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### Reaction methods for producing nitrogenous phosphoryl compounds that are in situ fluid compositions

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#### Abstract

Methods are disclosed for the manufacture of nitrogenous phosphoryl compounds as fluid compositions utilizing a reaction medium comprising one or more aprotic organic liquids that are not reactive with one or more phosphoryl chlorides functional groups and optionally one or more non-aqueous organic liquids as processing aides that result in fluid products for delivering the resulting nitrogenous phosphoryl compounds to the surface of a substrate. Methods are disclosed that eliminate the need for one or more of the following: 1) purifying of one or more nitrogenous phosphoryl compounds, 2) stripping of volatile organic solvents, 3) removal of inorganic by-products, 4) generating solid nitrogenous phosphoryl compounds, 5) packaging one or more solid nitrogenous compounds, and 6) dissolving the solid nitrogenous phosphoryl compounds into a liquid delivery system for application to the surface of a substrate. Methods are disclosed that lower the overall manufacturing cost, wherein said fluid nitrogenous phosphoryl compositions comprise liquid solutions, fluid colloids, and fluid suspensions. Fluidized nitrogenous phosphoryl compounds are applied onto fertilizer solid surfaces using simple blending equipment that commingle the composition with solid granules allowing farmers and small co-ops access to high tech, state of the art fertilizers at lower cost.

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5883297	12/1998	Sulzer et al.	N/A	N/A
5955630	12/1998	Cheng et al.	N/A	N/A
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## Background/Summary

(1) The present application claims priority under 35 USC 119(e) to U.S. Provisional Application 63/161,064 filed Mar. 15, 2021, the entire contents of which are incorporated by reference.

### FIELD OF THE INVENTION

(1) The invention relates to low cost processes for the manufacture of nitrogenous phosphoryl compounds that are in situ fluid compositions by elimination of the costly process steps that exist with the current state of art. Said low cost processes result in ready to use fluid products that can be applied directly to a substrate. Of particular interest are nitrogenous phosphoryl compounds that possess biologically active properties.

### BACKGROUND OF THE INVENTION

(2) Current methods to make liquid nitrogenous phosphoryl compositions are based on one or more steps selected from the group consisting of: 1. reacting phosphoryl trichloride and/or thiophosphoryl trichloride with one or more alkylamines utilizing a liquid reaction medium for the preparation of monoalkylamide phosphoryl dichlorides and/or monoalkylamide thiophosphoryl dichlorides and the resulting by-product alkyl ammonium chlorides, wherein said reaction medium comprises one or more volatile, non-reactive, aprotic organic liquids, a. if a hydrochloric acid absorber is not utilized to keep the one or more alkylamines from forming the corresponding one or more alkylammonium chloride salts, then recovery of the alkylamine from the by-product, alkyl ammonium chloride, is required to free the one or more alkylamines to react with phosphoryl chloride functional groups, 2. reacting the remaining one or more alkylamide phosphoryl dichloride and/or one or more alkylamide thiophosphoryl dichloride with ammonia or ammonium generating compounds to form the desired nitrogenous phosphoryl compounds, 3. separating inorganic chloride by-products from the desired nitrogenous phosphoryl compounds, 4. removing the volatile, non-reactive aprotic organic liquid and HCl absorbent (if a tertiary amine used), 5. purifying nitrogenous phosphoryl compounds and isolating the solid, waxy, temperature sensitive, sticky nitrogenous phosphoryl compounds to generate a flowable powder composition, 6. packaging and transporting the flowable powder composition comprising nitrogenous phosphoryl compounds, 7. dissolving the flowable powder composition comprising nitrogenous phosphoryl compounds within a non-aqueous organic liquid delivery system capable of imparting a continuous liquid solution coating comprising nitrogenous phosphoryl compounds onto the surface of substrates.

(3) A generic flow diagram for the process to yield liquid nitrogenous phosphoryl compositions is shown in Scheme 1:

(4) ##STR00001## wherein: a) X<sup>sup.2</sup> is one or more members selected from the group consisting of i) P=O and ii) P=S, wherein the nitrogen of the nitrogenous portion of said compounds are bonded to the "P", b) Z<sup>sup.e</sup> is H (hydrogen) R<sup>sup.19</sup> is one or more alkyl radicals selected from the group consisting of i) —C<sub>sub.1</sub>H<sub>sub.3</sub>, —C<sub>sub.2</sub>H<sub>sub.5</sub>, —C<sub>sub.3</sub>H<sub>sub.7</sub>, and —C<sub>sub.4</sub>H<sub>sub.9</sub>.

(5) The current processing technologies yield high purity nitrogenous phosphoryl compounds, wherein the nitrogenous phosphoryl compounds' performance is adequate at low levels application to plant growth medium. However, steps to drive the synthesis to a higher purity, flowable powder composition and consequently dissolve the powder into a solvating delivery system carries a cost burden that limits its utility on low cost substrates such as urea.

(6) Patent No. GB898302, Hoechst Ag, proposed a method for producing nitrogenous phosphoryl compounds by reacting the N-substituted phosphoric or thiophosphoric acid amide dichloride with ammonia using chloroform as an aprotic, non-reactive organic liquid. The process included separating the ammonium chloride formed and distilling off the chloroform. The crude product was

obtained as residue and could be purified by recrystallization in organic liquids such as benzene. The nitrogenous phosphoryl compounds generated were suggested to be used as components in fixing agents for fixing pigments in a wear-resistant manner on fibrous material. While this process will generate a composition containing the desired nitrogenous phosphoryl compounds, the utilization of chloroform and benzene as processing organic liquids have health and environmental issues, and the resulting residue has to be purified and then dispersed in an organic liquid to liquefy for ease of use.

(7) A series of patents by Kolc and Swerdloff (U.S. Pat. Nos. 4,517,003, 4,517,004, and 4,530,714) propose generating several nitrogenous phosphoryl compounds using generic laboratory methods where temperatures and pressures are not critical with suggested temperature ranges of  $-80^{\circ}\text{C}$ . to  $200^{\circ}\text{C}$ ., wherein the exact proportions of the reactants are not critical, and the order of reaction is not critical. They suggested the potential inclusion of inert reaction organic liquids such as ethyl ether, carbon tetrachloride, methylene chloride, benzene, dioxane, toluene, xylene, tetrahydrofuran, methyl sulfoxide (DMSO), and dimethylformamide. These types of reactions produce a hydrogen chloride by-product, which will react with amines producing amine chloride salts. If this hydrogen chloride by-product is not absorbed or removed, the formation of the amine chloride salts will interfere with the further reaction of the amines with the phosphoro-chlorides. Kolc and Swerdloff suggest using hydrogen chloride acceptors which are non-reactive with phosphoro-chlorides to allow the desired amine to remain free to participate in the reaction. These hydrogen chloride acceptors can form precipitates in the Kolc and Swerdloff mixtures, which they suggest can be removed by such conventional procedures as extraction, filtration, or centrifugation. The reaction methods are small-scale laboratory experiments designed to yield a composition comprising the desired compounds, wherein the desired compounds can be at least partially separated by such conventional procedures as evaporation and be at least partially purified by conventional procedures such as distillation and extraction, for the purpose of testing the compound's biological activity. However, these reaction methodologies are poorly suited for commercial industrial processes. These methods work well in a laboratory where poor reaction controls can be overcome with purification steps to yield the desired nitrogenous phosphoryl compounds, but they prove to be difficult to be instituted in an industrial manufacturing environment due to high processing cost, low yields, and the hazards associated with the recommended organic liquids, such as flammability and toxicity. More dangerously, two of the suggested organic liquids enumerated as being non-reactive reaction mediums, (dimethyl sulfoxide and dimethylformamide) are in fact known to be phosphoryl chloride reactive organic liquids in a highly exothermic matter.

(8) Sulzer (U.S. Pat. No. 5,770,771) describes a continuous process for the preparation of hydrocarbylthiophosphoric triamides where ammonium and N-hydrocarbylammoniothiophosphoryl dichloride are mixed in a reaction chamber in a ratio of 16:1, producing a reaction mixture comprising hydrocarbylthiophosphoric triamide and a trialkylamine (HCl absorber). The ammonium chloride co-product formed is kept in solution in the ammonium which has been added in large excess. The residence time of the reaction mixture in the reactor is from 1 to 10 minutes. The product of interest is separated off by means of distillation. Although not stated, the pressure requirement for the reaction vessel to contain the 11 mole excess of ammonia would be 3700 to 4300 psi. This high pressure requirement necessitates the utilization of high pressure specialty equipment and safety equipment resulting in significantly higher costs to produce the hydrocarbylthiophosphoric triamides.

(9) Sulzer (U.S. Pat. No. 5,872,293) teaches the use of at least 16-fold molar excess of ammonia during the ammonia reaction to form the desired nitrogenous phosphoryl compounds. The ammonia is present in a sufficient excess amount relative to the ammonium chloride to form a separate liquid phase in which the ammonium chloride solids dissolve. The ammonium chloride liquid phase is separated from the remainder of the mixture comprising at least one inert liquid organic solvent, a tertiary amine, and the desired nitrogenous phosphoryl compounds. The pressure

requirement for the reaction vessel to contain the 11 mole excess of ammonia would be 3700 to 4300 psi. This high pressure requirement necessitates the utilization of high pressure specialty equipment and safety equipment resulting in much higher costs to produce the hydrocarbylthiophosphoric triamides.

(10) Sulzer (U.S. Pat. No. 5,883,297) teaches a process that involves separated or recovered nitrogenous phosphoryl compounds continuously introduced as a stream of liquid containing nitrogenous phosphoryl compounds into a wiped film evaporator operating at a temperature range of about 60°–140° C., and at a pressure that avoids solids formation on the heating surface of the wiped film evaporator. The process allows for the continuous collection of the nitrogenous phosphoryl compounds.

(11) Huttenlock et al. (WO 2007/054392) describe a process for the preparation of alkylthiophosphoric triamides from ammonia and alkylthiophosphoryl dichloride by which gaseous ammonia is passed through an amido-dichloride solution and allowed to react. The residence time of the reaction mixture in the reactor is 60 minutes. The nitrogenous phosphoryl compounds of interest are isolated by means of phase separation, precipitated in the phase by lowering the temperature, and purified via a filtration step.

(12) Cheng (U.S. Pat. No. 5,955,630) teaches a method for the recovery of the desired nitrogenous phosphoryl compounds wherein the reaction mixtures are continuously fed to a wiped film evaporator which removes the volatiles (i.e., the reaction medium solvent and trialkylamine) from the desired nitrogenous phosphoryl compounds and process conditions to ensure that there is no solids formation on the heated surfaces of the wiped film evaporator. The invention also reveals the liquid mixture containing desired nitrogenous phosphoryl compounds is continuously fed to the wiped film evaporator wherein hot nitrogen (about 65° C., atmospheric pressure) is passed upwardly in countercurrent flow to the down-flow product stream to further reduce the small residual solvent content of the desired nitrogenous phosphoryl compounds to about a 0.5% solvent maximum.

(13) Kysilka (U.S. Pat. No. 8,513,460) teaches a method to make a purity of at least 98% of the desired nitrogenous phosphoryl compounds by utilizing toluene (aromatic solvent) as Solvent System One for two equivalents of hydrocarbylamine to react with one equivalent of phosphoryl or thiophosphoryl chloride and then react with four equivalents of ammonia. One equivalent of hydrocarbylamine forms the monoamide of phosphoryl or thiophosphoryl chloride, which is soluble in the aromatic phase. The second equivalent of hydrocarbylamine forms the corresponding hydrocarbylammonium chloride which is insoluble in the aromatic phase and is removed by filtration. The resulting hot filtrate is cooled to a temperature range of 0 to 25° C. where the N-(hydrocarbyl)phosphoric or thiophosphoric triamide precipitates. Purity was increased by repeated recrystallization of the crude product from toluene.

(14) Wissemeier (U.S. Pat. No. 8,075,659) discloses the preparation of a mixture of a first primary or secondary amine and a second primary or secondary amine. A method of preparing the preparation utilizing known process technologies of reacting thiophosphoryl chloride first with a mixture and subsequently with ammonia-preparations with improved urease-inhibitory effect which comprise at least two different (thio)phosphoric triamides. The method also relates to urea-comprising fertilizers which comprise these preparations. The invention furthermore relates to a method of preparing these preparations, to the use of these preparations in the fertilization with urea-comprising fertilizers, and to the use of urea-comprising fertilizers which comprise these preparations in agriculture or in horticulture.

(15) Many of these inventions are focused on producing high purity, solid nitrogenous phosphorous compounds. The drive to achieve high purity, solid nitrogenous phosphoryl compounds that are marketed as flowable powders requires difficult processing steps that drive up the manufacturing cost. What is required are low cost, fluid nitrogenous phosphoryl compositions that meets expected levels of performance and compositions that can be applied to substrates utilizing simple blending

equipment. Such an invention would allow farmers and small co-ops access to state of the art fertilizer at a low cost and will lead to mass adoption of treated urea, improving overall food production while lowering the amount of pollution in our water and air.

#### BRIEF SUMMARY OF THE INVENTION

(16) In an embodiment, lower cost methods to make ready-to-use fluid compositions comprising nitrogenous phosphoryl compounds are disclosed. These fluid compositions possess the pre-requisite properties to enable their application onto the surface of granular solids utilizing simple blending equipment that commingle the fluid composition with solid granules, allowing farmers and small co-ops access to high tech, state of the art fertilizers at low cost.

(17) In an embodiment, lower cost methods to make ready-to use compositions comprise one or more process steps selected from the group consisting of a) maintaining the desired nitrogenous phosphoryl compounds in a fluid state throughout its processing b) not utilizing a low boiling point, non-reactive aprotic liquid as the reaction medium that requires its removal from the ready-to-use composition c) not removing the inorganic chloride by-products, d) not using expensive HCl absorbers, e) avoiding the difficulties and cost associated with the solid nitrogenous phosphoryl compounds' purification steps, f) avoiding the steps of collecting and packaging the waxy, sticky, temperature and oxygen sensitive solid nitrogenous phosphoryl compounds as flowable powder and g) avoiding the step of dissolving the nitrogenous phosphoryl compounds powder into a non-aqueous, organo liquid delivery system. In a variation, the disclosed methods result in fluid nitrogenous phosphoryl compositions wherein the nitrogenous phosphoryl compounds are dispersed within a non-aqueous, organo liquid delivery system (NOLDS) as a solution, a colloid or a suspension. In a variation, the nitrogenous phosphoryl compounds' compositions are fluid at a temperature range of about  $-40$  to  $100^{\circ}$  C. In another variation, the fluid formulations' compositional weight percent comprises nitrogenous phosphoryl compounds at about 20% to about 95%.

(18) In an embodiment, the present invention provides low cost processes for preparing fluid nitrogenous phosphoryl compositions wherein the nitrogenous phosphoryl compounds comprise one or more structures selected from the group consisting of:

(19) ##STR00002##

and Structure II wherein the Structure I Z.sup.1 functional group is not present

(20) ##STR00003## a. wherein: i. R.sup.2 is one or more members selected from the group consisting of CH.sub.3, C.sub.2H.sub.5, C.sub.3H.sub.7, C.sub.4H.sub.9, C.sub.5H.sub.11, C.sub.6H.sub.13, C.sub.6H.sub.5, C.sub.7H.sub.7, OX.sup.1, OCH.sub.3, OC.sub.2H.sub.5, OC.sub.3H.sub.7, OC.sub.4H.sub.9, OC.sub.5H.sub.11, OC.sub.6H.sub.13, and HNR.sup.4, wherein (1) R.sup.4 is one or more members selected from the group consisting of H, CH.sub.3, C.sub.2H.sub.5, C.sub.3H.sub.7, C.sub.4H.sub.9, C.sub.5H.sub.11 and C.sub.6H.sub.13, (2) X.sup.1 is one or more members selected from the group consisting of (a) H, Na, Li, and K, (b) NH.sub.4, (c) one or more organoamines selected from the group consisting of mono C.sub.1-6 amine, di C.sub.1-6 amine, tri C.sub.1-6 amine, mono ethanol amine, diethanol amine, triethanol amine, monoisopropanol amine, diisopropanol amine, triisopropanol amine, ethylene diamine diethylene triamine, triethylene tetraamine, and tetraethylene pentamine, ii. R.sup.1 is one or more members selected from the group consisting of CH.sub.3, C.sub.2H.sub.5, C.sub.3H.sub.7, C.sub.4H.sub.9, C.sub.5H.sub.11 and C.sub.6H.sub.13, iii. R.sup.3 is one or more members selected from the group consisting of H, CH.sub.3, C.sub.2H.sub.5, C.sub.3H.sub.7, C.sub.4H.sub.9, C.sub.5H.sub.11 and C.sub.6H.sub.13, iv. Z.sup.1 is one or more members selected from the group consisting of Oxygen and Sulfur or optionally Z.sup.1 is not present.

(21) In an embodiment, one or more nitrogenous phosphoryl compounds are the reaction product of one or more phosphoryl chloride functional groups with one or more nitrogenous compounds within Organic Liquid System One wherein said Organic Liquid System One comprises the liquid reaction medium for making the desired nitrogenous phosphoryl compounds. In a variation, the

composition of Organic Liquid System One comprises one or more aprotic organic liquids. In another variation, the composition of Organic Liquid System One comprises one or more aprotic organic liquids that are non-reactive with phosphoryl chlorides. In another variation, one or more non-phosphoryl chloride reactive aprotic organic liquids of Organic Liquid System One are optionally added individually during the reaction steps to produce one or more fluid nitrogenous phosphoryl compositions.

(22) In an embodiment, Organic Liquid System Two is optionally added after the reaction consumes all or most of the phosphoryl chlorides functional groups to improve the methods of making one or more fluid nitrogenous phosphoryl compounds. Organic Liquid System Two impart one or more properties to the nitrogenous phosphoryl liquid compositions selected from the group consisting of a) assisting in removing reaction by-product chloride salts from the fluid compositions, b) maintaining the by-product chloride salts in a stable or semi-stable suspension c) lowering viscosity and improving fluidity of the fluid compositions at temperatures of  $(-20)^{\circ}\text{C}$ . or less, d) improving fluidity under summer storage conditions, e) improving evenness and homogeneity of the fluid coating onto urea, f) improving temperature stability of the nitrogenous phosphoryl compounds, and g) improving shipping, storage, and pumping of fluid nitrogenous phosphoryl compositions.

(23) In an embodiment, the composition of Organic Liquid System Two comprises one or more organic liquids selected from the group consisting of a) aprotic organic liquids and b) protic organic liquids. In another variation, the non-aqueous organic liquid delivery system that delivers the dispersed, desired nitrogenous phosphoryl compounds as an even, continuous coating solution or as a stable suspension to a substrate comprises Organic Liquid System Two.

(24) In an embodiment, one of the novel aspects of the methods to make compounds and compositions of the present invention is the lower cost of manufacturing one or more fluid nitrogenous phosphoryl compositions through the elimination of some of the standard processing steps. In a variation, the innovative methods to make yield adequate levels of the desired nitrogenous phosphoryl compounds to meet expected levels of performance in plant growth mediums at reduced cost.

(25) In an embodiment, the disclosed methods do not require many of the required steps of the prior art. Thus, the present methods comprise one or more steps selected from the group consisting of: a) no filtering of dangerous composition containing toxic, flammable reaction mediums that require highly specialized equipment to remove the inorganic by-products safely, b) not requiring specialized equipment to handle the purification of the desired nitrogenous phosphoryl compounds' solids, c) not requiring multiple washing with volatile solvents for purification of one or more desired nitrogenous phosphoryl compounds, d) not requiring shipping and storage of the one or more nitrogenous phosphoryl compounds' in solid forms that have been shown to be less stable versus those dispersed in an organic liquid, e) not requiring an overhead equipment for removal of Organic Liquid System One from fluid compositions, e) not requiring separate solids dissolving step to create a solution, or a colloid, or a suspension of the one or more desired nitrogenous phosphoryl compounds, f) not requiring removal of chloride by-products from fluid compositions, g) reducing the amount of waste generation, and h) eliminating the step required to make fluid compositions comprising one or more nitrogenous phosphoryl compounds.

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## Description

### DETAILED DESCRIPTION

#### Definitions

(1) Phosphoryl chloride functional group(s) refers one or more functional group selected from the group consisting of a)  $\text{Cl}-\text{P}=\text{O}$ ,  $\text{Cl}-\text{P}$ , and  $\text{Cl}-\text{P}=\text{S}$ .

- (2) Liquid refers to a state of matter.
- (3) Suspension refers to solid particles dispersed within a liquid.
- (4) Colloid refers to a mixture that has particles ranging from between 1 and 1000 nanometers in diameter, yet is still able to remain evenly distributed throughout the liquid
- (5) Fluid refers to a liquid, a colloid or a suspension as defined above.
- (6) Solution refers to a solute being completely dissolved in an organic liquid.
- (7) Nitrogenous phosphoryl compound refers to a structure wherein one or more nitrogen containing compounds are bonded through nitrogen to a phosphorus atom.
- (8) % is used to denote weight percent.
- (9) loading refers to the weight percent of one or more nitrogenous phosphoryl compounds dissolved in an organic liquid.
- (10) In an embodiment, phosphoryl chlorides comprise one or more members selected from the group consisting of a) phosphorous trichloride (PCl<sub>3</sub>), b) phosphoryl trichloride (POCl<sub>3</sub>), and c) thiophosphoryl trichloride (PSCl<sub>3</sub>)
- (11) In an embodiment, the present invention provides low cost methods of making one or more fluid nitrogenous phosphoryl compositions (phosphoryl trichloride and/or thiophosphoryl trichloride compositions) wherein one or more nitrogenous phosphoryl compounds comprise one or more members selected from the group consisting of:

(12) ##STR00004##

and Structure II wherein the Structure I Z<sup>sup.1</sup> functional group is not present

(13) ##STR00005## b. wherein: i. R<sup>sup.2</sup> is one or more members selected from the group consisting of CH<sub>3</sub>, C<sub>2</sub>H<sub>5</sub>, C<sub>3</sub>H<sub>7</sub>, C<sub>4</sub>H<sub>9</sub>, C<sub>5</sub>H<sub>11</sub>, C<sub>6</sub>H<sub>13</sub>, C<sub>6</sub>H<sub>5</sub>, C<sub>7</sub>H<sub>7</sub>, OX<sup>sup.1</sup>, OCH<sub>3</sub>, OC<sub>2</sub>H<sub>5</sub>, OC<sub>3</sub>H<sub>7</sub>, OC<sub>4</sub>H<sub>9</sub>, OC<sub>5</sub>H<sub>11</sub>, OC<sub>6</sub>H<sub>13</sub>, and HNR<sup>sup.4</sup>, wherein (1) R<sup>sup.4</sup> is one or more members selected from the group consisting of H, CH<sub>3</sub>, C<sub>2</sub>H<sub>5</sub>, C<sub>3</sub>H<sub>7</sub>, C<sub>4</sub>H<sub>9</sub>, C<sub>5</sub>H<sub>11</sub>, C<sub>6</sub>H<sub>5</sub>, and C<sub>6</sub>H<sub>13</sub>, (2) X<sup>sup.1</sup> is one or more members selected from the group consisting of (a) H, Na, Li, and K, (b) NH<sub>4</sub>, (c) one or more organoamines selected from the group consisting of mono C<sub>1-6</sub> amine, di C<sub>1-6</sub> amine, tri C<sub>1-6</sub> amine, mono ethanol amine, diethanol amine, triethanol amine, monoisopropanol amine, diisopropanol amine, triisopropanol amine, ethylene diamine diethylene triamine, triethylene tetraamine, and tetraethylene pentamine, ii. le is one or more members selected from the group consisting of CH<sub>3</sub>, C<sub>2</sub>H<sub>5</sub>, C<sub>3</sub>H<sub>7</sub>, C<sub>4</sub>H<sub>9</sub>, C<sub>5</sub>H<sub>11</sub> and C<sub>6</sub>H<sub>13</sub>, iii. R<sup>sup.3</sup> is one or more members selected from the group consisting of H, CH<sub>3</sub>, C<sub>2</sub>H<sub>5</sub>, C<sub>3</sub>H<sub>7</sub>, C<sub>4</sub>H<sub>9</sub>, C<sub>5</sub>H<sub>11</sub> and C<sub>6</sub>H<sub>13</sub>, iv. Z<sup>sup.1</sup> is one or more members selected from the group consisting of Oxygen and Sulfur or optionally Z<sup>sup.1</sup> is not present.

(14) In an embodiment, the non-reactive, Organic Liquid System One comprises one or more aprotic organic liquids, wherein said one of more aprotic organic liquids are selected from the group consisting of a) aprotic organic liquids with a boiling point less than or equal to 120° C. and b) aprotic organic liquids with a boiling point greater than 120° C. In a variation, the non-reactive, Organic Liquid System One meets one or more of the properties selected from the group consisting of a) good solvency of the intermediates b) good solvency of final nitrogenous phosphoryl compounds, c) low cost, d) readily available, e) does not degrade the nitrogenous phosphoryl compounds, f) low viscosity, g) poor solvency of the nitrogenous phosphoryl compounds and h) easily recyclable. In a variation, the non-reactive, Organic Liquid System One comprises said one or more aprotic organic liquids with a boiling point less than or equal to 120° C. selected from the group consisting of a) one or more hydrocarbons selected from the group consisting of i) one or more paraffinic liquids, ii) one or more cycloparaffinics, and iii) one or more aromatic hydrocarbons, b) one or more liquid halocarbons c) one or more halohydrocarbons, and d) one or more ethers selected from the group consisting of i) 1,4-dioxane, ii) 1,3-dioxolane, iii)



methyltetrahydrofuran, iv) dimethoxyethane, v) 1,3-dioxane, vi) 1,3-dioxolane, vii) 2,2-dimethyl-1,3-dioxolane, viii) diethyl ether, ix) tetrahydrofuran, and x) tetrahydropyran, e) d-limonene, f) 1,2-dimethyloxyethane and f) one or more esters selected from the group consisting of i) C.sub.1-C.sub.6 alkylformate and ii) C.sub.1-C.sub.6 alkylacetate.

(15) In another variation, the non-reactive, Organic Liquid System One comprises said one or more aprotic organic liquids with a boiling point greater than 120° C. selected from the group consisting of a) one or more alkylene carbonates selected from the group consisting of i) ethylene carbonate, ii) propylene carbonate and iii) butylene carbonate b) one or more 1-alkyl-2-pyrrolidone selected from the group wherein the alkyl functionality is a C.sub.1-C.sub.8 alkyl radical, c) one or more hydrocarbons selected from the group consisting of i) one or more paraffinic liquids, ii) one or more cycloparaffinics, iii) one or more aromatic hydrocarbons, iv) one or more aliphatic hydrocarbons, v) one or more acyclic hydrocarbons, vi) one or more unsaturated hydrocarbon, and vii) one or more polyalphaolefins, d) one or more C.sub.1-C.sub.18 fatty triglycerides, e) one or more organo phosphorous liquids selected from the group consisting of hexamethylphosphoramide and one or more trialkylphosphates selected from the structure:

(16) ##STR00006## wherein: R.sup.22 is alkyl radical —C.sub.1H.sub.3 to —C.sub.6H.sub.13 R.sup.23 is alkyl radical —C.sub.1H.sub.3 to —C.sub.6H.sub.13 R.sup.24 is alkyl radical —C.sub.1H.sub.3 to —C.sub.6H.sub.13 f) sulfolane, g) 2-methoxyethyl ether, h)

cyclohexylpyrrolidone, i) one or more polyols capped with C.sub.1-C.sub.6 organic acid wherein the polyol portion is selected from the group consisting of (i) ethylene glycol, (ii) 1,3 propylene glycol, (iii) 1,2 propylene glycol, (iv) butylene glycol, (v) trimethylol propane, (vi) trimethylol ethane, (vii) pentaerythritol, (viii) sorbitol, (ix) sorbitan, (x) glucose, (xi) fructose, (xii) galactose and (xiii) glycerin, j) one or more alkylene glycol alkyl ethers acetates selected from the group consisting of (i) dipropylene glycol methyl ether acetate, (ii) tripropylene glycol methyl ether acetate, and (iii) tripropylene glycol butyl ether acetate, k) isophorone, l) one or more diesters selected from the group consisting of (i) C.sub.1-C.sub.6 dialkylsuccinate, (ii) C.sub.1-C.sub.6 dialkyladipate, (iii) C.sub.1-C.sub.6 dialkylmethylglutarate, (iv) C.sub.1-C.sub.6 dialkyl glutarate, (v) C.sub.1-C.sub.6 dialkyl ethylsuccinate, and (vi) C.sub.1-C.sub.6 dialkyl maleate, m) dimethyl-2-imidazolidinone, n) one or more fatty acid alkyl esters wherein the one or more fatty acid functionality is selected from the group consisting of C.sub.8-C.sub.18 fatty acids and wherein the alkyl functionality is one or more C.sub.1-C.sub.6 alkyl radicals, and or) one or more dialkylene glycols dialkyl ethers, (glymes) selected from the structure:

R.sup.11—O—(R.sup.12—O).sub.n—R.sup.13 wherein: R.sup.11 is one or more alkyl radicals selected from the group consisting of C.sub.1 to C.sub.6, R.sup.12 is one or more alkylene radicals selected from the group consisting of C.sub.1 to C.sub.4, R.sup.13 is one or more alkyl radicals selected from the group consisting of C.sub.1 to C.sub.6, and n is one or more integers selected from the group of 1 to 10.

(17) In a variation, said Organic Liquid System One comprises one or more aprotic organic liquids with a boiling point greater than 120° C. wherein the non-aqueous organic liquid delivery system for imparting nitrogenous phosphoryl compounds onto the surface of substrates comprises the Organic Liquid System One wherein the remaining Organic Liquid System One has a boiling point greater than 120° C. In a variation, the Organic Liquid System One comprising said one or more aprotic organic liquids with a boiling point greater than 120° C. meets one or more of the requirements selected from the group consisting of: a) being environmentally safe, b) having a flashpoint above 145° F., c) being rated safe for contact with humans and animals, d) improving the processibility/costing of the method to make the fluid nitrogenous phosphoryl compositions, i) increasing the percent nitrogenous phosphoryl compounds and their precursors loading at processing temperatures, ii) lowering viscosity of reaction mixtures, iii) improving separation phase for removal of reaction by-products, iv) incorporating the chloride salt by-products into a stable suspension, e) serving as the delivery system or a component of a delivery system that

delivers the desired nitrogenous compound to the surface of a substrate, f) being non-reactive with phosphoryl chloride functional groups, g) assisting in stabilizing the inorganic by-product salts in a liquefied suspension, and h) not causing the degradation of the nitrogenous phosphoryl compounds. (18) In an embodiment, the Organic Liquid System One comprises one or more aprotic organic liquids with a boiling point less than or equal to 120° C., which is removed by methods known by those skilled in the art and optionally replaced by Organic Liquid System Two after the reaction has consumed all or most of the phosphoryl chloride functional groups. In a variation, the Organic Liquid System One comprising one or more aprotic organic liquids with a boiling point less than or equal to 120° C. is removed and optionally replaced by Organic Liquid System Two after the removal of all or most of the salt by-products of the reaction to form solutions of the desired nitrogenous phosphoryl compounds. In a variation, the composition of Organic Liquid System Two comprises one or more organic liquids selected from the group consisting of 1) aprotic organic liquids and 2) protic organic liquids wherein one or more of said aprotic organic liquids are selected from the group consisting of: a) dimethyl sulfoxide, b) one or more sulfoxide(s) selected from the group consisting of dialkyl, diaryl, or alkylaryl sulfoxide(s) selected from the formula structure:

$R_{sup.9}S(O)_{sub.x}R_{sup.10}$  wherein i)  $R_{sup.9}$  and  $R_{sup.10}$  are each independently a  $C_{sub.1-6}$  alkylene group, an aryl group or  $C_{sub.1-6}$  alkylenearyl group, ii) or  $R_{sup.9}$  and  $R_{sup.10}$  with the sulfur to which they are attached form a 4 to 8 membered ring wherein  $R_{sup.9}$  and  $R_{sup.10}$  together are a  $C_{sub.1-6}$  alkylene group which optionally contains one or more atoms selected from the group consisting of O, S, Se, Te, N, and P in the ring, iii) and x is 1 or 2, c) one or more alkylene carbonates selected from the group consisting of i) ethylene carbonate, ii) propylene carbonate, and iii) butylene carbonate, d) one or more polyols capped with  $C_{sub.1-6}$  organic acid wherein the polyol portion is selected from the group consisting of i) ethylene glycol, ii) 1,3 propylene glycol, iii) 1,2 propylene glycol, iv) butylene glycol, v) trimethylol propane, vi) trimethylol ethane, vii) pentaerythritol, viii) sorbitol, ix) sorbitan, x) glucose, xi) fructose, xii) galactose and xiii) glycerin, e) one or more alkylene glycol alkyl ethers acetates selected from the group consisting of i) dipropylene glycol methyl ether acetate, ii) tripropylene glycol methyl ether acetate, and iii) tripropylene glycol butyl ether acetate, f) one or more diesters selected from the group consisting of i)  $C_{sub.1-6}$  dialkylsuccinate, ii)  $C_{sub.1-6}$  dialkyladipate, iii)  $C_{sub.1-6}$  dialkylmethylglutarate, iv)  $C_{sub.1-6}$  dialkyl glutarate, v)  $C_{sub.1-6}$  dialkyl ethylsuccinate, and vi)  $C_{sub.1-6}$  dialkyl maleate, g) one or more 1-alkyl-2-pyrrolidone selected from the group wherein the alkyl functionality is a  $C_{sub.1-8}$  alkyl radical, h) one or more members selected from the group consisting of i) dimethylacetamide, ii) dimethylformamide, iii) dimethyl-2-imidazolidinone, iv) isophorone, v) hexamethylphosphoramide, vi) 2-methoxyethyl ether, vii) N,N-dimethyldecanamide, viii) N,N-dimethyloctanamide, ix) limonene, x) sulfolane i) one or more trialkylphosphates selected from the group represented by the structure:

(19) ##STR00007## wherein:  $R_{sup.22}$  is alkyl radical  $-C_{sub.1}H_{sub.3}$  to  $-C_{sub.6}H_{sub.13}$   $R_{sup.23}$  is alkyl radical  $-C_{sub.1}H_{sub.3}$  to  $-C_{sub.6}H_{sub.13}$   $R_{sup.24}$  is alkyl radical  $-C_{sub.1}H_{sub.3}$  to  $-C_{sub.6}H_{sub.13}$  j) one or more dialkylene glycols dialkyl ethers, (glymes) selected from the structure:

$R_{sub.11}-O-(R_{sup.12}-O)_{sub.n}-R_{sup.13}$  wherein:  $R_{sup.11}$  is one or more alkyl radicals selected from the group consisting of  $C_{sub.1}$  to  $C_{sub.6}$ ,  $R_{sup.12}$  is one or more alkylene radicals selected from the group consisting of  $C_{sub.1}$  to  $C_{sub.4}$ ,  $R_{sup.13}$  is one or more alkyl radicals selected from the group consisting of  $C_{sub.1}$  to  $C_{sub.6}$ , and n is one or more integers selected from the group of 1 to 10, k) one or more hydrocarbons selected from the group consisting of i) one or more paraffinic liquids, ii) one or more cycloparaffinics, iii) one or more aromatic hydrocarbons, iv) aliphatic hydrocarbons, v) acyclic hydrocarbons, vi) unsaturated hydrocarbon, and vii) polyalphaolefins, l) one or more fatty acid alkyl esters wherein the one or more fatty acid

functionality is selected from the group consisting of C.sub.8-C.sub.18 fatty acids and wherein the alkyl functionality is one or more C.sub.1-C.sub.6 alkyl radicals, and m) one or more C.sub.1-C.sub.18 fatty triglycerides,

and wherein said protic organic liquid is one or more members selected from the group consisting of: a) one or more alkanols selected from the group consisting of C.sub.1-C.sub.10 alkanols, b) one or more polyols selected from the group consisting of i) trimethylol propane, ii) trimethylol ethane, iii) pentaerythritol, iv) sorbitol, v) sorbitan, vi) glucose, vii) fructose, viii) galactose, and ix) glycerin, c) one or more polyalkylene glycols selected from the group consisting of poly(C.sub.1-C.sub.10 alkylene) glycols, d) one or more dioxolane compound selected from the group consisting of i) 2,2-dimethyl-1,3-dioxolane-4-methanol and ii) 2,2-diisobutyl-1,3-dioxolane-4-methanol, e) one or more alkylene glycol alkyl ethers selected from the structure:

(20) ##STR00008## wherein R.sup.14 is one or more members selected from the group consisting of CH.sub.3, C.sub.2H.sub.5, C.sub.3H.sub.7, C.sub.6H.sub.5, and C.sub.4H.sub.9, R.sup.15 is one or more members selected from the group consisting of H and

(21) ##STR00009## wherein R.sup.17 is one or more members selected from the group consisting of H and CH.sub.3, and f is an integer between 1 and 15, and R.sup.16 is one or more members selected from the group consisting of H and CH.sub.3, f) one or more alkyl lactates selected from the group consisting of i) ethyl lactate, ii) propyl lactate and iii) butyl lactate, g) one or more alkanolamines selected from the group consisting of alkanolamines selected from the structure:

(22) ##STR00010## wherein R.sup.5 is one or more members selected from the group consisting of CH.sub.3, C.sub.2H.sub.5, C.sub.2H.sub.4OR.sup.8, C(CH.sub.3).sub.2CH.sub.2OH, CH(C.sub.2H.sub.5)CH.sub.2OH, CH.sub.3CH(OH)CH.sub.2, and C.sub.3H.sub.6OH, R.sup.6 is one or more members selected from the group consisting of H, CH.sub.3, C.sub.2H.sub.5, C.sub.2H.sub.4OR.sup.8 and C.sub.3H.sub.6OH, R.sup.7 is one or more members selected from the group consisting of H, C.sub.2H.sub.4OR.sup.8 and C.sub.3H.sub.6OH, wherein R.sup.8 is (C.sub.2H.sub.4O).sub.gH wherein g is an integer between 1 and 10, h) and glycerol carbonate.

(23) A generic flow diagram for the improved process to yield lower cost of one or more liquid nitrogenous phosphoryl compositions is shown below in scheme 2:

(24) In an embodiment, a process to yield one or more low cost, fluid nitrogenous phosphoryl compositions comprise one or more steps selected from the group consisting of: 1. reacting one or more phosphoryl chloride functional groups within Organic Liquid System One with one or more alkylamines to make one or more monoalkylamide phosphoryl dichlorides and/or one or more monoalkylamide thiophosphoryl dichlorides, wherein the by-product, hydrochloric acid (HCl) is consumed by one or more methods selected from the group consisting of: a. reacting ammonia or other alkali compounds with the hydrochloric acid from one or more alkylammonium chlorides freeing the one or more alkylamines to fully react to form one or more monoalkylamide phosphoryl dichlorides and/or one or more monoalkylamide thiophosphoryl dichlorides, b. utilizing an HCl absorbent such as one or more trialkylamine to absorb the HCl, 2. reacting one or more monoalkylamide phosphoryl dichlorides and/or one or more monoalkylamide thiophosphoryl dichloride with ammonia or an ammonia generating compound resulting in desired one or more nitrogenous phosphoryl compounds, 3. removing inorganic chloride by-products by methods known by those skilled in the art such as, a) filtration, b) sedimentation and removal of organic layer, c) liquefaction of the inorganic salt by-products and subsequent draining off of by-products i) liquefaction of ammonium chloride with excess ammonia and pressurization of the reaction vessel #1, ii) liquefaction of chloride by-products with water addition which has been optionally de-oxygenated, optionally deionized and/or optionally distilled, 4) not removing the inorganic by-products wherein the inorganic by-products are maintained in a stable or semi-stable fluid suspension within the NOLDS, 5) removal of volatile components of Organic Liquid System One

through methods known in the art such as reduced pressures and temperatures less than 100° C., a) optionally Organic Liquid System One contains components with boiling point above 120° C. and therefore are not removed from said one or more low cost, fluid nitrogenous phosphoryl compositions, and 6) adding Organic Liquid System Two, wherein Organic Liquid System Two meet one or more of the following: a) are environmentally safe, b) have a flashpoint above 145° F., c) rated safe for contact with humans and animals, e) serves as the delivery system or components of a delivery system that delivers the desired nitrogenous compound to the surface of a substrate, f) does not degrade the one or more nitrogenous phosphoryl compounds, wherein a fluid composition comprising at least one nitrogenous phosphoryl compound at levels of 20-80% wherein the composition is fluid at (-20) to 55° C., and wherein the one or more low cost, fluid nitrogenous phosphoryl compositions comprise one or more physical states selected from the group consisting of a) liquid solutions, b) colloid and c) suspension.

(25) In a variation, the method to make fluid compositions comprising one or more nitrogenous phosphoryl compounds at levels of 20-90% wherein the composition is fluid at: (-40) to 100° C., (-20) to 80° C., (-20) to 70° C., (-10) to 70° C., 0 to 70° C., 10 to 70° C., 20 to 70° C., (-20) to 60° C., 20 to 60° C., or 25 to 55° C. In an embodiment, the method further comprises one or more methods/steps selected from the group consisting of a) polishing filtration of the fluid nitrogenous phosphoryl compounds to clear solution compositions, b) no filtration, pump to storage tank allow solids to settle to the bottom of the tank for removal, and c) maintain the inorganic by-products in a stable fluid suspension.

(26) In another variation, the method to make the fluid compositions comprising one or more nitrogenous phosphoryl compounds at levels of 20-90%, 30-85%, 40-85%, 50-90%, 50-80%, 20-30%, 20-40%, 20-50%, 30-40%, 30-50%, 40-50%, 40-60%, 50-70%, or 50-60% wherein the composition is fluid at (-40) to 100° C. Methods to make one or more fluid nitrogenous phosphoryl compositions further comprises one or more methods selected from the group consisting of pumping a) to storage tanks, b) to shipping containers and/or bulk transportation, c) to a vessel for further formulation optimization, and d) to application setups to impart a continuous homogenous liquid coating solution or a stable suspension comprising nitrogenous phosphoryl compounds.

(27) In an embodiment, one or more nitrogenous phosphoryl compounds comprise adducts of the reaction of a) one or more phosphoryl chlorides selected from the group consisting of: i) phosphoryl trichloride, ii) phosphoryl trichloride and iii) thiophosphoryl trichloride with b) one or more alkylamines selected from the group consisting of i) one or more monoalkyl amines and ii) one or more dialkyl amines wherein the alkyl functionalities of one or more monoalkyl amines and/or one or more dialkyl amines is selected from the group consisting of (a) —C.sub.1H.sub.3, (b) —C.sub.2H.sub.5, (c) —C.sub.3H.sub.7, (d) —C.sub.4H.sub.9, (e) (f) —C.sub.6H.sub.5, and (g) —C.sub.6H.sub.13. In a variation, said alkyl functionalities comprise linear or branched alkyl groups.

(28) In a variation, the molar ratio of the reactants of a) one or more said phosphoryl chlorides to b) one or more said alkylamines comprises one or more ratios of one or more phosphoryl chlorides:one or more alkylamines is selected from the group consisting of a) 1:2, b) 1:1.75, c) 0.75:1, d) 1:1.5, e) 1:1.25, f) 1:1.2, g) 1:1.1, h) 1:1.05, i) 0.95:1, and j) 1:1.3.

(29) In an embodiment, the temperature of the reaction in Step 1 of said one or more phosphoryl chlorides with one or more alkylamines comprises one or more members selected from the group consisting of a) -40° C. to 100° C., b) -40° C. to 80° C., c) -40° C. to 60° C., d) -30° C. to 60° C., e) -30° C. to 40° C., f) -30° C. to 20° C., g) -20° C. to 20° C., h) -20° C. to 10° C., i) -20° C. to 0° C., j) -20° C. to -10° C., k) -10° C. to 10° C., and l) -10° C. to 0° C. In a variation, the temperature of the reaction in Step 1 of said one or more phosphoryl chlorides with one or more said alkylamines is selected to optimize the selectivity of each of the chlorides on phosphoryl chlorides to result in the formation of primarily monoalkylamide phosphoryl chlorides.

(30) In an embodiment, the temperature of the reaction in Step 1a of said one or more

alkylammonium chlorides with one or more ammonia and/or ammonia generating compounds comprises one or more members selected from the group consisting of a)  $-40^{\circ}\text{C.}$  to  $100^{\circ}\text{C.}$ , b)  $-40^{\circ}\text{C.}$  to  $80^{\circ}\text{C.}$ , c)  $-40^{\circ}\text{C.}$  to  $60^{\circ}\text{C.}$ , d)  $-30^{\circ}\text{C.}$  to  $60^{\circ}\text{C.}$ , e)  $-30^{\circ}\text{C.}$  to  $40^{\circ}\text{C.}$ , f)  $-30^{\circ}\text{C.}$  to  $20^{\circ}\text{C.}$ , g)  $-20^{\circ}\text{C.}$  to  $20^{\circ}\text{C.}$ , h)  $-20^{\circ}\text{C.}$  to  $10^{\circ}\text{C.}$ , i)  $-20^{\circ}\text{C.}$  to  $0^{\circ}\text{C.}$ , j)  $-20^{\circ}\text{C.}$  to  $-10^{\circ}\text{C.}$ , k)  $-10^{\circ}\text{C.}$  to  $10^{\circ}\text{C.}$ , and l)  $-10^{\circ}\text{C.}$  to  $0^{\circ}\text{C.}$  In a variation, Step 1a of the reaction releases one or more alkylamines from their chloride salts to further react with phosphoryl chlorides. In a variation, a hydrochloric acid absorbent is utilized in Step 1 eliminating the requirement for Step 1a.

(31) In an embodiment, the temperature of the reaction in Step 1 and Step 1a are selected to take advantage of the selectivity of each of chlorides on phosphoryl chlorides to optimize the formation of the monoalkylamide phosphoryl chloride. In a variation, the agitation speed is maintained at a rate to ensure quick mixing of amine and ammonia reactants. In another variation, the agitation is slowed at some point during the ammonia gas charge.

(32) In an embodiment, HCl reactants comprise one or more members selected from the group consisting of a) ammonia, b) ammonium bicarbonate, c) ammonium carbonate, d) ammonium hydroxide, e) magnesium carbonate, f) magnesium oxide, g) magnesium hydroxide, h) calcium carbonate, i) calcium oxide, j) calcium hydroxide, k) potassium carbonate, l) potassium bicarbonate, m) sodium carbonate, and n) sodium bicarbonate.

(33) In an embodiment, an HCl absorbent comprises one or more trialkylamines wherein the alkyl portion of said one or more trialkylamine is selected from the group consisting of i) methyl, ii) ethyl, iii) propyl, iv) butyl and mixtures thereof.

(34) In an embodiment, because the overall process results in fluid nitrogenous phosphoryl compositions, Organic Liquid System One optionally comprises organic liquids that can be added at any time during the reaction phase of the process and that meet one or more of the following criteria: a) are environmentally safe, b) have a flashpoint above  $145^{\circ}\text{F.}$ , c) are rated safe for contact with humans and animals, d) improve processibility/cost of the method to make one or more fluid nitrogenous phosphoryl compositions, i) increase % nitrogenous phosphoryl compounds and their precursors loading of Organic Liquid System One at processing temperatures, ii) lower viscosity of reaction mixtures, iii) improve separation phase for removal of reaction by-products, iv) possess poor dissolution of one or more nitrogenous phosphoryl compounds, v) assist in stabilizing the inorganic chloride by-product salts in a fluid suspension, e) serve as the delivery system or a component of a delivery system that delivers the one or more nitrogenous phosphoryl compounds onto the surface of a substrate, f) is non-reactive with phosphoryl chloride functional groups, and/or g) does not degrade the nitrogenous phosphoryl compounds.

(35) In an embodiment, a stable suspension composition comprises a) one or more nitrogenous phosphoryl compounds, b) one or more inorganic chloride salts and an Organic Liquid System One comprises one or more aprotic organic liquids non-reactive to phosphoryl chloride functional group, wherein said Organic Liquid System One comprises said one or more aprotic organic liquids with a boiling point greater than  $120^{\circ}\text{C.}$  and wherein said one or more aprotic organic liquids are selected from the group consisting of: a) one or more alkylene carbonates, b) one or more 1-alkyl-2-pyrrolidone selected from the group wherein the alkyl functionality is a C.sub.1-C.sub.8 alkyl radical, c) one or more hydrocarbons, d) one or more C.sub.1-C.sub.18 fatty triglycerides, e) one or more organo phosphorous liquids selected from the group consisting of hexamethylphosphoramide and one or more trialkylphosphates selected from the structure:

(36) ##STR00011## wherein: R.sup.22 is alkyl radical  $-\text{C}_{1-6}\text{H}_{3-13}$  R.sup.23 is alkyl radical  $-\text{C}_{1-6}\text{H}_{3-13}$  R.sup.24 is alkyl radical  $-\text{C}_{1-6}\text{H}_{3-13}$ , f) sulfolane, g) 2-methoxyethyl ether, h) cyclohexylpyrrolidone, i) one or more polyols capped with C.sub.1-C.sub.6 organic acid, j) one or more alkylene glycol alkyl ethers acetates, k) isophorone, l) one or more diesters, m) dimethyl-2-imidazolidinone, n) one or more fatty acid alkyl esters wherein the one or more fatty acid functionality is selected from the group consisting of C.sub.8-C.sub.18 fatty acids and wherein the

alkyl functionality is one or more C.sub.1-C.sub.6 alkyl radicals, and o) one or more dialkylene glycols dialkyl ethers, (glymes) selected from the structure:

R.sub.11—O—(R.sub.12—O).sub.n—R.sub.13 wherein: R.sub.11 is one or more alkyl radicals selected from the group consisting of C.sub.1 to C.sub.6, R.sub.12 is one or more alkylene radicals selected from the group consisting of C.sub.1 to C.sub.4, R.sub.13 is one or more alkyl radicals selected from the group consisting of C.sub.1 to C.sub.6, and n is one or more integers selected from the group of 1 to 10.

(37) In a variation, said one or more alkylene carbonates are selected from the group consisting of i) ethylene carbonate, ii) propylene carbonate and iii) butylene carbonate

(38) In a variation, said one or more hydrocarbons are selected from the group consisting of i) one or more paraffinic liquids, ii) one or more cycloparaffinics, iii) one or more aromatic hydrocarbons, iv) one or more aliphatic hydrocarbons, v) one or more acyclic hydrocarbons, vi) one or more unsaturated hydrocarbon, and vii) one or more polyalphaolefins.

(39) In a variation, said one or more polyols capped with C.sub.1-C.sub.6 organic acid wherein the polyol portion is selected from the group consisting of i) ethylene glycol, ii) 1,3 propylene glycol, iii) 1,2 propylene glycol, iv) butylene glycol, v) trimethylol propane, vi) trimethylol ethane, vii) pentaerythritol, viii) sorbitol, ix) sorbitan, x) glucose, xi) fructose, xii) galactose and xiii) glycerin.

(40) In a variation, said one or more alkylene glycol alkyl ethers acetates selected from the group consisting of (i) dipropylene glycol methyl ether acetate, (ii) tripropylene glycol methyl ether acetate, and (iii) tripropylene glycol butyl ether acetate.

(41) In a variation, said one or more diesters selected from the group consisting of (i) C.sub.1-C.sub.6 dialkylsuccinate, (ii) C.sub.1-C.sub.6 dialkyladipate, (iii) C.sub.1-C.sub.6 dialkylmethylglutarate, (iv) C.sub.1-C.sub.6 dialkyl glutarate, (v) C.sub.1-C.sub.6 dialkyl ethylsuccinate, and (vi) C.sub.1-C.sub.6 dialkyl maleate.

(42) In a variation, one or more particles within said stable suspension composition comprises one or more members selected from the group a) one or more chloride salts, b) one or more nitrogenous phosphoryl compounds, and c) one or more fusion particles. Not to be bound by theory and based on belief and observations, said fusion particles are formed in-situ during the reaction of one or more phosphoryl chlorides with one or more alkylamines. In another variation, said one or more fusion particles comprise one or more chloride salts and one or more nitrogenous phosphoryl compounds. In another variation, the unexpected stability of the suspension is the result of the formation of small particles in situ and the presence of said fusion particle.

(43) In a variation, said stable suspension composition further comprises Organic Liquid System Two.

(44) In a variation, said stable suspension composition further comprises one or more members selected from the group consisting of a) one or more colorants, b) one or more scents, c) one or more odor masking agents, d) one or more nitrogenous phosphoryl compound organic stabilizers, and e) one or more surfactants.

(45) In a variation, when an excellent nitrogenous phosphoryl compound solvent, such as DMSO, is combined with said stable suspension compositions, the results are a) a destabilizing or the suspension and b) the particles are difficult to remove through filtration due to the presence of the formation of a cohesive, sticky residue. In another variation, ammonium chloride is insoluble in DMSO.

(46) In an embodiment, liquid solution compositions comprise methods to make that result in one or more nitrogenous phosphoryl compounds completely dissolved into a solution within a non-aqueous, organo liquid delivery system resulting in one or more fluid nitrogenous phosphoryl compositions. In a variation, said non-aqueous, organo liquid delivery system comprises low volatility Organic liquid System One wherein low volatility Organic Liquid System One comprises aprotic organic liquid that are non-reactive with phosphoryl chloride functional groups and wherein said low volatility Organic Liquid System One comprises organic liquids with boiling points

greater than or equal to 120° C. In another variation, said non-aqueous, organo liquid delivery system comprises one or more member selected from the group consisting of a) low volatility Organic Liquid System One and b) Organic Liquid System Two wherein one or more fluid phosphoryl nitrogenous compositions meet the following: a) are environmentally safe, b) having a flashpoint above 145° F., c) are rated safe for contact with humans and animals, d) having % loading of one or more phosphoryl nitrogenous compounds of 20-95% and are fluid at temperature range of -40 to 100° C. e) can be applied to nitrogen source fertilizer granules through application by commercial sprayers and metered applicators onto fertilizer granules mobilized by mixing, stirring and/or blending to ensure an even coating of the solution of nitrogenous phosphoryl compounds on the surfaces of said nitrogen source fertilizer granules through commingling of said solution with said nitrogen source fertilizer granules.

(47) ##STR00012## wherein: a) X is one or more members selected from the group consisting of i) P=O, ii) P, and iii) P=S b) Z is one or more members selected from the group consisting of i) one or more alkyl radicals selected from the group consisting of (a) —C.sub.1H.sub.3, (b) —C.sub.2H.sub.5, (c) —C.sub.3H.sub.7, (d) —C.sub.4H.sub.9, (e) —C.sub.5H.sub.11, (f) —C.sub.6H.sub.5, and g) H (hydrogen) c) R.sup.18 is one or more alkyl radicals selected from the group consisting of (a) —C.sub.1H.sub.3, (b) —C.sub.2H.sub.5, (c) —C.sub.3H.sub.7, (d) —C.sub.4H.sub.9, (e) —C.sub.5H.sub.11, and (f) —C.sub.6H.sub.5.

(48) In an embodiment, methods to make liquid solutions of one or more nitrogenous phosphoryl compositions comprise a reaction of one or more phosphoryl chlorides with one or more alkylamines in an aprotic, nonreactive organic solvent medium in reaction vessel #1 according to methods to make known by those in the art.

(49) In a variation, said reaction medium is charged into reaction vessel #1 wherein said reaction medium comprises Organic Liquid System One, wherein said Organic Liquid System One comprises one or more non-phosphoryl chloride reactive aprotic organic liquids selected from the group consisting of one or more aprotic organic liquids with a boiling point less than or equal to 120° C. In a variation, said Organic Liquid System One comprises one or more aprotic organic liquids selected from the group consisting of a) one or more hydrocarbons selected from the group consisting of i) one or more paraffinic liquids, ii) one or more cycloparaffinics, and iii) one or more aromatic hydrocarbons, b) one or more liquid halocarbons c) one or more halohydrocarbons, and d) one or more ethers selected from the group consisting of i) 1,4-dioxane, ii) 1,3-dioxolane, iii) methyltetrahydrofuran, iv) dimethoxyethane, v) 1,3-dioxane, vi) 1,3-dioxolane, vii) 2,2-dimethyl-1,3-dioxolane, viii) diethyl ether, ix) tetrahydrofuran, and x) tetrahydropyran, e) d-limonene, f) 1,2-dimethyloxyethane and f) one or more esters selected from the group consisting of i) C.sub.1-C.sub.6 alkylformate and ii) C.sub.1-C.sub.6 alkylacetate.

(50) In a variation, the composition of reaction vessel #1 further comprises one or more aprotic organic liquids that are non-reactive with one or more phosphoryl chlorides and wherein the one or more aprotic organic liquids have a boiling point greater than 120° C.

(51) In a variation, reaction vessel #1 has been deoxygenated and is filled with an inert gas.

(52) In a variation, the contents of reaction vessel #1 are adjusted to a temperature range of (-40) to 20° C. In another variation, the contents of reaction vessel #1 are adjusted one or more temperature ranges of (-40) to (-20)° C., c) (-40) to 0° C., d) (-40) to (-10)° C., e) (-30) to 20° C., f) (-30) to 10° C., g) (-30) to (-10)° C., h) (-20) to 20° C., i) (-20) to 10° C., j) (-20) to 0° C., k) (-20) to (-10)° C., l) (-10) to 20° C., m) (-10) to 10° C., n) (-10) to 0° C., o) 0 to 20° C., and p) 0 to 10° C.,

(53) In a variation, the contents of reaction vessel #1 are under strong agitation.

(54) In a variation, said one or more alkylamines are slowly charged to reaction vessel #1.

(55) In a variation, the charging of said one or more alkylamines is above the surface and/or sub-surface of the contents in reaction vessel #1. In another variation, the rate of charging said one or more alkylamines to the contents of reaction vessel #1 is dependent on maintaining the temperature

of the contents of reaction vessel #1 at less than 5° C., In another variation, the rate of charging said one or more alkylamines to the contents of reaction vessel #1 is dependent on maintaining the temperature of the contents of reaction vessel #1 at less than one or more of the following temperatures i) (-20)° C., ii) (-15)° C., iii) (-10)° C., iv) (-5)° C., v) 0° C., vi) 5° C., and vii) <(-20)° C.,

(56) In a variation, said one or more alkylamines is premixed with a portion of said Organic Liquid System One and then slowly charged to reaction vessel #1,

(57) In a variation, said one or more phosphoryl chlorides comprise one or more phosphoryl trichlorides and wherein the molar ratio range of the one or more said alkylamines to the one or more said phosphoryl trichlorides is 0.7:1.5. In another variation, the molar ratio of said one or more alkylamines to said one or more phosphoryl trichlorides is selected from the group consisting of (a) 0.7:1.0, (b) 0.8:1.0, (c) 0.9:1.0, (d) 0.95:1.0, (e) 1.0:1.0, (f) 1.05:1.0, (g) 1.1:1.0, (h) 1.2:1.0, (i) 1.3:1.0, (j) 1.4:1.0, (k) 1.5:1.0, (l) 0.7:1.0 and m) 0.9:1.0,

(58) In a variation, the composition of the contents do not comprise one or more trialkylamines that are utilized as HCl absorbants,

(59) In an embodiment, ammonia gas and/or ammonia generating compounds are charged to the contents of reaction vessel #1 under agitation.

(60) In a variation, said ammonia gas and/or ammonia generating compounds are charged very slowly, initially, to the contents of reaction vessel #1.

(61) In a variation, the charging of said ammonia gas and/or ammonia generating compound is above the surface and/or sub-surface of the said contents in reaction vessel #1.

(62) In a variation, the rate of charging said ammonia gas and/or ammonia generating compound is dependent on maintaining the temperature of the contents of reaction vessel #1 at less than 100° C. In another variation, the rate of charging said ammonia gas and/or ammonia generating compound is dependent on maintaining the temperature of the contents of reaction vessel #1 at less than one or more of the following temperatures i)  $\leq(-20)^{\circ}\text{C.}$ , ii)  $\leq(-15)^{\circ}\text{C.}$ , iii)  $\leq(-10)^{\circ}\text{C.}$ , iv)  $\leq(-5)^{\circ}\text{C.}$ , v)  $\leq(-0)^{\circ}\text{C.}$ , f)  $\leq 5^{\circ}\text{C.}$ , vi)  $<(-20)^{\circ}\text{C.}$ , vii)  $\leq 5^{\circ}\text{C.}$ , viii)  $\leq 10^{\circ}\text{C.}$ , ix)  $\leq 15^{\circ}\text{C.}$ , x)  $\leq 20^{\circ}\text{C.}$ , xi)  $\leq 25^{\circ}\text{C.}$ , xii)  $\leq 30^{\circ}\text{C.}$ , xiii)  $\leq 40^{\circ}\text{C.}$ , xiv)  $\leq 50^{\circ}\text{C.}$ , xv)  $\leq 60^{\circ}\text{C.}$ , xvi)  $\leq 70^{\circ}\text{C.}$ , xvii)  $\leq 80^{\circ}\text{C.}$ , xviii)  $\leq 90^{\circ}\text{C.}$ , and xix)  $\leq 100^{\circ}\text{C.}$

(63) In a variation, the rate of charge of ammonia gas and/or ammonia generating compound can be optionally slow in the beginning with very slow agitation.

(64) In a variation, the temperature of the contents of reaction vessel #1 are initially at  $\leq(-10)^{\circ}\text{C.}$ . In another variation, the temperature of the contents of reaction vessel #1 are maintained at a temperature range of (-10) to 5° C. through the first ¼ to ½ of the ammonia gas and/or ammonia generating compound, and allowed to slowly increase temperatures to a range of 30 to 100° C. for the remaining ¾ to ½ of the remaining charge of ammonia gas and/or ammonia generating compound.

(65) In an embodiment, upon completion of the charging of ammonia gas and/or ammonia generating compound, the contents of reaction vessel #1 are mixed for an additional 5-120 additional minutes at a temperature range of 30-100° C.

(66) In a variation, the contents of reaction vessel #1 are mixed for additional minutes comprising one or more time ranges selected from the group consisting of (1) 5-120 minutes, (2) 10-120 minutes, (3) 20-120 minutes, (4) 30-120 minutes, (5) 40-120 minutes, (6) 50-120 minutes, (7) 1-120 minutes, (8) 60-120 minutes, (9) 5-60 minutes, (10) 10-60 minutes, (11) 20-60 minutes, (12) 30-60 minutes, (13) 40-60 minutes, and (14) 50-60 minutes.

(67) In another variation, the temperature of reaction vessel #1's contents are maintained at one or more of the following temperature ranges (1) 30 to 40° C., (2) 30 to 50° C., (3) 30 to 60° C., (4) 30 to 70° C., (5) 30 to 80° C., (6) 30 to 90° C., (7) 30 to 100° C., (8) 40 to 60° C., (9) 40 to 70° C., (10) 40 to 80° C., (11) 40 to 90° C., (12) 40 to 100° C., (13) 50 to 60° C., and (14) 50 to 70° C.

(68) In an embodiment, upon completion of the reaction phase, the one or more inorganic chloride



by-products in reaction vessel #1 are removed from the contents of reaction vessel #1 by one or more methods known by those skilled in the art selected from the group consisting of a) filtration, b) sedimentation and removal of organic layer, and c) liquefaction of the inorganic chloride salt by-products and subsequent draining off of by-products, i) liquefaction through excess ammonia or ammonium hydroxide, ii) liquefaction through excess ammonia or ammonium hydroxide, wherein the reaction vessel #1 is pressurized with excess ammonia to decrease the amount of water present, and iii) liquefaction through dissolving by-products with water which has been optionally de-oxygenated.

(69) In an embodiment, the remaining organic contents from reaction vessel #1 is heated to less than 70° C. and placed under a vacuum of 0.1-40 mm of Hg to strip out the volatile components of Organic Liquid System One.

(70) In a variation, Organic Liquid System Two is charged to organic contents of reaction vessel #1 as needed to maintain fluidity of the organic contents from reaction vessel #1. In another variation, Organic Liquid System Two is charged to the contents of the stripped organic content from reaction vessel #1 after all of the volatile components of Organic Liquid System One have been removed resulting in a liquid solution. In another variation, said liquid solution of one or more nitrogenous phosphoryl compounds has any remaining inorganic chloride salt by-products removed by filtration and/or sedimentation.

(71) In a variation, a colorant is added to said liquid solution of one or more nitrogenous phosphoryl compounds.

(72) In a variation, a scent is added to said liquid solution of one or more nitrogenous phosphoryl compounds.

(73) In a variation, a buffer is added to said liquid solution of one or more nitrogenous phosphoryl compounds.

(74) In a variation, one or more surfactants are added to the contents of reaction vessel #1, wherein the one or more surfactants comprises one or more members selected from the group consisting of (a) one or more cationic surfactants, (b) one or more amphoteric surfactants, (c) one or more Zwitterionic surfactants, (d) one or more anionic surfactants and (e) one or more nonionic surfactants.

(75) In a variation, Organic Liquid Systems Two and high boiling point members of Organic Liquid System One serves as the delivery system or components of a delivery system that delivers the one or more nitrogenous phosphoryl compounds to the surface of a substrate,

(76) In a variation, said liquid solutions comprise one or more nitrogenous phosphoryl compounds at levels of 20-80% and wherein said liquid solution is fluid at (-25) to 55° C.

(77) ##STR00013## wherein: a) X is one or more members selected from the group consisting of i) P=O, ii) P, and iii) P=S b) Z is one or more members selected from the group consisting of i) one or more alkyl radicals selected from the group consisting of (a) —C.sub.1H.sub.3, (b) —C.sub.2H.sub.5, (c) —C.sub.3H.sub.7, (d) —C.sub.4H.sub.9, (e) —C.sub.5H.sub.11, (f) —C.sub.6H.sub.5, and g) H (hydrogen) c) R<sup>sup.18</sup> is one or more alkyl radicals selected from the group consisting of (a) —C.sub.1H.sub.3, (b) —C.sub.2H.sub.5, (c) —C.sub.3H.sub.7, (d) —C.sub.4H.sub.9, (e) —C.sub.5H.sub.11, and (f) —C.sub.6H.sub.5.

(78) In an embodiment, methods to make of one or more nitrogenous phosphoryl compounds that result in fluid suspensions compositions comprise a reaction of one or more phosphoryl chlorides with one or more alkylamines in an aprotic, nonreactive organic solvent medium in reaction vessel #1 according to methods to make known by those in the art.

(79) In a variation, said reaction medium is charged into reaction vessel #1 wherein said reaction medium comprises Organic Liquid System One, wherein said Organic Liquid System One comprises one or more non-phosphoryl chloride reactive aprotic organic liquids selected from the group consisting of one or more aprotic organic liquids with a boiling point greater than 120° C. selected from the group consisting of a) one or more alkylene carbonates selected from the group

consisting of i) ethylene carbonate, ii) propylene carbonate and iii) butylene carbonate b) one or more 1-alkyl-2-pyrrolidone selected from the group wherein the alkyl functionality is a C.sub.1-C.sub.8 alkyl radical, c) one or more hydrocarbons selected from the group consisting of i) one or more paraffinic liquids, ii) one or more cycloparaffinics, iii) one or more aromatic hydrocarbons, iv) one or more aliphatic hydrocarbons, v) one or more acyclic hydrocarbons, vi) one or more unsaturated hydrocarbon, and vii) one or more polyalphaolefins, d) one or more C.sub.1-C.sub.18 fatty triglycerides, e) one or more organo phosphorous liquids selected from the group consisting of hexamethylphosphoramide and one or more trialkylphosphates selected from the structure:

(80) ##STR00014## wherein: R.sup.22 is alkyl radical —C.sub.1H.sub.3 to —C.sub.6H.sub.13 R.sup.23 is alkyl radical —C.sub.1H.sub.3 to —C.sub.6H.sub.13 R.sup.24 is alkyl radical —C.sub.1H.sub.3 to —C.sub.6H.sub.13 f) sulfolane, g) 2-methoxyethyl ether, h) cyclohexylpyrrolidone, i) one or more polyols capped with C.sub.1-C.sub.6 organic acid wherein the polyol portion is selected from the group consisting of i) ethylene glycol, ii) 1,3 propylene glycol, iii) 1,2 propylene glycol, iv) butylene glycol, v) trimethylol propane, vi) trimethylol ethane, vii) pentaerythritol, viii) sorbitol, ix) sorbitan, x) glucose, xi) fructose, xii) galactose and xiii) glycerin, j) one or more alkylene glycol alkyl ethers acetates selected from the group consisting of (i) dipropylene glycol methyl ether acetate, (ii) tripropylene glycol methyl ether acetate, and (iii) tripropylene glycol butyl ether acetate, k) isophorone, l) one or more diesters selected from the group consisting of (i) C.sub.1-C.sub.6 dialkylsuccinate, (ii) C.sub.1-C.sub.6 dialkyladipate, (iii) C.sub.1-C.sub.6 dialkylmethylglutarate, (iv) C.sub.1-C.sub.6 dialkyl glutarate, (v) C.sub.1-C.sub.6 dialkyl ethylsuccinate, and (vi) C.sub.1-C.sub.6 dialkyl maleate, m) dimethyl-2-imidazolidinone, n) one or more fatty acid alkyl esters wherein the one or more fatty acid functionality is selected from the group consisting of C.sub.8-C.sub.18 fatty acids and wherein the alkyl functionality is one or more C.sub.1-C.sub.6 alkyl radicals, and o) one or more dialkylene glycols dialkyl ethers, (glymes) selected from the structure:

R.sup.11—O—(R.sup.12—O).sub.n—R.sup.13 wherein: R.sup.11 is one or more alkyl radicals selected from the group consisting of C.sub.1 to C.sub.6, R.sup.12 is one or more alkylene radicals selected from the group consisting of C.sub.1 to C.sub.4, R.sup.13 is one or more alkyl radicals selected from the group consisting of C.sub.1 to C.sub.6, and n is one or more integers selected from the group of 1 to 10.

(81) In another variation, said Organic Liquid System One comprises one or more non-phosphoryl chloride reactive aprotic organic liquids selected from the group consisting of aprotic organic liquids with a boiling point greater than 160° C.

(82) In another variation the composition of reaction vessel #1 further comprises one or more aprotic organic liquids with a boiling point less than or equal to 120° C.

(83) In a variation, reaction vessel #1 has been deoxygenated and is filled with an inert gas.

(84) In a variation, the contents of reaction vessel #1 are adjusted to a temperature range of (−40) to 20° C. In another variation, the contents of reaction vessel #1 are adjusted one or more temperature ranges of (−40) to (−20)° C., c) (−40) to 0° C., d) (−40) to (−10)° C., e) (−30) to 20° C., f) (−30) to 10° C., g) (−30) to (−10)° C., h) (−20) to 20° C., i) (−20) to 10° C., j) (−20) to 0° C., k) (−20) to (−10)° C., l) (−10) to 20° C., m) (−10) to 10° C., n) (−10) to 0° C., o) 0 to 20° C., and p) 0 to 10° C.,

(85) In a variation, the contents of reaction vessel #1 are under strong agitation.

(86) In a variation, said one or more alkylamines are slowly charged to reaction vessel #1.

(87) In a variation, the charging of said one or more alkylamines is above the surface and/or sub-surface of the contents in reaction vessel #1. In another variation, the rate of charging said one or more alkylamines to the contents of reaction vessel #1 is dependent on maintaining the temperature of the contents of reaction vessel #1 at less than 5° C., In another variation, the rate of charging said one or more alkylamines to the contents of reaction vessel #1 is dependent on maintaining the temperature of the contents of reaction vessel #1 at less than one or more of the following

temperatures i)  $(-20)^{\circ}\text{C.}$ , ii)  $(-15)^{\circ}\text{C.}$ , iii)  $(-10)^{\circ}\text{C.}$ , iv)  $(-5)^{\circ}\text{C.}$ , v)  $0^{\circ}\text{C.}$ , vi)  $5^{\circ}\text{C.}$ , and vii)  $<(-20)^{\circ}\text{C.}$ ,

(88) In a variation, said one or more alkylamines is premixed with a portion of said Organic Liquid System One and then slowly charged to reaction vessel #1,

(89) In a variation, said one or more phosphoryl chlorides comprise one or more phosphoryl trichlorides and wherein the molar ratio range of the one or more said alkylamines to the one or more said phosphoryl trichlorides is 0.7:1.5. In another variation, the molar ratio of said one or more alkylamines to said one or more phosphoryl trichlorides is selected from the group consisting of (a) 0.7:1.0, (b) 0.8:1.0, (c) 0.9:1.0, (d) 0.95:1.0, (e) 1.0:1.0, (f) 1.05:1.0, (g) 1.1:1.0, (h) 1.2:1.0, (i) 1.3:1.0, (j) 1.4:1.0, (k) 1.5:1.0, (l) 0.7:1.0 and m) 0.9:1.0,

(90) In a variation, the composition of the contents does not comprise one or more trialkylamines that are utilized as HCl absorbants.

(91) In a variation, after completing the charge of one or more alkylamines to reaction vessel #1, the contents of reaction vessel #1 are mixed for additional minutes comprising one or more time ranges selected from the group consisting of (1) 5-200 minutes, (2) 10-120 minutes, (3) 20-120 minutes, (4) 30-120 minutes, (5) 40-120 minutes, (6) 50-120 minutes, (7) 1-120 minutes, (8) 60-120 minutes, (9) 5-60 minutes, (10) 10-60 minutes, (11) 20-60 minutes, (12) 30-60 minutes, (13) 40-60 minutes, and (14) 50-60 minutes.

(92) In a variation, the temperature of reaction vessel #1's contents are maintained at  $(-40)$  to  $20^{\circ}\text{C.}$  In another variation, the contents of reaction vessel #1 are adjusted to one or more temperature ranges of  $(-40)$  to  $(-20)^{\circ}\text{C.}$ , c)  $(-40)$  to  $0^{\circ}\text{C.}$ , d)  $(-40)$  to  $(-10)^{\circ}\text{C.}$ , e)  $(-30)$  to  $20^{\circ}\text{C.}$ , f)  $(-30)$  to  $10^{\circ}\text{C.}$ , g)  $(-30)$  to  $(-10)^{\circ}\text{C.}$ , h)  $(-20)$  to  $20^{\circ}\text{C.}$ , i)  $(-20)$  to  $10^{\circ}\text{C.}$ , j)  $(-20)$  to  $0^{\circ}\text{C.}$ , k)  $(-20)$  to  $(-10)^{\circ}\text{C.}$ , l)  $(-10)$  to  $20^{\circ}\text{C.}$ , m)  $(-10)$  to  $10^{\circ}\text{C.}$ , n)  $(-10)$  to  $0^{\circ}\text{C.}$ , o)  $0$  to  $20^{\circ}\text{C.}$ , and p)  $0$  to  $10^{\circ}\text{C.}$

(93) In a variation, upon completing the charging of one or more alkylamines to the contents of reaction vessel #1, the ammonium functionality of the one or more alkyl ammonium chlorides is displaced through the addition one or more alkalis comprising one or more metal cations selected from the group consisting of (1) Na, (2) K, (3) Ca, (4) Mg, (5) Zn, (6) Mn, (7) Fe, (8) Cu, (9) Co, (10) Mo, and (11) Ni. In another variation, after the contents of reaction vessel #1 have mixed for an extra period of time, the agitation is slowed until the surface of the contents are slowly moving and then said one or more alkalis can be sifted onto the surface of the contents of reaction vessel #1.

(94) In an embodiment, the one or more alkylamines comprise one or more alkyl functionalities selected from the group consisting of i) one or more linear alkyl functionalities and ii) one or more branched alkyl functionalities and wherein the one or more i) linear alkyl functionalities and ii) branched alkyl functionalities are selected from the group consisting of (i) —C.sub.1H.sub.3, (ii) —C.sub.2H.sub.5, (iii) —C.sub.3H.sub.7, (iv) —C.sub.4H.sub.9, (v) —C.sub.5H.sub.11, (vi) —C.sub.6H.sub.5, and (vii) —C.sub.6H.sub.13

(95) In an embodiment, ammonia gas and/or ammonia generating compounds are charged to the contents of reaction vessel #1 under agitation.

(96) In a variation, said ammonia gas and/or ammonia generating compounds are charged very slowly, initially, to the contents of reaction vessel #1.

(97) In a variation, the charging of said ammonia gas and/or ammonia generating compound is above the surface and/or sub-surface of the said contents in reaction vessel #1.

(98) In a variation, the rate of charging said ammonia gas and/or ammonia generating compound is dependent on maintaining the temperature of the contents of reaction vessel #1 at less than  $100^{\circ}\text{C.}$  In another variation, the rate of charging said ammonia gas and/or ammonia generating compound is dependent on maintaining the temperature of the contents of reaction vessel #1 at less than one or more of the following temperatures i)  $\leq(-20)^{\circ}\text{C.}$ , ii)  $\leq(-15)^{\circ}\text{C.}$ , iii)  $\leq(-10)^{\circ}\text{C.}$ , iv)  $\leq(-5)^{\circ}\text{C.}$ , v)  $\leq(0)^{\circ}\text{C.}$ , f)  $\leq 5^{\circ}\text{C.}$ , vi)  $<(-20)^{\circ}\text{C.}$ , vii)  $\leq 5^{\circ}\text{C.}$ , viii)  $\leq 10^{\circ}\text{C.}$ , ix)  $\leq 15^{\circ}\text{C.}$ , x)  $\leq 20^{\circ}\text{C.}$ , xi)  $\leq 25^{\circ}\text{C.}$ , xii)  $\leq 30^{\circ}\text{C.}$ , xiii)  $\leq 40^{\circ}\text{C.}$ , xiv)  $\leq 50^{\circ}\text{C.}$ , xv)  $\leq 60^{\circ}\text{C.}$ , xvi)  $\leq 70^{\circ}\text{C.}$ , xvii)  $\leq 80^{\circ}\text{C.}$ , xviii)  $\leq 90^{\circ}\text{C.}$ ,

and xix)  $\leq 100^{\circ}\text{C}$ .

(99) In a variation, the rate of charge of ammonia gas and/or ammonia generating compound can be optionally slow in the beginning with very slow agitation.

(100) In a variation, the temperature of the contents of reaction vessel #1 are initially at  $\leq (-10)^{\circ}\text{C}$ . In another variation, the temperature of the contents of reaction vessel #1 are maintained at a temperature range of  $(-10)$  to  $5^{\circ}\text{C}$ . through the first  $\frac{1}{4}$  to  $\frac{1}{2}$  of the ammonia gas and/or ammonia generating compound, and allowed to slowly increase temperatures to a range of 30 to  $100^{\circ}\text{C}$ . for the remaining  $\frac{3}{4}$  to  $\frac{1}{2}$  of the remaining charge of ammonia gas and/or ammonia generating compound.

(101) In an embodiment, upon completion of the charging of ammonia gas and/or ammonia generating compound, the contents of reaction vessel #1 are mixed for an additional 5-120 additional minutes at a temperature range of  $30$ - $100^{\circ}\text{C}$ .

(102) In a variation, after completing the charge of ammonia and/or ammonia generating compounds, the contents of reaction vessel #1 are mixed for additional minutes comprising one or more time ranges selected from the group consisting of (1) 5-120 minutes, (2) 10-120 minutes, (3) 20-120 minutes, (4) 30-120 minutes, (5) 40-120 minutes, (6) 50-120 minutes, (7) 1-120 minutes, (8) 60-120 minutes, (9) 5-60 minutes, (10) 10-60 minutes, (11) 20-60 minutes, (12) 30-60 minutes, (13) 40-60 minutes, and (14) 50-60 minutes.

(103) In another variation, the temperature of reaction vessel #1's contents are maintained at one or more of the following temperature ranges (1) 30 to  $40^{\circ}\text{C}$ ., (2) 30 to  $50^{\circ}\text{C}$ ., (3) 30 to  $60^{\circ}\text{C}$ ., (4) 30 to  $70^{\circ}\text{C}$ ., (5) 30 to  $80^{\circ}\text{C}$ ., (6) 30 to  $90^{\circ}\text{C}$ ., (7) 30 to  $100^{\circ}\text{C}$ ., (8) 40 to  $60^{\circ}\text{C}$ ., (9) 40 to  $70^{\circ}\text{C}$ ., (10) 40 to  $80^{\circ}\text{C}$ ., (11) 40 to  $90^{\circ}\text{C}$ ., (12) 40 to  $100^{\circ}\text{C}$ ., (13) 50 to  $60^{\circ}\text{C}$ ., and (14) 50 to  $70^{\circ}\text{C}$ .

(104) In an embodiment, upon completion of the reaction of the contents of reaction vessel #1, the contents of reaction vessel #1 are cooled to  $<40^{\circ}\text{C}$ . and packaged.

(105) In a variation, upon completion of the reaction of the contents of reaction vessel #1, the ammonium functionality of the ammonium chloride is displaced through the addition one or more alkalis comprising one or more metal cations selected from the group consisting of (1) Na, (2) K, (3) Ca, (4) Mg, (5) Zn, (6) Mn, (7) Fe, (8) Cu, (9) Co, (10) Mo, and (11) Ni.

(106) In a variation, the one or more particles within the fluid suspension of nitrogenous phosphoryl compounds comprise (1) one or more chloride salts, (2) one or more nitrogenous phosphoryl compounds, and (3) one or more in situ "binary fused particles" of one or more chloride salts and one or more nitrogenous phosphoryl compounds.

(107) In a variation, upon completion of the reaction of the contents of reaction vessel #1, a colorant is optionally added to the contents of reaction vessel #1,

(108) In a variation, upon completion of the reaction of the contents of reaction vessel #1, a scent is optionally added to the contents of reaction vessel #1,

(109) In a variation, upon completion of the reaction of the contents of reaction vessel #1, a buffer is optionally added to the contents of reaction vessel #1,

(110) In a variation, upon completion of the reaction of the contents of reaction vessel #1, Organic Liquid System Two is optionally added to the contents of reaction vessel #1.

(111) In a variation, upon completion of the reaction of the contents of reaction vessel #1, one or more surfactants are added to the contents of reaction vessel #1, wherein the one or more surfactants comprises one or more members selected from the group consisting of (a) one or more cationic surfactants, (b) one or more amphoteric surfactants, (c) one or more Zwitterionic surfactants, (d) one or more anionic surfactants and (e) one or more nonionic surfactants.

(112) In a variation, said Organic Liquid System Two comprises one or more organic liquids selected from the group consisting of (1) aprotic organic liquids and (2) protic organic liquids wherein one or more of said aprotic organic liquids are selected from the group consisting of: (a) dimethyl sulfoxide, (b) one or more sulfoxide(s) selected from the group consisting of dialkyl, diaryl, or alkylaryl sulfoxide(s) selected from the formula structure:

R.sup.9S(O)xR.sup.10 wherein (i) R.sup.9 and R.sup.10 are each independently a C.sub.1-C.sub.6 alkylene group, an aryl group or C.sub.1-C.sub.3 alkylenearyl group, (ii) or R.sup.9 and R.sup.10 with the sulfur to which they are attached form a 4 to 8 membered ring wherein R.sup.9 and R.sup.10 together are a C.sub.1-C.sub.6 alkylene group which optionally contains one or more atoms selected from the group consisting of O, S, Se, Te, N, and P in the ring, (iii) and x is 1 or 2, (c) one or more alkylene carbonates selected from the group consisting of (i) ethylene carbonate, (ii) propylene carbonate, and (iii) butylene carbonate, (d) one or more polyols capped with C.sub.1-C.sub.6 organic acid wherein the polyol portion is selected from the group consisting of (i) ethylene glycol, (ii) 1,3 propylene glycol, (iii) 1,2 propylene glycol, (iv) butylene glycol, (v) trimethylol propane, (vi) trimethylol ethane, (vii) pentaerythritol, (viii) sorbitol, (ix) sorbitan, (x) glucose, (xi) fructose, (xii) galactose and (xiii) glycerin, (e) one or more alkylene glycol alkyl ethers acetates selected from the group consisting of (i) dipropylene glycol methyl ether acetate, (ii) tripropylene glycol methyl ether acetate, and (iii) tripropylene glycol butyl ether acetate, (f) one or more diesters selected from the group consisting of (ii) C.sub.1-C.sub.6 dialkyladipate, (iii) C.sub.1-C.sub.6 dialkylmethylglutarate, (iv) C.sub.1-C.sub.6 dialkyl glutarate, (v) C.sub.1-C.sub.6 dialkyl ethylsuccinate, and (vi) C.sub.1-C.sub.6 dialkyl maleate, (g) one or more 1-alkyl-2-pyrrolidone selected from the group wherein the alkyl functionality is a C.sub.1-C.sub.8 alkyl radical, (h) one or more members selected from the group consisting of (i) dimethylacetamide, (ii) dimethylformamide, (iii) dimethyl-2-imidazolidinone, (iv) isophorone, (v) hexamethylphosphoramide, (vi) 2-methoxyethyl ether, (vii) N,N-dimethyldecanamide, (viii) N,N-dimethyloctanamide, (ix) limonene, and (x) sulfolane (i) one or more trialkylphosphates selected from the group represented by the structure:

(113) ##STR00015## wherein: R.sup.22 is alkyl radical —C.sub.1H.sub.3 to —C.sub.6H.sub.13 R.sup.23 is alkyl radical —C.sub.1H.sub.3 to —C.sub.6H.sub.13 R.sup.24 is alkyl radical —C.sub.1H.sub.3 to —C.sub.6H.sub.13 (j) one or more dialkylene glycols dialkyl ethers, (glymes) selected from the structure:

R.sup.11—O—(R.sup.12—O).sub.n—R.sup.13 wherein: R.sup.11 is one or more alkyl radicals selected from the group consisting of C.sub.1 to C.sub.6, R.sup.12 is one or more alkylene radicals selected from the group consisting of C.sub.1 to C.sub.4, R.sup.13 is one or more alkyl radicals selected from the group consisting of C.sub.1 to C.sub.6, and n is one or more integers selected from the group of 1 to 10, (k) one or more hydrocarbons selected from the group consisting of (i) one or more paraffinic liquids, (ii) one or more cycloparaffinics, (iii) one or more aromatic hydrocarbons, (iv) aliphatic hydrocarbons, (v) acyclic hydrocarbons, (vi) unsaturated hydrocarbon, and (vii) polyalphaolefins, (l) one or more fatty acid alkyl esters wherein the one or more fatty acid functionality is selected from the group consisting of C.sub.8-C.sub.18 fatty acids and wherein the alkyl functionality is one or more C.sub.1-C.sub.6 alkyl radicals, and (m) one or more C.sub.1-C.sub.18 fatty triglycerides, and wherein said protic organic liquid is one or more members selected from the group consisting of: (a) one or more alcohols selected from the group consisting of the family of C.sub.1-C.sub.10 alkanols, (b) one or more polyols selected from the group consisting of (i) trimethylol propane, (ii) trimethylol ethane, (iii) pentaerythritol, (iv) sorbitol, (v) sorbitan, (vi) glucose, (vii) fructose, (viii) galactose, and (ix) glycerin, (c) one or more polyalkylene glycols selected from the group consisting of poly(C.sub.1-C.sub.10 alkylene) glycols, (d) one or more dioxolane compound selected from the group consisting of (i) 2,2-dimethyl-1,3-dioxolane-4-methanol and (ii) 2,2-diisobutyl-1,3-dioxolane-4-methanol, e) one or more alkylene glycol alkyl ethers selected from the structure:

(114) ##STR00016## wherein R.sup.14 is one or more members selected from the group consisting of CH.sub.3, C.sub.2H.sub.5, C.sub.3H.sub.7, C.sub.6H.sub.5, and C.sub.4H.sub.9, R.sup.15 is one or more members selected from the group consisting of H and.

(115) ##STR00017## wherein R.sup.17 is one or more members selected from the group consisting of H and CH.sub.3, and f is an integer between 1 and 15, and wherein R.sup.16 is one

or more members selected from the group consisting of H and CH.sub.3, (f) one or more alkyl lactates selected from the group consisting of (i) ethyl lactate, (ii) propyl lactate and (iii) butyl lactate, (g) one or more alkanolamines selected from the group consisting of alkanolamines selected from the formula structure:

(116) ##STR00018## wherein R.sup.5 is one or more members selected from the group consisting of CH.sub.3, C.sub.2H.sub.5, C.sub.2H.sub.4OR.sup.8, C(CH.sub.3).sub.2CH.sub.2OH, CH(C.sub.2H.sub.5)CH.sub.2OH, CH.sub.3CH(OH)CH.sub.2, and C.sub.3H.sub.6OH, R.sup.6 is one or more members selected from the group consisting of H, CH.sub.3, C.sub.2H.sub.5, C.sub.2H.sub.4OR.sup.8 and C.sub.3H.sub.6OH, R.sup.7 is one or more members selected from the group consisting of H, C.sub.2H.sub.4OR.sup.8 and C.sub.3H.sub.6OH, and wherein R.sup.8 is (C.sub.2H.sub.4O).sub.gH wherein g is an integer between 1-10, (h) and glycerol carbonate.

(117) In a variation, said one or more cationic surfactants comprises one or more members selected from the group consisting of (a) isostearylamine propyl ammonium chloride, (b) olealkonium chloride, (c) triethylmethylammonium chloride, (d) methyl triethanol ammonium methyl sulfate, (e) ethyl triethanol ammonium ethyl sulfate, (f) coconut trimethylammonium chloride, (g) coconut trimethylammonium methyl sulfate, (h) coconut dimethylethylammonium ethyl sulfate, (f) lauryl trimethylammonium chloride, (g) lauryl trimethylammonium methyl sulfate, (h) lauryl dimethylethylammonium ethyl sulfate, (i) one or more fatty acid amido propyl trimethyl ammonium chloride wherein the one or more fatty acids is selected from the group consisting of (1) capric acid, (2) caprylic acid, (3) lauric acid, (4) isostearic acid, and (5) oleic acid, (j) one or more fatty acid amido propyl trimethyl ammonium methyl sulfates wherein the one or more fatty acids is selected from the group consisting of (1) capric acid, (2) caprylic acid, (3) lauric acid, (4) isostearic acid, and (5) oleic acid, (k) one or more fatty acid amido propyl ethyl dimethyl ammonium ethyl sulfate wherein the one or more fatty acids is selected from the group consisting of (1) capric acid, (2) caprylic acid, (3) lauric acid, (4) isostearic acid, and (5) oleic acid, (m) methyl triethanol ammonium chloride, (n) N-ethyl-N,N-dipolyethoxyethyl-N-tallowalkylammonium ethosulfate, (o) N-methyl-N,N-dipolyethoxyethyl-N-tallowalkylammonium methosulfate, and (p) N-methyl-N,N-dipolyethoxyethyl-N-tallowalkylammonium chloride.

(118) In a variation, one or more nonionic surfactants comprises (1) one or more alkoxy functional groups wherein, alkoxy refers to the reaction of one or more alkoxides selected from the group consisting of (a) ethylene oxide, (b) propylene and (c) mixtures thereof with one or more functional groups wherein said one or more functional groups are selected from the group consisting of (a) one or more alkanolamines, (b) sorbitan monooleate, (c) sorbitan trioleate, (d) one or more alcohols wherein the one or more alcohols are selected from the group consisting of (i) octanol, (ii) isooctanol, (iii) 2-ethyl hexanol, (iv) nonanol, (v) decanol, (vi) isodecanol, (vii) undecanol, (viii) lauryl alcohol, (ix) tridecyl alcohol, (x) oleyl alcohol, (xi) linoleyl alcohol, (xii) linolenyl alcohol, and mixtures thereof, (e) tallow amine, (f) lauryl amine, (g) oleyl amine and (h) one or more fatty acids wherein the one or more fatty acids are selected from the group consisting of (i) capric acid, (ii) caprylic acid, (iii) lauric acid, (iv) oleic acid, (v) isostearic acid, (vi) linoleic acid, (vii) linolenic acid, (viii) ricinoleic acid, and mixtures thereof, (2) one or more alkanolamines, (3) sorbitan monooleate, (4) sorbitan trioleate, and (5) one or more ethylene oxide/propylene oxide block copolymers.

(119) In a variation, one or more anionic surfactants comprises (1) one or more alkyl diphenyl oxide disulfonates, (2) linear alkylbenzene sulfonates, (3) one or more alpha olefin sulfonates, (4) alkyl sulfates, (5) alkyl ethoxy sulfates, (6) alkyl sulfonates, (7) alkyl alkoxy carboxylates, (8) monoalkyl phosphates, (9) dialkyl phosphates, (10) sarcosinates, (11) sulfosuccinates, (12) isethionates, and (13) taurates.

(120) In an embodiment, the present invention relates to a method to make one or more nitrogenous phosphoryl compounds, wherein said one or more nitrogenous phosphoryl compounds comprise one or more alkylphosphoric triamides selected from Structure I:

(121) ##STR00019## wherein: a) R.sup.2 is one or more members selected from the group consisting of CH.sub.3, C.sub.2H.sub.5, C.sub.3H.sub.7, C.sub.4H.sub.9, C.sub.5H.sub.11, C.sub.6H.sub.13, C.sub.6H.sub.5, C.sub.7H.sub.7, OX', OCH.sub.3, OC.sub.2H.sub.5, OC.sub.3H.sub.7, OC.sub.4H.sub.9, OC.sub.5H.sub.11, OC.sub.6H.sub.13, and HNR.sup.4, wherein i) R.sup.4 is one or more members selected from the group consisting of H, CH.sub.3, C.sub.2H.sub.5, C.sub.3H.sub.7, C.sub.4H.sub.9, C.sub.5H.sub.11, C.sub.6H.sub.5, and C.sub.6H.sub.13, ii) X.sup.1 is one or more members selected from the group consisting of (1) H, Na, Li, and K, (2) NH.sub.4, (3) one or more organoamines selected from the group consisting of mono C.sub.1-6 amine, di C.sub.1-6 amine, tri C.sub.1-6 amine, mono ethanol amine, diethanol amine, triethanol amine, monoisopropanol amine, diisopropanol amine, triisopropanol amine, ethylene diamine diethylene triamine, triethylene tetraamine, and tetraethylene pentamine, b) R.sup.1 is one or more members selected from the group consisting of CH.sub.3, C.sub.2H.sub.5, C.sub.3H.sub.7, C.sub.4H.sub.9, C.sub.5H.sub.11 and C.sub.6H.sub.13, c) R.sup.3 is one or more members selected from the group consisting of H, CH.sub.3, C.sub.2H.sub.5, C.sub.3H.sub.7, C.sub.4H.sub.9, C.sub.5H.sub.11 and C.sub.6H.sub.13, d) Z.sup.1 is Oxygen or Sulfur, wherein said method to make one or more alkylphosphoric triamides results in liquid solution compositions wherein said method to make comprises the following steps: a) reaction of one or more phosphoryl chlorides, wherein said one or more phosphoryl chlorides comprise one or more phosphoryl trichlorides selected from the structure:

PZ.sup.1Cl.sub.3, wherein Z.sup.1 is one or more members selected from the group consisting of Oxygen and Sulfur, with one or more alkylamines in a reaction medium wherein said reaction medium comprises Organic Liquid System One resulting in one or more alkyl dichlorophosphoryl amides selected from the structure

R.sup.1PZ.sup.1Cl.sub.2 wherein R.sup.1 is one or more members selected from the group consisting of CH.sub.3, C.sub.2H.sub.5, C.sub.3H.sub.7, C.sub.4H.sub.9, C.sub.5H.sub.11 and C.sub.6H.sub.13 and one or more alkyl ammonium chlorides, b) reaction of said one or more alkyl ammonium chlorides with ammonia and/or one or more ammonia generating compounds to generate ammonium chloride and one or more alkylamines for reaction with one or more phosphoryl chlorides, c) reaction of said one or more alkyl dichlorophosphoryl amides with ammonia and/or one or more ammonia generating compounds to generate ammonium chloride and the one or more alkylphosphoric triamides of Structure I, d) removal of ammonium chloride by-products by one or more methods selected from the group consisting of i) filtration, ii) sedimentation and removal of organic layer, (1) optionally utilizing ultrasonic probes to enhance sedimentation iii) liquefaction of the inorganic salt by-products and subsequent draining off of by-products by one or more methods selected from the group consisting of: (1) liquefaction of ammonium chloride with excess ammonia and pressurization of the reaction vessel #1, (2) liquefaction of chloride by-products with water addition which has been optionally de-oxygenated, optionally deionized and/or optionally distilled, (3) combinations thereof, e) consolidation of the organic composition and removal of the volatile components of said Organic Liquid System One by one or more methods selected from the group consisting of i) reduced pressure, ii) heating the organic composition to less than 100° C. and iii) combinations thereof, f) replacing the volatile components of Organic Liquid System One with Organic Liquid System Two by one or more methods selected from the group consisting of i) during the process of removal of the volatile components of Organic Liquid System One, ii) after the process of removal of the volatile components of Organic Liquid System One and iii) combinations thereof, resulting in said one or more alkylphosphoric triamides compounds in liquid solution compositions, wherein said liquid solutions comprise 40-70% wt. of said one or more alkylphosphoric triamide compounds and wherein said liquid solution compositions are fluid at -20° C., wherein said Organic Liquid System One comprises one or more aprotic organic liquids that are non-reactive with phosphoryl chloride functional groups and wherein one or more ammonia

generating compounds are selected from the group consisting of a) ammonium carbonate and b) ammonium bicarbonate.

(122) In an embodiment, said one or more phosphoryl chlorides comprises PSCl.sub.3 and wherein said PSCl.sub.3 reacts with said one or more alkylamines and with ammonia to yield said one or more alkylphosphoric triamides selected from Structure I:

(123) ##STR00020## wherein: a) R.sup.2 is HNR.sup.4, wherein i) R.sup.4 is H, b) le is C.sub.4H.sub.9, wherein C.sub.4H.sub.9 is a linear alkyl functionality, c) R.sup.3 is H, and d) Z.sup.1 is Sulfur.

(124) In an embodiment, said Organic Liquid System One comprises one or more aprotic liquids that are not reactive with one or more phosphoryl chlorides, and wherein said one or more aprotic liquids are selected from the group consisting of a) one or more aprotic liquids that have a boiling point equal to or below 120° C., b) one or more aprotic liquids that have a boiling point above 120° C., and c) mixtures thereof, and wherein said one or more aprotic liquids that have a boiling point equal to or below 120° C. comprise one or more aprotic liquids selected from the group consisting of a) one or more hydrocarbons selected from the group consisting of i) one or more paraffinic liquids, ii) one or more cycloparaffinics, and iii) one or more aromatic hydrocarbons, b) one or more liquid halocarbons c) one or more halohydrocarbons, and d) one or more ethers selected from the group consisting of i) 1,4-dioxane, ii) 1,3-dioxolane, iii) methyltetrahydrofuran, iv) dimethoxyethane, v) 1,3-dioxane, vi) 1,3-dioxolane, vii) 2,2-dimethyl-1,3-dioxolane, viii) diethyl ether, ix) tetrahydrofuran, and x) tetrahydropyran, e) d-limonene, f) 1,2-dimethoxyethane and f) one or more esters selected from the group consisting of i) C.sub.1-C.sub.6 alkylformate and ii) C.sub.1-C.sub.6 alkylacetate.

(125) In an embodiment, said reaction medium comprising said Organic Liquid System One, wherein said Organic Liquid System One comprises one or more aprotic solvents with a boiling point greater than 120° C. selected from the group consisting of: a) one or more alkylene carbonates selected from the group consisting of i) ethylene carbonate, ii) propylene carbonate, and iii) butylene carbonate, b) one or more polyols capped with C.sub.1-C.sub.6 organic acid wherein the polyol portion is selected from the group consisting of i) ethylene glycol, ii) 1,3 propylene glycol, iii) 1,2 propylene glycol, iv) butylene glycol, v) trimethylol propane, vi) trimethylol ethane, vii) pentaerythritol, viii) sorbitol, ix) sorbitan, x) glucose, xi) fructose, xii) galactose and xiii) glycerin, c) one or more alkylene glycol alkyl ethers acetates selected from the group consisting of i) dipropylene glycol methyl ether acetate, ii) tripropylene glycol methyl ether acetate, and iii) tripropylene glycol butyl ether acetate, d) one or more diesters selected from the group consisting of i) C.sub.1-C.sub.6 dialkylsuccinate, ii) C.sub.1-C.sub.6 dialkyladipate, iii) C.sub.1-C.sub.6 dialkylmethylglutarate, iv) C.sub.1-C.sub.6 dialkyl glutarate, v) C.sub.1-C.sub.6 dialkyl ethylsuccinate, and vi) C.sub.1-C.sub.6 dialkyl maleate, e) one or more 1-alkyl-2-pyrrolidone selected from the group wherein the alkyl functionality is a C.sub.1-C.sub.8 alkyl radical, f) one or more members selected from the group consisting of i) dimethylacetamide, ii) dimethylformamide, iii) dimethyl-2-imidazolidinone, iv) isophorone, v) hexamethylphosphoramide, vi) 2-methoxyethyl ether, vii) N,N-dimethyldecanamide, viii) N,N-dimethyloctanamide, ix) limonene, x) sulfolane g) one or more trialkylphosphates selected from the group represented by the structure:

(126) ##STR00021## wherein: R.sup.22 is alkyl radical —C.sub.1H.sub.3 to —C.sub.6H.sub.13 R.sup.23 is alkyl radical —C.sub.1H.sub.3 to —C.sub.6H.sub.13 R.sup.24 is alkyl radical —C.sub.1H.sub.3 to —C.sub.6H.sub.13 h) one or more dialkylene glycols dialkyl ethers, (glymes) selected from the structure:

R.sup.11—O—(R.sup.12—O).sub.n—R.sup.13 wherein: R.sup.11 is one or more alkyl radicals selected from the group consisting of C.sub.1 to C.sub.6, R.sup.12 is one or more alkylene radicals selected from the group consisting of C.sub.1 to C.sub.4, R.sup.13 is one or more alkyl radicals selected from the group consisting of C.sub.1 to C.sub.6, and n is one or more integers selected from the group of 1 to 10, i) one or more hydrocarbons selected from the group consisting of i) one



or more paraffinic liquids, ii) one or more cycloparaffinics, iii) one or more aromatic hydrocarbons, iv) aliphatic hydrocarbons, v) acyclic hydrocarbons, vi) unsaturated hydrocarbon, and vii) polyalphaolefins, j) one or more fatty acid alkyl esters wherein the one or more fatty acid functionality is selected from the group consisting of C.sub.8-C.sub.18 fatty acids and wherein the alkyl functionality is one or more C.sub.1-C.sub.6 alkyl radicals, and k) one or more C.sub.1-C.sub.18 fatty triglycerides.

(127) In an embodiment, the molar ratio of the one or more said alkylamines to the one or more said phosphoryl trichlorides is 0.7 to 1.5:1.

(128) In an embodiment, the temperature of the reaction of one or more phosphoryl chlorides with one or more alkylamines is (-40) to 20° C.

(129) In an embodiment, said Organic Liquid System One is charged to a reaction vessel and the temperature of the contents of said reaction vessel is cooled to (-40) to 20° C. and then said one or more phosphoryl trichlorides are charged to said reaction vessel and cooled to (-40) to 20° C.

(130) In an embodiment, said one or more alkylamines are charged slowly and sub-surface of the composition comprising said a) Organic Liquid System One and one or more phosphoryl trichlorides, and wherein the agitation of the contents of said reaction vessel is at medium to high speed resulting in a composition comprising a) Organic Liquid System One, b) said one or more phosphoryl trichlorides, c) said one or more alkyl dichlorophosphoryl amides and d) one or more chloride salts of said one or more alkylamines.

(131) In an embodiment, the one or more chloride salts of said one or more alkylamines are reacted with ammonia freeing the one or more alkylamines to react with said phosphoryl trichlorides.

(132) In an embodiment, the temperature of contents of said reaction vessel are maintained at less than 0° C.

(133) In an embodiment, initially a very slow agitation speed of the contents of said reaction vessel is selected so as to reduce the mixing of the surface of the contents of said reaction vessel to low, wherein said ammonia is slowly charged to the headspace of said reaction vessel to react with said one or more alkyl ammonium chlorides.

(134) In an embodiment, a temperature of contents of said reaction vessel are maintained at less than 0° C.

(135) In an embodiment, after about ¼ the total ammonia charge is completed, the agitation speed is then increased to medium or high and the temperature of the contents of said reaction vessel are slowly allowed to increase to less than 100° C., and wherein the temperature is controlled by cooling medium and by addition rate of ammonia and wherein the addition of ammonia results in the consumption of said one or more phosphoryl chlorides.

(136) In an embodiment, the method to make further comprises an addition to the contents of the reaction vessel of one or more members selected from the group consisting of: a) a colorant, b) a scent, c) a buffer, d) one or more surfactants, wherein the one or more surfactants comprises one or more members selected from the group consisting of (1) one or more cationic surfactants, (2) one or more amphoteric surfactants, (3) one or more Zwitterionic surfactants, (4) one or more anionic surfactants and (5) one or more nonionic surfactants.

(137) In an embodiment, the method to make does not comprise one or more of the following steps: a) recovery and purification of solid said one or more alkylphosphoric triamides compounds, b) packaging of solid said one or more alkylphosphoric triamides, c) shipping and storage of solid said one or more alkylphosphoric triamides, and/or d) dispersing of the solid said one or more alkylphosphoric triamides in a liquid delivery system.

(138) In an embodiment, said Organic Liquid System Two comprises: a) dimethyl sulfoxide, b) one or more sulfoxide(s) selected from the group consisting of dialkyl, diaryl, or alkylaryl sulfoxide(s) selected from the formula structure:

R.sup.9S(O)xR.sup.10 wherein i) R.sup.9 and R.sup.10 are each independently a C.sub.1-C.sub.6 alkylene group, an aryl group or C.sub.1-C.sub.3 alkylenearyl group, ii) or R.sup.9 and R.sup.10

with the sulfur to which they are attached form a 4 to 8 membered ring wherein R.sup.9 and R.sup.10 together are a C.sub.1-C.sub.6 alkylene group which optionally contains one or more atoms selected from the group consisting of O, S, Se, Te, N, and P in the ring, iii) and x is 1 or 2, c) one or more alkylene carbonates selected from the group consisting of i) ethylene carbonate, ii) propylene carbonate, and iii) butylene carbonate, d) one or more polyols capped with C.sub.1-C.sub.6 organic acid wherein the polyol portion is selected from the group consisting of i) ethylene glycol, ii) 1,3 propylene glycol, iii) 1,2 propylene glycol, iv) butylene glycol, v) trimethylol propane, vi) trimethylol ethane, vii) pentaerythritol, viii) sorbitol, ix) sorbitan, x) glucose, xi) fructose, xii) galactose and xiii) glycerin, e) one or more alkylene glycol alkyl ethers acetates selected from the group consisting of i) dipropylene glycol methyl ether acetate, ii) tripropylene glycol methyl ether acetate, and iii) tripropylene glycol butyl ether acetate, f) one or more diesters selected from the group consisting of i) C.sub.1-C.sub.6 dialkylsuccinate, ii) C.sub.1-C.sub.6 dialkyladipate, iii) C.sub.1-C.sub.6 dialkylmethylglutarate, iv) C.sub.1-C.sub.6 dialkyl glutarate, v) C.sub.1-C.sub.6 dialkyl ethylsuccinate, and vi) C.sub.1-C.sub.6 dialkyl maleate, g) one or more 1-alkyl-2-pyrrolidone selected from the group wherein the alkyl functionality is a C.sub.1-C.sub.8 alkyl radical, h) one or more members selected from the group consisting of i) dimethylacetamide, ii) dimethylformamide, iii) dimethyl-2-imidazolidinone, iv) isophorone, v) hexamethylphosphoramide, vi) 2-methoxyethyl ether, vii) N,N-dimethyldecanamide, viii) N,N-dimethyloctanamide, ix) limonene, x) sulfolane i) one or more trialkylphosphates selected from the group represented by the structure:

(139) ##STR00022## wherein: R.sup.22 is alkyl radical —C.sub.1H.sub.3 to —C.sub.6H.sub.13 R.sup.23 is alkyl radical —C.sub.1H.sub.3 to —C.sub.6H.sub.13 R.sup.24 is alkyl radical —C.sub.1H.sub.3 to —C.sub.6H.sub.13 j) one or more dialkylene glycols dialkyl ethers, (glymes) selected from the structure:

R.sup.11—O—(R.sup.12—O).sub.n—R.sup.13 wherein: R.sup.11 is one or more alkyl radicals selected from the group consisting of C.sub.1 to C.sub.6, R.sup.12 is one or more alkylene radicals selected from the group consisting of C.sub.1 to C.sub.4, R.sup.13 is one or more alkyl radicals selected from the group consisting of C.sub.1 to C.sub.6, and n is one or more integers selected from the group of 1 to 10, k) one or more hydrocarbons selected from the group consisting of i) one or more paraffinic liquids, ii) one or more cycloparaffinics, iii) one or more aromatic hydrocarbons, iv) aliphatic hydrocarbons, v) acyclic hydrocarbons, vi) unsaturated hydrocarbon, and vii) polyalphaolefins, l) one or more fatty acid alkyl esters wherein the one or more fatty acid functionality is selected from the group consisting of C.sub.8-C.sub.18 fatty acids and wherein the alkyl functionality is one or more C.sub.1-C.sub.6 alkyl radicals, and m) one or more C.sub.1-C.sub.18 fatty triglycerides,

and wherein said protic organic liquid is one or more members selected from the group consisting of: a) one or more alkanols selected from the group consisting of C.sub.1-C.sub.10 alkanols, b) one or more polyols selected from the group consisting of i) trimethylol propane, ii) trimethylol ethane, iii) pentaerythritol, iv) sorbitol, v) sorbitan, vi) glucose, vii) fructose, viii) galactose, and ix) glycerin, c) one or more polyalkylene glycols selected from the group consisting of poly(C.sub.1-C.sub.10 alkylene) glycols, d) one or more dioxolane compounds selected from the group consisting of i) 2,2-dimethyl-1,3-dioxolane-4-methanol and ii) 2,2-diisobutyl-1,3-dioxolane-4-methanol, e) one or more alkylene glycol alkyl ethers selected from the structure:

(140) ##STR00023## wherein R.sup.14 is one or more members selected from the group consisting of CH.sub.3, C.sub.2H.sub.5, C.sub.3H.sub.7, C.sub.6H.sub.5, and C.sub.4H.sub.9, R.sup.15 is one or more members selected from the group consisting of H and

(141) ##STR00024## wherein R.sup.17 is one or more members selected from the group consisting of H and CH.sub.3, and f is an integer between 1 and 15, and R.sup.16 is one or more members selected from the group consisting of H and CH.sub.3, f) one or more alkyl lactates selected from the group consisting of i) ethyl lactate, ii) propyl lactate and iii) butyl lactate, g) one

or more alkanolamines selected from the group consisting of alkanolamines selected from the structure:

(142) ##STR00025## wherein R.sup.5 is one or more members selected from the group consisting of CH.sub.3, C.sub.2H.sub.5, C.sub.2H.sub.4OR.sup.8, C(CH.sub.3).sub.2CH.sub.2OH, CH(C.sub.2H.sub.5)CH.sub.2OH, CH.sub.3CH(OH)CH.sub.2, and C.sub.3H.sub.6OH, R.sup.6 is one or more members selected from the group consisting of H, CH.sub.3, C.sub.2H.sub.5, C.sub.2H.sub.4OR.sup.8 and C.sub.3H.sub.6OH, R.sup.7 is one or more members selected from the group consisting of H, C.sub.2H.sub.4OR.sup.8 and C.sub.3H.sub.6OH, wherein R.sup.8 is (C.sub.2H.sub.4O).sub.gH wherein g is an integer between 1 and 10, h) and glycerol carbonate.

(143) In an embodiment, the invention relates to a method to make one or more nitrogenous phosphoryl compounds, wherein said one or more nitrogenous phosphoryl compounds comprise one or more alkylphosphoric triamides selected from the Structure I:

(144) ##STR00026## wherein: a) R.sup.2 is one or more members selected from the group consisting of CH.sub.3, C.sub.2H.sub.5, C.sub.3H.sub.7, C.sub.4H.sub.9, C.sub.5H.sub.11, C.sub.6H.sub.13, C.sub.6H.sub.5, C.sub.7H.sub.7, OX', OCH.sub.3, OC.sub.2H.sub.5, OC.sub.3H.sub.7, OC.sub.4H.sub.9, OC.sub.5H.sub.11, OC.sub.6H.sub.13, and HNR.sup.4, wherein i) R.sup.4 is one or more members selected from the group consisting of H, CH.sub.3, C.sub.2H.sub.5, C.sub.3H.sub.7, C.sub.4H.sub.9, C.sub.5H.sub.11, C.sub.6H.sub.5, and C.sub.6H.sub.13, ii) X.sup.1 is one or more members selected from the group consisting of (1) H, Na, Li, and K, (2) NH.sub.4, (3) one or more organoamines selected from the group consisting of mono C.sub.1-6 amine, di C.sub.1-6 amine, tri C.sub.1-6 amine, mono ethanol amine, diethanol amine, triethanol amine, monoisopropanol amine, diisopropanol amine, triisopropanol amine, ethylene diamine diethylene triamine, triethylene tetraamine, and tetraethylene pentamine, b) R.sup.1 is one or more members selected from the group consisting of CH.sub.3, C.sub.2H.sub.5, C.sub.3H.sub.7, C.sub.4H.sub.9, C.sub.5H.sub.11 and C.sub.6H.sub.13, c) R.sup.3 is one or more members selected from the group consisting of H, CH.sub.3, C.sub.2H.sub.5, C.sub.3H.sub.7, C.sub.4H.sub.9, C.sub.5H.sub.11 and C.sub.6H.sub.13, d) Z.sup.1 is one or more members selected from the group consisting of Oxygen and Sulfur,

wherein said method to make one or more alkylphosphoric triamides compounds results in fluid compositions, wherein said fluid composition comprises one or more physical states selected from the group consisting of a) colloid and b) suspension, wherein said method to make comprises the following steps: a) reaction of one or more phosphoryl chlorides, wherein said one or more phosphoryl chlorides comprise one or more phosphoryl trichlorides selected from the structure: PZ.sup.1Cl.sub.3 wherein Z.sup.1 is one or more members selected from the group consisting of Oxygen and Sulfur, with one or more alkylamines in a reaction medium wherein said reaction medium comprises Organic Liquid System One resulting in one or more alkyl dichlorophosphoryl amides selected from the structure

R.sup.1PZ.sup.1Cl.sub.2 wherein R.sup.1 is one or more members selected from the group consisting of CH.sub.3, C.sub.2H.sub.5, C.sub.3H.sub.7, C.sub.4H.sub.9, C.sub.5H.sub.11 and C.sub.6H.sub.13 and one or more alkyl ammonium chlorides, b) reaction of said one or more alkyl ammonium chlorides with ammonia and/or one or more ammonia generating compounds to generate ammonium chloride and the one or more alkylamines for reaction with one or more phosphoryl chlorides, c) reaction of said one or more alkyl dichlorophosphoryl amides with ammonia and/or one or more ammonia generating compounds to generate ammonium chloride and said one or more alkylphosphoric triamides compounds in fluid compositions, wherein said fluid compositions comprise one or more physical states selected from the group consisting of a) colloid and b) suspensions, wherein said fluid compositions comprise 40-70% wt. of said one or more alkylphosphoric triamide compounds and wherein said fluid compositions are fluid at -20° C., wherein said Organic Liquid System One comprises one or more aprotic organic liquids that are

non-reactive with phosphoryl chloride functional groups and have a boiling point above 120° C. and wherein one or more ammonia generating compounds are selected from the group consisting of a) ammonium carbonate and b) ammonium bicarbonate.

(145) In an embodiment, said one or more phosphoryl chlorides comprises PSCl.sub.3, and wherein said PSCl.sub.3 reacts with said one or more alkylamines and with ammonia to yield said one or more alkylphosphoric triamides selected from Structure I:

(146) ##STR00027## wherein: a) R.sup.2 is HNR.sup.4, wherein i) R.sup.4 is H, b) R.sup.1 is C.sub.4H.sub.9, wherein C.sub.4H.sub.9 is a linear alkyl functionality, c) R.sup.3 is H, and d) Z.sup.1 is Sulfur,

(147) In an embodiment, the reaction medium comprises said Organic Liquid System One, wherein said Organic Liquid System One comprises one or more aprotic liquids that are not reactive with one or more phosphoryl chlorides, wherein said one or more aprotic liquids have a boiling point above 120° C. are selected from the group consisting of: a) one or more alkylene carbonates selected from the group consisting of i) ethylene carbonate, ii) propylene carbonate, and iii) butylene carbonate, b) one or more polyols capped with C.sub.1-C.sub.6 organic acid wherein the polyol portion is selected from the group consisting of i) ethylene glycol, ii) 1,3 propylene glycol, iii) 1,2 propylene glycol, iv) butylene glycol, v) trimethylol propane, vi) trimethylol ethane, vii) pentaerythritol, viii) sorbitol, ix) sorbitan, x) glucose, xi) fructose, xii) galactose and xiii) glycerin, c) one or more alkylene glycol alkyl ethers acetates selected from the group consisting of i) dipropylene glycol methyl ether acetate, ii) tripropylene glycol methyl ether acetate, and iii) tripropylene glycol butyl ether acetate, d) one or more diesters selected from the group consisting of i) C.sub.1-C.sub.6 dialkylsuccinate, ii) C.sub.1-C.sub.6 dialkyladipate, iii) C.sub.1-C.sub.6 dialkylmethylglutarate, iv) C.sub.1-C.sub.6 dialkyl glutarate, v) C.sub.1-C.sub.6 dialkyl ethylsuccinate, and vi) C.sub.1-C.sub.6 dialkyl maleate, e) one or more 1-alkyl-2-pyrrolidone selected from the group wherein the alkyl functionality is a C.sub.1-C.sub.8 alkyl radical, f) one or more members selected from the group consisting of i) dimethylacetamide, ii) dimethylformamide, iii) dimethyl-2-imidazolidinone, iv) isophorone, v) hexamethylphosphoramide, vi) 2-methoxyethyl ether, vii) N,N-dimethyldecanamide, viii) N,N-dimethyloctanamide, ix) limonene, x) sulfolane g) one or more trialkylphosphates selected from the group represented by the structure:

(148) ##STR00028## wherein: R.sup.22 is alkyl radical —C.sub.1H.sub.3 to —C.sub.6H.sub.13 R.sup.23 is alkyl radical —C.sub.1H.sub.3 to —C.sub.6H.sub.13 R.sup.24 is alkyl radical —C.sub.1H.sub.3 to —C.sub.6H.sub.13 h) one or more dialkylene glycols dialkyl ethers, (glymes) selected from the structure:

R.sub.11—O—(R.sup.12—O).sub.n—R.sup.13 wherein: R.sup.11 is one or more alkyl radicals selected from the group consisting of C.sub.1 to C.sub.6, R.sup.12 is one or more alkylene radicals selected from the group consisting of C.sub.1 to C.sub.4, R.sup.13 is one or more alkyl radicals selected from the group consisting of C.sub.1 to C.sub.6, and n is one or more integers selected from the group of 1 to 10, i) one or more hydrocarbons selected from the group consisting of i) one or more paraffinic liquids, ii) one or more cycloparaffinics, iii) one or more aromatic hydrocarbons, iv) aliphatic hydrocarbons, v) acyclic hydrocarbons, vi) unsaturated hydrocarbon, and vii) polyalphaolefins, j) one or more fatty acid alkyl esters wherein the one or more fatty acid functionality is selected from the group consisting of C.sub.8-C.sub.18 fatty acids and wherein the alkyl functionality is one or more C.sub.1-C.sub.6 alkyl radicals, and k) one or more C.sub.1-C.sub.18 fatty triglycerides.

(149) In an embodiment, said reaction medium further comprises an Organic Liquid System One, wherein said Organic Liquid System One comprises one or more aprotic solvents with a boiling point less than or equal to 120° C. selected from the group consisting of a) one or more hydrocarbons selected from the group consisting of i) one or more paraffinic liquids, ii) one or more cycloparaffinics, and iii) one or more aromatic hydrocarbons, b) one or more liquid halocarbons c) one or more haloalkylhydrocarbons, and d) one or more ethers selected from the group

consisting of i) 1,4-dioxane, ii) 1,3-dioxolane, iii) methyltetrahydrofuran, iv) dimethoxyethane, v) 1,3-dioxane, vi) 1,3-dioxolane, vii) 2,2-dimethyl-1,3-dioxolane, viii) diethyl ether, ix) tetrahydrofuran, and x) tetrahydropyran, e) d-limonene, f) 1,2-dimethoxyethane and f) one or more esters selected from the group consisting of i) C.sub.1-C.sub.6 alkylformate and ii) C.sub.1-C.sub.6 alkylacetate.

(150) In an embodiment, the molar ratio of the one or more said alkylamines to the one or more said phosphoryl trichlorides is 0.7 to 1.5:1.

(151) In an embodiment, the temperature of the reaction of one or more phosphoryl chlorides with one or more alkylamines is (-40) to 20° C.

(152) In an embodiment, said Organic Liquid System One is charged to a reaction vessel and the temperature of the contents of said reaction vessel is cooled to (-40) to 20° C. and then said one or more phosphoryl trichlorides is charged to said reaction vessel and cooled to (-40) to 20° C.

(153) In an embodiment, said one or more alkylamines are charged slowly and sub-surface of the composition comprising said a) Organic Liquid System One and b) said one or more phosphoryl trichlorides, wherein the agitation of the contents of said reaction vessel is at medium to high speed resulting in a composition comprising a) Organic Liquid System One, b) said one or more phosphoryl trichlorides, c) said one or more alkyl dichlorophosphoryl amides and d) one or more chloride salts of said one or more alkylamines.

(154) In an embodiment, the one or more chloride salts of said one or more alkylamines are reacted with ammonia freeing the one or more alkylamines to react with said phosphoryl trichlorides.

(155) In an embodiment, a temperature of contents of said reaction vessel are maintained at less than 0° C.

(156) In an embodiment, initially a very slow agitation speed of the contents of said reaction vessel is selected so as to reduce the surface mixing with the reaction medium below the surface and wherein said ammonia is slowly charged to the headspace of reaction vessel to react with said one or more alkyl ammonium chlorides.

(157) In an embodiment, a temperature of contents of said reaction vessel are maintained at less than 0° C.

(158) In an embodiment, after about ¼ the total ammonia charge is completed, the agitation speed is increased to medium or high and the temperature of the contents are slowly allowed to increase to less than 100° C., wherein the temperature is controlled by cooling medium and by addition rate of ammonia and wherein the addition of ammonia results in the consumption of said one or more phosphoryl chlorides.

(159) In an embodiment, the method to make further comprises an addition to the contents of the reaction vessel of one or more members selected from the group consisting of: a) Organic Liquid System Two to reaction vessel contents, wherein Organic Liquid System Two comprises one or more organic liquids selected from the group consisting of a) one or more aprotic organic liquids and b) one or more protic organic liquids wherein said one or more aprotic liquids are selected from the group consisting of: i) dimethyl sulfoxide, ii) one or more sulfoxide(s) selected from the group consisting of dialkyl, diaryl, or alkylaryl sulfoxide(s) selected from the formula structure: R.sup.9S(O)xR.sup.10 wherein (1) R.sup.9 and R.sup.10 are each independently a C.sub.1-C.sub.6alkylene group, an aryl group or C.sub.1-C.sub.3 alkylenearyl group, (2) or R.sup.9 and R.sup.10 with the sulfur to which they are attached form a 4 to 8 membered ring wherein R.sup.9 and R.sup.10 together are a C.sub.1-C.sub.6 alkylene group which optionally contains one or more atoms selected from the group consisting of O, S, Se, Te, N, and P in the ring, (3) and x is 1 or 2, iii) one or more alkylene carbonates selected from the group consisting of (1) ethylene carbonate, (2) propylene carbonate, and (3) butylene carbonate, iv) one or more polyols capped with C.sub.1-C.sub.6 organic acid wherein the polyol portion is selected from the group consisting of (1) ethylene glycol, (2) 1,3 propylene glycol, (3) 1,2 propylene glycol, (4) butylene glycol, (5) trimethylol propane, (6) trimethylol ethane, (7) pentaerythritol, (8) sorbitol, (9) sorbitan, (10)

glucose, (11) fructose, (12) galactose and (13) glycerin, v) one or more alkylene glycol alkyl ethers acetates selected from the group consisting of (1) dipropylene glycol methyl ether acetate, (2) tripropylene glycol methyl ether acetate, and (3) tripropylene glycol butyl ether acetate, vi) one or more diesters selected from the group consisting of (1) C.sub.1-C.sub.6 dialkylsuccinate, (2) C.sub.1-C.sub.6 dialkyladipate, (3) C.sub.1-C.sub.6 dialkylmethylglutarate, (4) C.sub.1-C.sub.6 dialkyl glutarate, (5) C.sub.1-C.sub.6 dialkyl ethylsuccinate, and (6) C.sub.1-C.sub.6 dialkyl maleate, vii) one or more 1-alkyl-2-pyrrolidone selected from the group wherein the alkyl functionality is a C.sub.1-C.sub.8 alkyl radical, viii) one or more members selected from the group consisting of (1) dimethylacetamide, (2) dimethylformamide, (3) dimethyl-2-imidazolidinone, (4) isophorone, (5) hexamethylphosphoramide, (6) 2-methoxyethyl ether, (7) N,N-dimethyldecanamide, (8) N,N-dimethyloctanamide, (9) limonene, (10) sulfolane ix) one or more trialkylphosphates selected from the group represented by the structure:

(160) ##STR00029## wherein: R.sup.22 is alkyl radical —C.sub.1H.sub.3 to —C.sub.6H.sub.13 R.sup.23 is alkyl radical —C.sub.1H.sub.3 to —C.sub.6H.sub.13 R.sup.24 is alkyl radical —C.sub.1H.sub.3 to —C.sub.6H.sub.13 x) one or more dialkylene glycols dialkyl ethers, (glymes) selected from the structure:

R.sup.11—O—(R.sup.12—O).sub.nR.sup.13 wherein: R.sup.11 is one or more alkyl radicals selected from the group consisting of C.sub.1 to C.sub.6, R.sup.12 is one or more alkylene radicals selected from the group consisting of C.sub.1 to C.sub.4, R.sup.13 is one or more alkyl radicals selected from the group consisting of C.sub.1 to C.sub.6, and n is one or more integers selected from the group of 1 to 10, xi) one or more hydrocarbons selected from the group consisting of (1) one or more paraffinic liquids, (2) one or more cycloparaffinics, (3) one or more aromatic hydrocarbons, (4) aliphatic hydrocarbons, (5) acyclic hydrocarbons, (6) unsaturated hydrocarbon, and (7) polyalphaolefins, xii) one or more fatty acid alkyl esters wherein the one or more fatty acid functionality is selected from the group consisting of C.sub.8-C.sub.18 fatty acids and wherein the alkyl functionality is one or more C.sub.1-C.sub.6 alkyl radicals, and xiii) one or more C.sub.1-C.sub.18 fatty triglycerides, and wherein said one or more protic organic liquid are selected from the group consisting of: i) one or more alkanols selected from the group consisting of C.sub.1-C.sub.10 alkanols, ii) one or more polyols selected from the group consisting of (1) trimethylol propane, (2) trimethylol ethane, (3) pentaerythritol, (4) sorbitol, (5) sorbitan, (6) glucose, (7) fructose, (8) galactose, and (9) glycerin, iii) one or more polyalkylene glycols selected from the group consisting of poly(C.sub.1-C.sub.10 alkylene) glycols, iv) one or more dioxolane compound selected from the group consisting of (1) 2,2-dimethyl-1,3-dioxolane-4-methanol and (2) 2,2-diisobutyl-1,3-dioxolane-4-methanol, v) one or more alkylene glycol alkyl ethers selected from the structure:

(161) ##STR00030## wherein R.sup.14 is one or more members selected from the group consisting of CH.sub.3, C.sub.2H.sub.5, C.sub.3H.sub.7, C.sub.6H.sub.5, and C.sub.4H.sub.9, R.sup.15 is one or more members selected from the group consisting of H and

(162) ##STR00031## wherein R.sup.17 is one or more members selected from the group consisting of H and CH.sub.3, and f is an integer between 1 and 15, and R.sup.16 is one or more members selected from the group consisting of H and CH.sub.3, vi) one or more alkyl lactates selected from the group consisting of (1) ethyl lactate, (2) propyl lactate and (3) butyl lactate, vii) one or more alkanolamines selected from the group consisting of alkanolamines selected from the structure:

(163) ##STR00032## wherein R.sup.5 is one or more members selected from the group consisting of CH.sub.3, C.sub.2H.sub.5, C.sub.2H.sub.4OR.sup.8, C(CH.sub.3).sub.2CH.sub.2OH, CH(C.sub.2H.sub.5)CH.sub.2OH, CH.sub.3CH(OH)CH.sub.2, and C.sub.3H.sub.6OH, R.sup.6 is one or more members selected from the group consisting of H, CH.sub.3, C.sub.2H.sub.5, C.sub.2H.sub.4OR.sup.8 and C.sub.3H.sub.6OH, R.sup.7 is one or more members selected from the group consisting of H, C.sub.2H.sub.4OR.sup.8 and C.sub.3H.sub.6OH, wherein R.sup.8 is

(C.sub.2H.sub.4O).sub.gH wherein g is an integer between 1 and 10, viii) and glycerol carbonate, b) a colorant, c) a scent, e) a buffer, f) one or more surfactants, wherein the one or more surfactants comprises one or more members selected from the group consisting of (a) one or more cationic surfactants, (b) one or more amphoteric surfactants, (c) one or more Zwitterionic surfactants, (d) one or more anionic surfactants and (e) one or more nonionic surfactants.

(164) In an embodiment, the method to make does not comprise one or more of the following steps: a) removing inorganic chloride salts, b) stripping of volatile components of Organic Liquid System One, c) handling and purifying volatile components recovered from the stripping of reaction vessel contents d) handling of waste by-products and waste from purification steps e) recovering and purifying solid said one or more alkylphosphoric triamides compounds, f) packaging of solid said one or more alkylphosphoric triamides, g) shipping and storage of solid said one or more alkylphosphoric triamides, and/or h) dissolving of the solid said one or more alkylphosphoric triamide in a liquid delivery system.

(165) In an embodiment, the present invention relates to a method to make one or more nitrogenous phosphoryl compounds, wherein said method comprises using specific gravity to create differential levels in a reaction medium wherein a first reactant and a second reactant react to generate an intermediate third reactant and an intermediate fourth reactant, said intermediate third reactant having a different specific gravity than the specific gravity of the first reactant, the specific gravity of the second reactant, and the specific gravity of the intermediate fourth reactant, wherein the different specific gravity causes the intermediate third reactant to rise to a surface of the reaction medium, wherein a fifth gaseous reactant is introduced at the surface of the reaction medium allowing the fifth gaseous reactant to react with the intermediate third reactant to generate the one or more nitrogenous phosphoryl compounds.

(166) In an embodiment, when the intermediate third reactant is completely reacted, the reaction medium is homogenous and no longer has differential levels based upon specific gravity,

(167) In an embodiment, the intermediate fourth reactant subsequently reacts with the fifth gaseous reactant to generate one or more nitrogenous phosphoryl compounds.

(168) In an embodiment, the reaction medium is kept at a temperature below 0° C.

(169) In an embodiment, the reaction medium is agitated at a rate that allows the third reactant to rise to the surface.

(170) In an embodiment, the fifth gaseous reactant is introduced below the surface of the reaction medium.

(171) In an embodiment, the reaction medium comprises one or more organic liquids that are non-reactive with phosphoryl chloride functional groups.

(172) In an embodiment, the one or more organic liquids comprise one or more aprotic liquids that have a boiling point equal to or below 120° C. selected from the group consisting of a) one or more hydrocarbons selected from the group consisting of i) one or more paraffinic liquids, ii) one or more cycloparaffinics, and iii) one or more aromatic hydrocarbons, b) one or more liquid halocarbons c) one or more halohydrocarbons, and d) one or more ethers selected from the group consisting of i) 1,4-dioxane, ii) 1,3-dioxolane, iii) methyltetrahydrofuran, iv) dimethoxyethane, v) 1,3-dioxane, vi) 1,3-dioxolane, vii) 2,2-dimethyl-1,3-dioxolane, viii) diethyl ether, ix) tetrahydrofuran, and x) tetrahydropyran, e) d-limonene, f) 1,2-dimethoxyethane and f) one or more esters selected from the group consisting of i) C.sub.1-C.sub.6 alkylformate and ii) C.sub.1-C.sub.6 alkylacetate.

(173) In an embodiment, the method further comprises adding between 1-10 wt. % water or ammonium hydroxide.

(174) In an embodiment, the method further comprises a filtering step. In a variation, the filtrate from the filtering step is ammonium chloride. In a variation, the ammonium chloride can be collected and used as a nitrification inhibitor.

(175) In an embodiment, the first reactant is PSCl.sub.3.

- (176) In an embodiment, the second reactant is a C.sub.1-C.sub.6 n-alkyl amine.
- (177) In an embodiment, the intermediate third reactant is a C.sub.1-C.sub.6 alkyl ammonium chloride.
- (178) In an embodiment, the intermediate fourth reactant is a n-C.sub.1-C.sub.6 alkyl thiophosphoric dichloro monoamide.
- (179) In an embodiment, the fifth gaseous reactant is ammonia.
- (180) In an embodiment, the reaction medium comprises a first organic liquid that is replaced by a second organic liquid.
- (181) In an embodiment, the second organic liquid is one or more liquids that can be used to deliver the one or more nitrogenous phosphoryl compounds to a substrate. In a variation, the one or more nitrogenous phosphoryl compounds are used as urease inhibitors.
- (182) In an embodiment, the substrate is a fertilizer granule.
- (183) In an embodiment, the fertilizer granule is a urea granule.
- (184) In an embodiment, the second organic liquid comprises DMSO (dimethyl sulfoxide) or NMP (n-methyl pyrrolidone).
- (185) The following Examples are presented to illustrate certain embodiments of the present invention:

(186) TABLE-US-00001 Rxt ID #: MCT00 7088 Lot # 20B08

#### Example 1

(187) 51.71 grams PCl.sub.3, 37.66 grams THF and 27.54 grams butyl amine were cooled to -20° C. A pre-cooled vessel was purged with N.sub.2 and then 18.33 grams of THF and 51.76 grams PCl.sub.3 were charged. A reaction vessel was purged with N.sub.2 and the 27.54 grams butyl amine, 54.28 grams ammonium carbonate and 18.33 grams THF were charged and mixed keeping temperature below -20° C. by utilizing a chilling bath set at -30° C. The mixture of PCl.sub.3/THF was slowly charged to reaction vessel over a 21 hour period at a rate wherein the temperature was desired to be maintained at a range of -10 to -20° C. while ensuring the off-gassing of carbon dioxide did not lead to excessive foaming. Fluidity of reaction vessel contents was extremely thick causing extreme difficulty in maintaining desired temperature range as the temperature range actually was -5 to 5° C. 67 additional grams of THF were charged in an effort to improve the fluidity of reaction vessel contents, replace THF loss to the reaction vessel's overhead system, and dissolve solids buildup. After charging was completed, the contents of the reaction vessel were mixed for 1 hour @ -10° C. 27.88 grams of ammonia gas was fed sub-surface of the contents of the reaction vessel while allowing the temperature to slowly increase to 40° C. The reaction vessel was sealed to allow a slight pressure build of <10 psi and held for two hours. Contents of reaction vessel were extremely viscous and an additional 100 grams of THF were charged to the reaction vessel. After completion of the ammonia charge, 25.0 grams dimethyl sulfoxide was charged but had little impact on the fluidity of the contents of the reaction vessel. Contents of reaction vessel were too thick to effectively remove THF and ammonium chloride. Process was terminated and reaction vessel contents were disposed of.

(188) The difficulties encountered in trying to produce a highly concentrated triamide composition suggested that the weight percent of the Organic Liquid System One would need to be increased.

(189) TABLE-US-00002 Rxt ID #: MCT00 7089 Lot # 20B13

#### Example 2

(190) 56.30 grams PCl.sub.3, 274.21 grams THF and 29.88 grams butyl amine were cooled to -20° C. A pre-cooled vessel was purged with N.sub.2 and then 137.11 grams of THF and 56.30 grams PCl.sub.3 were charged. A reaction vessel was purged with N.sub.2 and the 29.88 grams butyl amine, 48.62 grams ammonium bicarbonate and 137.11 grams THF were charged and mixed keeping temperature at a range of -10 to -5° C. by utilizing a chilling bath set at -30° C. The mixture of PCl.sub.3/THF was slowly charged to reaction vessel at a rate wherein the temperature was desired to be maintained at a range of -10 to 5° C. while ensuring the off-gassing of carbon



dioxide did not lead to excessive foaming. Fluidity of reaction vessel contents was acceptable. Reaction vessel contents were heated to 40° C. to convert butyl ammonium chloride to butyl amine and ammonium chloride and to assist in off gassing CO.sub.2 from the ammonium bicarbonate. Reaction vessel contents were cooled to -5° C. and ammonia gas charge sub-surface of reaction vessel contents began. After exotherm associated with the reaction of phosphoryl chlorides and ammonia had subsided, cooling was removed and contents of reaction vessel were allowed to rise to 41° C. and held for one hour.

(191) Agitation and heat were removed and contents were examined after 12 hours for sedimentation of inorganic chloride salts. The amount of sedimentation was insignificant. Agitation was initiated for the contents of the reaction vessel, contents were heated from 5° C. to 30° C. and 124 grams of deoxygenated distilled water was charged and mixed 15 minutes and then allowed to stand for 1 hour. 332.57 grams of the top layer was removed and charged to another reaction vessel. 29.38 grams of DMSO were charge to the top layer, content of the reaction vessel were heated to 40° C. and placed under vacuum resulting in a pressure reading of 20 mm of Hg. After removal of THF, the contents were cooled, off-loaded, and packaged.

(192) TABLE-US-00003 Rxt ID #: MCT00 7090 Lot # 20B20

#### Example 3

(193) 42.24 grams PCl.sub.3, 205.73 grams THF and 22.5 grams butyl amine were cooled to -20° C. A pre-cooled vessel was purged with N.sub.2 and then 102.87 grams of THF and 42.24 grams PCl.sub.3 were charged. A reaction vessel was purged with N.sub.2 and the 22.5 grams butyl amine, 44.33 grams ammonium carbonate and 102.87 grams THF were charged and mixed keeping temperature at a range of -10 to -5° C. by utilizing a chilling bath set at -30° C. The mixture of PCl.sub.3/THF was slowly charged to reaction vessel at a rate wherein the temperature was desired to be maintained at a range of -10 to 5° C. while ensuring the off-gassing of carbon dioxide did not lead to excessive foaming. Fluidity of reaction vessel contents was acceptable. Reaction vessel contents were heated to 40° C. to convert butyl ammonium chloride to butyl amine and ammonium chloride and to assist in off gassing CO.sub.2 from the ammonium carbonate. Reaction vessel contents were cooled to -5° C. and ammonia gas charge sub-surface of reaction vessel contents began. After exotherm associated with the reaction of phosphoryl chlorides and ammonia had subsided, cooling was removed and contents of reaction vessel were allowed to rise to 40° C. and held for one hour. Agitation and heat were removed and contents were examined after 12 hours for sedimentation of inorganic chloride salts. The amount of sedimentation was insignificant. Agitation was initiated for the contents of the reaction vessel, contents were heated from 5° C. to 30° C. and 100 grams of deoxygenated distilled water was charged and mixed 15 minutes and then allowed to stand for 1 hour. Top layer was removed and charged to another reaction vessel. Content of the reaction vessel were heated to 40° C. and placed under vacuum resulting in a pressure reading of 20 mm of Hg After removal of THF, 22.04 grams of DMSO were charge to the stripped top layer. The contents were cooled, off-loaded and packaged.

(194) TABLE-US-00004 Rxt ID #: MCT00 7091 Lot # 20B23

#### Example 4

(195) 61.37 grams PCl.sub.3, 159.38 grams THF and 32.73 grams butyl amine were cooled to -20° C. A pre-cooled reaction vessel was purged with N.sub.2 and then 159.38 grams of THF, 15.75 grams sulfur, and 61.37 grams PCl.sub.3 were charged and placed under agitation. 32.73 grams butyl amine, was slowly charged over a two hour period to the contents in the reaction vessel keeping temperature at a range of -10 to 0° C. by utilizing a chilling bath set at -30° C. Contents were mixed for 30 minutes, the agitation speed was reduced and 21.23 grams of ammonium bicarbonate was charged to the surface of the reaction vessel contents and mixed at slow speed for 30 minutes. Agitation speed was increased and contents heated to 40° C. Off gassing of CO.sub.2 was controlled by the temperature of the reaction vessel contents. Reaction vessel contents were cooled to -5° C. and ammonia gas charge sub-surface of reaction vessel contents began. After

exotherm associated with the reaction of phosphoryl chlorides and ammonia had subsided, cooling was removed and contents of reaction vessel were allowed to rise to 40° C. and held for one hour. 157 grams of deoxygenated distilled water was charged and mixed 15 minutes and then allowed to stand for 1 hour. Top layer was removed and charged to another reaction vessel, content of the reaction vessel were heated to 40° C. and placed under vacuum resulting in a pressure reading of 20 mm of Hg. After removal of THF, 32.07 grams of DMSO were charge to the stripped top layer, the contents were cooled, off-loaded and packaged.

(196) TABLE-US-00005 Rxt ID #: MCT00 7092 Lot # 20C14

#### Example 5

(197) 70.92 grams PCl.sub.3, 230.33 grams ethyl acetate and 37.77 grams butyl amine were cooled to -20° C. A pre-cooled reaction vessel was purged with N.sub.2 and then 230.33 grams of ethyl acetate, 18.18 grams sulfur, and 70.92 grams PCl.sub.3 were charged and placed under agitation. 37.77 grams butyl amine, was slowly charged over a two hour period to the contents in the reaction vessel keeping temperature at a range of -10 to 0° C. by utilizing a chilling bath set at -30° C. Contents were mixed for 15 minutes, the agitation speed was reduced and 24.81 grams of ammonium carbonate was charged to the surface of the reaction vessel contents and mixed at slow speed for 30 minutes. Agitation speed was increased and contents heated to 40° C. Off gassing of CO.sub.2 was controlled by the temperature of the reaction vessel contents. Reaction vessel contents were cooled to -5° C. and ammonia gas charge sub-surface of reaction vessel contents began. After exotherm associated with the reaction of phosphoryl chlorides and ammonia had subsided, cooling was removed and contents of reaction vessel were allowed to rise to 55.7° C. and held for one hour. Content of the reaction vessel were heated to 40° C. and placed under vacuum resulting in a pressure reading of 20 mm of Hg. After removal of THF, 42.31 grams of DMSO were charge to very thick reaction vessel contents. The contents were charged to a Buchner funnel under vacuum to remove finished product from inorganic salts. The contents were too thick and filtration resulted in extremely low yield of liquid product of less than 20 grams.

(198) TABLE-US-00006 Rxt ID #: MCT00 7093 Lot # 20C25

#### Example 6

(199) 88.53 grams PSCl.sub.3, 204.12 grams THF and 38.28 grams butyl amine were cooled to -20° C. A pre-cooled reaction vessel was purged with N.sub.2 and then 204.12 grams of THF, 24.83 grams of ammonium bicarbonate and 88.53 grams PSCl.sub.3 were charged and placed under agitation. 38.28 grams butyl amine, was slowly charged over a two hour period to the contents in the reaction vessel keeping temperature at a range of -10 to 0° C. by utilizing a chilling bath set at -30° C. Contents were mixed for 15 minutes, the agitation speed was increased and the contents slowly heated to 40° C. Off gassing of CO.sub.2 was controlled by the temperature of the reaction vessel contents. Reaction vessel contents were cooled to -5° C. and ammonia gas charge sub-surface of reaction vessel contents began. After exotherm associated with the reaction of phosphoryl chlorides and ammonia had subsided, cooling was removed and contents of reaction vessel were allowed to rise to 46.3° C. and held for 30 minutes. 200 grams of deoxygenated distilled water was charged and mixed 15 minutes and then allowed to stand for 1 hour. Top layer was removed and charged to reaction vessel #2 while the bottom layer was disposed of. The contents of the reaction vessel #2 were heated to 40° C. and placed under vacuum resulting in a pressure reading of 20 mm of Hg After removal of THF, 37.5 grams of DMSO were charged to the contents of reaction vessel #2 and mixed for 15 minutes. The contents of reaction vessel #2 were cooled, off-loaded and packaged

(200) TABLE-US-00007 Rxt ID #: MCT00 7094 Lot # 20C30

#### Example 7

(201) 83.95 grams PSCl.sub.3, 193.26 grams THF and 36.24 grams butyl amine were cooled to -20° C. A pre-cooled reaction vessel was purged with N.sub.2 and then 193.26 grams of THF, and 83.95 grams PSCl.sub.3 were charged and placed under agitation. 36.24 grams butyl amine, was

slowly charged over a two hour period to the contents in the reaction vessel keeping temperature at a range of  $-10$  to  $0^{\circ}$  C. by utilizing a chilling bath set at  $-30^{\circ}$  C. Contents were mixed for 15 minutes, the agitation speed was increased and the contents slowly heated to  $40^{\circ}$  C. Reaction vessel contents were cooled to  $-5^{\circ}$  C. and 28.57 grams of ammonia carbonate was slowly charged to reaction vessel contents in about 30 minutes. After exotherm associated with the reaction of phosphoryl chlorides and ammonia had subsided, cooling was removed and contents of reaction vessel were allowed to rise to  $40^{\circ}$  C. and held for 30 minutes. 200 grams of deoxygenated distilled water was charged and mixed 15 minutes and then allowed to stand for 1 hour. Top layer was removed and charged to reaction vessel #2, the contents of reaction vessel #2 were heated to  $40^{\circ}$  C. and placed under vacuum resulting in a pressure reading of 20 mm of Hg. After removal of THF, 37.5 grams of DMSO were charged to contents of reaction vessel #2 and mixed for 15 minutes. The contents of reaction vessel #2 were filtered, cooled, off-loaded and packaged

(202) TABLE-US-00008 Rxt ID #: MCT00 7095 Lot # 20D02

#### Example 8

(203) 87.41 grams PSCl.sub.3, 201.58 grams THF and 37.80 grams butyl amine were cooled to  $-20^{\circ}$  C. A pre-cooled reaction vessel #1 was purged with N.sub.2 and then 201.58 grams of THF, and 87.41 grams PSCl.sub.3 were charged and placed under strong agitation. 37.8 grams butyl amine, was slowly charged through reaction vessel headspace over a two hour period to the contents in the reaction vessel keeping temperature at a range of  $-10$  to  $0^{\circ}$  C. by utilizing a chilling bath set at  $-30^{\circ}$  C. Contents were mixed for 30 minutes, the agitation speed was then slowed as the contents began cooling to  $-10^{\circ}$  C. if necessary. Reaction vessel #1 contents were cooled to  $-10^{\circ}$  C. and 42.42 grams of ammonia was slowly charged to reaction vessel #1 sub-surface of the contents of reaction vessel #1. After 30 minutes of charging ammonia to reaction vessel #1, agitation speed was increase to medium speed. After exotherm associated with the reaction of phosphoryl chlorides and ammonia had subsided, cooling was removed and contents of reaction vessel were allowed to rise to  $40^{\circ}$  C. and held for 30 minutes. 200 grams of deoxygenated distilled water was charged and mixed 15 minutes and then allowed to stand for 1 hour. Top layer was removed and charged to reaction vessel #2, the contents of reaction vessel #2 were heated to  $40^{\circ}$  C. and placed under vacuum resulting in a pressure reading of 20 mm of Hg. After removal of THF, 37.03 grams of DMSO were charged to contents of reaction vessel #2 and mixed for 15 minutes. The contents of reaction vessel #2 were filtered, cooled, off-loaded and packaged.

(204) TABLE-US-00009 Rxt ID #: MCT00 7096 Lot # 20D11

#### Example 9

(205) 84.57 grams PSCl.sub.3, 194.71 grams THF and 36.52 grams butyl amine were cooled to  $-20^{\circ}$  C. A pre-cooled reaction vessel #1 was purged with N.sub.2 and then 194.71 grams of THF, and 84.57 grams PSCl.sub.3 were charged and placed under strong agitation. 36.52 grams butyl amine, was slowly charged sub-surface of the contents in reaction vessel #1 over a two hour period maintaining the temperature at a range of  $-10$  to  $0^{\circ}$  C. by utilizing a chilling bath set at  $-30^{\circ}$  C. Contents were mixed for 30 minutes, the agitation speed was then slowed wherein the surface of the contents was barely moving as the contents began cooling to  $-10^{\circ}$  C. if necessary. When reaction vessel #1 contents were cooled to  $-10^{\circ}$  C. and 28.78 grams ammonium carbonate powder was slowly added to the surface of the contents in reaction vessel #1 with the surface barely moving. Agitation was slowly increased over a two hour period to medium speed. After two hour period, 33.95 grams of ammonia began to be charged to the headspace of the reaction vessel #1 and temperature range was maintained at  $-10$  to  $0^{\circ}$  C. After  $4\frac{1}{2}$  hours, ammonia charge was switched to sub-surface and the temperature of the contents of reaction vessel #1 were allowed to increase to  $40$  to  $50^{\circ}$  C. Hold temperature at  $40$  to  $50^{\circ}$  C. for 2 hours and then charge 200 grams of deoxygenated distilled water, mixed 15 minutes and then allowed to stand for 1 hour. Top layer was removed and charged to reaction vessel #2, the contents of reaction vessel #2 were heated to  $40^{\circ}$  C. and placed under vacuum resulting in a pressure reading of 20 mm of Hg After removal of THF, 35.77 grams

of DMSO were charged to contents of reaction vessel #2 and mixed for 15 minutes. The contents of reaction vessel #2 were filtered, cooled, off-loaded and packaged.

(206) TABLE-US-00010 Rxt ID #: MCT00 7097 Lot # 20D18

#### Example 10

(207) 90.14 grams PSCl.sub.3, 207.52 grams THF and 38.92 grams butyl amine were cooled to  $-20^{\circ}\text{C}$ . A pre-cooled reaction vessel #1 was purged with N.sub.2 and then 207.52 grams of THF, and 90.14 grams PSCl.sub.3 were charged and placed under strong agitation. 38.92 grams butyl amine, was slowly charged sub-surface of over a two hour period to the contents in the reaction vessel keeping temperature at a range of  $-10$  to  $0^{\circ}\text{C}$ . by utilizing a chilling bath set at  $-30^{\circ}\text{C}$ . Contents were mixed for 30 minutes, the agitation speed was then slowed wherein the surface of the contents was barely moving as the contents began cooling to  $-10^{\circ}\text{C}$ . if necessary. When reaction vessel #1 contents were cooled to  $-10^{\circ}\text{C}$ . and 45.23 grams of ammonia began to be charged to the headspace of the reaction vessel #1 and temperature range was maintained at  $-10$  to  $0^{\circ}\text{C}$ . After 30 minutes of charging ammonia to reaction vessel #1, agitation speed was increase to medium speed. After exotherm associated with the reaction of phosphoryl chlorides and ammonia had subsided, ammonia was charged to the sub-surface of the contents of reaction vessel #1, cooling was removed and contents of reaction vessel were allowed to rise to  $40^{\circ}\text{C}$ . and held for 120 minutes. 200 grams of deoxygenated distilled water was charged and mixed 15 minutes and then allowed to stand for 1 hour. Top layer was removed and charged to reaction vessel #2, the contents of reaction vessel #2 were heated to  $40^{\circ}\text{C}$ . and placed under vacuum resulting in a pressure reading of 20 mm of Hg. After removal of THF, 38.12 grams of DMSO were charged to contents of reaction vessel #2 and mixed for 15 minutes. The contents of reaction vessel #2 were filtered, cooled, off-loaded and packaged.

(208) TABLE-US-00011 Rxt ID #: MCT00 7098 Lot # 20D22

#### Example 11

(209) 79.49 grams PSCl.sub.3, 183.01 grams THF and 34.32 grams butyl amine were cooled to  $-20^{\circ}\text{C}$ . A pre-cooled reaction vessel #1 was purged with N.sub.2 and then 183.01 grams of THF, and 79.49 grams PSCl.sub.3 were charged and placed under strong agitation. 34.32 grams butyl amine was slowly charged sub-surface of over a two hour period to the contents in the reaction vessel keeping temperature at a range of  $-10$  to  $0^{\circ}\text{C}$ . by utilizing a chilling bath set at  $-30^{\circ}\text{C}$ . Contents were mixed for 30 minutes, the agitation speed was then slowed wherein the surface of the contents was barely moving as the contents began cooling to  $-10^{\circ}\text{C}$ . if necessary. When reaction vessel #1 contents were cooled to  $-10^{\circ}\text{C}$ . and 39.89 grams of ammonia began to be charged to the headspace of the reaction vessel #1 and temperature range was maintained at  $-10$  to  $0^{\circ}\text{C}$ . After 30 minutes of charging ammonia to reaction vessel #1, agitation speed was increase to medium speed. After exotherm associated with the reaction of phosphoryl chlorides and ammonia had subsided, ammonia was charged to the sub-surface of the contents of reaction vessel #1, cooling was removed and contents of reaction vessel were allowed to rise to  $40^{\circ}\text{C}$ . and held for 60 minutes. Charge 200 grams of deoxygenated distilled water was charged and mixed 15 minutes and then allowed to stand for 1 hour. Top layer was removed and charged to reaction vessel #2, the contents of reaction vessel #2 were heated to  $40^{\circ}\text{C}$ . and placed under vacuum resulting in a pressure reading of 20 mm of Hg. After removal of THF, 33.62 grams of DMSO were charged to contents of reaction vessel #2 and mixed for 15 minutes. The contents of reaction vessel #2 were filtered, cooled, off-loaded and packaged.

(210) TABLE-US-00012 Rxt ID #: MCT00 7099 Lot # 20D25

#### Example 12

(211) 74.01 grams PSCl.sub.3, 170.40 grams THF and 31.96 grams butyl amine were cooled to  $-20^{\circ}\text{C}$ . A pre-cooled reaction vessel #1 was purged with N.sub.2 and then 170.40 grams of THF, and 74.01 grams PSCl.sub.3 were charged and placed under strong agitation. 31.96 grams butyl amine was slowly charged sub-surface of over a two hour period to the contents in the reaction

vessel keeping temperature at a range of  $-10$  to  $0^{\circ}\text{C}$ . by utilizing a chilling bath set at  $-30^{\circ}\text{C}$ . Contents were mixed for 30 minutes, the agitation speed was then slowed wherein the surface of the contents was barely moving as the contents began cooling to  $-10^{\circ}\text{C}$ . if necessary. When reaction vessel #1 contents were cooled to  $-10^{\circ}\text{C}$ . and 37.14 grams of ammonia began to be charged to the headspace of the reaction vessel #1 and temperature range was maintained at  $-10$  to  $0^{\circ}\text{C}$ . After 30 minutes of charging ammonia to reaction vessel #1, agitation speed was increase to medium speed. After exotherm associated with the reaction of phosphoryl chlorides and ammonia had subsided, ammonia was charged to the sub-surface of the contents of reaction vessel #1, cooling was removed and contents of reaction vessel were allowed to rise to  $40^{\circ}\text{C}$ . and held for 60 minutes. Turn off agitation and allow standing for 12 hours. Little or no separation was observed. 200 grams of deoxygenated distilled water was charged and mixed 15 minutes and then allowed to stand for 1 hour. Top layer was removed and charged to reaction vessel #2, the contents of reaction vessel #2 were heated to  $40^{\circ}\text{C}$ . and placed under vacuum resulting in a pressure reading of 20 mm of Hg. After removal of THF, 31.30 grams of DMSO were charged to contents of reaction vessel #2 and mixed for 15 minutes. The contents of reaction vessel #2 were filtered, cooled, off-loaded and packaged.

#### Example 13

(212) TABLE-US-00013 Rxt ID #: MCT00 7101 Lot # 20E30

(213) 75.65 grams  $\text{PSCl.sub.3}$ , 174.17 grams THF and 32.66 grams butyl amine were cooled to  $-20^{\circ}\text{C}$ . A pre-cooled reaction vessel #1 was purged with  $\text{N.sub.2}$  and then 174.17 grams of THF, and 75.65 grams  $\text{PSCl.sub.3}$  were charged and placed under strong agitation. 32.66 grams butyl amine was slowly charged sub-surface of over a two hour period to the contents in the reaction vessel keeping temperature at a range of  $-10$  to  $0^{\circ}\text{C}$ . by utilizing a chilling bath set at  $-30^{\circ}\text{C}$ . Contents were mixed for 30 minutes, the agitation speed was then slowed wherein the surface of the contents was barely moving as the contents began cooling to  $-10^{\circ}\text{C}$ . if necessary. When reaction vessel #1 contents were cooled to  $-10^{\circ}\text{C}$ . and 37.96 grams of ammonia began to be charged to the headspace of the reaction vessel #1 and temperature range was maintained at  $-10$  to  $0^{\circ}\text{C}$ . After 30 minutes of charging ammonia to reaction vessel #1, agitation speed was increase to medium speed. After exotherm associated with the reaction of phosphoryl chlorides and ammonia had subsided, ammonia was charged to the sub-surface of the contents of reaction vessel #1, cooling was removed and contents of reaction vessel were allowed to rise to  $40^{\circ}\text{C}$ . and held for 60 minutes. 32.00 grams of DMSO were charged to contents of reaction vessel #1. The contents of reaction vessel #1 were filtered utilize multiple washing. Filter cake was extremely sticky. Filtrate was cooled, off-loaded and packaged.

(214) TABLE-US-00014 Rxt ID #: MCT00 7102 Lot # 20F07

#### Example 14

(215) 71.74 grams  $\text{PCl.sub.3}$ , 164.70 grams THF and 38.21 grams butyl amine were cooled to  $-20^{\circ}\text{C}$ . A pre-cooled reaction vessel #1 was purged with  $\text{N.sub.2}$  and then 164.70 grams of THF, and 71.74 grams  $\text{PCl.sub.3}$  were charged and placed under strong agitation. 38.21 grams butyl amine was slowly charged sub-surface of over a two hour period to the contents in the reaction vessel keeping temperature at a range of  $-10$  to  $0^{\circ}\text{C}$ . by utilizing a chilling bath set at  $-30^{\circ}\text{C}$ . Contents were mixed for 30 minutes, the agitation speed was then slowed wherein the surface of the contents was barely moving as the contents began cooling to  $-10^{\circ}\text{C}$ . if necessary. When reaction vessel #1 contents were cooled to  $-10^{\circ}\text{C}$ . and 44.41 grams of ammonia began to be charged to the headspace of the reaction vessel #1 and temperature range was maintained at  $-10$  to  $0^{\circ}\text{C}$ . After 30 minutes of charging ammonia to reaction vessel #1, agitation speed was increase to medium speed. After exotherm associated with the reaction of phosphoryl chlorides and ammonia had subsided, ammonia was charged to the sub-surface of the contents of reaction vessel #1, cooling was removed and contents of reaction vessel were allowed to rise to  $40^{\circ}\text{C}$ . and held for 60 minutes. 30.25 grams of DMSO were charged to contents of reaction vessel #1. The contents of reaction

vessel #1 were cooled, off-loaded and packaged.

#### Example 15

(216) 100 grams of Example 14 was charged to reaction vessel #1 with chilled overhead to return THF to reaction vessel #1 and heated to 60-65° C. under agitation. 9.82 grams of Calcium Carbonate was slowly charged to contents of reaction vessel #1. Contents were held at 60-65° C. until foaming subsided. Contents were cooled to <25° C., agitation was stopped, and contents were allowed to sit for 12 hours. Minimal separation had occurred. The contents of reaction vessel #1 were off-loaded and packaged.

(217) Exchanging a calcium ion for an ammonium ion did not result in appreciable sedimentation of the chloride salt particles.

#### Example 16

(218) A number of organic liquids were each added to a 25 gram sample of Example 14 to evaluate improving sedimentation of inorganic chloride particles present in Example 14 within 24 hours of standing. Results are listed in Table below.

(219) TABLE-US-00015 Grams added/ Clear layer in 25 grams of inches of a Organic Liquid  
Example 14 4 inch sample dimethyl 25 1 sulfoxide dimethyl 8 <1 sulfoxide triethyl phosphate 2 0  
tributyl phosphate 2 <0.25 100 viscosity 0.3 0 polydimethyl siloxane triethyl methyl 2 0  
ammonium chloride butyl carbonate 2 0 triethanolamine 2 <0.5 ammonium 4 1 ⅞ hydroxide/28%  
Large amounts of additional liquids seem to break the stability of the suspension.

Unexpectedly, the suspension was fairly stable for 24 hours.

(220) TABLE-US-00016 Rxt ID #: MCT00 7103 Lot # 20F14

#### Example 17

(221) 71.33 grams PCl.sub.3, 168.12 grams THF and 39.89 grams butyl amine were cooled to -20° C. A pre-cooled reaction vessel #1 was purged with N.sub.2 and then 168.12 grams of THF, and 71.33 grams PCl.sub.3 were charged and placed under strong agitation. 39.89 grams butyl amine was slowly charged sub-surface of over a two hour period to the contents in the reaction vessel keeping temperature at a range of -10 to 0° C. by utilizing a chilling bath set at -30° C. 5.15 grams of tributyl phosphate were added to the contents of reaction vessel #1. Contents were mixed for 30 minutes, the agitation speed was then slowed wherein the surface of the contents was barely moving as the contents began cooling to -10° C. if necessary. When reaction vessel #1 contents were cooled to -10° C. and 39.00 grams of ammonia began to be charged to the headspace of the reaction vessel #1 and temperature range was maintained at -10 to 0° C. After 30 minutes of charging ammonia to reaction vessel #1, agitation speed was increase to medium speed. After exotherm associated with the reaction of phosphoryl chlorides and ammonia had subsided, ammonia was charged to the sub-surface of the contents of reaction vessel #1, cooling was removed and contents of reaction vessel were allowed to rise to 40° C. and held for 60 minutes. Agitation speed was reduced and 100 grams of ammonium hydroxide were charge to reaction vessel #1. The contents were mixed with slow agitation for 15 minutes and then the agitation was stopped. Separation occurred within 15 minutes and the bottom layer was drawn off from reaction vessel #1. The remaining contents of reaction vessel #1 were heated to 50° C. and placed under vacuum resulting in a pressure reading of 20 mm of Hg. After removal of THF, 30.88 grams of DMSO was charged to contents of reaction vessel #1. The contents of reaction vessel #1 were filtered, cooled, off-loaded and packaged.

(222) TABLE-US-00017 Rxt ID #: MCT00 7104 Lot # 20F20

#### Example 18

(223) 58.07 grams PCl.sub.3, 77.34 grams THF, 77.34 grams propylene carbonate and 40.21 grams butyl amine were cooled to -20° C. A pre-cooled reaction vessel #1 was purged with N.sub.2 and then 77.34 grams of THF, 77.34 grams propylene carbonate and 58.07 grams PCl.sub.3 were charged and placed under strong agitation. 40.21 grams butyl amine was slowly charged sub-surface of over a two hour period to the contents in the reaction vessel keeping temperature at a

range of  $-10$  to  $0^{\circ}$  C. by utilizing a chilling bath set at  $-30^{\circ}$  C. Contents were mixed for 30 minutes, the agitation speed was then slowed wherein the surface of the contents was barely moving as the contents began cooling to  $-10^{\circ}$  C. if necessary. When reaction vessel #1 contents were cooled to  $-10^{\circ}$  C. and 33.79 grams of ammonia began to be charged to the headspace of the reaction vessel #1 and temperature range was maintained at  $-10$  to  $0^{\circ}$  C. After 30 minutes of charging ammonia to reaction vessel #1, agitation speed was increase to medium speed. After exotherm associated with the reaction of phosphoryl chlorides and ammonia had subsided, ammonia was charged to the sub-surface of the contents of reaction vessel #1, cooling was removed and contents of reaction vessel were allowed to rise to  $40^{\circ}$  C. and held for 60 minutes. The contents were mixed with slow agitation for 15 minutes and then the agitation was stopped. After 12 hours little of no separation occurred. 77.34 grams DMSO were charge and mixed for 15 minutes and then allowed to settle for six hours. No separation could be observed. 100 grams ammonium hydroxide was charged and mixed slowly for 15 minutes and allowed to set for 12 hours. No separation was observed. The contents of reaction vessel #1 were disposed of.

(224) In an embodiment the presence of propylene carbonate and DMSO caused issues with the separation of the ammonium hydroxide solution containing composition from the triamide containing composition.

(225) TABLE-US-00018 Rxt ID #: MCT00 7105 Lot # 20G11

#### Example 19

(226) 71.95 grams PSCl.sub.3, 93.68 grams THF, 93.44 grams propylene carbonate and 40.38 grams butyl amine were cooled to  $-20^{\circ}$  C. A pre-cooled reaction vessel #1 was purged with N.sub.2 and then 93.68 grams of THF, 93.44 grams propylene carbonate and 71.95 grams PSCl.sub.3 were charged and placed under strong agitation. 40.38 grams butyl amine was slowly charged sub-surface of over a two hour period to the contents in the reaction vessel keeping temperature at a range of  $-10$  to  $0^{\circ}$  C. by utilizing a chilling bath set at  $-30^{\circ}$  C. Contents were mixed for 30 minutes, the agitation speed was then slowed wherein the surface of the contents was barely moving as the contents began cooling to  $-10^{\circ}$  C. if necessary. When reaction vessel #1 contents were cooled to  $-10^{\circ}$  C. and 36.10 grams of ammonia began to be charged to the headspace of the reaction vessel #1 and temperature range was maintained at  $-10$  to  $0^{\circ}$  C. After 30 minutes of charging ammonia to reaction vessel #1, agitation speed was increase to medium speed. After exotherm associated with the reaction of phosphoryl chlorides and ammonia had subsided, ammonia was charged to the sub-surface of the contents of reaction vessel #1, cooling was removed and contents of reaction vessel were allowed to rise to  $40^{\circ}$  C. and held for 60 minutes. 93.68 grams of DMSO were charged to contents of reaction vessel #1 and then reaction vessel #1 was placed under vacuum resulting in a pressure reading of 20 mm of Hg. After removal of THF, sample was split into two samples; sample #1 was placed in a  $50^{\circ}$  C. oven for 24 hour and sample #2 was placed in a freezer at  $-1^{\circ}$  F. for 24 hours. Sample #1 showed a slight separation at the top and sample #2 was fluid. The appearance of contents of reaction vessel #1 was a tannish-white suspension.

(227) The particles in the suspension proved to be very resilient. The presence of DMSO did not result in the extraction of NBPT and the sedimentation of the by-product ammonium chloride.

(228) TABLE-US-00019 Rxt ID #: MCT00 7106 Lot # 20H22

#### Example 20

(229) 91.36 grams PSCl.sub.3, 118.96 grams THF, 118.65 grams propylene carbonate and 51.28 grams butyl amine were cooled to  $-20^{\circ}$  C. A pre-cooled reaction vessel #1 was purged with N.sub.2 and then 118.65 grams of THF, 118.65 grams propylene carbonate and 91.36 grams PSCl.sub.3 were charged and placed under strong agitation. 51.28 grams butyl amine was slowly charged sub-surface of over a two hour period to the contents in the reaction vessel keeping temperature at a range of  $-10$  to  $0^{\circ}$  C. by utilizing a chilling bath set at  $-30^{\circ}$  C. Contents were mixed for 30 minutes, the agitation speed was then slowed wherein the surface of the contents was

barely moving as the contents began cooling to  $-10^{\circ}\text{C}$ . if necessary. When reaction vessel #1 contents were cooled to  $-10^{\circ}\text{C}$ . and 36.10 grams of ammonia began to be charged to the headspace of the reaction vessel #1 and temperature range was maintained at  $-10$  to  $0^{\circ}\text{C}$ . After 30 minutes of charging ammonia to reaction vessel #1, agitation speed was increase to medium speed. After exotherm associated with the reaction of phosphoryl chlorides and ammonia had subsided, ammonia was charged to the sub-surface of the contents of reaction vessel #1, cooling was removed and contents of reaction vessel were allowed to rise to  $40^{\circ}\text{C}$ . and held for 60 minutes. Contents were cooled and packaged.

(230) MCT-710601

#### Example 21

(231) 30 grams of ammonium hydroxide/28% were charge to 50 grams of Example 20, mixed for 15 minutes and then allowed to settle for two hours. Separation was very poor most likely due to the presence of propylene carbonate. Sample was disposed of.

(232) MCT-710602

#### Example 22

(233) 27.92 grams of DMSO were charged to 100 grams of Example 20 in reaction vessel #1 and then mixed and heated to  $40$ - $50^{\circ}\text{C}$ . Contents of reaction vessel #1 were placed under vacuum resulting in a pressure reading of 20 mm of Hg. After removal of THF, contents were cooled and packaged. After two weeks of setting, sample began to show small amount of separation with a clear layer at the top.

(234) The particles of the suspension did not collapse even with the presence of DMSO in which NBPT was highly soluble and in which ammonium chloride is very insoluble. The appearance of the sample was very viscous. Without being bound to theory, it is believed that the DMSO has been absorbed onto the particles in the suspension.

#### Example 23

(235) 65.0 grams of DMSO were charged to 150 grams of Example 20 in reaction vessel #1 and then mixed and heated to  $40$ - $50^{\circ}\text{C}$ . 39 grams of calcium carbonate were slowly charged. Contents were placed under vacuum wherein the rate of the depth of vacuum was controlled by the formation of foam within reaction vessel #1. Contents of reaction vessel #1 reached a vacuum reading of 20 mm of Hg, then the vacuum was broken with N.sub.2 subsurface sparge. After removal of THF, contents were cooled and packaged. After two weeks of setting, sample began to show small amount of separation with a clear layer at the top.

(236) Conversion of the ammonium chloride to calcium chloride even with DMSO present did not result in a breakdown of the suspension.

(237) TABLE-US-00020 Rxt ID #: MCT00 7107 Lot # 20J14

#### Example 24

(238) 83.27 grams  $\text{PSCl.sub.3}$ , 88.48 grams THF, 88.48 grams propylene carbonate and 51.28 grams butyl amine were cooled to  $-20^{\circ}\text{C}$ . A pre-cooled reaction vessel #1 was purged with N.sub.2 and then 88.48 grams of THF, 88.48 grams propylene carbonate and 83.27 grams  $\text{PSCl.sub.3}$  were charged and placed under strong agitation. 51.28 grams butyl amine was slowly charged sub-surface of over a two hour period to the contents in the reaction vessel keeping temperature at a range of  $-10$  to  $0^{\circ}\text{C}$ . by utilizing a chilling bath set at  $-30^{\circ}\text{C}$ . Contents were mixed for 30 minutes, the agitation speed was then slowed wherein the surface of the contents was barely moving as the contents began cooling to  $-10^{\circ}\text{C}$ . if necessary. When reaction vessel #1 contents were cooled to  $-10^{\circ}\text{C}$ . and 41.78 grams of ammonia began to be charged to the headspace of the reaction vessel #1 and temperature range was maintained at  $-10$  to  $0^{\circ}\text{C}$ . After 30 minutes of charging ammonia to reaction vessel #1, agitation speed was increase to medium speed. After exotherm associated with the reaction of phosphoryl chlorides and ammonia had subsided, ammonia was charged to the sub-surface of the contents of reaction vessel #1, cooling was removed and contents of reaction vessel were allowed to rise to  $40^{\circ}\text{C}$ . and held for 60 minutes.



Contents were cooled and packaged.

(239) MCT 710701

#### Example 25

(240) 35 grams of calcium carbonate were charged to 160 grams of Example 24 in reaction vessel #1 and then mixed and heated to 45-50° C. Contents of reaction vessel #1 were allowed to off-gas and then 30 grams of DMSO was slowly charged. Contents were placed under vacuum until the contents of reaction vessel #1 reached a vacuum reading of 20 mm of Hg. The vacuum was broken with N.sub.2 subsurface sparge. After removal of THF, contents were cooled and packaged. Sample was extremely thick upon setting for 24 hours.

(241) MCT 710702

#### Example 26

(242) 26.15 grams of DMSO were charged to 100 grams of Example 20 in reaction vessel #1 and then mixed and heated to 40-50° C. Contents of reaction vessel #1 were placed under vacuum resulting in a pressure reading of 20 mm of Hg. After removal of THF, contents were cooled and packaged. Sample was extremely thick upon setting for 24 hours.

(243) The suspension was found to be extremely resilient. Addition of DMSO caused a thickening of the suspension indicating that the particles of the suspension had surprisingly absorbed the DMSO. Not to be bound by theory, the suspension particles seemed to a fused particle of chloride salt and NBPT, wherein the NBPT portion of the fused particles had absorbed the DMSO. Surprisingly the CaCl.sub.2 insolubility in propylene carbonate and DMSO did not settle out indicating that the fused particle could not be collapse with organic solvents that NBPT was very soluble in and that chloride salts were not very soluble in.

(244) TABLE-US-00021 Rxt ID #: MCT00 710800 Lot # 20K17

#### Example 27

(245) 166.14 grams PSCl.sub.3, 216.04 grams THF, 216.04 grams propylene carbonate and 93.25 grams butyl amine were cooled to -20° C. A pre-cooled reaction vessel #1 was purged with N.sub.2 and then 216.04 grams of THF, 216.04 grams propylene carbonate and 166.14 grams PSCl.sub.3 were charged and placed under strong agitation. 93.25 grams butyl amine was slowly charged sub-surface of over a two hour period to the contents in the reaction vessel keeping temperature at a range of -10 to 0° C. by utilizing a chilling bath set at -30° C. Contents were mixed for 30 minutes, the agitation speed was then slowed wherein the surface of the contents was barely moving as the contents began cooling to -10° C. if necessary. When reaction vessel #1 contents were cooled to -10° C. and 83.36 grams of ammonia began to be charged to the headspace of the reaction vessel #1 and temperature range was maintained at -10 to 0° C. After 30 minutes of charging ammonia to reaction vessel #1, agitation speed was increase to medium speed. After exotherm associated with the reaction of phosphoryl chlorides and ammonia had subsided, ammonia was charged to the sub-surface of the contents of reaction vessel #1, cooling was removed and contents of reaction vessel #1 were allowed to rise to 40° C. and held for 60 minutes. Contents were cooled and packaged.

(246) TABLE-US-00022 Rxt ID #: MCT00 7111 Lot # 21B09

#### Example 28

(247) 62.14 grams PSCl.sub.3, 161.61 grams propylene carbonate and 34.88 grams butyl amine were cooled to -20° C. A pre-cooled reaction vessel #1 was purged with N.sub.2 and then 161.61 grams propylene carbonate and 62.14 grams PSCl.sub.3 were charged and placed under strong agitation. 34.88 grams butyl amine was slowly charged sub-surface of over a two hour period to the contents in the reaction vessel keeping temperature at a range of -10 to 0° C. by utilizing a chilling bath set at -30° C. Contents were mixed for 30 minutes, the agitation speed was then slowed wherein the surface of the contents was barely moving as the contents began cooling to -10° C. if necessary. When reaction vessel #1 contents were cooled to -10° C. and 31.18 grams of ammonia began to be charged to the headspace of the reaction vessel #1 and temperature range was

maintained at  $-10$  to  $0^{\circ}$  C. After 30 minutes of charging ammonia to reaction vessel #1, agitation speed was increase to medium speed. After exotherm associated with the reaction of phosphoryl chlorides and ammonia had subsided, ammonia was charged to the sub-surface of the contents of reaction vessel #1, cooling was removed and contents of reaction vessel #1 were allowed to rise to  $40^{\circ}$  C. and held for 60 minutes. Contents were heated to  $60-70^{\circ}$  C., then cooled and packaged.

#### Example 29

(248) TABLE-US-00023 Rxt ID #: MCT00 7154 Lot # 22B07 rxt SU 681.05

(249) 57.59 grams  $\text{PSCl.sub.3}$ , 132.59 grams ethyl acetate and 24.87 grams butyl amine were cooled to  $-20^{\circ}$  C. A pre-cooled reaction vessel #1 was purged with  $\text{N.sub.2}$  and then the THF and the  $\text{PSCl.sub.3}$  were charged, cooled to  $-20$  to  $-10^{\circ}$  C., and placed under strong agitation utilizing baffles to ensure more turbulence and improved mixing of reactants. 24.87 grams of butyl amine was slowly charged sub-surface by a syringe pump with a charge setting of 1 ml/min to the contents in the reaction vessel keeping temperature at a range of  $-20$  to  $-10^{\circ}$  C. by utilizing a chilling bath set at  $-20^{\circ}$  C. and adding dry ice as necessary to maintain temperature range. Agitation was set at 300 rpms at the start of the butyl amine charge and increased to 500 rpms as viscosity increased. Contents were then mixed for 30 minutes, and the agitation speed was then slowed 150 rpms wherein the surface of the contents was barely moving as the contents were cooled to  $-20$  to  $0^{\circ}$  C. 28.9 grams of ammonia were charged to the headspace of the reaction vessel #1 and temperature range was maintained at  $-15$  to  $-5^{\circ}$  C. After exotherm associated with the reaction of phosphoryl chlorides and ammonia had subsided, extra ammonia was charged to the sub-surface of the contents of reaction vessel #1, and cooling was removed. Temperature was allowed to increase to  $40$  to  $50^{\circ}$  C., and held for 180 minutes and agitation was slowly increased to 900 rpms. Contents were weighed and sampled wherein the weight of the contents was 231.88 grams (loss of 30.42 grams of solvent).

#### Example 29a

(250) 18.36 grams of 28%  $\text{NH.sub.4OH}$  were charged to reaction vessel #1, mixed and held at  $40-50^{\circ}$  C. for 20 minutes. The reaction vessel #1 contents were then charged to a Buchner funnel and a liquid was removed under vacuum of about 50 mmHg. 26.52 grams of ethyl acetate were charged to reaction vessel #1 for rinsing out vessel and the rinse ethyl acetate was charged to the Buchner funnel and allowed to sit with no vacuum for 30 minutes and then filtered. The solid residual was weighed at 69.61 grams with % solids of 83.35%. The liquid of both the first filtration and of the second filtration were added to a stripping flask and a sample was collected for analysis. % non-volatile for the liquid was 32.67%. The ethyl acetate was removed at  $30-50^{\circ}$  C. about 20 mmHg and 45.1 grams of solids were collected (85.32% of expected solids).

#### Example 29b

(251) 45.1 grams of N-methyl pyrrolidone were charged to the stripping flask containing Example 29a contents and mixed for 15 minutes and then off loaded and packaged. Appearance of sample was dark.

(252) In an embodiment, liquifying the by-product, ammonium chloride, results in a large amount of liquid waste with residual solvent present that would need to be stripped out. In an embodiment this extra handling and the subsequent disposal cost of a large amount of liquid waste would increase the overall production cost. In a variation, it is believed that the collapse of the suspension particles and subsequent filtration is more cost effective. In another variation, the solid residual resulting from filtration, could have a market value driving down the cost of producing a liquified NBPT

#### Example 30

(253) TABLE-US-00024 Rxt ID #: MCT00 7154 Lot # 22A11 rxt SU 665.04

(254) 57.59 grams  $\text{PSCl.sub.3}$ , 132.59 grams ethyl acetate and 24.87 grams butyl amine were cooled to  $-20^{\circ}$  C. A pre-cooled reaction vessel #1 was purged with  $\text{N.sub.2}$  and then the THF and the  $\text{PSCl.sub.3}$  were charged, cooled to  $-20$  to  $-10^{\circ}$  C., and placed under strong agitation utilizing

baffles to ensure more turbulence and improved mixing of reactants. 24.87 grams of butyl amine was slowly charged sub-surface by a syringe pump with a charge setting of 1 ml/min to the contents in the reaction vessel keeping temperature at a range of  $-20$  to  $-10^{\circ}$  C. by utilizing a chilling bath set at  $-20^{\circ}$  C. and adding dry ice as necessary to maintain temperature range. Agitation was set at 300 rpms at the start of the butyl amine charge and increased to 500 rpms as viscosity increased. Contents were then mixed for 30 minutes, and the agitation speed was then slowed 150 rpms wherein the surface of the contents was barely moving as the contents were cooled to  $-20$  to  $0^{\circ}$  C. 29.32 grams of ammonia were charged to the headspace of the reaction vessel #1 and temperature range was maintained at  $-15$  to  $-5^{\circ}$  C. After exotherm associated with the reaction of phosphoryl chlorides and ammonia had subsided, extra ammonia was charged to the sub-surface of the contents of reaction vessel #1, and cooling was removed. Temperature was allowed to increase to  $40$  to  $45^{\circ}$  C., and held for 180 minutes and agitation was slowly increased to 900 rpms. Contents were weighed and sampled wherein the weight of the contents was 236.04 grams.

#### Example 30a

(255) 18.63 grams of 28%  $\text{NH}_4\text{OH}$  were charged to reaction vessel #1, mixed and held at  $40$ - $45^{\circ}$  C. for 20 minutes. The reaction vessel #1 contents were then charged to a Buchner funnel and a liquid was removed under vacuum of about 50 mmHg. 26.91 grams of ethyl acetate were charged to reaction vessel #1 for rinsing out vessel and the rinse ethyl acetate was charged to the Buchner funnel and allowed to sit with no vacuum for 30 minutes and then filtered. The solid residual was weighed at 68.69 grams with % solids of 74.72%. The liquid of both the first filtration and of the second filtration were added to a stripping flask. The ethyl acetate was removed at  $30$ - $50^{\circ}$  C. about 20 mmHg and 49.22 grams of solids were collected (85.32% of expected solids).

#### Example 30b

(256) 29.51 grams of DMSO, 4.28 grams of propylene carbonate, and 0.43 grams of triethanolamine were charged to the stripping flask containing Example 30a contents and mixed for 15 minutes and then off loaded and packaged. Product was fluid with some color development.

#### Example 31

(257) TABLE-US-00025 Rxt ID #: MCT00 7112 Lot # 21C07

(258) 114.79 grams  $\text{PSCl}_3$ , 278.27 grams propylene carbonate and 64.43 grams butyl amine were cooled to  $-20^{\circ}$  C. A pre-cooled reaction vessel #1 was purged with  $\text{N}_2$  and then the propylene carbonate and the  $\text{PSCl}_3$  were charged, cooled to  $-20$  to  $-10^{\circ}$  C., and placed under strong agitation. 64.43 grams of Butyl amine were slowly charged sub-surface of over a two hour period to the contents in the reaction vessel keeping temperature at a range of  $-10$  to  $0^{\circ}$  C. by utilizing a chilling bath set at  $-30^{\circ}$  C. Contents were mixed for 30 minutes, and then the agitation speed was then slowed wherein the surface of the contents was barely moving as the contents were cooled to  $-10^{\circ}$  C. When reaction vessel #1 contents were cooled to  $-10^{\circ}$  C., began charging 57.6 grams of ammonia to the headspace of the reaction vessel #1 and temperature range was maintained at  $-10$  to  $0^{\circ}$  C. After 30 minutes of charging ammonia to reaction vessel #1, agitation speed was increase to medium speed. After exotherm associated with the reaction of phosphoryl chlorides and ammonia had subsided, extra ammonia was charged to the sub-surface of the contents of reaction vessel #1, cooling was removed and contents of reaction vessel #1 were allowed to rise to  $40^{\circ}$  C. and held for 60 minutes. Contents were heated to  $94.6^{\circ}$  C., then cooled and packaged. Process yielded 508.11 (approximately 99% of theoretical yield) grams of a flowable suspension. After 2 month of storage, a small clear layer developed at the top of the sample while the sample itself was fluid.

#### Example 32

(259) TABLE-US-00026 Rxt ID #: MCT00 7113 Lot # 21D08

(260) 113.5 grams  $\text{PSCl}_3$ , 221.86 grams propylene carbonate and 63.7 grams butyl amine were cooled to  $-20^{\circ}$  C. A pre-cooled reaction vessel #1 was purged with  $\text{N}_2$  and then the propylene

carbonate and the PSCl.sub.3 were charged, cooled to -20 to -10° C., and placed under strong agitation. Butyl amine was pre-mixed with 54.67 grams of propylene carbonate; however mixture produced an exotherm and was disposed of. 54.67 grams of propylene carbonate at -20° C. was charged to reaction vessel #1 and then 63.7 grams of butyl amine was slowly charged sub-surface of over a two hour period to the contents in the reaction vessel keeping temperature at a range of -20 to -10° C. by utilizing a chilling bath set at -30° C. and adding dry ice as necessary to maintain temperature range. Contents were mixed for 30 minutes, and the agitation speed was then slowed wherein the surface of the contents was barely moving as the contents were cooled to -10° C. When reaction vessel #1 contents were cooled to -20 to -10° C., began charging 56.95 grams of ammonia to the headspace of the reaction vessel #1 and temperature range was maintained at -20 to -10° C. After 30 minutes of charging ammonia to reaction vessel #1, agitation speed was increase to medium speed. After exotherm associated with the reaction of phosphoryl chlorides and ammonia had subsided, extra ammonia was charged to the sub-surface of the contents of reaction vessel #1, cooling was removed and contents of reaction vessel #1 were allowed to rise to 40° C. and held for 60 minutes. Contents were heated to 111.1° C., then cooled and packaged. Process yielded 517.69 grams (approximately 100% of expected yield) of a flowable suspension.

#### Example 33

(261) TABLE-US-00027 Rxt ID #: MCT00 7127 Lot # 21K01

(262) 107.86 grams PSCl.sub.3, 259.82 grams glycerol triacetate and 60.55 grams butyl amine were cooled to -20° C. A pre-cooled reaction vessel #1 was purged with N.sub.2 and then the propylene carbonate and the PSCl.sub.3 were charged, cooled to -20 to -10° C., and placed under strong agitation. 60.55 grams of butyl amine was slowly charged using a syringe pump at a rate of 35 ul/min sub-surface of over a two hour period to the contents in the reaction vessel keeping temperature at a range of -20 to -10° C. by utilizing a chilling bath set at -30° C. and adding dry ice as necessary to maintain temperature range. Contents were mixed for 30 minutes, and the agitation speed was then slowed wherein the surface of the contents was barely moving as the contents were cooled to -10° C. When reaction vessel #1 contents were cooled to -20 to -10° C., began charging 53.53 grams of ammonia to the headspace of the reaction vessel #1 and temperature range was maintained at -20 to -10° C. After 30 minutes of charging ammonia to reaction vessel #1, agitation speed was increase to medium speed. After exotherm associated with the reaction of phosphoryl chlorides and ammonia had subsided, excess ammonia was charged to the sub-surface of the contents of reaction vessel #1, cooling was removed and contents of reaction vessel #1 were allowed to rise to 100-100° C. and held for 60 minutes. Contents were then cooled and packaged. Process yielded a thick suspension.

#### Example 34

(263) TABLE-US-00028 Rxt ID #: MCT00 7128 Lot # 21K02

(264) 109.7 grams PSCl.sub.3, 285.33 grams propylene carbonate and 61.58 grams butyl amine were cooled to -20° C. A pre-cooled reaction vessel #1 was purged with N.sub.2 and then the propylene carbonate and the PSCl.sub.3 were charged, cooled to -20 to -10° C., and placed under strong agitation. 61.58 grams of butyl amine were slowly charged sub-surface of over a two hour period to the contents in the reaction vessel keeping temperature at a range of -20 to -10° C. by utilizing a chilling bath set at -30° C. and adding dry ice as necessary to maintain temperature range. Contents were mixed for 30 minutes, and the agitation speed was then slowed wherein the surface of the contents was barely moving as the contents were cooled to -10° C. When reaction vessel #1 contents were cooled to -20 to -10° C., began charging 55.05 grams of ammonia to the headspace of the reaction vessel #1 and temperature range was maintained at -20 to -10° C. After 30 minutes of charging ammonia to reaction vessel #1, agitation speed was increase to medium speed. After exotherm associated with the reaction of phosphoryl chlorides and ammonia had subsided, excess ammonia was charged to the sub-surface of the contents of reaction vessel #1, cooling was removed and contents of reaction vessel #1 were allowed to rise to 40° C. and held for

60 minutes. Contents were heated to 60-70° C., then cooled and packaged. Process yielded a flowable suspension.

#### Example 35

(265) TABLE-US-00029 Rxt ID #: MCT00 7129 Lot # 21K26

(266) 60.39 grams PSCl.sub.3, 139.02 grams tetrahydrofuran (THF) and 26.07 grams butyl amine were cooled to -20° C. A pre-cooled reaction vessel #1 was purged with N.sub.2 and then the THF and the PSCl.sub.3 were charged, cooled to -20 to -10° C., and placed under strong agitation. 26.07 grams of butyl amine were slowly charged sub-surface of over a two hour period to the contents in the reaction vessel keeping temperature at a range of -20 to -10° C. by utilizing a chilling bath set at -20° C. and adding dry ice as necessary to maintain temperature range. Contents were mixed for 30 minutes, and the agitation speed was then slowed wherein the surface of the contents was barely moving as the contents were cooled to -10° C. When reaction vessel #1 contents were cooled to -20 to -10° C., began charging 30.30 grams of ammonia to the headspace of the reaction vessel #1 and temperature range was maintained at -20 to -10° C. After 30 minutes of charging ammonia to reaction vessel #1, agitation speed was increase to medium speed. After exotherm associated with the reaction of phosphoryl chlorides and ammonia had subsided, extra ammonia was charged to the sub-surface of the contents of reaction vessel #1, cooling was removed and contents of reaction vessel #1 were allowed to rise to 40° C. and held at 40 to 55° C. for 180 minutes.

#### Example 35a

(267) 100 grams of 28% ammonium hydroxide and 100 grams of distilled water were stirred in. The contents were charged to a separatory funnel and allowed to sit for 3 hours. The bottom layer was removed and the top layer weighed and determined to be 227.35 grams. The solids of the top layer were determined to be 16.5% resulting in a calculated total solids weight of 37.5 grams. The process resulted in 63% of expected yield.

#### Example 36

(268) TABLE-US-00030 Rxt ID #: MCT00 7131 Lot # 21K09

(269) 56.98 grams PSCl.sub.3, 131.19 grams tetrahydrofuran (THF) and 31.98 grams butyl amine were cooled to -20° C. A pre-cooled reaction vessel #1 was purged with N.sub.2 and then the THF and the PSCl.sub.3 were charged, cooled to -20 to -10° C., and placed under strong agitation. 31.98 grams of butyl amine were slowly charged sub-surface of over a two hour period to the contents in the reaction vessel keeping temperature at a range of -20 to -10° C. by utilizing a chilling bath set at -20° C. and adding dry ice as necessary to maintain temperature range. Agitation was increase as contents became thicker from 80 rpms to 540 rpms during butyl amine charge. Contents were mixed for 30 minutes, and the agitation speed was then slowed wherein the surface of the contents was barely moving as the contents were cooled to -10° C. When reaction vessel #1 contents were cooled to -20 to -10° C., agitation reduced to 80 rpms and then began slowly charging 28.6 grams of ammonia to the headspace of the reaction vessel #1 while temperature range was maintained at -20 to -10° C. After 30 minutes of charging ammonia to reaction vessel #1, agitation speed was increase to 250 rpms. After exotherm associated with the reaction of phosphoryl chlorides and ammonia had subsided, excess ammonia was charged to the sub-surface of the contents of reaction vessel #1, cooling was removed and contents of reaction vessel #1 were allowed to rise to 40 to 60° C. and held for 60 minutes. Contents were then cooled 60° C. and 200 grams of 28% ammonium hydroxide and 100 grams of distilled water were stirred in. The contents were charged to a separatory funnel and allowed to sit for 3 hours. The bottom layer was removed and the top layer was determined to be 136.16 grams. The solids of the top layer were determined to be 28.83% resulting in a calculated total solids weight of 39.25 grams. The process resulted in 61.8% of expected yield.

#### Example 37

(270) TABLE-US-00031 Rxt ID #: MCT00 7132 Lot # 21K10

(271) 60.04 grams PSCl.sub.3, 145.46 grams propylene carbonate and 33.7 grams butyl amine were cooled to  $-20^{\circ}\text{C}$ . A pre-cooled reaction vessel #1 was purged with N.sub.2 and then the propylene carbonate and the PSCl.sub.3 were charged, cooled to  $-20$  to  $-10^{\circ}\text{C}$ ., and placed under strong agitation. 30.13 grams of butyl amine was slowly charged sub-surface of over a two hour period to the contents in the reaction vessel keeping temperature at a range of  $-20$  to  $-10^{\circ}\text{C}$ . by utilizing a chilling bath set at  $-20^{\circ}\text{C}$ . and adding dry ice as necessary to maintain temperature range. Contents were mixed for 30 minutes, and the agitation speed was then slowed 60 rpms wherein the surface of the contents was barely moving as the contents were cooled to  $-10^{\circ}\text{C}$ . When reaction vessel #1 contents were cooled to  $-20$  to  $-10^{\circ}\text{C}$ ., began charging 30.13 grams of ammonia to the headspace of the reaction vessel #1 and temperature range was maintained at  $-20$  to  $0^{\circ}\text{C}$ . After 30 minutes of charging ammonia to reaction vessel #1, agitation speed was increase to medium speed. After exotherm associated with the reaction of phosphoryl chlorides and ammonia had subsided, excess ammonia was charged to the sub-surface of the contents of reaction vessel #1, cooling was removed and contents of reaction vessel #1 were allowed to rise to  $40^{\circ}\text{C}$ . and held for 60 minutes. Contents were heated to  $60$  to  $70^{\circ}\text{C}$ ., then cooled and packaged. Process yielded 267.3 grams (approximately 100% of expected yield) of a slightly thick but flowable suspension.

#### Example 38

(272) TABLE-US-00032 Rxt ID #: MCT00 7133 Lot # 21K11

(273) 55.54 grams PSCl.sub.3, 133.77 grams ParaOil 40 (hydrocarbon) and 31.17 grams butyl amine were cooled to  $-20^{\circ}\text{C}$ . A pre-cooled reaction vessel #1 was purged with N.sub.2 and then the ParaOil 40 and the PSCl.sub.3 were charged, cooled to  $-20$  to  $-10^{\circ}\text{C}$ ., and placed under strong agitation. 31.17 grams of butyl amine was slowly charged sub-surface of over a two hour period to the contents in the reaction vessel keeping temperature at a range of  $-20$  to  $-10^{\circ}\text{C}$ . by utilizing a chilling bath set at  $-20^{\circ}\text{C}$ . and adding dry ice as necessary to maintain temperature range. Contents were mixed for 30 minutes, and the agitation speed was then slowed 60 rpms wherein the surface of the contents was barely moving as the contents were cooled to  $-10^{\circ}\text{C}$ . When reaction vessel #1 contents were cooled to  $-20$  to  $-10^{\circ}\text{C}$ ., began charging 27.87 grams of ammonia to the headspace of the reaction vessel #1 and temperature range was maintained at  $-20$  to  $0^{\circ}\text{C}$ . After 30 minutes of charging ammonia to reaction vessel #1, agitation speed was increase to medium speed. After exotherm associated with the reaction of phosphoryl chlorides and ammonia had subsided, excess ammonia was charged to the sub-surface of the contents of reaction vessel #1, cooling was removed and contents of reaction vessel #1 were allowed to rise to  $40^{\circ}\text{C}$ . and held for 60 minutes. Contents were heated to  $60$  to  $70^{\circ}\text{C}$ ., then cooled and packaged. Process yielded 248.01 grams (approximately 100% of expected yield) of a very viscous suspension. Reaction vessel #1 was extremely difficult to clean.

#### Example 39

(274) TABLE-US-00033 Rxt ID #: MCT00 7134 Lot # 21K13

(275) 53.27 grams PSCl.sub.3, 128.3 grams tetrahydrofuran (THF) and 29.9 grams butyl amine were cooled to  $-20^{\circ}\text{C}$ . A pre-cooled reaction vessel #1 was purged with N.sub.2 and then the THF and the PSCl.sub.3 were charged and placed under strong agitation. 29.9 grams of butyl amine were slowly charged sub-surface of over a two hour period to the contents in the reaction vessel keeping temperature at a range of  $-20$  to  $-5^{\circ}\text{C}$ . by utilizing a chilling bath set at  $-20^{\circ}\text{C}$ . and adding dry ice as necessary to maintain temperature range. Agitation was increase as contents became thicker from 80 rpms to 540 rpms during butyl amine charge. Contents were mixed for 30 minutes, and the agitation speed was then slowed wherein the surface of the contents was barely moving as the contents were cooled to  $-10^{\circ}\text{C}$ . When reaction vessel #1 contents were cooled to  $-20$  to  $-10^{\circ}\text{C}$ ., agitation reduced to 80 rpms and then began slowly charging 26.73 grams of ammonia to the headspace of the reaction vessel #1 while temperature range was maintained at  $-20$  to  $-10^{\circ}\text{C}$ . After 30 minutes of charging ammonia to reaction vessel #1, agitation speed was

increase to 250 rpms. After exotherm associated with the reaction of phosphoryl chlorides and ammonia had subsided, excess ammonia was charged to the sub-surface of the contents of reaction vessel #1, cooling was removed and contents of reaction vessel #1 were allowed to rise to 40 to 60° C. and held for 60 minutes. After three hours, contents were cooled to <40 C. 40 grams of extra THF were added to add in fluidity. Process yielded 277.81 grams of a fluid suspension.

#### Example 40

(276) TABLE-US-00034 Rxt ID #: MCT00 7136 Lot # 21K16

(277) 58.91 grams PSCl.sub.3, 126.24 grams propylene carbonate and 27.98 grams butyl amine were cooled to -20° C. A pre-cooled reaction vessel #1 was purged with N.sub.2 and then the propylene carbonate and the PSCl.sub.3 were charged, cooled to -20 to -10° C., and placed under strong agitation. 27.98 grams of butyl amine was slowly charged sub-surface of over a two hour period to the contents in the reaction vessel keeping temperature at a range of -20 to -10° C. by utilizing a chilling bath set at -20° C. and adding dry ice as necessary to maintain temperature range. Contents were mixed for 30 minutes, and the agitation speed was then slowed 60 rpms wherein the surface of the contents was barely moving as the contents were cooled to -10° C. When reaction vessel #1 contents were cooled to -20 to -10° C., began charging 29.56 grams of ammonia to the headspace of the reaction vessel #1 and temperature range was maintained at -20 to 0° C. After 30 minutes of charging ammonia to reaction vessel #1, agitation speed was increase to medium speed. After exotherm associated with the reaction of phosphoryl chlorides and ammonia had subsided, Extra ammonia was charged to the sub-surface of the contents of reaction vessel #1, cooling was removed and contents of reaction vessel #1 were allowed to rise to 40° C. and held for 60 minutes. Contents were heated to 60 to 70° C., then cooled and packaged. Process yielded 251.17 grams (approximately 103.5% of expected yield) of a thick, flowable suspension. The higher than expected yield is the result of excess ammonia that was charged to ensure that all phosphoryl chloride functional groups were eliminated in the final composition.

#### Example 41

(278) TABLE-US-00035 Rxt ID #: MCT00 7137 Lot # 21K17

(279) 60.04 grams PSCl.sub.3, 145.46 grams propylene carbonate and 33.7 grams butyl amine were cooled to -20° C. A pre-cooled reaction vessel #1 was purged with N.sub.2 and then the propylene carbonate and the PSCl.sub.3 were charged, cooled to -20 to -10° C., and placed under strong agitation. 33.7 grams of butyl amine was slowly charged sub-surface of over a two hour period to the contents in the reaction vessel keeping temperature at a range of -20 to -10° C. by utilizing a chilling bath set at -20° C. and adding dry ice as necessary to maintain temperature range. Contents were mixed for 30 minutes, and the agitation speed was then slowed 60 rpms wherein the surface of the contents was barely moving as the contents were cooled to -10° C. When reaction vessel #1 contents were cooled to -20 to -10° C., began charging 33.28 grams of ammonia to the headspace of the reaction vessel #1 and temperature range was maintained at -20 to 0° C. After 30 minutes of charging ammonia to reaction vessel #1, agitation speed was increase to medium speed. After exotherm associated with the reaction of phosphoryl chlorides and ammonia had subsided, extra ammonia was charged to the sub-surface of the contents of reaction vessel #1, cooling was removed and contents of reaction vessel #1 were allowed to rise to 40° C. and held for 60 minutes. Contents were heated to 60 to 70° C., then cooled and packaged. Process yielded 269.75 grams (approximately 103% of expected yield) of a very thick suspension. The higher than expected yield is the result of excess ammonia that was charged to ensure that all phosphoryl chloride functional groups were eliminated in the final composition.

#### Example 42

(280) TABLE-US-00036 Rxt ID #: MCT00 7139 Lot # 21K31

(281) 60.04 grams PSCl.sub.3, 145.46 grams ethyl acetate and 33.7 grams butyl amine were cooled to -20° C. A pre-cooled reaction vessel #1 was purged with N.sub.2 and then the propylene carbonate and the PSCl.sub.3 were charged, cooled to -20 to -10° C., and placed under strong

agitation. 33.7 grams of butyl amine was slowly charged sub-surface of over a two hour period to the contents in the reaction vessel keeping temperature at a range of  $-20$  to  $-10^{\circ}$  C. by utilizing a chilling bath set at  $-20^{\circ}$  C. and adding dry ice as necessary to maintain temperature range. Contents were mixed for 30 minutes, and the agitation speed was then slowed 60 rpms wherein the surface of the contents was barely moving as the contents were cooled to  $-10^{\circ}$  C. When reaction vessel #1 contents were cooled to  $-20$  to  $-10^{\circ}$  C., began charging 29.47 grams of ammonia to the headspace of the reaction vessel #1 and temperature range was maintained at  $-20$  to  $0^{\circ}$  C. After 30 minutes of charging ammonia to reaction vessel #1, agitation speed was increase to medium speed. After exotherm associated with the reaction of phosphoryl chlorides and ammonia had subsided, extra ammonia was charged to the sub-surface of the contents of reaction vessel #1, cooling was removed and contents of reaction vessel #1 were allowed to rise to  $40^{\circ}$  C. and held for 60 minutes. Contents were heated to  $60$  to  $75^{\circ}$  and held for one hour. Process yielded 236.18 grams (approximately 94.95% of expected yield) of a fluid suspension.

#### Example 42a

(282) 200 grams of distilled water was charged to reaction vessel #1, mixed and held at  $60^{\circ}$  C. for 20 minutes. The reaction vessel #1 contents were then charged to a separatory funnel and allowed to sit for 3 hours. The bottom layer was removed and the top layer was filtered and 196.7 grams were collected. Percent solids were ran on the solution at  $70^{\circ}$  C. and found to be 18.32%. The resulting recovered solids were calculated to be 36.11 grams or 62.3% of expected recovery. The solid residue was also collected. Wash water was observed to be clear but also to have a strong sulfur odor.

(283) In an embodiment, the lack of deoxygenating the wash water resulted in the breakdown of some of the NBPT resulting in a strong sulfur odor.

#### Example 43

(284) TABLE-US-00037 Rxt ID #: MCT00 7140 Lot # 21L05

(285) 62.77 grams PSCl.sub.3, 144.5 grams tetrahydrofuran (THF) and 27.1 grams butyl amine were cooled to  $-20^{\circ}$  C. A pre-cooled reaction vessel #1 was purged with N.sub.2 and then the THF and the PSCl.sub.3 were charged, cooled to  $-20$  to  $-10^{\circ}$  C., and placed under strong agitation. 27.1 grams of butyl amine was slowly charged sub-surface by a syringe pump with a charge setting of 1 ml/min to the contents in the reaction vessel keeping temperature at a range of  $-20$  to  $-10^{\circ}$  C. by utilizing a chilling bath set at  $-20^{\circ}$  C. and adding dry ice as necessary to maintain temperature range and increasing agitation from 150 to 450 rpms to maintain fluidity during reaction. Contents were mixed for 30 minutes, and the agitation speed was then slowed 60 rpms wherein the surface of the contents was barely moving as the contents were cooled to  $-8^{\circ}$  C. When reaction vessel #1 contents were cooled to  $-20$  to  $-0^{\circ}$  C., began charging 31.50 grams of ammonia to the headspace of the reaction vessel #1 and temperature range was maintained at  $-8.4$  to  $15^{\circ}$  C. After 30 minutes of charging ammonia to reaction vessel #1, agitation speed was increase to medium speed. After exotherm associated with the reaction of phosphoryl chlorides and ammonia had subsided, extra ammonia was charged to the sub-surface of the contents of reaction vessel #1, cooling was removed and contents of reaction vessel #1 were allowed to rise to  $40$  to  $60^{\circ}$  C. and held for 180 minutes.

#### Example 43a

(286) TABLE-US-00038 Rxt ID #: MCT00 714101 Lot # 21L05

(287) 101.16 grams of distilled, de-oxygenated water were charged to reaction vessel #1 containing 285.86 grams of Ex 45, mixed and held at  $60^{\circ}$  C. for 20 minutes. The reaction vessel #1 contents were then charged to a separatory funnel and allowed to sit for 3 hours. The bottom layer was removed. Another 70 grams distilled, de-oxygenated water was charged to reaction vessel #1 for rinsing the residuals out and then added to the top layer. The separatory funnel was shaken and the contents allowed to separate. The bottom layer was removed and disposed of, the top layer was filtered and 182 grams were collected. Percent solids were ran on the solution at  $70^{\circ}$  C. and found



to be 26.7%. The resulting recovered solids were calculated to be 47.31 grams or 76.4% of expected recovery. Wash water was observed to be hazy. 33.09 grams of DMSO were added to reaction vessel #1 contents. THF was stripped out up to a temperature of 71° C. and replaced by 0.3 grams of triethanolamine, 10 grams of propylene glycol and 3.92 additional grams of DMSO. Product was clear but dark in color.

#### Example 44

(288) TABLE-US-00039 Rxt ID #: MCT000 7142 Lot # 21L10

(289) 101.16 grams of distilled, de-oxygenated water were charged to reaction vessel #1 containing 238.9 grams of Ex 40, mixed and held at 60° C. for 20 minutes. The reaction vessel #1 contents were then charged to a separatory funnel and allowed to sit for 3 hours. The bottom layer was removed. Another 20 grams distilled, de-oxygenated water was charged to reaction vessel #1 for rinsing the residuals out and then added to the top layer. The separatory funnel was shaken and the contents allowed to separate. The bottom layer was removed and disposed of, the top layer was filtered and 179.49 grams were collected. Percent solids were ran on the solution at 70° C. and found to be 28.83%. The resulting recovered solids were calculated to be 39.11 grams or 66.11% of expected recovery. Wash water was observed to be hazy. THF was stripped out at a temperature of 40-50° C. and then replaced by 21.05 grams of DMSO, 0.24 grams of triethanolamine, and 2.37 grams of propylene glycol were added to contents

#### Example 45

(290) TABLE-US-00040 Rxt ID #: MCT00 7143 Lot # 21L14

(291) 106.83 grams PSCl.sub.3, 214.42 grams ethyl acetate and 46.12 grams butyl amine were cooled to -20° C. A pre-cooled reaction vessel #1 was purged with N.sub.2 and then the ethyl acetate and the PSCl.sub.3 were charged, cooled to -20 to -10° C., and placed under strong agitation. 46.12 grams of butyl amine was slowly charged sub-surface by a syringe pump with a charge setting of 1 ml/min to the contents in the reaction vessel keeping temperature at a range of -20 to -10° C. by utilizing a chilling bath set at -20° C. and adding dry ice as necessary to maintain temperature range and increasing agitation from 360 to 520 rpms to maintain fluidity during reaction. Contents were mixed for 30 minutes, and the agitation speed was then slowed 60 rpms wherein the surface of the contents was barely moving as the contents were cooled to -8° C. When reaction vessel #1 contents were cooled to -20 to -0° C., began charging 53.6 grams of ammonia to the headspace of the reaction vessel #1 and temperature range was maintained at -0 to -10° C. After 30 minutes of charging ammonia to reaction vessel #1, agitation speed was increase to medium speed. After exotherm associated with the reaction of phosphoryl chlorides and ammonia had subsided, extra ammonia was charged to the sub-surface of the contents of reaction vessel #1, cooling was removed and contents of reaction vessel #1 were allowed to rise to 40 to 50° C. and held for 180 minutes.

(292) 84.43 grams of DMSO, 16.72 grams of propylene carbonate and 0.84 gram of triethanolamine were added to reaction vessel #1 and mixed for 15 minutes. 200 grams of distilled, deoxygenated water were added to contents of reaction vessel #1, mixed for 15 minutes and then allowed to sit for three hours. Resulting product did not split and was in fact extremely thick and could not be filtered.

#### Example 46

(293) TABLE-US-00041 Rxt ID #: MCT00 7149 Lot # 21M08 rxt SU 559.3

(294) 50.16 grams PSCl.sub.3, 115.48 grams tetrahydrofuran (THF) and 21.66 grams butyl amine were cooled to -20° C. A pre-cooled reaction vessel #1 was purged with N.sub.2 and then the THF and the PSCl.sub.3 were charged, cooled to -20 to -10° C., and placed under strong agitation. 21.66 grams of butyl amine was slowly charged sub-surface by a syringe pump with a charge setting of 1 ml/min to the contents in the reaction vessel keeping temperature at a range of -20 to -10° C. by utilizing a chilling bath set at -20° C. and adding dry ice as necessary to maintain temperature range. Agitation was set at 150 pms to control froth formation during reaction.

Contents were mixed for 30 minutes, and the agitation speed was then slowed 114 rpms wherein the surface of the contents was barely moving as the contents were cooled to  $-11^{\circ}\text{C}$ . When reaction vessel #1 contents were cooled to  $-20$  to  $-0^{\circ}\text{C}$ ., began charging 25.17 grams of ammonia to the headspace of the reaction vessel #1 and temperature range was maintained at  $-15$  to  $-5^{\circ}\text{C}$ . After 30 minutes of charging ammonia to reaction vessel #1, agitation speed was increase to medium speed. After exotherm associated with the reaction of phosphoryl chlorides and ammonia had subsided, extra ammonia was charged to the sub-surface of the contents of reaction vessel #1, cooling was removed.

#### Example 46a

(295) Contents of reaction vessel #1 were allowed to rise to  $10^{\circ}\text{C}$ . and held for 30 minutes and then sampled for % NBPT.

#### Example 46b

(296) Contents of reaction vessel #1 were allowed to rise to  $20^{\circ}\text{C}$ . and held for 30 minutes and then sampled for % NBPT.

#### Example 46c

(297) Contents of reaction vessel #1 were allowed to rise to  $30^{\circ}\text{C}$ . and held for 30 minutes and then sampled for % NBPT.

#### Example 47

(298) TABLE-US-00042 Rxt ID #: MCT00 7150 Lot # 21M10 rxt SU 568.38

(299) 57.54 grams PSCl.sub.3, 132.47 grams tetrahydrofuran (THF) and 24.84 grams butyl amine were cooled to  $-20^{\circ}\text{C}$ . A pre-cooled reaction vessel #1 was purged with N.sub.2 and then the THF and the PSCl.sub.3 were charged, cooled to  $-20$  to  $-10^{\circ}\text{C}$ ., and placed under strong agitation. 24.84 grams of butyl amine was slowly charged sub-surface by a syringe pump with a charge setting of 1 ml/min to the contents in the reaction vessel keeping temperature at a range of  $-20$  to  $-10^{\circ}\text{C}$ . by utilizing a chilling bath set at  $-20^{\circ}\text{C}$ . and adding dry ice as necessary to maintain temperature range. Agitation was set at 150 pms and increased to 300 rpms over the course of the charge. Contents were mixed for 30 minutes, and the agitation speed was then slowed 100 rpms wherein the surface of the contents was barely moving as the contents were cooled to  $-11^{\circ}\text{C}$ . When reaction vessel #1 contents were cooled to  $-20$  to  $-0^{\circ}\text{C}$ ., began charging 28.87 grams of ammonia to the headspace of the reaction vessel #1 and temperature range was maintained at  $-15$  to  $-5^{\circ}\text{C}$ . After exotherm associated with the reaction of phosphoryl chlorides and ammonia had subsided, extra ammonia was charged to the sub-surface of the contents of reaction vessel #1, cooling was removed and agitation was increased to 350 rpms. Temperature was allowed to increase to  $30^{\circ}\text{C}$ . and held for 90 minutes. Contents were weighed and sampled wherein the weight of the contents was 249.43 grams (estimated loss of 40.29 grams related to potential solvent loss)

#### Example 47a

(300) 18.34 grams of distilled, de-oxygenated water were charged to reaction vessel #1 mixed and held at  $23^{\circ}\text{C}$ . for 20 minutes. The reaction vessel #1 contents were then charged to a Buchner funnel and a liquid was removed under vacuum of about 50 mmHg. 78 grams of THF was charged to reaction vessel #1 for rinsing out vessel and the rinse THF was charged to the Buchner funnel and allowed to sit with no vacuum for 30 minutes and then filtered. The liquid of both the first filtration and of the second filtration were added to a stripping flask. The THF was removed at  $30$ - $50^{\circ}\text{C}$ . about 20 mmHg and 35.5 grams of solids were collected.

#### Example 47b

(301) 29.7 grams of DMSO, 3.14 grams of propylene carbonate, and 0.16 grams of triethanolamine were charged to the stripping flask containing Example 47a contents and mixed for 15 minutes and then off loaded and packaged.

#### Example 48

(302) TABLE-US-00043 Rxt ID #: MCT00 7150 Lot # 21M27 rxt SU 620.12

(303) 51.8 grams PSCl.sub.3, 119.26 grams tetrahydrofuran (THF) and 22.37 grams butyl amine were cooled to  $-20^{\circ}\text{C}$ . A pre-cooled reaction vessel #1 was purged with N.sub.2 and then the THF and the PSCl.sub.3 were charged, cooled to  $-20$  to  $-10^{\circ}\text{C}$ ., and placed under strong agitation. 22.37 grams of butyl amine was slowly charged sub-surface by a syringe pump with a charge setting of 1 ml/min to the contents in the reaction vessel keeping temperature at a range of  $-20$  to  $-10^{\circ}\text{C}$ . by utilizing a chilling bath set at  $-20^{\circ}\text{C}$ . and adding dry ice as necessary to maintain temperature range. Agitation was set at 33 pms and increased to 100 rpms over the course of the charge to reduce foam formation. Contents were mixed for 30 minutes, and the agitation speed was then slowed 50 rpms wherein the surface of the contents was barely moving as the contents were cooled to  $-11^{\circ}\text{C}$ . When reaction vessel #1 contents were cooled to  $-20$  to  $-0^{\circ}\text{C}$ ., began charging 26.0 grams of ammonia to the headspace of the reaction vessel #1 and temperature range was maintained at  $-15$  to  $-5^{\circ}\text{C}$ . After exotherm associated with the reaction of phosphoryl chlorides and ammonia had subsided, extra ammonia was charged to the sub-surface of the contents of reaction vessel #1, cooling was removed and agitation was increased to 95 rpms. Temperature was allowed to increase to  $45^{\circ}\text{C}$ . and held for 180 minutes. Contents were weighed and sampled wherein the weight of the contents was 215.44 grams.

(304) TABLE-US-00044 Rxt ID #: MCT00 715201 Lot # 22A02 rxt SU 620.12

Example 48a

(305) 12.57 grams of 28% ammonium hydroxide were charged to 166.99 grams of Example 48, mixed and held at  $40^{\circ}\text{C}$ . for 20 minutes. The reaction vessel #1 contents were then charged to a Buchner funnel and a liquid was removed under vacuum of about 50 mmHg. 18.15 grams of THF was charged to reaction vessel #1 for rinsing out vessel and the rinse THF was charged to the Buchner funnel and allowed to sit with no vacuum for 30 minutes and then filtered. The liquid of both the first filtration and of the second filtration were added to a stripping flask. The solids content of the collected liquid was 32%.

(306) In an embodiment, the use of ammonium hydroxide for breaking the fused suspension particle results in further converting any residual phosphoryl chloride functional groups to phosphoryl amide groups.

(307) TABLE-US-00045 Rxt ID #: MCT00 7150 Lot # 21M28 rxt SU 846.58

Example 49

(308) 51.48 grams PSCl.sub.3, 118.52 grams tetrahydrofuran (THF) and 22.23 grams butyl amine were cooled to  $-20^{\circ}\text{C}$ . A pre-cooled reaction vessel #1 was purged with N.sub.2 and then the THF and the PSCl.sub.3 were charged, cooled to  $-20$  to  $-10^{\circ}\text{C}$ ., and placed under strong agitation. 22.23 grams of butyl amine was slowly charged sub-surface by a syringe pump with a charge setting of 1 ml/min to the contents in the reaction vessel keeping temperature at a range of  $-20$  to  $-10^{\circ}\text{C}$ . by utilizing a chilling bath set at  $-20^{\circ}\text{C}$ . and adding dry ice as necessary to maintain temperature range. Agitation was set at 300 rpms. Contents were mixed for 30 minutes, and the agitation speed was then slowed 75 rpms wherein the surface of the contents was barely moving as the contents were cooled to  $-12^{\circ}\text{C}$ . When reaction vessel #1 contents were cooled to  $-20$  to  $-0^{\circ}\text{C}$ ., began charging 25.83 grams of ammonia to the headspace of the reaction vessel #1 and temperature range was maintained at  $-15$  to  $-5^{\circ}\text{C}$ . After exotherm associated with the reaction of phosphoryl chlorides and ammonia had subsided, extra ammonia was charged to the sub-surface of the contents of reaction vessel #1, and cooling was removed. Temperature was allowed to increase to  $50$  to  $55^{\circ}\text{C}$ . and held for 180 minutes. Contents were weighed and sampled wherein the weight of the contents was 206.79 grams.

(309) TABLE-US-00046 Rxt ID #: MCT00 715201 Lot # 22A02 rxt SU 620.12

Example 49a

(310) 14.6 grams of 28% ammonium hydroxide were charged to 193.96 grams of Example 49, mixed and held at  $40^{\circ}\text{C}$ . for 20 minutes. The reaction vessel #1 contents were then charged to a Buchner funnel and a liquid was removed under vacuum of about 50 mmHg. 21.08 grams of THF

was charged to reaction vessel #1 for rinsing out vessel and the rinse THF was charged to the Buchner funnel and allowed to sit with no vacuum for 30 minutes and then filtered. The liquid of both the first filtration and of the second filtration were added to a stripping flask. The solids content of the collected liquid was 49.12%.

#### Example 50

(311) A number of the examples were tested for inhibition performance. The experimental samples were applied to urea using standard overhead mixer with an anchor agitator. The amount of the sample to charge was determined by the specific gravity of the sample times the volumetric treatment level. For example:

(312) Determining the amount of Example 42 to be charged at a rate of 3 quarts/ton of urea:

(313) Specific gravity=1.08 gm/ml=9.0 lbs/gal At an application level of 3 quart/ton of urea=6.75 lbs of Example 42/2000 lbs of urea The application level would be 0.336% of Example 42

(314) 200 grams of urea was placed in a vessel, agitation was set so not to sling the urea out of the vessel and the calculated amount of the experimental sample was dripped onto the agitating urea. After completing the sample addition, the urea was agitated for an additional minute to insure uniform coverage.

#### Example 51

(315) In 143.5 cubic inch plastic containers with a hole in the side large enough for a tube attached Draeger am-5000 meter to be safely inserted, 200 grams of West Texas Soil (Estacado Clay Loam) with a 10% pH of 8 to 9 and a moisture content of 25 to 30%. The surface area of the soil was calculated to 47.8 sq inches. Exactly 0.75 grams of each urea sample was added to the surface of the soil and the plastic container and its hole were sealed. The headspace of each sealed container was analyzed for ppm ammonia by using a Draeger am-5000 meter attached to the tube which was inserted into the plastic container's hole and the pump was engaged 15 minutes pulling the contents of the container's headspace through a NH<sub>3</sub> detector. The readings were captured in the Draeger meter and downloaded at the end of sample readings. The total NH<sub>3</sub> readings were calculated each day for each sample tested. Charts below show the performance of Examples versus urea and versus a similarly formulated inhibitor sample. The test was repeated after the urea was stored for one month and for two months.

(316) TABLE-US-00047 Initial Measuring Ammonia Generation Sample Days Ran ID 3 4 5 6 7 10 11 Total Example 42\* 46 0 102 98 90 53 6 395 NBPT 9 10 4 4 14 97 88 226 urea 1257 1263 1283 980 855 533 222 6393 \*Example 42 was adjusted to a calculated level of 20% and 1% of a colorant for ensuring adequate coating and coverage of the urea particles.

(317) TABLE-US-00048 One Month Storage Measuring Ammonia Generation Sample Days Ran ID 3 4 5 6 7 10 11 12 13 Total Example 28 37 82 193 325 399 231 55 12 1362 29 NBPT 111 51 57 98 236 349 251 301 153 1607 Urea 1043 1763 1351 1458 1099 844 650 544 382 9134

(318) TABLE-US-00049 Two Month Storage Measuring Ammonia Generation Sample Days Ran ID 1 5 6 7 8 9 13 14 15 16 Total Example 37 78 5 10 211 337 223 47 116 62 1089 29 NBPT 103 99 32 129 159 130 152 66 49 68 884 Urea 307 1196 1129 1091 918 798 202 199 125 74 5732

(319) Urea treated with Example 29 had equivalent performance to NBPT in controlling ammonia generation over a two month period.

#### Example 52

(320) Testing was performed on Example 31 (approximately 25% NBPT) versus N Yield (approximately a 20% NBPT). To compensate for the differences in concentration, N Yield was applied at a 3 quart/ton rate while Example 31 was applied at a 2.67 quart/ton rate

(321) TABLE-US-00050 Measuring Ammonia Generation Sample Days Ran ID 3 4 7 Total Example 31 340 234 655 1229 N Yield 273 131 301 705 Urea 202 603 341 1146

(322) Example 31 was a suspension that was tested as a urease inhibitor and compared to N Yield. Ammonia generation readings were surprising. Example 31 was performing as a nitrification inhibitor. Testing was conducted to determine if ammonium chloride had nitrification inhibition

properties.

#### Example 53

(323) N Yield was compared to N Yield+NH<sub>4</sub>Cl

(324) TABLE-US-00051 wt. in grams Distilled Water Solution 0.25 999.75 of N Yield Solution 0.5 999.5 of NH<sub>4</sub>Cl N Yield N Yield NH<sub>4</sub>Cl N Yield solution 20 N Yield solution 20 NH<sub>4</sub> solution NH<sub>4</sub> solution 2 urea 1.5 urea 1.5 Water 2 Water 23.5 23.5 mix with stir plate mix with stir plate

(325) Charge 11.75 grams of solution by dropping onto soil sample

(326) TABLE-US-00052 Measuring Ammonia Generation Sample Days Ran ID 3 4 5 6 Total NH<sub>3</sub> N Yield 241 140 226 186 792 N Yield + NH<sub>4</sub>Cl 660 271 303 241 1475

(327) Surprisingly, NH<sub>4</sub>Cl seemingly was performing as a nitrification inhibitor even in the presence of NBPT.

#### Example 54

(328) NH<sub>4</sub>Cl performance as a nitrification inhibitor was compared to dicyandiamide (DCD), a well-known nitrification inhibitor.

(329) TABLE-US-00053 21L1609 21L16010 RM gms gms 34% DCD solution in DMSO 24.26 DMSO 27.71 12.50 Ammonium Chloride 17.00 Water 57.68 12.94 Liquid Blue Dye 0.62 0.30

(330) The compositions were applied to urea at a 4 quart/ton rate.

(331) TABLE-US-00054 Sample ID 1 5 6 7 8 9 13 14 15 16 Total NH<sub>4</sub>Cl 308 1586 1495 1149 932 727 308 155 53 100 6505 21L1609 DCD 316 1346 1230 1280 1,152 1,095 358 99 — 49 6609 21L16010 Urea 307 1196 1129 1091 918 798 202 202 125 74 5735

(332) NH<sub>4</sub>Cl performance as a nitrification inhibitor was equivalent to DCD.

#### Example 55

(333) A few of the Examples were evaluated by HPLC for % NBPT. The following HPLC method was utilized

##### NBPT Isocratic HPLC Method

(334) 1. Diluent=50:50 acetonitrile:water 2. Internal Standard Solution To a 200-mL volumetric flask, add the following: 1.59 g acetanilide. Bring to volume with Diluent. 3. Sample: To a 100-mL volumetric flask, add the following: 100 mg NBPT equivalent sample, 10 mL internal standard (IS) Acetanilide (75 mg/mL). For Example: 26% NBPT Product

(335)  $\frac{100\text{mgNBPT}}{0.26} = 385\text{mgproduct}$  weighted Bring to volume with diluent. Mix well. 4. Transfer a portion through a Nylon 0.45-μm syringe filter into an HPLC vial. 5. Inject on LC/UV.

##### HPLC/UV Conditions

##### Column Information

(336) Phase: Zorbax SB-C.sub.18

(337) Diameter: 4.6 mm

(338) Length: 250 mm

(339) Particle Size: 5 μm

(340) Mobile Phase: 54:46 H.sub.2O:ACN

(341) Pump: Shimadzu LC-10AT, 1.0 ml/min Isocratic

(342) Autoinjector: Shimadzu SIL-10AXL

(343) Detector Type: Shimadzu SPD-10A Detector @ 214 nm

(344) Controller: Shimadzu SCL-10A

(345) Data Acquisition System: Empower 2, Build 2154

(346) Volume Injection: 1

(347) Column Oven: Timberline Instruments; Temperature=40° C.

(348) Average Retention Time:

(349) NBPT=3.6 min IS=4.1 min

Run Time: 15 min

(350) TABLE-US-00055 Process % Yields Example ID HPLC results Expected Results NBPT

Example 35 16.55% 30% 55.17% Example 42 17.05% 23.3% 73.2% Example 29 17.8% 23.3% 76.39% Note: Samples that utilized propylene carbonate could not be ran as the HPLC solution were not clear due to poor solubilization of the suspended particles that were removed by the Nylon 0.45- $\mu$ m syringe filter giving unreliable results.

(351) All references cited herein are incorporated by reference in their entireties for all purposes.

## Claims

1. A fluid suspension composition comprising: a) one or more chloride salts, wherein the one or more chloride salts are inorganic, wherein the one or more chloride salts comprise one or more cations selected from the group consisting of a) magnesium, b) calcium, c) zinc, d) manganese, e) copper, f) cobalt, g) molybdenum, by nickel, and mixtures thereof, b) one or more nitrogenous phosphoryl compounds, c) one or more “binary fused particles”, and d) a non-aqueous, organic liquid delivery system, wherein the binary fused particles comprise one or more chloride salts and one or more nitrogenous phosphoryl compounds, wherein the non-aqueous, organic liquid delivery system comprises Organic Liquid System One, wherein the Organic Liquid System One comprises one or more non-phosphoryl chloride reactive aprotic organic liquids comprising the following properties: a) a boiling point greater than 120° C., by environmentally safe, c) a flashpoint above 145° F., d) rated safe for contact with humans and animals, e) a delivery system or a component of a delivery system that delivers the desired one or more nitrogenous phosphoryl compounds and the one or more inorganic chloride salts to the surface of the nitrogen source fertilizer granule, and f) stabilizes the inorganic by-product salts in a liquefied suspension, wherein the Organic Liquid System One is the reaction medium of one or more phosphoryl chlorides with one or more nitrogenous compounds, and wherein the fluid suspension is applied as a continuous homogenous liquid stable suspension to a surface of nitrogen source fertilizer granules to impart one or more properties to the nitrogen source fertilizer granules selected from the group consisting of a) urease inhibition and b) nitrification inhibition.

2. The fluid suspension composition of claim 1, wherein the composition further comprises an Organic Liquid System Two, wherein the Organic Liquid System Two imparts one or more properties to the fluid suspension composition selected from the group consisting of: a) assisting in removing reaction by-product chloride salts from the fluid compositions, b) maintaining the by-product chloride salts in a stable or semi-stable suspension, c) lowering viscosity, d) improving fluidity at temperatures of (−20° C.) or less, e) improving fluidity under summer storage conditions, f) improving evenness and homogeneity of the coating onto the nitrogen source fertilizer granule, g) improving temperature stability, and h) improving shipping, storage, and pumpability.

3. The fluid suspension composition of claim 2, wherein the Organic Liquid System Two comprises one or more organic liquids selected from the group consisting of one or more additional aprotic organic liquids and one or more protic liquids.

4. The fluid suspension of claim 3, wherein the one or more additional aprotic solvents comprise a) dimethyl sulfoxide, b) one or more sulfoxide(s) selected from the group consisting of dialkyl, diaryl, or alkylaryl sulfoxide(s) selected from the formula structure:

$R_{sup.9}S(O)_xR_{sup.10}$  Wherein i)  $R_{sup.9}$  and  $R_{sup.10}$  are each independently a  $C_{sub.1-6}$  alkylene group, an aryl group or  $C_{sub.1-3}$  alkylenearyl group, ii)  $R_{sup.9}$  and  $R_{sup.10}$  with the sulfur to which they are attached form a 4 to 8 membered ring wherein  $R_{sup.9}$  and  $R_{sup.10}$  together are a  $C_{sub.1-6}$  alkylene group which optionally contains one or more atoms selected from the group consisting of O, S, Se, Te, N, and P in the ring, iii) and x is 1 or 2, c) one or more alkylene carbonates selected from the group consisting of i) ethylene carbonate, ii) propylene carbonate, and iii) butylene carbonate, d) one or more polyols capped with  $C_{sub.1-6}$  organic acid wherein the polyol portion is selected from the group consisting of i) ethylene

glycol, ii) 1,3 propylene glycol, iii) 1,2 propylene glycol, iv) butylene glycol, v) trimethylol propane, vi) trimethylol ethane, vii) pentaerythritol, viii) sorbitol, ix) sorbitan, x) glucose, xi) fructose, xii) galactose and xiii) glycerin, e) one or more alkylene glycol alkyl ethers acetates selected from the group consisting of i) dipropylene glycol methyl ether acetate, ii) tripropylene glycol methyl ether acetate, and iii) tripropylene glycol butyl ether acetate, f) one or more diesters selected from the group consisting of i) C.sub.1-C.sub.6 dialkylsuccinate, ii) C.sub.1-C.sub.6 dialkyladipate, iii) C.sub.1-C.sub.6 dialkylmethylglutarate, iv) C.sub.1-C.sub.6 dialkyl glutarate, v) C.sub.1-C.sub.6 dialkyl ethylsuccinate, and vi) C.sub.1-C.sub.6 dialkyl maleate, g) one or more 1-alkyl-2-pyrrolidone selected from the group wherein the alkyl functionality is a C.sub.1-C.sub.8 alkyl radical, h) one or more members selected from the group consisting of i) dimethylacetamide, ii) dimethylformamide, iii) dimethyl-2-imidazolidinone, iv) isophorone, v) hexamethylphosphoramide, vi) 2-methoxyethyl ether, vii) N,N-dimethyldecanamide, viii) N,N-dimethyloctanamide, ix) limonene, x) sulfolane, i) one or more trialkylphosphates selected from the group represented by the structure: ##STR00033## wherein: R.sup.22 is alkyl radical-C.sub.1H.sub.3 to —C.sub.6H.sub.13 R.sup.23 is alkyl radical-C.sub.1H.sub.3 to —C.sub.6H.sub.13 R.sup.24 is alkyl radical-C.sub.1H.sub.3 to —C.sub.6H.sub.13 j) one or more dialkylene glycols dialkyl ethers, (glymes) selected from the structure: R.sup.11—O—(R.sup.12—O).sub.n—R.sup.13 wherein: R.sup.11 is one or more alkyl radicals selected from the group consisting of C.sub.1 to C.sub.6, R.sup.12 is one or more alkylene radicals selected from the group consisting of C.sub.1 to C.sub.4 R.sup.13 is one or more alkyl radicals selected from the group consisting of C.sub.1 to C.sub.6, and n is one or more integers selected from the group of 1 to 10, k) one or more hydrocarbons selected from the group consisting of i) one or more paraffinic liquids, ii) one or more cycloparaffinics, iii) one or more aromatic hydrocarbons, iv) aliphatic hydrocarbons, v) acyclic hydrocarbons, vi) unsaturated hydrocarbon, and vii) polyalphaolefins, l) one or more fatty acid alkyl esters wherein the one or more fatty acid functionality is selected from the group consisting of C.sub.8-C.sub.18 fatty acids and wherein the alkyl functionality is one or more C.sub.1-C.sub.6 alkyl radicals, and m) one or more C.sub.1-C.sub.18 fatty triglycerides.

5. The fluid suspension of claim 3, wherein the one or more protic solvents comprise: a) one or more alkanols selected from the group consisting of C.sub.1-C.sub.10 alkanols, b) one or more polyols selected from the group consisting of i) trimethylol propane, ii) trimethylol ethane, iii) pentaerythritol, iv) sorbitol, v) sorbitan, vi) glucose, vii) fructose, viii) galactose, and ix) glycerin, c) one or more polyalkylene glycols selected from the group consisting of poly(C.sub.1-C.sub.10 alkylene) glycols, d) one or more dioxolane compounds selected from the group consisting of i) 2,2-dimethyl-1,3-dioxolane-4-methanol and ii) 2,2-diisobutyl-1,3-dioxolane-4-methanol, e) one or more alkylene glycol alkyl ethers selected from the structure: ##STR00034## wherein R.sup.14 is one or more members selected from the group consisting of CH.sub.3, C.sub.2H.sub.5, C.sub.3H.sub.7, C.sub.6H.sub.5, and C.sub.4H.sub.9, R.sup.15 is one or more members selected from the group consisting of H and ##STR00035## wherein R.sup.17 is one or more members selected from the group consisting of H and CH.sub.3, and f is an integer between 1 and 15, and R.sup.16 is one or more members selected from the group consisting of H and CH.sub.3, f) one or more alkyl lactates selected from the group consisting of i) ethyl lactate, ii) propyl lactate and iii) butyl lactate, g) one or more alkanolamines selected from the group consisting of alkanolamines selected from the structure: ##STR00036## wherein R.sup.5 is one or more members selected from the group consisting of CH.sub.3, C.sub.2H.sub.5, C.sub.2H.sub.4OR.sup.8, C(CH.sub.3).sub.2CH.sub.2OH, CH(C.sub.2H.sub.5)CH.sub.2OH, CH.sub.3CH(OH)CH.sub.2, and C.sub.3H.sub.6OH, R.sup.6 is one or more members selected from the group consisting of H, CH.sub.3, C.sub.2H.sub.5, C.sub.2H.sub.4OR.sup.8 and C.sub.3H.sub.6OH, R.sup.7 is one or more members selected from the group consisting of H, C.sub.2H.sub.4OR.sup.8 and C.sub.3H.sub.6OH, wherein R.sup.8 is (C.sub.2H.sub.4O).sub.gH wherein g is an integer

between 1 and 10, h) and glycerol carbonate.

6. The fluid suspension composition of claim 1, wherein the one or more nitrogenous phosphoryl compounds comprise one or more alkylphosphoric amides selected from Structure I:

##STR00037## wherein: a) R<sup>sup.2</sup> is one or more members selected from the group consisting of CH<sub>sub.3</sub>, C<sub>sub.2</sub>H<sub>sub.5</sub>, C<sub>sub.3</sub>H<sub>sub.7</sub>, C<sub>sub.4</sub>H<sub>sub.9</sub>, C<sub>sub.5</sub>H<sub>sub.11</sub>, C<sub>sub.6</sub>H<sub>sub.13</sub>, C<sub>sub.6</sub>H<sub>sub.5</sub>, C<sub>sub.7</sub>H<sub>sub.7</sub>, OX<sup>sup.1</sup>, OCH<sub>sub.3</sub>, OC<sub>sub.2</sub>H<sub>sub.5</sub>, OC<sub>sub.3</sub>H<sub>sub.7</sub>, OC<sub>sub.4</sub>H<sub>sub.9</sub>, OC<sub>sub.5</sub>H<sub>sub.11</sub>, OC<sub>sub.6</sub>H<sub>sub.13</sub>, and HNR<sup>sup.4</sup>, wherein i) R<sup>sup.4</sup> is one or more members selected from the group consisting of H, CH<sub>sub.3</sub>, C<sub>sub.2</sub>H<sub>sub.5</sub>, C<sub>sub.3</sub>H<sub>sub.7</sub>, C<sub>sub.4</sub>H<sub>sub.9</sub>, C<sub>sub.5</sub>H<sub>sub.11</sub>, C<sub>sub.6</sub>H<sub>sub.5</sub>, and C<sub>sub.6</sub>H<sub>sub.13</sub>, ii) X<sup>sup.1</sup> is one or more members selected from the group consisting of (1) H, Na, Li, and K, (2) NH<sub>sub.4</sub>, (3) one or more organoamines selected from the group consisting of mono C<sub>sub.1-6</sub> amine, di C<sub>sub.1-6</sub> amine, tri C<sub>sub.1-6</sub> amine, mono ethanol amine, diethanol amine, triethanol amine, monoisopropanol amine, diisopropanol amine, triisopropanol amine, ethylene diamine diethylene triamine, triethylene tetraamine, and tetraethylene pentamine, b) R<sup>sup.1</sup> is one or more members selected from the group consisting of CH<sub>sub.3</sub>, C<sub>sub.2</sub>H<sub>sub.5</sub>, C<sub>sub.3</sub>H<sub>sub.7</sub>, C<sub>sub.4</sub>H<sub>sub.9</sub>, C<sub>sub.5</sub>H<sub>sub.11</sub> and C<sub>sub.6</sub>H<sub>sub.13</sub>, c) R<sup>sup.3</sup> is one or more members selected from the group consisting of H, CH<sub>sub.3</sub>, C<sub>sub.2</sub>H<sub>sub.5</sub>, C<sub>sub.3</sub>H<sub>sub.7</sub>, C<sub>sub.4</sub>H<sub>sub.9</sub>, C<sub>sub.5</sub>H<sub>sub.11</sub> and C<sub>sub.6</sub>H<sub>sub.13</sub>, d) Z<sup>sup.1</sup> is one or more members selected from the group consisting of Oxygen and Sulfur.

7. The fluid suspension composition of claim 6, wherein the Structure 1 comprises the following: a) wherein R<sup>sup.1</sup>=H b) wherein R<sup>sup.2</sup>=NHR<sup>sup.4</sup> i) wherein R<sup>sup.4</sup>=H c) wherein R<sup>sup.3</sup>=C<sub>sub.4</sub>H<sub>sub.9</sub>, and d) wherein Z<sup>sup.1</sup>=sulfur.

8. The fluid suspension composition of claim 6, wherein the fused binary particle comprises a) Structure 1, i) wherein R<sup>sup.1</sup>=H, ii) wherein R<sup>sup.2</sup>=NHR<sup>sup.4</sup>, iii) wherein R<sup>sup.4</sup>=H, iv), c) wherein R<sup>sup.3</sup>=C<sub>sub.4</sub>H<sub>sub.9</sub>, and d) wherein Z<sup>sup.1</sup>=sulfur and b) one or more chloride salts comprising one or more cations selected from the group consisting of i) magnesium, ii) calcium, and iii) mixtures thereof.

9. The fluid suspension composition of claim 1, wherein the fluid suspension meets the following: a) environmentally safe, b) having a flashpoint above 145° F., c) rated safe for contact with humans and animals, d) % loading of one or more phosphoryl nitrogenous compounds of 20-95%, e) fluid at temperature range of -40 to 100° C., and f) can be applied to the nitrogen source fertilizer granules through application by commercial sprayers and metered applicators onto the nitrogen source fertilizer granules mobilized by mixing, stirring and/or blending to ensure an even coating of the nitrogenous phosphoryl compounds on the surfaces of said nitrogen source fertilizer granules through commingling of said fluid suspension composition with said nitrogen source fertilizer granules.

10. The fluid suspension composition of claim 9, wherein the coated nitrogen source fertilizer granule possesses urease inhibition and nitrification inhibition properties.

11. The fluid suspension composition of claim 1, wherein said Organic Liquid System One comprises one or more aprotic liquids that are not reactive with one or more phosphoryl chlorides, and wherein said one or more aprotic liquids are selected from the group consisting of: a) one or more alkylene carbonates selected from the group consisting of i) ethylene carbonate, ii) propylene carbonate, and iii) butylene carbonate, b) one or more polyols capped with C<sub>sub.1</sub>-C<sub>sub.6</sub> organic acid wherein the polyol portion is selected from the group consisting of i) ethylene glycol, ii) 1,3 propylene glycol, iii) 1,2 propylene glycol, iv) butylene glycol, v) trimethylol propane, vi) trimethylol ethane, vii) pentaerythritol, viii) sorbitol, ix) sorbitan, x) glucose, xi) fructose, xii) galactose and xiii) glycerin, c) one or more alkylene glycol alkyl ethers acetates selected from the group consisting of i) dipropylene glycol methyl ether acetate, ii) tripropylene glycol methyl ether acetate, and iii) tripropylene glycol butyl ether acetate, d) one or more diesters selected from the group consisting of i) C<sub>sub.1</sub>-C<sub>sub.6</sub> dialkylsuccinate, ii) C<sub>sub.1</sub>-C<sub>sub.6</sub> dialkyladipate, iii)



C.sub.1-C.sub.6 dialkylmethylglutarate, iv) C.sub.1-C.sub.6 dialkyl glutarate, v) C.sub.1-C.sub.6 dialkyl ethylsuccinate, and vi) C.sub.1-C.sub.6 dialkyl maleate, e) one or more 1-alkyl-2-pyrrolidone selected from the group wherein the alkyl functionality is a C.sub.1-C.sub.8 alkyl radical, f) one or more members selected from the group consisting of i) dimethylacetamide, ii) dimethylformamide, iii) dimethyl-2-imidazolidinone, iv) isophorone, v) hexamethylphosphoramide, vi) 2-methoxyethyl ether, vii) N,N-dimethyldecanamide, viii) N,N-dimethyloctanamide, ix) limonene, x) sulfolane, g) one or more trialkylphosphates selected from the group represented by the structure: ##STR00038## wherein: R.sup.22 is alkyl radical-C.sub.1H.sub.3 to —C.sub.6H.sub.13 R.sup.23 is alkyl radical-C.sub.1H.sub.3 to —C.sub.6H.sub.13 R.sup.24 is alkyl radical-C.sub.1H.sub.3 to —C.sub.6H.sub.13 h) one or more dialkylene glycols dialkyl ethers, (glymes) selected from the structure: R.sup.11—O—(R.sup.12—O).sub.n—R.sup.13 wherein: R.sup.11 is one or more alkyl radicals selected from the group consisting of C.sub.1 to C.sub.6, R.sup.12 is one or more alkylene radicals selected from the group consisting of C.sub.1 to C.sub.4, R.sup.13 is one or more alkyl radicals selected from the group consisting of C.sub.1 to C.sub.6, and n is one or more integers selected from the group of 1 to 10, i) one or more hydrocarbons selected from the group consisting of i) one or more paraffinic liquids, ii) one or more cycloparaffinics, iii) one or more aromatic hydrocarbons, iv) aliphatic hydrocarbons, v) acyclic hydrocarbons, vi) unsaturated hydrocarbon, and vii) polyalphaolefins, j) one or more fatty acid alkyl esters wherein the one or more fatty acid functionality is selected from the group consisting of C.sub.8-C.sub.18 fatty acids and wherein the alkyl functionality is one or more C.sub.1-C.sub.6 alkyl radicals, and k) one or more C.sub.1-C.sub.18 fatty triglycerides.

12. The fluid suspension composition of claim 1, wherein the fused binary particle is formed in situ within the reaction medium from the reaction of one or more phosphoryl chlorides with one or more nitrogenous compounds wherein HCl by-product of the reaction is then reacted with one or more members selected from the group consisting of: magnesium carbonate, magnesium oxide, magnesium hydroxide, calcium carbonate, calcium oxide, calcium hydroxide, and combinations thereof.

13. The fluid suspension composition of claim 1, wherein the Organic Liquid System One meet the following: a) non-reactive with phosphoryl chloride functional groups, and b) not causing the degradation of the one or more nitrogenous phosphoryl compounds.

14. The fluid suspension composition of claim 1, wherein the composition further comprises one or more members selected from the group consisting of: a) colorant, b) scent, c) buffer, and d) one or more surfactants, wherein the one or more surfactants are selected from the group consisting of one or more cationic surfactants, one or more amphoteric surfactants, one or more Zwitterionic surfactants, one or more anionic surfactants and one or more nonionic surfactants.

15. The fluid suspension composition of claim 1, wherein the fluid suspension composition is low cost due to a low-cost manufacturing process that: a) does not have a by-product waste stream, b) does not require a by-product removal step, c) does not have a solvent exchange step, and d) has reduced cycle time.

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