



# briefs

## ARTICLES IN THIS ISSUE

### STAFF-INDUSTRY COLLABORATIVE REPORT

#### New Ammonium Nitrate Process

Conventional ammonium nitrate plants carry out neutralization reaction of ammonia and nitric acid in a solution. In Commercial Solvents Corporation's new Stengel process, employed in their 225-ton-per-day plant at Sterlington, La., the reaction takes place in a packed tubular reactor with no water added other than that contained in the nitric acid. The packing serves as a reaction and condensation surface.

Molten ammonium nitrate flows out the bottom of a reactor and through a separator which removes steam and any unreacted ammonia. The ammonium nitrate drops through a packed column with a heated air stream passing countercurrently. It serves to reduce moisture to almost any desired level. The molten salt is then flaked on a water-cooled stainless steel belt, ground, screened, coated, and bagged.

The advantage from the elimination of the need for evaporating large quantities of liquid is obvious. The flaking step also has attractions that make it preferred over prilling, crystallizing, or graining. The process is considered to be safe because of the blanket of steam which covers the molten salt during its stay in the reactor-separator and because of the very small amount of material held up in the equipment at any one time. As an extra safety precaution, the reactor and separator are placed in a concrete barricade.

#### STENGEL PROCESS AMMONIUM NITRATE

*Albert S. Hester, J. J. Dorsey, Jr., and J. T. Kaufman*

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#### PILOT PLANT

#### Economical Vinyl Chloride Process

Gases from the Schoch, Sachse, and Wulff processes contain about 10% acetylene. If vinyl chloride can be made directly from these gases without the prior separation of acetylene, the cost of vinyl chloride should be reduced appreciably.

Chlorine can be introduced directly into the dilute gas mixture where it reacts with hydrogen to form hydrogen chloride. In this exothermic reaction the acetylene content of the gas is increased by about 10%. At 150° C. over a mercuric chloride-barium chloride catalyst the hydrogen chloride added readily to the acetylene to produce vinyl chloride with yields four to five times those reported in German plants. Sulfur compounds and higher acetylenes must be removed as they act as catalyst poisons.

This new process has several advantages over the usual process. Acetylene produced from hydrocarbons is cheaper than carbide acetylene, the vinyl chloride can be removed from the product gas more readily than acetylene, and the dilute gas prevents localized heating which destroys catalyst activity. A pure vinyl chloride that passes polymerization tests is readily made by this process.

#### VINYL CHLORIDE FROM DILUTE ACETYLENE GASES

*R. Emerson Lynn, Jr., and Kenneth A. Kobe*

University of Texas, Austin, Tex.

I&EC—April 1954



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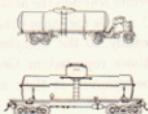
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## GENERAL ENGINEERING, DESIGN, AND PROCESS DEVELOPMENT ARTICLES

## Production of High Purity Tantalum and Niobium

I&amp;EC—April 1954

New methods for producing pure tantalum and niobium from ores and synthetic mixtures have been proposed periodically, but they have not proved satisfactory for more than a few specified conditions. The need for large amounts of high purity tantalum and niobium has increased tremendously in recent years. The development of liquid-liquid extraction techniques for the separation of other metal systems has offered a new approach to this problem.

Tantalum and niobium products of greater than 99% purity have been obtained when the feed mixture was contacted with methyl isobutyl ketone in a perforated plate pulse column. The process involves the preferential extraction of tantalum by the ketone from a hydrofluoric acid solution. A subsequent addition of hydrochloric acid to the aqueous raffinate and recontacting with fresh ketone selectively extracts the niobium from the remaining impurities. The process can be accomplished in either single-stage mixer-settler equipment or in the multistage pulse column.

This new extraction process accomplishes the separation of high purity metals from the starting material, regardless of the total metal content or the relative tantalum to niobium ratio of the ores, thereby allowing for the use of large stocks of low grade ores, or ores in which the metal ratio is below a marketable value.

## SEPARATION OF TANTALUM AND NIOBIUM BY LIQUID-LIQUID EXTRACTION

J. R. Werning, K. B. Higbie, J. T. Grace, B. F. Speece, and H. L. Gilbert ..... 644  
U. S. Bureau of Mines, Albany, Ore.

## Economic Syntheses of Substituted Styrenes

I&amp;EC—April 1954

The economic syntheses of methylstyrene and dimethylstyrene are desired to satisfy the demands of the plastics industry for additional vinyl aromatic sources and also to produce thermoplastic resins having physical properties equivalent to, or better than, styrene.

A new method of preparing ring substituted methylstyrenes is the vapor-phase cracking of unsymmetrical diarylethylenes using clay catalysts at exceedingly short contact times. The diarylethylenes are readily synthesized from toluene or xylene with either acetylene or acetaldehyde, using an appropriate acid catalyst system. The heat distortion temperature of methylstyrene and dimethylstyrene polymers exceed that of styrene and are therefore promising as replacements for styrene in thermoplastic moldings. In other respects their properties are comparable to styrene.

The utilization of readily available raw materials such as toluene or xylene and acetylene or acetaldehyde for the economic syntheses of methylstyrene and dimethylstyrene is an important contribution toward meeting the future demands of the plastics industry.

## METHYLSYRENES AND DIMETHYLSYRENES FROM DIARYLETHYLENES

J. R. Dixon and K. W. Saunders ..... 652  
Stamford Research Laboratories, American Cyanamid Co., Stamford, Conn.

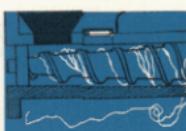
## Analysis of Adiabatic Plastics Extrusion

I&amp;EC—April 1954

The increasing importance of extrusion in the plastics industry has created a great deal of interest in the development of improved extrusion techniques and designs. However, much of this work has had to proceed on a purely empirical basis and it is desirable, for the guidance of future development work, to enlarge the theoretical background of plastics extrusion.

One problem of theoretical interest is to relate the performance of an extruder to its design and operating variables and to the physical properties of the plastic. This paper presents the solution of a special case, the adiabatic operation of melt extruders. By definition, an extruder operates adiabatically when there is no transfer of heat either to or from the plastic—i.e., when all of the heat and pressure energy is generated by mechanical working. In this paper, equations are derived which relate melt temperatures and pressures to distance along the screw channel. With these equations the extrusion rate, the temperature and pressure at the die, and the total power requirements can be calculated.

This analysis shows that with adiabatic operation it is possible to control independently the extrusion rate and temperature without using external heaters and that there are some potential advantages to be



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**Briefs**

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gained by this mode of operation. For example, higher extrusion rates are possible because the rate is not limited by the amount of heat that can be transferred through the barrel. Excessive temperature gradients in the plastic can be avoided since the heat is generated within the plastic and is not transferred to it from a high temperature source.

**THEORY OF ADIABATIC EXTRUDER OPERATION****James M. McKelvey** .....

E. I. du Pont de Nemours &amp; Co., Polychemicals Department, Experimental Station, Wilmington, Del. ....

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**Flowmeter from Pipe Fittings****I&EC—April 1954**

A simple, inexpensive flowmeter, built around a pair of ordinary screwed pipe tees and taking advantage of the ability of one tee to measure the impact pressure of the flowing fluid while the other simultaneously measures the static pressure, is described in this paper.

The meter was built and tested in sizes ranging from  $\frac{1}{4}$  to  $1\frac{1}{2}$  inches. Water was the fluid studied. A standard meter configuration was adopted, and differential coefficients, expressed as the number of velocity heads, were determined for the standard meter. The effect of modification of each component of the meter was systematically studied. The effect of Reynolds number on the differential was also investigated.

By adhering to the flowmeter specifications as developed, and by employing the coefficients obtained, fluid flow measurements with an accuracy of a few per cent can be made with this meter.

**PIPE TEE FLUID FLOWMETER****Lee Garwin and Spencer H. Landes** .....

Oklahoma A. &amp; M. College, Stillwater, Okla. ....

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**Consecutive versus Parallel Reactions****I&EC—April 1954**

Examples of coupled reversible reactions are commonly encountered in practice, both in the form of consecutive reactions and of parallel reactions. This paper presents solutions to the first-order rate equations for these complex reactions in a form sufficiently simplified to permit ready application.

By the use of dimensionless ratios and groups, all four reaction rate constants were eliminated from the general differential equations for coupled, first-order, reversible reactions. Solutions were then obtained for the two most important cases—consecutive reactions and parallel reactions—and techniques of application to practical kinetic studies were developed. Certain conditions under which the kinetics of parallel reversible reactions may resemble closely those of consecutive reactions are described and a means of distinguishing between the two cases is presented.

These results make possible a direct approach to the kinetics of certain complex reactions which previously could be studied only with difficulty.

**KINETICS OF COUPLED REVERSIBLE REACTIONS****Gerard N. Vriens** .....

American Cyanamid Co., Bound Brook, N. J. ....

669

**18TH ANNUAL UNIT PROCESSES SYMPOSIUM****I&EC—April 1954**

Unit process technology is advanced by contributions reporting the synthesis of a reasonably priced diphenylmercury; superiority of the recycle-type reactor over a single column reactor in the production of ethylene chlorhydrin; various oxygen and halogen combinations for nitrating propane to lower weight nitro-paraffins; factors affecting the mononitration of 2-methylnaphthalene; the alkylation of isobutane with propylene by means of a free-radical chain mechanism at high pressure; and the use of a rare earth oxide or a manganese oxide—bismuth oxide catalyst in the oxidation of ammonia at low temperatures. A 6-ton-per-day pilot plant pyrolysis of oil shale is described. Suspension copolymerization of vinyl chloride and vinyl acetate confirmed the view that this is bulk polymerization within suspended droplets. Optimum conditions are divulged for the mononitration of *m*-xylene, thus completing the information on the mononitration of the three xylenes. Actual plant operations are described for the continuous production of nitrotoluene and also hexamethylenetetramine in Germany.

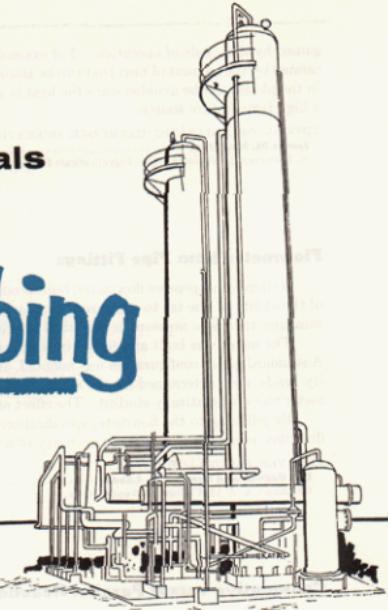
**UNIT PROCESSES IN CHEMICAL ENGINEERING** .....

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**GENERAL TECHNICAL AND SCIENTIFIC ARTICLES****Use of Plastic Films for Gas Separation**

I&amp;EC—April 1954

In order to evaluate the possible applications of gas separation by diffusion through plastic films, it is necessary to have quantitative data on this operation. In addition, the need exists for equipment designs which will permit economic operation.

Data on permeability of four plastic membranes to seven gases and vapors were obtained over a range of temperatures. Separation data are reported for one binary mixture and six multicomponent mixtures. A model of a diffusion cell was built and tested, which has certain advantages over older types.

The process has possible commercial applications, and the data may be used in design calculations.

## SEPARATION OF GASES BY PLASTIC MEMBRANES

**David W. Brubaker, Karl Kammermeyer, and James O. Osburn** ..... 733  
Chemical Engineering, State University of Iowa, Iowa City, Iowa

**Ultrasound in Plastics Processing**

I&amp;EC—April 1954

Although extensive information is available on the effect of ultrasound upon the solutions of high polymers, relatively little is known about its effect on the pure melts of these polymers. This work was undertaken to study the potential importance of ultrasound in the processing of thermoplastics.

In the ranges of temperatures and frequencies investigated, ultrasonic energy had no depolymerizing effect upon thermoplastic melts. Absorption of ultrasonic energy was found to depend on the frequency of the sound waves, and could rapidly increase the temperatures of the melts. It was noted that ultrasound could cause molecular orientation in molten polymer.



Ultrasound may be considered for applications in plastics processing. It could prove useful for rapid and local internal heating of both polar and nonpolar plastics. Thus, it provides a new means for welding and heat-sealing these materials, and may aid in other forming operations. The fact that ultrasound may cause molecular orientation without physical deformation may also be of practical interest.

## EFFECT OF ULTRASOUND ON THERMOPLASTIC MELTS

**Ernest C. Bernhardt** ..... 742  
Institut für Technische Physik, Technische Hochschule, Darmstadt, Germany

**Stability Limits of Shielded Flames**

I&amp;EC—April 1954

With unshielded Bunsen burners only flash back and lean-type blowoff of flames can be observed. A Smithells tube affixed to the burner or an annular stream of inert gas around the burner port permits the observation of both rich-type and lean-type blowoff as well as flash back. As ambient air is excluded in many burner applications, it was believed desirable to evaluate the stability limits of flames under similar conditions by using air-excluding devices to shield the flame.

Flame-stability experiments with a variety of hydrocarbons and with hydrogen sulfide on shielded Bunsen burners show that consistent data can be obtained for lean and rich blowoff and for flash-back limits. The diameter of the Smithells tube is critical for rich ethylene flames at low burner-jet velocities. The data from experiments on a Bunsen-Smithells burner with binary fuel mixtures of propane and ethylene fit an empirical equation which permits prediction of the behavior of fuel mixtures from the behavior of the components or deduction of the stability limits of the components if the behavior of a series of mixtures is known. Stability studies with isobutane-ethylene flames on Bunsen-Smithells and on inert-shielded burners show that at lean blowoff and at flash back, behavior of mixtures is predictable by the empirical equation based on additivity of stability limits. At rich blowoff the experimental data deviate slightly from the requirements of the empirical equation, indicating the presence of an inhibiting effect.

This work should lead to a better understanding of the behavior of multicomponent fuel mixtures.

## FLAME-STABILITY STUDIES ON SHIELDED BUNSEN BURNERS

**Philip F. Kutz** ..... 746  
Battelle Memorial Institute, Columbus 1, Ohio

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**Vapor-Liquid Equilibrium Data on Xylenes**

I&amp;EC—April 1954

The economical separation of *p*- and *m*-xylenes has presented a difficult problem. The possibility of separation by extractive distillation was explored by the determination of vapor-liquid equilibrium data. These experimental data have been used to illustrate the validity of different thermodynamic correlations.

Two different experimental approaches have been used in the determination of vapor-liquid equilibrium data: determination of relative volatility of two xylene isomers directly in the presence of an optimum amount of a solvent, and determination of vapor-liquid equilibrium data of each isomer with a solvent in the complete range of concentration. Although some solvents show an effect upon the relative volatility of xylene isomers, the method of extractive distillation is less likely to be economical. Infrared radiation did not significantly affect the vapor-liquid equilibrium compositions of a boiling mixture.

Vapor-liquid equilibrium data obtained by the second method were treated by the methods of correlation proposed by Wohl, Redlich and Kister, and Gil mont *et al.* The constants in the correlation equations were computed, and the calculated equilibrium data compared well with the experimental results. The formation of xylene azeotropes with 2-ethyl hexaldehyde was noted. The experimental data check well with the values predicted by Meissner's method.

**VAPOR-LIQUID EQUILIBRIUM. *m* AND *p*-XYLEMES IN DIFFERENT SOLVENTS**

Ju Chin Chu and O. P. Kharbanda; R. F. Brooks and S. L. Wang..... 754  
Polytechnic Institute of Brooklyn, Brooklyn, N. Y.; Washington University, St. Louis, Mo.

**Thermodynamic Criterion for Vapor Pressure Equations**

I&amp;EC—April 1954

Equations representing the vapor pressure of a pure substance are written in a variety of forms, some of which give unsatisfactory values for the heat of vaporization as a function of temperature and are known to extrapolate vapor pressures erroneously. Studies were undertaken to find the suitability of various equations for use in various limits of reduced temperature.

The form of the curve for  $\Delta H/\Delta Z$  as a function of temperature has been found to be the same for a variety of substances, and to have a shape requiring certain mathematical characteristics of the vapor pressure equation.

This provides a qualitative test for the various vapor pressure equations, and a basis for a generalized vapor pressure equation. It is also recommended for testing the consistency and smoothness of data for saturation properties.

**FORM OF A WIDE-RANGE VAPOR PRESSURE EQUATION**

Warden Wareing ..... 762  
Shell Development Co., Emeryville, Calif.

**Relation of Specific Surface to Explosibility of Dusts**

I&amp;EC—April 1954

An attempt was made to obtain a better understanding of the fundamental nature of dust explosions. The use of three different sugars made possible a study of dust explosions as affected by the number of carbon atoms in the molecule, the specific surface of the material, and the concentration of the explosive dust cloud.

The minimum explosive concentration, optimum explosive concentration, maximum explosion pressure, and rates of pressure increase are functions of the specific surface and number of carbon atoms in the molecule. There exists for any particular material a "critical point" which is characterized by a minimum optimum explosion concentration and an absolute maximum explosion pressure. An empirical relationship between explosion pressure and time is presented.

The use of specific surface rather than particle size as a measure of explosibility offers a new approach to the study of dust explosions, which can be utilized to express explosibility of dusts by the simple equation of the logistic.

**EXPLOSIVE PROPERTIES OF SUGAR DUSTS**

R. L. Meek and J. M. DallaValle ..... 763  
Georgia Institute of Technology, Atlanta, Ga.



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## Water-Soluble Water Repellent

Following the discovery that paper could be rendered water repellent by treatment with chromyl chloride vapor, the reaction products of chromyl chloride with certain organic materials were investigated with the objective of finding a hydrophobic agent that could be applied from aqueous solution.

This led to the development of stearato chromic chloride, a complex compound in which stearic acid is combined with basic trivalent chromic cations to form a water-soluble, surface-active product which is strongly adsorbed from aqueous solution by negatively charged surfaces, such as cellulose, proteins, and glass. The adsorbed film is hydrophobic, and when dried, the complex becomes insoluble in water. In the monomolecular adsorbed layer, each stearate group covers an area of 33 square A. The insolubilization of the complex appears to be due to hydrolysis of the chloride and polymerization of the resulting basic chromic cations.

Stearato chromic chloride is currently being employed as a hydrophobing agent for paper, felt, and glass fiber products, and as an antisticking agent where adhesion to surfaces is to be avoided.

### STEARATO CHROMIC CHLORIDE

R. K. Iler ..... 766  
Grasselli Chemicals Department, Experimental Station, E. I. du Pont de Nemours & Co., Inc., Wilmington 98, Del.

## Paint and Printing Ink Studies

I&amp;EC—April 1954

Loss of drying on aging has been a serious problem in the paint and printing ink industries for many years. This study was undertaken to learn the nature of this difficulty and means of avoiding it. Alumina hydrate lakes were studied as a group, since a poll of the printing ink industry showed them to be the most serious offenders.

Studies of loss of drying on aging of printing inks containing alumina hydrate lake pigments have shown that the loss increases with increasing acidity and moisture content of the pigments. It is also severe in the presence of Turkey-red oil and lakes containing excessive dye. The experimental data indicate that this loss is due to the adsorption of the cobalt onto the pigments. Two adsorption mechanisms are suggested for different lake pigments: One involves the extraction of cobalt into the acidic water film surrounding the pigment particles and the other involves reaction of the cobalt with oil-soluble organic sulfonic acids where these are present.

A cobalt adsorption test has been developed to distinguish those alumina hydrate lakes which are liable to produce loss of drying on aging. These pigments can either be avoided or corrective agents can be used.

### LOSS OF DRYING ON AGING. PROPERTIES OF HYDRATE LAKES

Donald M. Nace and William C. Walker ..... 769  
National Printing Ink Research Institute, Lehigh University, Bethlehem, Pa.

## Modified Casein Stabilizers for Latex Paint

I&amp;EC—April 1954

The reaction between protein stabilizers and polyvalent cations in a latex paint usually causes a great viscosity increase and makes the paint unusable. Proteins modified to be insensitive to polyvalent cations could help prevent this. Modification of a protein could affect its capacity to make stable emulsions, thereby improving freeze-thaw stability.

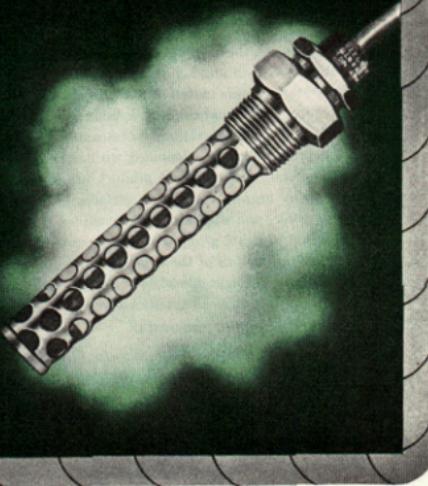
Enzymatic digestion or chemical modification of casein or alpha-soybean protein increases the calcium compatibility of these proteins. Latex paint formulations made with such modified proteins have good viscosity stability. When an enzyme-digested casein is used as the stabilizer, the paint also has good freeze-thaw stability. By using suitably modified proteins for stabilizers, latex paints with good viscosity stability can be formulated with hard water or even calcium-extended pigments.

An enzyme-modified casein or alpha-soybean protein will permit formulation of a latex paint of good freeze-thaw stability, which will not require heated cars for shipment or storage.

### MODIFIED PROTEINS FOR STABILIZING LATEX PAINTS

Kenneth S. Ronai and Samuel M. Weisberg ..... 774  
National Dairy Research Laboratories, Inc., Oakdale, L. I., N. Y.

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WORKS**

The Dewcel element is a thermometer bulb (liquid-filled or electric-resistance type) jacketed with lithium-chloride-impregnated woven glass tape. Over this are wound two spaced gold or silver wires connected to an AC source. The lithium chloride absorbs moisture, allowing current to flow, generating heat, and raising the temperature. Equilibrium temperature is reached when vapor-pressure of the moist salt exactly balances that of the surrounding air or gas. The System translates this temperature into direct readings of dew point.

Thus, Foxboro Dew Point Instruments give direct readings or control of dew point from -50°F. to 142°F. at working temperatures from -40°F. to 220°F. Readings easily converted to absolute or relative humidity.

\*Trade Mark

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## Mold Prevention on Optical Glass

Binoculars were treated with a foil containing radium, a radioactive lacquer, and polonium plating and exposed in a chamber which simulated tropical temperatures and relative humidities.

At the end of the exposure, the optical glass of the telescopes treated with polonium was free from mold growth. The radium foil prevented mold growth in only half of the telescopes tested. The radioactive lacquer was ineffective as a fungistat. However, the amount of radioactivity present in the radium-treated samples was less than that in the polonium-treated binoculars. The Porro prisms were the most difficult optical elements to fungus-proof. The binoculars treated with radioactive foil and polonium showed droplet formation on the internal optical glass elements. The metallic parts plated with polonium were heavily corroded.

Ionizing radiation from radium and polonium may be used to prevent mold growth on optical glass, but the amounts of radioactive materials required to protect a binocular completely against fungus growth are considered too hazardous to personnel. Radioactive materials may be used for specialized optical instruments which cannot be fungus-proofed by other means.

### RADIOACTIVE MATERIALS IN PREVENTION OF MOLD GROWTH IN OPTICAL INSTRUMENTS

*Sigmund Beck and Leonard Teitel*.....

Frankford Arsenal, Philadelphia, Pa.

778

## Effect of Heat on Gum Rosin

I&EC—April 1954

Practically all gum rosin is processed at temperatures between 155° and 290° C. by both the original oleoresin distiller and the final derivative manufacturer. This study was undertaken to determine the changes taking place in this temperature range.

These changes fall into two categories: Moderate heating for short periods of time brings about an increase in softening point, a more negative optical rotation, a slight decrease in acid number, and an increase in its tendency to crystallize; higher temperatures or prolonged heating at moderate temperatures cause an opposite trend in most of these changes—i.e., a decrease in softening point, a more positive optical rotation, a greater decrease in acid number, a decrease in the tendency to crystallize, and an improvement in color grade.

The results of this study should permit a more accurate prediction of the changes likely to occur in processing at temperatures in the range covered.

### THERMAL ISOMERIZATION OF GUM ROSIN

*J. S. Stinson and Ray V. Lawrence*.....

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784

## Catalytic Production of Dioxolanes and Trioxocanes

I&EC—April 1954

Cyclic acetals of ethylene glycol and commercially available aldehydes have been prepared by three procedures, using cation exchange resins as catalysts in conversions of 60 to 95%. The yields are essentially quantitative, in that no by-products were detected and the starting materials can be recycled. Cyclohexanone, the most reactive ketone, gave the dioxolane with a 57% conversion. The formation of ketals is much more difficult and the conversions are low, but good yields can be obtained by starting with ethylene glycol and ethyl orthoformate.

Formaldehyde reacts with diethylene glycol in the presence of sulfonic acid-type cation exchange resins to form an eight-membered cyclic acetal, 1,3,6-trioxocane. This is stable in neutral or slightly alkaline conditions but polymerizes in the presence of trace amounts of acids to give a water-soluble wax having a melting point below body temperature. Depolymerization occurs on heating, particularly in the presence of trace amounts of acids. Other trioxocanes have been prepared from diethylene glycol and isobutyraldehyde, dichloroacetaldehyde, and 2-ethylhexaldehyde. The trioxocanes prepared from 2-ethylhexaldehyde exist as viscous sirups only in polymeric forms, which break down to the monomer on heating, especially in the presence of trace amounts of acids, and repolymerize as the vapors condense. An equilibrium apparently exists between the monomers and the resulting polymers. Aldehydes having two  $\alpha$ -hydrogens do not form trioxocanes in appreciable yields, but those having only one  $\alpha$ -hydrogen form trioxocanes in fair yield.

Cation exchange resins of the sulfonic acid type are excellent catalysts for the formation of dioxolanes and some trioxocanes and offer several advantages over conventional catalysts. These catalysts may be used in packed columns, permitting continuous processes for the formation of acetals.

### CATALYSIS WITH CATION-EXCHANGE RESINS

*Melvin J. Astle, Joel A. Zaslawsky, and Paul G. Lafatys*.....

Case Institute of Technology, Cleveland 6, Ohio

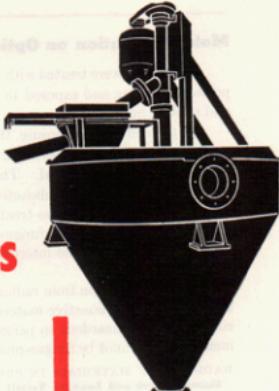
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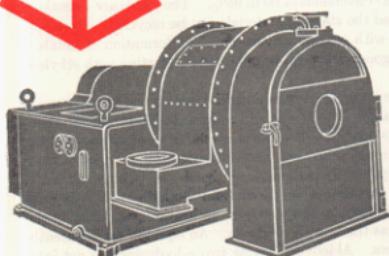
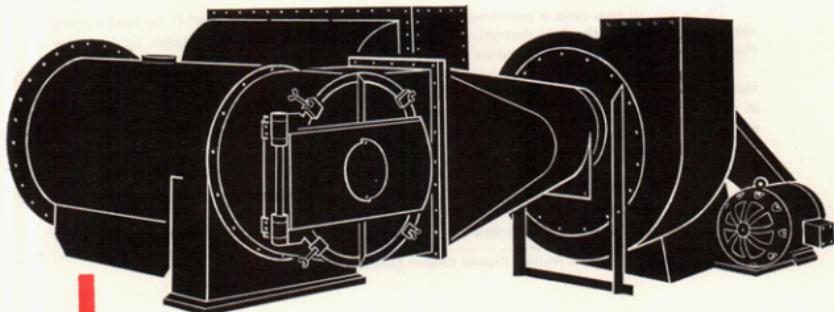
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Vol. 46, No. 4

## Pyridine as a Water Pollutant

As part of a series of investigations of the pollutive characteristics of specific organic materials, pyridine and several related bases were studied.

These materials proved to be subject to complete destruction by microorganisms found in polluted surface waters. The effective microorganisms apparently are relatively scarce in sewage but somewhat more numerous in polluted surface waters. Hence, a biochemical oxygen demand assay, made with a sewage inoculum not providing appropriate pyridine-using seed, will not reveal the oxygen usage which the compound will cause in a polluted surface water.

Because the materials studied are believed to be similar in their pollutive characteristics to many materials found in process wastes, it is suggested that a conventional biochemical oxygen demand test may not reveal the total material subject to biological attack in the receiving stream which the process waste contains.

### PERSISTENCE OF PYRIDINE BASES IN POLLUTED WATER

**M. B. Ettlinger, R. J. Lishka, and R. C. Kramer.....** 791  
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## Oil- and Freeze-Resistant Elastomers

The aircraft and automotive industries require an elastomer that is flexible at low temperatures and maintains its dimensions and physical properties after prolonged contact with various fuels and oils. Because existing polymers do not meet these exacting requirements, a large number of experimental polymers were made by emulsion polymerization in a pilot plant and their compounds examined. Copolymers were made from butadiene and various amounts of styrene, acrylonitrile, methacrylonitrile, alkyl acrylates, vinylidene chloride, diethyl fumarate, diethyl chloromaleate, and unsaturated ketones; some tripolymers of these monomers were also prepared and evaluated.

Improvements in oil and solvent resistance of polybutadiene realized by copolymerization with other monomers were invariably accompanied by a decrease in the excellent freeze resistance of the polybutadiene.

None of the compounds had both the desired oil and freeze resistance, but the data may offer leads for this and other purposes.

### EVALUATION OF DIENE-TYPE ELASTOMERS

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## Butadiene Copolymers with Acrylonitrile

This research was undertaken to learn the effects of incorporating pendant alkyl and nitrile groups into polybutadiene molecule.

This was accomplished by copolymerizing butadiene with  $\alpha$ -methyl-,  $\alpha$ -ethyl-, and  $\alpha$ -n-pentylacrylonitrile in weight ratios of 95 to 5, 85 to 15, and 75 to 25. The stress-strain properties, Gehman low-temperature properties, and oil resistance of the compounded and vulcanized copolymers were measured. The freeze points of the cured polymers are related to the length of the alkyl group and to the total nitrogen content of the polymer. The oil resistance of the cured polymer is decreased by increasing the chain length of the alkyl substituents.

The alkylacrylonitriles do not appear to represent improvements over acrylonitrile, as exemplified by Perbunan 18, as far as the balance between low-temperature and solvent-resistant properties is concerned. More practical work is needed to make a final answer certain.

### COPOLYMERS OF $\alpha$ -ALKYLACRYLONITRILES WITH 1,3-BUTADIENE

**C. S. Marvel and R. Thomas Stiehl; W. K. Taft and B. G. Labbe.....** 804  
University of Illinois, Urbana, Ill.; Government Laboratories, University of Akron, Akron, Ohio

## Vulcanization Accelerators

Although sulfenamides derived from mercaptobenzothiazole possess particular merit because of their delayed action properties, the increasing acceptance of reinforcing furnace blacks has intensified scorch problems for which these conventional accelerators offer only a partial solution. Accordingly, a search was

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made for more delayed action accelerators in the class of 4-alkyl thiazoles. This class was selected for study because it has remained essentially undeveloped, whereas derivatives of mercaptobenzothiazole have been extensively evaluated.

Preliminary evaluation showed that 5-carbomethoxy- or 5-carbethoxy-4-methyl-2-thiazolesulfenamides in which the amido group was morpholinyl or *tert*-butylamido were stable compounds which possessed more delayed action than commercial delayed action accelerators and were equal in accelerating strength. More extensive evaluation confirmed that in Hevea HAF black tread base the experimental accelerators possessed more delayed action. Stocks containing these materials exhibited moduli, tensile, and heat build-up equivalent to the control, lower permanent set, and, with one exception, less static compression. In GR-S 1500 HAF black tread the results were similar. However, the cut growth was excessive in either base and aged flexings were deficient in Hevea.

More delayed action accelerators for use with the new furnace blacks are indicated. The continually rising cost of aromatics may render the 4-alkyl thiazoles attractive from the economic standpoint.

VULCANIZATION CHARACTERISTICS OF A SERIES OF 4-METHYL-5-SUBSTITUTED 2-THIAZOLETHIOLS AND THEIR DERIVATIVES

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**Mechanism of Antioxidant Action**

I&EC—April 1954

Data presented in a previous communication on the effect of oxygen concentration on rate of oxidation of certain amine-inhibited black vulcanizates were not consistent with a simple chain-stopping mechanism of antioxidant action. The present study provides additional information and includes typical antioxidants of both amine and phenolic types.

Studies at different partial pressures of oxygen in combination with nitrogen show that the variation of rate of oxygen absorption in the constant-rate stage with oxygen concentration involves both a square-root and a first-power term, the latter being negligible in some cases. Rate data for all stocks studied, including variations of accelerator type, carbon loading, antioxidant type, and antioxidant concentration, are correlated on the basis of the same rate equation.

A mechanism of oxidation and antioxidant action is postulated, consistent with the observed behavior, which indicates that the antioxidant may function in at least four ways, some of which are beneficial, while others are harmful. Many anomalous facts, such as optimum antioxidant concentrations, catalysis of light-activated oxidations by some antioxidants, the difference in effectiveness of phenols and amines in gum and black stocks, and the role of carbon black in the oxidation, are capable of plausible explanation on the basis of this postulated mechanism of antioxidant action.

OXIDATION AND ANTIOXIDANT ACTION IN RUBBER VULCANIZATES

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**Alkali Lignin and Humic Acid for Reinforcing Rubber**

I&EC—April 1954

The incorporation of alkali lignin into rubber compounds by coprecipitation from latex has previously been shown to yield vulcanizates with tensile strengths, hardness, and tear resistance comparable with those of black-reinforced compounds. The properties of the vulcanizate depend, however, on the process of coprecipitation used.

Treatment of the lignin with hexamethylenetetramine in the presence of the rubber yields a vulcanizate in which tensile properties and hardness comparable with those of a black-reinforced compound are combined with the resilience of a pure gum compound. Lignosulfonic acid resulting from the acid wood pulp process has not shown the same promise as alkali lignin as a reinforcing filler in rubber. By an ion exchange technique it may be modified to yield results similar to those given by alkali lignin.

A material chemically related to lignin and potentially abundant is humic acid, obtained by the alkaline extraction of weathered outcrop coal. This material behaves in rubber much like alkali lignin, yielding products with the same general level of properties.

COMPOUNDING NATURAL RUBBER WITH LIGNIN AND HUMIC ACID

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