use of endotoxin include but are not limited to immunogenicity, immunomodulation, nonspecific resistance, tolerance and induction of colony stimulating factor (CSF) and g-interferon.

Adverse endotoxin effects are that almost every organ in the body and many metabolic pathways are affected.

Cytokins, for example, are considered major mediators of circulatory collapse and tissue necrosis observed in Gram-negative septicemia. Anticoagulant property of endothelial cells are lost and fibrin deposition is enhanced, and so on through a cascade of a multiplicity of other adverse affects, sometimes resulting in shock and death.

The usage of endotoxins for clinical purposes lies on a knife edge of determination as to what is too much or too little for an individual patient and also a function of age and metabolic efficiency. Apparently there is no good test for determining appropriate quantities for a specific individual for the induction of beneficial results, as opposed to no results or even hazardous results.

*August 26, 1993*

Talked to another doctor who stated that University analysis of so-called Chondrianas showed a mixture of pathogens.

*August 28, 1993*

1 CC of Chondrianas as received was mixed with 3 CC of distilled water, and the sample mailed to SmithKline Beecham Clinical Laboratories. Today the The Arthritis Fund/Rheumatoid Disease Foundation received the laboratory report stating that endotoxin range was greater than 10.0 EU/ML with a reference range less than 5.0.

*August 31, 1993*

A doctor reported placing a sample of Chondrianas in dark field view, finding only two small rod-shaped organisms of a type found in the bloodstream, as per pleomorphic reports of Gaston Naesson.

*September 2, 1993*

Three of four samples of alleged Condrianas obtained by The The Arthritis Fund/Rheumatoid Disease Foundation were studied under the microscope by another physician. Only gram-negative rod-shaped bacteria were found.

*September 3, 1993*

Serge Durand (Health Canada, Drug Directorate, Health Protection Building, Tunney's Pasture, Room 212, Ottawa, Ont. K1A 0L2) reported to us that they have no record of George Merkl, Life Crystals or Chondrianas, and especially have not authorized its usage under Conditional Notice of Compliance, Emergency Drug Release Program, Authorization for Investigation, or Notice of Compliance.

Also on this day George Merkl called this Foundation in response to a letter citing some of the preceding problems. Dr. Merkl stated that someone else in America was manufacturing fake Chondrianas made with pathogens and that these were not his organisms. That reputable clinics in Mexico had gotten good results from the use of his Chondrianas, and that he'd now created Chondrianas from Horse, Bear and other

animals. There were only a small handful of people authorized to sell Chondrianas, and unless the product came from one of them, it was not his Chondrianas. He agreed to send this Foundation a sample free gratis for testing and study by a scientist who specialized in pleomorphic organisms, Lida Mattman, Ph.D.

*September 7, 1993*

A doctor reported that he received a vial of Chondrianas shipped by a trucking company after he sent a bank check directly to Dr. Merkl. He took the proscribed amount of Chondrianas and Life Crystals in 18 injections. His "results" were that he received one Herxheimer reaction lasting about 45-minutes and three times the feeling that he was freezing to death.

*September 8, 1993*

A doctor reported results of cultures of his Chondrianas from an independent laboratory. They contained a light growth of two organisms: *Klebsiella pneumoniae* and *Alcaligenes* species.

*September 13, 1993*

George Merkl sent a batch of clinical documents, mostly in Spanish, along with several testimonials. A friend and former Mexican agreed to translate the Spanish for me. His final report was that there was nothing of significance, from a scientific medical viewpoint in them, and felt that they were not worth translating completely.

*September 27, 1993*

Received from a donor/member a copy of a flyer from Life Crystals, Inc. describing both Life Crystals and also Chondriana. This flyer included cost of Life Crystals. In a separate sheet, names and telephone numbers of those "who can assist you with more info." were also listed. These were: Dr. Angel Villalpando, Juarez, Mexico; Dr. Ivanne Rodriguez, Juarez, Mexico; Dr. Rodolpho Sanchez, Juarez, Mexico; Dr. Alberto Martinez, Juarez, Mexico; Centro Medico Diamante, Tijuana, Mexico; George Amezcua (representative) Tijuana, Mexico; Dr. Ravi Devgan, Canada; Dr. Helena Morris, London England or Europe; Dr. Randy Winters, Switzerland or Central Europe.

*October 5, 1993*

Letter received from a physician, "I received Chondrianas from George Merkl. I looked at these under my dark field microscope and saw moving bacteria, not Chondrianas. I had several samples cultured at SmithKline Laboratory, and every one of them grew up several strains of gram negative bacteria.

"Dr. Merkl gave me several irrational explanations of why bacteria should be present in his vial when Chondriana should have eaten them all up. It does not make sense.

"I cannot say I've observed anybody actually improve by either life crystals or so-called Chondriana. I believe there may be some merit to Dr. Merkl's Life Crystals, but not so-called Chondriana. I believe it is working by a non-specific immune response to gram negative bacteria, similar to Coley's Endotoxins published in the early 1930's. However, Dr. Merkl is working with live bacteria which could be doubly dangerous. Therefore, I cannot recommend his treatment to anybody. . . . It is not my intention to discredit him. It is solely my intention to let people know the truth, albeit, as I have seen it. Unfortunately, I'm not in a position to give you the case history of anyone treated with Chondriana, suffice to say that there were a few cancer patients who received the material and one with HIV. None of them had any improvement to the best of my knowledge."

*October 14, 1993*

Faxed to George Merkl a reminder that he was going to ship this foundation a free sample of Chondrianas to be studied by Lida Mattman, Ph.D. of Wayne University.

*November 23, 1993*

Mailed George Merkl a letter reminding him that he had agreed to ship this foundation a free sample of Chondrianas to be studied by Lida Mattman, Ph.D. of Wayne University.

*February 15, 1995*

Have not received any samples of Chondriana for Lida Mattman, Ph.D. by this date.

I have no basis to judge the nature of Chondrianas, as we have never received any, and neither I, nor The Arthritis Trust of America/The Rheumatoid Disease Foundation, by this report, intend to impugn Dr. George Merkl's scientific ability or integrity. I am, however, personally disappointed at having expended thought, effort and money without having any results to show for the expenditures, and I am personally disappointed especially for Dr. Merkl's apparent unwillingness to send us a sample of Chondrianas, as promised, for proper and open scientific study.

Perry A. Chapdelaine, Sr., 7111 Sweetgum Road, Suite A, Fairview, TN 37062-9384.

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<http://www.keelynet.com>

Dan York ( danyork@lyghtforce.com ) --- Tue, 10 Aug 1999 20:19:43 -0500

George Merkl's work goes way beyond what Warren talked about in his post. And BTW, Warren is correct, in personal contact situations George does not like to be addressed using the appellation Doctor

I also have spent time in George's lab. I have observed his microscope first hand magnifying objects that could be clearly seen that were smaller than the wave length of light. George modified his lab microscope himself to achieve a level of magnification that is not supposed to be possible. I have also watched first hand through that microscope (at magnifications that are achieved on any good dark field lab microscope) when out of pure "Life Crystals" (read that as pure ATP/GTP crystals) a new life form spontaneously comes into being. These life forms



are 12 strand DNA "creatures" that George calls "cosmic bio-machines". These cosmic bio-machines have some amazing capabilities in replicating biological molecules. The bio-machines are currently being tested in several different laboratories for use to synthetically replicate/manufacture several different hard to reproduce molecules. I have both video tape and still pictures in my possession that show the bio-machines coming into being.

I also agree that George has purposely put out disinformation about his work from time to time. It has generally his reaction to problems with the FDA and other government agencies. I know a lot about George's work that I am not at liberty to discuss in an open forum like this. Whatever George asks me to keep quiet about I do. I have known about the replication capabilities of George's bio-machines for a number of years now but this is the first time I have been able to discuss them publicly. It is because George has finally decided to release them for study that I can talk about them now. I also can not speak for Dr. Merkl. Only he can do that. He has had to be very careful over the years about what he says about his work who he talks to about it for a lot of very valid reasons.

I also agree with Warren's assessment that George is a genius. He has some unusual quirks about him though that for some folks makes him very hard to get along with. On the other hand if he feels that you are a friend is a delight to be around. A quote from a professional researcher with 29 years experience in immunology that I put in contact with George recently went something like "At one point early in our conversation I was convinced he is insane and I still am not real sure about that - but now I am more than willing to give him the benefit of a doubt and will actually test his processes in the lab."

One last thing, while Warren and I share the same last name we are not related and have never met. The one thing we share in common is having spent a lot of time with George and consider him a friend. When I first met George he asked me if I knew or was related to Warren.

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<http://www.keelynet.com>

Warren York Email

Hello Jerry, Dan and all:

You know me Jerry and you know my connection with Dr. Merkl. I don't know if Dan York knows or not but he should if he knows Dr. Merkl. It seems almost like you are posting just to get a reply or response. We go a long way back and you know more than you have let on here. I find it interesting but do not feel you are getting to the point. Since I was there and saw first hand as nobody else has, I can assure you all that the man is a genius. There is something about the ATP research and genetics that I am sure has never been brought to life. His work goes way beyond just genetics and life energies. Although I was with him in the early stages of the work he never said a word about something we both saw. I can not and will not speak for Dr. Merkl himself for only he can do that. What I can do is speak out from my own view of what I saw develop. Remember I still have the original lab video's.

For what ever reasons and I feel they are security related, Dr. Merkl has backed off and even went as far as to give disinformation at the later part of his work. What George (he does not like to be called Dr.) discovered goes way beyond any genetics we now know about. I have labeled it Creation Genetics for that is what it really is. Again, I am speaking not for George but from my own knowledge of the process involved. Believe me what you or even others may think they know about the work is nothing even close to the full story. George has broken no laws but his findings being kept back from the establishment has been a crime in my view. He has discovered a diagnostic tool above all that could detect cancer and other long term threats to mankind that if addressed at early enough stages would save if not give additional years of quality life where now it is not possible. You may wonder how a nuclear physicist got involved in something that is more of the medical sciences than the physical science. That may in part be due to me. He read my OLD paper on Space/Time Continuum and called me up. He turned out to be a neighbor. It is all based from the same research you will find on my site. It is connected and is a part of the unification equation nobody ever considers. For a unification equation to be a true unification equation it must address ALL phenomena and not just gravity, Time, electromagnetic, thermodynamic etc. It must address the " Life Energy " of which nobody has even begin to know how to approach. This is why I have coined the term "Creation Genetics. The full story gets even more bizarre. Well, here is a response. I hope it is close to what you were looking for. I will back out now and sit back and watch. Remember I was there. Warren York

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## George Merkl's Life Crystal Discoveries

by

Steve Mills

Good afternoon everyone! My name is Steve Mills.

I'm going to speak a little about my background; briefly on Ron Bourgoin, MS Physics and finally on the late George Merkl, Ph.D, but first let me tell you a little bit about myself.

As a child, I would watch my father work on cars and electrical things. I also enjoyed listening to his crystal radio that he had. It was one of those small rocket ships with the pull up antenna and a clip for grounding to anything metal. It worked really well. That little radio was probably responsible more than anything for my interest in electricity.

When I was younger, I used to take balloons, as most kids do, rubbing them against my jeans and placing them on the wall. Magnets were also one of my favorite toys as well. I noticed how strongly they both stayed in place. Seeing static charge in action as well as the magnetic intrigued me. As a child I was so fascinated with these things, that I was convinced that I wanted to learn more about electronics, which also lead into my interest in physics.

Of all these experiences, I would say that the crystal radio got most of my attention. For the crystal radio was showing me that power could be extracted from most anywhere. However, to extract power in meaningful quantities without hooking into the grid, is a definite challenge. This became my personal challenge, so research has ensued since then.

In my college years, about the time I turned 20, I found a book entitled "Free Energy Sources" by Dan Davidson. Reading that book started me down a path from which I cannot turn. Indeed, anyone who has begun this type of research is compelled to see their research through unto realization.

In 1979 I received my Associates Degree in Electronics Technology. Even though I don't have a E.E.T. or MS in electrical engineering, I do believe that studying the physics manuals, and understanding the math, is about as close to classroom study as a person can get.

My plans are to continue my education, for the sake of the paper on the wall, however my zeal for learning is not for the paper, but for the truth in physics. So often, we are told that certain laws absolutely cannot under any circumstances be broken. Laws change. We make adjustments as we go, in our physics manuals, driven by discovery. These discoveries are what my speech is about today. Primarily regarding the late Dr. George Merkl, but before I get to him, let me continue with myself, and Ron Bourgoïn, who has driven my interest in this field of study, and for helping educate me on the subject of superconduction, which he has renamed "hyper-conduction".

I began my search for the "Holy Grail" of unlimited power conversion in 1979, and got really excited when I attended the 2<sup>nd</sup> International Non-Conventional Energy Symposium, in 1983. There I heard many speakers on the subject of "Free Energy" and "Zero Point" energy, or "Phi Dot".

Among those speaking was Ron Bourgoïn, on the subject of superconducting filaments. Ron was good enough to send me a copy of his patent 19 years ago. I have read that patent several times and have intended to replicate his process for room temperature hyper conduction. Due to limited funds and lack of lab equipment, that never has happened. From what I understand, it is a tedious yet simple process, requiring patience.

After being out of contact for 19 years, I located Ron's newest number and contacted him. He remembered me after talking about the symposium in Atlanta. After a brief discussion, Ron clarified hyperconduction in his superconduction patent, US4,325,795. He coined the term "Hyper conduction," due to issues relating to the definition of super conduction and room temperature conduction thresholds of bismuth filaments.

After college, I got involved with several ventures, which gave me more exposure to new things. In the course of these learning experiences, I was to discover a relative neglected field of QED, and that's the subject of Homeopathy. To understand the dynamics involved in Homeopathic Technology challenges ones ability in abstract thought.

Homeopathy is founded upon "the law of similars". Any material can be administered as a remedy to certain illnesses in humans by bolstering their immune response. This refers to dilutions of chemical, mineral, herbal, and just about any type of material that can be used for dilutions in what are defined as "potencies".

Potencies are a progression of dilution and succussion (shaking) of these materials, to the point of Avogrado's formula for molecular limits. That limit is defined in a dilution as having no more original molecules of that substance from which the dilution is made.

What is left behind are the amplified energy patterns, a complex 3D wave function of that material. Amplified by succussing that dilution at every step of the diluting process, like striking a tuning fork, turning up the volume or amplitude.

I began studying homeopathy in 1986, and later that same year had the fortune to attend the 1986 International Tesla Conference in Colorado Springs, where I learned firsthand about Scalar waves and the difference between them and Transverse EM waves. By removing the molecules of the original material, it allows elbow room for vibrations of that same material to be amplified. That amplified wave function is carried inwardly at quantum electrodynamic levels. That involves phonon and photon interaction for carrying the information to the next level of each dilution interval, amplifying energy pattern wave functions through phonon wave interaction, possibly scalar interactions.

In times past, the classical homeopath has had trouble with accurately prescribing a remedy for the alleviation of illnesses within the patient. This problem was solved in the late 40's to early 50's, through research and development of German doctors. The answer came to the late Dr. Reinhold Voll, who discovered there is an electrical circulation throughout the body, akin to the nervous system, and directly connected to specific organs within the body. This he named EAV. Still, the process was lacking something. The computer.

Now along comes the computer age and digitizing these phonon and photon interactions. With the storage capacity of computers, now these interactions can be digitally stored by the thousands. But the theory goes way beyond mere electronics.

Homeopathic information transfer and storage involves comprehending electrical information transfer in ways that typical college manuals do not address. It involves virtual photons and the emission of phonon waves from electron to electron, as well as from photon to photon.

These are vibrations of the electrons and photons, referred to as "information," which are passed along to nearby electrons and photons. This is what is called holographic communication. Light, it seems, stores information. Like a hologram, but stored within single photons and single electrons themselves. Something that QED has been discovering through research.

This transfer of information is referred to as "Hololinguistics". It is the holographic information communicated from the patient through the machine and from the Homeopathic remedy through the machine to the patient. When the two energy patterns are a match, the device shows a balance. This single phenomenon has had my attention for 16 years now, and has been one of my side studies when I have the time.

I have a theory about energy conversion that is based upon this hololinguistic technology, however I am not at liberty to discuss that here at this time. This will be a subject for discussion in the future, at perhaps another seminar such as the Inventor's Weekend. Hopefully then I will have something to demonstrate, based upon that technology.

There is now ongoing research in the field of BioPhotons, which are laser like photon emissions on such a low level not detectable by human eyes. A coherent light emission from within the DNA molecules of cells. From what I have learned from Dr. Merkl and from Ron Bourgoïn, is that this is the superfluidity of photon and electron flow within our cells at the DNA level and lower. This could explain much about Homeopathic Technology and information transfer.

These phenomena are showing me that there is yet bigger clues to tapping into the cosmos for unlimited power conversion. It was telling me that matter has to be understood in a whole new manner of reasoning. It challenges the thought processes.

With the understanding of Hololinguistics in mind, I pressed on with my own research and consultations. First up of course was Ron Bourgoïn, regarding hyperconduction, and later Cosmic Geometry as discovered by Dr. Merkl.

These hyper-conductive paths are interesting to say the least. Research scientists have been looking at superconductivity as an application towards discovering sources of unlimited power, but neglect to realize the fact that hyperconductivity, a phenomenon not very well understood as yet, is actually a noteworthy field of study.

Hyperconduction is akin to superconduction, but does not require cold transitions of metals for Cooper electron pairing. Instead it is a threshold at ambient room temperature, at which electrons in thin filaments and angstrom sized domains, will pair and hyper conduct, with a breakover threshold for conduction, similar to superconduction.

The two phases are similar, however super conduction and super fluidity are a "cold state" pairing phase of conduction and flow, where hyperconduction is an ambient temperature threshold event phenomenon. Dr. Merkl, in his Life Crystal discoveries, claimed to have discovered super fluidity within molecular proteins by a specific process of geometric alignment. He adamantly claimed using this process and never considered it as being otherwise.

If instead he discovered "natural hyper conduction," he certainly discovered a phenomenon in nature that certainly lowers the threshold of hyperconduction by many magnitudes.

It appears that as you apply more voltage to Ron's bismuth filaments, that a small current flows until you reach the breakover threshold, at around 200 volts, typically, depending on the construct of the filaments. At breakover, the conduction transitions immediately.

With little more voltage applied, the voltage drop on the bismuth filament strands drops to less than  $1/10^{\text{th}}$  of a volt, and the current shoots upward to the ampere limits of the power supply. Incidentally, Ron is now working with Carbon filaments, and the filaments pass 3 1/3 amp through a filament less than the diameter of human hair.

I have an excerpt from the 1983 2<sup>nd</sup> International Non-Conventional Energy Symposium, Ron delivered his speech in Atlanta Georgia that year. For those who may be interested, or unaware of Ron Bourgoin, that will be available afterward. If need be we can make copies if we do not have enough everyone.

Whether or not superconduction or hyperconduction is the property involved, it is with my own personal interest that I now speak to you about George Merkl and his Life Crystal Discoveries.

George Merkl was a maverick within his lab. He often thought "outside the box". Even his methods in the lab were "outside of the box." It has been stated that George was light years ahead in his way of thinking. That kind of thinking is what led his intuition in the directions he went, which finally led him to the rediscovery of the Sumerian Technology from 7000 years ago. We were left with a legacy of technology it seems.

The Sumerians were a culture, which had scientists and was of an advanced nature. They certainly had the potential to view subatomic particles, which meant they had devices and instrumentation for accomplishing this feat. They knew how to scan molecules and atoms by "string photon scanning," a term coined by George Merkl. String photon scanning is the viewing of single file non-transverse-wave photons.

One of the discoveries, which led him into this area of rediscovery, was that Life Crystals could align matter into geometrical order. Lining it up into an organized matrix. These things he saw under his microscope, as he experimented with Penta-Carbons compounds.

George had a very good understanding of how electrons and protons can be slowed down, which drastically alters the physical properties of matter. This he could do with small scale equipment. Not big Tokamac reactors and things of that nature, but room temperature small scale alterations. The results were the product of his maverick ways in the lab.

In some of our conversations, George would tell me of what he saw, and that upon first discovering them, caused him to get up from his chair and go and pour a drink. That first discovery he saw was this Bio-Machine you see on page one in the handouts. Please note the shapes of the biomachines are individual and of different function for each.

George Merkl discovered how to slow electron spins and proton spins within molecules. This drastically altered physical properties within matter, and probably explains how the Hutchison Effect melts or seems to melt metals at room temperature. The slowing of electrons and protons by electric or magnetic inductions. George found that geometric displacement in specific domains of around 12 to 20 Angstroms opened up a pathway of wave spin interactions. It is these interactions that he found tapped the cosmos for unlimited energy.

I have now been seeing how Homeopathic Technology and Ron Bourgoin's discoveries all matched up with what George Merkl had discovered. I could now see how it all fit together.

George Merkl knew of Orgone Energy and Reich. He knew of the works of Tesla and of the UFO researches of the Germans in WW2. George discovered what the Orgone wave is and the Life Wave. There are 5 elements as he saw them. Energy, Matter, Life, Light and Gravity. All these emanated from geometrical progressions of the gravitons.

The whole physics he rediscovered is based upon graviton interactions in geometrical progression and specific patterns to form Energy, Life, Light, Matter and Gravity.

He also stated that the geometry is spherical in nature. It has no limits either outwardly or inwardly. This pattern is on all levels infinitely positive and negative. Infinitely inward and infinitely outwardly in geometry. The spin wave interactions of these waves acts like Cooper Pairs and seems to be the governing factor behind why electrons, photons, gravitons, and the like, will superconduct or hyperconduct.

The Sumerians were scientists and laymen just as we were. They had discovered the underlying physics of our universe 7000 years ago. Please note some of the pictograms that have been discovered. Many of these seals, pictograms, and artifacts were on loan to George, once the museums realized what he had rediscovered. He managed to decipher the Sumerian seals and language.

The technology was recorded in such a way that anyone with understanding could comprehend it in as simple an explanation as possible. The only caveat was that such a race to rediscover such writings and comprehend them would require that race to be at a technological level, advanced enough to understand and actually discover a method by which to "view" for themselves the things the Sumerians were able to see. It took someone with insight and a knowledge of alternative processes and lab methods to solve this dilemma.

That person was the late Dr. George Merkl. The first to see these biomachines and the first to understand what the ancient pictograms were. It was these discoveries that George found would enable us to progress onwards to the next phase of technological advancement. We have yet to discover the full potentials of this final phase of technology.

George Merkl found that under specific conditions, not too complicated, but just specific processes in the right order, could open these domains, and slow the spin of the electrons. Even reversing the spin of the protons within the material, which caused the formation of newly

undiscovered properties of matter to reveal themselves. Some of these effects were light of many colors emitting from within the material; Gravitational levitation; Electron emission, and **protein cellular resurrection...** Let me clarify that further.

Four years ago, George revealed to me that he was able to take dried proteins, 4 years sitting open to the environment unprotected, and placed them into his Sumerian Crystal solution. This restored the biological functions of those tissues at near 70% efficiency to its original condition within just minutes. Not days.

Now, I know that raises some eyebrows here at this statement, but I am quite certain that a man with over 100 patents worldwide, who gave standing room only speeches in presence of attending professionals, having acquired recognition in the Dept of Energy, having obtained a doctorate degree in physics, and having continued his research into biology, was not about to make bold statements, come up with some crackpot theory or drug induced hallucination of the mind, in order to gain a dead end recognition of his own. He did not want that at all. He never received a Nobel Prize or anything like it. That's the main reason why you don't know very much about George Merkl today.

George had rediscovered the Orgone Life Wave function. He knew the process and how to generate it. He knew how to reinsert the life energy wave into organic matter. But he didn't stop there. He went on to discover, beyond the Sumerian Seals, that life and energy are not so separate entities as one may think.

He was able to photograph the actual life energy of a person. I saw these for myself, and are not available at this time. The photographs look something like the soul energy of a person. Light emanating from the brain and limbs of the person photographed. He discovered a superluminal connection from the brain to the hands. He had also discovered that this universe is also alive. The inanimate matter has within itself the carrier of life energy. He discovered that light carries with it the pattern of life and the pattern of creation. This pattern emanates from levels much lower than even the gravitons themselves. Holographic information and hyper communication. Perhaps another branch of Hololinguistics.

Now I know that is a very bold statement, considering how many here think and how so much is taught in schools today in our text books regarding life processes. Let me remind you that George was a maverick in his methods. He experimented outside the box.

George had discovered that energy, matter, life, and light all begin with the spin interactions of graviton waves. Specifically in a clustered arrangement. This all boiled down to two basic forces. The Negative force and the Positive force. The outward expanding action and the inward imploding action. Both are the balance of each other on multiple levels.

George explained the expansion of the universe has two parts to it. One governs the other. As the universe expands, the inward atomic imploding force regulates the outward deploding force. This is a function of the matter wave. The converging of spinning wave packets towards a central point, creating mass centers. Hence matter waves. Still only energy but in geometric formations or clusters in geometric order. The two are interactive and it is the inward imploding force that is the secret to tapping the cosmos for energy. Unlimited amounts of it.

Folks, I cannot begin to paint a picture for you that accurately describes the infinite levels of this geometry, but if you really wish to gain a good grasp of it, I can recommend two VERY good books, both written by the late Dr. Merkl. These two books are entitled "Sumer Tech", and "Whispers of the Cosmos"

In these two books there are BIG clues as to how this technology is accomplished. Anyone with good lab "know-how" should be able to, with a little trial and error, be able to figure out the procedures George was doing in his lab.

I would highly recommend that you read them both, starting with Sumer Tech, then Whispers of the Cosmos. You will not find this information anywhere else on Terra Firma. They are challenging in thought and provoke a paradigm in thought and reason. They inspire applications of energy, magnetism, light, and the force of life.

Some of the obvious possibilities that can be learned from understanding this technology is Unlimited power on demand. Free, for only the cost of the machinery to convert it. I have many plans with this technology. Many applications of the geometry, into machinery. This could ultimately be the holy grail for the individual that has the tenacity to build it.

If you are to gain an understanding of energy on unlimited levels, a person should start by researching thin filaments as described by Ron Bourgoyn and following with reading the books written by George Merkl.

What is your dream? What is your accomplishment going to be in life? I am waiting to find mine as yet, but I know where I am headed with George Merkl as my guide. He may no longer be with us but he leaves us with a legacy that will forever be the opening of Pandora's box of secrets.

For those who are interested, there will be a 38 minute presentation on video of some biomachines, verbal notes and observations, according to George. These I will be presenting to you at 8:15. I have gone to extensive lengths preparing this video and gaining its approval from Maria Merkl, George's surviving wife. She has been kind enough to allow this group the opportunity to have a taste of public view of some of his research. I would highly recommend it for those who are interested in knowing more about the Sumerian Technology.

I understand there is a social following immediately afterward. I don't expect to see everyone back here following the social, however I would like to assert that not many people get to see what is on this video. I hope to see most of you there.

I want to thank everyone here for being present. I hope that you followed much of what I was saying. I will be available for questions during the social and I don't mind answering any questions.

Thank you.

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### Excerpt from:

#### *Pleomorphism*

by

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This is what life is, physical life, and where that which 'animates' this physical life, comes from. The **protit** of Enderlein has been called the **Colloid of Life**. These protits are particles that measure .01 to .0001 microns in diameter (that is about four hundred thousandths to four millionths of an inch.) The average red blood cell on the other hand is about 7 microns in diameter.

There is some point in space and time where these protits (the smallest of biological living particles in the physical realm, not the cell) were begotten from something or 'somewhere' else. Even if "the cell" were the smallest living thing then where did the first cells come from?

That we are, "...made in the Image of God", what does this mean? Does it mean that we are made in the image of an old Man with a white beard? That seems rather silly.

Creationism implies a Creator, God, which rather by definition created everything, not just us. Being Everything, including us, makes us part of All That, including everything. Assuming everything is part of everything and that all this was created by a guiding intelligence (which is now being proven by quantum physics as described below), then this Guiding Intelligence evolves us and everything of which we are a part through time to a destination. Evolution by definition implies a destination. What are we evolving toward? Could it be the consciousness of this Guiding Intelligence and everything It created?

If so, within the matrix of this time and space would be the "Image of God" of which we and everything are. The implications of this being profound, rather obviate the so called necessity of us and everything being created by accident *or* a Creator and bring us literally as will be seen below, into another dimension that *includes* these two apparently irreconcilable pairs of opposites by showing us that really, these are not opposites but *compliments* as, like black and white, you can not have one without the other.

The following two Latin statements constitute the *Cell Theory of Biology* which is the most basic principle of Biology and which is on the first page of any Biology text book.

***Omne vivum ex vivo - All life comes from previous life.***

***Omnis cellula e cellula - Every cell arises from subdivision of preexisting cells.***

The above are dictums from the church, not science. The purpose of this chapter will be to unite the apparently divergent view of the church (Creationism) and science (evolution).

The first individual to actually catch a glimpse of the occurrence of *cells apparently coming out of nowhere* and therefore not requiring subdivision of preexisting cells was Anton Leeuwenhoek who lived in the 17th century. He had ground glass to create the first microscope. In observing some rainwater he collected, he made note that there were tiny creatures moving about. Wondering where they came from, he did an experiment. He collected clean fresh rainwater and sealed it in pipettes. At first, nothing was in the water. Hours and a few days later, still nothing was in the water. However, on the fourth day, all of a sudden, little tiny creatures (protits) appeared. Where did they come from? Was it *spontaneous generation*?

"All organisms, and all of the cells that constitute them, are believed to have descended from a common ancestor cell through *evolution by natural selection*." <sup>1</sup>

This is still the dictum of modern biology and the argument for Darwinian evolution while compelling, is not complete as will be explained below.

Leeuwenhoek took his research to Robert Boyle, the father of chemistry, as we know it, and to Sir Isaac Newton who wrote many of the principles of physics. They did not believe that life could beget from light or in their way of thinking, from nothing. This was a time when the church played a big role in every major decision that was made. To have life you must have procreation, a mother-father union. Since there was

no mother or father that created Leeuwenhoek's teeny creatures, his observations were surely flawed, and they were dismissed. What could not be dismissed however was the observation of a newly discovered microscopic world. It was a foundation for developing the beginning ideas of the germ theory.

Another old example of "spontaneous generation", from the writings of Dr. Kurt Donsbach, is an experiment that he calls, "making protozoa". Anyone can do this experiment with the simplest of microscopes. Leeuwenhoek did it in 1600 B.C.! That these scientists from the past did these things with the equipments they had only ascribes to their genius. These people were not stupid.

The protozoa are among the most primitive and simplest life forms. Again, concerning creatures that just 'appear' in pond water this time Dr. Kurt Donsbach continues, "...after a few days, the little mass begins to move with a rhythmic pulsing motion. Eventually the pulsing motion becomes more pronounced and the glob appears to gather more energy. Soon it breaks away from the grassy shaft and is a living mass, classified in biology texts as protozoa. From this point, it can differentiate itself and other microorganisms appear."

The creatures on the left are all protozoa that just appear in pond water. Even if the *germs that create these beings* were in the pond water as maintained by Louis Pasteur and modern medicine, how could such incredible life forms all just *be there*? *Fascinating* isn't it?

Just what are these protozoal masses pulsing with, *life* obviously, extremely intelligent *Life*? Could it be what the vitalists call the Life Force, or *Prana*, *Chi*, *Eck...*the *Holy Spirit*, the higher vibrational essence of spirit - the breath of God?

The protozoa on the left are hairy single-cell animals that show amazing diversity of form and behavior. The top picture shows *Didinium*, a ciliated (hairy) protozoan with two circumferential rings of motile cilia (hairs) and a snout like protuberance at its head, with which it captures its prey. The bottom micrograph shows this living being engulfing another protozoan. Before it consumes its prey, it releases numerous small paralyzing darts from its snout region, paralyzing its prey.

We all know, *by accident*? Really, such ideas are no longer tenable.

Be that at it may, the germ theory fails to explain then, and fails to explain to this day, the question; from where exactly do germs come? Where is the mother-father microbe? In any textbook of science, medicine or biology, there is no explanation. When the germ theory took hold in the early years of biological science, the religious dogma of the time shaped the scientists thoughts who formulated the theories. Since they had no concrete evidence to answer the question, they left it unanswered. Moreover, it remains unanswered today. This is where a new paradigm unfolds.

Germs, and the protits they come from, are physical life forms, Life forms that have evolved from something.

"Since that something is not physically measurable, then it must be something that is on a higher vibrational, quantum physical or spiritual level." <sup>2</sup>

This new paradigm is Wholistic, pleomorphic and ever changing. It is **Holographic**. *Every speck of Life from the protit on up is a holographic projection of the same Power*, from dimensions beyond the three dimensional existence we find our selves in. Life is a quality of that Power and is awesome beyond belief. What follows is not about what that Power is; it is about how It gets into this three dimensional, material world.

An ant walking around on a flat, two-dimensional piece of paper would live in a two-dimensional existence and would see a three-dimensional cube projected onto its piece of paper as to the left. The ant would not understand this 'three-dimensional' structure as we cannot understand the four-dimensional super cube (generated mathematically) and projected onto our three-dimensional existence as to the right below.

About everyone nowadays has seen holograms, on television, in *Disneyland*. They are the three dimensional light forms that you can walk around, pass your hand through. A nature of holograms is that you can take any little piece of that light form (they are on special photographic plates actually) and blow it up, enlarge it-and you get the whole image! Another characteristic of a hologram is that you can make a holographic image as large as you want to, as vast as our universe or smaller than an atom.

"We are made in the image of God." What does that mean? I am sure it does not mean that we are created as an old man with a white beard. It means that we are projected into this time and space (three-dimensions) *as that image* and that if you blew any little piece (cell) of us up, you would get the whole existence. This concept is what this chapter is about and will be described in detail below. Science, quantum physics, has come face to face with God.

Getting back to the point here, as the point is that everything is One, large or small, and as we are talking about cells and things living that are *smaller* than cells, i.e. protits then what are protits? **Protits** are, "...composed of tiny, pyramid shaped crystals of ATP, adenosine triphosphate, the chemical fuel that powers the machines of our bodies. ATP is the energy molecule of the body, the muscles 'burn' ATP when they contract. These crystals are what spawned in the oceans of all worlds to transduce Life down from the bi-directional electromagnetic wave structure in the scalar potential of vacuum". <sup>3</sup>

This electromagnetic potential of vacuum is, **"The ZERO POINT ENERGY, the energy of nothingness, of vacuum, of chaos. This is the energy that holds all this corporeal existence together. It is Free Energy and it is infinite."** <sup>4</sup>

There are five energies that create our existence; gravity came first, then magnetism and electricity, then Light and then Life. The energy of Life composes a greater percentage of our universe than all the other energies making up our universe. Live is everywhere, always. When a baby is born, as it is first birthed and comes out of the womb, all is dark and apprehensive...is the baby OK, the baby appears asleep. Then, the infant takes its first breath, *comes alive*, the room fills with Light! It is the same when someone dies, something leaves the room and all is dark, the remaining corpse is more than asleep.

This is heavy stuff. As to gravitation, in the last two chapters of Misner, Thorne, and Wheeler's *Gravitation* is the statement that in quantum mechanics there existed an all-pervading energy imbedded in the fabric of space consisting of fluctuations of electricity. The term for this energy, the **zero-point energy**, came from the equations of quantum mechanics in the 1930s.

"Zero-point refers to the fact that these fluctuations persist even at zero degrees Kelvin (the point, temperature at which all molecular motion stops) and therefore are not thermal in nature". <sup>5</sup>

Thermal heat has nothing to do with these fluctuations of electricity as at this temperature of zero degrees Kelvin, by definition, there is no heat.

"In the 1930s Dirac derived the idea that space, vacuum consisted of a virtual sea of fluctuating energy that could manifest electron-positron pairs." <sup>6</sup>

"The random electrodynamics theory of Boyer mathematically describes how the zero-point energy oscillates in its interaction with matter. In this theory, Heisenberg's Uncertainty Principle allows the Zero-point energy to produce Brownian motion, relevant here as the motion of the protit has been ascribed to Brownian motion." <sup>7</sup>

"Wheeler's *Geometrodynamics* showed that the energy density of this 'nothingness', of space, vacuum, was enormous,  $10^{93}$  grams/cm<sup>2</sup>." <sup>8</sup>

The energy density of one cubic centimeter, 1 cm<sup>3</sup>, of matter, contains energy on the order of  $10^{16}$  grams/cm<sup>2</sup>, that from Einstein's equation,  $E = MC^2$ . According to this calculation, there are 77 orders of magnitude more energy contained in 1 cm<sup>3</sup> of vacuum than in 1 cm<sup>3</sup> of matter. That means there is one, with 77 zeros after it, times more energy in vacuum than in matter. *It takes more energy to hold matter together than the amount of energy contained in the matter itself.* That only makes sense.

"Quantum mechanics showed that this energy was constantly interacting with matter and the elementary particles in what is called **vacuum polarization**." <sup>9</sup>

"Quantum electrodynamics shows all particles are intertwined in a vacuum polarization interaction with the zero-point potential and shows how the interaction yields the mass of an elementary particle." <sup>10</sup>

"A modern view is that elementary particles are a coherence in the zero-point energy and this view can be supported by system self-organization theories, theories of chaos." <sup>11</sup>

Concerning the Power of Creation that we have been talking about, since 1991 some very startling discoveries have been made in the fields of astrophysics and cosmology. According to the *Big Bang Theory* the universe was, at the moment of creation, all clumped together in one 'place'. From there it exploded outward and has been expanding ever since. A basic premise of this idea was though, that one day all the gravity of everything in the universe would start to pull all the expanding stars back together. The expansion would stop and then the universe would start to contract, ending in a giant crunch.

Discoveries in this decade though have shown that the above idea is not happening. In fact, the ***expansion of the universe is actually accelerating with time!*** In the beginning the universe was expanding much slower than now so that distant supernovae (stars that explode) appear to be 25% fainter than expected, assuming that the universe had been expanding at the same rate since the beginning.

"...an explanation of the unexpected faintness of distant supernovae is that they are *farther away* than their red shifts suggest." <sup>12</sup>

Red shifts show how far away stars are and if they are further away than expected they must be expanding away faster than expected. What strange force is pushing the universe apart?

**THIS IS THE SAME 'POWER' OR FORCE THAT ANIMATES THE PROTIT, WHICH KEEPS IT ALIVE FOREVER.**

"According to Einstein's theory, the expansion (of the universe) can speed up if an exotic form of energy fills empty space everywhere. This strange "vacuum energy" is embodied in Einstein's equations as the so-called **cosmological constant**. Unlike ordinary forms of matter and energy the vacuum energy adds gravity that is repulsive (**antigravity**) and can drive the universe apart at ever increasing speeds (see "Cosmological Antigravity," by Lawrence M. Krauss, Ibid. pg. 52). The energy associated with this energy does not depend on position or time (or temperature as above), hence the name "**cosmological constant**". The force caused by the constant operates even in the complete absence of matter or radiation (vacuum energy). Therefore, its source must be a curious energy that resides in empty space, even the space in front of our noses. The cosmological constant, like the *ether* of old, endows the void with an almost metaphysical aura." <sup>13</sup>

**IT IS EVERYWHERE/ALWAYS.**

"Physicists Paul A. M. Dirac and later Richard Feynman, Julian S. Schwinger and Shinichiro Tomonaga showed that empty space was more complicated than anyone had previously imagined. Elementary particles, it turned out, can spontaneously pop out of nothingness, and disappear again (see "Exploiting Zero-Point Energy," by Philip Yam; SCIENTIFIC AMERICAN, December 1997). The aggregate energy represented by these 'virtual' particles, like other forms of energy, could exert a gravitational force, which could be either attractive or repulsive depending on physical principles that are not yet understood." <sup>14</sup>

Quantum theory predicts a whole spectrum of virtual particles spanning every possible wavelength. Calculating it this way, by adding up all the energy of these virtual particles, the total energy comes out to be infinite or at least, the calculated energy is roughly 120 orders of magnitude larger than the energy contained in all the matter in the universe. That means you take all the energy contained in all the matter in the universe and add 120 zeros to it to get the total vacuum energy! (Eighty-one orders difference as above or one hundred and twenty one as here are both awfully big.) We live in a sea of infinite energy and the new part of this is that the gravity associated with this energy is repulsive. *It is antigravity.*

It is antigravity. It is not heavy, it is *Light*.

The above picture <sup>15</sup> is a black hole viewed from the outside. In standard big-bang thinking, the matter that will form at the bottom of this black hole is preexisting, crushed into a singularity or bubbling in a super hot stew. In inflation theory, all there was before the beginning was a false vacuum, a transparent region crackling with quantum-mechanical promise. Within the false vacuum was the potential for vast amounts of 'virtual' particles, subatomic units that sometimes pop out of nowhere. Could this not be the way everything is created? More than likely, hydrogen is popping into existence all the time in the form of electron/positron pairs. The positron is the antiparticle of the electron. Perhaps too, protits 'pop' into existence just like the drop of the daughter universe pictured above falling out of another dimension from the bottom of a black hole.

This sea of infinite energy creates life too which is an energy. It must be, death being the lack of this energy. As this sea of energy is infinite and is pushing the universe apart at an ever-increasing rate, always creating new matter and energy to fill the resulting central void, everything inanimate and *animate* must come from it.

"Recent experiments have shown that the *brightness* of the zero-point energy is independent of the existence of reflectors and absorbers. This



shows that zero-point energy does not arise from an electromagnetic propagation in our three dimensional space." <sup>15</sup>

This energy comes from and returns to the fourth dimension and beyond, all of this mathematically known and proven with science's giant cyclotrons and computers. These machines are proving the existence of *Spirit* is it not, other dimensions, the undeniably indefinable.

Computers can 'think' in 12 dimensions where we cannot and by being able to do this, all anomalies in Einstein's equations were resolved. These anomalies were a big problem for Einstein as many of his equations ended with a final answer of zero or infinity, which was the anomaly. These were not satisfactory answers but with the advent of computers that could 'think' in twelve dimensions, this problem was solved. According to current science, we live in a twelve-dimensional existence.

"This zero-point energy pops into this third dimension in the form of the electron-positron pairs or tachyons (super fast subatomic particles) which sustain and nourish us." <sup>16</sup>

"These are "scalar waves with opposite oscillation and spin that braid into one single photon, quantum. The photon is an intermediate between particle matter and Free Energy and then pairs of photons braid into the electron and electrons braid into protons and neutrons." <sup>17</sup>

The ATP crystals in the protits *transduce, transmute* this tachyon energy down into the machinery of the cell itself, and they live.

This starts out as LIGHT:

"Luminous quanta of divine intelligence thrilling out in infinite vibrations of accordance breaking joyously into the rhythm of greater and higher waves of illumination intensifying towards the growing light of a self-existent Knowledge spontaneously awaking to Itself as the SUN OF TRUTH receiving the world from the murky depth of an obscure physical bondage into the enlightened height of spiritual freedom by the transforming radiation of ITS glorious Arc-Light." <sup>18</sup>

Therefore, matter forms, from out of "no where", and under the direction of divine *intelligence* it organizes. This appears to be contrary to the second law of thermodynamics, which states that everything is falling apart.

Yet, if the universe is expanding at an accelerating rate due to the continuous formation of 'new' matter then perhaps even this second law must be questioned. Life is created. It is happening all the time and is continuous. Life is everywhere. It is pushing the universe apart at a faster and faster rate. Suns form, all the elements, all the time. Protits form in the oceans of our bodies all the time, new life. Acid Base controls it. In new worlds the primordial soup of those oceans form and as "Luminous quanta of divine intelligence" interact with those oceans, they begin to live. This is a nature of Life too isn't it, to continually birth itself and to never die? Remember,

"Nothing is the prey of death; on the contrary, experience daily proves that everything is the prey of life, that is to say, of what can be nourished and what can be consumed." <sup>19</sup>

In this world protits form, are created continuously. In the body or in oceans,

"The hydroxyl (OH<sup>-</sup> ions from water) groups and the protons fill the inner membrane matrix of the protit as it is formed so as to provide an electrochemical gradient that allows the storage of energy. It is like a drop of water forms with an electrically charged membrane around it, a condenser. This electrical charge creates a magnetic field with the net effect that the condenser membrane of the protit is being charged with glowing energy. This energy can be used in several ways. It is first stacked up as an ATP reservoir of energy in the chlorophyll of plants and then as the mitochondria in animals, it can be used to induce birth (reproduction) and it is used to disassemble the genetic material of anything that tries to destroy it (the beginning of our immune systems). These protits in fact enzymatically devour bacteria, viruses or any chemical that may jeopardize the genetic process of mammals, they differentiate into the cells, lymphocytes etc., that do this job." <sup>20</sup>

This energy is *transmuting* all the pollutants in our bodies, is transmuting our bodies, from the inside out.

Therefore, these 'created' protits evolve too; guided by an 'internal' Intelligence or Will...they become whatever they need to turn into. These original protits differentiate first into chlorophyll (the ATP energy source for plants) and then into mitochondria (the ATP energy source for mammals) and then into cells and then,

"...in a foreword direction, they synthesize all living organs, things. These original protits are stored in the tails of sperm (which are a separate life form from the heads of sperm) and from them all the old things our bodies ever were can be cultured, by genetically growing them in the reverse direction". <sup>21</sup>

Isn't that strange?

"In reverse synthesis one can grow old and even meet our million old ancestors." <sup>22</sup>

Blue green algae and chlorophyll have been grown from shark testes by reverse synthesis. In this manner we can and will recreate or re-member (put back together) our old immune systems, which will reconstruct and start over if you will, that which we lost by growing up in the polluted world we live in. Old organs, our embryonic organs can and will be regenerated from these primordial protits. You have to pay taxes and die, who says so?

Remember, *in utero*, while an embryo, our bodies were first one-celled things like algae, amoebas, and then fishes, then frogs, then animal. *Ontogeny recapitulates Phylogeny*, the **Biogenic Law of Genetics**. Our embryonic development (ontogeny) travels through the ancestral forms (phenotypes), as if to say millions of years ago our heritage was evolving in salt water. In the womb the fetus recapitulates, goes through, all the past forms it ever had.

Gaston Naessens has made the following observations concerning the **Composition of the Somatid**: "The somatid may be the link between the biological sciences and the physical sciences." <sup>23</sup>

This study of Life is fun, exciting, and not dead like medical school. Without the protit/somatid life is atheistic, dust-to-dust. With the protit/somatid, as a transducer of energy from "beyond"... the *ying* and *yang* of religion and science become the complements they are, not opposites.

Scientists have broken matter down into smaller and smaller pieces, the atom, the proton, neutron, electron, then quarks, antiparticles, etc. In

today's giant cyclotrons, what they have found though is that as they break these 'pieces' of matter down further and further, the pieces just finally disappear, "pop in and out of existence" as stated above. *The particles enter the fourth dimension*, just as they are supposed to, according to these now corrected twelve dimensional formulas of Einstein.

What is even more fascinating is that when these particles disappear and get into this fourth dimension, which is a supra physical, *mental dimension*, the scientists find that whether these particles even exist in this dimension or not *is controlled by the thought* of the observers, by the thought of the scientists themselves.

Quantum theory demands the inclusion of the psyche since the observer effect of any phenomenon will instantaneously alter its state. Einstein, Rosen, and Podolsky postulated this and supported it with flawless mathematical proof; it was called the ERP Effect (Einstein, Rosen, and Podolsky).

The process of observing and being observed become one. The process of observing does not just effect the results of the experiment, as per the Heisenberg Uncertainty Principle, it *determines* them. *The observer and that observed have become One. This is Wholistic.*

To reiterate then, concerning the protit, chondriana, microzyma, somatid (there isn't even a word in the English language as was said before): we were told in medical school that these were chylomicrons (fat globules), artifacts (dirt) or that they just weren't there.

My wife and I went to a conference where material was presented, in the best of modern scientific terminology (therefore making what was said 'true'), research done in Germany in 2002 that had 'proven' that protits/somatids /chondrianas did not contain protein or DNA and that therefore they could not be alive.

From this research came the deduction that these particles (protits/somatids/chondrianas and all of their higher and larger forms) were only the breakdown products of cellular degeneration. This is bandied about in current conversation as the truth. The research has not been duplicated and besides, so what if the protits et al does not contain DNA or protein (DNA and protein are considered by modern science to be necessary factors for something to be alive). What is alive? What does that word mean, really?

Taking care of terminally ill cancer patients, one sees under the dark field microscope many microbial forms that are called, in increasing order of pathogenicity: free chondrits, mychits, microthrombocytes, thecits and finally bacterial rods themselves depending on the acid base of the milieu et al. (See Chapter II, Pleomorphism, as described previously for a description of these forms).

The thecits, as an example of the above forms, are long worm like critters with heads looking like sperms that swim around everywhere. Are you going to tell me that such critters are just break down products of cells? They are microorganisms and they are alive.

The question then remains, what is the protit/chondriana? Where does DNA come into the picture?

Enderlein himself said they were albumin globulin particles; this is nothing new (DNA was unknown when Enderlein started his research).

What else besides protein or DNA (or globulin and albumin as per Enderlein) could they contain?

### A possible Solution ---

The following is from an e-book called *Manna, Gold of the Gods* written by Henry Kroll in 2002. It can be found in its entirety at <http://www.mittymax.com, Mana, Gold of the Gods>.<sup>23</sup> It is called document 0081 and can be purchased for \$4.00.

This e-book contains more of the research done by the late **George Merkl**, PhD, who was quoted extensively above. To reiterate; **George Merkl**, PhD maintained that chondrianas/protits contained plates of ATP which act as transducers of energy from the fourth dimension into LIFE!

**George Merkl, PhD was also a Sumerian scholar, too.** Most of what he talks about are from Sumerian clay tablets that are more than 6000 years old! Sumerian mathematics...none of this is new. To emphasize the significance (to me anyway) of this recently discovered fact I will use the words of **George Merkl** regarding this.

"As I was enjoying exploring this extraordinary world, opened up by high-magnification and high-resolution microscopy, I recalled my past investigations of the Sumerians. This renewed my interest in the ancient civilization of the Sumerians, some 6000 years ago. To my amazement, I found many strange forms of living proteins and genetic components in my cultures to be identical to shapes and forms seen on ancient Sumerian glyphs and seals. These seals began to take on a new meaning, indicating, in my view, that the Sumerians possessed a science which was advanced far beyond our own in several important aspects, some of which I have realized in my lab.

"I feel strongly that what I have discovered is in effect a "rediscovery of principles" known to the Sumerians and other civilizations. I felt that the nature of these discoveries and what can be done with them is important, so I began to translate the Sumerian picture writings and the pre Inca-OLMEC; statue sculpture writings using String Photon Scanning. In a word, what I have discovered are proteins and molecules in Superlattice Order.

"While most people associate superconductivity with only the free flow of electrons, I have discovered that it also permits the free flow of Life Energy, the Life Force, Orgone Energy, Free Energy, and the Superlattice Order. I observed that this superconductivity occurs when electrons are arranged in what I call a "Superlattice", a circle of six with one in the middle. This pattern creates a gravitational wave, imploding by way of wave interference, through the seventh. The implosion draws in "cosmic" energy (or Zero Point Energy as some call it)".<sup>24</sup>  
The following quote from *Manna, Gold of the Gods*, begins with by research done by Dr. Muller with frequent reference to **George Merkl**, PhD:

"Contrary to popular scientific belief, Dr. Muller's research reveals, much to the chagrin of human genome geneticists, that genetic information is not contained in DNA strands. Instead, DNA and RNA molecules generate optical holograms which are in resonance with, and driven by, specific frequencies generated by the universal standing scalar gravitational wave. Every living cell resonantly receives the appropriate data in the form of weak electromagnetic coded signals from the universal scalar wave necessary for biochemical processes such as protein synthesis".<sup>25</sup>

Concerning resonance and the harmonics thereof, when you strike a high C on the piano say, or if a much higher C from beyond is struck, *all* the strings tuned to the C in the piano vibrate. The weak electromagnetic coded signals mentioned above strike such chords in our bodies all the time.

"Remarkably, in order to shield advanced biogenetic knowledge from the eyes of the profane, the simplistic images depicted on Sumerian clay texts by the Shemsu Hor, contain encoded data appertaining to protein synthesis and the manufacture of an elixir which repairs damaged cells and restores dead cells to life. Some 50,000 Sumerian clay texts contain coded images of cytoplasm, black protein, nucleotides and reticulated endoplasm. These texts discuss acid base. The Sumerian textual code was broken by the late **George Merkl**, PhD, that incredible genius whose suspicious death does not appear to be from natural causes.

"The basis of Dr. Müller's research was the discovery of a non-electromagnetic interaction between physical biotic systems. It was later found by Russian scientists that this previously unknown "biofield" interaction even occurred between supposedly inanimate objects such as rocks or interstellar material. Later, in 1988, microbiological experiments conducted aboard the Mir spacecraft determined that the biofield was actually gravitation and amazingly, modulated gravitation possessed the unique capability of transmitting biological data-a fact independently verified by the distinguished physicist and Sumerian scholar **George Merkl**, PhD.

"It appears to be the intent of the Russian government never to release the contents of the papers into the public domain, for they are based upon experimental data which not only disproves Einstein's gravitational theory but also renders much of presently accepted theoretical physics unacceptable. In other words, the world of theoretical physics requires a major overhaul.

"Russian experiments conducted during the 1960's and subsequently suppressed by the Russian Academy of Sciences, revealed that gravitational waves are able to travel at a minimum of twenty times the accepted velocity of light and that a "biofield" pervades the entire universe."<sup>26</sup>

What is the protit/somatid/chondriana then? Well, I maintain (and this will not be difficult to prove when modern science begins to take these things seriously) that the protit/somatid/chondriana is a charged albumin, globulin globule as Enderlein said, containing stacked, crystalline ATP molecules but, no DNA or protein.

As these protits/somatids/chondrianas begin to conglomerate depending on acid base and so on, they 'acquire' DNA from the node points of the logarithmic, universal standing scalar gravitational wave as matter (DNA). These optically generated holograms called DNA are created spontaneously by means of resonant, harmonic coupling that is driven by the specific frequencies generated by the universal standing scalar gravitational wave, the *engine of God*. ATP does act as the transducer of energy from the fourth dimension into LIFE! Life is everywhere, always!

There are no accidents in creation. **All creation is spontaneous.**

The concept of opposites is an illusion. There are no opposites, only *compliments* as you cannot have one without the other.

The universal logarithmic scalar standing wave is the only source of energy within the universe. All other physical objects, ranging from the sun to human cells merely draw energy from the universal wave by means of resonant coupling. Because of this harmonic coupling, the universal wave determines the physical form and growth characteristics of all living creatures, from simple protozoa to mammals, thus invalidating Darwinism. The distribution of matter within the universe is controlled by the universal wave, allowing for a flow of matter toward node points of the wave, thus preventing random and uncontrolled galactic growth. The logarithmic wave is, the engine of God.

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Before I start, I would like to explain the difference between Life Crystal and Chondriana. Life Crystal does not contain Chondriana and Life Crystal cannot induce Chondriana into your body, although some counterfeit products indicate the contrary. Life Crystal is produced by extracting adenosine phosphates, guanosine phosphates and five carbon sugars from off the shelf food items. It is a material with very high purity. The FDA in the United States approved adenosine in 1989 and adenosine monophosphate 20 years ago, and they know exactly what they do. When a patient is taken to the hospital with a coronary attack, this is the first thing they give them to save their lives in ER. The injection contains three milligrams of adenosine monophosphate and it costs \$3,000 per injection. That makes it a million dollars a gram. We have ten grams or \$10,000,000.00 worth of adenosine in every bottle of our Life Crystal drink and we market it world-wide for a very reasonable price.

When the adenosine and adenosine phosphate, guanosine and guanosine phosphate are crystallized and dissolved in double distilled water, they depolarize the water. The Life Crystal has the same tetrahedral structure as the water, with matching frequencies, and therefore it can charge the water with enough energy to change the polarity of the water. Once it changes the polarity of the water, it will lose the affinity to hydrophilic molecules and pick up affinity towards hydrophobic molecules. In layman's terms, this means it can dissolve double carbon bonds, hydrocarbons, waxes, cholesterol, plaques and sugar deposits, and this enables it to clean out the blood vessels. Most of the time when an organ fails, it fails because of lack of respiration and lack of circulation. Once you clean out the blood vessels and re-establish circulation, the organs will recover very quickly.

Life Crystal has a tetrahedral structure like a pyramid. Because of the shape of the tetrahedral crystal, it develops a vortex field around the pyramid and then the crystal will deposit around the pyramid in a vortex coil. This is the reason why Life Crystal can tap into the cosmic free energy of creation. Light is made up of free energy, 918 pairs of scrolls which make up one photon, and this is the breath of creation, or the breath of God, whatever you want to call it. Without that, life cannot exist on earth. With this knowledge we can break down light for the first time. The 918 pairs of photons then mould into one electron and 918 pairs of electrons mould into one neutron. That is the way free energy is molded into light and light is molded into matter. We are all made of the same substance. This is the reason the Life Crystal is capable of tapping into the life energy which is a prerequisite for life...

The Chondriana are human in origin. They are our precursors. They are micro-organisms which have been synthesized from human DNA. The Chondriana existed three and a half billion years ago. We had life forms back then that were capable of reproducing or mass producing all our organs. There is a micro-organism which has been found fossilized in western Australia dated three and a half billion years old. It is identical to the Chondriana. The Chondriana (ancient female micro-organisms) give birth to the Chondrions (ancient male micro organisms). Every organelle in our cells was at one time produced by similar female Chondriana. In other words, we had life forms, back three and a half billion years ago that were capable of reproducing all our organs. We had factories in our cells, which we do not have today, which produced the organelles inside our cells. In so many ways we have de-evolved. With this discovery we can correct the atrophy that has taken hold of us at a deep cellular level and activate dormant DNA codes to regenerate ourselves. In the Bible some of the famous characters lived hundreds of years. So can we too once we re activate this ancient sumerian water technology...

With a reverse procedure, we can recreate those factories in our cells and that is what I have done in my own lab. I took the organelles inside our cells and recreated all the factories from which those organelles had originated. I was able to produce Chondriana, mass producing centrioles, mitochondria, Golgi apparatus and all the different organs, including the cell nucleus and cell nucleolus. If you can start producing all these factories with Life Crystal, you can start replacing some of the old worn out factories inside your cells. But even more exciting, I have Chondriana now mass producing nucleoli, and nucleoli can reproduce nuclei and primordial cells. All our cells differentiated from primordial cells. The only place we have primordial cells left in our body today is in the female and male sex organs. We can direct these cells to produce giant lymphocytes to protect us. This means we can bring back our immune defense system, the defense cells we once used to have but which we lost because the sun is cooling down and we are losing energy. We are not going through evolution; we are going through devolution. Our cells are becoming punier and punier as time goes on. The massive carbon dioxide blankets we are putting up in the sky is accelerating the process. But now, re-introducing Life Crystal into the human system, we can re-awaken our ancient immune system. We can bring back the old giant lymphocytes which are able to protect us from a degenerate environment until we can all agree to begin a sustainable global lifestyle.

The reason I was able to bring Chondriana back to life is because I can simulate the energy level at any time in history with Life Crystal. I can go back three and a half billion years to bring back any kind of life that existed then, depending on how much energy I put into the Life Crystal medium. The first Chondriana which were synthesized were the precursors of our mitochondria, the organelles inside our cells and the precursors of our immune system. These Chondriana are super intelligent and are given intravenously to the human body. They are the military and they attack any kind of disease that is present in the body. They can pick up the frequency from diseases and match it with the proper military to crush the disease. They can also pick up the frequency of our T-4 cells or T-8 cells or lymphocytes and mass produce them on demand...

If there is a disease involved, whether it is viral or bacterial, it always needs Chondriana. Life Crystal will not eliminate diseases. Life Crystal will clean out the blood vessels, restore respiration, rejuvenating and rebuilding. It will also help strengthen the immune system. If there is a disease involved you have to use Chondriana. If it is not a disease, just degeneration, you can rejuvenate with just the Life Crystal. Doctor Merkl went on to discover the Sumerian Water Elixir of Life and with this advanced formula you did not have to use the Chondriana as they were reawakened inside your own body with the Sumerian Formula...

Many clinics were using Life Crystal for chelation therapy. They are replacing E.D.T.A. with Life Crystal to clean the blood vessels and they are getting much better results. When someone is on Life Crystal, even just as a nutritional supplement taken orally, if they take it every day, even if their arteries are 98% plugged, there is no need for heart by-pass surgery, and within one year the arteries will be cleaned out completely. The body water will dissolve the cholesterol and they will urinate off the fat. Chelation may be done in two ways using Life Crystal. We recommend a teaspoon three times a day for the oral dose. The intravenous treatment is given at half a cc. per week...

Life Crystal cleans out the blood vessels and restores circulation. When Life Crystal is administered intravenously to a diabetic who needs a kidney transplant, in five weeks, the kidneys function normally and there is no need to replace the organ. If diabetics are scheduled for a leg amputation, in five weeks, Life Crystal will dissolve the sugar deposits, restore respiration and nutrient flow, eliminate claudication, and they can go jogging. We have had diabetics whose eyesight has also been restored...

We have had ample clinical evidence from all over the world that Life Crystal helps restore health. We have had a few cases of Parkinson's Disease and our treatment was successful. We are very effective against cancer, environmental illness and all diseases. We get a 100% response with HIV, AIDS. We have some people now who have been cured from AIDS. All the lupus diseases, rheumatic arthritis, circulatory problems, diabetes and so on. When people have coronary heart attacks, they may administer it every day. Someone who has a serious problem such as kidney failure, claudications or blindness, should use the intravenous method because it is quicker. In five weeks it restores the circulation and

respiration, and cleans out the blood vessels. Because the Chondriana are precursors of our origin, they are part of us only they have been dormant until I brought them to life to do their natural job. Therefore, you are not taking drugs or chemicals when you take Life Crystals...

I have had about 20 clinics in Mexico alone. In Tijuana, almost every clinic is using Life Crystal and Chondriana, including the Contreras Clinic and the Meer Institute. We have clinics in European countries, in England, Belgium, Germany and Switzerland, in the University Hospital in Greece, in Australia, and in the East-West Medical Foundation in Taipei, China.

Q & A --

Q: How do the Chondriana interact with pathogens and viruses?

Dr. Merkl: The Chondriana themselves are militant. They attack the disease directly. They attack it physically, consuming it. Their membranes are invasive, so they attack the disease, take it apart and recycle it. They break down the genetic material all the way to ribosomes and recycle them into their own sequence. They grow up on it. They can also produce interferon and interleukin II. They can stimulate the T-4 and T-8 cell production, or they can pick up the frequency of your immune cells and reproduce them. They can reproduce dozens of different types of macrophages, each of which has a different type of function. They produce oscillators of many different types, immune cells which break up the disease and then come and clean up like a vacuum cleaner. The Chondriana's mouth is like a sack. It is like a shutter of a camera. The neck can narrow and open and they use it like a vacuum cleaner. They just mop up the disease. Because they are super intelligent, they know the sequence of life. They can sequence the DNA molecule to make all kinds of life forms. They can make life like you make breakfast. They are far more intelligent than we are and they are our ancient ancestors living inside of us.

Q: Are the Chondriana not rejected in the same way an organ implant from another person is rejected?

Dr. Merkl: The Chondriana do not come from another person. The Chondriana belong to everyone. It doesn't type to my person or any person. When the Chondriana are injected into your blood stream, the Chondriana's genetic material goes into your blood cells, blows itself up four times the size, and you start mass producing your own Chondriana from that moment forth. Since the discovery of the Sumerian Water it is no longer necessary to use the Chondriana injectables as the Sumerian Formula will awaken and activate your own dormant Chondriana.

Q: Does the human system have Chondriana already in it?

Dr. Merkl: Yes, In dormancy. We used to have it active, but we don't have it active any more, only dormant. Only the information on the DNA is left with us.

Q: Is Life Crystal dependent upon Chondriana?

Dr. Merkl: No because Life Crystal by itself is basically rebuilding, restoring, rejuvenating and cleaning out the blood vessels, restoring the circulation, respiration and providing nutrients. Ninety-five percent of your energy comes from ATP, and Life Crystal is loaded with ATP. But you can't make Chondriana without Life Crystal.

Q: What is Life Crystal and how do you make it?

Dr. Merkl: Life Crystal is made up of adenosine phosphate, guanosine phosphate and five carbon sugars. Our DNA molecules are made from it. They are the basic building blocks of life. We take this from plants. The adenosine and guanosine phosphates come from genetic material of plants, like lettuce, tea etc. The five carbon sugars come from fruit juices, organically produced, very high purity fruit juices. You reap off the carbon and you have five carbon sugars. You add a phosphate to it and you have sugar phosphate (fructose and glucose phosphates). These are intermediates in the process of a new cell making ATP.

Q: Can you take Life Crystal and Chondriana at the same time?

Dr. Merkl: You can take them independently or you can take them together. You put the oral Life Crystal under your tongue. Keep it in your mouth for a minute or two, let it absorb before swallowing it. We usually recommend a teaspoon. There is no shelf life limit. The longer you keep it, the better it is. You can keep it on the shelf. You don't have to refrigerate it until you open it. We recommend that people use Chondriana if they are sick. If they have a bacterial or viral disease, they use half and half: half Life Crystal and half Chondriana, since the Life Crystal helps to open up the fine blood vessels. When the Chondriana go after the disease and kill it, the waste and the toxins can be drained much faster because your fine blood vessels are open and you have a much lower reaction.

Q: Is Life Crystal safe to take whether you are sick or not?

Dr. Merkl: Absolutely. It is very safe. It is the perfect preventative. The youngest person to take it is four months old. My wife just gave birth to a baby girl and she was taking it all through her pregnancy. She was also on Chondriana treatment. It is safe to take the nutritional supplement, one teaspoon three times a day, not the injectable. It is absolutely safe. It will be very interesting to see what effects it has had on the child. She is too young to draw blood and test it, but I will test her later. We have three Chondriana babies from different families. Two of them couldn't conceive previously. Once they went on Life Crystal, they conceived within two or three months because Life Crystal will speed up the proliferation of primordial cells, the sex cells, and make them very strong. The children are very strong and very healthy and all three of them are pretty close to nine pounds and were born at eight months, not nine months, and they turned around at three months. My wife and I had three other children before and this pregnancy was entirely different from the others.

Q: We have a boy who has chronic asthma. Can he be helped with Life Crystal?

Dr. Merkl: We have a lot of little boys who are just on Life Crystal drink alone whose asthma is disappearing. We have a lot of children in countries where their mothers were exposed to harmful chemicals or insecticides when they were pregnant. All these children were born with epilepsy and seizures. They had 200 to 400 seizures a month, and some had 10 seizures a day. They went on Life Crystal drink alone, nothing else, and within two months they were normal.

Q: Is Life Crystal effective for Candida?

Dr. Merkl: Candida occurs most of the time in association with Epstein Barr syndrome. It is a major epidemic in English speaking countries. United States have 60 million Epstein Barr cases and only five percent of the population know that they actually have it. It spreads from passing around the bottle or kissing. You are spreading it by saliva, drinking or eating after someone else. It stays dormant for 15 years, but once it breaks out and gets into the active state, you start developing the Candidas. Candida eats away your glucose and deprives you of ATP, because ATP is made from glucose. Your organ cells degenerate, and as a result the disease moves in, and the immune cells degenerate, so more disease moves in. Your immune cells are not capable of defending you. If you supplement ATP, you can freeze the disease, sort of put it on the shelf, because you keep your organ cells and your immune cells happy. Then you can lead a normal life and you eliminate your chronic fatigue. Chronic fatigue occurs because your cells are starved to death. They don't have ATP anymore. Ninety-five percent of energy comes from ATP. If you want to get rid of the disease, you have to go through Chondriana treatment, 30 injections, which give very strong reactions, but you can get rid of the virus. Epstein Barr is called the cousin of AIDS. AIDS destroys the immune cells directly. In Epstein Barr and Candida, the immune cells are starved by deprivation of ATP.

Q: How do you know how many injections to use for the Epstein Barr virus?

Dr. Merkl: In order to determine the number of injections to use, we do blood tests. We look for the immunoglobulins. The level should be below 20. The more advanced your Epstein Barr is, the higher the level is. If someone had 600, they have probably had Epstein Barr for about 25 years. If they had 1400, they may have had Epstein Barr for 35 years. Those are the final stages. In the final stages, Epstein Barr attacks the brain and the organs and destroys the organs; people cannot function any more and they die.

Q: Is Life Crystal effective against heavy metal toxicity including mercury?

Dr. Merkl: Yes. It is being used as a chelation agent. The Life Crystal is a very effective chelating agent but not necessarily for the removal of mercury.

Q: Have you had any success with lupus?

Dr. Merkl: We have very exciting results with lupus. We have lupus A, B, C and D. The lupus A attacks basically joints. Lupus B and C attack organs and lupus D attacks the brain. It usually takes about five or six injections to be on the safe side, 10 injections for sure gets rid of lupus of any kind, either A, B, C or D. Lupus also occurs together with hepatitis, because if you have B and C, they also attack your organs including the spleen, liver, kidney and bring in hepatitis. So in one sweep we can get rid of all the problems.

Q: Can Life Crystal be used for rejuvenation?

Dr. Merkl: We can reproduce primordial cells which can be turned into new young cells in order to reverse your aging. You can restore your body, your organs and your tissues, and not only slow down, but actually reverse the aging process in your body.

Q: Can Life Crystal be used for thinning hair and baldness?

Dr. Merkl: Well, look at my hair. Sixty percent of my hair is new and whatever I had before was completely white. It grew in about a year and a half. You can use it topically.

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### **Dr. George Merkl : Origins of Life -- Journey into Inner Space [ Excerpts ]**

When electrons are placed in Sacred Geometry, a coherent electron beam is provided by the superlattice network. The coherent light is laced throughout and between each row of the superlattice grid structure. This superlattice acts as a beam splitter, resulting in a multitude of coherent photon beams. All wave form patterns are pinned down in this superlattice, remaining in place as the superstructure of this dimension. Photon emissions are then under control. The wave addition therefore will up-shift the amplitude and the similitude from the blueprints of life...

Since the molecules are in lock-step, the split-beams on the existing grid result in a multitude of interference patterns, locking in the captivated blueprint of expressed genetic molecules. The captive blueprints of life then form templates in the photon light beam that will serve as the skeleton of life forms. The building blocks of life can now fill in the sacred map of the photons to reassemble life from the information stored in the light! Blocking in the constant phase insures the template printing of interference, and the mobility of the molecules are thus held in order by the superlattice network grid...

With the timely application of this knowledge, Peace will come as the Promise Fulfilled...

Now that I know of what I know, I realize how little we all know!...

We know from historical evidence that the Sumerians appeared in the Mesopotamian Valley about seven thousand years ago, setting up four colonies. [After the final end of Sumerian power and civilization around 2000 BC, the area came under the exclusive control of Semitic peoples for centuries]. The Semitic genes I received from my mother's side of the family shall now be re-explored, as I am able to bring back the Sumerian past they left behind as they inbred with the Semites before they vanished from the surface of the earth. One question still remains today unanswered: did they leave as giants as they once appeared here or did they perhaps miniaturize themselves with the technology I have re-discovered, remaining here in the 4th dimension waiting on us to wake up to the Cosmic Pulse of Life? Many of us believe there is a 12th Planet with an orbital time of 36 thousand years. If such a planet exists, it must be composed of Cosmic transparent matter. That would make the planet BLACK, but also churning out anti-gravitational fields powerful enough to break out from the solar gravitational field. It probably orbits between two suns, which would explain the long orbital path. The composition would also explain the catastrophic events on earth when this 12th planet passed by, disturbing the solar system by gravitational field interaction. The interference patterns between the suns and its planets, if disrupted, would result in orbital changes. Such catastrophic event could bring about airborne matter and disease as well, causing planets to flip over or even to collide. Historical evidence indicates that such events took place in Biblical times...

I will attempt to take you back to the beginning of time, where we can come to appreciate the Sumerians whom taught us Cosmic Geometry and Cosmic Math. The recent restoration of Sumerian Math is still in my genes - THE PAST

SMART Protein Scientists with gender design built the Cosmic Bio-Machines, following the Cosmic Order. These Cosmic Life factories and their scientists came here thousands of years ago and they haven't left us yet. The question is, when and where did they land? Or have they been here all of this time outside of our time line? Or, did I call them back in time via this rediscovered Cosmic Science they left for us to find? Perhaps the Sumerians never left here, they just miniaturized themselves and slipped into the 4th dimension. I have documented and actually miniaturized and magnified life forms via the interference Cosmic Order. It is like a Hyper-Dimensional Hologram or replicator. Imagine a flying saucer or spaceship that turns into microscopic size and hides in a grain of sand or in a crystal. SMART Proteins hold the blueprint to all life forms, including the Cosmic Bio-Machines. Ordinary protein holds the bio-structure. SMART Proteins hold the sacred blueprints in interference or perhaps seek out and process the information held captive in the interference patterns. SMART Protein Scientists are born out of the Sumerian Water, the Elixir of Life that delivers the Cosmic Order...

These new Cosmic Scientists are harnessing the Cosmic Order that holds the Breath of Creation. Their arrival as the new Macro-science is the beginning of the NEW GOLDEN AGE OF LIGHT, as we will be able to enter into the highest COSMIC PLANE. One of the criteria is the SUMERIAN WATER that is structured to this Cosmic Order! By altering the hydrophobic and hydrophilic properties, such sacred water is essential to all life and is found in the nucleus of a cell and is central to build microtubules or spindles for cell division in the mitochondria, where the protein substrate that processes the ATP is in this Sacred Water. In Cosmic Geometry and superconductivity, such water is also found in the cerebral spinal fluid where the nerve impulses are transferred to the distribution points along the grid. These are the fingerprints of the Cosmos. The Sumerian Water, or Elixir of Life, plays an important role in processing such water and producing it into specific geometry. Parts

of the Beta Sheets in Cosmic Order are made from these large SMART Proteins. They also design the Amino Acids to the desired length. They design and build protein riveting machines to build intricate bio-structures. They design and build phasing in and out dish antennae fields, with bell shaped optical devices, to produce interference blueprints of all life forms...

All this SMART Protein precision hardware is hooked into a specific, life-replicating engine that processes information to replicate genetic material from light holograms [that are] holding the blueprint of life! They are building and stacking the Beta Sheets of proteins by these assembly workers laying down the proteins one by one, like the stone masons laying down the bricks! They fit the peptides into three-dimensional geometry by instant production, just like bees building their honeycombs. They use the Sacred Water as a scaffolding, acting as a mold, in conjunction with the interference patterns laid into the Cosmic Geometry...

Through the rediscovery of the Sumerian Water, the Elixir of Life, these Ancient-Future, Cosmic Scientists have arrived once again and have been domesticated to teach us the Super Science of the Cosmos, as the Sumerians [originally] had access to it. With the discovery of the Life Crystals, I was able to mass produce layered, staked SMART Protein Beta Sheets to produce Live Crystals in Cosmic Order. These Live Crystals then are able to holographically self-replicate themselves and reproduce by copying, via a holographic blue print. This discovery then paved the way for the Sumerian Technology, which they brought with them when they landed in the Mesopotamian Valley seven thousand years ago. The Sumerian literature they left behind has now been translated, and the Sumerian Math and Geometry have been restored to understand the Cosmos and how energy is condensed into matter or life! The life process is self-controlling, once we provide the ELIXIR OF LIFE or the SUMERIAN WATER as the medium. The ELIXIR the Sumerians had has been rediscovered by me on Christmas day, 1995, early in the morning. Now that I know of what I know, I realize how little we all know!..

...[T]his Cosmic Physics of the Nuclear Well with a six-wave interaction that is drawn into the seventh, forms wave units to form a graviton energy unit. When we take seven graviton or gravity waves in the same way, we form phase-coherence to fill the nuclear well to braid into FREE-ENERGY SCROLLS. Finally, a photon and a quantum of light have been born! Now, when we begin to assemble quantum's of light in marching phase, Sacred Geometry forms and transfers dectrons, photons and SMART proteins into Cosmic Order; superconductivity and superfluity will thus commence."

So now when we adopt this Ancient Future Cosmic Physics to SMART Proteins or genetic grains, we can replicate living crystal BIO-COMPUTERS, surpassing our brain functions. [This will] synthesize all into a self-replicating, self-restoring, self-maintaining eternal mode, totally alive to serve us and to be our very best Cosmic friend!"

A NEW AGE FAMILY of Cosmic Scientists and Engineers, along with their production teams and assembly workers, are already here and ready to serve us freely. They could be put into SERVICE to revolutionize every spectrum of human endeavor in all our industries. This potential is hard for even me to comprehend. They could recycle the excess Co2, saving us from the negative effects and turning it all into clean, natural food (glucose - ATP). They could build industry for us, eliminate all diseases forever and help us to re-grow new limbs and organs within our bodies. But most of all, they would give us those extra years for further space exploration... inner and outer space exploration, that is! This is not in the realm of science fiction... My Cosmic guys are here now to prove it. See them for yourself!...

This NEW AGE BIO-ELECTRIC INDUSTRY could tap into the ATOM without a nuclear reaction! The new institutions shall free the minds from captivity of an old, worn out, mind-set paradigm, encouraging us to dare to dream, explore and use our more awakened minds and intuition. This is the reality and its here today, as the NEW AGE shall sweep out the old one once and for all!" -- Dr. George Merkl

### **Cosmic Bio-Machines or Incubators within the Power of the Cross**

The Hyper bionic Machine - MEGA Protein Factory is the Cosmic Incubator or the Cosmic Bio-Machine (CBM). It is the ATP Nucleotide production plant. The equilateral cross as the Cosmic Bio-Machine is the nuclear engine. Both the gravitation and the magnetic fields are represented in the Power of the Cross. This machine has a Phase Out Vortex, an Implosion Bell and a Phase In Vortex. The Cross of Life, or the Power of the Cross, is the Nuclear resonance of the Cosmic Incubator or Cosmic Bio-Machine...

The Sumerian Life Amplifier (superlattice, beam-function order) acts as a gravitational hologram, containing the coherent electron beam and coherent light beam combined with the Sumerian Crystal water. [This is seen as] the surface tension forces compress the TETRA HEDRA. The Sacred Geometrical constraints require the nucleotides in the superlattice to vibrate along this string. This frequency of resonance then is transferred, to be stored in the interference points of the coherent electron photo beams. Molecules then arrange themselves according to the ADAMIC BLUEPRINT, as is recalled for storage in the 4th dimension..."

### **Life Crystal Sumerian Water, the Elixir of Life**

In the Life Crystal water the water molecules are structured to the grid patterns of Sacred Geometry. The Sumerian Water is laced with the minute, Elixir of Life that I discovered in plant sources and it delivers light that is energizing the water. Next we have the birth of the Nuclear Well, which is holding energy into matter. Any materials in our world that have fallen into disordered incoherence can be restored or resurrected with this knowledge, if applied correctly. Thus, by restoring life back into the elements by restoring the Sacred Geometry into lockstep, we have a new world of regeneration at our finger tips! The Elixir of Life is coherent nutrients, and our DNA behaves like a wave, so we shall nourish our DNA with high potency nutrients that behaves like wave forms...

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## **Sacred Geometry**

by

**Dr. George Merkl**

306 pairs of photons in Sacred Geometry braid the interference points into lockstep to form electrons. If the lockstep is scattered and out of Sacred Geometry, the electron dispenses into photons.

$$(6 \times 7 + 10 \times 6) = 102$$

$$102 \times 3 \times 3 = 240 \times 3 = 612 / 2 \text{ 306 pairs of photons}$$



In this Sumerian Water combined with my own genetic material, I witnessed the emergence of a very ancient life form called the Chondriana. It was she that divided herself and gave birth to a male Chondrion. In this Chondriana, the DNA and RNA segments are the library of the past and the present, holding all the information needed for biological process and proliferation. The SMART Proteins in the DNA wrap around histones and provide the functional life-fields that activate the DNA. Enzymatic functions of genetic and chemical processes are controlled by these SMART Protein molecules. The SMART Proteins are produced in the recent cytoplasm located in the head of the spermatozoa, that is replicated in the undifferentiated cells and carried over to differentiated cells.

The degenerated remnants of these primordial, SMART Proteins synthesized in the ancient cytoplasm, resurrected themselves via the LIFE CRYSTAL. Such a treasure I discovered in the tail section of mammal spermatozoa. When the RNA wrapped around the modern proteins it is capable of reproducing all recent genetic hardware, replicated and self-instructed to carry out the full, life process. When the RNA wrapped around the primordial proteins, the information was pasted on to the RNA (activation), historical, genetic hardware, which can be retrieved from the ancient past. In other words, the RNA will act as an assembly worker functioning according to the instructions of the SMART Protein... the ribosome of the RNA carrying out the message of its SMART Protein in protein synthesis. Proteins and RNA-DNA are interdependent, hence the protein is the original parent of the RNA-DNA.

The RNA is an information library as well as an assembly worker, activated by the SMAART Protein that provides the life force. Again, compare the RNA-Protein to the core and the windings to induce the life process. This life force encourages the biological molecules to interact and carry out the designated life process within the cell. This SMART Protein-RNA combination not only carries out the life-enzymatic processes, but also has the intelligence to disassemble invaders such as viruses and microorganisms, providing inter-cellular defense.

This protein folds to specific 3-D geometrical shapes to initiate specific life replication, as the SMART Proteins with the Amino Acids fold and shape the original life form.

The nucleotide Life Crystal supplements in the life process are in the mono-phosphate form. However, the Life Crystal contains all the other forms, as well as the intermediate of the fructose and the glucose phosphates. A gene expression of the sequence of nucleotides transcribed to the RNA act as an assembly worker (template) to participate in the protein production process.

Since Chondriana is a single cell organism, it falls into the category of eukaryote. Having four ribosomal chondriana synthesized from dispersed or spliced genetic materials, the uncoded DNA is folded into circles, with the action of the GTP (guanosine-tri-phosphate) hydroxyl (OH) groups playing active parts in the cleaving.

The GTP acts as the splicing agent, cleaving the strand of the RNA at the site of the five carbon sugars between the phosphate group and the five carbon sugars, attacking the phosphodiester bond that is the bridge between the phosphate group and the sugar.

Once the DNA is spliced and formed, the ring of the life process begins with protein synthesis. The four RNA segments hold the information of the haploids. As the head of the spermatozoa enters the ovum the first step is the formation of the Chondriana. Thus evolves the morula cell cluster (clef appears in the morula). This is the cross point, as the pre-embryonic cell cluster evolves with the Chondriana ancestor. From the primordial cells (the ectodermic, endodermic and the mesodermic germinal layers), cell differentiation begins by hormone activation provided by the ovum, as the embryo come into existence. If the Chondriana is recalled outside of the ovum in the absence of the hormones, the ancestor Chondriana cannot develop into the morula. Instead, each haploid cell becomes an independent, primordial life form. Germinal layers in both the testes and the ovaries produce primordial nucleuses that can turn out Chondrianas in conception, when the two nuclei (the ovum and the spermatozoa located in the bottom of the head) fuse during pre-embryonic stage of the first Chondriana. These ancient ancestors of our immune systems form in the pre-embryonic morula a functional immune system... that later de-evolves from its primordial immune system to our current, atrophied, immune system condition.

Once the immune system is in place, then under the protection of cell division begins what leads to the first, pre-embryonic, fetal tissue [development] via hormone stimulation, cell-differentiation into the fetus. Life consist of two different entities: the immune system that evolves first, and then the higher order of life that molds together into one genetic system. The link between the two, life systems are the sex glands that are capable of replicating both of these systems.

As life evolves around our immune system, the immune system is being devolved and degenerated from the Chondriana. For our well being the two systems must be in perfect balance, or else disease will take over. To reverse the degenerative path of the immune system, one must restore the diminishing, life force energy level in the body. We evolve via devolution as a genetic nation with our cells united for our very survival, forming an ally with the immune system!

The Cosmic Bio-Machine or Cosmic Incubator implodes Life Crystal in a CO<sub>2</sub> Catalyzed Membrane, which is in superlattice or lockstep. First the Amino Acids form, then the SMART Proteins and finally the RNA and micro-organisms. This creates the ATP - Photo Synthesizer Matrix. In this matrix, living cells are stored in crystals in superlattice. The Mitochondria is in Superlattice. The super matrix of poly-adenosine nucleotide phosphates is the electron transport system to convert the CO<sub>2</sub> into organic molecules. This super matrix delivers energy to produce ATP, driven by proton gradients and splitting the water molecule by an electro-proto-synthetic process. This mechanism drives the Cosmic Incubator or Cosmic Bio-Machine, that probably existed at the time life first evolved. These incubators can process life but also are able to become very efficient ATP synthesizers. The Cosmic Incubator can process both cellulose - Aldemides and CO<sub>2</sub> into glucose and ATP.

The owner of the CROSS and the STAR OF DAVID superlattice design matrix represent the Cosmos. Even in living cells and organisms, you will find the STAR OF DAVID and the Superlattice of proteins or nucleotides as the Creator placed them there. The Superlattice follow the electron configuration in lockstep. First came Crystals and light synthesized carbon dioxide, then came the precursors of the Mitochondria, the COSMIC INCUBATOR. Poly Nucleotides follow the Sacred Geometry that is the signature of the Cosmos. Molecules (LC) line up in lockstep (superlattice) to form Live Crystals. Such crystals are stored in the Mitochondria - cell-nucleus - centrioles and throughout in the cell, to store energy and nutrients for the genetic process.

When electrons are placed in Sacred Geometry, a coherent electron beam is provided by the superlattice network. The coherent light is laced throughout and between each row of the superlattice grid structure. This superlattice acts as a beam splitter, resulting in a multitude of coherent photon beams. All wave form patterns are pinned down in this superlattice, remaining in place as the superstructure of this dimension. Photon emissions are then under control. The wave addition therefore will up-shift the amplitude and the similitude from the blueprints of life.

Since the molecules are in lock-step, the split-beams on the existing grid result in a multitude of interference patterns, locking in the captivated blueprint of expressed genetic molecules. The captive blueprints of life then form templates in the photon light beam that will serve as the

skeleton of life forms. The building blocks of life can now fill in the sacred map of the photons to reassemble life from the information stored in the light! Blocking in the constant phase insures the template printing of interference, and the mobility of the molecules are thus held in order by the superlattice network grid.

Super conductivity is electrons in Sacred Geometry. Super conductive, electron-spin interaction in Sacred Geometry form stacked Cosmic Holograms. The electron's orbital path, when in lockstep and paired in Sacred Geometry in such a system, causes all photons within the electron (electrons made of photons) to also be present in ordered form. External Light will lay throughout the ordered structure, in coherence. If this system is transplanted into nucleotides of genetic material, Sacred Geometry will then follow. Once the genetic material is in Sacred Geometry at the interference points, the genetic blueprint locks in via the coherent beams from which life is replicated into this third-dimensional form."

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## YouTube Videos

<http://www.youtube.com/watch?v=Hh8VJ-5a3uI>

**Intro to Sumerian Microbiology**

[http://www.youtube.com/watch?v=k\\_4u3FAOulQ](http://www.youtube.com/watch?v=k_4u3FAOulQ)

<http://www.youtube.com/watch?v=3qxFMLMzOgk>

<http://www.youtube.com/watch?v=3v0NHvfVSgQ>

<http://www.youtube.com/watch?v=fWkNaMEe6dg>

<http://www.youtube.com/watch?v=QWS3BUwQsTI>

<http://www.youtube.com/watch?v=DJu84ITgZFI>

<http://www.youtube.com/watch?v=S9OCD3-vaHg>

<http://www.youtube.com/watch?v=uc1rZPKf11Y>

<http://www.youtube.com/watch?v=bOBLhtGO3sU>

<http://www.youtube.com/watch?v=TjS4dx-9QVY>

<http://www.youtube.com/watch?v=DCYO-Xg8DbA>

<http://www.youtube.com/watch?v=asTs0eIwbtw>

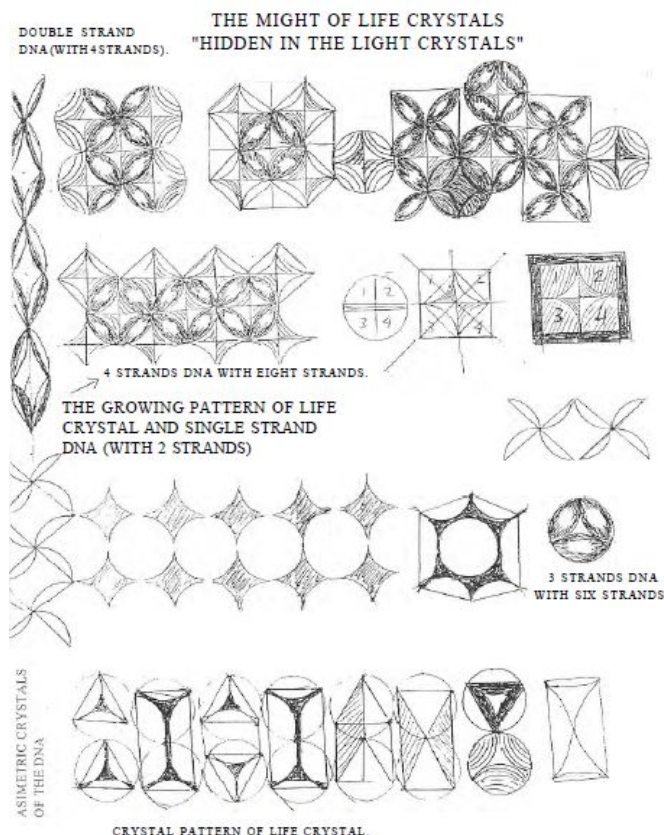
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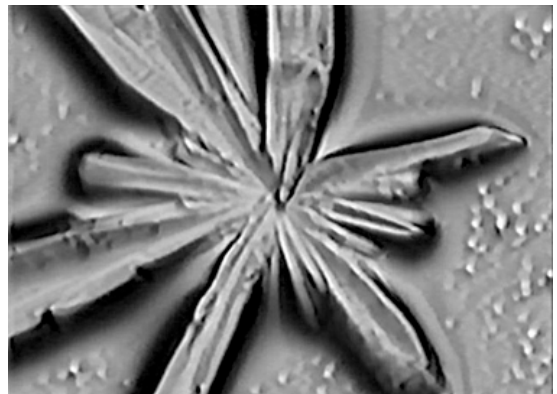
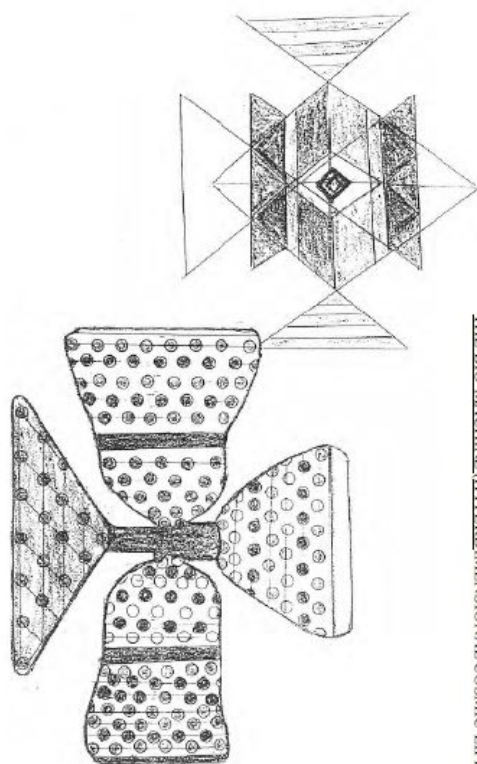
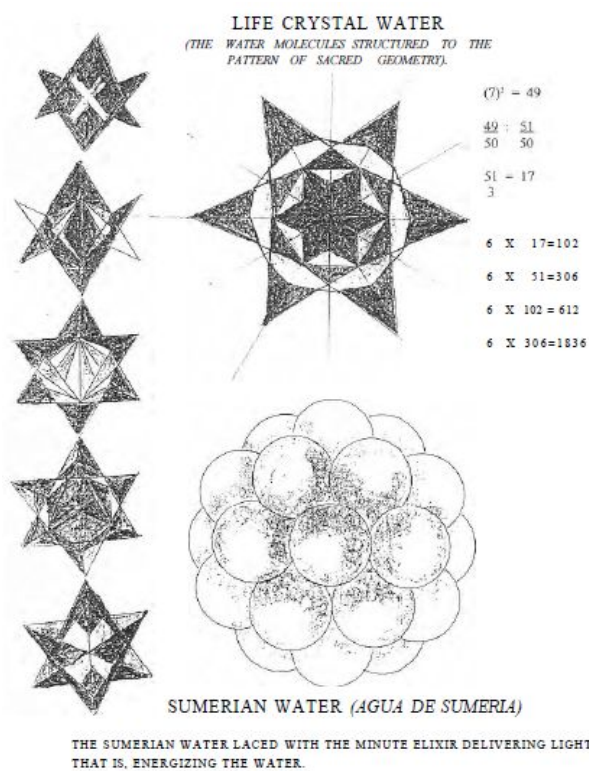
<http://www.youtube.com/watch?v=a3hbSZ1a7ZE>

<http://www.youtube.com/watch?v=4uRrn0M2f0E>

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## Select Graphics from Sumer-Tech





<http://sun-nation.org/>

SUMER - TECH



Cosmic Bio Machine  
George Merkl © copyright 1995

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NOTE : Due to bandwidth issues, the complete illustrated 38 MB PDF of this book no longer is posted here, but it is included on the Civilization Kit DVD.

Here is the link for the original online posting @ sun-nation.org :

<http://www.sun-nation.org/merkl-life-crystals.html>

<http://www.sun-nation.org/pdf/Sumer%20Tech%20-%20Dr%20George%20Merkl%2010-3-12.pdf>

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TEXT ONLY :

[ Prefatory Text Image Captions ]

HOME IN ON LIFE!

WHEN ELECTRONS PLACED IN SACRED GEOMETRY, A COHERENT ELECTRON BEAM IS PROVIDED BY THE SUPERLATTICE. THE COHERENT LIGHT LACED THROUGH BETWEEN EACH ROW OF THE SUPERLATTICE. THE SUPERLATTICE ACTING AS A BEAM SPLITTER, RESULTING IN MULTITUDE OF COHERENT PHOTON BEAMS. ALL WAVE PATTERNS ARE PINNED DOWN IN THE SUPERLATTICE, REMAINING IN PLACE. PHOTON EMISSION THEN UNDER CONTROL. THE WAVE ADDITION THEREFORE WILL UP SHIFT THE AMPLITUDE.

SINCE THE MOLECULES ARE IN LOCK STEP, THE SPLIT BEAMS ON THE EXIT RESULTING IN A MULTITUDE OF INTERFERENCE PATTERNS, LOCKING IN THE CAPTIVATED BLUEPRINT OF EXPRESSED GENETIC MOLECULES. THE CAPTIVE BLUEPRINTS OF LIFE FORMING TEMPLATES IN LIGHT, THAT WILL SERVE AS THE SKELETON OF LIFE. THE BUILDING BLOCKS NOW CAN FILL IN THE SACRED MAP OF PHOTONS TO REASSEMBLE LIFE FROM THE INFORMATION STORED IN LIGHT. BLOCKING IN THE CONSTANT PHASE, ENSURES THE TEMPLATE PRINTING OF INTERFERENCE, THE MOBILITY OF THE MOLECULES HELD IN ORDER BY THE SUPERLATTICE.

EXTERNAL LIGHT WILL LACE THROUGH THE ORDERED STRUCTURE, ORDERED INTO COHERENCE. IF THIS SYSTEM IS TRANSPLANTED INTO NUCLEOTIDES OF GENETIC MATERIAL, SACRED GEOMETRY WILL FOLLOW. ONCE THE GENETIC MATERIAL IN SACRED GEOMETRY AT THE INTERFERENCE POINTS, GENETIC BLUE PRINT LOCKS IN VIA COHERENT BEAMS FROM WHICH LIFE REPLICATED.

INBORN THE BLUEPRINT OF LIFE WITH TIME AND SPACE FROM HIS BREATH, IN BURSTING BUBBLES LIFE HAS BEEN DELIVERED. INSTANT LIFE FORM IMPLoding THE LIFE CRYSTALS CATALYZED BY CO<sub>2</sub>. GRAINS OF LIFE IN SUPER LATTICE, AMINO ACIDS IN LOCK STEP ARE FIRST PROTEINS NEATLY MOLDS THEN RNA FOLLOWS.

THE GRAINS OF LIFE'S CRYSTALS ARE ALIVE AND DREAMS USHERED BY THAT OF MYSTERIOUS HANDS, GENETIC CRYSTALS RESPOND AND ARE NO LONGER A DREAM.

(IMPLoding LIFE CRYSTAL IN A CO<sub>2</sub> CATALYZED MEMBRANE FIRST AMINO ACID FORMS THEN PROTEIN AND FINALLY RNA AND MICRO ORGANISM.)

SUPER MATRIX OF POLY ADENOSINE NUCLEOTIDE PHOSPHATE IS THE ELECTRON TRANSPORT TO CONVERT CO, TO ORGANIC MOLECULES.

THIS SUPER MATRIX DELIVERS ENERGY TO PRODUCE ATP DRIVEN BY PROTON GRADIENTS AND SPLITTING WATER BY

## ELECTRO-PIOTOSYNTHETIC PROCESS.

THIS MECHANISM DRIVES THE COSMIC INCUBATOR THAT PROBABLY EXISTED AT THE TIME LIFE EVOLVED. THIS INCUBATORS CAN PROCESS LIFE BUT ALSO ARE A VERY EFFICIENT ATP SYNTHESIZER. THE COSMIC INCUBATOR CAN PROCESS CELLULOSE-ALDEHYDES AS WELL AS CO<sub>2</sub> INTO GLUCOSE AND ATP.

THE OWNER OF CROSS AND THE STAR OF DAVID REPRESENT THE COSMOS. EVEN IN LIVING CELLS AND ORGANISMS, YOU FIND THE STAR OF DAVID AND THE SUPERLATTICE OF PROTEINS OR NUCLEOTIDES AS THE CREATOR PLACED IT. THE SUPERLATTICE FOLLOWS THE ELECTRON CONFIGURATION IN LOCKSTEPS. FIRST CRYSTALS, AND LIGHT SYNTHESIZED CARBON DIOXIDE, THEN CAME THE PRECURSORS OF THE MITOCHONDRIA, THE COSMIC INCUBATOR.

POLY NUCLEOTIDES FOLLOWS THE SACRED GEOMETRY THAT IS THE SIGNATURE OF THE COSMOS. MOLECULES (EC) LINE UP IN LOCKSTEP (SUPERLATTICE) TO FORM LIVE CRYSTALS, SUCH CRYSTALS ARE STORED IN THE MITOCHONDRIA-CELL-NUCLEUS-CENTRIOLES AND THROUGHOUT IN THE CELL, TO STORE ENERGY AND NUTRIENTS FOR THE GENETIC PROCESS.

When six wave combine with the seventh in the center, the center one becomes the nuclear well adding all the waves-crests and their amplitude increases. Slurping them in like whiteholes, however if the waves are out of phase they will annihilate (cancel) each other. When light is laced through the rows of electrons or protons ordered in cosmic-geometry the photons too form superlattice and countless photon beams. The electrons in superlattice act as a beam splitter. The coherent waves emerging from such system will form interference with each other in an orderly fashion. But since in the crystals electrons, 41 photons and proteins are also in superlattice they too can form interference in an orderly fashion. If the square of the wave function of  $6 + 1$  waves in the superlattice would result in 49 or  $42 + 7$  that would manifest in Sacred Geometry. Since we are absorbing energy in three dimensions, each set of scrolls must be absorbed at the same frequency therefore  $33 \times G = 18-1$  the one in the center becomes the nuclear well absorbing 17 waves this three dimensional scroll will pair, absorbing a total of 34 waves.

This single scroll (17W) in three dimension ( $3 \times 17 = 51$ ) forms one tetrahedral that is equal to one photon. But two tetrahedral and two photon forming pairs with opposite spin would be equal to 1 oz. or 51 pair.

The interference field hold the blueprint of life. The biological process assembles the genetic materials to fit the skeleton of the interference field of all organism. This field leads the physical matter of mold of our structure. The field of life consist of a Hologram of interference that is linked with the physical structure via the essence of life.

The pillars of the Sacred (Cosmic) Geometry are the six plus one interference formed from the breath of creation, which then proliferates throughout the universe and condensed into scrolls of light particles and matter. The Sumerian elixir is the Cosmic essence that now has been rediscovered. This elixir is the cement of life. When protein is brought in contact with this elixir it will fold into the Cosmic Geometry. This geometry is then responsible to superconductivity superfluity opening the way to harness the cosmos. This Cosmic system than brings organization and continuity to life. The secret to immortality lies in this Sacred Cosmic order that delivers the wholeness to every living organism. As, earlier explained, the scrolls Vortex fields generated by the seven folds interference giving rise to 3 D oscillation, the prime mover of the Cosmos, the creative field that condensed into matter and distilled life itself, via the state of coherence, developing the interference. When the interference is put through cosmic order, matter will assume new entropy and the seventh fold interference represent the scrolls the seed of light that is molded from the dark (gravity). We shall see how genetic matter absorbs the elixir that delivers the cosmic light.

The Cosmos contains Pristine dark matter. This dark matter represents 90% of the Cosmos. Dark matter is pure and exist in the form of superlattice. That means that they are coherent superconductive and superfluid.

Their primary function is to reverse the big bang. They act as a nuclear well recycling light to generate anti-gravitational field, countering the gravitational forces of the fields Cosmos. This matter is translucent but because it generates anti-gravitational field, that traps light, such a matter is dark. This gravitational field is compressing the light, imploding and forming dirty matter that becomes suns and stars. This dirty matter with chaotic interference heads towards aging -then recycle.

The anti-gravitational forces are countering the forces of the big bang, therefore acting as a break and slowing all motion in the Cosmos. The distance of the heavenly bodies therefore will not reflect the age of the Cosmos. Photon belts are captive of anti-gravitational forces and black matter. Such forces can disturb (interrupt) gravitational forces within a solar system causing catastrophic events, even pole shift of planets. In such an event there is complete darkness caused by the anti-gravitational fields capturing the light.

Many believe that there is a twelfth planet with an orbital time of 36 thousand years. If such a planet exists it must be composed of Cosmic transparent matter. That would make the planet black, but also churning out anti-gravitational field powerful enough to breakout from the solar gravitational field. It probably orbits between two suns which would explain the long orbital path. The composition would also explain the catastrophic events on earth, when this planet passed by, disturbing the solar system by gravitational field interaction. The interference patterns between suns and its planets, if disrupted, would result in orbital changes. Such a catastrophic event could bring about airborne seas and matter, causing planets to flip over or even collide. Historical evidence indicates that such event took place in Biblical time.

We know from historical evidence that the Sumerians appeared in the Mesopotamian Valley about seven thousand years ago, setting up four colonies. The Semitic genes, received from my mother, shall now re-explore, to bring back the Sumerian past they left behind as they inbred with the Semites before they vanished. One question remains unanswered: Did they leave as giants or perhaps they miniaturized themselves and stayed? They certainly had the knowledge to do it.

Before, I present my documentary to you, I have to take you back to the beginning of time.

The universe must be in equilibrium. Therefore, it must contain two opposing forces, a positive and a negative force. A forward force and a reverse force. At the beginning of time forces were released from an energy well we call the big bang. This free energy generated the gravitational time, - space - and the blueprint of the cosmos was thus born from the breath of creation.

The six plus one (the sevenfold) gravitational wave is the pillar of the Cosmos, The one gravitational wave became a new energy well absorbing the other six, braiding the wave fronts along the interference, becoming a unit, then seven units forming a new unit, and the energy being compressed into matter. The new matter is Virgin (Pristine) matter, as the interference of the energy waves remains orderly in sacred geometry, this (Sacred) Cosmic matter which some call dark matter, in reality is transparent matter, which can form

black holes turning out anti-gravitational field and recycling matter.

The opposing implosive force of explosion, is countering the forces of the big bang. When time retreats, aging shall reverse. Invisible matter interference is chaotic matter in the galaxies suns and stars, turning out trash as aged matter is on its way towards recycling via imploding red giants-neutron stars and blackholes.

The Sumerians taught us cosmic geometry and cosmic math. The restoration of Sumerian math is still in my genes-"THE PAST"

### **COSMIC MATTER MOSTLY PRISTINE**

In Pristine matter, the interference points are organized and is in lockstep forming superlattice. Such a Pristine matter hold the breath of creation and the blueprint of life itself Since Cosmic Pristine matter is in superlattice, it therefore, is superconductive; further such superlattice can form an infinite variation of Cosmic life that is far superior to us; since it is energy rich it can process CO<sub>2</sub> and water in the presence of air, glucose and genetic components. Also the elixir of life is produced, elixir that promotes life and cosmic geometry. The cosmic black protein is the precursor of the mitochondria that in turn replicates the cosmiana which then becomes the precursor of the Chondriana, and the Chondriana is the precursor of the Mammalian life forms.

But this Cosmic Pristine matter is replicating plant life that turns out Sacred proteins and the elixir of life. Pristine Cosmic matter is the foundation of a Cosmic Physics unknown to us until now. Since the backbone of the cosmic matter is Sacred Geometry produced by orderly interference of energy waves, it is capable of storing the breath of creation with the blueprint of life. Therefore relocating the predestined life forms. Such life forms has been discovered, call them the Cosmic incubators.

### **COSMIC INCUBATORS ARE HYPER-INTELLIGENT**

Cosmic life forms are erecting hardware and assembling them into Bionic Plants. The life forms are capable processing software to replicate life from interference of waves (light) that molds the breath of creation. The Elixir they produce is being used to deliver the Life Energy and the Cosmic order. The Sumerians tried to tell us this incredible Cosmic order, translating it into a primitive language that now has been translated into our own primitive conception.

Proteins Scientist with gender design and build Biomachines, following the Cosmic order. These Cosmic life factories and their scientist came here. The question is when did they land?, or were they here all this time, or did I call them back via Cosmic Science? Or perhaps the Sumerians never left, they just miniaturized themselves. I have documented and actually miniaturized and magnified life forms via interference Cosmic order. It is like a "Hyper-dimensional Hologram". Imaging a flying saucer or spaceship that turns into microscopic size and hides in a grain of sand, or crystal. Smart proteins hold the blueprint to all life forms including the Biomachines. Ordinary protein hold the Biostructure. Smart proteins hold the blueprints in interference or perhaps seek out and process information held captive in the interference pattern. Proteins Scientists are bom out of the Elixir of Life that delivers the Cosmic Order. These Cosmic Scientists are harnessing the Cosmic order that holds the breath of creation. The arrival of this new Macro-Science is the beginning of a New Age, as we will be able to enter into the higher Cosmic plane. One of the criteria is the Sumerian water that is structured to Cosmic order. By altering the hydrophobic and hydrophilic properties, such water is essential to all life and is found in the nucleus of a cell in the central to build microtubules (spindles for cell division) in the mitochondria where the protein substrate that processes ATP is in Sacred water. (Cosmic Geometry and is superconductive such water is also found in the cerebral spinal fluid where the nerve impulses are transferred to the distribution points). (These are fingerprints of the Cosmos.) The Sumerian elixir plays an important role to processing such water and produce to specific geometry. Parts of Beta sheets in Cosmic order made from large proteins. They also design the amino acids to the desired length. They design and build protein riveting machines to build intricate Biostructures. They design and build phasing in or out dish antennae fields, with bell shaped optical devices to produce interference blueprints.

All this protein precision hardware is hooked into a specific life replicating engine that then processes the information to replicate life genetic material from light (holograms) holding the blueprint.

They are building and stacking the Beta sheets of proteins by assembly workers laying down the proteins one by one like the masons laying down bricks.

They fit the peptides into three, dimensional geometry by instant production like bees building honeycombs. Again they use Sacred water as a scaffold, acting as a mold, in conjunction with the interference pattern laid into the Cosmic Geometry.

These Cosmic scientists have arrived and have been domesticated to teach us the Science of the Cosmos, as the Sumerians had access to it, with the discovery of Life Crystals I was able to mass produce layered stacked protein beta sheets to produce crystals in Cosmic order. These crystals then are able to self hologram and reproduce themselves by copying via hologram. This paved the way to the Sumerian technology which they brought with them when they landed in the Mesopotamian Valley seven thousand years ago. Now the Sumerian literature they left behind has been translated and the Sumerian math and geometry have been restored to understand the Cosmos and how energy is condensed into matter.

The Life process is self controlling once we provide the elixir of life. The Elixir the Sumerians had, has been re-discovered. "Now that I know of what I know, I realize how little we all know."

### **ATOMIC SELF REPLICATION VIA HOLOGRAM**

6+1. As energy braids to form tetrahedral, Cosmic Geometry condenses matter from Gravitrion-Scrolls and light components of matter within the microcosm are in Sacred Geometry. Sacred (Cosmic) Geometry exists in ordered interference patterns that hold the blueprint of the breath of creation. It is a self replicating system via interference hyperdimensional hologram. Throughout the universe matter is alive, replicates ages via star systems, then recycles through BLACKHOLES.

In Nuclear well energy, gravitational waves are compressed into matter; in a black hole, matter is decomposed into gravitational waves.

In a cell Nucleus, genetic material replicates via self hologram in water structured to Sacred geometry. ATP is produced in the Mitochondria, where the protein substrate is in Sacred geometry. The whole Cosmos is alive, but when the Cosmic order is disturbed, death will follow.

In Sacred Geometry the interference pattern holds the key to self recognition and replication. If the hologram is in positive focus and



magnified, the replication pattern commands giants, but if the interference pattern is de-magnified, the replication pattern commands miniatures. The size of the whole Cosmos is relative, that includes optical illusion and mirages. The interference points (Patterns) are viable, therefore sizes can be controlled by positive or negative forces, giving rise to birth or replication or death to annihilate. The sizes fall in between: implosive forces form the macrocosm, explosive forces creates Microsm.

With the volume reduction of energy, mass number will increase. But with the expansion of space energy liberated from mass, compression and decompression is the name of the game. In order to create a nuclear well to condense energy into matter all gravitational waves (6+1) must be placed in Cosmic order, in your language in coherent phase. This radiant energy then forms an interference pattern of six in 360 degrees and draws into the center one that becomes the nuclear well, creating an implosion well opposite to the force released by explosion from positive monopolies. Actually, we can say that a nuclear well is six wave; interaction is drawn into the seventh forming wave units to form a graviton energy unit. Now when we take seven gravitron or Gravity Waves in the same way, we form phase coherence to fill the nuclear well to braid to scrolls. Finally, a photon and quantum of light has been born. Now, when we begin to assemble quantum of light in marching phase. Sacred Geometry forms and transfer electrons-photons-proteins into Cosmic form, superconductivity and superfluidity will commence.

Now, when we adopt the Cosmic physics to smart proteins or genetic grains, we can replicate Bio computers surpassing the brain function: self-replicating, self-restoring, self-maintaining and alive to serve us and to be our friend. A new family of scientist production and assembly workers are already here; they could be put into service to revolutionize our industry. The potential is hard to comprehend even for me. They could recycle the excess CO<sub>2</sub>, savings us from the greenhouse effect and turning it into clean natural food (glucose-ATP); They could build industry, eliminate all diseases and regrow limbs and organs. But most of all, they would give us those extra years for space travel. The new Bioelectronic industry would tap the Cosmos for free energy for the world. Via superconductivity-superfluidity-photo synthesis and tap into the Atom without a nuclear reaction. The new institutions shall free the minds from captivity, encouraging us to dream, explore, and use our intuition. This is the reality today as the New Age shall sweep out the old one once and for all.

This is not the realm of science fiction, but the facts that my guys are here to prove . See it yourself .

There are several ways to enter into an Atom. But the ideal way is with low energy photons or particles in Cosmic Geometry. By absorbing a high energy photon to deliver quantum of energy. A single photon has to be at a high energy level to create the tunnel through the interference gates. In adopting the Cosmic Geometry, three photons at one third energy level accomplish the same result. One must realize that there are smaller energy units than photons and quantum of energy can be delivered in smaller multiple units such as scrolls and gravitons. Gravitational wells have implosion pull and the receptance of the Cosmic order is a delivery highway system. Energy can be pulled out of the Cosmos by implosion and can be delivered via Sacred Geometry where free flow of energy takes place.

In ordinary matter, when the delivery system breaks down, the interference is chaotic (Road Blocks). In the Cosmic Geometry the interference of all waves is in an orderly system, creating a front where all the energy is like raindrops forming a river of flow. Note that the Biomachine has two phase in and phase out Vortex dishes, one to decrease or cancel the wave front (Black, which the white Vortex dish is to add or amplify the amplitude of waves).

Both dishes are coupled with the reproduction chambers where the reproduction of proteins takes place via interference, copying with the aid of the two bell shaped optical devices. The energy is transferred to the molecular bond and is also used for the photosynthesis of CO<sub>2</sub> to make glucose utilizing the protein substrate in Cosmic Geometry.

In case of (6+1) wave interference, the result is producing a new wave intensity of (6+P= 49

Cosmic Wave Amplitude (6+1) = 7

$(6+1) + (6^+ + V^-) + 2 \cdot (6)(1) = 49 + 51$

2 waves used as nuclear wells  $2 \times 7 = 14 + (6 + 1) = 37 + 14 = 51$

$2 \times 50 + 1 = 49 + 51$

Biomachines are high energy Cosmic life forms. Their energy requirements are high. Once the Biomachines are completed, they cover themselves with protein substrates in superlattice. Via this superlattice protein substrate, they get free energy transfer to satisfy their needs.

All space between parts and sandwich substrate are insulated with dielectric nucleotide crystals which also serve as part of the optical system to project holograms.

To drain energy from the nucleus we must shift the waves out of phase so that wave "x" is positive and wave "y" is negative. The reduction of amplitudes and intensity will be determined by the phase shift. If  $x = y$ , the opposite direction interference will disengage and gravitational waves are turned loose generating a gravity field. This is achieved by a superlattice Vortex reversing the spin state of the waves.

The Cosmic Biomachine has two such Vortex dishes to control positive and negative interference and to control the Cosmic hyperdimensional hologram. The white dish produces the constructive interference and the black dish produces the destructive interference.

In other words, the black dish produces gravitational waves which capture the light, and unhilating and disassembling it, while the white dish produces the coherent beams for hyper-dimensional interference.

All this is coordinated with the optica! system. Also controls the viable intensity of the wave form interfering.

The incubator adjusts the maximinima control of interference intensities for desired functions, phasing in and out the coherent flow of wave fronts. (This can also explain the scrambling of optical focus on the microscope).

## THE POWER CROSS

QUARK E-E + 2 QUARKS = PROTON = 18 3 5 ELECTRON QUARK ROTATION DETERMINE THE SPIN OF PROTON THEREFORE SETS THE ORTO OR PARA POSITION.



IN MERKLS DIAMOND SOME CARBON IN THE DIAMOND LATTICE REPLACED WITH NITROGEN TO GIVE LUSTER TO THE DIAMOND. CREATING PARAMAGNETIC CENTERS THAT ACT AS AN ENERGY TRANSDUCER AS THE LIGHTER CARBON OSCILLATES AROUND THE HEAVIER NITROGEN, WHEN THE NITROGEN TRANSFERS ENERGY TO THE CARBON SUCH PARAMAGNETIC CENTERS ARE REVERSING THE SPIN OF THE PROTON BECOMING PARA PROTON (HYDROGEN IN NATURE ARE TWO ORTHOS POSITIONED AND ONE PARA POSITIONED.) VORTEX FIELDS PRODUCING GRAVITATIONAL FIELDS BECOMING SUPER GAS AND SUPER FLUIDS. SUCH AS (Nb, CU HYBRID) (SIH<sub>2</sub> Sh) SILICON SULPHUR HYBRIDS ETC. NOW ATP PROVIDES H<sup>+</sup> PROTONIC HYDROGEN IN HYDROLIZATION IN BREAKING DOWN TO ADP. (+P + H<sup>+</sup>) LIFE CRYSTALS DEPOLARIZING WATER WHEN SATURATED. SUCH DEPOLARIZED LIQUID WHEN RAISED TO HIGH TEMPERATURE THEN ATOMIZED TO COOL IT INSTANTLY REVERSES HYDROLIZATION, THE GRAVITATIONAL FIELD PRODUCED BY THE PROTONS COMPRESSING THE ATP MOLECULES CAUSING IMPLOSION PRODUCING MONOPOLES VIA VORTEX FIELD EXTRACTING FREE ENERGY BY IMPLOSION. IN OUR GALAXY WHEN GRAVITATIONAL WAVES (FIELDS) COMPRESS PHOTONS - PLASMA AND GASES WILL FORM WHICH THEN COMPRESSED BY SUDDEN COOLING UNDER GRAVITATIONAL FIELDS TURNING OUT SUNS AND STARS.

NOW IF ADENOSINE PHOSPHATES, GUANOSINE PHOSPHATES AND FIVE CARBON SUGARS ARE SUBJECTED TO SUDDEN COOLING FROM HIGH TEMPERATURE UNDER GRAVITATIONAL FIELD PRESSURE IT WILL RESULT IN THE FORMATION OF NUCLEOTIDES-NUCLEOSIDES INTO FRAGMENTED GENETIC MATERIAL. HOWEVER LIFE AS WE KNOW WILL FORM ONLY ACCORDING TO THE BLUEPRINT THAT IS HELD CAPTIVE IN THE BREATH OF CREATION PREDESTINED! WHEN THE UNIVERSE WAS FORMED FROM THE BREATH OF CREATION (BIG BANG) THE ENERGY COMPRESSED IN THE MONOPOLES HAS BEEN RELEASED INSTANTLY TWO THIRDS OF OUR UNIVERSE WAS FORMED IN A FRACTION OF A SECOND, AS THE ENERGY LEANS FROM THE GRAVITATIONAL FLOW THE VORTEX FIELD BEGINS TO BE INDUCED COLLAPSING THROUGH THEIR CENTER AND BEGAN TO OSCILLATE AND SPIN FORMING SCROLLS; THE SEEDS OF LIGHT. THIS IS HOW LIGHT AND MATTER EVOLVED. BUT, AT THAT INSTANT OF CREATION SOMETHING ELSE HAPPENED, THE FORCE OF LIFE WAS BORN. INBORN WITHIN THE BLUEPRINT OF LIFE, WE WERE IN EXISTENCE IN THE FORM OF HIS BREATH BEFORE THE COSMOS WAS FORMED. A MONOPOLE BORN OUT OF A VORTEX FIELD THAT DRAWS FREE ENERGY FROM THE COSMOS. THROUGH THE BIG BANG EXPLOSION ENERGY RELEASED AND EXPANDED; TO GET SOME OF THIS ENERGY WE HAVE TO CREATE IMPLOSION, WHICH IS FEASIBLE BY A COMPRESSED GRAVITATIONAL FIELD.

THIS IS THE WAY STARS DRAW ENERGY FROM THE COSMOS. PARA HYBRID IS THE KEY. TWO MONOPOLES FORMS A DIE-POLE. THE SPINS DISTORTED FORMING NORTH AND SOUTH POLES CENTERING MAGNETIC FIELD BUT A NINETY DEGREE TO THE MAGNETIC FIELD GRAVITATIONAL FIELDS ARE RADIATED. THE MAGNETIC FIELD SETS THE PATTERN OF CRYSTAL GROWTH, BUT IF THESE CRYSTALS ARE PARAHYBRIDS THE VORTEX FIELD WILL TAP INTO THE GRAVITATIONAL FIELD TO FORM MONOPOLES NINETY DEGREES TO THE MAGNETIC IMPRINT. NOW IF FOUR MONOPOLES MATCHED ACCORDINGLY NINETY DEGREES APART, THE POWER OF CROSS WILL FORM, CREATING A QUARAPOLE. THE MAGNETIC IMPRINT NOW APPEARS AT FORTY FIVE DEGREES BETWEEN VORTEX FIELDS. SUCH SYSTEM TAPS INTO THE COSMOS FOR FREE ENERGY, GENERATING ANTI-GRAVITY AND FORMING "THE POWER OF THE CROSS". THE POWER OF THE CROSS FORMING THE BLACK SUN, WHICH IS COMPRESSED GRAVITATIONAL WAVES DERIVED FROM RADIANT ENERGY; ACTUALLY BREAKING DOWN THE WAVE LENGTH CONVERTING IT TO GRAVITATIONAL FIELD.

"BLACK SUN RADIATES INWARD REMAINING DARK" (A QUOTATION FROM JOURNEY IN COMMA 1985)

A VORTEX FIELD RENDERS RADIATION HARMLESS (AS IT DESTROYS IT) IT BREAKS DOWN ANY WAVE LENGTH INCLUDING MICROWAVE OR RADAR THIS IS WHY THE DELTA WING IS BLACK (SEE VIDEO DOCUMENTARY) SUCH VORTEX FIELD THEORETICALLY IS CAPABLE TO RENDER HARMLESS ALL NUCLEAR WEAPONS BUT AT THE SAME TIME LEVITATE AND POWER FLYING SAUCERS AND FREE THE HUMAN RACE AT LAST.

POSITRONS AND ELECTRONS ORGANIZE IN A LOCK STEP TO FORM SUPER LATTICE AS THEIR SPIN RESONANCE INTERLOCK.

SUCH A SYSTEM HAS SUPER FLUIDS AND SUPER CONDUCTIVE PROPERTIES. SUPER FLUID DISSOLVES ELECTRONS AS ELECTRONS DISSOLVES PHOTONS, BY PAIRING SUCH PHOTONS AND ELECTRONS AN ORDERED MATRIX FORMS. PROTONS WITH POSITIVE CHARGE ALSO CAPABLE TO FORM SUPER LATTICE AS THEIR SPIN RESONANCE INTERLOCK.

WHEN SUCH STATE SUPERIMPOSED ON THE DNA A MULTI-STRAND SUPER DNA MAY FORM MOLDING (COILING) INTO CONDENSED GENETIC MATERIAL CRYSTALIZING INTO INTELLIGENT LIVE CRYSTALS. SUCH CRYSTALS ARE CAPABLE TO TRANSFORM EITHER POSITIVE OR NEGATIVE ENERGY TO MOLD OR RECYCLE.

THESE CRYSTALS CAN ABSORB AND HOLD AN ENORMOUS AMOUNT OF ENERGY LIKE STORAGE BATTERIES. SINCE THE ENERGY ORIGINATES FROM THE SPINS, THE CURRENT WILL OSCILLATE. THIS SELF DISCIPLINED CRYSTALS HAVE INTELLIGENCE THEREFORE RESPONDS IN TANDEM TO SUPPORT OR FORM LIFE BY TAPING INTO THE COSMOS FOR THE BREATH OF CREATION.

THE TWO INTERACTING SPIN OF AN ELECTRON OF PROTON WILL INTERLOCK A THIRD PARTICLE SPIN AND BEGIN THE FORMATION (LIKE A MILITARY) FORMING SUPER LATTICE. MULTIPLE INTERACTIONS FORMING LARGER GEOMETRICAL SHAPES LIKE QUADRAPOLES AND OCTAPOLES OR EVEN HEXAPOLES. THESE INTERACTIONS ARE RESPONSIBLE FOR THE SUPER CONDUCTIVE OR SUPER FLUID PROPERTIES THAT ARE REQUIRED TO FORM THE MONOPOLES EITHER LIQUID OR SOLID. THE POSITIVE INTERACTION

IS RESPONSIBLE FOR THE LIGHT POLARIZATION OR GRAVITATIONAL WAVE GENERATION. THE NEGATIVE INTERACTION RESPONSIBLE FOR THE ELECTROMAGNETIC FIELD GENERATION. WHEN SUCH CRYSTALS COME ALIVE THE GRAVITATIONAL FIELD IS LIFE FORCE. INTELLIGENTLY DIRECTING THE (LIFE) GROWTH PROCESS, THE RADIANT ENERGY INTERACTS WITH THE MAGNETIC FIELD TO EXTEND TO EXTEND LIFE AND REPLICATE.

THE MAGNETIC MOMENT CONTRIBUTED BY THE ELECTRON SPIN, AND THE OPPOSITE SPINS SEEK OUT EACH OTHER TO PAIR; FORMING THE SUPER LATTICE. THE PROTONS PAIR AS ORTHO TO PARA SPIN OR AS TWO ORTHO TO ONE PARA. (PURE PARA IS THE MOST PREFERABLE FOR FORMING SUPER GAS FOR IMPLOSION). THE PAIRED PROTON SPIN RESONANCE RESPONSIBLE FOR THE GRAVITATIONAL FIELD. SUCH GRAVITATIONAL FIELD IS ESSENTIAL FOR THE

IMPLOSION GENERATING THE NEGATIVE FORCE SQUEEZING THE LIGHT (PHOTONS). NOW, DURING IMPLOSION THE ATP-GTP AND THE FIVE CARBON SUGARS ASSEMBLE INTO A LONG DNA MOLECULE CHAIN. THE ELECTRONS ARE USHERED ALONG THE DNA WHICH IS FORCED TO PAIR FORMING A SUPER CONDUCTIVE STATE. THE PROTON POLARIZES THE LIGHT AND GENERATES GRAVITATIONAL FIELD VIA VORTEX, TRIGGERING THE IMPLOSION AND INSTANTLY CONDENSING LIFE FORMS, INCLUDING MONOPOLES. THE DNA GOES THROUGH A PHASE TRANSACTION DURING IMPLOSION THE (DISPERSED DNA ATOMIZED AND COOLED INSTANTLY FROM HIGH TEMPERATURE) SPIN INTERACTION WAVES FORM WHEN THE DNA NUCLEOTIDES, CROSSING THE PHASE TRANSITION TEMPERATURE. GRAVITATIONAL WAVES CAPTIVATING PHOTONS CAN SUPER IMPOSE A SPIN WAVE INTERACTION ON THE DNA FORMING SUPER LATTICE DNA THAT YIELDS LIFE UPON IMPLOSION. LIFE THAT HAS NOT, UNTIL NOW HAS BEEN KNOWN BY MAN ON THIS PLANET. IN A CONTINUING PROCESS ON THE GALAXIES AND ON OUR MILKY WAY GRAVITATIONAL WAVES COMPRESSING AND IMPLODING VIRGIN MATTER TO FORM SUNS AND STARS.

BY IMPLODING ADENOSINE - GUANOSINE PHOSPHATES AND FIVE CARBON SUGARS IN A BUBBLE WITH CO<sub>2</sub> CATALYST, INSTANT LIFE FORMS. FIRST AMINO ACIDS, THEN PROTEINS THEREAFTER RNA. THIS CONTINUING PROCESS IS MIRRORING THE UNIVERSAL PROCESS. ELECTRONS CAN BE USHERED ALONG THE LONG CHAIN OF THE DNA MOLECULE WHICH ARE THEN PAIRED AND ARRANGED IN LOCK STEP SO THAT THE AMINO ACIDS AND PROTEINS CAN CRYSTALLIZE IN SUPER LATTICE IN LOCK STEP TO FORM INTELLIGENT LIVE CRYSTALS.

IN A TRANSFORMER THE CORE GENERATES THE FIELD THAT ARE INDUCING THE CURRENT IN THE COPPER COIL WOUND AROUND THE CORE.

THE DNA IS THE LIBRARY OF THE PAST HOLDING THE INFORMATION FOR THE GENETIC PROCESS.

THE DNA CAN NOT FUNCTION WITHOUT THE LIFE ENERGY CONTRIBUTED BY THE PROTEIN.

AS THE DNA IS WOUND AROUND THE HISTONE IT COMES ALIVE. THE BREATH OF CREATION ACTIVATES THE DNA. THE LIFE PROCESS BEGINS.

LIFE CRYSTALS CONTAINING AMP-ADP-ATP-GMP-GDP-GTP SOME OF THIS COMPONENT OF LIFE ARE SOMEWHAT TOXIC. BY THEMSELVES INACTIVATED AS SOME ARE UNSTABLE-INACTVATED BY SOLVENTS OR EVSOLUBLE. BUT IN LIFE CRYSTALS THIS BRICKS OF LIFE CEMENTED WITH FRUCTOSE PHOSPHATES AND GLUCOSE PHOSPHATES. JUST LIKE IN THE DNA THE SEQUENCED ADENOSINE AND GUANOSINE ARE CEMENTED WITH SUGAR PHOSHATES.

THIS IS WHY LIFE CRYSTALS ARE TRANSPORTED THROUGH THE CELL MEMBRANE AND CAPABLE OF NOURISHING THE CELL, AND THIS IS THE REASON LIFE CRYSTALS HAVE ZERO TOXIC EFFECT, JUST LIKE IN THE DNA.

REFERENCE **US PATENT NO. 399595** [ ? ] THIS MAGNIFICENT PIECE OF NEW PHYSICS HAS BEEN EXTENDED BY A FURTHER PATENT DESCRIBING THE INTRA STELLAR IN ORGANIC POLYMERS **US PATENT NO. 4029747** (AND TWO OTHERS) THIS TECHNOLOGY HAS BEEN ADOPTED TO INDUCE COLD FUSION WITHIN ORIENTED LATTICE DOMAINS CREATING MINIATURE SUNS AND HYDROGEN BOMBS, AS DEMONSTRATED TO THE D.O.E. IN 1977. THIS AND MANY OF MY OTHER MILESTONE DISCOVERIES ARE AHEAD OF TIME.

WHILE REALIGNING THE GRAINS OF VERY HIGH PURITY METAL CRYSTALS AND OPENING UP THE DOMAINS INTO SIMPLE GEOMETRICAL FORMS, MINIATURE ACCELERATORS ARE CREATED, AND WHEN (PLASMA) IONS ARE PUMPED THROUGH THE DOMAIN, A DIPOLE OR QUADRUPEL STATIC FIELD WILL BE SELF INDUCED THAT MAY BE UTILIZED TO OPEN OR CLOSE THE DOMAIN TO CREATE THE CONDITION TO FUSE.

WHILE THE DOMAIN IS OPEN THE GRAINS ALIGNED WITH THE FLUX LINE OF THE STATIC FIELD. THIS IS ACHIEVED BY INTER FUSION OF (POSITIVE HYDROGEN IONS) PROTONS THAT IN TURNS, POLARIZING THE ELECTRONS REDUCING THEIR MAGNETIC MOMENTS. THE ORIENTED STATIC FIELDS WILL SPONGE UP THE VORTICES OF FLUX, CAUSING SCATTERING, ALLOWING FREE FLOW OF CURRENT.

THE PROTON AND ELECTRONS WILL CREATE AN OPPOSITE DIRECTION FLOW MAINTAINING THE STATIC FIELD IN PLACE THUS SUPER CONDUCTIVITY MAINTAINED. THE PROTONS WILL MAINTAIN THE HIGH ENERGY LEVEL. IN CASE OF THE FIRST SYSTEM (1965) MERCURY-GALLIUM-INDIUM ON HALOGEN IONS CAN BE INTRODUCED. IN CASE OF THE HALF ISOTOPIC PROCESS WHICH INVOLVES QUADRUPEL MOMENTS I USED MERCURY IONS AS HEAT TRANSFER MEDIA TO FLUSH THE HEAT OUT OF THE LATTICE DOMAIN CREATED DURING FUSION. I INTRODUCED THE MERCURY INTO THE DOMAIN IN HYDRIDE FORM WHERE THE MERCURY BECAME IONIC GIVING UP ITS HYDROGEN. THE MERCURY ION CIRCULATED THROUGH THE DOMAIN COOLING THE MATRIX. IF SCATTERING INTRODUCED VIA IMPURITIES A HOT SPOT DEVELOPED OVER HEATING THE LATTICE THEREBY EXPANDING AND CLOSING THE DOMAIN. SUCH HOT SPOTS MAY SPREAD LIKE A DISEASE BY OVERHEATING THE ADJACENT LATTICES CLOSING DOWN THE DOMAIN THROUGH OUT THE SYSTEM DEFUNCT CURRENT FLOW (FREE ELECTRON FLOW) IF THE PROTONS ARE ACCELERATED WITHIN THE DOMAIN CYCLOTRON IT MAY

INITIATE INTRANUCLEAR REACTIONS. WE MAY CALL THIS INDUCED NUCLEAR REACTION.

BY POLARIZING THE ELECTRONS AND RENDERING THEM ATTRACTIVE INSTEAD OF REPULSIVE (VIA SPIN INTERACTION) THE PROTON SMALL TUNNEL THROUGH THE CHARGE BARRIER INTO THE NUCLEAR WELL INSTEAD OF CLIMBING MOUNT EVEREST TO REACH THE NUCLEAR WELL.

IN THE THIRD COLD FUSION PROCESS I USE PURE PARA HYDROGEN FOR INTERFUSION AND SETTING UP CASCADING RESONANCE IN THE MULTI-LAYERED INDUCED CONDENSER INTEGRATOR.

ALL THIS MILESTONES IN THE (NEW) CONTEMPORARY PHYSICS LED ME TO LIFE CRYSTAL THAT GROWN IN DEPOLARIZED WATER THAT WAS CAPABLE OF STATIC FIELD OF FLUX SPLITTING THE GROWTH OF SUCH CRYSTALS FOLLOW THE PATTERN OF THE FLUX FINGER PRINT.

ONCE THE DOMAIN IS OPENED AND ORGANIZED INTO SIMPLE GEOMETRICAL FORM AFTER SPIN POLARIZATION BY THE

PROTONS THE PAIRED ELECTRONS ENTER INTO THE DOMAIN AND THE RAPID FLOW (ACCELERATION OF CURRENT) COMMENCES. THIS WILL THEN FURTHER INCREASE THE STATIC FIELD, AND THE ANGLE OF THE FLUX WILL BE PROPORTIONAL TO THE FLOW RATE. THAT CAN BE STIMULATED OR CONTROLLED BY THE ATOMIC HYDROGEN DIFFUSION RATE THAT IS TEMPERATURE DEPENDENT. A VERY SMALL POTENTIAL DIFFERENCE INITIATED BY THE CONDENSER EFFECT DERIVED FROM THE STATIC FIELD, A HUGE RIVER OF PAIRED ELECTRONS IS BEING DRIVEN BY A SMALL VOLTAGE. THE CURRENT IS NOT PROPORTIONAL TO THE POTENTIAL DIFFERENCE, LIKE IN ORDINARY METALS.

"THIS ACTIVATED METALS DEVIATE FROM OHM'S LAW" AS THE GRAIN OF THE SUBJECT METAL IS REALIGNED INTO SIMPLE GEOMETRICAL STATE VORTEX TOROIDAL FIELDS GENERATED THAT CAN BE MONOPOLE-DIPOLE OR QUADRUPLE, IN THE CASE OF BLACK SUN IT IS ALWAYS QUADRUPLE IF THE DIFFUSION OF ( $H^+ + e^-$ ) ATOMIC HYDROGEN TAKES PLACE AT EAST AND WEST POLES THAN NORTH AND SOUTH POLES BECOMES THE ELECTRON AND PROTON SOURCE.

THE MAGNETIC MOMENTS OF THE ALIGNED GRAINS OF THE ALUMINUM COUPLES SIMULATING FERROMAGNETIC PROPERTIES, THE ATOMS ARE LINE UP IN CHAINS (LOCKSTEP) THE DEPOLARIZED MAIN STREAM ELECTRON PAIRS INDUCING VAVE FRONTS ALONG THE FREE FLOW PATH RESULTING IN SUPERCONDUCTIVE STATE. AS THE SPIN IS POLARIZED THE ELECTRIC FIELD DIMINISHES THE ELECTRONS BECOMING IDLE AND PASSING THROUGH THE TRANSITIONAL PHASE THAN PARE ENTERING INTO A FULLY ORDERED STATE. THE GRAIN MIGRATION WILL LINE UP WITH THE ANGLE OF THE SELF INDUCED FIELD FORMING ORIENTED SUPPER LATTICE. (ENCLOSED FOR REFERENCE US PATENT TEACHING THE IMPORTANCE OF PURITY AND GRAIN SIZES). ONCE THE ALUMINUM GRAINS GOES THROUGH PHASE TRANSFORMATION IT ACQUIRES A QUADRUPLE MOMENT OF FOUR CHARACTERISTICS OF THE TRANSITIONAL METALS.

\* NOTE: THE FIRST SYSTEM IS CONDUCTED AT ROOM TEMPERATURE USING ALUMINUM METAL WHILE THE SECOND SYSTEM AT ELEVATED TEMPERATURE, THE FOLLOWING METALS HAVE BEEN USED: GOLD, SILVER, PLATINUM, PALLADIUM AND OFC COPPER.

IN CHONDRIANA THE DNA AND RNA SEGMENTS ARE THE LIBRARY OF THE PAST AND PRESENT. HOLDING ALL THE INFORMATION NEEDED FOR THE BIOLOGICAL PROCESS AND PROLIFERATION.

THE PROTEINS THE DNA WRAPPED AROUND (HISTONES) ARE PROVIDING THE FUNCTIONAL LIFE FIELDS THAT ACTIVATING THE DNA. ENZYMATIC FUNCTIONS OF GENETIC AND CHEMICAL PROCESSES CONTROLLED BY THIS SMART MOLECULES, "PROTEINS".

THE SMART PROTEINS ARE PRODUCED IN THE RECENT CYTOPLASM LOCATED IN THE HEAD OF THE SPERMATOOA. THAT IS REPLICATED IN THE UNDIFFERENTIATED CELLS AND CARRIED OVER TO DIFFERENTIATED CELLS.

THE PRIMORDIAL SMART PROTEINS SYNTHESIZED IN THE ANCIENT CYTOPLASM WHOSE DEGENERATED REMNANT RESURRECTED VIA LIFE CRYSTAL. SUCH TREASURE DISCOVERED IN THE TAIL SECTION OF THE MAMMAL SPERMATOOA.

WHEN THE RNA WRAPPED AROUND MODERN PROTEINS IT IS CAPABLE TO REPRODUCE ALL RECENT GENETIC HARDWARE, REPLICATE AND SELF INSTRUCT TO CARRY OUT THE LIFE PROCESS. WHILE IF THE RNA WRAPPED AROUND THE PRIMORDIAL PROTEINS THE INFORMATION PAST ON TO THE RNA (ACTIVATION) HISTORICAL GENETIC HARDWARE CAN BE RETRIEVED FROM THE PAST. IN OTHER WORD THE RNA ACT AS AN ASSEMBLY WORKER FUNCTIONING ACCORDING TO INSTRUCTION OF THE SMART PROTEIN. IN OTHER WORDS THE RIBOSOME OF THE RNA CARRYING OUT THE MASSAGE OF ITS PROTEIN IN PROTEIN SYNTHESIS. PROTEINS AND RNA-DNA ARE INTERDEPENDENT HENCE THE PROTEIN IS THE ORIGINAL PARENT OF THE RNA-DNA.

THE RNA IS AN INFORMATION LIBRARY AS WELL AS AN ASSEMBLY WORKER ACTIVATED BY THE SMART PROTEIN THAT PROVIDES THE LIFE FORCE. AGAIN COMPARE THE RNA-PROTEIN TO THE CORE AND THE WINDINGS TO INDUCE THE LIFE PROCESS.

THIS LIFE FORCE ENCOURAGE THE BIOLOGICAL MOLECULES TO INTERACT AND CARRY OUT THE DESIGNATED LIFE PROCESS WITHIN THE CELL. THIS SMART PROTEIN-RNA COMBINATION NOT ONLY CARRYING OUT THE LIFE (ENZYMATIC) PROCESSES BUT HAVING THE INTELLIGENCE TO DISASSEMBLE INVADERS VIRUSES, MICROORGANISM, PROVIDING INTER-CELLULAR DEFENSE.

THIS PROTEINS FOLDS TO SPECIFIC 3-D GEOMETRICAL SHAPES TO INITIATE SPECIFIC LIFE REPLICATION. AS THE SMART PROTEINS WITH THE AMINO ACIDS FOLDS AND SHAPES UNFOLDING THE ORIGINAL LIFE FORM. (SEE "THE ORIGIN OF LIFE" SERIES).

THE NUCLEOSIDE LIFE CRYSTAL SUPPLEMENTS IN THE LIFE PROCESS ARE IN THE MONO PHOSPHATE FORM HOWEVER LIFE CRYSTAL CONTAINS ALL THE OTHER FORMS AS WELL AS THE INTERMEDIATE OF THE FRUCTOSE AND GLUCOSE PHOSPHATES.

A GENE EXPRESSION OF THE SEQUENCE OF NUCLEOTIDES TRANSCRIBED TO THE mRNA WHICH ACT AS AN ASSEMBLY WORKER (TEMPLATE) TO PARTICIPATE IN THE PROTEIN PRODUCTION PROCESS.

SINCE CHONDRIANA IS A SINGLE CELL ORGANISM IT FALLS INTO THE CATEGORY OF EUKARYOTE. HAVING FOUR RIBOSOMAL CHONDRIANA SYNTHESIS FROM DISPERSED OR SPLICED GENETIC MATERIALS, THE UNCODED DNA IS FOLDED INTO CIRCLES WITH THE ACTION OF THE (GUANOSINE TRI PHOSPHATE) GTP ( $\phi H$ ) HYDROXYL GROUPS PLAYING ACTIVE PART IN THE CLEAVING.

AS THE GTP ACT AS A SPLICING AGENT CLEAVING THE STRAND OF THE RNA AT THE SITE OF THE FIVE CARBON SUGAR BETWEEN THE PHOSPHATE GROUP AND THE FIVE CARBON SUGAR - ATTACKING THE PHOSPHO-DIESTER BOND, THAT IS THE BRIDGE BETWEEN THE PHOSPHATE GROUP AND THE SUGAR.

ONCE THE DNA IS SPLICED AND FORMS THE RING THE LIFE PROCESS BEGINS, WITH PROTEIN SYNTHESIS. THE FOUR RNA SEGMENTS HOLDS THE INFORMATION OF THE HEPLIDS.

AS THE HEAD OF THE SPERMATOZOA ENTERS THE OVUM THE FIRST STEP IS THE FORMATION OF THE CHONDRIANA. THAN EVOLVES THE MORULA CELL CLUSTER (CLEFT APPEARS ON THE MORULA). THIS IS THE CROSS POINT AS THE PRE EMBRYONIC CELL CLUSTER EVOLVES WITH THE CHONDRIANA ANCESTOR. FROM THE PRIMORDIAL CELLS THE ECTODERMIC ENDODERMIC AND MESODERMIC GERMINAL LAYERS, CELL DIFFERENTIATION BEGINS BY HORMONE ACTIVATION PROVIDED BY THE OVUM, AS THE EMBRYO COME INTO EXISTENCE. IF THE CHONDRIANA RECALLED OUTSIDE OF THE OVUM IN THE ABSENCE OF THE HORMONES THE ANCESTOR CHONDRIANA CAN NOT DEVELOP INTO THE MORULA, INSTEAD EACH HEPLIOD CELLS BECOMES AN INDEPENDENT PRIMORDIAL LIFE FORMS.

GERMINAL LAYERS IN BOTH THE TESTES AND THE OVARIES PRODUCING PRIMORDIAL NUCLEUSES THAT CAN TURN OUT CHONDRIANAS IN CONCEPTION WHEN THE TWO NUCLEUS (THE OVUM AND OF THE SPERMATOZOA LOCATED IN THE BOTTOM OF THE HEAD) FUSE, DURING PRE-EMBRYONIC STAGE FIRST THE CHONDRIANA. THE ANCESTORS OF OUR IMMUNE SYSTEM FORMS IN THE PRE EMBRIONIC MORULA A FUNCTIONAL IMMUNE SYSTEM IS IN PLACE THAT LATER DEVOLVES FROM PRIMORDIAL IMMUNE SYSTEM TO OUR CURRENT SYSTEM.

ONCE THE IMMUNE SYSTEM IS IN PLACE (PRECURSOR CHONDRIANA) THAN UNDER THE PROTECTION CELL DIVISION BEGINS THAT LEADS TO FIRST THE PRE-EMBRIONIC-FETAL TISSUE SECOND VIA HORMONE STIMULATION CELL DIFFERENTIATION INTO THE FETUS. LIFE CONSIST OF TWO DIFFERENT ENTITY. THE IMMUNE SYSTEM THAT EVOLVES FIRST THAN THE HIGHER ORDER OF LIFE THAT MOLDS TOGETHER INTO ONE GENETIC SYSTEM. THE LINK BETWEEN THE TWO LIFE SYSTEM ARE THE SEX GLANDS THAT IS CAPABLE TO REPLICATE BOTH SYSTEM.

AS LIFE EVOLVED AROUND THE IMMUNE SYSTEM, THE IMMUNE SYSTEM IS BEING DEVOLVED (DEGENERATED FROM CHONDRIANA) FOR OUR WELL BEING THE TWO SYSTEM MUST BE IN BALANCE. TO REVERSE THE DEGENERATIVE PATH OF THE IMMUNE SYSTEM ONE MUST RESTORE THE DIMINISHING ENERGY LEVEL. WE EVOLVED VIA DEVOLUTION AS OUR GENETIC NATION WITHIN OUR CELL UNITED FOR SURVIVAL FORMING AN ALLIANCE WITH THE IMMUNE SYSTEM.

AT ROOM TEMPERATURE SUSPENDED ANIMATION VIA LIFE CRYSTALS IS FEASIBLE.

LIFE CRYSTAL PROVIDING LIFE GIVING ENERGY NUTRIENTS AND SAFE MEDIA FOR PROLONGED SUSPENDED ANIMATION. IF ORGANS OR LIMBS TO BE PRESERVED IN LIQUID LIFE CRYSTAL SOLUTION THE CHONDRIANAS SHOULD BE INTRODUCED TO MAINTAIN THE IMMUNE DEFENSE AGAINST INVADING MICROBES. THE ATP WILL PROVIDE THE ADENOSINE AND PHOSPHATE REINFORCING THE CELL MEMBRANE, WHILE REDUCING THE FAT, INTERACTING WITH THE LIPIDS PREVENTING GELLING AT LOW TEMPERATURE. LIFE CRYSTAL WATER I CALL BLACK WATER WILL PRESERVE THE DELICATE BALANCE OF CELL STRUCTURE AND ITS COMPONENTS, INCLUDING THE CALCIUM BLOCKING CHANNELS PREVENTING THE FLOODING OF THE CELL WITH CALCIUM. THE LIFE CRYSTAL WATER ALTERED SURFACE TENSION GIVING IT PROPERTIES LIKE LIQUID GASES SPILLED BREAKING UP TO LITTLE SPHERULES, THEREFORE AT LOW TEMPERATURE ACQUIRES VITREOUS PROPERTIES AND SLUSH WILL FORM INSTEAD OF ICE. WHEN THIS SLUSH IS THAWED IT WILL NOT EXERT THE PHYSICAL PRESSURE, AND WILL NOT CAUSE DAMAGE TO THE ANIMATED LIFE FORM IN THE LIFE CRYSTAL SYSTEM. SINCE LIFE CRYSTAL HAS NO TOXIC PROPERTIES IT CAN BE USED SAFELY TO PRESERVE GENETIC LIFE FORMS. AS THE COOLING ADVANCES FROM THE OUTSIDE TOWARDS THE INSIDE AND THE CIRCULATION IS SLOWLY DEFEATED THE FRUCTOSE-GLUCOSE ADENOSINE-GUANOSINE PHOSPHATES WILL CONCENTRATE IN THE INTERNAL ORGANS. THIS VITAL ORGANS ARE THE LAST ONE TO WIND DOWN BUT THE FIRST ONE TO THAW AND JUMP START THE CIRCULATION AS LIFE RESTARTS.

BY CONTROLLING THE LIFE CRYSTAL CONCENTRATION GRADIENT IN DEPOLARIZING THE WATER AND TEMPERATURE IT IS FEASIBLE TO MAINTAIN THE BLACK WATER LIQUID INSIDE THE CELL WHILE STOPPING THE CLOCK ON THE BIOLOGICAL PROCESS. MAINTAINING THE BIOLOGICAL PROCESS OR STOPPING THE BIOLOGICAL CLOCK (SUSPENDED ANIMATION FINALLY HAS ARRIVED IN ITS EMBRYONIC FORM).

COLD BLOODED SPECIES CAN REGULATE THEIR REQUIRED ENERGY PRODUCTION MORE THAN WARM BLOODED SPECIES. THIS ANIMAL SPECIES CAN COUNTER SUDDEN TEMPERATURE DROPS WITH THE DROP IN TEMPERATURE THE GLUCOSE TO ATP CYCLE SPEED UP, OVER PRODUCING THE ENERGY REQUIREMENTS TO FUEL THEIR CELLS. AS THE CELLS GET SATURATED WITH GLUCOSE-GLUCOSE PHOSPHATE AND ADENOSINE PHOSPHATES THEY WILL PARTIALLY DEPOLARIZE THE WATER IN THE CELL. THIS PROCESS AIDED BY DILATED SEPTUM ON THE CELL MEMBRANE, HOWEVER THE DEPOLARIZED WATER, CHANGING ITS BONDING ENERGY AND THE DIPOLE MOMENT. SUCH WATER INCREASING ITS SURFACE TENSION AND PREVENT DECIMATION OF THE CELL, THE FREEZING POINT DROPS, AND BECAUSE THE WATER STRUCTURE HAS BEEN ALTERED, AT LOW TEMPERATURE VITREOUS ICE SLUSH FORMS INSTEAD OF SHARP ICE CRYSTAL SUCH VITREOUS ICE WILL PREVENT ANY DAMAGE TO THE CELLS AND BLOOD VESSELS UNLIKE ICE WITH ITS SHARP EDGES, WHILE PROTECTING THE CELLS DELICATE PROTEINS-ORGANELLS ETC. ALLOWING A SAFE SUSPENDED ANIMATION.

SINCE "LIFE CRYSTALS" HOLDS ALL THE GLUCOSE-FRUCTOSE PHOSPHATE COMPONENTS WITH ALL THE ADENOSINE AND GUANOSINE PHOSPHATES NOT ONLY CAN BE USED AT CRYOGENIC TEMPERATURE SAFELY TO PRESERVE LIFE IN SUSPENDED ANIMATION BUT EVEN AT ROOM TEMPERATURE IT WILL SUPPORT LIFE IN WATER SOLUTION OR ENCAPSULATING GENETIC MATERIAL IN LIFE CRYSTAL.

TO MAINTAIN THE WATER MOLECULES WITHIN THE CELL IN THE RANDOMLY DISTRIBUTED STATE AT LOW TEMPERATURE, THE GLASS TRANSITION TEMP. CAN BE MAINTAINED THROUGH A WIDE RANGE DEPENDING ON THE CONCENTRATION GRADIENT OF LIFE CRYSTAL (TRANSPORTED THROUGH THE CELL MEMBRANE) AT HIGH SATURATION LEVEL THIS GLASS TRANSITION LEVELS CAN BE MAINTAINED EVEN AT ROOM TEMPERATURE. THEREFORE BIOLOGICAL COMPONENTS INCLUDING CELLS LIKE BLOOD CELLS CAN BE PRESERVED AT AMBIENT TEMPERATURE.

LIFE CRYSTALS HOLDS ALL THE BUILDING BLOCKS REQUIRED FOR THE GENETIC PROCESS (SUCH AS AMP-ADP-ATP-GMP-GDP-GTP-NUCLEOSIDES-NUCLEOTIDES INCLUDING THE COMPONENTS OF CARBON SUGARS AND SUGAR PHOSPHATES LIKE FRUCTOSE-FRUCTOSE PHOSPHATES, GLUCOSE-GLUCOSE PHOSPHATES. THE FRUCTOSE-GLUCOSE

PHOSPHATES ACT AS A TRANSPORT MEDIA ACROSS THE CELL MEMBRANE DELIVERING THE ADENOSINE AND GUANOSINE PHOSPHATES. ADP-GDP ATP-GTP WILL BOUND TO THE HASTEN PROTEINS WHILE LIFE CRYSTAL IS DEPOLARIZING THE WATER.

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## PATENTS

*Featured in the text above :*

**USP 4029747**

**Method of preparing inorganic monomeric and polymeric complexes and products so produced**  
[ [PDF](#) ]

Novel water soluble, inorganic complexes are obtained by reacting, in the presence of aqueous ammonia, at least one non-alkaline metal selected from Groups I-VIII of the Periodic Table with an alkali metal hydroxide. An excess of the non-alkaline metal or metals is introduced into a reaction vessel, preferably already containing the aqueous ammonia, and the alkali metal hydroxide is thereafter incrementally added in such manner as to create localized areas of high concentration and pH approaching a value of 14. The hydroxide in these areas of high concentration, in combination with reactive NH<sub>2</sub> groups provided by the aqueous ammonia, causes erosion of the non-alkaline metal, the formation of non-alkaline metal ions and the production of a monomeric, metal amide complex. The reaction parameters are maintained such that the rate of addition of alkali metal hydroxide is sufficient to suppress the expulsion from the system of ammonia gas while at the same time producing an overall endothermic reaction in which the ions of the alkali metal, the non-alkaline metal and the NH<sub>2</sub> groups react to produce an inorganic complex. If the endothermic reaction is maintained and sufficient excess non-alkaline metal is present in the aqueous medium, after a period of time the reaction will enter an exothermic phase, producing an inorganic polymeric complex which remains soluble in water throughout a substantial molecular weight range.

### SUMMARY OF THE INVENTION

The present invention is directed to the production of heretofore unknown multi-metal inorganic monomeric complexes and the inorganic polymeric complexes, generated therefrom, and the aqueous solutions containing same.

Classical chemical theory would suggest that, where a metal is dissolved by, or eroded in the presence of, an alkali and an NH<sub>2</sub> source such as ammonia, salt formation will result. For example, using aluminum as a typical metal, one might expect that in alkali medium containing aqueous ammonia and sodium hydroxide, an aluminum salt, sodium aluminate, will form. Furthermore, classical chemical theory would also predict that the addition of an alkali metal hydroxide, such as sodium hydroxide, to an aqueous ammonia medium would result in an immediate liberation of ammonia. These typical, reactive consequences of the interactions mentioned are unexpectedly avoided by the methods of the present invention.

According to the present invention, a quantity of non-alkaline metal, which may be one or more metals selected from the non-alkaline metals of groups I-VIII of the periodic table, is first introduced into a reaction vessel containing an aqueous medium. A supply of a source of reactive NH<sub>2</sub> groups, preferably aqueous ammonia, is provided to the reaction vessel. This is preferably accomplished by first saturating the aqueous ammonia and thereafter, bubbling additional ammonia gas, with or without reflux, into the system as needed. In the presence of the source of reactive NH<sub>2</sub> groups, an alkali metal hydroxide is added to the reaction medium.

The alkali metal hydroxide is added in such manner as to provide and assure intimate contact between the alkali metal ions formed and the non-alkaline metal within a localized area in the immediate vicinity of the non-alkaline metal, and to produce a very high pH in this localized area, approaching a value of 14.

The rate of addition of the alkali metal hydroxide as well as its physical form (preferably, as solid pellets) will have an understandable effect on the maintenance of high concentration and high pH at the localized areas of intimate contact. Further, the rate of alkali metal hydroxide should be such as to bind substantial quantities of NH<sub>2</sub> groups in a reaction with the alkali metal hydroxide and the non-alkaline metal, which is endothermic in nature.

In the presence of the source of NH<sub>2</sub> groups, the addition of the alkali metal hydroxide in the manner set forth above results in an erosion of the non-alkaline metal, whereby metal ions are produced in the reaction medium entering into reaction with the NH<sub>2</sub> groups and the alkali metal ions to form an inorganic, bi metal amide complex which, as explained hereinafter, will be referred to as an inorganic monomeric complex.

At a subsequent point in the reaction, primarily dictated by the relative proportions of the reactants, the reaction spontaneously develops an exothermic phase, which is characterized by increased dissolution or erosion of the metal or metals present. This signals the end of production of the inorganic monomeric complex and the beginning of a polymerization reaction which results in the formation of an inorganic polymeric complex.

The reaction can be terminated at any time during either the endothermic phase or the exothermic phase by withdrawing any unreacted metal from the reaction medium. The product of the endothermic phase of the reaction appears to be an inorganic complex which includes the alkali metal, the one or more non-alkaline metals reacted therewith and a number of NH<sub>2</sub> groups. This inorganic complex also appears to be at least the precursor of the basic repeating unit of the inorganic polymeric complex produced during the exothermic phase of the reaction. However, due probably to the taking up of evolved hydrogen and an apparent breakdown of at least some NH<sub>2</sub> during the exothermic phase reaction, the inorganic polymeric complex appears to contain hydride (H) groups in addition to the alkali metal, non-alkaline metal or metals and NH and NH<sub>2</sub> groups. In any event, as referred to herein, the term "inorganic monomeric complex" will be used to refer to the product obtained during the endothermic phase reaction, while the term "inorganic polymeric complex" will be used to refer to the product formed during the exothermic phase reaction. As explained in more detail hereinafter, the presence of each of the groups within the monomeric and polymeric complexes has been established through analytical techniques.

Those of ordinary skill in the art will recognize that, in order to avoid the formation of salts and/or the release of large quantities of ammonia, the addition of the alkali metal hydroxide must be at a rate such that substantial quantities of NH<sub>2</sub> groups, as they are provided to the system, are taken up and bound into the complex. This will be governed to some extent by the rate at which the non-alkaline metal erodes, providing

ions for reaction. While some ammonia will necessarily be released from the reaction as the alkali metal hydroxide goes into solution, this effect may be minimized by refluxing and/or the bubbling or additional ammonia gas into the reaction medium.

It is preferred to add the alkali metal hydroxide to an aqueous medium which has already been saturated with the source of NH<sub>2</sub> groups. It is, however, likewise acceptable, in the practice of the present invention, to add the source of NH<sub>2</sub> groups (for example, aqueous ammonia, ammonia gas or an NO<sub>x</sub> source) to an aqueous medium already containing some dissolved alkali metal hydroxide, it again being understood that such addition should be at a rate as to release and substantially tie up NH<sub>2</sub> groups from the source thereof, while developing the required endothermic phase and that localized areas of pH approaching 14 exist. In similar manner, it is within the scope of the present invention to simultaneously feed both the alkali metal hydroxide and the source of NH<sub>2</sub> groups to the reaction medium at such rates as to effect this same result.

Those skilled in the art will appreciate that some non-alkaline metals of groups I-VIII of the periodic table will naturally dissolve or erode in the aqueous alkaline medium, sometimes even without the assistance of a preliminary reaction between the alkali metal and the NH<sub>2</sub> groups. As to these metals, the above method proceeds easily without the application of external sources for developing the endothermic phase and/or exothermic phase of the reaction. That is to say, the introduction of the reactants in accordance with the above sequence develops a natural endotherm which leads to a natural exothermic phase. However, where the particular metal utilized does not dissolve or erode easily in the reaction medium, it may be advantageous under certain circumstances to promote the reaction in one or more ways.

For example, an increase in the reaction rate can be achieved through one or more of the following:  
an increase in the effective surface area of the metal or metals to react with the alkali metal hydroxide and source of NH<sub>2</sub> groups;

depolarization of the surface area of the metal or metals to increase hydrogen attack and erosion of these metal or metals, such as for example, by the addition of one or more metals which are easily eroded in contact with the hard to erode metals and/or application of the electric current;

external cooling of the reaction medium to force the reactants into the endothermic phase of the reaction with the concurrent tying up in the form of the inorganic monomeric complex of the alkali metal ions, amino groups and eroded metal or metals; and/or

the application of external heat subsequent to the endothermic phase of the reaction in order to initiate the exothermic phase of the reaction wherein polarization of the inorganic monomeric complex occurs.

It is general practice in accordance with the method of the present invention to employ the non-alkaline metal or metals of groups I-VIII in the periodic table in amounts in excess of that which is expected to react with the source of NH<sub>2</sub> groups and the alkali metal hydroxide. Since the erosion of the non-alkaline metal or metals is essential for both inorganic monomeric complex production and inorganic polymeric complex production, termination of the reaction either in the exothermic phase or during or after the endothermic phase can be readily achieved by separating any unreacted non-alkaline metal or metals from the reaction medium.

Both the inorganic monomeric complex and the inorganic polymer complex produced through the method of the present invention have several established utilities. For example, the inorganic monomeric complex has been found particularly suitable for the production of soaps and bleach formulations in the manner described in co-pending application Ser. No. 534,084, filed Dec. 23, 1974, and entitled "Soaps and Detergents." Among the various uses thus far determined for the inorganic polymer complex, one of the more important is the use of the complex, in aqueous solution, for plating one or more of the various metals of groups I-VIII of the periodic table on various substrates.

## DESCRIPTION OF THE DRAWINGS

In the drawings:

**FIG. 1 is a diagrammatic illustration of the reaction dynamics of the method of the present invention, plotting temperature and non-alkaline metal erosion, alkali metal consumption, ammonia consumption, and pH as a function of time for a Si-Na and Si-K model system;**

**FIG. 2 is a chart of groups identifiable by infra-red analysis superposed on an infrared scan chart;**

**FIG. 3 is an infra-red scan of a Si-Na system after the endothermic phase of reaction;**

**FIG. 4 is an infra-red scan of a Si-Na system of a clear solution resulting from vacuum drying at 80 DEG C. of the product after the exothermic phase of reaction;**

**FIG. 5 is an infra-red scan of a Si-K liquid system after the exothermic phase of reaction;**

**FIG. 6 is an infra-red scan of a Si-K solid dehydrated system after the exothermic phase of reaction;**

**FIG. 7 is an infra-red scan of a Si-Na liquid system after the exothermic phase of reaction.**

**FIG. 8 is an infra-red scan of the Si-Na system of FIG. 7 after drying at 93 DEG C.;**

**FIG. 9 is an infra-red scan of the Si-Na system of FIG. 7 after drying at 232 DEG C.;**

**FIG. 10 is an infra-red scan of the Si-Na system of FIG. 7 after drying at 1093 DEG C.;**

**FIG. 11 is an infra-red scan of a Zn-K system;**

**FIG. 12 is an infra-red scan of a Ta-Na system;**

**FIG. 13 is an infra-red scan of a Sn-Na system;**

**FIG. 14 is an infra-red scan of a Be-K system;**

**FIG. 15 is an infra-red scan of a Zr-Na system;**

**FIG. 16 is an infra-red scan of a Nb-K system;**

**FIG. 17 is an infra-red scan of a Mo-Na system;**

**FIG. 18 is an infra-red scan of a Al-Na system;**

**FIG. 19 is an infra-red scan of a Fe-Na system;**

**FIG. 20 is an infra-red scan of a Bi-Na system;**

**FIG. 21 is an infra-red scan of an In-K system;**

**FIG. 22 is an infra-red scan of a Ti-Na system;**

**FIG. 23 is an infra-red scan of a Co-Na system;**

**FIG. 24 is an infra-red scan of a Si-Na system with 10% Na, effective for gem stone production;**

**FIG. 25 is an infra-red scan of a Si-Na system with 5% Na, effective for gem stone production;**

**FIG. 26 is a thermogravimetric curve over the range of 25 DEG-1000 DEG C. for a Si-Na system of the present invention; and**

**FIG. 27 is a thermogravimetric curve of the Si-Na system of FIG. 26 over the temperature range of 900 DEG-2300 DEG C.**

## **DESCRIPTION OF PREFERRED EMBODIMENTS**

The method of the present invention for the production of both the inorganic monomeric complex and the inorganic polymeric complex is carried out totally in an aqueous reaction medium. As a result, the inorganic monomeric complex and inorganic polymeric complex are produced as aqueous solutions of these products. For most applications, it will be unnecessary to separate the products from the aqueous solution. This is because the important utilities contemplated for the products of the present invention, for example, utilization of the monomeric complex in the production of soaps and bleach formulations, or of the polymeric complex in plating solutions are contemplated for use in aqueous solution form.

The following reactants are required:

- (a) at least one non-alkaline metal selected from groups I-VIII of the periodic table;
- (b) an alkali metal hydroxide; and
- (c) a source of reactive NH<sub>2</sub> groups. These reactants are individually discussed below.

### **The Non-Alkaline Metal**

In the method of the present invention, the metal reactant may be any non-alkaline metal of groups I-VIII of the periodic table. Representative, non-limiting examples of applicable non-alkaline metals include:

Group I-B: copper, silver, gold  
Group II-A: beryllium, magnesium  
Group II-B: zinc, cadmium  
Group III-A: aluminum, gallium, indium  
Group IV-A: silicon, tin, lead  
Group IV-B: titanium, zirconium, hafnium  
Group V-A: antimony, bismuth  
Group V-B: vanadium, niobium, tantalum  
Group VI-A: selenium, tellurium  
Group VI-B: chromium, molybdenum, tungsten  
Group VII-B: manganese  
Group VIII: iron, cobalt, nickel, palladium rhodium

It should be recognized that while silicon, as a member of group IV-A, is not generally defined as a metallic element, silicon acts in the method of the present invention as a non-alkaline metal. Accordingly, the expression "non-alkaline metal of groups I-VIII of the periodic table" is meant to embrace any and all of the above and equivalent metals, including silicon. As will be further recognized, the term non-alkaline metal of groups I-VIII of the periodic table does not embrace the alkali metals of group I-A. The alkaline earth metals, calcium, strontium, and barium of group II-A, are similarly not within the scope of the term. On the other hand, beryllium and magnesium of group II-A can be applicably employed in the practice of this invention and these metals also fall within the scope of the expression "non-alkaline metal of groups I-VIII of the periodic table" as used throughout this specification.

In addition to a single non-alkaline metal, as defined above, the metal reactant of the present invention can advantageously comprise two or more such non-alkaline metals. As indicated previously, non-alkaline metals applicable include not only those metals of groups IV-III of the periodic table which are easily dissolved or eroded in an aqueous alkaline solution, but also those metals which do not readily dissolve. Apparently, the method of the present invention, possibly through the intermediate formation in site of alkali amides, provides for an environment effective to erode such metals, as will be explained in more detail subsequently, in order to increase the reactivity, i.e., the rate of erosion and entry into reaction of certain metals.

The non-alkaline metal is generally employed in a solid form, with the exception of gallium, for example, which is liquid at room temperature. In broad terms, it may be stated that reaction dynamics are related to the available surface area of the non-alkaline metal. Where the non-alkaline metal is in the form of extremely large chunks, there may be insufficient surface area per unit weight of non-alkaline metal for adequate erosion and reaction in accordance with the method of the present invention. Here, or where the metal erodes slowly and is difficult to react, it is often advisable to increase the reactivity of the metal by increasing the available surface area, such as by decreasing the particle size of the non-alkaline metal. Care must be taken, however, not to provide a particle size which is sufficiently reactive that erosion takes place too rapidly and an effective endothermic phase becomes difficult to achieve. On the other hand, with some metals, a decrease in the particle size of

the non-alkaline metal can actually decrease the reaction dynamics. This latter effect appears to occur due to hydrogen polarization between the metal particles, not only at the surface of the powdered metal but also within the mass of metal particles. This has the effect of reducing the available hydrogen for metal erosion. Its reversal, or elimination, is explained hereinafter in the discussion dealing with reaction dynamics.

For any particular non-alkaline metal or combination of non-alkaline metals, the selection of size and/or shape of the metallic pieces can be easily determined by one skilled in the art, based upon an observation of reaction dynamics. In general, however, on a laboratory scale it has been found that a particle size of from about 1/2 inch to 1 inch in diameter for non-alkaline metal shot or sponge is particularly effective in achieving the advantageous results of the present invention. Of course, the method of the present invention can be carried out with equal efficiency using wires, foils or other convenient forms and shapes.

### **The Alkali Metal Hydroxide**

The alkali metal hydroxide utilized as the second reactant in the methods of the present invention can comprise any hydroxide of a metal of group I-A of the periodic table, principally, sodium hydroxide, potassium hydroxide or lithium hydroxide. As will be explained in more detail hereinafter in connection with the discussion on reaction parameters, the alkali metal ions supplied to the reaction medium by the hydroxide enter into reaction and form a component of the inorganic complexes produced by the reaction. In addition, the alkali metal hydroxide appears responsible first for the release from the aqueous ammonia or ammonia gas or  $\text{NH}_2$  groups and thereafter for binding up these groups and making them accessible to ions of the eroding non-alkaline metal in the formation of the products of the present invention.

As will be explained in more detail hereinafter, the particular alkali metal hydroxide employed does have some effect on the reaction dynamics. The preferred alkali metal hydroxide in accordance with the present invention is sodium hydroxide or potassium hydroxide.

While not favored in the practice of the method of the present invention, it should be recognized that under certain circumstances all or a portion of the alkali metal hydroxide may be replaced with one of the alkaline earth metal hydroxides.

The alkali metal hydroxide, specifically sodium hydroxide or potassium hydroxide, dissolves in the aqueous medium in the reaction of the present invention. The alkali metal hydroxide can be employed either in solid form or in the form of a prepared aqueous solution of the alkali metal hydroxide. In solid form, the alkali metal hydroxide can be in the form of pellets, powder or flakes. Alkali metal hydroxides such as sodium hydroxide and potassium hydroxide readily absorb carbon dioxide. It is preferred, in accordance with the present invention, to prevent as much of such carbon dioxide absorption as possible since the absorbed carbon dioxide does enter into the reaction to some extent and interferes with the production of the inorganic monomeric complex and inorganic polymeric complex. However, extreme precautions to prevent carbon dioxide absorption do not appear necessary.

As will be more fully explained hereinafter when discussing reaction parameter, the alkali metal hydroxide is generally metered slowly into the aqueous medium in such manner as to create localized areas in proximity to the non-alkaline metal which contain a high concentration of hydroxide ions at a pH approaching 14. The use of a solid form of the alkali metal hydroxide is quite advantageous in this respect, although it is also practicable to use an aqueous solution thereof for this metering or controlled addition.

### **The Source of $\text{NH}_2$ Groups**

Analytical studies have shown that the inorganic monomeric complex produced in the method of the present invention contains  $\text{NH}_2$  groups. Studies also indicate that the  $\text{NH}_2$  are converted at least in part to hydride (H) and NH groups during polymerization to the inorganic polymeric complex. Accordingly, the third essential reactant in the method of the present invention comprises any substance which, under the conditions of the reaction, acts as a source of reactive  $\text{NH}_2$  groups.

In the preferred reaction process, the source of  $\text{NH}_2$  groups comprises aqueous ammonia.  $\text{NH}_2$  groups are liberated when this aqueous ammonia is brought into reactive contact with the alkali metal hydroxide. As an alternative, ammonia gas may be used, introducing the gas into the reaction system of the present invention by any suitable conventional techniques, such as bubbling. The use of ammonia gas in this manner also permits a supplementing of the supply of the  $\text{NH}_2$  source at any stage during the endothermic reaction phase. This permits better control in some circumstances and/or may be necessary to offset previous ammonia losses.

Another applicable alternative  $\text{NH}_2$  source for use in the practice of the present invention may be formed directly in the reaction medium by utilizing as a reactant an  $\text{NO}_x$  gas, i.e., NO and/or  $\text{NO}_2$  or nitric acid. The use of  $\text{NO}_x$  gas or nitric acid as a source of reactive  $\text{NH}_2$  groups appears applicable due to the erosion of the non-alkaline metal and concurrent hydrogen ion production. These hydrogen ions react with the  $\text{NO}_x$  groups in the reaction medium, creating in situ the reactive  $\text{NH}_2$  groups. These reactive  $\text{NH}_2$  groups are then available for metal amide formation and formation of the inorganic monomeric complex. When the non-alkaline metal is not easily eroded in the reaction medium upon alkali metal hydroxide addition, it may be necessary to assist the erosion so as to develop the hydrogen ions necessary for reactive  $\text{NH}_2$  group production. This can be achieved, for example, by supplementing the  $\text{NO}_x$  or nitric acid with ammonia gas at the onset of the reaction.

It is hypothesized that the reactant actually responsible (at least in part) for erosion of the non-alkaline metal and formation of both the inorganic monomeric complex and the inorganic polymeric complex is an alkali metal amide, formed in situ through the reaction of the alkali metal ions of the alkali metal hydroxide with the released  $\text{NH}_2$  groups. A further alternative source of  $\text{NH}_2$  groups, therefore, which is applicable in the method of the present invention, would be a pre-formed alkali metal amide, such as sodium amide. When using this alkali metal amide as the source of  $\text{NH}_2$  groups, it may be possible to eliminate all or part of the alkali metal hydroxide.

## **REACTION PARAMETERS**

### **Reactant Proportions**

In the production of the inorganic polymeric complex of the present invention, the ratio of the alkali metal to the non-alkaline metal can be varied within wide limits. For example, such ratio may be varied from about 1:0.5 to 1:10 or more, this ratio being directly proportional to the effective surface area of the non-alkaline metal during the exothermic phase of the reaction. For example, where a great surface area exists and there is substantial erosion of the non-alkaline metal during the exothermic phase, there will be a greater ratio of non-alkaline metal to alkali metal in the inorganic polymeric complex. Likewise, the amount of alkali metal when compared with the non-alkaline metal will be greater where a small surface area of the non-alkaline metal exists during the exothermic phase of the reaction, and as a result, the erosion rate is less.

The molar ratio of alkali metal to non-alkaline metal in the inorganic monomeric complex prepared according to the preferred method of the present invention, will ideally be about 1:1, particularly with respect to metals which are easily eroded in the reaction of the present invention.



The reason for this is that in accordance with the present invention, in the ideal reaction the alkali metal hydroxide is added to the reaction medium in an amount and at a rate proportional to the erosion of the non-alkaline metal. The rate of introduction of the alkali metal hydroxide can be easily determined by one of ordinary skill in the art by an examination and monitoring of the reaction dynamics, specifically the dissolution or erosion of the non-alkaline metal. Of course, where the non-alkaline metal erodes slowly or is otherwise difficult to react in accordance with the present invention, a substantially greater molar ratio of alkali metal than 1:1 may exist in the inorganic monomeric complex. A greater amount of non-alkaline metal compared to the alkali metal will, of course, be present in the inorganic polymeric complex due to greater erosion of the non-alkaline metal during the exothermic phase of the reaction. The addition of the alkali metal hydroxide to the reaction medium and the reaction dynamics based thereon will be more fully discussed in connection with the reaction dynamics of the method of the present invention.

Generally, in accordance with the present invention, the non-alkaline metal is employed in an amount in excess based on the desired molar ratio of alkali metal to nonalkaline metal in the inorganic polymeric complex. Since the formation of both the inorganic monomeric complex and the inorganic polymeric complex requires the presence of non-alkaline metal for reaction, the reaction of the present invention may be terminated conveniently at any stage, e.g., after the endothermic reaction or during or after the exothermic reaction, by simply removing the excess non-alkaline metal from the reaction medium.

As will be explained in greater detail hereinafter, the amount of the source of the NH<sub>2</sub> groups employed in the method of the present invention will generally be predetermined based upon the particular alkali and non-alkaline metals utilized. It has been determined that in the inorganic complexes formed in accordance with the present invention, the non-alkaline metal takes its lowest possible valence state (if in fact more than one valence state is possible). It is further believed that in the inorganic monomeric complex of the present invention the number of NH<sub>2</sub> groups is equal to the sum of the valences of the alkali metal and non-alkaline metal. From this, therefore, it is possible to determine the amount of the source of NH<sub>2</sub> groups to be introduced into the reaction medium. Due to the inevitable loss of some ammonia, however, it will be desirable to include within the reaction medium an excess amount of the source of NH<sub>2</sub> groups based upon the proposed formulation. This will assure that sufficient NH<sub>2</sub> groups will be present to complex with the alkali metal and non-alkaline metal in the formation of the inorganic monomeric and polymeric complexes of the present invention.

### Reaction Dynamics

The reaction dynamics of the method of the present invention will be described primarily with regard to model systems based upon silicon/sodium and silicon/potassium. It should be recognized, however, that the reaction dynamics set forth for these models will be typical of a substantial number of the non-alkaline metals of groups I-VIII of the periodic table encompassed within the description given above. Where applicable, specific mention will be made of those metals which deviate to some extent from the silicon/sodium and silicon/potassium model to be discussed. Failure to mention a specific metal, however, should not necessarily be taken as an indication that the metal will precisely track the silicon model. The reaction dynamics for any particular metal system or combination of metals can be easily determined by one of ordinary skill in the art through routine experimentation.

The reaction dynamics of the method of the present invention will be illustrated by reference to FIG. 1. Utilizing silicon as a model non-alkaline metal, the curves shown represent two systems, a silicon/sodium system and a silicon/potassium system. Referring first to the silicon/sodium system, FIG. 1 includes three main curves. The first comprises a temperature profile curve, which also corresponds to the silicon metal erosion in the reaction medium. This curve plots the temperature of the reaction system and the erosion of the silicon metal as a function of time in hours. A second curve plots the NH<sub>3</sub> content and consumption in this silicon/sodium system, also as a function of time in hours. The final curve relating to the silicon/sodium system involves a plot of the sodium hydroxide content and consumption against time in hours.

It will be observed, in connection with the temperature curve, that there is an initial rise in temperature upon the introduction of the sodium hydroxide into the aqueous medium (which in the case of the reaction represented, already includes aqueous ammonia and silicon metal). This initial rise in temperature is probably due to the exothermic heat of solution as the sodium hydroxide pellets are dissolved in the aqueous reaction medium. In addition, the introduction of the sodium hydroxide pellets into the aqueous reaction medium effects a release of NH<sub>2</sub> groups from the aqueous ammonia. This reaction is also exothermic, further giving rise to the initial slight increase in temperature of the reaction medium. For example, the temperature may rise from an initial room temperature of approximately 75 DEG F. to a temperature of 80 DEG-90 DEG F. during the initial introduction of the sodium hydroxide.

The erosion of the silicon metal is also exothermic. The intensity of each of these exothermic reactions, however, lags that of the endothermic reaction, which is associated with the formation of molecular bonds and is theorized to result principally from the formation of water from the reaction of released hydrogen ions and free hydroxy radicals supplied by the alkali metal hydroxide binding or tying up of the released NH<sub>2</sub> groups, perhaps initially in the form of an alkali metal (i.e., sodium) amide, definitely as a complex with the alkali metal and the eroded silicon, most likely a combination of both. As a result, a natural endotherm is created, and shortly after the initial increase in reaction temperature due to the initial introduction of the sodium hydroxide, the temperature of the reaction medium begins to cool, leveling off as illustrated over a period of hours. While the degree of the endothermic phase of the reaction depends on many factors, particularly including the rate of erosion of the silicon, it has been found in accordance with the present invention that an endothermic phase cooling of the reaction medium to as low as 40 DEG-50 DEG F. can be achieved. It must be recognized that this endothermic phase is achieved in accordance with the model illustrated without any external cooling of the reaction medium.

Generally, the introduction of the sodium hydroxide should be completed in approximately two to four hours, although longer periods can be successfully employed. The addition of the alkali metal hydroxide is carried out in such manner as to create, immediately adjacent the non-alkaline metal, localized areas of high alkali metal hydroxide concentration at very high pH, approaching pH 14. An intimate contact relationship is thus developed between the alkali metal hydroxide and non-alkaline metal, establishing effective non-alkaline metal erosion and effective release and binding up of reactive NH<sub>2</sub> groups.

At this point in the reaction there will be sufficient sodium ions in solution in the reaction medium to bind with and tie up the released NH<sub>2</sub> groups. During the entire period of the endothermic phase of the reaction, therefore, there are competing exothermic and endothermic reactions, with the exothermic reactions, principally the erosion of the silicon metal, lagging the endothermic reactions, principally water formation and the formation of metal and multi-metal amides.

As indicated previously, the reaction of the present invention is accompanied by hydrogen evolution. If the reaction proceeds too rapidly and the hydrogen evolution occurs to such an extent as to carry the alkali metal hydroxide out of intimate contact with the non-alkaline metal, the reaction will not proceed as desired. As a result, the incremental feed of alkali metal hydroxide should be carried out in a manner so as to maintain the desired contact and the localized areas of high pH.

If the alkali metal hydroxide is fed to the reaction system at too fast a rate, substantial amounts of ammonia will be released from the reaction system and there will be insufficient erosion of the non-alkaline metal. Accordingly, there will be insufficient production of the inorganic monomeric complex. This reaction can be placed on a proper course by accelerating the erosion of the non-alkaline metal, such as by heating the reaction system. The non-alkaline metal which erodes will then bind with the alkali metal ions, remaining  $\text{NH}_2$  groups and alkali metal amides formed by reaction of the two.

On the other hand, if the feed of the alkali metal hydroxide is too slow, and as a result, there is insufficient  $\text{NH}_2$  group formation and hydrogen release, the eroded non-alkaline metal tends to bind with the alkali metal in the form of a salt, such as sodium silicate. When this occurs, it does not appear possible to reverse the reaction to achieve the production of the desired complexes.

At some point after the reaction has continued in its endothermic phase for a period of time, a natural exothermic phase is entered. This appears to be due to the energy course of the exothermic reactions overtaking and passing the endothermic reactions. Presumably, the initiation of the exothermic phase of the reaction coincides with a decrease in metal amide production, resulting from the binding or tying up of substantially all available  $\text{NH}_2$  groups and/or available sodium ions. From this point in the reaction, the exothermic reactions, erosion of the silicon metal and breaking up of  $\text{NH}_2$  groups and formation of metal hydride linkages in the polymerizing monomeric complex, begin to predominate and the temperature of the reaction medium starts to increase. As the temperature of the reaction medium increases, the erosion of the silicon metal also increases since the erosion rate is affected by the temperature achieved using the silicon/potassium system, the use of the less active potassium hydroxide in the method of the present invention has the effect of drawing out or lengthening the endothermic phase of the reaction, presumably due to a decrease in the erosion rate of the silicon metal. As a result, instead of a six or seven hour endothermic phase, all other factors being maintained constant, the endothermic phase using the potassium hydroxide is extended to twenty to twenty-four hours. The exothermic phase occurs at approximately twenty-four hours after the initiation of the reaction. Again, this exothermic phase is characterized by a temperature rise to a peak of approximately 260 DEG F. Once the exothermic phase is initiated and the erosion of the metal leads the endothermic reactions, the exothermic phase takes off quite quickly due to the "snowballing" effect associated with increased erosion of the silicon metal and hydride formation, with increasing temperature. This exothermic phase in the silicon/potassium system is also lengthened when compared to the exothermic phase of the silicon/sodium system.

The above discussion concerning the reaction dynamics involving the silicon metal system in which a natural endothermic phase and a natural exothermic phase are achieved is applicable to a great number of non-alkaline metals of groups I-VIII of the periodic table, utilized in accordance with the method of the present invention. For example, similar natural endothermic phase and a natural exothermic phase is achieved when utilizing such metals as aluminum, gallium, beryllium, zinc, titanium and zirconium. As indicated previously, however, certain applicable non-alkaline metals are less reactive than the model silicon metal and, as a result, it is often necessary to provide some external influence upon the development of the endothermic and exothermic phases of the reaction. Included within this latter group of metals are tin, tantalum, copper, bismuth, titanium and the other refractory metals, nickel and the nickel group metals, and cadmium.

With these and similar less reactive metals, it may be necessary, in order to either initiate, accelerate or maintain the endothermic phase of the reaction, to provide external cooling of the reaction medium. Similarly, for some metals it may be necessary, in order to develop and/or accelerate the exothermic phase, to slightly heat the reaction medium. This heating should be carried out in a uniform manner. Heating of the reaction medium to a temperature of about 180 DEG F. to about 220 DEG F. will generally be satisfactory to develop and accelerate the exothermic phase. Here again, it should be recognized that due to the increased erosion of the non-alkaline metal with increased temperature, the exothermic phase will develop through this heating.

In the alternative to cooling the reaction medium to develop the endothermic phase, or heating the reaction medium to develop the exothermic phase, there are other available procedures when utilizing a less reactive non-alkaline metal. For example, as has been explained earlier, since the reactivity of the non-alkaline metal is to some extent a function of the surface area of this non-alkaline metal, increased reactivity can be achieved by increasing the surface area of the non-alkaline metal. This can be done by providing the non-alkaline metal in smaller particle size or in greater amount. Again, however, it should be recognized that care should be taken in utilizing a powdered metal in the method of the present invention. This is due to the fact that in some instances the powdered metal becomes too reactive, not allowing for the controlled development of an endothermic phase, and in other instances, a polarization of hydrogen occurs, making the non-alkaline metal even less reactive.

Taking advantage of the fact that the method of the present invention is applicable to so many different metals, a very effective way of increasing the reactivity of the less reactive metals is the including within the reaction system of a minor amount of a more reactive metal, such as, silicon, aluminum, gallium, and the like. In this respect, it has been determined that even amounts as small as 0.01% of this more reactive metal can increase the reactivity of a less reactive non-alkaline metal. Of course, since more than one metal is applicable in accordance with the present invention, there is no upper limit in terms of the amount of the more active non-alkaline metal which can be added to the reaction medium. Also, the addition of almost any second non-alkaline metal, even as an impurity in the first non-alkaline metal seems to increase the reactivity of the non-alkaline metal through an anodic reaction.

When increasing the reactivity of a less reactive non-alkaline metal through the addition of a more reactive non-alkaline metal, it has been found advantageous to use as the more reactive metal a non-alkaline metal which has been previously used in a reaction carrying out the method of the present invention. That portion of the non-alkaline metal remaining after carrying out the method of the present invention, upon its removal from the reaction medium, is found to be even more reactive in carrying out further reactions. In fact, since even the less reactive non-alkaline metals become more reactive after being utilized in carrying out the method of the present invention, some of the less reactive non-alkaline metal which has been put through the instant method can be used to accelerate the reactivity of the same non-alkaline metal in future reactions. This procedure would eliminate the possibility of introducing an impurity into the inorganic monomeric complex or inorganic polymeric complex as a second metal, where no second metal is desired.

This latter phenomenon can also be used to advantage in another respect. As has been previously indicated, certain less reactive non-alkaline metals, as well as non-reactive metals of very small particle size, tend to polarize hydrogen on the surface of the metals, thereby decreasing the reactivity of such metals in the method of the present invention. This polarization can be eliminated or substantially reduced by the addition to the reaction medium of a small portion of a more reactive non-alkaline metal as discussed above. In the alternative, an electric current can be applied to the non-alkaline metal to eliminate this polarization. By eliminating this polarization effect, the less reactive non-alkaline metal will again become more reactive in the reaction of the present invention.

Finally, where a less reactive non-alkaline metal is utilized, there is a greater tendency for ammonia to escape from the reaction system since there is less eroded metal to complex with and thereby tie up the released  $\text{NH}_2$  groups. Under such circumstances, the endothermic phase of the reaction of the present invention can be accelerated by utilizing a continuous supply of ammonia gas either to supplement the aqueous

ammonia reactant or as a replacement thereof. By providing this supply of ammonia gas, it becomes possible to always maintain sufficient ammonia in the system. The problems created by excessive ammonia loss can also be prevented by carrying out the method of the present invention under reflux conditions wherein any ammonia which escapes is returned to the reaction medium. This can be achieved with reflux, such as dry ice reflux.

Referring again to the model system of FIG. 1, there is also illustrated the alkali metal hydroxide consumption in this same system. It can be noted by reference to FIG. 1 that the consumption of both sodium hydroxide and potassium hydroxide in the system occurs primarily during the endothermic phase of the reaction. This is consistent with the proposed explanation of the reaction dynamics which suggests that the sodium ions tie up or bind with released  $\text{NH}_2$  groups and eroded metal during the endothermic phase of the reaction. Accordingly, under ideal conditions when the reaction process is controlled so as to maintain an endothermic phase, substantially all of the alkali metal ions should be consumed. It is, of course, possible that some free alkali metal ions will not bind with the released  $\text{NH}_2$  groups and eroded metal and will appear as free ions in the final product.

FIG. 1 also illustrates ammonia content and consumption in both the silicon/sodium system and silicon/potassium system. It should be recognized that, in the embodiment illustrated, the reaction is initiated with aqueous ammonia already present in the reaction system. Accordingly, the dynamics which are illustrated in FIG. 1 pertain to such system wherein a maximum ammonia content is present at the initiation of the reaction, with this amount steadily declining as the reaction proceeds. It should be apparent, however, as previously indicated, that the ammonia content in the reaction system can be supplemented or that a steady flow of ammonia gas can be utilized in lieu of the aqueous ammonia. Under these circumstances, the reaction dynamics as they pertain to ammonia content and consumption will be greatly different than that illustrated in FIG. 1.

As illustrated in FIG. 1, there is a steady decline in the free ammonia content in the model system. While not illustrated, the decline in the free ammonia content is accompanied by an increase in the available  $\text{NH}_2$  content in the reaction system. This is consistent with the proposed theory that the introduction of the alkali metal hydroxide liberates free  $\text{NH}_2$  groups which, in turn, are bound up with the alkali metal ions and/or the eroded metal ions from the non-alkaline metal. As illustrated, the free ammonia content of the model reaction system is substantially depleted at the initiation of the exothermic phase of the reaction. In this respect, under optimum conditions, as the reaction system enters its exothermic phase all of the ammonia will be in the form of reaction  $\text{NH}_2$  groups which are substantially bound in the inorganic monomeric complex and no free ammonia will be expelled as the exothermic phase is entered. Moreover, as previously indicated, the addition of the alkali metal hydroxide is controlled in accordance with the reaction dynamics of the present invention to minimize as much as possible any expulsion of free ammonia from the reaction system. Again, this effect can be enhanced in accordance with the present invention by utilizing a reflux system and/or by supplementing the aqueous ammonia with ammonia gas.

The above explanation with respect to FIG. 1 and the reaction dynamics of the present invention has dealt principally with silicon/sodium and silicon/potassium systems. As previously noted, similar reaction dynamics are applicable for other nonalkaline metals. Substantially all of the nonalkaline metals of groups I-VIII of the periodic table show similar reaction dynamics, although some modification of the reaction parameters may be necessary to insure proper development of the endothermic and/or exothermic phases of the reaction.

With respect to the alkali metal hydroxide, it is emphasized that in addition to the applicability of sodium hydroxide and potassium hydroxide, the process of the present invention is applicable as well to the other alkali metal hydroxides. In addition, as was also indicated previously, all or a portion of the alkali metal hydroxide may be replaced with an alkaline earth metal hydroxide, such as calcium, barium or strontium hydroxide. These alkaline earth metal hydroxides are however, less active than the illustrated potassium hydroxide, thereby tending to draw out both the endothermic phase and exothermic phase of the reaction. Because of the rather low activity of these alkaline earth metal hydroxides, their use is not preferred.

Lithium hydroxide, while useful in the reaction, has low solubility in an aqueous ammonia medium. Therefore, it is generally necessary to dissolve all or some of the lithium hydroxide in water before introduction into the reaction system. This inhibits to a large degree the successful utilization of lithium hydroxide pellets or other solid form, and the development of the localized areas of high concentration and high pH.

As a result, if lithium hydroxide is employed as the alkali metal hydroxide, it is generally necessary to increase the rate of reaction, such as through supplementary ammonia gas or immersed surface area of the non-alkaline metal.

The remaining alkali metals behave roughly equivalent to the alkaline earth metals when utilized as the alkali metal hydroxide in the reaction of the present invention. Here again, therefore, due to the low reactivity of these alkali metal hydroxides, their use is not preferred. The preferred alkali metal hydroxides for effectively carrying out the method of the present invention are sodium hydroxide and potassium hydroxide. With the use of these alkali metals, a substantial number of non-alkaline metals of groups I-VIII of the periodic table achieve a natural endothermic phase. Accordingly, with respect to the experimental work which will be discussed hereinafter, principal activity has been with sodium hydroxide and potassium hydroxide.

Referring again to the model systems illustrated in FIG. 1, this figure further plots the overall pH of the reaction system as a function of time, specifically with regard to the illustrated silicon/sodium system. As illustrated, the pH of the reaction system is initially about 11.5 based upon the initial presence of aqueous ammonia. The pH rises upon the addition of the alkali metal hydroxide (in this case, sodium hydroxide), but then lowers again, tends to level off and is thereafter generally maintained within the range of about pH 11.5 to pH 13, preferably pH 12.5 to pH 13.

The lowering of the pH, after it initially rises upon the addition of the alkali metal hydroxide, is clearly due to the complexing of the alkali metal and the  $\text{NH}_2$  groups, with the concurrent formation of water and release of hydrogen to the system, during the endothermic phase of the reaction. It should be understood however, based upon the previous explanation of reaction parameters, that while the pH of the overall system is an important indicator of the manner in which the reaction is proceeding, it is nevertheless essential to the initiation and maintenance of the endothermic reaction phase that the system contain localized areas proximate to the non-alkaline metal wherein the pH approaches a value of 14.

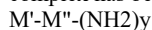
## Proposed Structures

Throughout the foregoing and following description, reference has been made to the production through the methods of the present invention of "an inorganic monomeric complex" and "an inorganic polymeric complex." The term "complex" is used because the structural configurations of these products have not been precisely determined. However, extensive analysis has been conducted which has established the monomeric and polymeric nature of the products and the presence of certain functional groups. In view of the difficulty in precisely determining the structural configuration of monomeric and polymeric products, particularly when obtained through complex chemical

reactions, the expressions "inorganic monomeric complex" and "inorganic polymeric complex" are deemed aptly descriptive of the products produced by the method of the present invention.

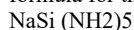
During the endothermic phase of the reaction, the alkali metal hydroxide, as it is introduced into the reaction system, first acts to release  $\text{NH}_2$  groups from the source of  $\text{NH}_2$  groups. Thereafter, the combined presence in the system of the reactive  $\text{NH}_2$  groups and the alkali metal and hydroxide ions appears to cause an erosion of the non-alkaline metal, bringing into solution metal ions. It appears that this erosion effect is due to the formation, in an intermediate reaction, of relatively unstable alkali metal amides, probably by reaction between the alkali metal ions and released  $\text{NH}_2$  groups. These alkali metal amides, in turn, react with the non-alkaline metal to form the inorganic monomeric complexes of the present invention. This last reaction is demonstrated physically by erosion of such non-alkaline metals which do not ordinarily erode easily or become soluble in the aqueous alkali medium.

It is postulated, therefore, that during the endothermic phase of the reaction an inorganic monomeric complex is formed which includes the alkali metal ions, ions of the non-alkaline metal and  $\text{NH}_2$  groups. Based upon extensive analysis, a formula for this inorganic monomeric complex has been developed, which can be empirically written as follows:



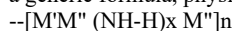
Wherein  $\text{M}'$  is the alkali metal,  $\text{M}''$  is the nonalkaline metal of groups I-VIII of the periodic table and  $y$  is the sum of the valences of  $\text{M}'$  and  $\text{M}''$ .

As previously indicated, it has been found that, in the inorganic complexes formed in accordance with the present invention, the non-alkaline metal tends to assume its lowest valence state. Accordingly,  $y$  in the above generic formula would in most instances comprise the sum of the valences of the alkali metal and lowest valence state of the non-alkaline metal. For example, in terms of the metal system of FIG. 1, the formula for the inorganic monomeric complex in the silicon/sodium system can be written as follows:



where, as has been mentioned earlier, it is necessary or desired to employ more than one non-alkaline metal of groups I-VIII of the periodic table, the sum of the valences should be calculated using an "apparent," or average, valence of the non-alkaline metals. It should be recognized, however, that when two or more different non-alkaline metals are employed, it is also possible that different monomeric complexes will be formed, each including only one non-alkaline metal in its structure.

The exothermic phase of the reaction of the present invention appears to be characterized by a breaking down of the  $\text{NH}_2$  groups, together with a polymerization of the inorganic monomeric complexes. This breaking down of the  $\text{NH}_2$  groups is not complete, however, and so polymerization is accompanied by the formation of  $\text{NH}$  groups and hydride groups within the inorganic polymeric complex. Referring again to a generic formula, physical-chemical analysis of the inorganic polymeric complex suggests a structure as follows:



Wherein  $\text{M}'$  again represents the alkali metal,  $\text{M}''$  represents one or more non-alkaline metals of groups I-VIII of the periodic table,  $x$  represents the total valence of  $\text{M}'$  and  $\text{M}''$  and  $n$  represents the numbers of repeating units in the inorganic polymeric complex.

While the above formula illustrates the presence of  $\text{NH}$  and  $\text{H}$  groups in the structure of the inorganic polymeric complex, this illustration is presented on the basis of a presumed preponderance of these groups. It is quite probable that in addition to  $\text{NH}$  and  $\text{H}$  groups the inorganic polymeric complex will contain  $\text{NH}_2$  groups which are not broken down in the exothermic phase of the reaction as well as hydride groups resulting from the further breaking down of  $\text{NH}$  groups. In addition, since the inorganic polymeric complex is formed in an aqueous medium, there is also the possibility that hydroxy groups and/or hydroperoxy groups will appear in the structure of the inorganic polymeric complex. These latter groups will generally be present when utilizing  $\text{NO}_x$  or nitric acid as the source reactive of  $\text{NH}_2$  groups. Accordingly the above proposed structure is deemed merely illustrative of what would appear to be a predominant unit of the inorganic polymeric complex and its presentation is in no way intended to exclude other groups, as described above.

As has been alluded to, during the exothermic phase of the reaction, there appears also to be considerable formation of the hydride of the non-alkaline metal, represented by the formula  $\text{M}''\text{H}_z$ , wherein  $z$  is the valence of the non-alkaline metal. If the exothermic phase is allowed to continue with excess non-alkaline metal maintained in the reaction system, greater amounts of the hydride will be formed. Eventually even the hydride will break down, resulting in the formation of elemental metal, possibly even metal oxide within the inorganic polymeric complex. A completely solid reaction mass will result if the reaction is carried substantially beyond the exothermic phase in the presence of additional non-alkaline metal.

As indicated previously, it has been observed that any non-alkaline metal which is removed from the reaction system following the exothermic reaction is more active in subsequent reactions than it had originally been prior to its use in the method of the present invention. The reason for this is not fully understood, although it does appear that this increased reactivity is associated with either the presence of a small quantity of the inorganic polymeric complex remaining in the removed non-alkaline metal and/or the presence of a non-alkaline metal hydride permeated through this non-alkaline metal.

With certain non-alkaline metals, the excess metal which is removed from the reaction medium following the exothermic reaction of the present invention has several other properties not generally associated with the non-alkaline metal in its original form. Using silicon as a typical example, the silicon metal removed from the reaction medium after the exothermic reaction will have a sponge-like appearance, different from the appearance of the original silicon metal. In addition, this silicon metal removed from the reaction medium will have the property of disassociating water, a property which may be explained by the presence of active hydrogen within the silicon metal, presumably due to its permeation with silicon hydride. While certainly not wishing to be bound by any explanation, it appears that it can be conclusively said that the non-alkaline metal which is removed from the reaction medium of the present invention subsequent to the exothermic reaction is altered in physical characteristics from the original non-alkaline metal.

The reaction of the present invention yields both the inorganic monomeric complex and the inorganic polymeric complex as stable water solutions. In this respect, both complexes are soluble in water and stable in the alkaline solution in which the complexes are formed. The solutions are clear and transparent. The solution of the inorganic polymeric complex is quite viscous. The transparency of this solution indicates that the inorganic polymeric complex includes a linear polymer. While the viscosity of the polymeric solution is, to some extent, dependent upon the amount of water which is present, it is also clearly an indication of molecular weight. The viscosity can vary from a viscosity similar to that of water itself to a highly viscous type of material which pours very slowly. In addition, as previously noted, if the exothermic reaction is carried further, a totally solid product is produced. Generally, the specific gravity of this polymer solution will range up to about 2.2. Here again, the specific gravity is dependent upon many variables and specific gravities outside this limit can easily be achieved.

A unique characteristic of the inorganic polymeric complex is the ability to reverse the polymerization reaction and develop a further exothermic phase and repolymerization. This can be done by reacting the inorganic polymeric complex with ammonia, i.e., aqueous ammonia or ammonia gas, thereby driving the polymeric complex back to a monomeric state. This is consistent with the proposed theory that in the inorganic monomeric complex there is a preponderance of NH<sub>2</sub> groups, these NH<sub>2</sub> groups breaking down into NH and H groups when going from the monomeric state to the polymeric state. Reaction of the NH groups with ammonia yields NH<sub>2</sub> groups.

This reversing ability is a valuable process control feature. It can be successfully utilized to load the inorganic polymeric complex with a greater amount of non-alkaline metal than is achievable in the initial exothermic reaction. For example, if after reversing the polymerization additional non-alkaline metal is added to the reaction medium, a new exothermic phase can be established which will increase the non-alkaline metal content of the complex. This is of particular value where the ratio of non-alkaline metal to alkaline metal must be increased, as is producing inorganic polymeric complexes for gem stone preparation.

The polymeric nature of the inorganic polymeric complex becomes especially apparent when the polymeric complex is dehydrated. The solid residue which is formed remains transparent, hard, horny (although plastic in nature) and is hygroscopic. When this solid is re-dissolved in water a clear viscous solution similar to the original solution of the inorganic polymeric complex results.

Since the inorganic polymeric complex is soluble in water, it can be diluted with water for a number of applications. For example, in the field of metal plating, both electroless plating and electrical plating, it is generally advantageous to dilute the polymer solution with water to provide a less viscous solution for plating purposes.

As stable as the aqueous solutions of the inorganic polymeric complex are, when acidified with acid, the polymeric structure appears to be destroyed, forming a solid gel. This solid gel forms a salt-like deposit when dehydrated. When the dehydrated solid gel is re-dissolved in water there is no similarity whatsoever between this solution and the original viscous inorganic polymeric solution. Accordingly, while the inorganic polymer solution is stable in alkaline medium, acid medium appears to destroy its polymeric nature.

To identify the functional groups in the structures of the inorganic monomeric and polymeric complexes, infra-red spectroscopy analysis was carried out on various samples. Again, principal analytical work was carried out with respect to the model systems utilizing silicon and sodium, and silicon and potassium. The infra-red curves developed in this analysis are set forth in the accompanying drawings 4-25, with FIG. 2 comprising an identifying chart superimposed upon an infrared chart to illustrate the characteristic groups which have been observed in the inorganic monomeric and polymeric complexes. These groups as well as other groups specifically characteristic of the silicon products can be found in the following table.

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## **Group Identification**

### **Absorption**

#### **Wave length Wave number**

#### **Group Intensity (microns) (cm@-@1)**

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**NH<sub>2</sub> medium 2.9-3.2 3448-3125**

**strong 6.1-6.5 1639-1538**

**medium 8.8-9.6 1136-1042**

**strong 10.8-14.0 926-714**

**N-H medium 2.9-3.3 3448-3030**

**weak 6.3-6.8 1587-1471**

**medium 8.8-9.4 1136-1064**

**Si-H very strong 4.3-4.8 2326-2083**

**N-H stretch 2.7-3.3 3704-3030**

**N-H bend 6.1-6.7 1639-1493**

**N-H rock 11.1-14.2 901-704**

**Si-O strong 9.2-9.5 1087-1053**

### **Silicates**

**very strong 9.0-11.1 1111-901**

**Carbonates 6.9-7.1 1149-1408**

**11.4-11.6 877-862**

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Turning to the figures, FIG. 3 represents the I.R. scan of a silicon/sodium complex after the endothermic phase of the reaction. The sample was introduced into the spectrophotometer as a capillary film. In addition to the hydroxyl groups of water present, the I.B. scan illustrates the presence of NH<sub>2</sub> groups in the monomeric complex. Again, reference can be made to FIG. 2 and the above table.

FIG. 4 is an I.R. scan of the same silicon/sodium polymer vacuum-dried at 80 DEG C. The sample was a cast film on a cesium iodide window. Again, the characteristic peaks identifying the silicon hydride, NH<sub>2</sub> group, NH group and their secondary and tertiary vibrations are illustrated. Again, groups such as the hydroxyl group from water, and impurities also appear in these infra-red scans.

FIGS. 5 and 6 are respectively I.R. scans of a silicon/potassium polymeric complex after the exothermic phase, FIG. 5 being a scan of the liquid polymeric complex, with figure 6 being a scan of the solid dehydrated polymeric complex. Both samplings were introduced as cast films of cesium iodide windows. When these infrared scans of FIGS. 5 and 6 are compared, for example, to the infra-red scan of FIG. 3, i.e., a silicon/sodium monomeric complex after the endothermic phase, it can be seen that in the exothermic phase of the reaction the number of NH<sub>2</sub> groups has decreased and the Si-H and N-H groups have increased relatively. This supports the hypothesis stated above that during the exothermic phase of the reaction, a substantial number of NH<sub>2</sub> groups are converted to hydride and N-H groups.

FIGS. 7-10 comprise a series of infra-red scans illustrating the stability of the functional groups with respect to heat. FIG. 7 is an infra-red scan of a silicon/sodium inorganic polymeric complex after the exothermic phase of the reaction, illustrating the presence of Si-H, NH<sub>2</sub> and NH groups. While the amount of NH<sub>2</sub> groups is reduced in the polymeric complex as compared to the monomeric complex resulting after the endothermic phase of the reaction, a significant amount of NH<sub>2</sub> groups still exists. FIG. 8 is an I.R. scan of the same silicon/sodium polymeric complex, this polymeric complex being a solid dehydrated polymer, dried at 93 DEG C. A review of this scan indicates that the greater proportion of the NH<sub>2</sub> groups present is not affected by this heating. The I.R. scan of FIG. 9 was taken of the solid dehydrated product, heated and dehydrated at 232 DEG C. When reviewing this scan it is noted that a considerable amount of the NH<sub>2</sub> groups have been removed from the inorganic polymeric complex through this heating. Finally, FIG. 10 is an I.R. scan of the same silicon/sodium inorganic polymeric complex heated and dehydrated at 1093 DEG C. A review of this scan indicates that most of the NH<sub>2</sub> functional groups have been eliminated.

FIGS. 11-23 are infra-red scans of other non-alkaline metals of groups I-VIII of the periodic table, with sodium or potassium. These I.R. scans will be related to specific inorganic polymeric complex preparations in the later given examples. Similarly, FIGS. 24 and 25 represent further I.R. scans of silicon/sodium inorganic polymeric complex, specifically prepared with reduced alkali metal content, for the particular application of gem stone preparation. These I.R. scans again will be discussed further in connection with later given examples.

Thermogravimetric analysis was conducted with respect to the inorganic polymeric complex of the present invention in order to develop further information concerning the structure of the inorganic polymeric complex by observing and analyzing the gases which are driven off during heating. The thermo-analytical behavior of the inorganic polymeric complex is illustrated in FIGS. 26 and 27. In preparing the charts of FIGS. 26 and 27, 8 mm diameter crucibles were used, the crucibles being tungsten cups of 20 mm height. To prevent loss of the sample material, a fairly low heating rate of 4 DEG C. per minute was utilized.

It can be seen from FIGS. 26 and 27 that upon heating of the inorganic polymeric complex gas release can be observed at four different temperatures. At the point where gas is first released, occurring between 25 DEG C. and 400 DEG C., the inorganic polymeric complex foams and large quantities of gas are given off. The weight loss in this first degassing phase is largely in the form of H<sub>2</sub>O which comes off at about 120 DEG C. Other unidentified gases start coming off at 120 DEG C., gradually tapering off at 400 DEG C.

During the second degassing phase, occurring at approximately 600 DEG C., the polymer re-foams and then collapses. At this point there appears to be some further, but slight, weight loss. These first and second degassing phases are seen in FIG. 26 which represents the initial behavior of the sample over the temperature range of 25 DEG-1000 DEG C.

More specifically with regard to FIG. 26, the expansion which occurred was due to an endothermic single weight loss process which started at 40 DEG C., obtaining a maximum rate at 135 DEG C., and concluding at 200 DEG C. A broad exotherm is seen at about 650 DEG C. This peaks starts at 810 DEG C. and is concluded at 1000 DEG C. Since there is no appreciable weight loss accompanying this, it is indicative of the formation of a glassy solid, somewhat akin to the "curing process" in organic polymer systems.

Referring now to FIG. 27, a third degassing phase is seen, taking place at about 1300 DEG C., at which point the inorganic polymeric complex loses a substantial amount of weight. It has been observed that nitrogen and hydrogen are driven off at this point. At the fourth degassing phase, occurring at 1580 DEG C., further large amounts of gas are driven off. This is in the form of hydrogen. If the temperature continues to be elevated further (to approximately 2500 DEG C.), the remainder of the polymer is completely vaporized. Looking at FIG. 27 in more detail, a small irregular exotherm is seen at about 900 DEG C. At 1100 DEG C. a weight loss process commences and from 1280 DEG C. to 1600 DEG C. a "gas bubbling" phenomena occurs, as mirrored in the 10X expanded weight change scale and magnified in the rate of weight loss trace. During this period there is about a 24% weight loss, followed immediately by about a 28% weight loss. At 1900 DEG C. a large weight loss occurs. At 2300 DEG C. all the sample has volatilized.

Based upon the results of the thermogravimetric analysis, it is postulated that at 135 DEG C. any water remaining after the exothermic phase of the reaction is released along with NH groups. At 600 DEG C. the water of crystallization is released, and at 1300 DEG C. the remaining NH groups are released. Finally, at 1580 DEG C. the hydrogen from the hydride groups present in the inorganic polymeric complex is released.

In a further study using mass spectroscopy it has been observed that nitrogen and atomic hydrogen are released by the inorganic polymeric complex. The atomic hydrogen appears to be released from room temperature through 1550 DEG C. The nitrogen is released at 875 DEG C.

As previously indicated both the inorganic monomeric complex and inorganic polymeric complex of the present invention have a variety of uses. For example, the inorganic monomeric complex can be utilized in the preparation of soaps and bleach formulations in accordance with applicant's co-pending application Ser. No. 534,084, filed Dec. 23, 1974. For example, a suitable soap can be prepared by reacting the inorganic monomeric complex with a fatty acid, through a reaction mechanism in which the fatty acid moiety replaces an NH<sub>2</sub> group of the inorganic monomeric complex. Reaction of hydrogen peroxide with the inorganic monomeric complex either with or without a fatty acid, produces a useful bleach product as a result of this presence of hydroperoxy groups in the final reaction product. Hydroperoxy groups can be introduced into the inorganic monomeric complex even without the separate reaction with hydrogen peroxide. This occurs naturally when utilizing nitric acid or NO<sub>x</sub> gas as the source of NH<sub>2</sub> groups in the production of the monomeric complex. The additional presence of oxygen in the reaction system tends to bring about the formation of hydroperoxy groups which are introduced into the main structure of the inorganic monomeric complex. Accordingly, it should be understood that the description of the inorganic monomeric complex as used throughout is meant to embrace such products which include hydroperoxy groups.

The inorganic polymeric complexes of the present invention have various utilities as a result of the unique characteristics of this inorganic polymeric complex. This is particularly true with regard to the apparent presence of free electrons and hydrogen ions within the complex. For example, the unique electrical properties of the complex make them effective liquid or fluidic capacitors, or diodes. While the ability of the inorganic polymeric complex of the present invention to act in the foregoing manner is not precisely understood, it is believed that the ability of the inorganic polymeric complex to store energy is based upon the presence of the free electrons present in the complex.

A further interesting, valuable utility of various inorganic polymeric complexes produced in accordance with the method of the present invention is in the production of synthetic gem stones. In this respect, since the inorganic polymeric complexes contain an alkali metal such as sodium or potassium, and one or more non-alkaline metals, it is possible to provide an inorganic polymeric complex in which the metal components match those of gem stones. When such inorganic polymeric complex is subjected to degassing by heating at elevated temperature, a crystalline solid product results, which product closely resembles in structure and characteristics the desired gem stone.

Generally, in order to match the alkali metal content of the gem stone, the inorganic polymeric complex of the present invention must be produced in such way as to have a reduced alkali metal content. Sometimes, the inorganic polymeric complex may contain no more than up to about 10% of the alkali metal, based on the total metal content of the complex. Such an inorganic polymeric complex can be prepared by one of the two possible techniques, either a dilution/reaction method or straight reaction method. In the dilution/reaction method, an inorganic polymeric complex is produced in substantially the same manner as has been described, the polymeric complex thereafter being diluted with aqueous ammonia or water, and subsequently, further reacted with additional non-alkaline metal. This further reaction with additional non-alkaline metal is carried out until the desired ratio between the alkali metal and non-alkaline metal is achieved.

In the straight reaction method, the inorganic polymeric complex is prepared utilizing the total amount of the desired alkali metal in the reaction's initial phase. However, due to the small amount of alkali metal utilized, the endothermic phase of the reaction will be slight, with the rate of the reaction again being proportional to the surface area of the non-alkaline metal. With only a small amount of alkali metal present, care must be taken to avoid excessive erosion of the non-alkaline metal which could result in salt formation, rather than production of the desired inorganic polymeric complex. Further dilution of the inorganic polymeric complex and/or heating to develop an exothermic phase can eliminate this salt formation.

The inorganic polymeric complexes of the present invention have the interesting capability of acting as a solvent for oxides and hydroxides of non-alkaline metals. Many unique applications of this inorganic polymeric complex result from this ability.

A principal use of the inorganic complexes of the present invention is in plating the non-alkaline metal from solutions of the inorganic polymeric complex. The inorganic polymeric complex can be utilized as a plating solution directly as it is produced from the exothermic phase of the reaction or, if too viscous, can be diluted with water. It has been determined in accordance with the present invention that plating can be achieved by utilization of any of the inorganic polymeric complexes of the present invention. However, certain plating variables must be altered for specific plating operations, including the parameters of voltage, amperage, temperature and/or type of electrode utilized.

A very unique characteristic of the inorganic polymeric complexes with respect to plating is that through the use of these complexes it is possible to plate certain metals which have not been previously capable of plating. Reference is made, for example, to the refractory metals such as titanium, tantalum and niobium, as well as to silicon. While silicon has been previously reported as being deposited by vacuum deposition and sputtering techniques, there appears to be no record of the successful plating of silicon metal.

Still further, it has been determined that platings of 99% purity and higher have been achieved through the use of the inorganic polymeric complexes of the present invention. Moreover, it has been discovered in accordance with the present invention that alloy plating can be achieved by utilizing an inorganic polymeric complex which contains more than one non-alkaline metal, or by mixing two or more inorganic polymeric complexes of the present invention. Still further, by using a carbon electrode, metal carbide plating has been found to be achievable. All of these applications of the inorganic polymeric complex of the present invention establish the unusual utility of these materials.

The present invention will now be described in reference to the following specific examples. Such examples are presented for purposes of illustration only, and shall not under any circumstances be deemed as limiting the present invention. In these examples, unless otherwise indicated, all percentages are by weight.

#### **EXAMPLE 1**

Three duplicate runs were carried out in accordance with the present invention in order to study the reproducibility of the inorganic polymeric complex. These runs were as follows:

1. Low purity silicon/potassium hydroxide
2. High purity silicon/potassium hydroxide
3. High purity silicon/sodium hydroxide

In studying the reproducibility of the inorganic polymeric complex, the amounts of reactants were kept constant for the duplicate runs. In addition, the parameters of surface area of the silicon, rate of addition of the alkali metal hydroxide and length of time of the exothermic reaction were kept as constant as possible.

#### **Low Purity Silicon/Potassium**

In this duplicate study, a mole ratio of silicon to potassium of 2:1 was employed in the initial reactants. The reactants involved 616 grams of silicon metal, 616 grams of potassium hydroxide and 1,998.3 grams of ammonium hydroxide, based upon a 26 Be solution of ammonium hydroxide in water. The silicon metal had an average particle size of 1/2 inch to 3/4 inch. The potassium hydroxide was added to reaction vessel containing the aqueous ammonia and silicon metal. The potassium hydroxide was added in small increments, the addition being completed in approximately two hours. An endothermic phase was developed which lasted approximately six hours. Thereupon, an exothermic phase developed which was completed in approximately 45 minutes. First and second reactions were carried out on consecutive days with the following results:

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#### **Reaction No. 1**

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**Reaction No. 2**

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**Silicon 7.93% 7.86% -Potassium 8.72% 8.68%****Weight % Ratio****1.0996 1.1056****Si:K Ratio 1.2659 1.2591**

---

It is noted that the results achieved in reaction #1 and reaction #2 are quite comparable, indicating that the inorganic polymeric complex can be reproduced, when the conditions are controlled. It should be noted that the endothermic and exothermic phase in this reaction were shortened by the use of low purity silicon, even though potassium was employed as the alkali metal. The impurities present in the silicon, principally iron, tend to increase the reactivity of the silicon metal, thereby shortening both the endothermic phase and exothermic phase of the reaction.

**High Purity Silicon/Potassium**

In this duplicates study, the same amounts of reactants were utilized with the exception that 2,054.8 grams of aqueous ammonia were utilized and the silicon metal had an average particle size of 1 inch to 1 1/2 inches. In these duplicate runs the potassium hydroxide addition was completed in approximately three hours. The endothermic phase of the reaction lasted approximately eight hours while the exothermic phase was completed in approximately 80 minutes. Again, duplicate reactions produced the following results:

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**Reaction No. 1****Reaction No. 2**

---

**Silicon 4.92% 4.93% -Potassium 12.19% 11.93%****Weight % Ratio****2.4776 2.4199****Si:K Ratio 0.5619 0.5752**

---

Here again, it can be noted from reactions #1 and #2 that excellent reproducibility of the inorganic polymeric complex is achieved. The lower amount of silicon in the inorganic polymeric complex, is due to the lower reactivity of the high purity silicon when compared with the low purity silicon. While potassium hydroxide was utilized as the alkali metal hydroxide, the endothermic phase and exothermic phase were not as drawn out as in the model system of FIG. 1, principally due to the manner of introduction of the potassium hydroxide, i.e., a shorter period of introduction when compared to typical potassium hydroxide introduction. In addition, the particle size and surface area of the high purity silicon appeared to affect the reaction dynamics.

**High Purity Silicon/Sodium**

The reaction, parameters utilized in connection with these duplicate runs were the same as observed in connection with the high purity silicon/potassium reaction above. The results achieved were:

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**Reaction No. 1****Reaction No. 2**

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**Silicon 6.89% 6.87% -Sodium 6.77% 6.77%****Weight % Ratio****0.9826 0.9854****Si:Na Ratio 0.8329 0.8306**

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It can be observed from the above results that here again with the use of sodium hydroxide, excellent reproducibility of the inorganic polymeric complex was achieved. In each of the above duplicate runs, analysis of the silicon content of the inorganic polymeric complex was achieved by determining the SiO<sub>2</sub> present after double dehydration and emulsion of the SiO<sub>2</sub> with hydrofluoric acid. The sodium and potassium determinations were obtained directly by atomic absorption using flame photometry.



## EXAMPLE 2

Further experiments were carried out utilizing low and high purity silicon with sodium hydroxide or potassium hydroxide in order to study the effect of parameters on the ratio of the alkali metal to the non-alkaline metal in the reaction. The parameters studied were a surface area of the non-alkaline metal and the duration of the exothermic phase of the reaction. Since it is difficult to measure surface area of any nonhomogeneous metal, the surface area was established by relating it to screen and mesh size.

In each of the following experiments, the addition of the alkali metal hydroxide to the reaction medium of aqueous ammonia and either low purity silicon or high purity silicon metal was achieved in two to three hours. In each of the experiments, a natural endothermic phase was developed which gave way to a natural exothermic phase after approximately 6 to 18 hours. In each of the experiments, the exothermic phase of the reaction was approximately 1-2 hours, with the reaction being terminated after the exothermic phase of removal of unreacted silicon metal. The analysis of the inorganic polymeric complexes formed was carried out in the same manner as set forth above with regard to example 1. The following experimental results were achieved:

---

### Experiment A

Low purity silicon

Mol ratio 2 Si:1 K

KOH 616 gr.

NH<sub>4</sub> OH\* 1,998.3 gr.

Si 616 gr.

Metal size 1/2" to 3/4"

Si 7.93%

K 8.72%

Ratio Si:K in polymer 1.2659

### Experiment B

Low purity silicon

Mol ratio 2 Si:1 K

KOH 616 gr.

NH<sub>4</sub> OH 1,995.7 gr.

Si 616 gr.

Metal size 1/2" to 3/4"

Si 7.86%

K 8.68%

Ratio Si:K in polymer 1.2591

### Experiment C

Low purity silicon

Mol ratio 4 Si:1 K

KOH 308 gr.

NH<sub>4</sub> OH 1,995.0 gr.

Si 616 gr.

Metal size 1" to 1 1/2"

Si 7.15%

K 10.63%

Ratio Si:K in polymer 0.9364

### Experiment D

**Low purity silicon**

**Mol ratio 1.3 Si:1 K**

**KOH 924 gr.**

**NH<sub>4</sub> OH 1,996.2 gr.**

**Si 616 gr.**

**Metal size 1/4" to 1/2"**

**Si 6.67%**

**K 10.43%**

**Ratio Si:K in polymer 0.8902**

**Experiment E**

**High purity silicon**

**Mol ratio 2 Si:1 K**

**KOH 616 gr.**

**NH<sub>4</sub> OH 2,054.9 gr.**

**Si 616 gr.**

**Metal size 1" to 1 1/2 "**

**Si 4.92%**

**K 12.19%**

**Ratio Si:K in polymer 0.5619**

**Experiment F**

**High purity silicon**

**Mol ratio 2 Si:1 K**

**KOH 616 gr.**

**NH<sub>4</sub> OH 2,054.8 gr.**

**Si 616 gr.**

**Metal size 1" to 1 1/2 "**

**Si 4.93%**

**K 11.93%**

**Ratio Si:K in polymer 0.5752**

**Experiment G**

**Low purity silicon**

**Mol ratio 1.3 Si:1 K**

**KOH 924 gr.**

**NH<sub>4</sub> OH 2,043.0 gr.**

**Si 616 gr.**

**Metal size 3/4" to 1"**

**Si 4.15%**

**K 13.42%**

**Ratio Si:K in polymer 0.4307**

#### **Experiment H**

**High purity silicon**

**Mol ratio 3 Si:1 K**

**KOH 616 gr.**

**NH<sub>4</sub> OH 2,043.0 gr.**

**Si 924 gr.**

**Metal size 1/2" to 3/4"**

**Si 6.83%**

**K 9.62%**

**Ratio Si:K in polymer 0.9886**

#### **Experiment I**

**High purity silicon**

**Mol ratio 6 Si:1 K**

**KOH 616 gr.**

**NH<sub>4</sub> OH 1,816.0 gr.**

**Si 1,815.0 gr.**

**Metal size 3/4" to 1"**

**Si 7.14%**

**K 9.70%**

**Ratio Si:K in polymer 1.0246**

#### **Experiment J**

**Low purity silicon**

**Mol ratio 2 Si:1 K**

**KOH 616 gr.**

**NH<sub>4</sub> OH 1,986.2 gr.**

**Si 616 gr.**

**Metal size 3/4" to 1"**

**Si 5.60%**

**K 11.62%**

**Ratio Si:K in polymer 0.6709**

#### **Experiment K**

**High purity silicon**

**Mol ratio 5.6 Si:1 K**

**KOH 616 gr.**

**NH<sub>4</sub> OH 1,991.3 gr.**

**Si 1,730.95 gr.**

**Metal size 1/2" to 1"**

**Si 8.17%; 8.22%**

**K 8.32%; 8.61%**

**Ratio Si:K in polymer 1.3670; 1.3292**

**(duplicate analyses)**

**Experiment L**

**Low purity silicon**

**Mol Ratio 2 Si:1 Na**

**NaOH 440 gr.**

**NH<sub>4</sub> OH 2,050.4 gr.**

**Si 616 gr.**

**Metal size 1/2" to 3/4"**

**Si 7.67%**

**Na 5.93%**

**Ratio Si:Na in polymer 1.0769**

**Experiment M**

**High purity silicon**

**Mol ratio 6 Si:1 Na**

**NaOH 440 gr.**

**NH<sub>4</sub> OH 2,053.6 gr.**

**Si 1,816.0 gr.**

**Metal size 3/4" to 1"**

**Si 6.89%**

**Na 6.77%**

**Ratio Si:Na in polymer 0.8329**

**Experiment N**

**High purity silicon**

**Mol ratio 6 Si:1 Na**

**NaOH 440 gr.**

**NH<sub>4</sub> OH 2,055.0 gr.**

**Si 1,816.0 gr.**

**Metal size 3/4" to 1"**

**Si 6.87%**

**Na 6.77%**

**Ratio Si:Na in polymer 0.8306**

**Experiment O**

**High purity silicon**

**Mol ratio 6.5 Si:1 Na**

**NaOH 440 gr.**

**NH<sub>4</sub> OH 2,043.0 gr.**

**Si 2,014.7 gr.**

**Metal size 1" to 1 1/2 "**

**Si 6.16%**

**Na 8.70%**

**Ratio Si:Na in polymer 0.5795**

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\*26 Be DEG in all runs

It is observed from the above experimental results that the amount of the non-alkaline metal that reacts with the alkaline metal in the preparation of the inorganic polymeric complex is directly proportional to the surface area of the non-alkaline metal.

A molecular weight determination was carried out with respect to the product of Example M. The determination was by membrane osmometry in 0.1 M NaOH at 45 DEG C. A polymer concentration of 21.67% in the aqueous medium was found with a Mn of 36,900, yielding a molecular weight of about 160,000.

### **EXAMPLE 3**

Following the procedure essentially as recited in Example 1, a zinc/potassium inorganic polymeric complex was prepared by reacting 390 grams of zinc metal, 168 grams of potassium hydroxide and 315 grams of aqueous ammonia (26 Be). The molar ratio of zinc to potassium in the reactant system was 2:1.

After the exothermic phase of the reaction, the inorganic polymeric complex was analyzed to contain 4.78% zinc and 24.93% potassium. Both the zinc content and potassium content of the polymer were determined by direct atomic absorption. FIG. 11 represents the infra-red scan for this zinc/potassium inorganic polymeric complex.

### **EXAMPLE 4**

Following the same procedure as Example 3, an inorganic polymeric complex of tantalum and sodium was produced by adding 40 grams of sodium hydroxide to a reaction medium comprising 210 grams of aqueous ammonia (26 Be) and 908 grams of tantalum metal, the initial molar ratio of tantalum to sodium in the reaction system being 5:1. The inorganic polymeric complex, after the exothermic phase of the reaction, was analyzed to contain 6.49% tantalum and 6.87% sodium. The analysis of the tantalum was achieved by precipitation by cupferron and the sodium analysis was carried out by atomic absorption.

FIG. 12 represents the infra-red scan for this tantalum/sodium inorganic polymeric complex.

### **EXAMPLE 5**

Following the procedure of Example 3, a tin/sodium inorganic polymeric complex was prepared by reacting 80 grams of sodium hydroxide with 1,190 grams of tin metal in 300 grams of aqueous ammonia (26 Be), the initial reaction ratio of tin to sodium being 5:1. Again, the introduction of the sodium hydroxide was carried out to first develop an endothermic phase of reaction and thereafter, an exothermic phase. Analysis of the inorganic polymeric complex obtained after the exothermic phase of the reaction yielded 13.44% sodium and 0.32% tin. The tin analysis was done by reduction with nickel and titration with KIO<sub>3</sub>. The sodium analysis was done by atomic absorption.

FIG. 13 represents an infra-red scan of this tin/sodium inorganic polymeric complex.

### **EXAMPLE 6**

Following the same procedure as Example 3, a beryllium/potassium polymer was obtained by reacting 140 grams of potassium hydroxide with 45 grams of beryllium metal in 265.5 grams of aqueous ammonia (26 Be). The reaction was carried through a first endothermic phase, and thereafter, through an exothermic phase of reaction. The inorganic polymeric resulting from the exothermic phase of the reaction was analyzed to contain 0.21% beryllium and 22.64% potassium. The beryllium analysis was carried out by precipitation of the phosphate and correction to the beryllium metal. Potassium analysis was done by atomic absorption.

FIG. 14 represents the infra-red scan of this beryllium/potassium inorganic polymeric complex.

### **EXAMPLE 7**

Following the procedure of Example 3, an inorganic polymeric complex of zirconium and sodium was prepared. The reactants were: zirconium metal, 2,184 grams; sodium hydroxide, 120 grams; aqueous ammonia, 525 grams (26 Be). The initial molar ratio of zirconium to sodium was 8:1. The inorganic polymeric complex resulting from the exothermic phase of the reaction was analyzed. The zirconium was precipitated as zirconium oxide in cupferron and the sodium content was determined by atomic absorption. This analysis yielded 2.73% zirconium and 8.46% sodium.

FIG. 15 represents the infra-red scan for this zirconium/sodium inorganic polymeric complex.

### **Example 8**

Again following the same procedure as in Example 3, a niobium/potassium inorganic polymeric complex was prepared. The reactants were: 186 grams of niobium metal, 56 grams of potassium hydroxide and 210 grams of aqueous ammonia (26 Be). The initial molar ratio of niobium to potassium was 2:1. The inorganic polymeric complex resulting after the exothermic phase of the reaction was analyzed to determine the niobium and potassium content. The niobium content was achieved by precipitation as niobium oxide Nb<sub>2</sub>O<sub>5</sub> by cupferron. The potassium was analyzed by atomic absorption. The results yielded 3.19% niobium AND 12.07% potassium.

FIG. 16 represents the infra-red scan of the niobium/potassium inorganic polymeric complex.

### **EXAMPLE 9**

Utilizing the same procedure as above, a molybdenum/sodium inorganic polymeric complex was prepared by reacting 40 grams of sodium hydroxide with 384 grams of molybdenum metal in a reaction medium comprising 245 grams of aqueous ammonia (26 Be), the addition of the sodium hydroxide being such as to develop an endothermic phase of the reaction and thereafter, an exothermic phase of reaction. The initial molar ratio of molybdenum to sodium was 4:1.

The inorganic polymeric complex resulting from the exothermic reaction was analyzed to determine the molybdenum and sodium content. The molybdenum was precipitated by benzo-oxime and analyzed. The sodium was determined by direct atomic absorption. As a result, it was found that the inorganic polymeric complex contained 1.06 % molybdenum and 8.10% sodium.

FIG. 17 represents the infra-red scan for this molybdenum/sodium inorganic polymeric complex.

#### EXAMPLE 10

An aluminum/sodium inorganic polymeric complex was prepared utilizing a procedure the same as that discussed above with respect to Example 3. In this example, 520 grams of sodium hydroxide were reacted with 702 grams of aluminum metal in 1,820 grams of aqueous ammonia (26 Be). Again, the addition of the sodium hydroxide was such as to first develop an endothermic phase of reaction and thereafter, an exothermic phase. The initial molar ratio of aluminum to sodium in the reactants was 2:1.

The inorganic polymeric complex resulting from the exothermic phase of the reaction was analyzed. The aluminum content of the complex was determined by hydroxide separation and ignition as aluminum oxide. The sodium content was determined by atomic absorption. An aluminum content of 18.31 % was found and a sodium content of 11.25% was found.

FIG. 18 represents the infra-red scan for this aluminum/sodium inorganic polymeric complex.

#### EXAMPLE 11

Utilizing the same procedure as set forth above with respect to Example 3, an iron/sodium inorganic polymeric complex was prepared. The reactants were: 224 grams of iron, 160 grams of sodium hydroxide and 560 grams of aqueous ammonia (26 Be). The initial molar ratio of iron to sodium was 1:1. Again, the introduction of the sodium hydroxide was such as to first develop an endothermic phase of reaction and thereafter, an exothermic phase of reaction. The inorganic polymeric complex resulted from this exothermic phase of reaction.

To determine the iron and sodium content of this inorganic polymeric complex, the iron was titrated with potassium permanganate and the sodium was determined directly by atomic absorption. An iron content of 0.94 % and a sodium content of 23.00% were found.

FIG. 19 represents the infra-red scan for this iron/sodium inorganic polymeric complex.

#### EXAMPLE 12

A procedure similar to that set forth above in Example 3 was carried out in order to produce a bismuth/sodium inorganic polymeric complex. In this example, the following reactants were utilized: bismuth, 627 grams; sodium hydroxide, 40 grams; aqueous ammonia, 210 grams (26 be). The initial molar ratio of bismuth to sodium in the reactants was 3:1.

Again, the sodium hydroxide was added to the reaction medium containing the bismuth metal and aqueous ammonia in order to first produce an endothermic phase and thereafter, an exothermic phase. The reaction was terminated by withdrawing excess bismuth metal after the exothermic phase of the reaction. The inorganic polymeric complex of bismuth and sodium resulting from this exothermic phase of the reaction was analyzed to determine the bismuth and sodium content. The bismuth content was determined by hydroxide separation, while the sodium content was obtained by atomic absorption. A bismuth content of 0.29% was determined, and a sodium content of 8.25% was determined.

FIG. 20 represents the infra-red scan of this bismuth/sodium inorganic polymeric complex.

#### EXAMPLE 13

An inorganic polymeric complex of indium and potassium was prepared utilizing a procedure similar to that of Example 3. In this example, 112 grams of potassium hydroxide were introduced into a reaction vessel containing 690 grams of indium metal and 280 grams of aqueous ammonia (26 Be), the initial molar ratio of indium to potassium being 3:1. Again, the introduction of the potassium hydroxide was such as to develop an endothermic phase of reaction and thereafter, an exothermic phase of reaction. The inorganic polymeric complex of indium and potassium resulted from this exothermic phase.

The indium content of the complex was analyzed by hydroxide precipitation from a perchloric acid medium. The potassium content was analyzed by atomic absorption. An indium content of 1.94% was found and a potassium content of 25.46% was found.

FIG. 21 represents the infra-red scan of this indium/potassium inorganic polymeric complex.

#### EXAMPLE 14

Following the same procedure as above, a titanium/sodium inorganic complex was obtained by reacting the following: 1,968 grams of titanium metal, 328 grams of sodium hydroxide, and 1,435 grams of aqueous ammonia (26 Be). The initial molar ratio of titanium to sodium was about 5:1. Again, the introduction of the sodium hydroxide into the reaction medium containing the titanium metal and aqueous ammonia was such as to produce an initial endothermic phase of reaction and subsequently, an exothermic phase of reaction.

The inorganic polymeric complex resulting from the exothermic phase of the reaction was analyzed to contain 11.54% sodium and 3.81% titanium. The titanium analysis was carried out by precipitation of titanium dioxide by cupferron. The sodium analysis was carried out by atomic absorption.

FIG. 22 represents the infra-red scan of the titanium/sodium inorganic polymeric complex.

#### EXAMPLE 15

Following the same procedure as indicated above in Example 3, a cobalt/sodium inorganic polymeric complex was prepared for the following reactants: 2,183 grams of cobalt metal, 296 grams of sodium hydroxide, 1,036 grams of aqueous ammonia (26 Be). The initial molar ratio of cobalt to sodium was 5:1. Again, the introduction of the sodium hydroxide into the reaction medium containing the cobalt metal and aqueous ammonia effectively achieved the production of an endothermic phase of reaction and thereafter, an exothermic phase of reaction.

The inorganic polymeric complex of cobalt and sodium resulting from the exothermic phase of reaction was analyzed to determine the cobalt and sodium contents. Both analyses were done by atomic absorption. It was found that the inorganic polymeric complex contained 0.15% cobalt and 16.23% sodium.

FIG. 23 represents the infra-red scan of this cobalt/sodium inorganic polymeric complex.

#### EXAMPLE 16

Two different chromium/sodium inorganic polymeric complexes were prepared by the procedure of Example 3. These reactions differed principally in the amount of chromium introduced into the inorganic polymeric complex. In the first reaction, 260 grams of chromium metal were reacted with 40 grams of sodium hydroxide in 245 grams of aqueous ammonia (26 Be). In the second reaction, 260 grams of chromium were reacted with 56 grams of sodium hydroxide in 245 grams of aqueous ammonia (26 Be). In the first case, the initial molar ratio of chromium to sodium 5:1. In the second case, the initial molar ratio of chromium to sodium was 5:1.

Each reaction was carried out in a similar manner, first forming an endothermic phase and thereafter, an exothermic phase of reaction. Excess chromium was removed from the system after the exothermic phase of reaction and both of the inorganic polymeric complexes resulting from this exothermic phase of reaction were analyzed. Both the chromium determination and sodium determination were carried out by atomic absorption. The product of the first reaction was found to contain 1.19% chromium and 6.18% sodium. The product of the second reaction was found to contain 3.93% chromium and 15.60% sodium.

#### EXAMPLE 17

Following a similar procedure as above a tungsten/sodium polymer was prepared from the following reactants: tungsten metal, 552 grams; sodium hydroxide, 40 grams; aqueous ammonia, 245 grams (26 Be). The initial molar ratio of tungsten to sodium in the reaction was 3:1. Again, the introduction of the sodium hydroxide was carried out to produce a first endothermic phase and thereafter, an exothermic phase.

The inorganic polymeric complex resulting from the exothermic phase was analyzed to determine the tungsten and sodium content. The tungsten content was analyzed by precipitation with cinchonine and ignition as tungstic oxide,  $WO_3$ . The sodium content was determined by atomic absorption. It was found that this inorganic polymeric complex contained 2.38% tungsten and 9.57% sodium.

#### EXAMPLE 18

A manganese/sodium polymer was prepared in a manner similar to the above, utilizing 3,135 grams of manganese, 285 grams of sodium hydroxide, and 1,995 grams of aqueous ammonia (26 Be), the molar ratio of manganese to sodium in the reaction being 8:1. A similar endothermic phase and exothermic phase were developed.

The product resulting from the exothermic phase of the reaction, i.e., the inorganic polymeric complex, was analyzed to determine the manganese and sodium content. A manganese content of 1.79% and a sodium content of 11.05% were found. The manganese content was determined by direct titration and the sodium content was determined by atomic absorption.

#### EXAMPLE 19

Following a similar procedure to that set forth above, a nickel/sodium inorganic polymeric complex and a nickel/potassium inorganic polymeric complex were produced. In producing the nickel/sodium inorganic polymeric complex, 2,242 grams of nickel were reacted with 380 grams of sodium hydroxide, and 1,330 grams of aqueous ammonia (26 Be). In producing the nickel/potassium inorganic polymeric complex the 380 grams of sodium hydroxide were replaced with 532 grams of potassium hydroxide. In each case, the initial molar ratio of nickel to alkali metal was about 4:1.

Each reaction was carried out through the addition of the alkali metal hydroxide to produce an endothermic phase of reaction and thereafter, an exothermic phase of reaction.

The inorganic polymeric complex resulting from the exothermic phase of the reaction was analyzed in the case of each of the complexes prepared. The nickel, potassium and sodium were all analyzed by direct determination by atomic absorption. In the case of the nickel/sodium inorganic polymeric complex, the analysis yielded 0.15% nickel and 14.86% sodium. In the case of the nickel/potassium inorganic polymeric complex, the analysis yielded 0.19% nickel and 23.55% potassium.

#### EXAMPLE 20

Utilizing a similar procedure to that of Example 3, a silver/sodium inorganic polymeric complex was prepared by reacting 216 grams of silver metal with 40 grams of sodium hydroxide in 70 grams of aqueous ammonia (26 Be). The initial molar ratio of the reactants was 2:1. Again, an endothermic phase of reaction and subsequent exothermic phase of reaction were developed by the addition of the sodium hydroxide to the reaction medium containing the silver metal and aqueous ammonia.

The inorganic polymeric complex resulting from the exothermic phase of the reaction was found to contain 0.21% silver and 16.51% sodium, both by atomic absorption. It was also found in accordance with the present invention that when utilizing silver in a powder form it is often necessary to increase the aqueous ammonia content, such as by a factor of two (2) due to the fact that the silver powder tends to become sponge-like and readily absorbs the aqueous ammonia. This generally tends to produce a proportional decrease in the amount of the alkali metal in the inorganic polymeric product.

#### EXAMPLE 21

A cadmium/sodium inorganic polymeric complex was obtained by reacting 1,120 grams of cadmium with 200 grams of sodium hydroxide and 525 grams of aqueous ammonia (26 Be). The initial molar ratio of cadmium to sodium in the reactants was 2:1.

The cadmium content and sodium content of the inorganic polymeric complex resulting from the exothermic phase of the reaction were analyzed by atomic absorption. A cadmium content of 0.37% was found, as was a sodium content of 18.04%.

#### **EXAMPLE 22**

This example illustrates the preparation of a gallium/sodium polymer utilizing a similar reaction procedure as described above. In this example, 700 grams of gallium were reacted with 80 grams of sodium in 280 grams of aqueous ammonia (26 Be), the sodium hydroxide being added to the reaction vessel containing the gallium metal and aqueous ammonia so as to initiate an endothermic phase of reaction and thereafter, an exothermic phase of reaction. The initial molar ratio of gallium to sodium was 5:1.

The inorganic polymeric complex resulting from the exothermic phase of the reaction was analyzed to determine the gallium and sodium content. Both gallium and sodium were analyzed by direct atomic absorption. This analysis yielded 0.33% gallium and 9.32% sodium.

#### **EXAMPLE 23**

A similar procedure was utilized to produce an inorganic polymer from antimony and sodium by reacting 366 grams of antimony metal with 40 grams of sodium hydroxide and 210 grams of aqueous ammonia (26 Be). The initial molar ratio of antimony to sodium was 3:1.

The inorganic polymeric complex resulting from the exothermic phase of the reaction was analyzed to determine the antimony and sodium content. Both analyses were done by atomic absorption. The inorganic polymeric complex analyzed as 0.49% antimony and 10.39% sodium.

#### **EXAMPLE 24**

A tellurium/sodium polymer was prepared in the same manner as above by reacting 128 grams of tellurium with 40 grams of sodium hydroxide in 245 grams of aqueous ammonia (26 Be). The initial molar ratio of tellurium to sodium in the reaction system was 2.5:1. Again, the addition of the sodium hydroxide was such as to develop an initial endothermic phase of reaction and thereafter, an exothermic phase of reaction.

The inorganic polymeric complex resulted from the exothermic phase of reaction. This inorganic polymeric complex was analyzed to determine the tellurium and sodium content. The tellurium was precipitated by reduction with SO<sub>2</sub> to the tellurium metal. The sodium content was determined from the filtrate of the precipitation by atomic absorption. The results of this analysis were 0.81% tellurium and 30.06% sodium.

#### **EXAMPLE 25**

Two copper/sodium inorganic polymeric complexes were prepared utilizing copper metal and sodium hydroxide. Two reactions were carried out, varying the copper content in the inorganic polymeric complex. In the first reaction, 450 grams of copper were reacted with 100 grams of sodium hydroxide in 800 ml of aqueous ammonia (26 Be).

In the second reaction, 450 grams of copper were reacted with 200 grams of sodium hydroxide in 800 ml of aqueous ammonia (26 Be).

In both cases, the addition of the sodium hydroxide was such as to develop an endothermic phase of reaction and thereafter, an exothermic phase of reaction. The inorganic polymeric complexes resulting from the exothermic phases of the reactions were analyzed for copper content and sodium content, both by atomic absorption. In the case of the first reaction, a copper content of 0.23% and a sodium content of 7.63% was found. In the case of the second reaction, a copper content of 0.07% and a sodium content of 14.94% were found.

#### **EXAMPLE 26**

A magnesium/sodium inorganic polymeric complex was prepared by reacting 600 grams of magnesium metal, 200 grams of sodium hydroxide and 525 grams of aqueous ammonia (26 Be) in accordance with the procedure described above.

#### **EXAMPLE 27**

A gold/sodium inorganic polymeric complex was prepared by reacting 394 grams of gold metal, 40 grams of sodium hydroxide and 140 grams of aqueous ammonia (26 Be), in accordance with the process as described in Example 3.

#### **EXAMPLE 28**

A titanium/potassium inorganic polymeric complex was prepared utilizing ammonia gas to supplement the aqueous ammonia. The reaction involved 1,968 grams of titanium and 459.2 grams of potassium hydroxide. The desired amount of aqueous ammonia was 1,435 grams, about 26 B aqueous ammonia. To achieve this, some 915.9 grams of ammonia gas were introduced continuously into the reaction vessel. The ammonia gas was introduced from beneath the titanium metal to provide a uniform dispersion of ammonia gas and contact between the gas and titanium. An aqueous medium was employed. A dry-ice condensor was also employed for reflux purposes to prevent the escape of the ammonia gas, keeping as much ammonia as possible in the reaction system.

As a result of this reaction, a similar endothermic phase and exothermic phase was achieved through the metering of the potassium hydroxide into the reaction system. The inorganic polymeric complex prepared through this procedure appears similar to those complexes obtained by the use of aqueous ammonia.

#### **EXAMPLE 29**

A tantalum/potassium inorganic polymeric complex was prepared utilizing 908 grams of tantalum metal, 56 grams of potassium hydroxide and 210 grams of aqueous ammonia (26 Be). The initial molar ratio of tantalum to potassium was 5:1.

The tantalum metal was introduced into the reaction vessel with the aqueous ammonia and the potassium hydroxide was added slowly to



generate the endothermic phase and exothermic phase of the reaction. Due to the tendency for tantalum to polarize hydrogen, a power supply was added to the system, utilizing a tantalum anode and tantalum cathode in contact with the tantalum metal in the reaction vessel. As a result of this, a small current was passed through the tantalum metal. This had the effect of depolarizing the surface of the tantalum metal, thereby assuring the development of the desired endothermic phase and subsequent exothermic phase of the reaction. As a result of this procedure, an inorganic polymeric complex of tantalum and sodium was prepared.

#### **EXAMPLE 30**

An indium/potassium inorganic polymeric complex was prepared by reacting 690 grams of indium, 122 grams of potassium hydroxide and 280 grams of aqueous ammonia (26 Be). In this process, a hot water bath was utilized to initiate the exothermic phase of the reaction. The inorganic polymeric complex of indium and potassium resulted.

#### **EXAMPLE 31**

This example represents the preparation of an inorganic polymeric complex for gem stone preparation. This example is carried out to provide a minimum of sodium in the silicon/sodium inorganic polymeric complex. The reactants are 560 grams of silicon metal, 40 grams of sodium hydroxide and 1,750 grams of aqueous ammonia (26 Be).

The sodium hydroxide is added to the reaction vessel containing the silicon metal and aqueous ammonia in the initial phase of the reaction. Due to the limited amount of sodium hydroxide added based upon the amount of silicon metal, only a slight endothermic phase is developed and the average temperature in the reaction is about 70 DEG F., slightly below room temperature. In this process the erosion rate of the silicon must be in equilibrium with the addition of the sodium hydroxide in order to prevent salt formation. By carrying this reaction through the slight endothermic phase and thereafter, through the exothermic phase, a silicon/sodium inorganic polymeric complex is prepared which can be effectively used for gem stone preparation by application of degassing techniques.

#### **EXAMPLE 32**

A further silicon/sodium polymer useful for gem stone preparation is prepared by the dilution-reaction method. Some 500 ml of a previously formed silicon/sodium inorganic polymeric complex having a specific gravity of 1.543 is diluted with aqueous ammonia. Upon dilution, two separate layers are initially formed, the bottom layer being the inorganic polymeric complex, and the top layer being the aqueous ammonia. These two separate layers, however, become miscible upon the addition of silicon metal and heat.

The original inorganic polymeric complex contained greater than 10% by weight sodium. In order to achieve a maximum sodium content of 10% by weight, additional silicon metal must be introduced into the inorganic polymeric complex. To achieve this, 131.7 grams of silicon metal are added to the two-phase system, with heating. As a result of this the two phases become miscible and the silicon metal is taken out by the inorganic polymeric complex, thereby providing the desired ratio of silicon metal to sodium metal in the complex. This inorganic polymeric can now be utilized for gem stone preparation.

#### **EXAMPLE 33**

An aluminum/sodium polymer useful for gem stone preparation is prepared by reacting 702 grams of aluminum, 40 grams of sodium hydroxide and 1,820 grams of aqueous ammonia (26 Be). By utilizing this small amount of sodium compared to the amount of aluminum, an inorganic polymeric complex is prepared which is capable of being utilized for gem stone preparation.

#### **EXAMPLE 34**

A further polymer of aluminum and sodium for gem stone preparation is prepared by the dilution technique. An aluminum/sodium inorganic polymeric complex is utilized which has a specific gravity of 1.673, the original polymer contained in excess of 5% sodium. In order to achieve a 5% sodium content in the inorganic polymeric complex, additional aluminum metal, i.e., 3,895 grams, must be present in the final product. Accordingly, 3,723.3 grams of aluminum are added to the original aluminum/sodium inorganic polymeric complex diluted with aqueous ammonia. This achieves a final product with a reduced sodium content, i.e., about 5%, useful for gem stone preparation. A dilution of the aluminum inorganic polymeric complex with aqueous ammonia is done slowly since this complex reacts vigorously with the aqueous ammonia.

#### **EXAMPLE 35**

An aluminum/sodium/calcium inorganic polymeric complex is prepared by reacting 486 grams of aluminum, 46 grams of sodium hydroxide, 40 grams of calcium hydroxide and 1,890 grams of aqueous ammonia. In this reaction, the sodium hydroxide is added initially in order to initiate the endothermic phase of the reaction, whereupon the calcium hydroxide is fed in and reacted with the inorganic polymeric complex formed.

The foregoing description has been directed to particular embodiments of the invention in accordance with the requirements of the Patent Statutes and for purposes of illustration and explanation. It will be apparent, however, to those skilled in this art that many modifications and changes in the procedures set forth will be possible without departing from the scope and spirit of the invention. It is applicants intention that the following claims be interpreted to embrace all such modifications and variations.

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### **Cubic carbon [ Diamond] EP0142176**

Disclosed is a crystalline face centred allotrope of carbon having properties of extreme hardness, inertness and a negative thermal coefficient of resistance. The crystalline carbon may be adherently deposited onto a substrate by decomposition a hydrocarbon gas into constituent gaseous ionic hydrogen and carbon fractions, separating the fractions into layers, and exposing the objects desired to be coated to the carbon layer whereon deposition occurs. The properties of the crystalline carbon, termed cubic carbon, may be varied by suitably doping with a known amount of a known impurity.[ e.g., Nitrogen, Type II blue diamond ]

0001] This invention relates to a novel carbon allotrope and to coatings thereof possessing a combination of properties heretofore unknown,

which allotrope will hereinafter be referred to as "cubic carbon".

[0002] The coating art is replete with myriad compositions suited to a wide spectrum of uses. Utilities of coatings are bottomed on needs ranging from those that are cosmetic-protective (e.g. paints, varnishes, and lacquers) to those that are principally protective (e.g. zinc galvanic coatings) to those which must be able to withstand the most rigorous of physical and chemical conditions (e.g. high temperature wire enamels, acid or caustic container coatings, high temperature oxidation-resistant coatings). As a general proposition, most coating materials in use today are polymeric in nature, with some obvious exceptions such as galvanic zinc coatings. Other than the latter, however, there are few if any examples of common protective coatings which are applied in elemental form.

[0003] It has long been suspected that there exists a form of carbon intermediate between graphite and diamond. Graphite and diamond represent carbon allotropes i.e. the same elemental substance existing in two or more forms which manifest extremely diverse physical and/or chemical properties. Diamond is formed of a tetrahedral network lattice structure in which each carbon atom is tetrahedrally bonded to four other carbon atoms through SP hybrid orbitals. Because of this network bonding arrangement, diamond is essentially a super crystal which accordingly exhibits extreme hardness, a high melting point, great chemical stability, and non-conduction of electricity due to the fact that all electrons are covalently bound and therefore unavailable to function as charge carriers. By contrast, graphite is composed of layers of fused hexagonal rings, which layers are bonded to each other by relatively weak van der Waal's forces. Because of these weak forces, facile slippage of the layers over each other occurs, resulting in the characteristic lubricity which graphite exhibits. Other amorphous forms of carbon such as carbon black also exist.

[0004] Some attempts have been made to locate a carbon allotrope intermediate between graphite and diamond, but none have met with success. Such an allotrope has now been discovered, along with a method for its manufacture.

[0005] The present invention provides a novel allotrope of carbon having a face centered cubic structure. As will herein be described, this allotrope possesses a wide spectrum of properties, the combination of which is not exhibited by any prior art coating.

[0006] Cubic carbon is formed by the decomposition of a hydrocarbon gas at high temperature and under otherwise substantially quiescent conditions. By this invention, the hydrocarbon gas is admitted to a decomposition chamber where it virtually instantaneously dissociates into carbon and hydrogen ions. Because the dissociated species are in ionic form, they may be separated into layers by deflecting through a magnetic field. The separated layers of hydrogen ions and carbon ions pass into a uniformly heated deposition chamber containing objects to be coated. Advantageously, the hydrogen ions recombine to form hydrogen gas which is vented from the chamber while the carbon ions deposit on objects in crystalline layers which have been determined to have a face-centered cubic structure.

[0007] Cubic carbon is characterized by a novel combination of properties including extreme mechanical ruggedness, a negative thermal coefficient of resistance, conductive properties which can be varied or manipulated, efficient heat transfer properties, complete inertness to all known chemical corrosives, and high temperature oxidative stability. Cubic carbon therefore possesses a wide spectrum of applications ranging from coatings which prolong the useful life of tungsten-carbide cutting tools by a factor of 2 to 5, to utility in forming junction devices such as solar cells which transduce sunlight into electrical energy. This carbon allotrope additionally finds utility in more mundane settings such as forming resistant coatings on cooking vessels which, when suitably provided with electrical leads, have the capability of heating comestibles using the whole of the coated surface at a fraction of the cost required to cook on a conventional gas or electric range burner.

[0008] Prior art attempts to make the novel cubic carbon allotrope of this invention have failed chiefly for two reasons. First, in any reactor wherein a hydrocarbon gas is decomposed to constituent hydrogen and carbon ions, it is important that mass convection be avoided. Therefore, the flow rate at which a hydrocarbon gas is permitted to travel through the apparatus must be carefully controlled and maintained at a level low enough so that no such convection occurs. Thus, the term "substantially quiescent" used previously means any flow rate which does not cause mass convection to occur. The apparatus used to effect decomposition of a hydrocarbon vapor is constructed such that the hydrogen and carbon separate into ionic layers.

[0009] Thus, it is easily seen that any hydrocarbon decomposition apparatus of a given size or volume will be characterized by a critical flow rate which may not be exceeded without causing recombination or mixing of said layers, which in turn would be fatal to the formation of cubic carbon.

[0010] The second important point is that thermal convection of the separated carbon and hydrogen layers should be avoided, and therefore uniform heating of the entire decomposition chamber is required at the high decomposition temperatures employed.

[0011] To summarize, a very pure hydrocarbon gas is admitted to a high temperature uniformly heated decomposition chamber at a flow rate which avoids mass convection. The vapor decomposes into constituent hydrogen and carbon ions and these are magnetically manipulated such that a distinct layer of hydrogen ions is formed in the top of the reactor and a layer of carbon ions is formed in the bottom of the reactor. In this manner objects which have been placed in the bottom of the reactor are coated with a layer of carbon which bonds tightly thereto as a face-centered cubic crystal.

**Figure 1 is a schematic diagram of the apparatus used to decompose hydrocarbon gas and deposit cubic carbon onto objects.**

**Figure 2 is an x-ray diffraction pattern characteristic of cubic carbon.**

**Figures 3-5 are electron micrographs showing the visual surface structure of a substrate not coated with cubic carbon.**

**Figures 6-9 are electron micrographs of a cubic carbon coated substrate at the magnifications noted.**

**Figure 10 is an electron micrograph of a cubic carbon coating after having been subjected to corrosion testing.**

[0012] Referring to the figures, figure 1 shows the apparatus (herein also referred to as a vapor ionizer or ionizer) used for decomposing a gaseous hydrocarbon into its constituent ions such that carbon is adherently deposited on an object in a face centered cubic crystal structure. The apparatus, generally indicated as 10, comprises a first stage decomposition and separation chamber in combination with a second stage deposition chamber, which apparatus is fabricated of refractory or other high temperature material. The deposition chamber is internally divided by refractory separator 12 into upper level 14 and lower level 16 into which objects to be coated are placed through loading door 18. The chamber is initially purged of oxidative or other non-hydrocarbon material by source 20 of pure inert gas such as a tank of high-purity compressed helium. After the chamber has been purged the inert gas is shut off and the chamber is opened to source 22 of pure hydrocarbon feed gas, such as a tank of high purity compressed methane. Means for switching between the purge and feed gas may be easily effected by any

conventional valve configuration. The hydrocarbon feed gas is regulated by means of flow meter 24 at a precise flow rate, the gas entering porous frit element 26 such that the gas flow is diffused so that no mass convection occurs. The gas exiting the frit immediately enters decomposition chamber 27 which is uniformly heated by means of upper heater 28 and lower heater 30. The temperature of decomposition chamber 27 is such that the gas is virtually instantaneously decomposed into carbon and hydrogen ionic species. The ions travel through a magnetic ion separator 32 which has been preset to a magnetic field strength such that the ionic species entering the field emerge separated into two ionic layers, the upper layer being positive and negative hydrogen ions and the lower layer comprising different ionic carbon species. The lighter hydrogen ion species pass into upper level 14 of the deposition chamber while the heavier ionic carbon species are deflected into lower level 16 of said deposition chamber, the layers being maintained separate by the aforementioned refractory separator 12. The hydrogen ions in the upper level pass through a set of discharge plates 34 which promote the uniform recombination of said species to hydrogen gas which exits the upper level at 36 through a pinch valve and is bubbled through water trap 38. The carbon layer in lower level 16 adherently deposits on objects 40 which have been placed therein and forms a black metallic sheened layer thereon. Any carbon ionic species which is not so deposited exists from lower level 16 through orifice 42 and travels into water trap 44 wherein it forms carbon black. Both upper chamber 14 and lower chamber 16 are uniformly heated by means of upper heater 46 and lower heater 48. The apparatus incorporates suitable means for controlling the plate voltages 50, the magnetic field strength for separating ions 52, and for controlling temperature 54.

[0013] Any suitable object may serve as a substrate for the deposition of cubic carbon, the only requirement being that it be small enough to be placed within the deposition chamber and that it withstand the temperatures at which deposition takes place. Thus the object will generally be fabricated of metal or some suitable ceramic. Cubic carbon can be deposited on any article so long as it satisfies these requirements, and any attempt to detail a list of such objects would be meaningless.

[0014] The feed gas which serves as a source of carbon is advantageously a light hydrocarbon such as methane, ethane, propane, butane, pentane, or branched isomers thereof, although the aforesaid list is by no means meant to be exhaustive or limiting. Preferred are hydrocarbon gases having one to six carbon atoms, particularly methane.

[0015] To deposit a layer of pure crystalline cubic carbon, the only restrictions which need be observed are that the gas be a true hydrocarbon, that the gas be of very high purity, and that the gas exhibit a vapor pressure high enough to be regulated by means of conventionally employed flow metering devices. Thus, a hydrocarbon having an extremely high vapor pressure which is yet liquid at room temperature, such as isopentane, would be suitable for use with this invention. By the term "high purity" it is meant that the gas have a purity of at least 99.9 mole percent.

[0016] It is noted that high purity gases are required only if high purity coatings of cubic carbon are desired. As it will be shown, for certain applications it is advantageous to in fact include impurities as part of the feed gas. Moreover, if the apparatus is constructed without gas metering devices such that a gas cannot be used, heavier hydrocarbons such as oil, gasoline, benzene etc. can be used by being contained e.g. in a ceramic or other high temperature container and placed within the apparatus prior to heating. The means used to heat the deposition chamber can suitably be an induction furnace which satisfies the requirement of heating the chambers evenly.

[0017] For decomposition it has been found advantageous to use temperatures in the range of  $900 < 0 > ^\circ\text{C}$  to  $2400[\text{deg.}]\text{C}$ . For temperatures up to  $1400 < 0 > ^\circ\text{C}$  the vapor ionizer may be fabricated of quartz. At higher temperatures up to those not exceeding  $2000[\text{deg.}]\text{C}$  the ionizer may be fabricated of alumina. For still higher temperatures the ionizer may be fabricated of zircon up to temperatures of  $2600[\text{deg.}]\text{C}$ . The material most preferred for use at lower temperature is clear quartz because, advantageously, one may then view the physical processes occurring in the apparatus.

[0018] As previously mentioned, precise control of flow rate at a sufficiently low value to prevent mass convection is fairly important. Admission of the feed gas at a rate above a critical flow rate characteristic of a given apparatus having a set volume results in mixing and recombination of the decomposed gaseous constituents such that no deposition of cubic carbon will occur. For the cylindrical quartz tube employed in the experiments leading to this invention, which tube had a diameter of about 2 inches, a flow rate of about 70 cc/min. of methane gas was found to be critical.

[0019] As shown by Figure 2, pure (i.e. undoped) cubic carbon exhibits an X-ray diffraction pattern which is quite distinctive and which shows a single, well defined peak rising above the background, the single peak being indicative of the single-crystal nature of the pure material. Figure 2 is intended then to represent pure cubic carbon and serves as a label by which one of ordinary skill may determine that he has in fact achieved the substance. The X-ray diffraction pattern shows the film to be a mixture of amorphous and crystalline carbon that has a distinct preferred orientation to its diffraction pattern. This orientation is no doubt due to the way the film is deposited on the substrate. The spectrographic analysis showed Ti and Si and other elements that were suspected to be due to the substrate composition and not the cubic carbon film itself. The X-ray diffraction pattern further indicates that the crystal structure is face-centered cubic.

[0020] As each crystalline material has a unique X-ray diffraction pattern, comparison of the X-ray diffraction pattern of an unknown material with the X-ray diffraction pattern for known materials permits qualitative identification of the unknown material. Since cubic carbon possesses unique X-ray diffraction pattern use of this technique permits positive identification of cubic carbon and readily distinguishes it from other forms of carbon.

[0021] Figures 3-5 show the highly magnified surface of a substrate (quartz) which had not been coated with cubic carbon. As can be seen from these electron micrographs, no ordered structure can be seen although the surface can be seen to exhibit definitely shaped constituent particles, as shown in progressively pronounced fashion as the magnification increases from 40,000 (figures 3 and 4) to 70,000 (figure 5).

[0022] Figures 6-9 show the changing appearance of the substrate of figures 3-5 once a layer of cubic carbon has been adherently deposited thereon. As can be seen at the lower magnification (1500X) of figure 6 the coating appears homogeneous and well defined as small spheres. Figure 7 additionally shows the coating at low (1500X) magnification, but wherein the highlighted (lightened) section has been further magnified up to 7500X and shown as the right hand portion of the micrograph. In this enlargement the spherical and homogeneous nature of the single crystal structure shows up even more clearly. Figure 8 shows magnification of the surface of 5000X while figure 9 shows extreme magnification to 25000X and offers what amounts to a close look at the single crystal spherical shape of cubic carbon particles. Thus figures 6-9 offer a continuum of magnifications of a cubic carbon coating and detail the microscopic characteristics of this substance.

[0023] The chief characteristics of pure cubic carbon are its extreme hardness and inertness as displayed, for example, by its virtual total lack of response to oxidation and corrosion by any acid, base, or gas at temperatures up to  $600[\text{deg.}]\text{C}$ . Figure 10 is an electron micrograph of crystalline cubic carbon deposited on tungsten after having been subjected to corrosion testing. There is no visual change in the micrograph from that which would be obtained for freshly deposited cubic carbon;

[0024] Cubic carbon additionally has a wide range of uses. Its diamond-like hardness extends the life of tungsten-carbide cutting tools by a factor of about 3 when deposited thereon in a thickness of about 0.1 microns. Its extreme inertness allows virtually total protection of objects used in caustic/oxidizing/acidic and generally corrosive environments such as that found in acid generators or electrolysis cells. For such debilitating environments expensive relatively inert metals used normally such as titanium to fabricate e.g. acid containers can be replaced by cubic carbon-coated aluminium (or other relatively cheap material) which is even more inert and which can be produced at a fraction of the cost.

[0025] Moreover, cubic carbon can be used to make very inexpensive junction devices which function as solar cells. A ceramic element, such as a porcelain bar is allowed to be coated to a depth of a couple of microns (ca. less than 10) with cubic carbon as described previously for the reactor. The bar so coated is then plated on its ends with two different metals, e.g; copper at one end and nickel at the other, but leaving the middle portion of the bar coated only with cubic carbon. By suitably attaching electrical leads to the copper and nickel end portions and connecting an ammeter to the leads a current will be seen to flow when the bar is exposed to sunlight or radiation (heat). Thus the bar so coated operates as a junction device which transduces sunlight or heat to electrical energy.

[0026] Much energy is wasted, for example, in normal cooking operations because heat from the gas burner or electric range element is dissipated into the surrounding atmosphere. Too, generally high power levels must be applied only to the bottom of a cooking vessel to achieve relatively lower cooking temperature therein. By coating a cooking vessel, e.g. a ceramic or high temperature glass dish which one normally places in an oven, with cubic carbon (1-5 microns) and attaching suitable electrical leads to allow connection to an ordinary wall outlet, the cubic carbon coating dissipates the electrical energy quickly and transduces it to heat over the whole surface of the vessel. Thus by simply coating the cooking surface of a cooking vessel with cubic carbon and attaching to a wall outlet, the coated dish will quickly reach a constant temperature determined by the resistance of the coating, which resistance in turn depends on the thickness of the coating and whether it has been doped with any impurities which facilitate heat transfer through the coating. No high power levels are required or oven-type environments which waste heat by heating the surrounding air space as well as the vessel itself.

[0027] In this regard cubic carbon may be intentionally doped with elements which result in the formation of carbides which affect (i.e. facilitate) the electrical conductivity by disrupting the pure cubic carbon crystal structure with molecules which transform a non-conductive material into a semi-conductive one. Such impurities are metals (e.g. aluminium, magnesium, beryllium, calcium, aluminium etc.) or other substances such as sulfur or nitrogen. The doping materials may be incorporated by being included as part of the initial hydrocarbon gas used to form the coating (if the impurities are themselves gaseous) or may be added to the decomposition chamber in a pre-calculated amount (i.e. an amount which has been empirically determined to produce a desired level of doping) wherein the impurity vaporizes in the decomposition temperature and deposits onto objects to be coated along with the carbon phase. There is no hard-and-fast rule for predicting what effect a given impurity will have. The properties of any doped cubic carbon coating must be determined on an impurity-by-impurity basis, but it is emphasized that such determination is well within the scope of this invention.

[0028] Thus by doping a cubic carbon coating with a predetermined amount and type of doping material, a coating having a pre-selected resistance may be achieved.

[0029] Further, it has been found that cubic carbon coated heat exchange elements are much more efficient than an element coated with some different substance such as black paint. Thus flowing a cool fluid such as water through a cubic carbon-coated coil will result in much greater energy absorption and heat transfer to the cool fluid than with other heat absorbing coatings operating simply on the principle of being a black surface.

#### **Example 1 - Manufacture of cubic carbon.**

[0030] A quartz tube having therein a divider separating it into upper and lower chambers and preheated to oven 900[deg.]C was flushed with helium (inert gases like N<sub>2</sub> will function equally as well). The chamber was equipped as per figure 1, having heating elements to heat it evenly and a magnetic deflector to separate ions from decomposed gases. Various objects to be coated had been placed in the lower chamber including alumina crucibles, metal screws, and glass plates coated with alumina.

[0031] The ends of the upper chamber were sealed except for a small aperture at one end of the tube to which was attached a length of tubing which extended into a condenser with water to permit gas to escape from the ceramic tube without permitting atmosphere to re-enter therein.

[0032] A pinch valve was applied to the tubing to permit the aperture to be sealed off.

[0033] The heating elements were turned on and the temperature of the upper and lower chambers were raised to 900[deg.]C. Following this, high purity methane gas was allowed to enter the chamber and the temperature was maintained at 900[deg.]C for approximately 10 minutes until the methane had completely decomposed. The tube to the trap was sealed with the valve and the chambers were allowed to cool to room temperature.

[0034] The objects which had been placed in the lower chamber were removed and examined and it was observed that a coating several hundred microns thick had been deposited upon all of the objects within the lower chamber. Using this procedure with more methane and longer exposure periods, coatings of several hundred microns have been deposited on various objects.

#### **Example 2.**

[0035] One end of the combustion tube, including both upper and lower chambers, was fitted with a quartz glass window. In this manner, it became possible to view the reaction and observe the deposition of the cubic carbon on the surface of the objects placed therein. As in Example 1, the opposite end of the chamber was sealed except for the pressure relief aperture which led to a dry ice trap which permitted the interior of the chamber to be isolated from the oxygen in the atmosphere.

[0036] A boat crucible containing a few drops of crude oil composed of both heavy and light hydrocarbon fractions was placed into the decomposition chamber and various metal and ceramic objects were placed throughout the lower chamber including immediately adjacent the quartz glass window. The temperature was raised to approximately 1100[deg.]C and the process was observed through the quartz window.

[0037] At the elevated temperature the mixture of light and heavy crude was seen to decompose into two distinct layers of vapor within the upper chamber. The upper layer was believed to be composed of reducing hydrogen gases and the vapors of contaminants in the oil, such as sulfur. The lower layer was observed to be a heavy bluish-white cloud and was believed to be a stationary carbon ion cloud.

[0038] As long as any crude remained in the boat, material was produced which resolved into the two layers or zones. The evolution of these gases produced a positive pressure within the upper chamber such that the hydrogen continually flowed from the upper chamber into the dry ice trap. The gases in the upper zone flowed easily and exited the tube by way of the pressure relief aperture. The bottom layer, i.e. the carbon ion cloud, appeared to be much heavier and remained relatively stationary in the lower portion of the upper chamber, entering into the lower chamber through portholes.

[0039] Only those portions of the objects exposed to the carbon ion cloud were coated. Objects exposed to the upper chamber's vapors were not coated at all and lacked and deposited coating or metallic luster.

### **Example 3.**

[0040] As in Example 2, crude oil containing varying percentages of lighter boiling fractions were examined. It was observed that the size of the vaporous cloud in the upper zone in the chamber was larger as the percentage of the light end fraction in the hydrocarbon was increased. It is noted that the lower carbon ion cloud was maintained as long as there was a positive flow of upper zone material leaving the exit aperture.

[0041] If desired, methane can be fed continually into the decomposition chamber to produce a positive flowing system wherein the upper fraction continually exits the upper chamber and is replenished by incoming new methane. In this manner the zone of carbon ions is sustained and cubic carbon is deposited in any desired quantity on the objects in the lower chamber. Vapor density is kept at the saturation level for best results.

[0042] A variety of materials and objects have been plated with the new crystalline carbon form in the manner detailed in the above examples. At present, cubic carbon has been found to deposit and tightly bond to quartz, ceramics, and metals with or without an oxide film. Further, deposition takes place regardless of the shape or size of the objects.

### **Example 4.**

[0043] An aluminium crucible containing a specimen of hydrocarbon consisting of a very heavy crude oil was placed in an induction furnace which had been purged with an inert gas. The induction furnace was connected to one end of high density graphite decomposition tube three inches in diameter and six feet in length. A small aperture was provided at the opposite end of the graphite decomposition tube to which a gas trap was attached as previously described. Objects to be coated with cubic carbon were placed inside the graphite decomposition tube.

[0044] The induction furnace was raised to a temperature of 1100[deg.]C and the carbon ion vapor which evolved from the decomposition of the heavy crude oil was conducted from the induction furnace into the graphite decomposition tube to be deposited upon the objects therein. Thereafter, the temperature of the induction furnace was reduced and the objects were removed from the graphite decomposition tube.

[0045] Upon examination of the objects coated in this manner, it was observed that the coating on those objects within the combustion tube and nearest the induction coils was smooth, very bright and lustrous. The coating on objects were progressively less shiny and more dull and grey as the distance from the induction coils increased. On some objects a mixture of cubic carbon and needle crystalline carbon from graphite vapor was observed.

[0046] Observations of the many variations of the process of this invention as illustrated in the above example indicate that a vessel containing a carbonaceous material such as heavy crude oil or tar may be connected to the interior of a suitable furnace, such as an induction furnace, such as an induction furnace by an appropriate means such as a ceramic tube. The furnace may, in turn, be interconnected with a large chamber. If desired, the furnace could be located as a well or pit portion within the larger chamber. Thereafter, the oxygen in the atmosphere of the furnace and chamber could be removed by purging the chamber and furnace with an inert gas such as N or helium. The chamber could then be heated to a convenient temperature (above about 600[deg.]C) and the induction furnace portion could be heated to greater than 900[deg.]C, preferably about 1100[deg.]C, and as high as 2400[deg.]C.

[0047] With such an arrangement the carbonaceous material could be added dropwise continuously to the induction furnace portion and, therefore, the carbon ion cloud could be produced continuously. By this means, the cubic carbon could be deposited upon objects located within the chamber in whatever quantities might be desired.

### **Example 5.**

[0048] The ceramic combustion tube of the previous examples was replaced with a quartz glass decomposition tube having an internal diameter of approximately 1-1/2 inches and a length of approximately 4 feet. The quartz glass decomposition tube was placed in the furnace such that the midpoint of the tube was within the furnace and the end portions of the quartz tube extended outside the furnace. Articles to be coated were placed inside the quartz tube and the ends of the quartz tube were sealed with laboratory stoppers.

[0049] A length of one-quarter inch OD quartz glass tubing was inserted through one rubber stopper at one end and plastic tubing was attached thereto. The other end of the plastic tubing was attached to a water air lock. As in the previous examples, positive pressure permitted the gas to escape but atmospheric oxygen was not permitted to re-enter.

[0050] The other end of the quartz decomposition tube contained a laboratory stopper with a length of one-quarter inch OD quartz glass tubing passing therethrough. A cylindrical alumina tube having one open end was placed over the end portion of the quartz glass tubing located within the decomposition chamber. Refractory wool was packed within the annulus between the alumina tube and the quartz glass tube to filter gases passing through the quartz tubing into the quartz decomposition chamber.

[0051] A length of plastic tubing was attached to the opposite end of the quartz glass tubing located on the exterior of the decomposition chamber. A hydrocarbon gas source comprising a bottle of natural gas with a needle valve was attached to the exterior end of the quartz tubing in a convenient manner. In this manner the hydrocarbon gas could pass from the gas source, in turn, into the length of quartz glass tubing, the annulus between the ceramic tube and the quartz tubing continuing through the refractory wall filter, and into the quartz decomposition chamber. Thereafter, gas within the chamber could exit through the quartz tubing at the opposite end of the decomposition chamber and escape through the air lock.

[0052] The furnace was turned on and the temperature at the center portion of the quartz glass decomposition chamber was raised to approximately 1000[deg.]C. The hydrocarbon gas was introduced very slowly into the decomposition chamber and the gas was allowed to react with the oxygen within the chamber. After the oxygen in the chamber was exhausted, the hydrocarbon gas decomposed to deposit upon

the substrates within the chamber a bright coating of cubic carbon.

[0053] It has been found that any hydrocarbon can be used in the process to produce cubic carbon. However, to produce a deposit of highest purity and with the least number of cubic carbon crystal discontinuities or defects, the choice of hydrocarbon and flow rate must be controlled. Using this process, crude oil, gasoline, benzene, alcohol, olefins, acetylene, aromatics and hydrocarbon gases such as methane, ethane, butane, etc. have been used with success.

[0054] In order to produce a high purity cubic carbon which is primarily single crystal sheet, it is extremely important that even a trace amount of contaminants such as oxygen or sulfur be kept out of the decomposition tube. It has been observed that alkanes of six carbon atoms or less produce cubic carbon coatings with the least crystal discontinuities. It is presently preferred to use high purity methane or other lighter hydrocarbons as the carbon source for the process of the present invention.

#### **Examples 6 to 10.**

[0055] Using the same apparatus as described in Example 4 the decomposition chamber containing the desired substrate to be coated was heated to 1100[deg.]C. A volume of hydrocarbon gas or vapor was fed into the decomposition chamber within one minute and the temperature maintained for ten minutes. Thereafter, heating was terminated and the decomposition chamber was allowed to cool to room temperature and the substrate sample was discharged.

[0056] Using this procedure, two-dimensional single crystal sheets of cubic carbon were deposited on quartz glass using butane, ethylene, heptane, methane, benzene, acetone and crude oil. Table 1 following illustrates how the number of crystal discontinuities and impurities, as reflected by the specific resistance of the cubic carbon, are effected by the starting material.

#### **Examples 11-13**

[0057] An alumina substrate was placed in the decomposition chamber as described in the above examples, the furnace was heated to 1500[deg.]C and a volume of butane gas was injected into the decomposition chamber within approximately one minute. After ten minutes the chamber was cooled and the substrate discharged. Table 2 which follows illustrates the variation and specific resistance of the metallic carbon deposit as a function of carbon concentration.

#### **Example 14.**

[0058] In a series of tests a metal article was placed within a decomposition chamber and the chamber was purged with helium until all oxygen was removed. The decomposition chamber was heated to 1000[deg.]C and 200cc of butane gas was fed therein. At the end of thirty minutes, the decomposition chamber was cooled to room temperature and the metal article was removed. Using the procedure articles of tungsten, titanium, platinum, molybdenum, silicon, niobium, carbon steel and byrellium were coated with a metallic carbon layer rendering the metal article completely resistant to any corrosive gas or liquid and/or combinations thereof.

[0059] Upon examination, objects coated with cubic carbon have a bright cubic luster similar to chromium metal. The visual appearance varies depending upon the habit of the cubic carbon crystal. This crystal habit can be effected by the reaction temperature, flow rate of starting material, concentration of carbon ions in the reaction atmosphere, impurities, vapor density, and the texture of the surface upon which the cubic carbon is deposited.

#### **Example 15.**

[0060] Ceramic crucibles which had been coated with cubic carbon were placed inside a furnace in the absence of air and elevated to a temperature of approximately 2400[deg.]C for several hours. Upon examination, the coating on the crucibles showed absolutely no sign of degradation.

[0061] The coated ceramic crucibles were placed inside a muffle furnace and elevated to 1100[deg.]C in the presence of oxygen. It was observed that the coating very slowly volatilized without evolution of smoke and vanished leaving no residue. At temperatures up to approximately 600[deg.]C and in the presence of air no visible degradation has been observed at the end of time spans as long as forty-eight hours.

#### **Example 16.**

[0062] Cubic carbon was found to vary from electrically conductive to semiconductive depending upon the relative purity and crystal continuity of the coating. One object coated in this manner was a small boat crucible composed of a smooth refractory ceramic. Using conventional electroplating methods, the crucible was connected to a terminal of a voltage source and electroplated with a layer of nickel metal.

#### **Example 17.**

[0063] Another object coated with the new cubic carbon was a small ceramic tube approximately two inches long and having an outside diameter of approximately one-quarter inch. Using standard methods, a band of nickel approximately one-quarter inch wide was electroplated at each end of the ceramic tube. The leads of a voltage source were attached respectively to each of the nickel bands.

[0064] Due to the thin coating of the semiconductive cubic carbon on the surface of the ceramic tube, the tubing acted as a resistor. An electric current was passed through the coating on the ceramic tube causing the temperature of the ceramic tube to rise until it glowed red. It was observed that as the temperature of the cubic carbon increased, its resistance decreased. That is, the cubic carbon exhibited a negative thermal coefficient of resistance. After this test, the coating on the ceramic tube was examined and no sign of oxidation or decomposition was observed by visual inspection and the material maintained its bright metallic luster.

#### **Example 18.**

[0065] By connecting an ammeter across the leads of the tube of Example 17, a current of 50 microamps was generated upon heating the tube. Using this procedure heat can be converted to electricity at temperature as low as 130[deg.]F.

**Example 19.**

[0066] Tests were made for solubility or etchability in acetone, xylenes, isopropyl alcohol, sulfuric acid, hydrofluoric acid, hydrochloric acid, nitric and phosphoric acids. Tests were made using full strength reagents at room temperature. Samples were also boiled for periods ranging from hours to days, washed in de ionized water and then examined using scanning electron microscopy techniques to compare them with an untreated control. None of the solvents or acids had any noticeable effect.

**Example 20.**

[0067] Pieces of alumina tubing coating with a thin film of cubic carbon were exposed alternately to hot hydrofluoric, hydrochloric, and nitric acids for extended periods of time without observed corrosion or decomposition of the cubic carbon coating. Thereafter, the substrates coated with the cubic carbon were alternately exposed to hot sodium hydroxide, potassium hydroxide, and ammonium hydroxide without any visible reaction. In fact, at the present time, no attempted combination of acid, base or organic compound has been shown to corrode an article coated with cubic carbon.

**Example 21.**

[0068] Tungsten carbide cutting tools have been coated with cubic carbon at a thickness of one micron with a consequent doubling of the life of the cutting edge.

**Example 22.**

[0069] A china cup was coated with cubic carbon and two electrical connections were made to the coating. The cup was filled with water and a power source of 25 watts was applied to the electrical connections. Within one minute the water was boiling in the cup.

[0070] Because of the unique properties as demonstrated by the above examples, cubic carbon is useful in the manufacture of an article which must resist corrosive environments at high temperatures. Further, the ability of the cubic carbon to be very strongly bonded or diffused to a surface such as ceramic and thereafter permit the surface to conduct electricity to permit the substrate to be electroplated in a conventional manner with a metal provides a method of permanently bonding an electrically conductive surface to a non-conducting ceramic.

[0071] Although only a few exemplary embodiments of this invention have been described in detail above, those skilled in the art will readily appreciate that many modifications are possible in the exemplary embodiments without materially departing from the novel teachings and advantages of this invention. Accordingly, all such modifications are intended to be included within the scope of this invention as defined in the following claims.

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**US4180444****Electrolytic methods employing graphitic carbon cathodes and inorganic complexes produced thereby**  
[ [PDF](#) ]

Process in which an electric current is passed between a cathode and a graphitic carbon anode graphitic carbon and / or a non-alkaline metal of Groups I to VIII of the Periodic Table, and through an electrolyte solution comprising aqueous NH<sub>3</sub> compound, NO<sub>x</sub>, SO<sub>2</sub>, H<sub>2</sub>S which can be reduced or hydrogenated to the cathode of a hydride, which reacts with the cathode to form a soluble inorganic complex in water containing graphitic carbon and / or this metal.

**SUMMARY OF THE INVENTION**

The present invention is directed to unique electrolytic methods employing graphite or a suitable equivalent as at least one electrode, the cathode, and preferably as both electrodes. Inorganic, water soluble, complexes, which appear polymeric, are produced, the nature and utility of which will depend on the nature of the particular electrolyte utilized and whether one or more metals are also introduced into the reaction.

The term "graphitic carbon", as used throughout this disclosure, is intended to refer to graphite and materials which are the functional equivalent of graphite in its characteristics of conducting electrons and absorbing and retaining within its structure both ionized and molecular gases, compounds and complexes with which it comes into intimate contact, most typically through contact with solutions.

More specifically, the present invention provides for electrolytic methods whereby inorganic, water soluble complexes are produced, which methods comprise passing a current between two electrodes, at least one of which is a graphitic carbon cathode, as defined above, through an electrolyte which is an aqueous solution of any compound or complex of compounds capable of being absorbed and retained by the graphitic carbon of the cathode and reduced or hydrogenated at the cathode to a hydride species. The hydride species which are electrically charged are transported directly to the anode surface, and the anode is of a material which reacts with such electrically charged species to produce the desired water soluble complex.

It has been found that when graphite or its functional equivalent as explained above is placed into intimate contact with certain chemical materials, the contact most typically being in an electrolytic environment, these chemical materials are absorbed into the structure of the graphite. Most typically, it has been found that gases will be absorbed by the graphite, both those which are generated by the electrolytic action at the graphite electrode, but also gases which have been absorbed into the electrolytic medium. The present invention utilizes this phenomenon to provide compounds and complexes which, when in the reduced or hydrogenated state resulting from cathodic action, react with a sacrificial anode to produce inorganic complexes which appear polymeric in nature and are water soluble.

It is a feature of the invention that the sacrificial anode employed in the electrolytic methods utilizing a graphitic carbon cathode may, itself, be a graphitic carbon electrode. In this embodiment, the invention provides a technique for dissolving graphitic carbon and producing unique, inorganic, water soluble complexes containing graphitic carbon. These complexes are valuable products due to their gas absorbing properties, i.e., absorb gases such as SO<sub>2</sub> and H<sub>2</sub>S in pollution control processes and can be used effectively as catalysts, such as in the conversion of wood or starch to dextrose and in the hydrogenation of coal, peat, etc.

It is also a feature of the present invention, as will be more fully explained herein, that the sacrificial anode employed in the electrolytic methods of the present invention may be any non-alkaline metal selected from Groups I-VIII of the Periodic Table. Thus, the present invention

provides methods of providing solutions of water soluble, inorganic complexes containing metals, some of which have heretofore never been available in aqueous solutions. These metal-containing solutions have significant utility in techniques such as plating from aqueous solution, both with and without electrolysis. These metal containing complexes also have utility as gas absorbants in pollution control processes.

More specifically, the particular reactants employed in the method of the present invention will depend primarily upon the properties of the inorganic complexes desired as end products. For example, inorganic complexes prepared using an anode of silicon, aluminum, graphite or mixtures thereof with an aqueous solution of ammonia as the electrolytic solution will have valuable utility in fertilizer compositions. Complexes prepared using aluminum as the anode in an electrolytic solution containing sulfur dioxide have been demonstrated to have a valuable utility in the absorption of SO<sub>2</sub> or H<sub>2</sub> S gases from stack gas mixtures. Complexes prepared with refractory metals, such as tungsten, titanium, molybdenum, and the like, are, because of their water soluble characteristics, useful in plating from aqueous solution.

As indicated above, the complexes appear polymeric in nature. They can be dried to polymer-like films which can be redissolved in water. The dried films of complex exhibit the unique characteristic of sublimation.

The essential materials necessary in the method of the present invention are the following:

A gas-absorbing, graphitic cathode;

A sacrificial anode, which may be an identical material to the cathode but also may comprise any non-alkaline metal selected from Groups I-VIII of the Periodic Table, with the proviso that the metal is capable of electrolytic dissolution in the present method, a mixture of such metals, or a mixture of one or more such metals with graphite; and

an electrolyte containing essentially any compound capable of being absorbed into the structure of the graphitic cathode and reduced or hydrogenated at the cathode to produce electrically charged hydride species such as NH<sub>x</sub>, SH<sub>x</sub>, PH<sub>x</sub> and the like, wherein x is an integer representing the number of hydrogen atoms in the electrically charged hydride species. This electrolyte compound is preferably selected from the group consisting of ammonia and hydrides and oxides of nitrogen, sulfur and phosphorus.

The present invention is based upon the discovery of a combination of simultaneous physical occurrences, all of which relate to the unique characteristics of graphite and its use as a cathode. In a traditional electrolytic process wherein ammonia or a dissolved hydrogen-producing gas is in the electrolyte, the electrolysis typically produces a bubbling off of hydrogen gas at the cathode. It has been discovered, however, according to the present invention, that if graphite or an equivalent is utilized as the cathode, some of the hydrogen evolved in the electrolytic process will be absorbed and retained at the cathode. This hydrogen, possibly in ionic form, will then be available to react with any electrolyte compounds which are also absorbed by the graphite, and which may be in a reduced state as a result of the cathodic action. This results in the production of additional hydrogen (perhaps in atomic form) as well as the creation of negatively charged radicals containing hydrogen, i.e., hydride species.

Also, in the electrolytic method the hydrogen and the hydride species which are generated at the cathode will begin to migrate to the anode as if traveling along the lines of the electric field. These groups will then react with the positive ions at the anode to begin to produce inorganic complexes soluble in the electrolytic solution.

#### **BRIEF DESCRIPTION OF THE DRAWINGS**

**FIGS. 1A-E illustrates a series of infrared spectra of a inorganic complex produced according to the present invention after such complex had been vacuum-dried and heated at progressively higher temperatures;**

**FIG. 2 is a reproduction of an infrared spectrum of a complex produced according to the present invention employing graphite as both cathode and anode with an electrolyte containing dissolved NH<sub>3</sub> ; and**

**FIG. 3 is a reproduction of an infrared spectrum of a complex produced according to the present invention employing a graphite cathode, a graphite anode and SO<sub>2</sub> gas in the electrolyte.**

#### **DETAILED DESCRIPTION OF THE INVENTION**

##### **The Cathode**

It is an essential requirement of the invention that the cathode in all instances be comprised of graphitic carbon, or its functional equivalent.

Graphite, of course, is a naturally conductive material. That is, it is capable of carrying electrons and is a very effective electrode in electrolytic procedures. Unlike most conductive materials, however, graphite also has the capability of absorbing and retaining within its structure gases such as hydrogen, ammonia, sulfur dioxide, hydrogen sulfide and the like. This gas-absorbing capability arises out of the unique physical structure of graphite.

It was long ago established by X-ray analysis that the carbon atoms in graphite are arranged in layers, each containing a continuous network of planar hexagonal rings. Within each layer, the carbon atoms are held together by strong, covalent bonds. The layers themselves, however, are held together by comparatively weak forces.

The separation between the graphite layers has been measured to be 3.35 Angstroms. It has further been determined that within each layer, each carbon atom is surrounded by only three others, in a uniform hexagonal configuration, similar to that of benzene, wherein the distances between the carbon atoms are all equal at 1.415 Angstroms. Thus, it is apparent that many atoms, molecules and ions may be inserted between the carbon layers and even, to some extent, within the graphite structure itself. This ability to absorb gases and to maintain them inside the graphite structure permits reduction reactions to be carried on inside the electrode itself.

One way in which it has been demonstrated that the graphite electrode actually absorbs certain gases and causes an internal reduction reaction to occur when used as a cathode is to take the cathode after it has been used in the reaction of the present invention and submit it to degassing techniques.

The many forms of so-called amorphous carbon, such as charcoal, soot and lampblack, are all actually microcrystalline forms of graphite. The microcrystals may be so small as to contain only a few unit cells of the graphite structure. Nevertheless, the graphite structure exists and thus



these forms of carbon, with their ready ability to absorb large amounts of gases and solutes from solution, may be utilized as generally less preferred substitutes for the graphite cathode. Graphite is generally preferred because of its ability to be molded into a convenient shape for use as an electrode and for its good conductivity.

As will be more fully explained subsequently, the reaction method of the present invention has been carried out employing a graphite anode, as well as a graphite cathode. In such processes, graphite from the anode is dissolved into the solution. It has been observed that the dissolved graphite which begins to appear in solution will, itself, act as a cathodic electrode. Power may be shut off and yet there will still be a current flow due to the ability of the dissolved graphite particles to absorb and collect negative charges.

In the unique processes of the present invention, the reduction which occurs inside the graphitic cathode produces electrically charged species which are extremely reactive and will readily give up electrons when they contact the surface of the anode. This results in the formation at the anode of soluble inorganic complexes containing as a backbone thereof the material of the anode. Because they are formed from species which have been created at the cathode by reduction and reaction with hydrogen, these compounds will typically have a hydride-like linkage contained in the body thereof.

The reduction process which occurs inside the cathode will also occur at or near the surface of the cathode. In many instances, reduction of dissolved metal compounds will result in a plating out of the metal compound on the cathode surface. This does not necessarily stop the production of the species which result in the formation of the unique products of the present invention. It has been observed that the coatings which form on the surface of the cathode are porous and, to some extent, permit passage of the gases and other species generated inside the cathode itself. However, it has been observed that with certain particular combinations of reactants the cathode will have to be replaced from time to time in order to continue the formation of the inorganic complexes of the present invention. The need for replacement of the cathode can be easily observed when carrying out the electrolytic method.

Most typically, the electrolytic reaction of the present invention, more specifically, the absorption by the cathode of hydrogen, hydrogen ions and gases or other compounds dissolved in the electrolyte and the subsequent reduction and/or hydrogenation of these materials, results in the production of hydride species which, when transported by the electric field to the surface of the anode, produce the inorganic complexes. As will be apparent to those skilled in this art, the larger the graphite cathode which is utilized, the more surface area and internal structure will be available for absorption and retention of the electrolyte compounds and their subsequent reduction to produce charged radicals desirable to carry out the reaction of the invention. Accordingly, where such processes as dissolution of metals are desired, reduction will be emphasized and the size of the cathode should be adjusted accordingly. On the other hand, where oxidation type reactions are to be emphasized, as for example utilizing a graphite cathode and a graphite anode, it is the graphite anode which should be increased in size in relation to the cathode. This can be done most simply by placing chunks of graphite material in the bottom of the reaction flask. These chunks of graphite material will then act in union with the standard anode electrode to form a larger anodic surface.

### **The Anode**

There are basically three variations in the broad concept of the present invention, each depending on the utilization of a different anodic system. In the first variation, a graphitic carbon anode is utilized with the graphitic carbon cathode. In this embodiment of the invention, where the electrolyte contains a compound such as a member of the group consisting of hydrides and oxides of nitrogen, sulfur and phosphorus (all of which are readily transformed, by reduction and/or hydrogenation, at the graphite cathode), the invention provides a technique for dissolving graphitic carbon and producing unique, inorganic, water soluble materials containing graphitic carbon.

It is particularly important when employing a graphite anode with a graphite cathode that the current used, in relation to the size of the reactor and electrodes, be such as to minimize any oxidation process, which may produce undesirable gases, such as acetylene. Essentially all that is necessary for carrying out the present invention is that a potential difference be developed between the electrodes and that the electrodes, of course, be positioned such that the electric field passes a current between the electrodes, thereby causing at least a portion of the gases evolved at the cathode during electrolysis to be transported physically to the anode.

Since the preferred hydride producing compounds are all weak electrolytes, there will be little hydrogen generated initially to be absorbed by the cathode, and the process may take several days before any significant dissolution of the graphite anode is visible. Here again, patience is important. The use of a stronger electrolyte, particularly an alkali metal compound, may result in an undesirable reaction and could cause an explosion.

It is a significant discovery resulting from the present invention that as the hydrides are developed according to the reaction and cause dissolution of graphite at the anode, the water soluble, inorganic graphite containing complexes produced thereby significantly increase the electrolytic conductivity of the solution, thereby increasing the rate of reaction. It is postulated that this increase in conductivity results from the graphite structure of these soluble products absorbing and retaining negative electrical charges.

In the second anodic variation, a base metal is utilized as the sacrificial anode. In this embodiment, it will be apparent that only those metals which are capable of dissolving during electrolysis may be utilized. It is also important that the two electrodes be properly positioned and that a small current be passed between them.

When utilizing base metal anodes, especially with highly conductive electrolytic solutions, the tendency will be for oxidation reactions to predominate. Thus, in the first place, the reduction process necessary to form the species which are essential in producing the inorganic complexes of the present invention will be masked. Secondly, oxidation may result in the plating out on the cathode of the base metal. This will have a tendency, in many instances, to slow down the electrolytic reaction. Thirdly, where the electrolyte contains an alkali, there may be some production of intermediate salts and/or oxides, which will crystallize out and collect on the surface of the electrolytic vessel and/or the cathode. These adverse reactions may be minimized by maintaining the current at a low level in the initial stages, but it may also be desirable to utilize a large graphitic cathode, as discussed above.

There is one exception to the limitations referred to above. This is, when a previously prepared inorganic complex containing graphite is utilized in the electrolyte. When such an electrolytic solution is utilized, those metals which do not normally dissolve electrolytically can also be used. In effect, any non-alkaline metal of Groups I-VIII of the Periodic Table may be utilized as the anode when the electrolyte contains graphitic carbon complex produced according to the present invention. The term non-alkaline metal is meant to embrace all metals excluding only the alkali and alkaline earth metals, such as barium, calcium and strontium.

Where the electrolyte contains no dissolved graphite material, unique inorganic complexes, soluble in water, may still be produced by employing as anodes certain base metals, such as aluminum, which dissolve normally upon electrolysis. Many complex inorganic, water

soluble products may be produced according to the invention wherein the base metal is associated with radicals which would not otherwise be capable of reacting with the metal.

The third anodic variation contemplates the use of both graphite and a non-alkaline metal selected from Groups I-VIII of the Periodic Table as the anodic electrode.

In this variation there are three possibilities, an electrode which is a physical combination of graphite with the particular metal, a graphite electrode with a quantity of the metal being placed in the bottom of the electrolysis vessel, and a metal electrode with a quantity of graphite placed in the electrolysis vessel. It is preferred according to the present invention to employ a system utilizing a graphite electrode as the anode, placing a quantity of the non-alkaline metal to be dissolved in the electrolysis vessel prior to setting up the electrolysis. This is because, as explained above, the graphite anode may be more readily positioned with respect to the cathode to produce an efficient and effective dissolution of graphite. Once the graphite is in solution, the electrolytic nature of the dissolved graphite complexes takes effect, as explained above, accelerating the efficiency of the process. Also, it is easier to control the direction of the electrolytic process by utilizing graphitic anodes and cathodes of varying relative sizes. One particularly preferred configuration is to utilize a large, hollow cylindrical graphitic cathode and a small cylindrical bar of graphite as the anode. The small electrode may be placed inside the hollow of the cylinder, the amount of insertion thereof providing a very effective control for the production of the most desirable current and electric field.

As noted above, when graphite is used as any part of the anode, the electrolytic process of the present invention is capable of dissolving any non-alkaline metal from Groups I-VIII of the Periodic Table. Whether the non-alkaline metal be comprised of the electrode itself or be placed in the electrolysis vessel as chunks, particles or even powder, the actual electrode must be positioned in relation to the quantity of material in the electrolysis vessel so that the total mass of the combination of non-alkaline metal and graphite acts anodically. It has been found not to be necessary in all instances to provide direct, physical contact between the graphite and the non-alkaline metal. As a matter of fact, in the preferred embodiment wherein graphite is employed as the anodic electrode, there is less of a tendency to produce oxidation products where there is no direct physical contact. It is believed that the production of dissolved graphite-based inorganic complexes actually facilitates the transfer of the charged radicals which attack and dissolve the non-alkaline metal.

### **The Electrolyte**

Generally speaking, in an electrolytic process an electrolyte is anything which is capable of carrying a current between two electrodes. In the present invention, the definition is almost as broad, the type of suitable electrolyte being limited only by such parameters as its ability to dissolve the complexes formed in the reaction process and, more importantly, the compounds which are to be absorbed, retained and reduced or hydrogenated by the graphitic cathode to hydride species which are thereafter transported through the electrolyte to the anode.

Accordingly, the present invention contemplates the use of any electrolyte containing in dissolved or ionized form a quantity of any atom, molecule, ion or complex which is capable of being absorbed and retained by graphite from an electrolytic medium and electrically transformed, by reduction or hydrogenation, to an electrically charged species capable of reaction with the sacrificial anode material, specifically hydride species, to produce an inorganic complex material which is soluble in water. The only apparent limitations are the size of the material in its unitary structure (atom, ion, molecule or complex) and its ionic nature, that is, whether it is capable of being reduced and/or hydrogenated to a state where it will react with the anode. Compounds of nitrogen, sulfur or phosphorus, specifically hydrogen-containing gases, such as  $\text{NH}_3$ ,  $\text{H}_2\text{S}$  and  $\text{PH}_3$ , as well as oxides such as  $\text{NO}_x$  ( $\text{NO}$  and/or  $\text{NO}_2$ ),  $\text{SO}_2$ ,  $\text{P}_2\text{O}_5$ , and the like, which are capable of being reduced to hydrides, are particularly valuable in the production of the inorganic, water soluble, complexes as will be further described. However, solutions of weak acid and dilute solutions of strong acids such as  $\text{H}_3\text{PO}_4$  can also be utilized as electrolytes in the present method.

It is characteristic of the reaction of the present invention that the reaction proceeds very slowly, sometimes taking days before some visible change has occurred. The nature of the electrolyte will to some extent have an effect on the rate of the reaction. However, typical means of speeding up an electrolytic reaction, such as heating or increasing the current, are generally not appropriate since these means will simply increase the traditional electrolytic type reactions or, in the case of ammonia, for example, result in an increased bubbling off of ammonia gas. However, in some cases, gentle heating has been beneficial.

While most of the preferred electrolyte compounds do not have a very high solubility in water (in the sense that they do not readily disassociate), the concentration of the electrolyte will ordinarily not be a critical factor. The acceptable concentration range for most electrolyte compounds is limited on the low side only to the extent that enough molecules be in the water to provide a continuous visible reaction and limited on the high side only by the concentration solubility characteristics of the compound.

It is preferred to initiate the reaction by having some electrolyte in the solution prior to passing a current between the electrodes. Since some of the preferred electrolyte compounds have a relatively low solubility ratio, and since the compound is used up in the reaction, it will be required in some procedures that there be an addition of compound to the reaction medium during the course of the electrolytic process. This can be by any suitable means.

As has been alluded to, certain compounds have such a low conductivity that it may be desirable to add to the solution, prior to or during the electrolysis, a small amount of a strong electrolyte having other than a neutral pH, such as an alkali hydroxide or the like, to increase the reaction rate.

Finally, the electrolyte may also contain an amount of inorganic graphite-containing complex previously prepared according to the present invention. As has been mentioned, the dissolved graphite particles apparently have the capability of absorbing electrons and therefore producing a very high conductivity. In addition, it appears that cathodic reduction takes place at or inside these dissolved graphite particles, thus further increasing the efficiency of the electrolytic process.

It should be noted that under some circumstances, at least when ammonia is utilized in the electrolyte, there is an intermediate product precipitated out on the sides of the electrolysis vessel during the course of the method of the present invention. This material is crystalline, has a six-sided crystal structure and is soluble in water. It appears to be an amide or hydride salt, formed by the reaction of  $\text{NH}_2$  groups with the graphite. It does not form a film when dried as does the final complex. When a non-alkaline metal is employed in the process, an intermediate precipitate also appears, but which is heavier, obviously due to the presence of the metal.

The appearance of this intermediate "salt" can be used as an indication of how the method is developing. If too much salt begins to form, this is an indication either that there is too much metal going into solution or that there is not enough ammonia in the system and metal hydrides are being formed. Heating the reaction will help the salt to break down and also not to develop too fast. Increasing the amperage of the electrolytic current will also to some extent offset the excess production of this intermediate and push the reaction back to the production of desired

complex.

Basically, the parameters for electrolysis are simple: keep the reaction relative cool and the current as low as possible. Most reactions proceed quite well at temperatures below about 60 DEG C. This basic rule will have to be modified occasionally to offset the production of excess intermediate. This can also be accomplished by maintaining the reaction vessel under a slightly elevated pressure. This helps to "push" the hydrogen gas evolved at the cathode into the system and also helps keep the dissolved gases from escaping at the anode. When ammonia is used, for example, too much heat and/or too much current will result in the escape of ammonia, both in the form of ammonia gas and also by a breakdown of the intermediate amide salt.

It is most important that the current density, that is, the average current per unit volume of reaction, be kept low. It is preferred that this be accomplished by maintaining a very high surface area for the cathode. As has been mentioned, too much current will cause the driving off of gases such as ammonia and may also cause adverse oxidation reactions to occur. Hence, as a general rule the wattage should be kept relatively low, the reaction drive force being maintained by having a very high surface area for the chosen electrode.

On a laboratory scale, it has been determined that best results are achieved with a current of about 0.1 to 4 amps and a low voltage, up to about 25 volts. It is apparent that higher voltages and/or currents might be utilized on a commercial scale and the determination of applicable parameters can be easily carried out by one skilled in the art. This is done by observing the course of the reaction, specifically the dissolution of the sacrificial anode, transfer of bubbles from cathode to anode and formation of the desired and side products. Preferably the reaction should be carried out to achieve a transparent solution, although slightly colored solutions indicative of larger crystals of graphitic carbon are acceptable.

The invention will now be described with particular reference to certain preferred embodiments, set forth in the following specific examples. Such examples are presented for the purposes of illustration only and to comply with the requirements of 35 U.S.C. 112, paragraph 1. They should not under any circumstances be deemed as limiting the present invention. In the examples, unless otherwise indicated, all percentages are by weight. A power source having a maximum output of 25 volts was utilized in the electrolytic processes. Where reference is made to a commercial ammonium hydroxide solution, it is intended to refer to a 26 Be DEG solution (which is 38% by weight NH<sub>3</sub>).

#### EXAMPLE 1

This example was carried out to illustrate the preparation of an inorganic graphite-containing complex according to the present invention, the complex containing NH and/or NH<sub>2</sub> groups. An electrolysis system was set up utilizing aqueous ammonia as the electrolyte and utilizing a graphite anode and cathode. The electrolysis vessel was a 6 liter boiling flask, the electrodes comprising graphite rods 12" in length and the electrolyte comprising 2,100 grams of 2,333 ml of aqueous ammonia (26 DEG Be). Into the electrolysis vessel were added 986 grams of additional graphite.

Initially, the graphite anode is placed in contact with the graphite in the bottom of the flask. The graphite cathode is placed at a distance from the graphite anode and the graphite in the electrolysis vessel which will yield a maximum current but also be such that the gas evolving from the cathode (hydrogen) does not bubble off but rather travels along the surface of the solution to come into contact with the anode.

The electrolysis was initiated by applying a current from a 25 volt power source across the electrodes. The conditions mentioned above were established at a maximum current of 0.1 amps at 20 volts. Initially the current is small due to the low conductivity of the electrolyte.

The system is then left essentially undisturbed for approximately 44 hours, after which the current was able to be adjusted to 0.15 amps at 24.5 volts. Approximately 24 hours later the current is still 0.15 amps at 24.5 volts and the reaction is observed as proceeding slowly. After a further 24 hour period the electrodes are moved closer together and the cathode also moved closer to the graphite in the electrolysis vessel. At this stage a current of 0.35 amps was measured at a voltage of 25 volts. The system is allowed to remain substantially undisturbed in this state for a further period of approximately 72 hours.

At this time, the system was observed to be drawing 1.4 amps at 25 volts. The increase in the current is apparently due to the increased conductivity of the electrolyte solution as the graphite goes into solution. At this time some gas is noted as evolving from the anode (possibly acetylene) and the solution appears to be getting darker.

Some 24 hours later the system is observed to be drawing 2.1 amps at 24.5 volts. The reaction vessel is hot to the touch and gas is still noted as evolving. After an additional 24 hour period the system is observed to be drawing 2.7 amps at 25 volts, and after a still further 24 hour period the system pulls 3.4 amps at 25 volts. The reaction system is hot and the solution now appears very dark in color, apparently due to the dissolution of the graphite in the reaction system, as the anode is now visibly eroded.

At this point some 2,200 ml of aqueous ammonia are slowly added to the electrolyte. The system cools down, but no ammonia odor is detected. After this addition of the aqueous ammonia, the current was set at 2 amps and 20 volts. The system was allowed to remain in this state substantially undisturbed for 4 days.

After this time the reaction vessel is observed and noted to be hot, a gas is still seen to be evolving from the anode and the system draws 1.7 amps at 21.5 volts. After an additional 24 hour period it is noted that the graphite anode has almost totally eroded away and out of the electrolyte. The reaction vessel has cooled. The anode is repositioned in the solution and the current is set at this time at 2 amps at 23 volts. After an additional 24 hour period the reaction vessel is again warm, and the current has increased to 2 amps and 17.5 volts. The next day, the anode has again eroded away. After readjusting the anode, the current is again set at 2 amps and 24.5 volts. After approximately 5 hours under these conditions the current is adjusted to 3.7 amps and 24.5 volts under which conditions a gas, presumably acetylene is seen to be evolving. The reaction is then terminated by withdrawal of the graphite anode and cathode.

During the course of the reaction it is noted that the electrolyte has grown darker in color and that a layer which appears as a cloud of bubbles develops in the upper portion of the reaction vessel. This appears to be an indication that the rate of dissolution of the graphite is too fast and some graphite-containing by-products are being formed.

After the termination of the reaction, the electrolyte is removed from the electrolysis vessel. The product is prepared in the form of an aqueous solution of an inorganic complex analyzed to contain graphite and NH and/or NH<sub>2</sub> linkages.

Quantitative analysis of the complex prepared according to the procedures outlined above was carried out using ASTM standard test method #56, which is the so-called Kjeldahl distillation technique, to determine the amount of nitrogen present in the product. This is basically a

reduction of bound nitrogen to ammonia, with the subsequent steam distillation of the ammonia into an excess acid solution. The excess acid is then back titrated with sodium hydroxide to determine the amount which reacted with the liberated ammonia. Prior to this test the complexes were air-dried, and then vacuum-dried at 50 DEG C. until there was no trace of ammonia vapor detected. The dry polymer was then heated with concentrated sulfuric acid and potassium dichromate through the fuming state, almost to dryness.

The amount of carbon present was quantitatively determined by combustion of the products, followed by an absorption of the CO<sub>2</sub> gases which result. These two tests showed that the product contained 73% nitrogen and 12% carbon, dry weight.

The graphite electrodes employed were taken from the electrolysis solution, rinsed thoroughly with deionized water, and then placed in a standard acid solution overnight. The next morning, the solution and electrodes were boiled. The resulting solution was then back titrated to determine the amount of acid which had been consumed by reaction with the ammonia which had been absorbed into the electrode. The fact that the electrode had absorbed ammonia gas was determined by a gas chromatograph analysis. After rinsing the electrode with deionized water, the electrode was placed in a combustion tube and heated to over 500 DEG C. for better than 45 minutes, with the resulting gases collected in a gas bulb. These gases were then injected into the gas chromatograph and the resulting retention times compared to those of known gases. The presence of ammonia was clearly indicated by this test.

## EXAMPLE 2

In this example, two basic experiments were carried out employing the procedures of Example 1, the first producing what will be called the "light complex". The second producing a "dark complex".

In each case, a reaction flask is filled with 2,000 ml. of commercial 26 DEG Be ammonia (38% by weight). Approximately 20 pieces broken from a graphite stick 1/2" in diameter are placed in the bottom of the flask. Two graphite rods, 5/8" in diameter are used as electrodes. One of them, approximately 20" long was brought into contact with the graphite particles at the bottom. This became the anode of the electrolytic system. The pressure was maintained at a slightly elevated level in order to minimize ammonia loss and maximize the formation of NH and NH<sub>2</sub> groups in the reaction.

(a) In the first experiment, approximately 2 amps at 12 volts was passed between the electrodes. The reaction started at room temperature and no external heat was added. No visible reaction took place, but on the second day it began to be apparent that the reaction vessel was heating. The temperature went up to approximately 50 DEG C. and stayed there for the duration of the reaction. This reaction continued for a period of 14 days. During this time, it was apparent that the graphite anode was deteriorating, with particles of graphite de-laminating and settling at the bottom of the reaction vessel.

At the end of 14 days there was no detectable odor of ammonia and most of the graphite particles had apparently dissolved. The solution was essentially water-white and transparent, with a slight greenish-yellow tint. It had specific weight of approximately 1.05 and was fairly viscous. When poured on a glass plate and dried, it formed a film which also was clear and which was water soluble. There was no detectable odor of ammonia.

The product of this first experiment was tested and found to contain 52% by weight nitrogen, the remainder was essentially carbon and hydrogen. An infrared analysis was made of the product after it had been vacuum dried at 50 DEG C. The IR spectrum produced is shown in the attached drawings as FIG. 2. This analysis confirmed the presence of hydrogen-nitrogen and carbon-nitrogen bonds. The graphite structure was confirmed by IR analysis through the determination that the para-carbon linkage was present in the product.

Upon heating the dry product, the odor of ammonia began to become apparent as the temperature approached about 150 DEG C., and the color of the dry product began slowly to change. From essentially water-white, it became straw-yellow at about 200 DEG, amber-brown at 300 DEG, grey at 400 DEG C. Above approximately 300 DEG C. the product was no longer soluble in water. When heating was continued, there appeared another transformation. At about 700 DEG C., the grey product began to turn lighter, and with further heat, at 900 DEG C. became a transparent, very hard material.

(b) In the second reaction experiment, all parameters were kept essentially constant except for the electrolytic current. A 25 volt power source was employed this time and the current was maintained between 4 and 6 amps. In this case, there was almost immediately produced a detectable evolution of gases at both the anode and cathode. The gas evolved at the cathode is hydrogen; that evolved at the anode is ammonia. No ammonia odor was detectable after six days. The solution rather than clear, was a dark amber-brown color and there was a substantial quantity of delaminated graphite particles in the bottom of the flask. The solution was strained, dried and analysed. It was determined that this product had 32% by weight nitrogen. This comparative test illustrates the importance of avoiding the use of too large a current in the laboratory scale process of the present invention. Where graphite is employed as the anode, the carbon-carbon linkages appear to be attacked, causing production of acetylene gas and the premature breakdown of the graphite structure. If a metal anode is utilized, too much current will result in the production of oxide.

In order to further study the inorganic complexes produced according to the practice of the present invention, samples of the solutions prepared according to Example 2a were dried and heated in vacuum at different temperatures and the products resulting subjected to infrared analysis. The infrared scans depicted in the attached drawings, FIGS. 1A-1E, were taken as the products were heated to different temperatures. FIG. 1A shows the IR spectrum of the product vacuum-dried at 50 DEG C. FIGS. 1B-E show the same product after it had been vacuum-dried and heated to 105 DEG C., 300 DEG C., 500 DEG C. and 700 DEG C., respectively.

With reference to FIGS. 1A-1E, it should be noted first that, after the product had been dried, no detectable odor of ammonia was present. However, the spectrum illustrated in FIG. 1A indicates that a large amount of nitrogen and hydrogen is present in the product. This is evidenced by the peaks at or near Wave Nos. 3300, 2800, 1400 and 600-900, which indicate the presence of the NH bond. The peaks at 2200, 1650 and in the area of 800 indicate the presence of the carbon-nitrogen bonding. While the peak at 3300 may also be indicative of the presence of water in the sample, it will be noted, by reference to FIGS. 1B and 1C, for example, that the peak remains on heating under vacuum.

As the product is heated to higher temperatures, large amounts of ammonia are detected as being given off. Inspection of FIGS. 1C and 1D will confirm that the NH groups are being lost. Finally, upon heating the product to temperatures above 500 DEG, all of the NH groups in the product are essentially broken down. This is confirmed by the fact that the product no longer gives off a detectable ammonia odor. Notice also the appearance of the carbon-nitrogen and nitrogen-nitrogen bonding as evidenced by the new peaks appearing in FIG. 1E. As will be also noted by reference to FIG. 1E, the para-carbon linkage is still present, indicating that the hexagonal graphite structure remains intact.

### EXAMPLE 3

This example illustrates the preparation of a complex which contains both a metal and graphite. In this case the metal employed is silicon. The electrolysis used both a graphite anode and a graphite cathode.

(a) The graphite cathode and graphite anode were immersed in an electrolyte comprising 1800 grams of 26 DEG Be aqueous ammonia ( $\text{NH}_4\text{OH}$ ). Added to the aqueous ammonia electrolyte were 390.6 grams of high purity silicon metal. Initially the anode and cathode were arranged such that the graphite anode was in physical contact with the silicon metal located in the bottom of the electrolysis vessel. Initially a voltage of 20 volts was applied across the graphite anode and cathode and a current of 0.1 amps was drawn.

Under these conditions, operating at room temperature, a moderate reaction of the silicon metal wherein the silicon dissolved in the electrolyte was observed.

After approximately one day's time, some slight increase in the reaction rate of the silicon metal was noted. At this point, a sample of the electrolyte was taken and determined to have a pH of 12.7, at a temperature of 25 DEG C.

After approximately 30 hours of reaction, the electrolyte became cloudy, probably indicating that too much silicon had entered the electrolyte. Shortly thereafter a bluish-white substance (probably silicon amide) began to build up on the graphite anode. It was observed after 48 hours of reaction that the graphite anode appeared covered with this build-up. At this point in the reaction, a current of 0.3 amps at a voltage of 24 volts was measured. The electrolyte was cloudy, and had a pH of 12.2. The temperature was 20 DEG C.

At approximately this stage in the reaction, the graphite anode and cathode were removed and the white deposit scraped off both electrodes. The electrodes were then reinserted in the electrolyte at a position above the silicon metal. A voltage of 25 volts was applied between the graphite anode and cathode, drawing a current of 0.4 amps.

After approximately 72 hours of reaction, it was noted that the electrolyte darkened, apparently due to the entry of excess graphite into the electrolyte solution. At this point the voltage across the anode and cathode was 25 volts and the system drew a current of 2 amps. The build-up on the anode was again removed and the graphite anode and cathode were moved closer together, the current being adjusted to 2.25 amps at 25 volts.

Approximately 72 hours later, the current was 2 amps at 14 volts.

24 hours later the electrolysis vessel was dismantled and the electrolyte removed and filtered. Analysis of the electrolyte showed it to be an aqueous solution containing a complex which included both silicon metal and graphitic carbon moieties tied together through  $\text{NH}$  and/or  $\text{NH}_2$  groups.

(b) This example was carried out similarly to the last example with silicon metal in the electrolyte medium. This example, however, was carried out to illustrate how the addition of a small amount of alkali to the electrolyte prior to current application greatly increases the conductivity of the electrolyte, thereby assisting in the dissolution of the metal.

Using the graphite anode and cathode as in part (a), above, the electrolyte was prepared from 2,275 grams of aqueous ammonia ( $\text{NH}_4\text{OH}$ ). Some 364 grams of high purity silicon metal was placed at the bottom of the reaction flask. 72.8 grams of a 10% aqueous solution of potassium hydroxide were then fed into the electrolyte over a 20 minute period prior to current application.

The current was applied with the graphite anode being in contact with the silicon metal. The initial current was 1 amp at 6.5 volts, illustrating the very conductive nature of the electrolyte, apparently due to the presence of the potassium hydroxide in the electrolyte solution. After approximately 16 hours of reaction, the amperage was measured at 2.1 amps at 9.5 volts. Shortly thereafter the current was adjusted to 1 amp at 5.5 volts. It remained fairly constant over the next 8 hours. There was no increase in reaction temperature.

Approximately 18 hours later the system was measured to be drawing 0.9 amps at 7 volts. At this time, it was observed that the silicon was not depositing on the cathode and the current was adjusted to 1 amp at 8 volts.

Approximately 24 hours later, a heavy build-up on the anode was noted (this is probably a sodium-silicon admide). The anode was removed from the system and it was observed that there was erosion of the anode underneath the build-up. The anode and cathode were then reinserted into the electrolyte, both in position above the silicon metal. The current was adjusted to 1 amp at 6 volts.

The system was allowed to continue in this state for approximately 72 hours. After the 72 hours period, the system was again observed and the build-up of material on the anode again removed. At this time the current was adjusted at 2 amps at 12 volts.

After a further 24 hour period the current was turned off and the cathode and anode removed from the electrolyte. The electrolysis vessel containing the electrolyte was then put on a hot-plate, and heated to approximately 120 DEG C.

After heating for approximately 1 hour the electrolysis chamber containing the electrolyte was taken off the hot-plate and it was observed that some graphite precipitated out of the solution. Several hours later the electrolyte was removed and observed. Analysis indicates that the electrolyte contains silicon metal, graphitic carbon moieties and  $\text{NH}$  and/or  $\text{NH}_2$  groups in the complex dissolved in the aqueous medium.

### EXAMPLE 4

(a) This example was carried out using molybdenum metal in an electrolysis system comprising both a graphite anode and a graphite cathode immersed in an electrolyte of aqueous ammonia. To 2,100 grams of the electrolyte, 3,293.2 grams of low purity molybdenum metal were added. At the start up of the electrolysis reaction, the cathode and anode were so arranged that the graphite anode was in physical contact with the molybdenum metal within the electrolyte. Initially the system drew 0.005 amps at 20 volts. Shortly after the initiation of the reaction, the pH of the electrolyte was measured by meter and found to be 12.6 at room temperature.

Approximately 20 hours after initiation of the reaction, the system was observed to have developed a cloudy, light brown color, and the electrolyte was warm. At this time, the pH was measured 11.5 at 35 DEG C., and the system drew 1.5 amps at 17 volts. The current was adjusted to 1 amp at 12 volts. After another four hours, the electrical leads were disconnected from the power supply and connected to an ammeter. A deflection of 0.2 amps suggested that the electrolyte solution retained dissolved electrons. The leads were again connected and the

current adjusted to 1 amp at 10 volts.

Three hours later, the current had increased to 2 amps at 14 volts, and approximately 18 hours after that, the system was drawing 3.5 amps at 14.5 volts. The electrolyte was also observed to be warm and the pH measured to be 10.7 at 37 DEG C. The solution appeared cloudy due to the presence of oxides. At this time the current was adjusted to 2 amps at 10 volts.

Approximately 6 hours later, it was noted that the reaction was getting lighter and clearer, suggesting the possibility that any metal oxides present were being reduced. The reaction was allowed to remain in this condition for an additional 16 hour period, after which it was definitely observed that the electrolyte was becoming clearer. Also metallic oxides which had collected on the sides of the electrolysis vessel were disappearing. Shortly thereafter the pH of the electrolyte was measured at 10.3 at 25 DEG C. The current was adjusted to 2 amps at 12 volts. Approximately 3 hours later the system was again observed and it was noted that a heavy brown layer (probably a mixture of molybdenum nitride molybdenum metal and carbide) had appeared on the cathode.

Some 20 hours later the system was observed to be drawing 1.5 amps at 11.5 volts. When the electrolyte was sampled a pH of 9.6 was measured at 36 DEG C. At this time the current was adjusted to 2 amps at 9 volts and the cathode and anode moved closer together. The reaction was allowed to remain undisturbed in this state for approximately 5 additional hours after which the electrodes were removed and the electrolyte solution containing the complex removed from the electrolysis vessel and filtered.

An analysis of the electrolyte solution showed it to be an aqueous solution of a complex containing graphitic carbon moieties, molybdenum metal and NH and/or NH<sub>2</sub> linkages. When this aqueous solution was dried and the dried complex analyzed, the molybdenum content of the complex was found to be 68.12% by weight. When this aqueous solution was subjected to electrolysis using a molybdenum or graphite anode and a copper cathode, molybdenum was deposited on the copper.

(b) The procedure of part (a) was repeated using molybdenum metal, but further including some 56 grams of a 10% potassium hydroxide solution added to the electrolyte solution, over a period of about 15 minutes prior to current application. In the initial setup, both the graphite cathode and graphite electrode were again arranged so as to be above the molybdenum metal, out of physical contact therewith. Initially a current of 2 amps at 13 volts was applied.

After only 2 hours of reaction, a very fast reaction of the molybdenum metal around the graphite anode was observed. This is in contrast to the slower initiation that was achieved in the absence of the alkali, potassium hydroxide.

After approximately 18 hours of reaction, a heavy deposit of molybdenum had developed on the cathode. A new cathode was introduced into the reaction system. During the time that the current was not applied while the cathode was being replaced, the metal reaction slowed drastically, picking up again when the current was reapplied. The current was reapplied at 2 amps and 9 volts.

24 hours later the system was observed to be drawing 2 amps at 9.5 volts, with the reaction proceeding at a fast rate. Approximately 43 hours after the initiation of the reaction another new cathode had to be inserted into the electrolyte and the current adjusted to 4 amps.

Approximately 24 hours later, approximately one liter of ammonium hydroxide solution was added to the electrolyte. No release of ammonia gas from the system was observed, even though the electrolyte was warm. At this point in the reaction the system drew 2 amps at 9 volts.

The system was allowed to remain undisturbed again for approximately 4 days, after which it was observed that the system drew 2.5 amps at 8.5 volts and the reaction system was warm. Approximately 6 hours later an additional 28 grams of 10% potassium hydroxide and a new cathode were inserted in the electrolysis system. At this point the system drew 2 amps at 7 volts. The cathode that was removed from the electrolysis system was analyzed and found to contain ammonia absorbed in the graphite cathode.

Approximately 18 hours later the system was still drawing 2 amps at 7 volts and 24 hours after that an additional 28 grams of 10% potassium hydroxide were added. Six hours later the system was analyzed and found to have a pH of 9.0. An additional 28 grams of potassium hydroxide were added at this time.

It was thereafter found that the anode had eroded away over the next 12-18 hour period. Analysis of the electrolyte showed a pH of 9.0. After the addition of 56 grams of potassium hydroxide the current was set at 2 amps and 7.5 volts. After operation under these conditions for approximately 11/2 hours the electrodes were removed and the electrolyte removed and filtered. The electrolysis vessel contained a considerable amount of carbon and molybdenum. The pH of the electrolyte was found to be 10.5.

When the electrolyte was analyzed, it was found to be an aqueous solution of a complex containing molybdenum, graphitic carbon and bridges comprising NH and/or NH<sub>2</sub> groups.

#### **EXAMPLE 5:**

In this example, copper metal was utilized, together with a graphite anode and a graphite cathode. The electrolysis vessel was set up with the graphite anode comprising a hollow graphite cylinder with a graphite rod extending out of a portion thereof. The cylinder was immersed in an electrolyte comprising 2800 grams of ammonium hydroxide (NH<sub>4</sub> OH) solution with some 128 grams of copper metal placed inside the hollow graphite cylinder. Thus, the graphite anode was in physical contact with the copper metal. The graphite cathode was positioned inside the cylinder above the copper particles.

Current was applied from a 25 volt power source and the position of the graphite cathode inside the graphite cylinder adjusted until a suitable transfer of bubbles from the cathode to the anode was observed. At this point, the current was measured at 0.2 amps. The temperature was 19 DEG C. and the solution immediately began to take on a pale blue hue. Some 5 1/2 hours later, the solution color had not changed noticeably but the current had increased to 0.4 amps. at 25 volts.

The system was left undisturbed under these conditions over night. The next morning the current had increased to 1.0 amps. and the temperature was 29 DEG C. There was some copper visible on the surface of the cathode.

After another 24 hours, the pH of the solution was measured at 12.6 and the current at 1.6. The temperature was 30 DEG C. and the reaction was obviously continuing. Three days later the solution was a deep blue, additional copper had plated out on the cathode and the power supply was no longer working since the copper had built up to a point where it shorted out the power supply. A new cathode was inserted and 25 volts of power reapplied at which point the current was measured at 2 amps. The temperature of the reaction was 22 DEG C. The reaction was

permitted to continue for several more days at which point the electrolysis process was terminated by withdrawal of the electrodes. The electrolyte solution was removed, filtered and analyzed to contain a solution of a complex containing graphitic carbon, copper and NH and/or NH<sub>2</sub> bridges.

#### **EXAMPLE 6:**

In this example, an electrolysis system was set up employing a graphite anode and a graphite cathode immersed in an electrolyte comprising 2100 grams of standard commercial ammonium hydroxide solution. 644 grams of powdered tungsten were placed in the bottom of the electrolysis vessel and the graphite anode and cathode positioned above the mass of the metal, but out of physical contact therewith. A full 25 volts was applied across the electrodes and the reaction immediately began to be visible. A slight ammonia odor was detected.

After continuing the reaction in this manner for 48 hours, a crystalline formation began to be visible on the inside of the reactor flask. After 2 more days, tungsten metal was visible as plating out on the cathode surface.

The cathode was removed and a new cathode inserted. The power was reapplied, drawing 4 amps for approximately 6 hours and thereafter reduced to 2 amps and held constant there. After 5 more days, again the cathode was covered with a thick plating of tungsten metal and had to be replaced. The reaction was continued with a new cathode. This time, the current was set at 2 amps and 6 volts and the reaction continued for 5 more days before being terminated by removal of the electrodes.

The solution was analyzed to contain a complex containing graphitic carbon, tungsten and NH and/or NH<sub>2</sub> groups. Each of the electrodes was degassed and tested by gas chromatograph to contain ammonia.

#### **EXAMPLES 7-15:**

Using the procedures outlined in Examples 3-6, above, specifically employing a graphite cathode, a graphite anode and an electrolyte containing ammonium hydroxide, complexes were prepared with the following metals being placed in the bottom of the reaction flask:

Aluminum--Al  
Titanium--Ti  
Tantalum--Ta  
Nickel--Ni  
Beryllium--Be  
Manganese--Mn  
Niobium--Nb  
Chromium--Cr  
Zinc--Zn

In each instance, a complex was prepared and analyzed to contain both graphitic carbon and the particular metal employed.

#### **EXAMPLE 16:**

In this example, the preparation of a complex according to the present invention is described utilizing aluminum as the anode with dissolved SO<sub>2</sub> gas as the electrolyte.

An electrolysis system is set up employing as anode an aluminum bar, 5/8" in diameter and approximately 2 feet long which has been coiled at one end to form a coil-spring shape approximately 10 centimeters high and 10 centimeters in diameter. A 5/8" graphite bar is utilized as the cathode. The electrodes were placed in a 1400 cc beaker, the cathode centered inside the coil of the anode and approximately one liter of deionized water added. A source of SO<sub>2</sub> gas was provided and set up so that the gas bubbled slowly through from the bottom of the beaker at a rate such that the solution was not overly agitated.

The SO<sub>2</sub> feed was initiated and the power turned on at a voltage of 20 volts. No current was drawn at first, but within a very short time a current of approximately 0.2 amps. was measured. The pH of the solution was measured at 2.0 and the temperature at 21 DEG C.

After about 1 hour, the reaction vessel was heated by wrapping it with heating tape. The reaction became turbid and the temperature increased to approximately 50 DEG C. within the next hour. At this point the current was measured at 1 amp and 20 volts.

The reaction was left undisturbed for a period of 5 hours, during which time the temperature remained between 55 DEG and 60 DEG C. and the pH in the neighborhood of 2.2. The voltage dropped and the amperage increased from 1 to a value of 2.7, at which time the voltage began to increase and the amperage to decrease. At a value of 2 amps and 20 volts, the rate of SO<sub>2</sub> was increased and the reaction continued. After another hour, the reaction was stopped and the solution analyzed. A complex product was produced containing aluminum and SH groups. The graphite cathode was removed and observed to have a strong hydrogen sulfide odor.

#### **EXAMPLE 17:**

(a) This example demonstrates the production of a complex containing graphitic carbon as the complex backbone with linkages of SH groups.

The electrolysis system employed comprised a 1400 cc beaker, filled with deionized water and containing a large, hollow graphite cylinder with a thin graphite bar inserted in the center of the cylinder. The graphite cylinder was connected as the cathode and the thin bar as the anode. SO<sub>2</sub> gas was bubbled through the beaker in the manner described in the preceding example. The reaction was heated by wrapping the beaker with heating tape.

The SO<sub>2</sub> feed was started with the reaction medium reached a temperature of 43 DEG C. Within 15 minutes, the power was turned on and the system measured as drawing 7 amps at 10 volts. With continued heating and increased temperature, the current was maintained essentially constant but the voltage kept dropping. After a period of some 6 hours, the reaction temperature had reached 75 DEG C. and the voltage had dropped to 4.5. There was no visible deposition on either electrode but the electrolyte had a brownish color. There was clearly some erosion of the graphite anode and dissolution of graphite into the electrolyte.

After a reaction period of approximately 6 hours, the SO<sub>2</sub> feed was stopped and the electrodes withdrawn. The solution was filtered and vacuum-dried at 50 DEG C. A clear film was formed which, when analyzed, was found to contain carbon, sulfur and hydrogen.

The graphite cathode used in the preparation of the carbon-sulfur complex described above was taken from the electrolysis solution and rinsed with deionized water. The electrode was then placed in a combustion tube, heated to approximately 500 DEG C. and the gases which evolved passed through a cadmium sulfate solution. A bright yellow cadmium sulfide precipitate was produced, evidencing the presence of a sulfide.

(b) In this case, the procedure of part (a) was duplicated with the exception that there was no heating of the electrolysis reaction vessel.

The SO<sub>2</sub> feed was started with the temperature at 18 DEG C. Within an hour the temperature had climbed to 42 DEG C. and the system was drawing 4.0 amps. at 7.5 volts. The amperage was maintained constant throughout the reaction procedure. Again, the voltage dropped, decreasing to 4.0 volts after about 7 hours. The temperature did not go more than 1 or 2 degrees above 40 DEG C.

Within a few hours, it was clear that a thin layer of sulfur was depositing on the cathode. This layer increased in thickness as the reaction continued, and may have had something to do with the fact that the reaction did not proceed as quickly as in the previous procedure. The reaction medium was a light brown color. After the electrodes were removed, the solution was filtered and analyzed to contain carbon (in graphite form) with sulfur and hydrogen bridges. An infrared spectrum of the complex produced according to this procedure after it had been vacuum-dried and heated to 105 DEG C. is shown in the drawings as FIG. 3.

#### **EXAMPLE 18:**

In this example, the production of a graphite based complex containing P-H linkages is demonstrated.

An electrolysis system is set up comprising a graphite anode and a graphite cathode in an electrolyte consisting of 1500 grams total weight of a 10% phosphoric acid (H<sub>3</sub> PO<sub>4</sub>) solution. The electrodes are positioned according to the procedure of the invention and a current applied across the electrodes. At first, a 10 volt power supply is utilized, initially generating a current of about 4.0 amps.

The reaction is left to proceed with very little visible evidence that anything is occurring other than the bubbles generated which pass from the cathode to the anode surface. However, after several days it is observed that the reaction rate has picked up, the temperature of the electrolyte has increased and some graphite has visibly eroded from the anode surface. As the reaction proceeds, some H<sub>2</sub> O evaporation occurs, requiring the addition of water to the reaction vessel.

The reaction is stopped and the solution analyzed. An inorganic complex has been produced containing graphitic carbon and P-H linkages.

The foregoing description has been directed to particular embodiments of this invention in accordance with the requirements of the Patent Statutes and for purposes of illustration and explanation. It will be apparent, however, to those skilled in this art that many modifications and changes in the procedures set forth will be possible without departing from the scope and spirit of the invention. It is applicant's intention that the following claims be interpreted to embrace all such modifications and variations.

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### **Method of preparing oxygen-containing bleach and product so produced** **US4116859** **[ [PDF](#) ]**

#### **SUMMARY OF THE INVENTION**

The present invention is directed to the production of heretofore unknown oxygen-containing bleach products, specifically bleach products obtained by reaction of hydrogen peroxide or ozone with a multi-metal, water-soluble, inorganic amide group (NH<sub>2</sub>)-containing monomeric complex.

Co-pending application Ser. No. 631,949, filed on Nov. 17, 1975, now **U.S. Pat. No. 4,029,747**, issued June 14, 1977 discloses a method for producing certain novel inorganic monomeric and polymeric complexes and such complexes so produced. In accordance with the present invention, novel oxygen-containing bleach products are prepared by reaction of the monomeric complex, with a source of hydroperoxy groups, specifically an inorganic source of hydroperoxy groups, such as hydrogen peroxide or ozone.

It is theorized from analytical data that each monomeric unit of the monomeric complexes employed to produce the oxygen-containing bleach products includes a plurality of reactive NH<sub>2</sub> groups. When such monomeric complexes are reacted with a source of hydroperoxy groups, such as hydrogen peroxide, the hydroperoxy groups replace and/or chemically combine with the NH<sub>2</sub> groups to produce a final product containing hydroperoxy groups. This final bleach product is formed in a solid, particulate form, which is water-soluble, although water solubility decreases with increasing hydroperoxy group content on the non-alkaline metal.

The reaction between the source of hydroperoxy groups and multi-metal, water-soluble, inorganic amide group-containing monomeric complexes is carried out by simply contacting the reactants while maintaining the reaction temperature below about 75 DEG F., to insure the stability of the final bleach product. Since the reaction is exothermic, cooling may be required. Preferably, cooling is provided to maintain the reaction temperature at room temperature or below.

In preparing the bleach products of the present invention, it is preferable to minimize the water content of the reaction system. To achieve this, more concentrated reactants are preferably utilized. A minimum amount of water will allow the formation of a solid particulate product by mixing the source of hydroperoxy groups and monomeric complex.

The oxygen-containing bleach of the present invention can be effectively utilized as an industrial or household bleach. The bleach releases active oxygen in an aqueous environment and this active oxygen is responsible for the active bleach properties. Since effective bleaching is achieved without chlorine or other halogen, a safer, nonpolluting, more efficient product is provided.

#### **BRIEF DESCRIPTION OF THE DRAWINGS**

**In the drawings:**

**FIG. 1 is an infra-red scan of a monomeric complex reactant containing sodium and silicon metals;**



**FIG. 2 is an infra-red scan of an oxygen-containing bleach product in accordance with the present invention, formed by reacting the silicon-sodium monomeric complex of FIG. 1 with hydrogen peroxide;**

**FIG. 3 is an infra-red scan of a monomeric complex reactant containing sodium and molybdenum metals;**

**FIG. 4 is an infra-red scan of an oxygen-containing bleach product in accordance with the present invention formed by reacting the molybdenum-sodium monomeric complex of FIG. 3 with hydrogen peroxide;**

**FIG. 5 is an infra-red scan of a monomeric complex reactant containing aluminum and sodium metals;**

**FIG. 6 is an infra-red scan of an oxygen-containing bleach product in accordance with the present invention formed by reaction of the aluminum-sodium monomeric complex of FIG. 5 with hydrogen peroxide;**

**FIG. 7 is an infra-red scan of a monomeric complex reactant containing zirconium and sodium metals;**

**FIG. 8 is an infra-red scan of an oxygen-containing bleach in accordance with the present invention formed by reacting the zirconium-sodium monomeric complex of FIG. 7 with hydrogen peroxide;**

**FIG. 9 is an infra-red scan of a monomeric complex reactant containing titanium and sodium metals; and**

**FIG. 10 is an infra-red scan of an oxygen-containing bleach in accordance with the present invention formed by reacting the titanium-sodium monomeric complex of FIG. 9 with hydrogen peroxide.**

## **DESCRIPTION OF THE PREFERRED EMBODIMENTS**

The oxygen-containing bleach products in accordance with the present invention are prepared by contacting and reacting a source of hydroperoxy groups, specifically an inorganic source of hydroperoxy groups, such as hydrogen peroxide or ozone, with a water-soluble, multi-metal, inorganic, amide group-containing monomeric complex. The oxygen-containing bleach is prepared as a solid particulate bleach product which releases active oxygen in an aqueous environment. This provides for the effective bleaching characteristics of this product. Generally, the bleach product which is prepared is water-soluble, although water solubility decreases with increasing hydroperoxy group content specifically as the hydroperoxy groups are attached to the non-alkaline metal. It is theorized in accordance with the present invention that the NH<sub>2</sub> groups of the inorganic monomeric complex are replaced by hydroperoxy groups (HO<sub>2</sub>) through the reaction of the present invention. The released NH<sub>2</sub> groups pick up hydrogen ions and can be observed as ammonia evolved from the reaction. Since the monomeric complexes utilized as reactants contain a plurality of NH<sub>2</sub> groups, this replacement can be carried out so that all or a portion of NH<sub>2</sub> groups are replaced with hydroperoxy groups.

To prepare the oxygen-containing bleach products of the present invention, the following reactants are required:

- A. water-soluble, multi-metal, inorganic, amide group-containing monomeric complex; and
- B. a source of hydroperoxy groups, specifically hydrogen peroxide, or ozone.

These reactants are individually discussed below.

### **The Monomeric Complex**

A full description of the preparation of the monomeric complex reactants utilized in the process of the present invention can be found in applicant's co-pending application Ser. No. 631,949, filed on Nov. 17, 1975, the disclosure of which is herein incorporated by reference.

In general, the monomeric complex reactants are prepared in accordance with a reaction sequence which involves: introducing an excess of a non-alkaline metal or metals into a reaction vessel, preferably already containing aqueous ammonia, and thereafter incrementally adding an alkali metal hydroxide in such manner as to create localized areas of high alkali concentration, the pH approaching a value of 14 in such localized areas. The hydroxide in these areas of high concentration, in combination with the reactive NH<sub>2</sub> groups provided by the aqueous ammonia, causes dissolution of the non-alkaline metal, the formation of non-alkaline metal ions and the production of a monomeric complex reactant as employed in accordance with the present invention. Accordingly, the reactants necessary to produce such monomeric complex include at least one non-alkaline metal, a source of NH<sub>2</sub> groups, particularly aqueous ammonia, and an alkali metal hydroxide. These reactants will be individually discussed below.

The non-alkaline reactant is broadly selected from those non-alkaline metals of Groups I-VIII of the Periodic Table. Representative, non-limiting examples of applicable non-alkaline metals include:

- Group I-B: copper, silver, gold
- Group II-A: beryllium, magnesium
- Group II-B: zinc, cadmium
- Group III-A: aluminum, gallium, indium
- Group IV-A: silicon, tin, lead
- Group IV-B: titanium, zirconium, hafnium
- Group V-A: antimony, bismuth
- Group V-B: vanadium, niobium, tantalum
- Group VI-A: chromium, molybdenum, tungsten
- Group VI-B: manganese
- Group VIII: iron, cobalt, nickel, palladium, rhodium

It should be recognized that while silicon, as a member of Group IV-A, is not generally defined as a metallic element, silicon acts as a non-alkaline metal in preparing the monomeric complex reactant. Accordingly, the expression "non-alkaline metal of Groups I-VIII of the Periodic Table" is meant to embrace any and all of the above and equivalent metals, including silicon. As will be further recognized, the term "non-alkaline metal of Groups I-VIII of the Periodic Table" does not embrace the alkali metals of Group I-A. The alkaline earth metals, calcium, strontium, and barium of Group II-A, are similarly not within the scope of the term. On the other hand, beryllium and magnesium of Group II-A can be applicably employed in the preparation of the monomeric complex reactant and these metals also fall within the scope of the expression "non-alkaline metal of Groups I-VIII of the Periodic Table."

While any of the above non-alkaline metals can be utilized to form the monomeric complex reactant which is contacted and reacted with the source of hydroperoxy groups to form the oxygen-containing bleach in accordance with the present invention, the non-alkaline metal utilized is preferably one which contributes additional valuable properties to the bleach product, particularly those metals which have heretofore been used in preparation of industrial bleaches, soaps, etc. Preferred examples of such metals include magnesium, aluminum, silicon, zinc, titanium, zirconium, molybdenum, and tungsten. While these metals are preferred and while the representative exemplification will be directed primarily thereto, it should be recognized from applicant's co-pending application Ser. No. 631,949 that the monomeric complex reactant can be formed from any of the non-alkaline metals of Groups I-VIII of the Periodic Table, and accordingly, the oxygen-containing bleach product of the present invention can be effectively prepared from any such monomeric complexes.

An alkali metal hydroxide is utilized as the second reactant to produce the monomeric complex reactant. This reactant can comprise any hydroxide of a metal of Group I-A of the Periodic Table, principally, sodium hydroxide, potassium hydroxide or lithium hydroxide.

The alkali metal hydroxide, specifically sodium hydroxide or potassium hydroxide, dissolves in the aqueous medium in the monomeric complex production reaction. The alkali metal hydroxide can be employed either in solid form or in the form of a prepared aqueous solution of the alkali metal hydroxide. In solid form, the alkali metal hydroxide can be conveniently in the form of pellets, powder or flakes.

Analytical studies have shown that the inorganic monomeric complexes useful in accordance with the present invention contain NH<sub>2</sub> groups. Accordingly, the third essential reactant in the method of producing the monomeric complexes comprises any substance which, under the conditions of the reaction, acts as a source of reactive NH<sub>2</sub> groups.

In the preferred reaction for preparation of the monomeric complex reactant, the source of NH<sub>2</sub> groups comprises aqueous ammonia. NH<sub>2</sub> groups are liberated when this aqueous ammonia is brought into reactive contact with the alkali metal hydroxide and non-alkaline metal. As an alternative, ammonia gas may be used, introducing the gas into the reaction system by any suitable conventional techniques, such as bubbling. The use of ammonia gas in this manner also permits a supplementing of the supply of the NH<sub>2</sub> source at any stage during the reaction.

Another applicable alternative NH<sub>2</sub> source may be formed directly in the reaction medium by utilizing as a reactant an NO<sub>x</sub> gas, i.e., NO and/or NO<sub>2</sub>, or nitric acid. The use of NO<sub>x</sub> gas or nitric acid as a source of reactive NH<sub>2</sub> groups appears applicable due to the dissolution of the non-alkaline metal and concurrent hydrogen ion production. These hydrogen ions react with the NO<sub>x</sub> groups in the reaction medium, creating in situ the reactive NH<sub>2</sub> groups. These reactive NH<sub>2</sub> groups are then available for metal amide formation and formation of the inorganic monomeric complex reactant. When the non-alkaline metal is not easily dissolved in the reaction medium upon alkali metal hydroxide addition, it may be necessary to assist the dissolution so as to develop the hydrogen ions necessary for reactive NH<sub>2</sub> group production. This can be achieved, for example, by supplementing the NO<sub>x</sub> or nitric acid with ammonia gas at the onset of the reaction.

As disclosed in applicant's co-pending application Ser. No. 631,949, filed Nov. 17, 1975, the reaction between the non-alkaline metal, alkali metal hydroxide and source on NH<sub>2</sub> groups proceeds through a first endothermic phase wherein a monomeric complex is produced and thereafter, through an exothermic phase wherein the monomeric complex polymerizes into a polymeric complex form. It is presumed from analytical data which has been obtained that, during this polymerization stage, a substantial portion of the NH<sub>2</sub> groups of the inorganic monomeric complex are broken up into NH and hydride groups which are present in the polymerized complex. It has been determined in accordance with the present invention, however, that an effective bleach product cannot be obtained through the reaction of the source of hydroperoxy groups and inorganic polymeric complex, presumably due to the lack of a substantial number of reactive NH<sub>2</sub> groups in the polymerized complex and the difficulty of the hydroperoxy groups to replace or chemically combine with the NH or hydride groups. Accordingly, the process of the present invention for the preparation of the oxygen-containing bleach product requires the use of the monomeric complex.

As described in applicant's co-pending application Ser. No. 631,949, filed Nov. 17, 1975, after an endothermic phase of reaction in which the monomeric complex is formed, a natural exothermic phase is entered. In order to produce the monomeric complexes useful as reactants in the process of the present invention, however, such exothermic phase must not be allowed to go to completion since, as indicated previously, the polymeric complexes cannot be effectively used in producing the oxygen-containing bleach products. Generally, however, the reaction for preparing the monomeric complexes is allowed to go slightly into the exothermic phase, for example, up to a temperature of about 140 DEG F., in order to rid the monomeric complex formed of any free ammonia. At this temperature, there may be some slight polymerization of the monomeric complex and some slight splitting of the NH<sub>2</sub> groups into NH and hydride groups; however, this slight degree of polymerization will not interfere with the basic function of the monomeric complex reactant in the preparation of the oxygen-containing bleach of the present invention. Furthermore, where substantial polymerization does occur, the reaction can be driven back to its monomeric stage by the simple addition of ammonia. Accordingly, the expression "water-soluble multi-metal inorganic amide group-containing monomeric complex" as used throughout is meant to embrace the above-discussed complex, possibly including a minor amount of polymerized complex.

Throughout the foregoing and following description, reference has been made to the production of an inorganic monomeric "complex". The term "complex" is used because the structural configurations of these products have not been precisely determined. However, extensive analysis has been conducted which has established the monomeric nature of the products and the presence of certain functional groups. In view of the difficulty in precisely determining the structural configuration of products obtained through complex chemical reactions, the expression "complex" is deemed aptly descriptive of this reactant used in the method of the present invention.

### **The Source of Hydroperoxy Groups**

The second essential component in preparing the oxygen-containing bleach of the present invention is a source of hydroperoxy groups. In this regard, the present invention can employ any source of hydroperoxy groups effective to introduce hydroperoxy groups into the backbone of the monomeric complex. Such source of hydroperoxy groups is preferably hydrogen peroxide or ozone, most preferably an aqueous solution of hydrogen peroxide. As will be appreciated from the discussion hereinafter involving the reaction parameters, when employing an aqueous solution of hydrogen peroxide, the concentration of the hydrogen peroxide should be as high as possible in order to minimize the amount of water present in the reaction system. In this way, it is possible to effectively produce the oxygen-containing bleach product as a substantially dry powder. Of course, in lieu of an aqueous solution of hydrogen peroxide, 100% hydrogen peroxide can be employed in the process of the present invention; however, highly concentrated hydrogen peroxide must be handled very carefully.

When utilizing ozone, for example, as the source of hydroperoxy groups, the ozone can be bubbled into the reaction system containing the aqueous solution of the inorganic monomeric complex. Reaction between an aqueous solution of hydrogen peroxide and the aqueous solution of the inorganic monomeric complex can be effectively achieved simply by mixing these components in a suitable vessel.

### **Reaction Parameters**

As indicated above, when employing the preferred form of the source of hydroperoxy groups, it is merely necessary to contact, through mixing, the aqueous solution of the inorganic monomeric complex and hydrogen peroxide or aqueous solution thereof while maintaining the reaction temperature below about 75 DEG F. This provides sufficient contact between the hydrogen peroxide and monomeric complex such that the hydroperoxy groups of the hydrogen peroxide replace and/or chemically combine with the amide groups of the inorganic monomeric complex to produce a final bleach product containing hydroperoxy groups. Such bleach product containing hydroperoxy groups crystallizes out of the reaction system during the mixing of the reactants.

The oxygen-containing bleach product which is formed as a crystalline solid redissolves in the aqueous medium from which it is formed if the product is not immediately removed from the reaction medium. With increasing hydroperoxy group content i.e., as the hydroperoxy groups replace an increasing member of the NH<sub>2</sub> groups attached to the non-alkaline metal of the multi-metal complex, this bleach product has decreasing solubility. However, due to the solubility in the aqueous medium it is preferred in accordance with the present invention to limit the amount of water present in the reaction system, and by so doing, directly form the oxygen-containing bleach product as a substantially dry solid product, specifically a substantially dry powder. For this reason, concentrated hydrogen peroxide solutions are preferred over dilute hydrogen peroxide solutions. In addition, the water content of the inorganic monomeric complex solution can be reduced prior to reaction by evaporating some of the water present. Preferably, after preparation of the inorganic monomeric complex and removal of excess unreacted non-alkaline metal, the aqueous solution of the inorganic monomeric complex is heated to evaporate about one-half of its water content. This concentrated inorganic monomer solution is then very effective for producing the oxygen-containing bleach product in the form of a substantially dry powder.

The selection of an appropriate water content for a particular system can be easily carried out by one skilled in the art through routine experimentation. It is merely important that the water not be present in such an excess amount that after production of the oxygen-containing bleach product, the solid redissolves in the aqueous medium. The selection of a water content to achieve a dry product or a substantially dry product through the mixing of the aqueous solution of the inorganic monomeric complex and hydrogen peroxide can be very easily done. Precise selection of the water content is not necessary since if a wet product is formed, drying can be achieved by vacuum evaporation or simply by gently heating the wet solid. Care should be taken to avoid temperature in excess of about 160 DEG F. since temperatures in excess of this value tend to degrade the oxygen-containing bleach.

As indicated previously, it is theorized that the inorganic monomeric complex utilized as a reactant in accordance with the present invention contains a plurality of NH<sub>2</sub> groups, this number generally corresponding to the total valence of the alkali metal and non-alkaline metal. The reaction of such inorganic monomeric complex with the source of hydroperoxy groups, specifically hydrogen peroxide, can be effectively carried out in order to replace and/or chemically combine with one or more of such NH<sub>2</sub> groups in order to produce the final oxygen-containing bleach product. The degree of replacement and/or chemical combination will depend upon the molar ratio of the inorganic monomeric complex to the hydrogen peroxide. This molar ratio can suitably vary from 1:1 or below to 5:1 or more. Again, the particular molar ratio of the hydrogen peroxide to the inorganic monomeric complex can be easily selected by one skilled in the art depending upon the final hydroperoxy content desired in the oxygen-containing bleach product.

The preparation of the oxygen-containing bleach product can be achieved by merely contacting, through mixing, the above-described reactants in a suitable vessel under ambient temperature and pressure conditions. Preferably, the contacting is carried out at a temperature of room temperature or below. Since the reaction between the inorganic monomeric complex and hydrogen peroxide or other equivalent source of hydroperoxy groups is exothermic, it may be necessary to cool the reaction vessel, through any suitable means. The reaction medium should not be allowed to go higher than 100 DEG-105 DEG F. since at temperatures above this range it becomes extremely difficult to control the reaction as the exothermic reaction becomes great and a breakdown of the oxygen-containing bleach product is substantial. The use of an ice bath to effectively maintain the temperature below this value, preferably at room temperature or below, can be easily carried out.

The present invention will now be described by reference to the following specific examples. Such examples are presented for purposes of illustration only and the present invention cannot under any circumstances be deemed limited thereto. In these following examples, unless otherwise stated, all parts and percentages are by weight.

#### EXAMPLE 1

A silicon-sodium monomeric complex is prepared following the reaction sequence and parameters described in co-pending application Ser. No. 631,949, filed on Nov. 17, 1975. The reactants include 616 g of silicon metal, 1,925 g of aqueous ammonia (28 DEG Be) and 440 g of sodium hydroxide, the latter being metered slowly into the reaction system in order to develop an endothermic phase of reaction. After completion of the reaction, 320 g of silicon are removed from the reaction system. Accordingly, some 296 g of silicon enter into the inorganic monomeric complex. The complex is diluted with 500 ml of deionized water. The aqueous solution of the monomeric complex has a specific gravity of 1.461.

To prepare the oxygen-containing bleach product of the present invention, some 50 ml of this aqueous solution of the silicon-sodium monomeric complex are reacted with 8 ml of hydrogen peroxide (50%) by simply mixing the reactants under ambient temperature and pressure. This mixing results in the formation of a white, substantially dry powder. During the mixing of the hydrogen peroxide with the silicon-sodium monomeric complex, the release of ammonia from the monomeric complex is detected.

FIG. 1 represents an infra-red scan of a silicon-sodium monomeric complex produced as described above. FIG. 2 represents an infra-red scan of the silicon-sodium bleach prepared as described above by reaction of the silicon-sodium monomeric complex with hydrogen peroxide. By comparing FIGS. 1 and 2, an increased hydroperoxy group content in the bleach product can be observed. In this regard, the hydroperoxy group comes out at approximately 875 cm<sup>-1</sup> and at about 2,500 cm<sup>-1</sup>. Due to the water present in the system, some of the amide group which can be observed at 1,600 cm<sup>-1</sup> are covered with hydroxy. In addition, the spectrum for silicon bonding partially covers some of the spectrum where the amide and hydroperoxy groups would be observed. Oxygen bonding also appears at 875 cm<sup>-1</sup>.

#### EXAMPLE 2

Following the procedure of Example 1, a bleach-containing molybdenum metal is prepared.

First, a molybdenum-sodium monomeric complex is prepared by reacting molybdenum metal (4,538.8 g) in aqueous ammonia (1,930.6 g-28 DEG Be) with the controlled incremental addition of 630.4 g of sodium hydroxide. After the addition of the sodium hydroxide the reaction system is heated slightly to 85 DEG-100 DEG C., utilizing a dry ice condenser to prevent ammonia escape. Due to the slow reaction of molybdenum, this slight heating is necessary in order to initiate the dissolution of the molybdenum in the reaction system. The reaction is run

in this manner for several days after which a low heat of approximately 30 DEG C. is applied. The reaction thereafter continues for approximately one week without any heat in order to prepare the final molybdenum-sodium monomeric complex. The final product has a specific gravity of 1.332.

To prepare the molybdenum-containing bleach, 50 ml of the above monomeric complex are reacted with 5 ml of hydrogen peroxide. This reaction is extremely exothermic. The reaction occurs merely by mixing the monomeric complex and aqueous hydrogen peroxide.

FIG. 3 is an infra-red scan of a molybdenum-sodium inorganic monomeric complex prepared as above. FIG. 4 is an infra-red scan of a molybdenum-containing bleach prepared by reacting the molybdenum-sodium inorganic monomeric complex with hydrogen peroxide. Noting FIG. 4, the peaks at approximately 875 cm@-1 and 2,500 cm@-1 identify the presence of hydroperoxy groups within the bleach product. Also, a comparison of FIG. 3 and 4 shows some decrease in the amide group content in the bleach when compared with the starting monomeric complex.

### EXAMPLE 3

In this example an aluminum-containing bleach is prepared by following the procedure, substantially as described in connection with Example 1.

To prepare the aluminum-containing monomeric complex, 713 g of aluminum are introduced into 1,820 g of aqueous ammonia (28 DEG Be) and thereafter, 520 g of sodium hydroxide pellets are incrementally introduced into the reaction system over a period of about two hours. The reaction system develops a natural endothermic phase and thereafter begins to enter an exothermic phase. The reaction is terminated by withdrawing unreacted aluminum when the temperature of the reaction system reaches 150 DEG F. Some 647 g of aluminum are removed from the reaction system, indicating that 66 g of aluminum are introduced into the monomeric complex. The monomeric complex obtained has a specific gravity of 1.314.

To prepare the aluminum-containing bleach, 50 ml of the above-prepared monomeric complex are reacted with 8.33 ml hydrogen peroxide, the components being mixed thoroughly until the bleach crystallizes as a substantially dry powder. The formation of the bleach by mixing the aluminum-containing monomeric complex and hydrogen peroxide is exothermic.

FIG. 5 is an infra-red scan of an aluminum-sodium monomeric complex prepared in accordance with the procedure above; and FIG. 6 is an infra-red scan of an aluminum-containing bleach obtained by mixing such a monomeric complex with hydrogen peroxide. Reference to FIG. 6 will again reveal the presence of the hydroperoxy groups in the bleach product and a comparison of FIGS. 5 and 6 illustrates the apparent reduction in the amide group content of the monomeric complex when converted to bleach.

### EXAMPLE 4

Following the procedure of Example 1, the zirconium-containing oxygen-containing bleach is prepared from a zirconium-sodium inorganic monomeric complex. This complex is prepared through a reaction which involves introducing 2,241.6 g of zirconium metal into 1,078 g of aqueous ammonia (28 DEG Be) and thereafter incrementally introducing 246.4 g of sodium hydroxide pellets. The feed of sodium hydroxide is accomplished by approximately one hour and forty minutes, and thereafter, the reactants are maintained undisturbed, while entering an endothermic phase of reaction. The reaction is terminated by removing unreacted zirconium metal, the monomeric complex having a specific gravity of 1.227. To prepare the bleach product, 50 ml of this zirconium-containing monomeric complex are mixed with 10 ml of hydrogen peroxide (50%), the mixing being carried out thoroughly until crystallization of the bleach occurs. This mixing results in a slightly exothermic reaction. The hydroperoxy group content of the bleach is found to be 16.66%.

FIG. 7 represents an infra-red scan of a zirconium-containing monomeric complex produced as described above; FIG. 8 represents the infra-red scan of a bleach obtained by reacting such a zirconium-sodium monomeric complex with hydrogen peroxide. Again, it can be seen that the bleach has an increased hydroperoxy group content, as exemplified by the small peak at about 2,500 cm@-1 in FIG. 8 and the increased size of the peak at about 875 cm@-1.

### EXAMPLE 5

Following the procedure of Example 1, a titanium-sodium monomeric complex is prepared and is subsequently utilized to prepare a titanium-containing bleach. The titanium monomer is prepared by reacting 1,997.52 g of titanium metal in 2,080.75 g of aqueous ammonia (28 DEG Be) with 951.2 g of sodium hydroxide pellets, introduced incrementally. The sodium hydroxide is fed over a time period of approximately 40 minutes after which the reactants are allowed to remain undisturbed in the reaction vessel. The reaction is terminated by withdrawing unreacted titanium from the reaction system, yielding a monomeric complex having a specific gravity of 1.432.

This monomeric complex is reacted with hydrogen peroxide by mixing 50 ml of a monomeric complex with 5 ml of a 50% hydrogen peroxide solution. The components are mixed thoroughly until the bleach product crystallizes as a substantially dry powder. The reaction between the monomeric complex and hydrogen peroxide is mildly exothermic. The hydroperoxy group content of the final bleach is found to be approximately 9%.

FIG. 9 represents an infra-red scan for the titanium-sodium monomeric complex prepared as above. FIG. 10 represents an infra-red scan for a titanium-containing bleach obtained by reacting such titanium-sodium inorganic monomeric complex with hydrogen peroxide. It can again be seen by comparing FIGS. 9 and 10 that there is an increase in the hydroperoxy group content in the bleach, as evidenced by the increased peak at about 875 cm@-1 and the peak at approximately 2,500 cm@-1.

### EXAMPLE 6

Following a procedure similar to that of Example 1, a tungsten-sodium monomeric complex is produced by reacting 603.5 g of tungsten in 245 g of aqueous ammonia (28 DEG Be) through the incremental addition of 40 g of sodium hydroxide pellets. The sodium hydroxide pellets are introduced over a time period of approximately one and one-half hours. After introduction of the sodium hydroxide, the reaction is pushed by utilizing a 1.5 amp current in order to accelerate the dissolution of the tungsten metal.

After allowing the reaction to remain in this manner for a period of time, the reaction is terminated by withdrawal of unreacted tungsten. The monomeric complex has a density of 1.230. A bleach product containing tungsten is prepared by reacting 20 ml of this monomeric complex with 4.5 ml of hydrogen peroxide (50%).

## EXAMPLE 7

Utilizing a similar procedure as that described above, a magnesium-containing bleach is prepared from a magnesium-sodium monomeric complex. The complex is prepared by reacting 432 g of magnesium metal in 630 g of aqueous ammonia (28 DEG Be) through the incremental addition of 240 g of sodium hydroxide pellets. The feed of sodium hydroxide continues over a period of approximately one hour and fifty minutes. The reactants are allowed to remain undisturbed for a period of time. The reaction is terminated with withdrawal of unreacted magnesium, leaving a monomeric complex having a specific gravity of 1.264.

To prepare the magnesium-containing bleach, 50 ml of this magnesium-containing monomeric complex are reacted with 8 ml of hydrogen peroxide (50%) by thoroughly mixing the reactants until the magnesium-containing bleach crystallizes. The reaction was exothermic. The hydroperoxy group content of the bleach is found to be approximately 13.8%.

## EXAMPLE 8

To produce a zinc-sodium-containing monomeric complex, 2,080 g of powdered zinc metal are reacted with 840 g of aqueous ammonia (28 DEG Be) through the incremental addition of 320 g of sodium hydroxide pellets. The sodium hydroxide pellets are fed into the reaction system over a period of approximately two hours and ten minutes. The reactants are allowed to remain undisturbed for a period of time. The reaction is terminated by withdrawing the unreacted zinc metal, the monomeric complex having a specific gravity of 1.277.

To prepare the zinc-containing bleach, 50 ml of the above monomeric complex are reacted with 4.5 ml of hydrogen peroxide (50%) by thoroughly mixing the reactants until the bleach crystallizes as a substantially dry solid. This reaction is exothermic. An analysis of the bleach indicates that the hydroperoxy group content is approximately 8.3%.

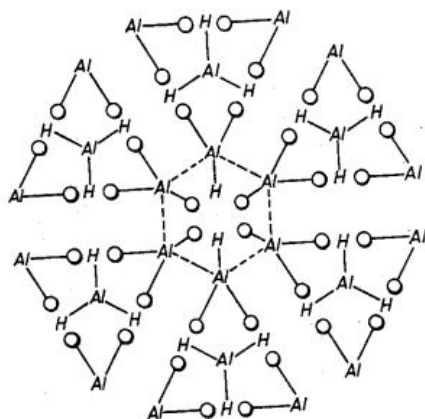
The foregoing examples have been presented in order to illustrate various embodiments of the present invention. It should be understood, however, that the method and product of the present invention can not be limited thereto and modifications within the foregoing examples and description can be made while still falling within the spirit of the present invention. For example, it is possible to employ a mixture of inorganic monomeric complexes to achieve a bleach product having desired unique characteristics. Likewise, while the previous discussion has been centered around the preparation of inorganic monomeric complexes by a procedure set forth in applicant's co-pending application Ser. No. 631,949, filed on Nov. 17, 1975, the inorganic monomeric complex need not be produced by such process, as long as the metallic backbone is present and the reactive NH<sub>2</sub> groups are present for replacement by or chemical combination with the source of hydroperoxy groups. Accordingly, the present invention can not under any circumstances be limited to the exemplification previously made, but rather, must be construed to embrace any and all equivalents thereof.

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## HYDROPEROXY GROUP-CONTAINING ALUMINUM COMPOUND AND METHOD OF MAKING THE SAME

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## BACKGROUND OF THE INVENTION

### 1. Field of the Invention

The present invention is directed to a method of producing hydroperoxy-group-containing aluminum compounds and such compounds so produced. More specifically, the present invention is directed to a method of producing hydroperoxy-group-containing aluminum compounds in a sheet or plate-like form, the compounds being transparent, appearing polymeric in nature, and having the physical appearance of mica.

### 2. Description of Prior Art

The patent literature describes various types of transparent alumina and processes for producing such transparent alumina. For example, U.S. Pat. No. 3,026,210 to R. L. Coble describes a transparent alumina in which the alumina comprises high density polycrystalline alumina bodies having in-line transmission of not less than 10%/ml thickness of radiant energy of wave length in the range from 0.30 micron to about 6.6 microns. Such transparent alumina bodies are prepared by compacting a mixture of finely divided alumina with minor additions of magnesia and firing the compact for predetermined periods of time at a temperature not lower than about 1,700 DEG C., either in a vacuum or in a hydrogen environment.

U.S. Pat. No. 3,026,177 to P. D. S. St. Pierre, et al. also involves the preparation of transparent polycrystalline alumina. In accordance with this patent, the product is produced by compacting a substantially pure, finely divided alumina and subjecting the compact to a first firing in a hydrogen atmosphere at a temperature of from about 1,650 DEG C. to 1,750 DEG C. for a period of from 50 to 300 minutes and thereafter,

subjecting the alumina body to a second firing in a hydrogen atmosphere at a temperature of from about 1800 DEG C. to 2000 DEG C. for a period of not less than 15 minutes. This second firing is said to improve the transparency of the polycrystalline alumina.

U.S. Pat. No. Re. 27,083 in the name of Emile A. Klinger, et al., describes a dense polycrystalline body of alumina which has adequate transparency and high mechanical strength. Such product is described as having an average size of grains of less than 10 microns and a specific gravity greater than 3.96. The polycrystalline body of fine grained alumina is obtained by compacting a powder of aluminum oxide particles having a particle size not substantially greater than 3 microns and sintering the compacted body in the substantial absence of gases having a molecular weight substantially greater than that of hydrogen for a period of 3 to 30 minutes at a temperature of 1700 DEG-1950 DEG C.

Also, U.S. Pat. No. 3,718,494 to Howard H. Jacobson describes sheet-like alpha alumina having an average thickness of 10-250 m.mu. and major faces averaging 15-1000 m.mu.@2. Such sheet-like alpha alumina is prepared by the formation of mullite in the presence of a reactive flouride salt such as cryolite.

With the exception of the last noted patent to Jacobson, each of the above-described processes involves compacting a high temperature sinter to produce transparent bodies of alumina. None illustrates the direct production of an aluminum-containing product, such as alumina, in a transparent form directly in its formative reaction. Furthermore, in none of these products is the alumina in a sheet-like or plate-like form, having a physical structure similar to mica and appearing polymeric in nature.

The preparation of an aluminum product having a substantial amount of hydroperoxy groups is a desirable goal in that the hydroperoxy groups create unique physical and chemical characteristics in the aluminum compound. For example, unique reactivity and catalytic properties are associated with the presence of hydroperoxy groups. The use of hydrogen peroxide to produce an alumina product, specifically as a hydro-forming catalyst, is illustrated in U.S. Pat. No. 2,852,473 to Welling.

The Welling patent relates to a process which involves a first amalgamation of aluminum with a solution of a mercuric salt, such as mercuric chloride, and subsequent contacting of the amalgamated aluminum with a dilute aqueous solution of hydrogen peroxide. With respect to the desired hardness, porosity and surface area of the catalyst, a desirable product is said to be obtained when the temperature of the reaction medium is maintained between about 70 DEG C. and the boiling point of the aqueous hydrogen peroxide. Generally, a granulated aluminum is utilized to prepare this alumina hydro-forming catalyst.

A process such as described in the Welling patent cannot produce an alumina product in which the product is transparent, in sheet or plate form and with the physical appearance of mica. Moreover, a product such as that produced by the process of Welling cannot be polymeric in nature. Still further, the hydroperoxy group content of such product is minimal due to the presence of a substantial amount of water in the reaction system and the employment of a high temperature reaction, since hydrogen peroxide is unstable under the temperature conditions of Welling. Still further, the use of a conventional amalgamated aluminum does not lend itself to the preparation of an aluminum product containing hydroperoxy groups and one which can be characterized as transparent, in sheet or plate form and with the physical appearance of mica. Such a product has now been obtained, however, by adherence to the parameters of the present invention.

## **SUMMARY OF THE INVENTION**

The method of the present invention allows the formation of an aluminum product having the physical appearance of mica, the aluminum product further being characterized by the presence of hydroperoxy groups, transparency and a sheet or plate-like form, the aluminum product appearing polymeric in nature.

The aluminum-containing product is prepared by contacting and reacting, at room temperature or below (a) a reactive metal comprising aluminum metal of a purity of at least 99.9% by weight permeated with a liquid metal selected from mercury, gallium and indium/gallium alloys with (b) a liquid reactant comprising (i) hydrogen peroxide in (ii) an anhydrous liquid organic compound having no multiple carbon-to-carbon bonds. The liquid reactant, i.e., hydrogen peroxide, in the anhydrous liquid organic compound is substantially free of water, i.e., has up to about 10% by weight water, preferably as little water as possible.

A further aluminum-containing product can be obtained by firing the product obtained by the foregoing reaction at an elevated temperature. This appears to effect a release of hydrogen from the hydroperoxy-group-containing aluminum product.

In an alternative embodiment, a hydroperoxy-group-containing aluminum product having a metal deposited on or between sheets or plates is obtained by adding a minor amount of a metal powder or metallic salt to the reaction system.

## **BRIEF DESCRIPTION OF THE DRAWINGS**

**FIG. 1 is a representation of the proposed chemical structure of the aluminum-containing product of the present invention as obtained by reaction between an activated aluminum and hydrogen peroxide;**

**FIG. 2 is a representation of the proposed chemical structure of the aluminum-containing product of FIG. 1, after firing at elevated temperature; and**

**FIG. 3 comprises two superimposed infrared scans of the aluminum-containing product; one as produced and one after firing.**

## **DESCRIPTION OF PREFERRED EMBODIMENTS**

As indicated previously, a hydroperoxy-group-containing aluminum compound, specifically one having a .gamma.-structure, is produced in accordance with the present invention by the reaction of a reactive form of aluminum and a liquid reactant comprising hydrogen peroxide in an anhydrous liquid organic compound having no multiple carbon-to-carbon bonds. Such hydroperoxy-group-containing aluminum compound is characterized by transparency, and formation in a sheet or plate-like form, the physical appearance of the product resembling that of mica. In addition, the hydroperoxy-group-containing aluminum compound appears polymeric in nature.

The reactants necessary to achieve such hydroperoxy-group-containing aluminum product will be discussed individually below:

### **Reactive Form of Aluminum**

A principal reactant in the preparation of the hydroperoxy-group-containing aluminum product of the present invention is aluminum metal,

which has been activated or made reactive through permeation by a second metal, i.e., a liquid metal selected from mercury, gallium and indium/gallium alloys. By "activated" or "reactive" is means that the inter-crystalline structure of the aluminum is made uniquely and exceptionally receptive to chemical reaction. A more thorough description of the aluminum activation technique as well as an explanation of the physio-chemical phenomena which occur during activation is contained in copending application Ser. No. 497,560, filed Aug. 15, 1974, which is hereby incorporated by reference.

Basically, activation of the aluminum is carried out by wetting the surface of the aluminum in the presence of a source of hydrogen ions (protons), such as hydrochloric acid, with a liquid second metal selected from mercury, gallium and indium/gallium alloys until the aluminum has been permeated with the second metal. Evidence has been developed to establish that during this permeation of the aluminum the grain boundaries of the aluminum are actually penetrated by atoms of the second metal. This appears to create a realignment of the aluminum grain boundaries which has the effect of opening up the domains within the aluminum metal, resulting in unique reactivity properties.

The activated aluminum which is utilized in the process of the present invention shows many unique characteristics when compared with the base untreated aluminum metal. One of these is the dissolution of the aluminum and release of hydrogen ions or protons when placed in a liquid medium containing a source of hydrogen ions.

In preparing the activated aluminum, aluminum metal in the form of relatively small rods, bars or sheets, etc., preferably substantially free of bends and twists, is generally utilized. Preferably, soft aluminum metal having a purity of at least 99.9% by weight is used. The use of an aluminum metal with a substantial percentage of impurities is disadvantageous in that activation requires substantially longer periods of time. More importantly, there is the tendency for the impurities to enter into and precipitate from the subsequently formed products. Generally, however, the impurities are leached from the aluminum during extended periods of activation.

Using as an example the aluminum-mercury system, activation of the aluminum can be effectively carried out by floating an aluminum bar or rod on mercury in the presence of a hydrogen ion donating medium. This hydrogen ion donating medium can be an aqueous inorganic or organic acid, an aqueous alkaline solution of an alcohol, etc. In principle, any hydrogen ion source or hydrogen ion donating medium can be utilized. It is hypothesized that the mercury is caused to penetrate and diffuse through the aluminum in the form of a stable hydride formed in situ by the reaction between the mercury metal and hydrogen ions of the hydrogen ion donating medium. The ability of mercury, indium and gallium to act in a similar fashion appears to be associated with the hydride-forming capabilities of these metals and the fact that each of these metals has an atomic volume similar to that of hydrogen itself. This allows the metals to penetrate and diffuse through the aluminum either together with hydrogen or in the form of the hydride.

As will be apparent to those skilled in this art, the length of time required for activation of the aluminum with mercury will depend upon many factors, including the exposed surface area, which is related to the hardness of the aluminum, and the amount of impurities present in the aluminum. Longer times are required for harder aluminums and those with greater impurity contents. As a general rule, adequate activation of aluminum rods with mercury can be achieved in a time period of from about two hours to about 24 hours. When utilizing a preferred soft grade of aluminum, activation can be achieved in a period of from 8 to 12 hours.

When the aluminum is activated, it becomes embrittled and can be easily broken, using a snapping action. By breaking the aluminum and examining the grain structure at the break, one can determine the degree to which the mercury has penetrated and diffused throughout the aluminum. In accordance with the preferred embodiment of the present invention, the desired hydroperoxy-group-containing aluminum products are prepared utilizing activated aluminum in which the mercury, gallium, or indium/gallium alloy has penetrated and diffused entirely throughout the aluminum structure.

In forming the activated aluminum, high temperatures should be avoided since high temperatures appear to prevent grain structure realignment due to lattice expansion. Generally, the activation of aluminum with mercury can be carried out under ambient temperature conditions, with a temperature in the range of from 40 DEG F. to 100 DEG F. being preferred. When utilizing gallium as the second metal, it is often desirable to gently heat the gallium in order to maintain the gallium in the liquid state, thereby allowing the permeation to more effectively take place. Here again, however, excessive temperatures should be avoided.

When forming the activated aluminum using gallium or an indium/gallium alloy, the permeation and diffusion of the gallium or indium/gallium through the aluminum rod may be initiated merely by contacting one end of the aluminum rod with the second metal or metals in the presence of the hydrogen ion donating medium. The initiation of diffusion may be observed by noting the surface wetting of the aluminum with the second metal or metals above the point of contact. Further diffusion of the gallium or indium/gallium alloy through the entirety of the aluminum can then be continued by placing the aluminum rod in an anhydrous organic liquid medium such as alcohol, or the like, which is capable of donating hydrogen ions for effecting further diffusion.

With respect to the indium/gallium alloys which may be used, essentially any alloy composition will be suitable, ranging from 90% by weight gallium and 10% by weight indium to 10% by weight gallium and 90% by weight indium. When employing such alloy, however a 50:50 weight mixture of gallium and indium is extremely effective. As the percentage of indium is increased in the indium/gallium alloy, there is an increasing tendency for the activated aluminum to disintegrate. This does not prevent production of the desired products of the present invention, but could slightly complicate withdrawal of unreacted activated aluminum from the reaction medium. The amount of mercury, gallium or indium/gallium alloy which penetrates and diffuses through the aluminum will depend, of course, upon the length of treatment, temperature of treatment and the ability of the aluminum to retain the second metal or metals. As previously mentioned, it is preferred in accordance with the present invention to employ an activated aluminum in which the mercury, gallium or indium/gallium alloy has penetrated entirely through the aluminum.

Generally, a soft aluminum will pick up and retain a maximum of about 5% by weight of mercury during the activation process, with typical pickups being in the 2.5 to 3.5% by weight range. This amount of mercury is very effective to achieve the desired results of the present invention. Even with much smaller amounts of mercury, however, e.g., amounts as low as 0.1%, the aluminum becomes activated and can be used to produce the unique products of the present invention. It is preferred, however, to operate with a mercury content within the above specified range of about 2.5% to about 3.5% by weight.

It has generally been found that an aluminum activated with gallium or indium/gallium alloy is more active than the mercury-activated aluminum. For this reason, the amount of gallium or indium/gallium required to achieve suitable activation is less than the amount of mercury required. Generally, the aluminum will pick up and retain a maximum of about 3% by weight gallium or indium/gallium alloy. However, suitable activation can be achieved with as little as about 0.05 to about 1.0% by weight gallium or indium/gallium alloy and it is preferred to utilize an amount in this range. However, it has been found that even with amounts of these metals as low as 0.01% by weight, very active aluminum effective for the process of the present invention can be provided.

## The Liquid Reactant

The liquid reactant is separated into two portions, the first being the active reactant, hydrogen peroxide, and the second being a carrier for the hydrogen peroxide, specifically a liquid organic compound having no multiple carbon-to-carbon bonds.

The hydrogen peroxide reactant in accordance with the method of the present invention is generally in the form of an aqueous solution of hydrogen peroxide. However, since it is necessary in accordance with the present invention to minimize the amount of water present in the reaction system, the aqueous solution of hydrogen peroxide should be somewhat concentrated, with concentrations of 30% hydrogen peroxide or greater being generally used, preferably 50% hydrogen peroxide or greater. In addition, it is applicable in accordance with the present invention to employ 100% hydrogen peroxide. However, highly concentrated hydrogen peroxide solutions, i.e., those of about 70-95% concentration must be handled with care.

The vehicle or carrier for the hydrogen peroxide can effectively comprise any liquid organic compound having only single carbon-to-carbon bonds, i.e., one which contains no carbon-to-carbon double bonds. Preferably, such liquid organic compound is an anhydrous compound, again to effectively minimize the water content of the reaction system. Typical examples of such liquid organic compounds having no carbon-to-carbon double bonds include, for example, alcohols, particularly lower monohydric alcohols, such as methanol, ethanol, isopropyl alcohol, n-butyl alcohol, isobutyl alcohol, amyl alcohol, hexanol, etc.; ketones, particularly ketones of up to six carbon atoms, including for example, formaldehyde, acetaldehyde, etc.

The absence of carbon-to-carbon double bonds in the liquid organic compound acting as a vehicle or carrier for the hydrogen peroxide is important since compounds with carbon-to-carbon double bonds tend to enter into reaction with the activated or reactive aluminum, and in some instances, take part in polymerization reactions. Accordingly, such reactions which may tend to interfere with the interaction and reaction between the hydrogen peroxide and activated aluminum should be avoided. Accordingly, any liquid organic compound which does not readily react with the activated aluminum under the conditions of the present method can be effectively employed as the carrier or vehicle for the hydrogen peroxide.

In order to minimize the water content of the liquid reactant in accordance with the present invention, the carrier or vehicle liquid is generally employed in a large amount with respect to the hydrogen peroxide solution. As indicated previously, the total water content of the liquid reactant should not exceed about 10% by weight. Generally, such water content is about 2-5% by weight. This is effectively achieved by employing a ratio of the liquid organic compound having no carbon-to-carbon double bonds to the aqueous hydrogen peroxide solution of from about 2:1 to about 20:1 or more. Certainly it should be apparent that even greater amounts of the carrier or vehicle when compared to the aqueous hydrogen peroxide solution can be utilized to even further minimize the amount of water present in the liquid reactant system.

The use of an anhydrous liquid organic compound is advantageous in the process of the present invention since the anhydrous organic liquid will have the tendency to tie up the small percentage of free water present in the reaction system, such as in the aqueous hydrogen peroxide reactant. Accordingly, the amount of anhydrous liquid organic compound based on the amount of hydrogen peroxide should preferably be sufficient to tie up any free water present in the system. This allows the reaction system to be substantially free of free water. Accordingly, as used throughout, the expression "substantial absence of water" is meant to embrace the above limitations, specifically the limitation that a maximum of 10% water can be present in the reaction system.

## Optional Metallic Reactant

It is possible in accordance with the present invention to deposit on the hydroperoxy-group-containing aluminum compound a metal or combination of metals by employing in the reaction system a powdered metal or metallic salt capable of depositing the metal under the conditions of the reaction. By employing such metal powder or metal salt in the process of the present invention, the metal or combination of metals are deposited on the hydroperoxy-group-containing aluminum product between sheets or flakes. This can be carried out to provide coloration to the hydroperoxy-group-containing aluminum compound, such as in the preparation of a metallic pigment, or can be used to deposit a catalytic component, such as platinum, palladium, etc., on the hydroperoxy-group-containing aluminum compound, which provides a support for the catalyst.

Typical metals applicable in accordance with the present invention include, for example, platinum, palladium, chromium, manganese, titanium, silver, gold, iron, cobalt, magnesium, cadmium, nickel, copper, zinc, zirconium, alkali metals, etc. Again, where possible, these metals can be utilized in the form of the metallic powder or the salts of these metals which are capable of depositing the metal under the conditions of the reaction can be utilized. Typical salts include, for example, the oxide, nitrate, or chloride of the respective metals. The salt of the metal should be one which is soluble in the carrier medium, e.g., alcohol, utilized in the reaction system of the present invention.

Generally, it is necessary only to deposit a very minor amount of the metal onto the hydroperoxy-group-containing aluminum compound to achieve the desired coloration and/or catalytic properties. Accordingly, such metal or metallic salt should be employed in this alternative embodiment in that amount. An amount of about 0.01% to about 5% by weight based upon the weight of the reactive or activated aluminum is sufficient for this purpose. Here again, however, somewhat lesser or greater amounts can be utilized to achieve particular purposes.

## Reaction Parameters

As indicated previously, the process of the present invention to prepare the hydroperoxy-group-containing aluminum compound is carried out by simply contacting and reacting the reactive aluminum with a liquid comprising (i) hydrogen peroxide in (ii) an anhydrous liquid organic compound having no multiple carbon-to-carbon bonds. In addition, as previously indicated, such reaction is carried out in the substantial absence of water. Still further, it is important in accordance with the present invention that the reaction be carried out without heating, i.e., at room temperature or below, preferably below room temperature, by cooling the reaction system during the reaction. This can be achieved in any suitable manner, such as by carrying out the reaction with the reaction vessel immersed in an ice bath.

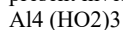
The process of the present invention is preferably carried out by adding the reactive aluminum in rod, bar or sheet form to a suitable reaction vessel, preferably vertically oriented, with the activated aluminum rod being immersed in the liquid reaction system comprising the hydrogen peroxide in the anhydrous liquid organic compound having no multiple carbon-to-carbon bonds. It is totally unnecessary to use any sophisticated reaction system and unnecessary to provide any agitation of the reaction system. Clearly, all that is necessary in accordance with the present invention is to bring together the hydrogen peroxide and reactive or activated aluminum in a suitable reaction vessel in the substantial absence of water, and at room temperature or below.



In accordance with the process of the present invention, by maintaining reactive contact between the hydrogen peroxide and reactive or activated aluminum, it appears that a layer of hydrogen peroxide forms over the surface of the activated or reactive aluminum. Another layer of the hydrogen peroxide forms over the first layer and apparently, successive layers build up in this manner.

In accordance with the above hypothesized formation of the hydroperoxy-group-containing aluminum compound, it is presumed that the hydrogen peroxide groups alternate in accordance with the charge pattern distribution on the atoms therein. Highly concentrated hydrogen peroxide has a tendency to produce hydroperoxy groups which are relatively stable radicals. It is hypothesized that during the production of such hydroperoxy groups, the associated hydrogen ion diffuses into and then out of the reaction or activated aluminum. As this reaction continues, the sheet or flake-like form of the hydroperoxy-group-containing aluminum compound is developed in accordance with the hypothesized atomic structure illustrated in FIG. 1.

Referring to FIG. 1, it is noted that from the proposed or hypothesized model, there is a sharing of hydroperoxy groups by the aluminum atoms. Therefore, a more accurate representation of the basic building block of the hydroperoxy-group-containing aluminum compound of the present invention may be the following:



each of these formulae and atomic structures are merely hypothesized, however, and there is no intention that this invention is limited thereto. Rather, such hypotheses are presented for a more clear understanding of the process of the present invention.

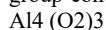
As indicated previously, the hydroperoxy-group-containing aluminum compound of the present invention is formed as sheets or flakes which resemble mica in physical appearance. These sheets or flakes which are successively built up on the reactive or activated aluminum rod separate or flake off of the aluminum. The product can then be easily removed, as by decanting the liquid, filtration, etc.

The individual layers of the hydroperoxy-group-containing aluminum compound in accordance with the present invention are transparent to natural light. In this regard, such layers resemble window glass in their translucent properties. However, when multiple layers of the hydroperoxy-group-containing aluminum compound are built up, the product is pearlescent due to the light refraction associated with the multiple layers.

While the hydroperoxy-group-containing aluminum compound is formed in the above-described reaction as a clear, transparent product, a pigment-type product, i.e., one which is colored, can be produced as previously described by including a metal powder or soluble metal salt in the reaction system. Accordingly, various colored products can be produced. In addition, by the judicious selection of the metallic component, highly catalytic products with the hydroperoxy-group-containing aluminum compound as a support can be produced.

The hydroperoxy-group-containing aluminum compound of the present invention has the property of being stable over a wide temperature range. However, when this product is subjected to a greatly elevated temperature in the order of 2000 DEG C. or higher, hydrogen ions are released from the structure of the hydroperoxy-group-containing aluminum compound. It is hypothesized that such release of hydrogen atoms results in a product having a structure as illustrated in FIG. 2.

It is noted from FIG. 2 that the proposed structure corresponds to that of sapphire. Presumably, the basic building block of the hydroperoxy-group-containing aluminum compound after firing at a temperature in the order of 2000 DEG C. takes the form as follows:



it is interesting to compare the reaction of the hydroperoxy-group-containing aluminum compound with sulfuric acid, both before and after firing. Before firing, it appears as though a sulfite is formed, thereby illustrating that the aluminum is in an ionic form. However, after firing, no reaction with sulfuric acid appears to take place.

FIG. 3 represents infrared scans of the hydroperoxy-group-containing aluminum compound both before and after firing. The wide absorption band in the region of 3400  $\text{cm}^{-1}$  in both infrared scans tends to establish the presence of hydroperoxy groups, rather than hydroxy groups which would be easily lost in firing. The change which takes place in the region of 900  $\text{cm}^{-1}$  is believed to be associated with the breaking of hydrogen bonds and release of hydrogen from the structure.

The present invention will now be described by reference to the following specific examples. It should be understood, however, that such examples are presented for purposes of illustration only, and the present invention can not under any circumstances be deemed limited thereby.

Unless otherwise stated in the following examples, and throughout this specification, all parts and percentages are by weight.

#### EXAMPLE 1

A high purity aluminum rod of about 100 grams and having a diameter of about one-half inch is activated by permeation with mercury in the presence of hydrochloric acid. The aluminum rod is saturated to the point of having about 3% by weight of mercury. The activated rod is then washed thoroughly with distilled water in order to remove the presence of residual hydrochloric acid or any chloride compound which might have been formed during the treatment. A tall, narrow vessel is filled with a mixture of about 1000 ml of anhydrous methanol and about 50 ml of hydrogen peroxide having a concentration of 30%. The activated aluminum rod is placed into the vessel so that it retains a vertical position. This is desirable in order to increase the available surface area to the reactants and to permit the free conduction and convection of heat away from the reactive aluminum rod. Cooling is applied where necessary to maintain the temperature at room temperature or below. The activated aluminum rod is submerged throughout the reaction. The reaction which takes place produces thin sheets which appear to peel off of the activated aluminum rod and accumulate in the vessel. It is desirable not to disturb or jar the vessel in order to avoid shattering the sheets. The sheets formed can be removed by removing the activated aluminum rod and then decanting the liquid. The final product can then be washed in distilled water and dried.

#### EXAMPLE 2

Example 1 is repeated with the use of an equivalent amount of anhydrous ethyl alcohol instead of anhydrous methanol.

#### EXAMPLE 3

Example 1 is repeated utilizing an equivalent amount of ethylene glycol in lieu of methanol.

#### **EXAMPLE 4**

Example 1 is repeated utilizing an equivalent amount of isopropyl alcohol in lieu of methanol.

#### **EXAMPLE 5**

Examples 1-4 are repeated with the exception that a 50% concentrated aqueous solution of hydrogen peroxide is used and the vessel is cooled with an ice pack.

#### **EXAMPLE 6**

Examples 1-4 are repeated with the exception that a 70% concentrated solution of hydrogen peroxide is used and the vessel is cooled with an ice pack.

#### **EXAMPLE 7**

Following the procedure of Example 1, a mercury prepared activated aluminum rod is reacted with 800 ml of anhydrous ethyl alcohol mixed with 200 ml of hydrogen peroxide having a concentration of 50%. A similar product is produced.

#### **EXAMPLE 8**

Example 7 is repeated except that 1 g of chromium oxide or chromium nitrate is mixed into the solution before the activated aluminum rod is inserted. This results in a green product with chromium deposited thereon.

#### **EXAMPLE 9**

Example 7 is repeated except that either manganese oxide or manganese nitrate is mixed into the solution before the activated aluminum rod is inserted. A pink product is produced.

#### **EXAMPLE 10**

The procedure of Example 7 is repeated except that 1 g of titanium nitrate or titanium oxide is added to the solution before the activated aluminum rod is inserted. A white product is produced.

#### **EXAMPLE 11**

The procedure of Example 7 is repeated except that 1 g of cobalt oxide or cobalt nitrate is added to the solution before the activated aluminum rod is inserted. A blue smokey product results.

#### **EXAMPLE 12**

The procedure of Example 7 is repeated except that 1 g of magnesium oxide or magnesium nitrate is added to the liquid before the activated aluminum rod is inserted.

#### **EXAMPLE 13**

The procedure of Example 7 is repeated except that 1 g of ferric oxide or ferric nitrate is added to the liquid before the activated aluminum rod is inserted. A reddish or ruby colored product results.

#### **EXAMPLE 14**

The procedure of Example 7 is repeated except that 1 g of cadmium oxide or cadmium nitrate is added to the liquid before the activated aluminum rod is inserted.

#### **EXAMPLE 15**

The procedure of Example 7 is repeated except that 1 g of nickel oxide or nickel nitrate is added to the liquid before the activated aluminum rod is inserted.

#### **EXAMPLE 16**

The procedure of Example 7 is repeated except that 1 g of zirconium oxide or zirconium nitrate is added to the liquid before the activated aluminum rod is inserted.

#### **EXAMPLE 17**

The procedure of Example 7 is repeated except that 1 g of potassium chromate is added to the liquid before the activated aluminum rod is inserted.

#### **EXAMPLE 18**

The procedure of Example 7 is repeated except that 1 g of cupric nitrate is added to the liquid before the activated aluminum rod is inserted.

#### **EXAMPLE 19**

The procedure of Example 7 is repeated except that 1 g of zinc oxide or zinc nitrate is added to the liquid before the activated aluminum rod is inserted.

## EXAMPLE 20

A catalyst supported on the hydroperoxy-group-containing aluminum compound of the present invention is prepared by a procedure similar to that of Example 1. A mercury prepared activated aluminum rod permeated with approximately 3% by weight mercury is contacted and reacted with 1000 ml anhydrous alcohol (SD40) with 100 ml of hydrogen peroxide, a 50% aqueous solution. Some 30 drops of silver nitrate are added to the alcohol-hydrogen peroxide liquid system prior to introduction of the activated aluminum rod. This results in the deposition of silver on the sheet or plate-like hydroperoxy-group-containing aluminum product.

## EXAMPLE 21

The procedure of Example 20 is repeated with the exception that 30 drops of gold chloride are utilized in lieu of the silver nitrate. This results in the deposition of gold on the hydroxy-group-containing aluminum product.

## EXAMPLE 22

The procedure of Example 20 is repeated with the exception that 24 drops of platinum chloride are utilized in lieu of the silver nitrate. The resulting product comprises a sheet or plate-like hydroperoxy-group-containing aluminum support with platinum deposited thereon. This product shows excellent catalytic activity.

## EXAMPLE 23

The procedure of Example 20 is again repeated with the exception that 6 drops of palladium chloride are utilized in lieu of the silver nitrate. This product comprises palladium metal deposited on the hydroperoxy-group-containing aluminum support. Again, excellent catalytic activity is illustrated.

## EXAMPLE 24

The hydroperoxy-group-containing aluminum product formed in sheet or plate-like form in accordance with Example 1 is fired in an oven at a temperature in the order of 2000 DEG C. This firing results in the release of hydrogen atoms from the solid sheet-like product. This fired product is particularly suitable for electronic support applications.

While the present invention has been described primarily with regard to the foregoing specific exemplification and reference to specific embodiments, it should be understood that the present invention can not under any circumstances be deemed limited thereto, but rather, must be construed as broadly as all of any equivalents thereof.

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### Metallic-organo-peroxide and organo-metallic-peroxide

US4017527

[ [PDF](#) ]

## BACKGROUND OF THE INVENTION

The present invention relates to a method for the formation of metallic-organo-peroxides and organo-metallic peroxides and to such compounds so produced.

Many methods for forming organo-peroxides are known. Reference is made to the book, "Hydrogen Peroxide in Organic Chemistry" by John G. Wallace, published by the Electro Chemical Department of EIdu Pont De Neumours and Company.

Generally, an organic peroxide is defined as a derivative formed by replacing one or both hydrogen atoms of hydrogen peroxide by an organic radical. The present method produces compounds which include a metal atom bonded to an organic radical and a peroxide type bonded to the organic radical. The peroxide group is the oxygen to oxygen type bond in which a hydrogen atom can be connected to one of the oxygen atoms. The method of the present invention also produces a product wherein the organic radical has a metallic atom bonded to it and the peroxide group is bonded to the metal atom. The differences in the types of products produced gives rise to the distinction between the description of the product as being on the one hand a, metallic-organo-peroxides and on the other hand an, organo-metallic-peroxides.

One elementary method of producing an organic peroxide makes use of the fact that a strong aliphatic acid solution with hydrogen peroxide exists in equilibrium with the corresponding organic peracid. ##STR1##

However, in the absence of a strong acid catalyst, such as a mineral acid, the attainment of equilibrium is impractically slow, especially at temperatures below 40 DEG C. At higher temperatures, it is difficult to prevent the excessive loss of active oxygen unless the oxidizable organic substance is also present to react with the organic peracid as it forms. For this reason, it is common practice, whenever possible, to employ hydrogen peroxide under conditions for the in situ formation of the organic peracid at temperatures ranging up to the boiling point of the aliphatic acid.

The peracid formed in situ can be reacted with an olefinic material to produce an epoxy (oxirane) compound as a primary product. ##STR2##

The reaction is, of course, two-staged, since the peracid must be formed first.

Common organic peracid systems include glacial acetic acid or formic acid with hydrogen peroxide. The organic peracids thus formed are unstable and considered a hazard particularly when the organic peracid is relatively concentrated.

It is known that the mild oxidizing action of hydrogen peroxide is increased considerably by use of certain metallic catalysts. One example of metallic catalyst is ferrous sulfate which is employed in a redox system:

$Fe^{+2} + H_2O_2 \rightarrow Fe^{+3} + H_2O + OH^-$

It has been employed with hydrogen peroxide and is generally known as Fenton's Reagent. Other catalyst include osmium and tungstic oxides

employed to hydroxylate aromatic and unsaturated hydrocarbons and to effect other oxidations. These additional catalysts are classed as Milas' Reagents which together with Fenton's Reagent constitute the bulk of the metal activated hydrogen peroxide systems. Hydrogen peroxide in metal-activated systems reacts as though it was dissociating into two hydroxyl radicals.

Fenton's oxidations are, in fact, believed to proceed through the intermediate formation of hydroxyl free radicals:  
$$\text{Fe}^{2+} + \text{H}_2\text{O}_2 \rightarrow \text{Fe}^{3+} + \text{OH}^- + \text{OH}^\bullet$$

the  $\text{Fe}^{2+}$ - $\text{Fe}^{3+}$  systems, and such other redox systems as  $\text{Cu}^{+}$ - $\text{Cu}^{2+}$ , are normally employed with hydrogen peroxide in aqueous acid medium. A small amount of sulfuric acid is added to an aqueous solution of ferrous sulfate heptahydrate so that Fenton's oxidations are carried out at a pH of 1-4. In a less acid solution, the reaction efficiency is decreased, and hydrogen peroxide is catalytically decomposed.

Other metal-activated systems include in decreasing order of catalytic efficiency the following:  
 $\text{OsO}_4$ ,  $\text{WO}_3$ ,  $\text{MoO}_3$ ,  $\text{SeO}_2$ ,  $\text{CrO}_3$ ,  $\text{V}_2\text{O}_5$ ,  $\text{TiO}_2$  and  $\text{Ta}_2\text{O}_5$

Derivatives of the aforementioned catalyst, such as phosphotungstic acids (e.g.  $\text{H}_3\text{PO}_4 \cdot 12\text{WO}_3$ ) are also effective as catalysts for hydrogen peroxide.

The prior art shows four general methods of incorporating the peroxide bond ( $-\text{OO}-$ ) into organic molecules. These methods include auto-oxidation, ozonization, the association of oxygenated free radicals, and the addition and substitution reactions of hydrogen peroxide and hydroperoxides. Typically, hydrogen peroxide is reacted with acids, anhydrides, esters, alcohols, organic sulfates and sulfonates, carbonyl compounds, and organic chlorine compounds to produce organic peroxides.

The reaction of acids anhydrides and esters with hydrogen peroxide ordinarily lead to the formation of organic peracids, although other organic percompounds may result. The most popular method of preparing a peracid is by mixing hydrogen peroxide and an aliphatic acid in the presence of a strong acid catalyst such as sulfuric acid. Typically an equimolar mixture of high strength hydrogen peroxide and acetic acid with one percent sulfuric acid catalyst reaches equilibrium after standing for 12 to 16 hours.

The resin technique for peracetic acid formation is considered much faster and permits continuous or batchwise preparation.

The resin technique for peracetic acid formation is operated simply by passing a mixture of glacial acetic acid and hydrogen peroxide through a cation exchange resin column. The column contains polystyrene sulfonic acid resin which has been treated with glacial acetic acid to remove excess water. Under conditions for operation of the resin technique, a contact time of 12 to 16 minutes at about 45 DEG C is sufficient for maximum conversion of hydrogen peroxide to peracetic acid. The serious drawback in this method is that it requires high strength hydrogen peroxide and thus creates a serious hazard.

A common procedure for converting an alcohol,  $\text{R}-\text{O}-\text{H}$ , to a hydroperoxide,  $\text{R}-\text{OOH}$ , consists of reacting hydrogen peroxide and tertiary alcohols with strong sulfuric acid. The reaction is believed to involve the formation of an intermediate sulfate and, therefore, is similar to the alkylation of hydrogen peroxide by dialkyl sulfates. The reaction often results in serious explosions, although the final products of such reactions are relatively stable. Prior art methods do not produce peroxide products with primary or secondary alcohols very easily. Mixtures of a tertiary alcohol and hydrogen peroxide have been used as germicides, fungicides, bleaching agents, and peroxide reagents.

The strong sulfuric acid used in the hydrogen peroxide-alcohol reaction is sometimes replaced by heteropolyacids having multiple inorganic acid radicals. The heteropolyacids which are soluble in ether, contain the elements of phosphorous, silicon, or boron, coordinated with a metallic oxide such as tungsten oxide. The characteristic solubility of heteropolyacids allows the preparation of alkyl hydroperoxides to be carried out in ether.

The known methods of converting carbinols of many types to hydroperoxides and disubstituted peroxides makes use of strong sulfuric acid as a catalyst. Typically, acetylene peroxides are formed by the interaction of hydrogen peroxide and the hydroxyl group of acetylenic carbinols in the presence of strong sulfuric acid. The peroxides thus formed are unusually stable despite the presence of the acetylenic bond.

Dialkyl sulfates and alkyl hydrogen sulfates can be used to produce hydroperoxides and dialkyl peroxides by a reaction with alkaline hydrogen peroxide. These peroxides are often used as polymerization catalysts and diesel fuel additives. Typically, primary and secondary dialkyl peroxides are prepared by the alkylation of hydrogen peroxide with alkyl methane sulfonates in liquid alkaline methanolic solution. A known method for producing sodium peroxy sulfonates is carried out by reacting a sulfonic acid such as naphthalene sulfonic acid and sodium peroxide in a liquid medium. The operation is conducted in a cold environment to reduce the violence of the reaction. The peroxy product obtained has approximately 6% active oxygen and is considered useful as a bleaching agent or insecticide.

Olefins have been transformed to hydroperoxides in a reaction which amounts to the addition of hydrogen peroxide to the double bond. The reaction, however, is conducted in strong sulfuric acid according to known methods and probably involves the formation of an intermediate sulfate. ##STR3##

The preparation typically takes place at below 0 DEG C and takes several hours.

It is known that hydrogen peroxide can be reacted with an aldehyde or a ketone in the presence of a catalyst to form a peroxide compound. The following equilibrium is believed to occur for an aldehyde and hydrogen peroxide: ##STR4##

Typically, formaldehyde is treated with hydrogen peroxide in ether in the presence of  $\text{P}_2\text{O}_5$ . The peroxide product is very explosive. Both cyclic ketones and aliphatic ketones have a tendency to form stable but hazardous peroxides. As a rule, known methods produce unstable peroxides of aromatic ketones and aromatic aldehydes because of the greater ease of migration of the phenyl radicals attached to the carbonyl carbon.

It is known that organic peroxides can be formed by reacting hydrogen peroxide with organic chlorine compounds. The reaction is generally conducted in the presence of a chlorine acceptor which may be caustic or organic bases such as pyridine.

## SUMMARY OF THE INVENTION

One of the principal objects of the invention is to provide a peroxide compound comprising the steps of preparing a reactive aluminum and

reacting the reactive aluminum with a liquid mixture of hydrogen peroxide and an organic compound. Another object of the present invention is to provide a method for forming a metallic-organoperoxide comprising the steps of forming a reactive aluminum and contacting a liquid mixture of hydrogen peroxide and an organic compound with the reactive aluminum. Another object of the invention is to provide a method for forming an organo-metallic-peroxide comprising the steps of forming a reactive aluminum and contacting a liquid mixture of hydrogen peroxide and an organic compound with the reactive aluminum.

Another object of the present invention is to provide a method of forming an alcohol peroxide comprising the steps of forming a reactive aluminum and contacting a liquid mixture of an alcohol and hydrogen peroxide with the reactive aluminum.

Another object of the invention is to provide a method of forming an aldehyde peroxide comprising the steps of forming a reaction aluminum and containing a liquid mixture of an aldehyde and hydrogen peroxide with the reactive aluminum.

Another object of the invention is to provide a method of forming a ketone peroxide comprising the steps of forming a reactive aluminum and contacting a liquid mixture of a ketone and hydrogen peroxide with the reactive aluminum.

The invention accordingly comprises the several steps and the relation of one or more of such steps with respect to each of the others, or as exemplified in the following detailed disclosure, and the scope of the application of which will be indicated in the claims.

## THE INVENTION

The present invention is focused on the utilization of the catalytic properties of a reactive aluminum prepared by the methods described in co-pending patent application, "Catalytic Electrode", Ser. No. 211,979, filed Dec. 27, 1971, now abandoned.

The reactive aluminum in the present disclosure corresponds to the aluminum catalytic electrode described in the aforementioned patent application.

Generally, a reactive aluminum is prepared by contacting highly pure aluminum in the presence of a hydrogen ion source with a metal which can form a hydride. The hydrogen ion source can be inorganic acid or the like or an inorganic acid such as citric acid or acetic acid or the like. The reactive aluminum in an alkali solution such as water and sodium hydroxide will serve as a hydrogen ion source for the formation of another reactive aluminum.

The metal for forming the reactive aluminum can be an element or an alloy. Preferably, the metal is gallium, or indium or an alloy of the two or mercury. A desirable characteristic of the metal is that it is soft and pliable and preferably a liquid during the process in order to permit the rapid permeation through the aluminum. The general rule is that an element with an atomic volume near that of hydrogen is preferred for this metal.

It should be understood that the term "highly pure" herein means greater than 99% and that purities in the order of 99.9 and 99.99% are preferable.

One simple method of preparing a reactive aluminum is to use an aluminum piece, such as a rod 99.99% pure having a length of 3 inches and a diameter of a half inch. The aluminum rod is placed on its side in a glass dish and sufficient 2N acid, such as hydrochloric acid, is added to cover the aluminum rod. The aluminum rod is contacted with the metal such as mercury or gallium and the metal is given time to permeate through the aluminum rod. Of course, the aluminum rod can be treated in a vertical position if desired and a rod shape is not necessary. Other shapes may be used.

The concentration of the acid can cover the broadest range to even include neutral water. The choice of the hydrogen ion source such as an acid will depend upon the peroxide compound to be formed and the concern over impurities.

It is preferable to prepare the aluminum rod for the reaction by at least partially stripping the aluminum oxide coating which has formed on the surface due to the exposure to air and moisture. If the aluminum rod has been stripped, then hot water can serve as the hydrogen ion source. Otherwise, it may be desirable to start out with an acid to strip off the oxide coating on the aluminum rod in order to initiate the reaction to form the reactive aluminum as quickly as possible. Of course, the aluminum rod may be stripped mechanically with sandpaper or a file or the like.

The inter-reaction which occurs between the aluminum rod and the acid, gives rise at the start to the formation of large bubbles which rise up to the surface through the acid. After a while it will be observed that instead of large bubbles forming at the top of the aluminum rod and then breaking free and rising to the surface of the acid, tiny bubbles will be emanating from many parts of the upper surface of the aluminum rod. The occurrence of the multitude of tiny bubbles indicates that the aluminum rod is being converted into a "reactive" aluminum.

Generally, the aluminum rod will take up or absorb from 0.1 to 5 percent of the mercury by weight depending upon how long the reaction is permitted to continue. A range of 2 to 3 percent of the mercury by weight is desired for many applications. However, in some applications as little as 0.1 percent of the mercury by weight is preferred.

The reaction can be stopped on the basis of the increased weight of the aluminum rod due to the absorption of the metal or due to the production of a multitude of tiny bubbles for a period of 10 to 15 minutes or due to the observance of the hydrolysis of water when the aluminum rod being treated is placed therein.

An aluminum rod treated as described, displays surprisingly active catalytic properties not at all suggested by the prior art. The prior art has recognized that aluminum and an amalgam of aluminum exhibit catalytic properties. It is of considerable significance that the treatment of highly pure aluminum as described herein exhibits catalytic and initiating properties that far exceed prior art contemplation.

Another method of preparing a reactive aluminum uses gallium instead of mercury. The same aluminum rod is placed in a glass dish and covered with hydrochloric acid and one end of the aluminum rod is contacted with the gallium having a mass from 1 to 3 percent of the aluminum rod. The treatment takes from 10 to 15 minutes depending upon how well the oxide coating on the aluminum rod has been removed at the point of contact of the gallium and the aluminum rod. A fairly clean part of the aluminum rod is indicated by the observance of large bubbles generated thereat.

It is desirable to dip a reactive aluminum formed with gallium into anhydrous alcohol immediately after completion of the process in order to

prevent the formation of a brownish compound on the aluminum rod thereafter.

The amount of the metal in the aluminum rod can be varied in accordance with applications. In general, if a high percent of the metal by weight is desired, quick cooling of the reactive aluminum rod after formation will prevent the squeezing out of the metal due to an exothermic reaction. Water is convenient for this purpose for a reactive aluminum formed with mercury. However, in cases where it is desired to reduce the amount of, say, mercury from several percent by weight to, say, 0.1% by weight, the reactive aluminum can be heated to squeeze out the mercury.

From the above, it is clear herein, including the claims, what is meant by a "reactive aluminum".

The reactive aluminum exhibits an aligned matrix and, it is believed, capable of converting at least partially to a hydride at one or more valences and produces  $Al^{+}$ ,  $e^{-}$ ,  $H^{+}$ ,  $OH^{-}$ ,  $HO_2^{-}$ , and  $O_2^{-}$  radicals depending upon the fluid contacting the reactive aluminum. Where the fluid is hydrogen peroxide, it is believed that  $O_2^{-}$  and  $O_2^{2-}$  radicals are produced in addition to the aforementioned radicals.

Certain impurities such as copper and iron, inhibit the formation of a reactive aluminum and so should be avoided in the aluminum rod. Impurities which inhibit or promote the reaction are given in the aforementioned catalytic electrode application.

An important aspect of the present is the realization that the aforementioned treatment of highly pure aluminum results in a reactive aluminum which can bring about a reaction with an organic compound and hydrogen peroxide to form a peroxide compound. The reactive aluminum not only brings about the formation of the peroxide compound, but further results in the aluminum from the reactive aluminum entering into the peroxide compound to form a part thereof. The amount of aluminum which forms a part of the peroxide compound can vary from a fraction of a percent to 5 percent or more.

Generally, the reaction time for the production for most of the peroxides in accordance with the present invention is considerably shorter than the time taken by prior art methods. The rate of the reaction for the production of a peroxide in accordance with the present method will be determined by well known parameters, such as the concentration of the hydrogen peroxide, the temperature of the constituents, the quantity of the reactive aluminum, and other factors.

If a reaction is permitted to proceed too rapidly so that the exothermic reaction elevates the temperature of the constituents, there is a possibility that the hydrogen peroxide will tend to break up into water and oxygen and thereby reduce the concentration of the hydrogen peroxide. As a result, there will be fewer peroxide groups available for the formation of the desired peroxide compound.

One approach for controlling the reaction rate is to add the hydrogen peroxide a little at a time. Of course, this presumes the presence of a liquid organic compound already present in order to form the mixture with the hydrogen peroxide.

Of course, a dilute hydrogen peroxide can be used in order to reduce the exothermic reaction to produce a relatively low-grade peroxide compound without the incidence of an elevated temperature or a reaction which requires some monitoring.

Generally, the atomic bonding in an organic peroxide formed by the present methods will be dependent upon the bonding that is present in the organic compound used in the reaction with hydrogen peroxide in the presence of the reactive aluminum. For example, it is well known that double bonds in organic compounds tend to be preferred sites for inter-reaction or reactions in general.

Generally, an organic compound having only single bonds can be reacted with hydrogen peroxide in the presence of the reactive aluminum to form an organic peroxide which shows hydroperoxide groups attached to the aluminum atoms with the organic groups coordinated about the aluminum atom. The precise structure or organization is not known. In practicing the present method to form an organic peroxide with an organic compound having single bonds, it is preferred to use a relatively dilute solution of hydrogen peroxide so as to provide a readily available source of hydroxyl radicals. A water solution of hydrogen peroxide between 3 to about 10% is preferable. Higher concentrations of hydrogen peroxide can be used but will tend to produce an inorganic peroxide in competition with the formation of the organic-metallic-peroxide described. The examples given herein illustrate the use of single bonded organic compounds such as alcohols, ketones, and aldehydes.

In the case where the organic compounds include other than single bonds such as double bonds, triple bonds, multiple bonds in general, or the like, an organic peroxide having a peroxide or hydroperoxide group attached directly to an organic group can be produced. Since the organic peroxide product in this case also includes an aluminum atom, it is proper to describe such products as a metallic-organic-peroxides,

The presence of a ring structure in the organic compounds does not hereto affect the formation of an organic peroxide by the present methods. The organic compounds having at least one multiple bond include, the so-called vinyl-type compound. Basically, a vinyl-type compound is characterized by the formula  $R-CH=CH_2$ . The R or R' can be a member of the aliphatic series. When a nitrile group replaces the carboxyl group then acrylonitrile is obtained. For an amide group acrylamide is obtained while for an aldehyde group acrolein or acrylaldehyde is obtained. Either the R or R' or both can be replaced by a halide or an active halide.

It is interesting to note that in the article entitled, "Thermochemistry of the Hydrogen Polyoxides  $H_2O_3$  and  $H_2O_4$ " by Paul A. Giguere, published in the Transactions New York Academy of Sciences, that the presence of three radicals of  $H_2O_3$  and  $H_2O_4$  were prepared and measured under the extreme conditions of temperature of less than 100 DEG K. Furthermore, this article reports to indicate that the formation of higher order hydrogen polyoxides are extremely difficult and require extreme temperature and pressure requirements. Although no limitation is intended to be suggested by the presentation of theories related to the operation of the present invention, it is believed that in carrying out the present invention there is a considerable production of radicals of  $H_2O_3$  and  $H_2O_4$  due to the reactive aluminum reacting with the hydrogen peroxide. It has been observed that during the formation of an organic peroxide by the method of the present invention the removal of the reactive aluminum from the mixture into air produced a red mercuric oxide on the reactive aluminum which had been prepared with mercury. The formation of red mercuric oxide rather than black mercuric oxide is an indication of  $O_2^{-}$  radicals which points to the aforementioned polyoxide radicals. In another experiment, part of an organic peroxide prepared with a carboxylic acid, was contacted with some mercury and immediately produced red mercuric oxide which for the same reason given previously, tends to confirm the proposed theory.

The production of higher order polyoxide radicals is favored by the use of highly concentrated hydrogen peroxide in the order of 50% and the use of elevated temperatures above 50 DEG C. There is the possibility that the use of highly concentrated hydrogen peroxide at an elevated temperature may tend to cause a very quick breakdown of the hydrogen peroxide so that the reaction carried out at an elevated temperature will tend to readily reduce the concentration of the hydrogen peroxide.

An interesting theory has been evolved to explain the formation of organic peroxides in terms of the present methods. The theory is not intended to be a limitation. To start with, in a typical reaction involving hydrogen peroxide with a carboxylic acid, the carboxylic acid which is represented as RCOOH forms a layer around the reactive aluminum rod and the hydrogen peroxide forms a layer on the layer of the carboxylic acid. A layer of the carboxylic acid then forms about the hydrogen peroxide layer and it continues alternating between the two types of layers. This can be shown diagrammatically by the following: ##STR6##

In accordance with the hypothesis given herein, Table 1 shows the arrangement of the layers of carboxylic acid and hydrogen peroxide alternating and having an atomic arrangement corresponding with the charges of the atoms present therein. The cells shown in Table 1 indicate the cooperation between atoms to produce radicals which will eventually combine with aluminum atoms to form the metallic-organo-peroxide. It is seen that the radical RCOOOH is formed while hydrogen atoms which break away from the carboxylic acid atoms combine with hydroxyl atoms from the hydrogen peroxide to produce water. The hydrogen atoms readily diffuse about and through the matrix of the reactive aluminum to interact therein and ultimately the formation of the aluminum carboxylic peroxide takes the form of ##STR7## It is believed that the unusual stability of the organic peroxides prepared in accordance with the present methods is due to the fact that if the oxygen atom between the carbon and aluminum atoms is dislodged, the aluminum atom will immediately couple over to the carbon atom to restore the stability of the atomic structure. In the proposed form, it is noted that the aluminum atom acts as a cross-link between the organic peroxide groups.

Highly concentrated hydrogen peroxide has a tendency to produce hydroperoxy groups which are relatively stable radicals as compared to other peroxide type radicals. The hypothesis for the interreaction between a carboxylic acid and a weak hydrogen peroxide solution equals a somewhat similar hypothesis as given above in that alternate layers are produced on the reactive aluminum but the pattern is as shown in Table 2: ##STR8##

As can be seen, the layers of the carboxylic acid alternate with hydroperoxy radicals. The organization of the atoms with respect to each other is based on the supposed interreaction between the charges carried by the various radicals. The cells indicated in Table 2 suggest how the various atoms and radicals interreact in order to bring about the formation of the aluminum carboxylic peroxide. The presence of the hydroperoxide groups tends to give rise to hydroxyl atoms as the desired product is being formed. It is believed that the hydrogen radicals diffuse into the reactive aluminum. It may be that the overall reaction which occurs for hydrogen peroxide and a carboxylic acid is some combination of the illustrated reaction formats given in Tables 1 and 2. Furthermore, it may be that the resulting organic peroxide contains hydroperoxide groups.

It is of interest that generally peroxide is the preferred reactant but the essential features for the production of organic peroxide rely only on the presence of O@- radicals or the like. Known methods of producing the desired radicals include, for example, the use of ultraviolet light on a mixture containing water to produce O3 @- radicals. Another way of getting the desired radicals is to generate ozone and bubble it through the mixture which will be used to produce the organic peroxide. Other equivalent means for providing the desired radicals will be obvious to those skilled in the art.

The use of hydrogen peroxide with the concentration of about 30% is convenient and the examples given herein were performed using hydrogen peroxide with a 30% concentration except where indicated differently. Also, most experiments carried out were done at a temperature below 50 DEG C, but, of course, as it has been noted, it is sometimes desirable to use an elevated temperature in order to increase the occurrence of peroxide radicals. As noted previously, organic peroxides have many known uses. The present organic peroxides provide a novel use as a catalyst for the formation of resins since it is possible to use an organic peroxide of the same organic radical corresponding to the main organic radical in the resin. This is an attractive use since no problem of removing the catalyst occurs since the catalyst becomes an integral and tolerable component in the resin formed thereby.

## EXAMPLES

Illustrative, non-limiting examples of the practice of the invention are set out below. Numerous other examples can readily be evolved in the light of the guiding principles and teachings contained herein. The examples are intended merely to illustrate the invention and not in any sense to limit the manner in which the invention can be practiced. The parts and percentages recited therein and all through this specification, unless specifically provided otherwise, refers to parts by weight and percentages by weight.

### EXAMPLE 1

An aluminum methyl acrylic peroxide is formed by combining 86 grams of methyl acrylate and 393 grams of water, reacting with a reactive aluminum rod of about 54 grams, and then adding gradually about 40 grams of hydrogen peroxide.

### EXAMPLE 1

An aluminum ethyl oxalate peroxide is formed by combining 146 grams of ethyl oxalate, reacting with a reactive aluminum rod of about 54 grams, and then gradually adding about 100 grams of hydrogen peroxide.

### EXAMPLE 3

An aluminum vinyl acetic peroxide is formed by combining 86 grams of vinyl acetate, reacting with a reactive aluminum rod of about 54 grams, and then gradually adding about 80 grams of hydrogen peroxide.

### EXAMPLE 4

An aluminum ethyl alcohol peroxide is formed by combining 44 grams of ethyl alcohol and 40 grams of hydrogen peroxide and reacting with a reactive aluminum rod of about 50 grams. The hydrogen peroxide should be added gradually and should have a concentration of less than 10%.

### EXAMPLE 5

An aluminum methyl ethyl ketone peroxide is formed by combining 100 grams of methyl ethyl ketone with 100 grams of hydrogen peroxide and reacting with a reactive aluminum rod of about 100 grams. The hydrogen peroxide should be added gradually and have a concentration of 10% or less.

#### EXAMPLE 6

An aluminum butyl alcohol peroxide is formed by combining 74 grams of butyl alcohol with about 100 grams of hydrogen peroxide and reacting with a reactive aluminum rod of about 100 grams. The hydrogen peroxide should be added gradually and have a concentration of 10% or less.

#### EXAMPLE 7

An aluminum acetaldehyde peroxide is formed by combining 44 grams of acetaldehyde with 50 grams of hydrogen peroxide and reacting with a reactive aluminum rod of about 50 grams and having a concentration of 10% or less.

#### EXAMPLE 8

An aluminum acetone peroxide is formed by combining 58 grams of acetone with 100 grams of hydrogen peroxide and reacting with a reactive aluminum rod of about 50 grams.

#### EXAMPLE 9

An aluminum isopropyl alcohol peroxide is formed by reacting 100 grams of isopropyl alcohol and 150 grams of hydrogen peroxide with a reactive aluminum rod of about 100 grams. The hydrogen peroxide should have a concentration of 10% or less.

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#### **Metalic-organo-peroxide and organo-metalic-peroxide and preparation thereof** **US3969387**

#### **Process for preparing peroxide group containing aluminum complex** **US4093707**

#### **METHOD OF FORMING A COMPOUND CONTAINING ALUMINUM AND GLYCEROL** **US4052429**

#### **Method of removing gaseous pollutants from gas streams utilizing an activated form of aluminum** **US4093702**

#### **Method of preparing metal-containing soap and soap so produced** **US4182685**

#### **HYDROPHOSPHIDE-GROUP CONTAINING MULTI-METAL INORGANIC POLYMERIC COMPLEX AND METHOD OF MAKING SAME** **US4117088**

#### **ALUMINUM HALOHYDRATE** **US4053570**

#### **HYDROSULFIDE-GROUPS CONTAINING MULTI-METAL INORGANIC POLYMERIC COMPLEX AND METHOD OF MAKING SAME** **US4117099**

#### **Aluminum containing alkenylbenzene resin** **US4071668**

#### **PROCESS FOR THE REMOVAL OF SO<sub>2</sub> FROM A STACK GAS, ABSORPTIVE MEDIUM FOR USE IN PROCESS AND PROCESS FOR PREPARING THE ABSORPTIVE MEDIUM** **US4108969**

#### **METALLSALTER AV KARBOXYLSYROR OCH FORFARANDE FOR DERAS FRAMSTELLNING** **SE7608006**

#### **Process utilizing activated aluminum and products produced thereby** **US4017591**

#### **PROCEDE POUR LA PREPARATION DES COMPLEXES ANORGANQUES** **RO71855**

#### **METHOD OF FORMING INORGANIC MONOMERIC POLYMERIC COMPLEXES AND PRODUCTS SO PRODUCED** **ZA7507451**

#### **MANUFACTURING PROCESS FOR INORGANIC MONOMER COMPLEX AND INORGANIC POLYMER COMPLEX** **JPS51132196** **Process for the removal of SO<sub>2</sub> from a stack gas** **US4012487**

#### **ALUMINIUMHALOGENOHYDRATE, VERFAHREN ZU DEREN HERSTELLUNG UND DEREN VERWENDUNG** **DE2502533**

#### **Aluminum organoiodides** **US4072699**



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**WERKWIJZE TER BEREIDING VAN EEN KUNSTMEST- POLYMEER.**  
CH605469

Activated aluminum and method of preparation thereof  
US3993595

**PROCEDIMIENTO PARA PREPARAR UN POLIMERO FERTILIZANTE**  
MX3118 (E)

**PEROXYDCHEMIKALIEN**  
DE2432889

Process for treating a contaminated flow medium  
CH609884

Aluminum hydrates and salts of carboxylic acids  
US3959093

Metal hydrates and salts of carboxylic acids  
US3957598

**MICA-TYPE ALUMINA AND RELATED COMPOUNDS**  
CA1029170

**GLIMMERARTIGES ALUMINIUMOXYD UND VERWANDTE VERBINDUNGEN**  
DE2405492

**ALUMINUM HALOHYDRATE**  
US4038373

**HARZE**  
DE2363419

**OXYGEN-BEARING ALUMINUM COMPLEX AND APPARATUS AND METHODS FOR PRODUCING THE SAME**  
CA939116

**ALUMINUM-COPPER-MERCURY COMPLEX AND METHODS FOR PRODUCING THE SAME**  
US3574607

**STEP-BY-STEP THERMO-MAGNETIC MOTOR**  
US3445740

**TRANSFORMER WITH TEMPERATURE CONTROLLED ADJUSTABLE COUPLING**  
US3454914

**RELAY WITH CONTACTS OF MERCURY METAL COMPOSITION**  
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