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(54) **PRODUCTION OF AMMONIUM PHOSPHATES**

HERSTELLUNG VON AMMONIUMPHOSPHATEN

PRODUCTION DE PHOSPHATES D'AMMONIUM

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Description

TECHNICAL FIELD

[0001] The present invention relates in general to production of ammonium phosphates from phosphorus-containing solutions.

BACKGROUND

[0002] Phosphate rock (apatite) is the primary commercial source of phosphorus. The majority of the world's phosphate production is used to manufacture fertilizers to sustain agricultural production. The quality of phosphorus reserves is declining and the cost of extraction and processing is increasing. Associated heavy metals like cadmium substituting calcium can be present in phosphate rock at high levels requiring separation. Several countries restrict heavy metal levels in fertilizers. For example, in Sweden P fertilizers having cadmium contents above 5 mg Cd / kg P are imposed with a tax. Some European fertilizer producers have switched suppliers importing only raw material that have set cadmium limits.

[0003] All water-soluble phosphate salts such as soluble fertilizers are derived from phosphoric acid. Phosphoric acid is produced commercially by either a 'wet' or a thermal process. Wet digestion of phosphate rock is the most common process. Thermal processing is energy intensive and therefore expensive. For that reason, quantities of acid produced thermally are much smaller and mainly used for production of industrial phosphates.

[0004] Phosphoric acid for fertilizer production is almost solely based on wet digestion of rock phosphate. The process is mainly based on dissolution of apatite with sulfuric acid. After dissolution of the rock, calcium sulfate (gypsum) and phosphoric acid are separated by filtration. To produce merchant-grade phosphoric acid, high acid concentrations are required and water is evaporated. Calcium sulfate exists in a number of different crystal forms depending on the prevailing conditions such as temperature, phosphorus concentration in the slurry, and level of free sulfate. Calcium sulfate is either precipitated as di-hydrate ($CaSO_4 \cdot 2H_2O$) or as hemi-hydrate ($CaSO_4 \cdot ½H_2O$). Phosphoric acid produced through this process is characterized by a low purity.

[0005] All ammonium phosphate salts are derived from phosphoric acid. Merchant-grade phosphoric acid, having a concentration of about 54% P_2O_5, is neutralized with ammonia to form either mono-ammonium phosphate (MAP) or di-ammohium phosphate (DAP) by controlling the ammonia to phosphoric acid mole ratio during the neutralization process. Ammonia is used in liquid or gaseous form. Liquid anhydrous ammonia is usually preferred since surplus heat from other systems is necessary for vaporizing liquid ammonia into a gaseous form. The neutralization of merchant-grade phosphoric acid with ammonia is usually performed in several stages using several reaction vessels. The mole ratio of ammonia to phosphoric acid in the pre-reactor/s is normally held at a level which gives the maximum solubility for the slurry (between 1.4 and 1.45 for production of DAP and usually less than 1 for production of MAP). For operation control, the ammonia to phosphoric acid mole ratio is determined by monitoring the pH of the slurry. Excess heat of reaction is removed from the pre-neutralizer/s by adding water to the reactor/s. Evaporation of the water cools the slurry. As the mole ratio of ammonia to phosphoric acid is increased over 1, un-reacted ammonia escapes from the reactor and the gaseous vapors released must be scrubbed with an acid. The slurry from the pre-neutralization reactor/s which usually contain between 16 to 23% water is usually fed into an ammoniator-granulator to complete the addition of ammonia for the desired product. Completion of the neutralization and additional evaporation of water results in solid particles being formed. It is necessary to recover the un-reacted ammonia from the gaseous vapors by scrubbing with an acid. Thereafter, the solid ammonium phosphates are usually dried in a separate reactor to reduce moisture content. Loss of ammonia from the dryer is usually recovered by scrubbing with acid. The solid ammonium phosphates are normally cooled by passing air through a cooling reactor.

[0006] For several applications such as fertigation (the application of water-soluble fertilizers in the irrigation water) and foliar fertilization (spraying fertilizers on leaves) there is a need for fully-soluble ammonium phosphates to avoid clogging of the fertigation equipment by non-dissolved solids. Wet-process phosphoric acid contains a substantial amount of impurities such as iron, aluminum, calcium, magnesium, cadmium, etc. which form water-insoluble solids upon neutralization with ammonia and thus fertilizer-grade ammonium phosphates are not completely water-soluble. Therefore, fully-soluble P fertilizers for fertigation purposes must be specially produced from purified phosphoric acid.

[0007] The current technology for phosphoric acid purification is based on extraction of impure wet-process phosphoric acid into an organic solvent (ketones, tri-alkyl phosphates, alcohols, etc.) followed by back extraction with water forming a dilute and pure phosphoric acid which is thereafter concentrated by water evaporation. Purified phosphoric acid is thereafter neutralized with ammonia forming fully-soluble ammonium phosphate products according to the procedure described above.

[0008] In general, two processes for solvent extraction of phosphoric acid can be identified: a) partial extraction of phosphoric acid from concentrated solutions, and b) complete extraction of phosphoric acid in the presence of other

acids or salts.

[0009] Partial extraction of phosphoric acid from concentrated phosphoric acid produced by digestion of apatite with sulfuric acid is the most common process. In this process, only part of the phosphoric acid is extracted into an organic phase. The remaining non-extracted phosphoric acid together with metal impurities is used for production of low-grade phosphate salts such as different fertilizers. Any solvent capable of solvating phosphoric acid can be used in this process, both solvents that have a reasonably constant distribution coefficient down to fairly low concentrations such as alcohols, and solvents which show very little extraction capacity for phosphoric acid below a specific threshold concentration, i.e., the distribution coefficient is very sharply concentration dependent such as for ethers, esters and selected ketones.

[0010] A different approach is to obtain complete extraction of phosphoric acid in the presence of high concentrations of other acids or salts. The addition of a second acid such as H_2SO_4 (US patent 3573005) or a salt such as $CaCl_2$ (US patent 3304157) can improve the distribution coefficient (the distribution ratio of solute between the organic and aqueous phases) of phosphoric acid even at fairly low phosphoric acid concentrations. Although the added acid is also extracted by the solvent its proportion in the organic solvent is normally less than that in the feed solution. Suitable solvents are alcohols, trialkyl phosphates such as tributyl phosphate, etc. which show reasonably constant distribution coefficients down to fairly low phosphoric acid concentrations. The method is recommended for extracting phosphoric acid from remaining impure phosphoric acid resulting from the partial extraction process. A main disadvantage of this approach is that the final aqueous phase is rich in the added acid (i.e. sulfuric acid) or salts together with impurities, which might not have a final use.

[0011] The disadvantages of the state-of-the art technologies for production of ammonium phosphates are numerous. The phosphoric acid as produced from the gypsum filter is not suitable for direct manufacture of ammonium phosphate salts. The acid must be further concentrated by water evaporation to a suitable phosphoric acid concentration (usually about 54% P_2O_5). Normally, concentration of phosphoric acid is done in three stages. The weak acid from the filter (28% P_2O_5) is evaporated to 40% P_2O_5 in a single stage vacuum evaporator. The acid is then clarified to remove precipitated solids and the clarified acid is then concentrated to 54% P_2O_5 in two stages. The inter-stage concentration is about 48% P_2O_5. The 54% P_2O_5 acid is used for ammonium phosphate production according to the procedure described above.

[0012] To concentrate acids through evaporation is a very energy-intensive process. The amount of steam required for concentrating phosphoric acid usually varies between 2.5 - 5 tons of steam per ton of phosphorus, depending on production conditions. The energy demand for concentration of phosphoric acid is a major production cost. Expensive equipment such as steam distribution systems, evaporators, effluent gas scrubbers, condensation systems, cooling water systems, liquid effluent treatment systems and acid storage facilities are necessary for production of merchant-grade phosphoric acid. Furthermore, additional equipment is needed for the neutralization of phosphoric acid with ammonia in several stages, drying, cooling and scrubbing of ammonia from gaseous vapors. A major disadvantage is that the quality of the ammonium phosphate product is set by the quality of the apatite raw-material. Produced ammonium phosphates of fertilizer grade are generally contaminated with heavy metals such as cadmium and are not fully-soluble and therefore not suitable for use in applications such as fertigation.

[0013] Production of completely-soluble ammonium phosphate salts (technical grade) is more complex and requires purification of merchant-grade phosphoric acid by solvent extraction prior to neutralization with ammonia. The energy costs for water evaporation in this process are much higher since the phosphoric acid needs to be concentrated twice: a) the acid must be concentrated prior to solvent extraction, and b) the purified phosphoric acid is dilute and has to be concentrated again by water evaporation. Additional equipment for production of fully-soluble ammonium phosphates includes facilities for pretreatment prior to solvent extraction, liquid-liquid extraction equipment, liquid-liquid stripping equipment and evaporators for concentrating purified acid.

[0014] US patent 3,298,782 describes a process for the purification of wet-process phosphoric acid which consists of a) extracting phosphoric acid from an aqueous phase to an alcohol-amine organic phase, b) separating the alcohol-amine phase from the aqueous phase, and c) recovering purified phosphoric acid from the alcohol-amine phase. The main objective was to recover purified phosphoric acid by back-extraction with water. In the text it is also mentioned that phosphate salts can be recovered from the alcohol-amine phase by reaction with a base. In one of the examples, an aqueous ammonia solution was used to strip the phosphate from the organic phase into an aqueous phase.

[0015] US patent 3,458,282 describes a method for purifying phosphoric acid by utilizing an amine dissolved in an organic diluent (e.g. kerosene) as an extractant phase to remove either certain impurities from phosphoric acid or to extract phosphoric acid from the aqueous phase. When phosphoric acid was extracted with the amine-diluent solvent, the main objective was to obtain purified aqueous phosphoric acid by back-extraction with water, or to obtain an aqueous phosphate salt solution by reaction with an aqueous base. In the patent text it is also mentioned that it may be possible to remove phosphate from the amine by vaporizing off the organic diluent and treating the remaining material with an aqueous solvent or a gas such as ammonia to precipitate phosphate. To vaporize and condense very large quantities of an organic diluent such as kerosene is both costly and complex.

[0016] US patent 3,894,143 describes a process for obtaining crystallized ammonium phosphate of good quality from wet-process phosphoric acid and ammonia. The process consists of a) forming a mixture of aqueous phosphoric acid

and acetone in which all components are miscible with water, b) precipitating impurities by addition of ammonia and separating the precipitated impurities to form a purified mixture, c) contacting the purified mixture with ammonia to produce ammonium phosphate crystals and a supernatant liquid, and d) Separating the ammonium phosphate crystals from the supernatant liquid and distilling the supernatant to separate the acetone for recycling. The disadvantages of this method include distillation of large quantities of acetone, limited yield of ammonium phosphates, and production of large quantities of dilute aqueous ammonium phosphate effluents. The process was therefore not applied in the industry.

[0017] In the published international patent application WO 2008/115121, a method and an arrangement for phosphorus recovery are disclosed. Phosphorus ions are extracted from solutions by adsorbing phosphorus ions in a scavenger and by releasing the phosphorus ions into an eluate during regeneration of the scavenger. The regeneration is performed by ammonia. Phosphate anions are precipitated in form of tri-ammonium phosphate upon introduction of excess amounts of ammonia. The ammonia remaining in solution after the precipitation of tri-ammonium phosphate is reused for regenerating the scavenger. Unfortunately, tri-ammonium phosphate is unstable at ambient temperature and atmospheric pressure resulting in the decomposition of the crystal accompanied with release of ammonia. Such unstable crystalline solid is not suitable for direct use in agriculture.

[0018] In the US patent 3,415,619, a process for making ammonium phosphate is disclosed. Water-soluble ammonium phosphate is achieved by extracting a substantially iron-free aqueous phosphoric acid, derived from the reaction of calcium phosphate-containing ore and a strong mineral acid, into a water-immiscible trialkyl phosphate extractant, separating the phosphoric acid-laden extractant from the residual aqueous phase, removing the calcium impurities therefrom, contacting the phosphoric acid-laden extractant with anhydrous ammonia at a temperature of between about 20 and 90°C, and separating solid, water-soluble ammonium phosphate from the extractant. The solid ammounium phosphate is indicated to be washed with low boiling hydrocarbon solvents to remove organic extractant adhesive thereto.

[0019] In the US patent 3,342,579, slowly soluble ammonium polyphosphate and methods for its manufacture are disclosed. An apparatus has a vessel with an agitator and inlets for a superphosphoric acid and anhydrous ammonia. The reacted material is filtered to obtain a solid product of crystals.

[0020] GB 636,035 discloses improvements of processes of producing diammonium phosphate. Mono-ammonium phosphate is introduced into a solution of diammonium phosphate in a reactor and anhydrous ammonia is fed into the reactor. Diammonium phosphate crystals are collected at the chamber bottom.

[0021] US 4,781,905 discloses a process for producing phosphoric acid and/or phosphates from wet-process phosphoric acid and an extractant therein. A crude acid is extracted with a water immiscible solvent mixture consisting of mixed trialkyl phosphine oxide and a dilutent. A part of P2O5 in the crude acid is extracted into the solvent mixture and the balance remains in the raffinate. Pure phosphoric acid or phosphates are produced by stripping the loaded solvent with appropriate aqueous phase and secondary calcium phosphate fertilizer is obtained by neutralizing the raffinate with calcium carbonate.

[0022] EP 0 248 256 A2 discloses a process for the production of ammonium phosphate where ore containing phosphate and calcium ions is attacked by with nitric acid, the resultant reactant mixture is treated with a polar organic solvent to form an aqueous solution containing traces of solvent, impurities and calcium nitrate and an organic extract containing phosphoric and nitric acid, the organic extract is purified and ammoniated to form a precipitate of the mono and/or diammonium phosphate, a light phase containing the solvent, and a heavy aqueous phase containing ammonium nitrate and ammonium phosphate, the light phase is separated and recycled to the extraction step, the ammonium phosphate precipitate is separated from the heavy aqueous phase, the separated ammonium phosphate is washed, the washing waters of the ammonium phosphate and part of the heavy aqueous phase containing ammonium nitrate and ammonium phosphate is recycled to the purification step while the aqueous solution from the purification step is cycled into the extraction step.

[0023] US 3,443,889 discloses a process for the manufacture of mono-ammonium phosphate crystals where the crystals are washed with a saturated solution of ammonium phosphate.

[0024] There is a need for an improve method for production of fully-soluble ammonium phosphates such as mono-ammonium phosphate (MAP) or di-ammonium phosphate (DAP), in which the costs associated with the concentration of phosphoric acid by evaporation of water are excluded.

SUMMARY

[0025] A general object of the present invention is to improve methods and devices for production of ammonium phosphate from phosphorus-containing solutions. A further object of the present invention is to provide a method for production of fully-soluble ammonium phosphates without the need for concentrating phosphoric acid by evaporation of water. Another object of the present invention is to provide a cost effective method for production of ammonium phosphates without the need for drying and scrubbing ammonia from effluent vapors. A further object of the present invention is to provide recovered ammonium phosphates in a form that easily can be utilized for fertilizing purposes.

[0026] The above objects are achieved by methods and devices according to the enclosed patent claims. In general

words, in a first aspect, a method for production of ammonium phosphates comprises providing of a phosphorus-loaded water immiscible liquid phase, adding of anhydrous ammonia to the water immiscible liquid phase, precipitating of mono-ammonium phosphate and/or di-ammonium phosphate from the water immiscible liquid phase and extracting of the precipitated mono-ammonium phosphate and/or di-ammonium phosphate from the water immiscible liquid phase. The method further comprises controlling of a temperature of the water immiscible liquid phase during the adding and precipitating to a predetermined temperature interval.

[0027] In a second aspect, an arrangement for production of ammonium phosphates comprises a mixing volume. The mixing volume has an inlet adapted for a phosphorus-loaded water immiscible liquid phase and an inlet adapted for adding of anhydrous ammonia into the water immiscible liquid phase. The arrangement further comprises a heat exchanger arranged in thermal contact with the water immiscible liquid phase. A controller is arranged for operating the heat exchanger to keep the water immiscible liquid phase in the mixing volume within a predetermined temperature interval. The arrangement also comprises a precipitate remover arranged for removing crystals of precipitated mono-ammonium phosphate and/or di-ammonium phosphate from the mixing volume.

[0028] Preferably, the phosphorus is extracted into the phosphorus-loaded water immiscible liquid phase from solutions by adsorbing phosphorus into a liquid scavenger having affinity for phosphorus, thereby creating the phosphorus-loaded water immiscible liquid phase. The phosphorus is removed by the addition of anhydrous ammonia from the liquid scavenger during regeneration of the scavenger. The temperature of the liquid scavenger is preferably maintained below its boiling point. The regenerated scavenger is preferably continuously recycled in order to extract phosphorus from further feed solutions.

[0029] The separated crystalline ammonium phosphates are washed, preferably with an aqueous solution in which the pH is controlled to a predetermined level. The scavenger initially adhering to the crystals is separated from the dense aqueous phase in a phase separator. The so separated scavenger is continuously recycled in order to extract phosphorus from a feed solution. The aqueous wash solution is also recycled for further washing.

[0030] The washed ammonium phosphate crystals are thereafter dried. The drying can preferably at least to a part be performed by heat obtained from the heat exchange process cooling the mixing of anhydrous ammonia with phosphoric acid.

[0031] The invention provides for extraction of phosphorus from process streams in form of high quality products such as ammonium phosphate fertilizers in an environmentally friendly and cost effective way. The invention enables production of MAP or DAP independent of the initial composition of the precipitated crystals. According to the invention, phosphorus can be recovered as a concentrated, water-soluble, inorganic product of a high quality, i.e. high phosphorus availability to plants and minor heavy metal contamination. Another advantage of the present invention is that it enables to reuse the scavenger without the need for distillating large quantities of liquid scavenger.

BRIEF DESCRIPTION OF THE DRAWINGS

[0032] The invention, together with further objects and advantages thereof, may best be understood by making reference to the following description taken together with the accompanying drawings, in which:

FIG. 1 is a block scheme of an embodiment of an arrangement for recovery phosphorus;

FIG. 2 is a block scheme of an embodiment of an arrangement for production of ammonium phosphates according to the present invention;

FIG. 3 is a flow diagram of an embodiment of a method according to the present invention; and

FIGS. 4-5 are block schemes of other embodiments of an arrangement for production of ammonium phosphates according to the present invention.

DETAILED DESCRIPTION

[0033] Throughout the drawings, the same reference numbers are used for similar or corresponding elements.

[0034] Some often used terminology in the present disclosure is to be interpreted as follows:

Scavenger- material having affinity for solute species, e.g. material adsorbing ions or acids, by ion association or solvation mechanisms. The term comprises different kinds of extractants contained in solvents,

Solvent - A liquid phase, typically organic, which preferentially dissolves extractable solute species from an aqueous solution.

Extractant - An active component, typically organic, of a solvent enabling extraction.

Solvent extraction (liquid liquid extraction) - The separation of one or more solutes from a mixture by mass transfer between immiscible phases in which at least one phase typically is an organic liquid.

Regeneration - The displacement from the scavenger of the ions or acids removed from the process solution to make the scavenger ready for reuse.

Diluent - A liquid, typically organic, in which an extractant is dissolved to form a solvent.

Raffinate - An aqueous phase from which a solute has been removed by extraction.

[0035] The main objective of the invention is to provide a simple and cost effective method for production of ammonium phosphates. The method enables production of ammonium phosphates without the need for concentrating phosphoric acid by water evaporation. Furthermore, the method enables production of fully-soluble and pure mono-ammonium phosphate or di-ammonium phosphate salts.

[0036] One possible general approach to production of ammonium phosphates from phosphoric acid is to extract phosphoric acid into an organic solvent and use ammonia as a mean to precipitate ammonium phosphates directly from the organic phase. As will be found further below, use of anhydrous liquid ammonia or gaseous anhydrous ammonia turns out to be favorable. Anhydrous liquid ammonia or gaseous anhydrous ammonia was not tested as a mean to precipitate ammonium phosphates directly from an organic phase in the US patent 3,298,782 the US patent 3,458,282. One reason for not testing such alternatives may be that there are several major difficulties associated with such general approach such as non sufficient phosphoric acid loading in the organic phase at low phosphoric acid concentrations, problems of heat generation and evaporation of the solvent, loss of expensive solvent adhering to precipitates, difficulties to predict the form of precipitated ammonium phosphate, and difficulties to separate impurities. The difficulties are described in the following text and must be overcome to give a commercially interesting approach. Solvents used for purification of phosphoric acid such as ketones, tri-alkyl phosphates and alcohols require high concentrations of phosphoric acid in the feed solution in order to obtain a sufficient high phosphoric acid loading in the organic phase for a liquid-liquid extraction process to be practical. The use of such solvents requires concentration of phosphoric acid by water evaporation prior to phosphoric acid extraction.

[0037] Even if sufficient high phosphoric acid loading can be achieved in the organic phase, for the liquid-liquid extraction process to be practical, then the reaction of ammonia with concentrated phosphoric acid is known to be highly exothermic which could lead to the evaporation of the solvent. Stripping a solvent, loaded with phosphoric acid, with an aqueous base generate a substantial amount of heat. The problem of such heat generation is described in US patent 4,112,118, which relates to a process for preparing phosphate salts from phosphoric acid extracted into an organic solvent by stripping with an aqueous base. In order to minimize heat production the mole ratio of base to phosphoric acid had to be reduced to between 0.1:1 and 0.5:1 in order to enable the process to be operational. This emphasizes the problem with heat evolution and evaporation of the solvent when precipitating mono-ammonium phosphate (MAP) or di-ammonium phosphate (DAP) with anhydrous ammonia directly in the organic phase since the ammonia to phosphoric acid mole ratio has to be above 1. It is also difficult to predict the amount of heat generated when reacting anhydrous ammonia and phosphoric acid in a specific solvent mixture since enthalpy data are specific for each solvent mixture and must therefore be determined experimentally.

[0038] In addition to the difficulties due to heat generation and evaporation of the solvent, large amounts of solvent are expected to remain adhering to the precipitated ammonium phosphate crystals and the loss of expensive solvent mixture would be economically unacceptable, at least in some applications. Removal of adhering solvent by distillation is difficult since the boiling point for solvents such as tributyl phosphate (289°C) exceeds the melting point for mono-ammonium phosphate (190°C). Furthermore, the process must be controlled to produce stable ammonium phosphate salts such as mono-ammonium phosphates or di-ammonium phosphates, which are the desired end products. Finally, methods for removal of impurities such as metals, silica, fluorine, etc. must be identified in order for such a process to be applicable.

[0039] All the now identified above mentioned difficulties led to that the above mentioned general approach was not tested and not implemented in the industry prior to the present invention.

[0040] Here below, an embodiment of a process for producing ammonium phosphates from a phosphorus-containing mineral according to the present invention is described in details in connection with Fig. 1. However, although being an advantageous approach, the present invention is not limited to recovery of phosphorus from minerals, but is applicable to many different systems providing phosphate ions / phosphoric acid. A similar process with minor modifications can be used e.g. for extracting phosphorus from ash of incinerated sewage sludge, ash of incinerated animal by-products, P rich streams within sewage treatment works, industrial effluents, etc.

[0041] An embodiment of an arrangement 100 for recovery phosphorus is shown in Fig. 1. Apatite concentrate 2 obtained by the beneficiation of mined phosphate rock is subjected to digestion with sulfuric acid 1 in a digester 4 according to known methods giving digested apatite 3. Known process schemes include di-hydrate, hemihydrate, hemi-hydrate-dihydrate, and dihydrate -hemihydrate processes. Calcium sulfate (gypsum) 5 and a phosphorous-containing aqueous solution 7, in this embodiment phosphoric acid, are thereafter separated by filtration in a digester separator 6. The filter-grade phosphoric acid 7 is optionally pretreated to remove impurities by known methods. The entire arrangement for digestion of apatite 4 and separation of impurities 6 can be seen as a pretreatment for providing a feed solution to a liquid-liquid extraction process, i.e. a phosphorus-containing aqueous solution 7. The feed solution is provided to an arrangement 10 for production of ammonium phosphates, in this embodiment provided by liquid-liquid extraction. Liquid-liquid extraction involves selective transfer of solute between two immiscible phases, an aqueous phase and an organic phase. The two immiscible phases are first thoroughly mixed in order to facilitate the transfer of solute and then separated.

[0042] In order to recover phosphate from phosphorus-containing aqueous solution 7, a liquid-liquid extraction process is utilized, where a feed aqueous solution containing phosphate ions / phosphoric acid is exposed to an organic phase (herby named scavenger). The phosphate ions / phosphoric acid are thereby extracted into the scavenger. This is described more in detail further below. In general terms, the arrangement 10 for production of ammonium phosphates derives ammonium phosphate 9 from the phosphorus-containing aqueous solution 7, giving a remaining process liquid 8, which preferably can be reused together with the sulfuric acid 1 for further digestion.

[0043] An embodiment of an arrangement 10 for production of ammonium phosphates is illustrated more in detail in Fig. 2. An extraction section 12 is arranged for allowing adsorbing of phosphorous from a phosphorous-containing aqueous solution 7 into a liquid scavenger 15 having affinity for phosphorous. An aqueous solution depleted in phosphorous leaves the extraction section 12. When used in conjunction with the arrangement 100 for recovery phosphorus shown in Fig. 1 the phosphorus depleted aqueous solution becomes the remaining process liquid 8. An outlet from the extraction section 12 for scavenger 15 loaded with phosphorous is connected to an inlet 22 for a phosphorus-loaded water immiscible liquid phase of a mixing volume 20, whereby the scavenger 15 loaded with phosphorous forms a phosphorus-loaded water immiscible liquid phase 14. As also will be discussed further below, an inlet for scavenger 15 depleted from phosphorous 16 to the extraction section 12 is connected, at least indirectly, to the mixing volume 20. This inlet for scavenger 15 depleted from phosphorous 16 is thus arranged for reusing regenerated scavenger 15 formed in the mixing volume 20 for further adsorbing of phosphorous in the extraction section 12.

[0044] Any organic solvent (scavenger) capable of removing phosphorus from aqueous solutions can be used in the liquid-liquid extraction of the extraction section. The mechanism of phosphorus extraction can be ion association, solvation of phosphoric acid or both. The composition of the scavenger should be selected according to the concentration of the phosphoric acid feed, presence of additional acids or salts, etc. in order to obtain a high loading capacity and an effective operational extraction process.

[0045] Processing dilute phosphoric acid streams requires the use of scavengers with a strong extraction power for phosphate. Liquid scavengers suitable for extracting phosphoric acid from dilute solutions are liquid amines. In general, primary, secondary and tertiary liquid amines can be used. Amine extractants have a low water-solubility, good miscibility with organic solvents, good chemical stability, high selectivity and a strong binding power enabling acid extraction from very dilute solutions. Preferably, amines should be selected having a nitrogen atom attached to a large organic molecule containing more than seven aliphatic or aromatic carbon atoms. Such organic amines are highly soluble in organic solvents and almost insoluble in water. In contact with an acid containing solution, the amine base reacts with the acid to form a protonated positive charge, which associates with the anion of the acid. Organic amines can extract more acid than the stoichiometric ratio of 1 acid molecule per 1 molecule of amine through solvation of additional neutral acid molecules. In concentrated phosphoric acid, up to four phosphate molecules are extracted per molecule of liquid amine. High concentration of amines can polymerize to form a third, non-wanted, separate phase. However, the formation of the non-wanted third phase can be avoided by dissolving the amines in another organic solvent which is a strong Lewis base such as tributyl phosphate or alcohols. Mixtures of solvating extractants such as tri butyl phosphate and liquid amines are preferably used together to efficiently extract phosphate at both high and low concentrations.

[0046] Solvating extractants are liquid organic molecules containing oxygen atoms (alcohols, esters, ethers, ketons, trialkyl phosphates, amides, etc.) which interact with phosphoric acid to form H-associations. During this mechanism, the extractant replaces part of the water molecules and solvates the phosphoric acid molecule in the organic phase. The binding of phosphoric acid is weak through H-association. Solvating extractants can be divided into two groups: a) solvents that have a reasonably constant distribution coefficient down to fairly low concentrations such as alcohols, tributyl phosphate, etc., and b) solvents which show very little extraction capacity for phosphoric acid below a specific threshold concentration, i.e., the distribution coefficient is very sharply concentration dependent such as for ethers, esters and selected ketones e.g. methyl isobutyl ketone. For processing of filter-grade phosphoric acid it is preferred to use mixtures of solvents that have a reasonably constant distribution coefficient down to fairly low concentrations such as tributyl phosphate and liquid amines which have a strong extraction power for phosphate even at very low concentrations due to an ion association mechanism.

[0047] By extracting phosphoric acid with two mechanisms coupled to each other, i.e., phosphate adsorption by ion association and solvation of neutral phosphoric acid, mixtures of solvating extractants such as tributyl phosphate and liquid amines such as trioctyl amine are effective scavengers both for highly concentrated as well as highly diluted phosphoric acid streams. The distribution coefficients involved in acid extraction by such scavengers are high, which means that the number of contact stages necessary, is low. The organic to aqueous volume ratio for extracting phosphoric acid from filter-grade phosphoric acid having a concentration of 5M can be below 10:1 and preferably below 5:1. The obtained phosphate concentration in the scavenger is preferably above 1M. In addition, mixture of solvating extractants and liquid amines are selective towards anions and do not bind positively charged metals, which means that metal contaminants are separated from the extracted phosphoric acid by remaining in the aqueous solution.

[0048] The filter-grade phosphoric acid is fed to a liquid-liquid extraction process characterized by the above described scavenger. The liquid-liquid extraction process is preferably a continuous liquid-liquid extraction process using preferably liquid-liquid extraction equipment such as pulsed-columns. However, any other liquid-liquid extraction equipment can be used such as, agitated columns, non-agitated columns, mixer settlers, inline mixers, centrifugal contractors, etc.

[0049] The raffinate, which is depleted in phosphate, is further treated to remove metal precipitates. It can then be used for apatite dissolution or gypsum washing, (see e.g. Fig. 1).

[0050] The scavenger which is loaded with phosphorus is optionally scrubbed to remove co-extracted impurities forming a phosphorus-loaded water immiscible liquid phase.

[0051] Returning to Fig. 2, the phosphorus-loaded water immiscible liquid phase 14 is thereafter treated with anhydrous ammonia to form crystalline ammonium phosphate directly in the scavenger. To this end, the arrangement 10 for production of ammonium phosphates comprises a mixing volume 20 having an inlet 22 for the phosphorus-loaded water immiscible liquid phase 14. The mixing volume has furthermore an inlet 24 for adding anhydrous ammonia 18 into the phosphorus-loaded water immiscible liquid phase 14. Anhydrous liquid ammonia or gaseous anhydrous ammonia can be utilized.

[0052] In order to form a solid ammonium phosphate crystal, several ammonia molecules react with several phosphoric acid or hydrogen phosphate molecules to form a crystal structure by H-bonding of ammonium molecules to phosphate molecules. The weak bonds easily dissociate in contact with water, which makes ammonium phosphate crystals highly water-soluble. It is known that several crystalline ammonium phosphate solid phases can be obtained by contacting ammonia, phosphoric acid and water at different concentrations and temperatures. The following crystalline solid phases are known: $(NH_4)_7H_2(PO_4)_3$, $(NH_4)_3PO_4$, $(NH_4)_3PO_4{\cdot}2H_2O$, $(NH_4)_3PO_4{\cdot}3H_2O$, $(NH_4)HPO_4{\cdot}H_2O_2$, $(NH_4)_2HPO_4$, $(NH_4)_2HPO_4{\cdot}2H_2O$, $NH_4H_2PO_4$, $(NH_4)_3H_2(PO_4)_4$, $NH_4H_5(PO_4)_2{\cdot}H_2O$, and $NH_4H_5(PO_4)_2$. Several of these crystalline ammonium phosphates are unstable at ambient temperature and atmospheric pressure resulting in the decomposition of the crystal into another structure accompanied with release of ammonia. Such unstable crystalline solid phases are not suitable for use in agriculture.

[0053] It is known that perfectly dry ammonia will not combine with perfectly dry hydrogen chloride to form the ammonium salt. Moisture is thus necessary to bring about the reaction. Extraction of phosphoric acid with scavengers such as tributyl phosphate is accompanied with co-extraction of water molecules. The mole ratio of co-extracted water to tributyl phosphate varies between 0.7 to 1.7 $[H_2O]_{org}/[TBP]_{org}$ depending on the concentration of phosphoric acid in the scavenger and the temperature

[0054] It has, however, surprisingly been found that by reacting anhydrous ammonia with phosphoric acid or phosphate molecules in organic scavengers (e.g. tributyl phosphate, mixtures of tributyl phosphate and alcohols, mixtures of tributyl phosphate and amines), the crystalline solid phase obtained has an ammonium to phosphate mole ratio which is about 1 and the crystals are stable at ambient temperature and atmospheric pressure. The crystalline solid phase was found to be composed primarily of mono-ammonium phosphate (MAP) $NH_4H_2PO_4$. A minor fraction of di-ammonium phosphate (DAP) $(NH_4)_2HPO_4$ was also present. Thus crystalline solid ammonium phosphates, surprisingly produced by contacting anhydrous ammonia with phosphate in the above described scavengers, can be used directly for agricultural purposes.

[0055] The precipitation of phosphorus from the above described organic solvents was found to be highly effective enabling phosphorus removal efficiency above 99%. The high stripping efficiency enables high operational capacity during extraction of phosphoric acid. In contrast to stripping with water, which is based on an equilibrium reaction leading to incomplete phosphorus stripping, the reaction of phosphorus with ammonia is not based on equilibrium and phosphorus stripping is complete.

[0056] It is known that anhydrous ammonia is soluble in different organic solvents such as ethanol (10% by weight at 25°C), methanol (16% by weight at 25°C), etc. However, the solubility of ammonia in tributyl phosphate is only 0.6% by weight at 20°C and the solubility decreases with increasing temperatures. Above 35°C the solubility of ammonia in tributyl phosphate is insignificant. Thus, the amount of residual ammonia in the scavenger after precipitation of phosphorus is very low. It was also found that there is a correlation between phosphorus loading in the organic solvent to pH and conductivity. Conductivity decreases and pH level increases with decreasing concentration of phosphoric acid in the solvent. Addition of ammonia can thereby be controlled by monitoring the conductivity and/or pH of the scavenger to enable operation without excess ammonia. To this end, again referring to Fig. 2, the arrangement 10 for production of

ammonium phosphates comprises a sensor 26, in this embodiment a sensor for monitoring of a conductivity of the water immiscible liquid phase, in the mixing volume 20. The arrangement 10 for production of ammonium phosphates further comprises an adder control unit 28 connected to the sensor 26 and arranged for controlling an amount of added anhydrous ammonia 18 in response to the monitored conductivity. In an alternative embodiment, the sensor 26 is a sensor for monitoring of a pH of the water immiscible liquid phase in the mixing volume 20, and the adder control unit 28 is consequently arranged for controlling an amount of added anhydrous ammonia 18 in response to the monitored pH.

[0057] Reaction of ammonia with phosphoric acid is as mentioned above highly exothermic and a substantial amount of heat is expected to be produced during this reaction. However, it was surprisingly found that the heat generated when neutralizing a solvent loaded with 1.42 M H_3PO_4 (~50°C $liter^{-1}$ solvent) is lower than required for vaporizing the solvent. The temperature of the scavenger can therefore in a practical manner be controlled by heat-exchanging to a temperature within a desired interval. The arrangement 10 for production of ammonium phosphates comprises a heat exchanger 30 arranged in thermal contact with the water immiscible liquid phase 15. In the embodiment of Fig. 2, the heat exchanger 30 is arranged in the mixing volume 20 for extracting heat from the water immiscible liquid phase 15 within the mixing volume 20. The temperature in the mixing volume 20 where the scavenger 15 and ammonia 18 is mixed is preferably measured by a thermometer 32 and this measure is used by a controller 34 for operating the heat exchange in such a way that the temperature of the water immiscible liquid phase 15 in the mixing volume 20 is held within a predetermined temperature interval. Preferably, the scavenger to be used in the extraction section 12 is cooled to a temperature which is below 60°C since lower temperatures favor phosphoric acid extraction by the scavenger 15.

[0058] In one particular embodiment, the cooling of the heat exchanger 30 can be achieved by vaporizing liquid anhydrous ammonia into a gaseous form. In such a manner cooling can be obtained by using ammonia which is an ingredient in the final product. This is indicated in Fig. 2 by the broken arrows 36 and 37. To that end, the arrangement 10 for production of ammonium phosphates comprises a source of liquid ammonia 19. A heater unit 23 is connected to the source of liquid ammonia 19 and is connected to or integrated with the heat exchanger 30. The heater unit 23 is arranged for utilizing at least a part of heat extracted in the heat exchanger 30 to produce gaseous ammonia, used as the anhydrous ammonia 18. This means that the inlet 24 for adding anhydrous ammonia 18 of the mixing volume 20 is connected for extracting the gaseous ammonia from the source of liquid ammonia 19.

[0059] Alternatively, cooling can be achieved by any other means such as heat exchange with cooling water. This alternative is preferable when it is desired to recover the generated heat for use in other processes or used for drying the recovered ammonium phosphate crystals, which will be discussed further below.

[0060] The crystalline solid ammonium phosphates are thereafter separated from the scavenger by known solid-liquid separation techniques such as filtration, decantation, centrifugation, etc. In Fig. 2, a precipitate remover 40 is arranged for removing crystals of precipitated mono-ammonium phosphate and/or di-ammonium phosphate from the mixing volume 20. The phosphorus-depleted scavenger 16 is then preferably continuously recycled in order to again extract phosphate from a feed solution in the extraction section 12.

[0061] Relatively large amounts of scavenger remain adhering to the separated ammonium phosphate crystals. These amounts are typically large enough that a loss of expensive solvent mixture generally would be economically unacceptable. It is therefore preferable to also recycle these amounts of scavenger. In the embodiment of Fig. 2, the arrangement 10 for production of ammonium phosphates comprises washing arrangement 50, in turn comprising a washer 52 connected to the precipitate remover 40. The washer 52 is arranged for washing the separated ammonium phosphate crystals. A drier 54 is connected to the washer 52 and is arranged for drying the washed crystals. A separator 60 is connected to the washer 52 and is arranged for separating residual scavenger 17 washed from the crystals. The separator 60 is thereby connected to the inlet to the extraction section 12 for scavenger depleted from phosphorous 16 for reusing the separated residual scavenger 17 for further adsorbing of phosphorous in the extraction section 12. The separator is also arranged for providing washing liquid depleted from residual scavenger 59 for reuse for washing crystals in the washer 52.

[0062] According to one embodiment of the present invention, the scavenger adhering to the separated ammonium phosphate crystals is removed by washing the ammonium phosphate crystals with a saturated aqueous ammonium phosphate solution. The scavenger initially adhering to the crystals forms a separate phase which typically is lighter than the dense aqueous phase and is as mentioned further above water immiscible. The two phases are thereby spontaneously separated from each other. The separator 60 of the present embodiment therefore is a phase separator arranged for separation of the scavenger and said saturated aqueous solution of ammonium phosphate. It was surprisingly found that the above mentioned wash procedure is highly efficient. The carbon content of the washed ammonium phosphate crystals was found to be lower than carbon contents of commercial high-purity ammonium phosphate salts. It is believed that the washing with saturated ammonium phosphate solution is a dynamic process in which ammonium phosphate crystals constantly dissolve and recrystallize enabling efficient removal of adhering solvent. The operation of the wash procedure is simple and is not energy intensive. The saturated ammonium phosphate solution which is separated from the crystals is continuously recycled for further washing. Make up of saturated ammonium phosphate solution is made by dissolving produced ammonium phosphate salts in aqueous solutions such as water, phosphoric acid, or other

acid/salt solutions. As mentioned also before, the separated water-immiscible scavenger is continuously recycled in order to extract phosphate from a feed solution.

[0063] The washed ammonium phosphate crystals are thereafter dried in the drier 54. The drying can preferably at least to a part be performed by heat obtained from the heat exchange process cooling the mixing of anhydrous ammonia with phosphoric acid. To that end the drier 54 is connected to the heat exchanger 30 as indicated by the broken arrows 37 and 39. The drier 54 is thereby arranged for utilizing at least a part of the heat extracted in the heat exchanger 30 for drying the washed crystals.

[0064] The produced ammonium phosphates are fully water-soluble, metal depleted and can be used for agricultural purposes such as fertilization or fertigation.

[0065] Another important advantage of the wash process according to a preferred embodiment of the present invention is that it enables to control the production of ammonium phosphates to produce either MAP or DAP independent of the initial composition of the precipitated crystals. If MAP is the desired end product, then the wash solution used is preferably composed of saturated aqueous solution of mono-ammonium phosphate. The pH of the slurry is controlled and adjusted to a value between 2 and 6, preferably between 3 and 5 and most preferably of about 4.1 by addition of e.g. phosphoric acid or ammonia. This procedure results in production of MAP independent of the initial composition of the precipitated crystals. In a similar way if DAP is the desired end product then the wash solution used is composed of saturated aqueous solution of di-ammonium phosphate. The pH of the slurry is controlled and adjusted to a value between 6 and 10, preferably between 7 and 9 and most preferably of about 8.3 by addition of e.g. ammonia. This procedure results in production of DAP independent of the initial composition of the precipitated crystals. In such a manner, production of both MAP and DAP is possible according to the invention. To this end, the washer 52 is further arranged for controlling a pH of the saturated aqueous solution of mono-ammonium phosphate and/or di-ammonium phosphate.

[0066] Fig. 3 illustrates a flow diagram of steps of a method according to an embodiment of the present invention. A method for production of ammonium phosphates begins in step 200. In step 210, a phosphorus-loaded water immiscible liquid phase is provided. Anhydrous ammonia is added to the water immiscible liquid phase in step 212. In one particular embodiment, the step 212 of adding comprises monitoring of a conductivity of the water immiscible liquid phase and controlling an amount of added anhydrous ammonia in response to the monitored conductivity. In another particular embodiment, the step 212 of adding comprises monitoring of a pH of the water immiscible liquid phase and controlling an amount of added anhydrous ammonia in response to the monitored pH. Mono-ammonium phosphate and/or di-ammonium phosphate is in step 214 precipitated from the water immiscible liquid phase. In step 216, a temperature of the water immiscible liquid phase during the steps of adding and precipitating is controlled to be situated within a predetermined temperature interval. As will be discussed more in detail further below, the actual step of controlling can be performed before, during and/or after the steps of adding and precipitating. The important feature is that it is ensured that the temperature during the adding and precipitating is kept within predetermined limits. It is of less importance when the actual instant of heat removal occurs. Step 216 may therefore be situated in time before, concurrent with and/or after the steps 212 and 214. The temperature controlling typically comprises extraction of heat from the water immiscible liquid phase. This heat may, at least to a part, be used for producing gaseous ammonia from liquid ammonia by means of heating. This gaseous ammonia can be used as the anhydrous ammonia added in step 212. In step 218 the precipitated mono-ammonium phosphate and/or di-ammonium phosphate is extracted from the water immiscible liquid phase.

[0067] In the embodiment illustrated in Fig. 3, the method further comprises a step 220, in which crystals of extracted precipitated mono-ammonium phosphate and/or di-ammonium phosphate is washed. In step 222, residual water immiscible liquid phase, i.e. typically scavenger (as discussed here below), washed from the crystals is separated. The separated residual scavenger is preferably reused for further adsorbing of phosphorous to obtain the phosphorus-loaded water immiscible liquid phase as indicated by the broken arrow 224. Similarly, washing liquid depleted from residual scavenger is reused for further washing of the crystals as indicated by the broken arrow 226. In this particular embodiment, the washing is performed with saturated aqueous solution of ammonium phosphate and the separating of residual scavenger is performed by phase separation of the scavenger and the saturated aqueous solution of ammonium phosphate. The washed crystals are dried in step 228. Preferably, the drying utilizes at least a part of the heat extracted from the step of controlling the temperature.

[0068] In a preferred embodiment, the pH of the saturated aqueous solution of mono-ammonium phosphate and/or diammonium phosphate is controlled to drive the chemical reactions to production of particular compositions of MAP and/or DAP. In particular, pure MAP can be obtained by acid pH and pure DAP can be obtained by slightly basic pH, as discussed above.

[0069] In the embodiment of Fig. 3, the step 210, providing a phosphorus-loaded water immiscible liquid phase in turn comprises adsorption of phosphorous from a phosphorous-containing aqueous solution into a liquid scavenger having affinity for phosphorous. This means that the scavenger loaded with phosphorous forms the phosphorus-loaded water immiscible liquid phase. The method according to the embodiment of Fig. 3 then also comprises the further step 230 of reusing regenerated scavenger formed by the step of extracting 218 for further adsorbing of phosphorous in step 210. The procedure ends in step 299.

[0070] As briefly mentioned above, the actual extraction of heat from said phosphorous-loaded water immiscible liquid phase can be performed in different ways. In the embodiment of Fig. 2, the heat exchanger 30 is integrated in the mixing volume 20. This is presently believed to be the preferred way, since it gives a well controlled temperature. However, alternatives are also possible. In Fig. 4, an embodiment is illustrated, where the heat exchanger 30 is arranged in contact with the water immiscible liquid phase leaving the precipitate remover 40. The controller 34 may still be controlled based on the temperature in the mixing volume 20 as measured by a thermometer 32. Alternatively, or in addition, a controller 34' can be operated based on the temperature of the scavenger entering the extraction section 12 by means of a thermometer 32'. In this way, the temperature of the scavenger entering the extraction section 12 is primarily controlled, which in turn will keep the temperature of the phosphorous-loaded water immiscible liquid phase within the mixing volume in the next cycle within the requested temperature interval, in particular if there is information about the assumed phosphorus content leaving the extraction section 12 with the phosphorous-loaded water immiscible liquid phase. In other world, by controlling the temperature of the scavenger entering the extraction section 12, an indirect control of the temperature in the mixing volume will also be achieved. This can be a good alternative in arrangements, where the initial phosphorous content is relatively stable or at least predictable. The scavenger entering the extraction section 12 may then be optimized in temperature regarding phosphorous affinity.

[0071] In Fig. 5, yet another embodiment is illustrated, where the heat exchanger 30 is arranged in contact with the water immiscible liquid phase leaving the extraction section 12 before entering the mixing volume 20. Here, the control can be based on either or both of a temperature in the mixing volume or a temperature of the loaded scavenger before entering the mixing volume 20. The temperature in the scavenger before entering the mixing volume is then measured by a thermometer 32" and using a controller 34". In this way, the temperature of the loaded scavenger is reduced, and the expected exothermic reactions in the mixing volume 20 will bring the water immiscible liquid phase to the predetermined temperature interval. This embodiment can be advantageous in applications where there are difficulties in combining the ammonia adding and precipitating with en efficient heat extraction.

[0072] The detailed embodiments above are only a few examples of how a method and an arrangement for production of ammonium phosphates may be arranged. The phosphorus-containing water immiscible liquid phase is preferably provided as described further above, but there are also other possibilities. The phosphorus-containing water immiscible liquid phase could be provided by any type of ion exchange process. The phosphorus-containing liquid phase could also be provided by other chemical processes, such as dissolution from solid phases. Likewise, the post-treatment of the precipitated MAP and/or DAP is also just one example, presently preferred, of how the MAP and/or DAP can be managed. Other more conventional techniques such as direct distilling of the precipitate in order to evaporate the scavenger or other solvent directly without any washing step. Furthermore, in certain applications, where the scavenger or other solvent is not very expensive and is harmless as impurity in the produced MAP/DAP, one may completely remove the washing procedure.

[0073] Tests have been performed on different systems in order to illustrate and verify the advantages obtained by methods and arrangements according to the principles described above. Some examples are presented here below.

EXAMPLE 1

[0074] An organic solvent composed of 80% tributyl phosphate and 20% heptanol by volume, having a pH value of 5.9, was loaded with 1.42 M H_3PO_4 by exposing the organic solvent to aqueous phosphoric acid. The two immiscible phases were first thoroughly mixed in order to facilitate the transfer of phosphoric acid and then separated. The loaded-organic solvent having a pH value of -0.4 was contacted with an excess of liquid anhydrous ammonia (> 50 g NH_3 $liter^{-1}$ solvent). Crystalline solids formed in the organic phase. The solids were separated from the organic solvent by centrifugation and decantation. The separated solids were washed several times with methanol and dried for 2 hours at 90°C. The recovered inorganic salt was composed of 12.3% N and 26.8% P corresponding to 98% $NH_4H_2PO_4$ and 2% $(NH_4)_2HPO_4$ by weight. The removal efficiency of phosphorus from the organic solvent was found to be as high as 99.4%.

EXAMPLE 2

[0075] The same experiment as described in example 1 was repeated with an organic solvent composed of 80% tributyl phosphate and 20% tri-octyl/decyl amine by volume. The recovered inorganic salt was composed of 13% N and 26.6% P corresponding to 90% $NH_4H_2PO_4$ and 10% $(NH_4)_2HPO_4$ by weight.

EXAMPLE 3

[0076] The same experiment as described in example 1 was repeated, the only difference being the use of a limited amount of liquid anhydrous ammonia (< 20g NH_3 $liter^{-1}$ solvent). The recovered inorganic salt was composed of 12.2% N and 26.9% P corresponding to 99% $NH_4H_2PO_4$ and 1% $(NH_4)_2HPO_4$ by weight.

EXAMPLE 4

[0077] The same experiment as described in example 3 was repeated, the only difference being the use of an organic solvent composed of 80% tributyl phosphate and 20% tri-octyl/decyl amine by volume. The recovered inorganic salt was composed of 12.4% N and 26.8% P corresponding to 97% $NH_4H_2PO_4$ and 3% $(NH_4)_2HPO_4$ by weight.

EXAMPLE 5

[0078] Measured amounts of liquid anhydrous ammonia were added to a solvent composed of 80% tributyl phosphate and 20% heptanol, loaded with 1.42 M H_3PO_4. The pH and conductivity of the solvent (22°C) as a function of amounts of added ammonia are shown in the following table 1:

Table 1. pH and conductivity of the solvent (22°C) as a function of amounts of added ammonia of example 5.

Added ammonia	pH	Conductivity
(g NH_3 liter^{-1} solvent)		**(mS/cm)**
0	-0.40	1.18
1	0.54	0.74
2.7	0.62	0.50
5.7	0.70	0.23
8.9	0.78	0.14
12	0.78	0.13
18.2	1.24	0.05
21	3.32	
23.3	4.9	0.02
25	7.6	0.01

EXAMPLE 6

[0079] The same experiment as described in example 5 was repeated, the only difference being the use of an organic solvent composed of 80% tributyl phosphate and 20% tri-octyl/decyl amine by volume. The pH and conductivity of the solvent (22°C) as a function of amounts of added ammonia are shown in the following table 2:

Table 2. pH and conductivity of the solvent (22°C) as a function of amounts of added ammonia of example 6.

Added ammonia (g NH_3 liter^{-1} solvent)	**pH**	**Conductivity (mS/cm)**
0	-0.09	3.73
1.0	0.12	2.98
3.0	0.55	2.76
4.3	0.79	2.47
6.0	0.89	1.53
7.3	1.27	1.47
9.0	2.29	1.24
11.0	2.67	1.11
13.0	2.78	
15.3	4.62	0.56
24.6	7.10	0.03

EXAMPLE 7

[0080] An excess of gaseous anhydrous ammonia was added to a solvent composed of 80% tributyl phosphate and 20% heptanol by volume, loaded with 1.42 M H_3PO_4. The temperature of the solvent increased from 22°C to 78°C.

EXAMPLE 8

[0081] The same experiment as described in example 7 was repeated, the only difference being the use of an organic solvent composed of 80% tributyl phosphate and 20% tri-octyl/decyl amine by volume. The temperature of the solvent increased from 23°C to 86°C.

EXAMPLE 9

[0082] Crystals of mono-ammonium phosphate were separated from a solvent composed of 80% tributyl phosphate and 20% heptanol by decantation. The separated crystals were fed into an aqueous solution saturated with mono-ammonium phosphate. The crystals were separated from the saturated aqueous solution by centrifugation and dried at 90°C. The organic solvent initially adhering to the crystals formed a separate phase above the aqueous phase. The carbon content of the washed mono-ammonium phosphate crystals was found to be lower than carbon contents of commercial high-purity mono ammonium phosphate salts. Similar results were obtained when using a solvent composed of 80% tributyl phosphate and 20% tri-octyl/decyl amine.

EXAMPLE 10

[0083] Crystals of mono-ammonium phosphate were fed into an aqueous solution saturated with di-ammonium phosphate. The pH of the aqueous solution was thereafter adjusted to a value of 8.3 by addition of gaseous anhydrous ammonia. The crystalline solids were thereafter separated from the saturated aqueous solution and dried. The solids were found to be composed of essentially di-ammonium phosphate. Thus, crystals of mono-ammonium phosphate could be converted into crystals of di-ammonium phosphate.

[0084] The embodiments described above are to be understood as a few illustrative examples of the present invention. It will be understood by those skilled in the art that various modifications, combinations and changes may be made to the embodiments in particular, different part solutions in the different embodiments can be combined in other configurations, where technically possible. The scope of the present invention is, however, defined by the appended claims.

Claims

1. A method for production of ammonium phosphates, comprising the steps of:

providing (210) a phosphorus-loaded water immiscible liquid phase (14);
adding (212) anhydrous ammonia (18) to said phosphorus-loaded water immiscible liquid phase (14);
precipitating (214) at least one of mono-ammonium phosphate and di-ammonium phosphate from said water immiscible liquid phase (14);
controlling (216) a temperature of the water immiscible liquid phase (15) during said steps of adding (212) and precipitating (214) to a predetermined temperature interval;
extracting (218) said precipitated at least one of mono-ammonium phosphate and di-ammonium phosphate from said water immiscible liquid phase (15);
washing (220) crystals of said extracted precipitated at least one of mono-ammonium phosphate and di-ammonium phosphate; and
drying (228) said washed crystals,

characterised in that
said step of washing (220) crystals comprises washing away residual water immiscible liquid phase (17) from said crystals of said extracted precipitated at least one of mono-ammonium phosphate and di-ammonium phosphate with a wash solution comprising saturated aqueous solution of ammonium phosphate;
and by the further steps of:

separating (222) said residual water immiscible liquid phase (17) washed from said crystals from said wash solution by phase separation of said residual water immiscible liquid phase washed from said crystals of said extracted precipitated at least one of mono-ammonium phosphate and di-ammonium phosphate and said wash solution;
reusing (224) said phase separated residual water immiscible liquid phase (17) for further adsorbing of phosphorous to be reused for further extraction; and
reusing (226) washing liquid depleted from residual water immiscible liquid phase (17) for further washing of

crystals in said step of washing crystals of said extracted precipitated at least one of mono-ammonium phosphate and di-ammonium phosphate.

2. The method according to claim 1, **characterised in that**
said wash solution used in said step of washing is composed of saturated aqueous solution of mono-ammonium phosphate or di-ammonium phosphate for production of mono-ammonium phosphate or di-ammonium phosphate, respectively.

3. The method according to claim 2, **characterised by** controlling a pH of said saturated aqueous solution of mono-ammonium phosphate to a pH of 2-6 or controlling a pH of said saturated aqueous solution of di-ammonium phosphate to a pH of 6-10 for driving chemical reactions to production of particular compositions of mono-ammonium phosphate or di-ammonium phosphate, whereby said controlling of a pH of said saturated aqueous solution of mono-ammonium phosphate comprises addition of phosphoric acid or ammonia and said controlling of a pH of said saturated aqueous solution of di-ammonium phosphate comprises addition of ammonia.

4. The method according to any of the claims 1 to 3, **characterised in that**
said step of providing (210) a phosphorus-loaded water immiscible liquid phase (14) comprises the step of adsorbing phosphorous from a phosphorous-containing aqueous solution into a liquid scavenger having affinity for phosphorous, whereby said scavenger loaded with phosphorous forms said phosphorus-loaded water immiscible liquid phase (14); and
by the further step of reusing regenerated said scavenger formed by said step of extracting (218) for further adsorbing of phosphorous.

5. The method according to any of the claims 1 to 4, **characterised in that** said step of adding (212) comprises monitoring of a conductivity or a pH of said water immiscible liquid phase (14) and controlling an amount of added anhydrous ammonia (18) in response to said monitored conductivity or pH.

6. The method according to any of the claims 1 to 5, **characterised in that** said step of controlling (216) a temperature comprises extraction of heat from said water immiscible liquid phase before, during and/or after said steps of adding (212) and precipitating (214).

7. An arrangement (10) for production of ammonium phosphates, comprising:

a mixing volume (20);
said mixing volume (20) having an inlet (22) for a phosphorus-loaded water immiscible liquid phase (14);
said mixing volume (20) having an inlet for adding anhydrous ammonia (18) into said water immiscible liquid phase (14);
a heat exchanger (30) arranged in thermal contact with said water immiscible liquid phase (15);
a controller (34) arranged for operating said heat exchanger (30) to keep said water immiscible liquid phase (14) in said mixing volume (20) within a predetermined temperature interval;
a precipitate remover (40) arranged for removing crystals of at least one of precipitated mono-ammonium phosphate and di-ammonium phosphate from said mixing volume (20)
a washer (52, 52') connected to said precipitate remover (40) and arranged for washing said crystals; and
a drier (54) connected to said washer (52, 52') and arranged for drying said washed crystals, **characterised by**
a separator (60) connected to said washer (52, 52') and arranged for separating residual water immiscible liquid phase (17) washed from said crystals;
said separator (60) being connected to said inlet to said extraction section (12) for water immiscible liquid phase (17) depleted from phosphorous for reusing said separated residual water immiscible liquid phase (17) for further adsorbing of phosphorous in said extraction section (12);
said separator (60) being further arranged for providing washing liquid (59) depleted from residual water immiscible liquid phase for reuse for washing crystals in said washer (52, 52');
said washer (52) is arranged for washing said crystals with saturated aqueous solution of ammonium phosphate; and
said separator (60) comprising a phase separator (58) arranged for separation of said water immiscible liquid phase (17) and said saturated aqueous solution of ammonium phosphate.

8. The arrangement according to claim 7, **characterised in that**
said washer (52) is arranged for using a wash solution composed of saturated aqueous solution of mono-ammonium

phosphate or di-ammonium phosphate for production of mono-ammonium phosphate or di-ammonium phosphate, respectively.

9. The arrangement according to claim 8, **characterised in that** said washer (52) is arranged for controlling a pH of said saturated aqueous solution of mono-ammonium phosphate and/or di-ammonium phosphate.

10. The arrangement according to claim 9, **characterised In that** said washer (52) is arranged for addition of phosphoric acid or ammonia to said saturated aqueous solution of mono-ammonium phosphate or of ammonia to said saturated aqueous solution of di-ammonium phosphate.

11. The arrangement according to any of the claims 7 to 10, **characterised by**
an extraction section (12), arranged for adsorbing phosphorous from a phosphorous-containing aqueous solution into a liquid scavenger having affinity for phosphorous;
an outlet from said extraction section (12) for scavenger loaded with phosphorous is connected to said inlet (22) for a phosphorus-loaded water immiscible liquid phase of said mixing volume, whereby said scavenger loaded with phosphorous forms said phosphorus-loaded water immiscible liquid phase (14);
an inlet to said extraction section (12) for scavenger depleted from phosphorous (16) is connected to said mixing volume (20) and arranged for reusing regenerated said scavenger formed in said mixing volume (20) by operation of said precipitation remover (40) for further adsorbing of phosphorous in said extraction section (12).

12. The arrangement according to any of the claims 7 to 11, **characterised by:**

one of:

a sensor (26) for monitoring of a conductivity of said water immiscible liquid phase (14); and
a sensor (26) for monitoring of a pH of said water immiscible liquid phase (14); and

an adder control unit (28) connected to said sensor (26) and arranged for controlling an amount of added anhydrous ammonia (18) in response to said monitored conductivity or pH.

13. The arrangement according to any of the claims 7 to 12, **characterised in that** said heat exchanger (30) is operating for extraction of heat from said phosphorous-loaded water immiscible liquid phase (14) entering said mixing volume (20), from said water immiscible liquid phase within said mixing volume (20) and/or from phosphorous-depleted water immiscible liquid phase (17) exiting said mixing volume (20).

14. The arrangement according to claim 13, **characterised by**
a source of liquid ammonia (19);
a heater unit (23) connected to said source of liquid ammonia (19) and connected to or integrated with said heat exchanger (30), said heater unit (23) is arranged for utilizing at least a part of said heat extracted in said heat exchanger (30) to produce gaseous ammonia;
said inlet (24) for adding anhydrous ammonia (18) of said mixing volume being connected for extracting said gaseous ammonia from said source of liquid ammonia (19).

Patentansprüche

1. Verfahren zur Herstellung von Ammoniumphosphaten, das folgende Schritte umfasst:

Bereitstellen (210) einer Phosphor-beladenen, mit Wasser nicht mischbaren, flüssigen Phase (14);
Zugabe (212) von wasserfreiem Ammoniak (18) zu der Phosphor-beladenen, mit Wasser nicht mischbaren, flüssigen Phase (14);
Präzipitieren (214) von mindestens einem aus Ammoniumdihydrogenphosphat und Diammoniumhydrogenphosphat aus der mit Wasser nicht mischbaren, flüssigen Phase (14);
Regeln (216) einer Temperatur der mit Wasser nicht mischbaren, flüssigen Phase (15) während der Schritte der Zugabe (212) und des Präzipitierens (214) auf ein festgelegtes Temperaturintervall;
Extrahieren (218) des präzipitierten, mindestens einen aus Ammoniumdihydrogenphosphat und Diammoniumhydrogenphosphat aus der mit Wasser nicht mischbaren, flüssigen Phase (15);

Waschen (220) von Kristallen des extrahierten, präzipitierten mindestens einen aus Ammoniumdihydrogenphosphat und Diammoniumhydrogenphosphat; und
Trocknen (228) der gewaschenen Kristalle,
dadurch gekennzeichnet, dass
der Schritt des Waschens (220) von Kristallen das Abwaschen jeglicher restlicher, mit Wasser nicht mischbarer, flüssiger Phase (17) von den Kristallen des extrahierten, präzipitierten mindestens einen aus Ammoniumdihydrogenphosphat und Diammoniumhydrogenphosphat mit einer Waschlösung, umfassend gesättigte, wässrige Lösung von Ammoniumphosphat, umfasst;
und durch folgende weitere Schritte:

Trennen (222) der restlichen, mit Wasser nicht mischbaren, flüssigen Phase (17), die von den Kristallen gewaschen wurde, von der Waschlösung durch Phasentrennung zwischen der restlichen, mit Wasser nicht mischbaren, flüssigen Phase, die von den Kristallen des extrahierten, präzipitierten mindestens einen aus Ammoniumdihydrogenphosphat und Diammoniumhydrogenphosphat gewaschen wurde, und der Waschlösung;
Wiederverwenden (224) der phasengetrennten restlichen, mit Wasser nicht mischbaren, flüssigen Phase (17) zur weiteren Adsorption von Phosphor zur Wiederverwendung für weitere Extraktion; und
Wiederverwenden (226) von Waschflüssigkeit, abgereichert an restlichen, mit Wasser nicht mischbaren, flüssigen Phase (17) zum weiteren Waschen von Kristallen in dem Schritt des Waschens von Kristallen des extrahierten, präzipitierten mindestens einen aus Ammoniumdihydrogenphosphat und Diammoniumhydrogenphosphat.

2. Verfahren nach Anspruch 1, **dadurch gekennzeichnet, dass** die in dem Waschschritt verwendete Waschlösung aus gesättigter, wässriger Lösung von Ammoniumdihydrogenphosphat oder Diammoniumhydrogenphosphat zur Herstellung von Ammoniumdihydrogenphosphat bzw. Diammoniumhydrogenphosphat zusammengesetzt ist.

3. Verfahren nach Anspruch 2, **gekennzeichnet durch** Regeln eines pH-Werts der gesättigten, wässrigen Lösung von Ammoniumdihydrogenphosphat auf einen pH-Wert von 2 bis 6 oder **durch** Regeln eines pH-Werts der gesättigten, wässrigen Lösung von Diammoniumhydrogenphosphat auf einen pH-Wert von 6 bis 10 zum Steuern chemischer Reaktionen zur Herstellung bestimmter Zusammensetzungen von Ammoniumdihydrogenphosphat oder Diammoniumhydrogenphosphat, wobei das Regeln eines pH-Werts der gesättigten, wässrigen Lösung von Ammoniumdihydrogenphosphat die Zugabe von Phosphorsäure oder Ammoniak umfasst und das Regeln eines pH-Werts der gesättigten, wässrigen Lösung von Diammoniumhydrogenphosphat die Zugabe von Ammoniak umfasst.

4. Verfahren nach einem der Ansprüche 1 bis 3, **dadurch gekennzeichnet, dass**
der Schritt des Bereitstellens (210) einer Phosphor-beladenen, mit Wasser nicht mischbaren, flüssigen Phase (14) den Schritt des Adsorbierens von Phosphor aus einer phosphorhaltigen, wässrigen Lösung in einem flüssigen Abfangreagenz umfasst, das eine Affinität für Phosphor aufweist, wobei das mit Phosphor beladene Abfangreagenz die Phosphor-beladene, mit Wasser nicht mischbare, flüssige Phase (14) bildet; und durch den weiteren Schritt der Wiederverwendung des regenerierten Abfangreagenzes, gebildet durch den Schritt des Extrahierens (218), zur weiteren Adsorption von Phosphor.

5. Verfahren nach einem der Ansprüche 1 bis 4, **dadurch gekennzeichnet, dass** der Schritt der Zugabe (212) das Überwachen einer Leitfähigkeit oder eines pH-Werts der mit Wasser nicht mischbaren, flüssigen Phase (14) und das Regeln einer Menge des zugegebenen wasserfreien Ammoniaks (18) in Reaktion auf die überwachte Leitfähigkeit oder den überwachten pH-Wert umfasst.

6. Verfahren nach einem der Ansprüche 1 bis 5, **dadurch gekennzeichnet, dass** der Schritt des Regelns (216) einer Temperatur eine Wärmeentnahme von der mit Wasser nicht mischbaren, flüssigen Phase vor, während und/oder nach den Schritten der Zugabe (212) und des Präzipitierens (214) umfasst.

7. Anordnung (10) zur Herstellung von Ammoniumphosphaten, umfassend:

einen Mischraum (20);
wobei der Mischraum (20) einen Einlass (22) für eine Phosphat-beladene, mit Wasser nicht mischbare, flüssige Phase (14) aufweist;
wobei der Mischraum (20) einen Einlass für die Zugabe von wasserfreiem Ammoniak (18) in die mit Wasser nicht mischbare, flüssige Phase (14) aufweist;

einen Wärmetauscher (30), der in thermischem Kontakt mit der mit Wasser nicht mischbaren, flüssigen Phase (15) angeordnet ist;
einen Controller (34), der zum Betrieb des Wärmetauschers (30) angeordnet ist,
um die mit Wasser nicht mischbare, flüssige Phase (14) in dem Mischraum (20) in einem festgelegten Temperaturintervall zu halten;
einen Präzipitat-Entferner (40), der zum Entfernen von Kristallen von mindestens einem aus präzipitiertem Ammoniumdihydrogenphosphat und Diammoniumhydrogenphosphat aus dem Mischraum (20) angeordnet ist;
eine Waschvorrichtung (52, 52'), die mit dem Präzipitat-Entferner (40) verbunden und zum Waschen der Kristalle angeordnet ist; und
einen Trockner (54), der mit der Waschvorrichtung (52, 52') verbunden und zum Trocknen der gewaschenen Kristalle angeordnet ist,
gekennzeichnet durch
eine Trennvorrichtung (60), die mit der Waschvorrichtung (52, 52') verbunden und zum Abtrennen der restlichen, mit Wasser nicht mischbaren, flüssigen Phase (17) angeordnet ist, die von den Kristallen gewaschen wurde;
wobei die Trennvorrichtung (60) mit dem Einlass zu dem Extraktionsbereich (12) für die mit Wasser nicht mischbare, flüssige Phase (17), die an Phosphor abgereichert ist, verbunden ist, um die abgetrennte, restliche, mit Wasser nicht mischbare, flüssige Phase (17) zur weiteren Adsorption von Phosphor in dem Extraktionsbereich (12) wiederzuverwenden;
wobei die Trennvorrichtung (60) weiter zur Bereitstellung von Waschflüssigkeit (59), die an restlicher, mit Wasser nicht mischbarer, flüssiger Phase abgereichert ist, zur Wiederverwendung zum Waschen von Kristallen in der Waschvorrichtung (52, 52') angeordnet ist;
wobei die Waschvorrichtung (52) zum Waschen der Kristalle mit gesättigter, wässriger Lösung von Ammoniumphosphat angeordnet ist; und
wobei die Trennvorrichtung (60) einen Phasentrenner (58) umfasst, der zum Trennen der mit Wasser nicht mischbaren, flüssigen Phase (17) und der gesättigten, wässrigen Lösung von Ammoniumphosphat angeordnet ist.

8. Anordnung gemäß Anspruch 7, **dadurch gekennzeichnet, dass** die Waschvorrichtung (52) zur Verwendung einer Waschlösung angeordnet ist, die aus gesättigter, wässriger Lösung von Ammoniumdihydrogenphosphat oder Diammoniumhydrogenphosphat zur Herstellung von Ammoniumdihydrogenphosphat bzw. Diammoniumhydrogenphosphat zusammengesetzt ist.

9. Anordnung nach Anspruch 8, **dadurch gekennzeichnet, dass** die Waschvorrichtung (52) zur Regelung eines pH-Werts der gesättigten, wässrigen Lösung von Ammoniumdihydrogenphosphat und/oder Diammoniumhydrogenphosphat angeordnet ist.

10. Anordnung nach Anspruch 9, **dadurch gekennzeichnet, dass** die Waschvorrichtung (52) zur Zugabe von Phosphorsäure oder Ammoniak zu der gesättigten, wässrigen Lösung von Ammoniumdihydrogenphosphat oder von Ammoniak zu der gesättigten, wässrigen Lösung von Diammoniumhydrogenphosphat angeordnet ist.

11. Anordnung nach einem der Ansprüche 7 bis 10, **gekennzeichnet durch** einen Extraktionsbereich (12), der zur Adsorption von Phosphor aus einer phosphorhaltigen, wässrigen Lösung in ein flüssiges Abfangreagenz mit Affinität für Phosphor angeordnet ist;
einen Auslass von dem Extraktionsbereich (12) für Abfangreagenz, beladen mit Phosphor, der mit dem Einlass (22) für eine Phosphor-beladene, mit Wasser nicht mischbare, flüssige Phase des Mischraums verbunden ist, wobei das mit Phosphor beladene Abfangreagenz die Phosphor-beladene, mit Wasser nicht mischbare, flüssige Phase (14) bildet;
einen Einlass zu dem Extraktionsbereich (12) für Abfangreagenz, abgereichert an Phosphor (16), der mit dem Mischraum (20) verbunden und zur Wiederverwendung des regenerierten Abfangreagenzes, gebildet in dem Mischraum (20) **durch** Betrieb des Präzipitat-Entferners (40), zur weiteren Adsorption von Phosphor in dem Extraktionsbereich (12) angeordnet ist.

12. Anordnung nach einem der Ansprüche 7 bis 11, **gekennzeichnet durch:**

einen aus:

einem Sensor (26) zur Überwachung einer Leitfähigkeit der mit Wasser nicht mischbaren, flüssigen Phase (14); und

einem Sensor (26) zur Überwachung eines pH-Werts der mit Wasser nicht mischbaren, flüssigen Phase (14); und
einer Zugabe-Regeleinheit (28), die mit dem Sensor (26) verbunden und zum Regeln einer Menge zugegebenen wasserfreien Ammoniaks (18) in Reaktion auf die überwachte Leitfähigkeit oder den überwachten pH-Wert angeordnet ist.

13. Anordnung nach einem der Ansprüche 7 bis 12, **dadurch gekennzeichnet, dass** der Wärmetauscher (30) zur Entnahme von Wärme aus der Phosphor-beladenen, mit Wasser nicht mischbaren, flüssigen Phase (14), die in den Mischraum (20) eintritt, aus der mit Wasser nicht mischbaren, flüssigen Phase in dem Mischraum (20) und/oder aus der an Phosphor abgereicherten, mit Wasser nicht mischbaren, flüssigen Phase (17), die den Mischraum (20) verlässt, arbeitet.

14. Anordnung nach Anspruch 13, **gekennzeichnet durch** eine Quelle für flüssigen Ammoniak (19);
eine Heizeinheit (23), die mit der Quelle für flüssigen Ammoniak (19) verbunden ist und mit dem Wärmetauscher (30) verbunden oder darin angeordnet ist, wobei die Heizeinheit (23) zur Nutzung mindestens eines Teils der in dem Wärmetauscher (30) entnommenen Wärme angeordnet ist, um gasförmigen Ammoniak zu erzeugen;
den Einlass (24) zur Zugabe von wasserfreiem Ammoniak (18) des Mischraums, der zur Entnahme des gasförmigen Ammoniaks aus der Quelle für flüssigen Ammoniak (19) verbunden ist.

Revendications

1. Procédé de production de phosphates d'ammonium, comprenant les étapes :

d'obtention (210) d'une phase liquide non miscible avec l'eau (14) chargée de phosphore ;
d'ajout (212) d'ammoniac anhydre (18) à ladite phase liquide non miscible avec l'eau (14) chargée de phosphore ;
de précipitation (214) d'au moins un phosphate parmi le phosphate de mono-ammonium et le phosphate de di-ammonium à partir de ladite phase liquide non miscible avec l'eau (14) ;
de régulation (216) à une température de la phase liquide non miscible avec l'eau (15) durant les étapes d'ajout (212) et de précipitation (214) sur un intervalle de température prédéfini ;
d'extraction (218) dudit précipité comprenant au moins un phosphate parmi le phosphate de mono-ammonium et le phosphate de di-ammonium de ladite phase liquide non miscible avec l'eau (15) ;
de lavage (220) des cristaux dudit produit extrait précipité constitué d'au moins un phosphate parmi le phosphate de mono-ammonium et le phosphate de di-ammonium ; et
de séchage (228) desdits cristaux,

caractérisé en ce que
ladite étape de lavage (220) des cristaux comprend l'élimination par lavage de la phase liquide non miscible avec l'eau (17) résiduelle desdits cristaux dudit produit extrait précipité constitué d'au moins un phosphate parmi le phosphate de mono-ammonium et le phosphate de di-ammonium avec une solution de lavage comprenant une solution aqueuse saturée de phosphate d'ammonium ;
et **par** les étapes ultérieures :

de séparation (222) de ladite phase liquide non miscible avec l'eau (17) résiduelle, obtenue par lavage desdits cristaux, et de ladite solution de lavage par une séparation de phases de ladite phase liquide non miscible avec l'eau résiduelle, obtenue par lavage desdits cristaux d'au moins un phosphate parmi le phosphate de mono-ammonium et le phosphate de di-ammonium dudit produit précipité extrait et ladite solution de lavage ;
de réutilisation (224) de ladite phase séparée de la phase liquide non miscible à l'eau (17) résiduelle pour une nouvelle adsorption de phosphore afin d'être réutilisée pour une nouvelle extraction ; et
de réutilisation (226) du liquide de lavage épuisé en la phase liquide non miscible à l'eau (17) résiduelle pour un autre lavage des cristaux dans ladite étape de lavage des cristaux dudit produit extrait précipité constitué d'au moins un phosphate parmi le phosphate de mono-ammonium et le phosphate de di-ammonium.

2. Procédé selon la revendication 1, **caractérisé en ce que**
ladite solution de lavage utilisée dans ladite étape de lavage est composée d'une solution aqueuse saturée de phosphate de mono-ammonium ou de phosphate de di-ammonium pour la production de phosphate de mono-ammonium ou de phosphate de di-ammonium, respectivement.

3. Procédé selon la revendication 2, **caractérisé par** une régulation à une valeur de pH de ladite solution aqueuse saturée de phosphate de mono-ammonium à un pH de 2 à 6 ou une régulation à une valeur de pH de ladite solution aqueuse saturée de phosphate de di-ammonium à un pH de 6 à 10 afin d'effectuer des réactions chimiques pour la production de compositions particulières de phosphate de mono-ammonium ou de phosphate de di-ammonium, où la régulation à une valeur de pH de ladite solution aqueuse saturée de phosphate de mono-ammonium comprend une addition d'acide phosphorique ou d'ammoniac et ladite régulation à une valeur de pH de ladite solution aqueuse saturée de phosphate de di-ammonium comprend l'addition d'ammoniac.

4. Procédé selon l'une quelconque des revendications 1 à 3, **caractérisé en ce que**
ladite étape d'obtention (210) d'une phase liquide non miscible à l'eau (14) chargée de phosphore comprend l'étape d'adsorption de phosphore à partir d'une solution aqueuse contenant du phosphore sur un fixateur liquide possédant une affinité pour le phosphore, où ledit fixateur chargé de phosphore forme ladite phase liquide non miscible à l'eau (14) chargée de phosphore ; et
par l'étape ultérieure de réutilisation dudit fixateur régénéré formé par ladite étape d'extraction (218) pour une nouvelle adsorption de phosphore.

5. Procédé selon l'une quelconque des revendications 1 à 4, **caractérisé en ce que** ladite étape d'ajout (212) comprend la surveillance d'une conductivité ou d'un pH de ladite phase liquide non miscible à l'eau (14) et la régulation d'une quantité d'ammoniac anhydre (18) ajoutée en réponse à ladite conductivité ou audit pH surveillés.

6. Procédé selon l'une quelconque des revendications 1 à 5, **caractérisé en ce que** ladite étape de régulation (216) à une température comprend l'extraction de chaleur de la phase liquide non miscible avec l'eau avant, pendant et/ou après lesdites étapes d'ajout (212) et de précipitation (214).

7. Installation (10) pour la production de phosphates d'ammonium, comprenant :

un espace de mélange (20) ;
ledit espace de mélange (20) possédant une entrée (22) pour une phase liquide non miscible à l'eau (14) chargée de phosphore ;
ledit espace de mélange (20) possédant une entrée pour ajouter de l'ammoniac anhydre (18) dans ladite phase liquide non miscible à l'eau (14) ;
un échangeur de chaleur (30) mis en contact thermique avec ladite phase liquide non miscible à l'eau (15) ;
un régulateur (34) conçu pour mettre en oeuvre ledit échangeur de chaleur (30) afin de conserver ladite phase liquide non miscible à l'eau (14) dans ledit espace de mélange (20) dans un intervalle de température prédéfini ;
un système pour prélever le précipité (40) conçu pour enlever les cristaux précipités d'au moins l'un, parmi le phosphate de mono-ammonium et le phosphate de di-ammonium, de l'espace de mélange (20)
un système de lavage (52, 52') en communication avec ledit système pour prélever le précipité (40) et conçu pour le lavage desdits cristaux ; et
un sécheur (54) en communication avec ledit système de lavage (52, 52') et conçu pour le séchage desdits cristaux lavés,

caractérisée par

un séparateur (60) en communication avec ledit système de lavage (52, 52') et conçu pour la séparation de ladite phase liquide non miscible avec l'eau (17) résiduelle provenant du lavage desdits cristaux ;
ledit séparateur (60) étant en communication avec ladite entrée vers ladite section d'extraction (12) pour la phase liquide non miscible à l'eau (17) épuisée en phosphore en vue d'une réutilisation de la phase liquide non miscible à l'eau (17) résiduelle pour une nouvelle adsorption de phosphore dans ladite section d'extraction (12) ;
ledit séparateur (60) étant en outre conçu pour l'obtention de liquide de lavage (59) épuisé en phase liquide non miscible avec l'eau résiduelle pour une réutilisation dans le but de laver les cristaux dans ledit système de lavage (52, 52') ;
ledit système de lavage (52) est conçu pour le lavage desdits cristaux avec une solution aqueuse saturée de phosphate d'ammonium ; et
ledit séparateur (60) comprenant un séparateur de phases (58) conçu pour la séparation de ladite phase liquide non miscible avec l'eau (17) et de la solution aqueuse saturée de phosphate d'ammonium.

8. Installation selon la revendication 7, **caractérisée en ce que**
ledit système de lavage (52) est conçu pour l'utilisation d'une solution de lavage composée d'une solution aqueuse

saturée de phosphate de mono-ammonium ou de phosphate de di-ammonium, pour la production de phosphate de mono-ammonium, respectivement, de phosphate de di-ammonium.

9. Installation selon la revendication 8, **caractérisée en ce que**
ledit système de lavage (52) est conçu pour la régulation à une valeur de pH de ladite solution aqueuse saturée de phosphate de mono-ammonium et/ou de phosphate de di-ammonium.

10. Installation selon la revendication 9, **caractérisée en ce que**
ledit système de lavage (52) est conçu pour l'addition d'acide phosphorique ou d'ammoniac à ladite solution aqueuse saturée de phosphate de mono-ammonium ou d'ammoniac à ladite solution aqueuse saturée de phosphate de di-ammonium.

11. Installation selon l'une quelconque des revendications 7 à 10, **caractérisée par**
une section d'extraction (12), conçue pour l'adsorption du phosphore à partir d'une solution aqueuse contenant du phosphore sur un fixateur liquide ayant une affinité pour le phosphore ;
une sortie de ladite section d'extraction (12) pour le fixateur chargé de phosphore est en communication avec ladite entrée (22) pour une phase liquide non miscible à l'eau chargée de phosphore dudit espace de mélange, ce par quoi ledit fixateur chargé de phosphore forme ladite phase liquide non miscible à l'eau (14) chargée de phosphore ;
une entrée vers ladite section d'extraction (12) pour le fixateur épuisé en phosphore (16) est en communication avec ledit espace de mélange (20) et conçue pour la réutilisation dudit fixateur régénéré formé dans ledit espace de mélange (20) en mettant en oeuvre ledit système de prélèvement du précipité (40) en vue d'une nouvelle adsorption de phosphore dans ladite section d'extraction (12).

12. Installation selon l'une quelconque des revendications 7 à 11, **caractérisée par :**

un des éléments parmi :

un capteur (26) chargé de la surveillance de la conductivité de ladite phase liquide non miscible à l'eau (14) ; et
un capteur (26) chargé de la surveillance du pH de ladite phase liquide non miscible à l'eau (14) ; et

une unité de contrôle additionneuse (28) connectée audit capteur (26) et conçue pour le contrôle d'une quantité d'ammoniac anhydre (18) ajoutée en réponse à ladite conductivité ou audit pH surveillé.

13. Installation selon l'une quelconque des revendications 7 à 12, **caractérisée en ce que** ledit échangeur de chaleur (30) est mis en oeuvre pour l'extraction de chaleur à partir de la phase liquide non miscible à l'eau (14) chargée de phosphore entrant dans ledit espace de mélange (20), à partir de ladite phase liquide non miscible à l'eau à l'intérieur dudit espace de mélange (20) et/ou à partir de la phase liquide non miscible à l'eau (17) épuisée en phosphore quittant ledit espace de mélange (20).

14. Installation selon la revendication 13, **caractérisée par**
une source d'ammoniac liquide (19) ;
une unité de chauffage (23) en communication avec ladite source d'ammoniac liquide (19) et en communication ou intégrée audit échangeur de chaleur (30), ladite unité de chauffage (23) étant conçue pour l'utilisation d'au moins une partie de ladite chaleur extraite **dans** ledit échangeur de chaleur (30) afin de produire de l'ammoniac gazeux ;
ladite entrée (24) pour l'ajout d'ammoniac anhydre (18) dudit espace de mélange étant en communication pour réaliser l'extraction dudit ammoniac gazeux à partir de ladite source d'ammoniac liquide (19).

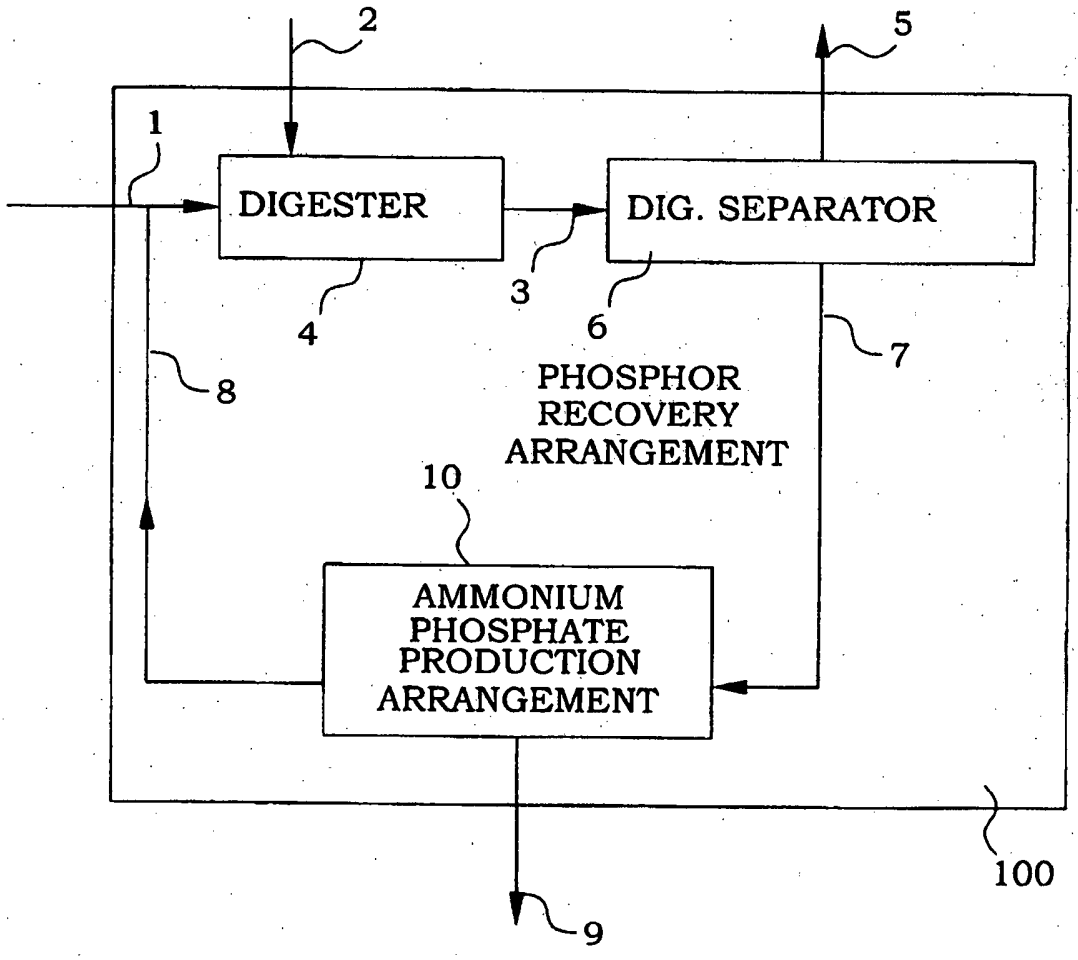
2
5
1
DIGESTER
DIG. SEPARATOR
3
4
6
7
8
PHOSPHOR
RECOVERY
ARRANGEMENT
10
AMMONIUM
PHOSPHATE
PRODUCTION
ARRANGEMENT
100
9

Fig. 1

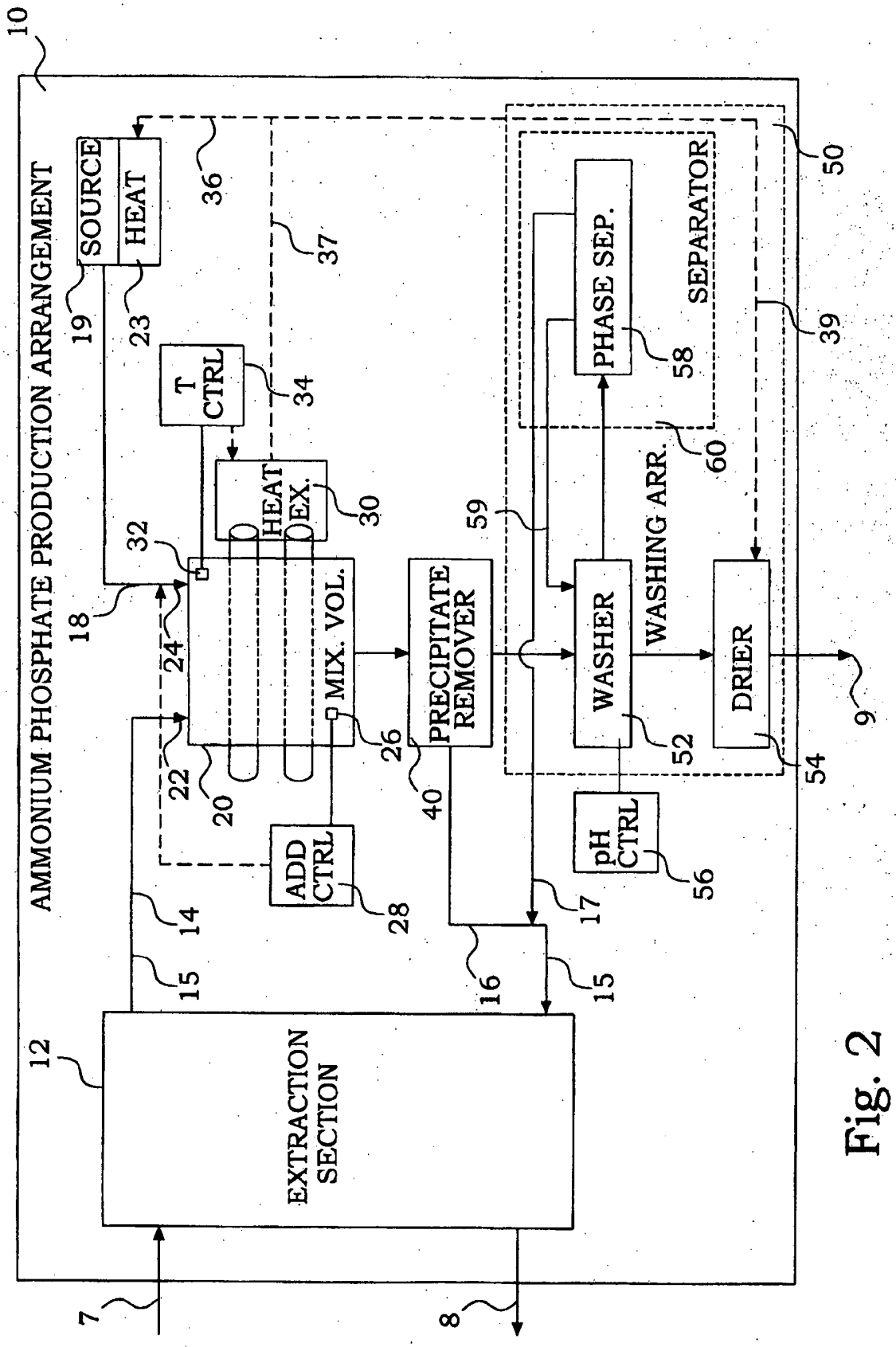
10
AMMONIUM PHOSPHATE PRODUCTION ARRANGEMENT
SOURCE
HEAT
19
23
36
37
50
T CTRL
34
HEAT EX.
30
32
18
24
22
20
MIX. VOL.
26
PRECIPITATE REMOVER
40
59
PHASE SEP.
58
SEPARATOR
39
60
WASHER
WASHING ARR.
52
DRIER
54
9
pH CTRL
56
ADD CTRL
28
14
15
16
17
12
EXTRACTION SECTION
7
8

Fig. 2

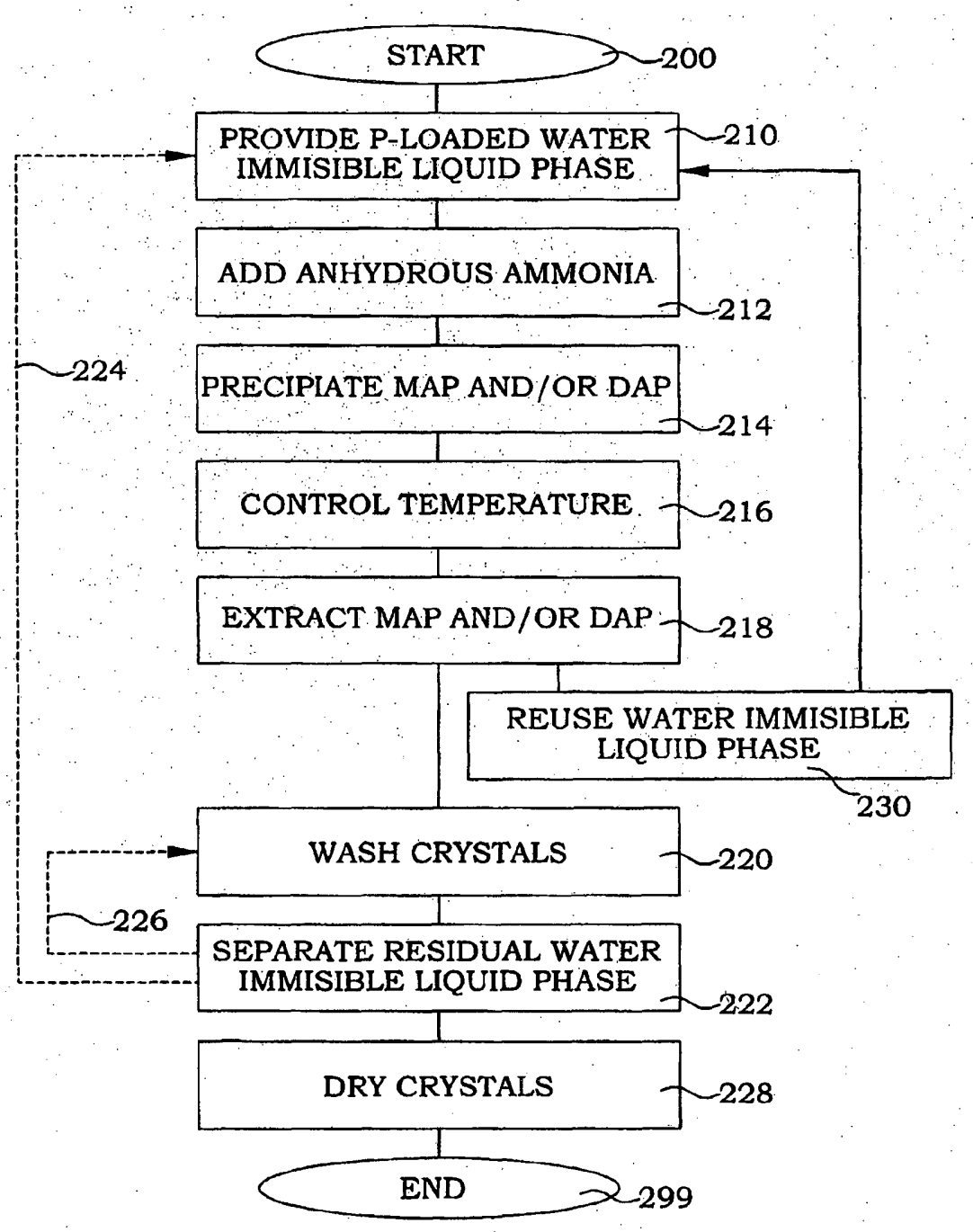
START
200
PROVIDE P-LOADED WATER IMMISIBLE LIQUID PHASE
210
ADD ANHYDROUS AMMONIA
212
224
PRECIPIATE MAP AND/OR DAP
214
CONTROL TEMPERATURE
216
EXTRACT MAP AND/OR DAP
218
REUSE WATER IMMISIBLE LIQUID PHASE
230
WASH CRYSTALS
220
226
SEPARATE RESIDUAL WATER IMMISIBLE LIQUID PHASE
222
DRY CRYSTALS
228
END
299

Fig. 3

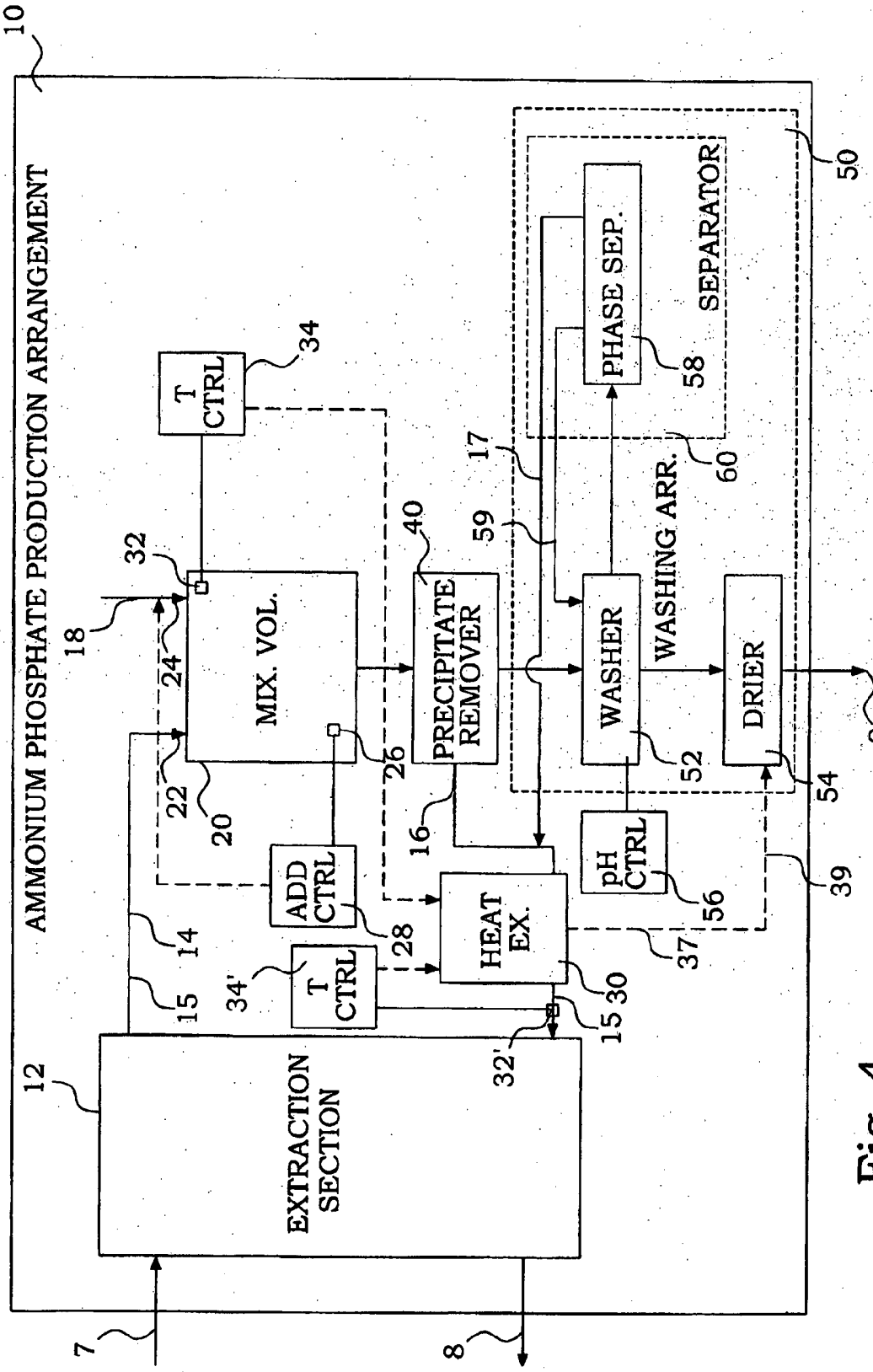
10
AMMONIUM PHOSPHATE PRODUCTION ARRANGEMENT
EXTRACTION SECTION
12
7
8
MIX. VOL.
18
24
22
20
32
T CTRL
34
ADD CTRL
28
T CTRL
34'
14
15
PRECIPITATE REMOVER
40
26
16
HEAT EX.
30
15
32'
37
pH CTRL
56
WASHER
52
WASHING ARR.
59
17
PHASE SEP.
58
SEPARATOR
60
50
DRIER
54
39
9

Fig. 4

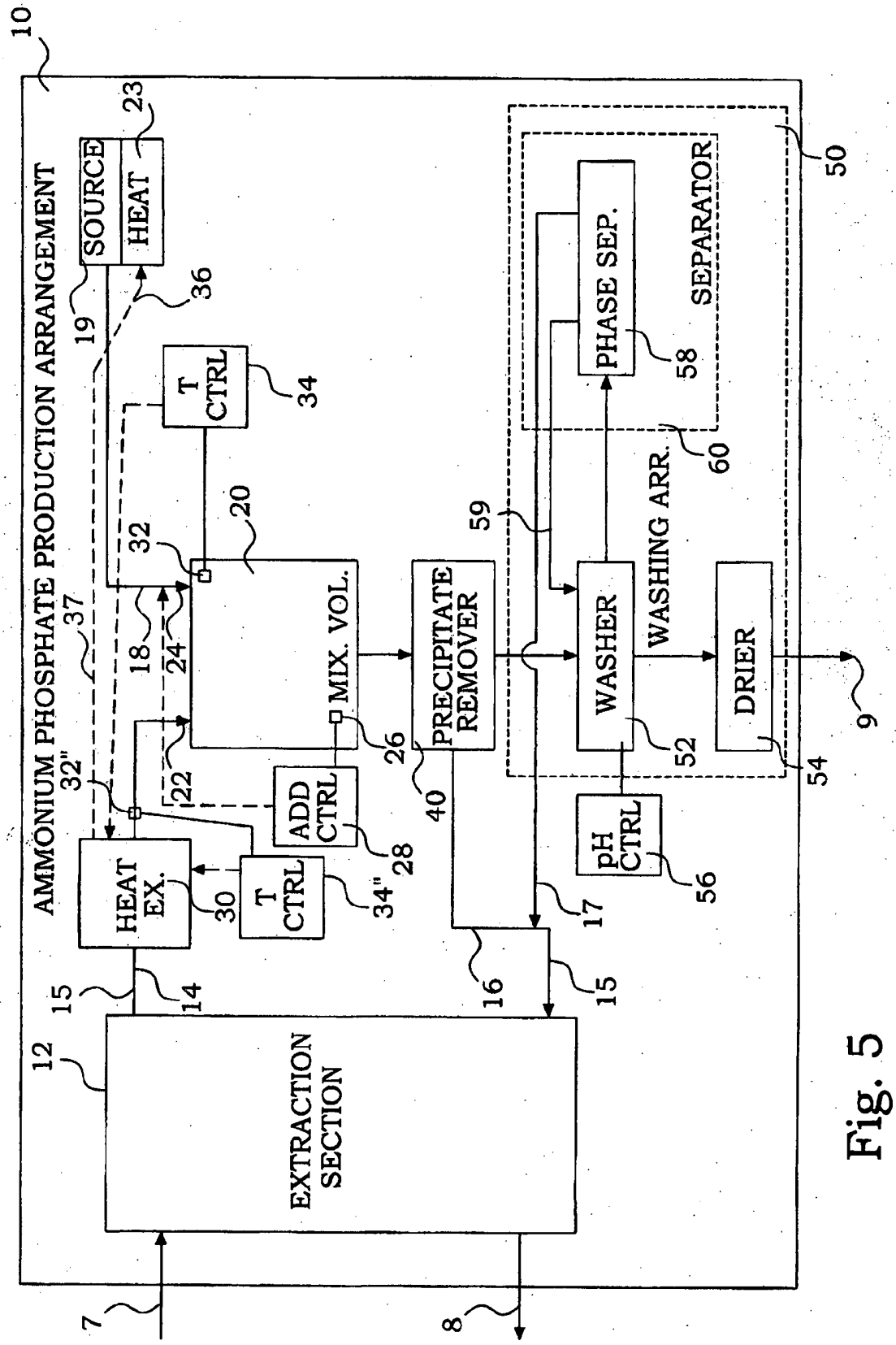
10
AMMONIUM PHOSPHATE PRODUCTION ARRANGEMENT
SOURCE
HEAT
23
19
36
T CTRL
34
20
32
37
18
24
MIX. VOL.
PRECIPITATE REMOVER
59
PHASE SEP.
SEPARATOR
50
58
60
WASHING ARR.
WASHER
DRIER
9
26
40
52
54
32"
22
ADD CTRL
28
HEAT EX.
30
T CTRL
34"
pH CTRL
56
17
16
15
15
14
12
EXTRACTION SECTION
7
8

Fig. 5

REFERENCES CITED IN THE DESCRIPTION

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Igénypontok

1. Ammónium-foszfátok előállítására szolgáló eljárás, amely a következő lépéseket foglalja magában:

foszforral terhelt, vízzel nem elegyedő folyékony fázis (14) előállítása (210);

vízmentes ammónia (18) hozzáadása (212) az említett foszforral terhelt vízzel nem elegyedő folyékony fázishoz (14);

a mono-ammónium-foszfát és a di-ammónium-foszfát közül legalább az egyik lecsapatása (214) az említett vízzel nem elegyedő folyékony fázisból (14);

a vízzel nem elegyedő folyékony fázis hőmérsékletének (15) szabályozása (216) egy előre meghatározott hőmérséklet-intervallumban az említett hozzáadás (212) és lecsapatás (214) lépései alatt;

az említett, lecsapatott mono-ammónium-foszfát és a di-ammónium-foszfát közül legalább az egyik extrahálása (218) az említett vízzel nem elegyedő folyékony fázisból (15);

az említett, extrahált és lecsapatott mono-ammónium-foszfát és di-ammónium- foszfát közül legalább az egyik kristályainak mosása (220); és

az említett mosott kristályok szárítása (228),

azzal jellemezve, hogy

az említett kristálymosási (220) lépés magában foglalja a maradék vízzel nem elegyedő folyékony fázis (17) lemosását az említett kristályokról, melyeket a mono-ammónium-foszfát és a di-ammónium-foszfát közül legalább az egyik lecsapatása és extrahálása után állítottak elő, ahol a mosóoldat ammónium-foszfát telített vizes oldatát tartalmazza, továbbá az alábbi lépéseket foglalja magában:

az említett maradék, vízzel nem elegyedő folyékony fázis (17) elválasztása (222) amely folyékony fázist az említett kristályokról az említett mosóoldattal mostak le, azon maradék, vízzel nem elegyedő folyékony fázisnak a nevezett mosóoldatról történő elválasztásával, amelyet a mono-ammónium-foszfát és a di-ammónium-foszfát közül legalább az egyik lecsapatása és extrahálása után kapott kristályokról mostak le

az említett fázisszeparált maradék vízzel nem elegyedő folyékony fázis (17) újrafelhasználása (224) a további extrakcióhoz újrahasználandó foszfor további adszorbeálására, és

a maradék, vízzel nem elegyedő folyékony fázisról lecsapolt mosófolyadék (17) újrafelhasználása (226) kristályok további mosására azon említett lépésben, amelyben a mono-ammónium-foszfát és a di-ammónium-foszfát közül legalább az egyik lecsapatott és extrahált kristályainak mosására kerül sor.

2. Az 1. igénypont szerinti eljárás, **azzal jellemezve, hogy**

az említett mosási lépésben felhasznált említett mosóoldat mono-ammónium-foszfát vagy di-ammónium-foszfát telített vizes oldatából áll, melynek rendeltetése, hogy mono-ammónium-foszfát illetve di-ammónium-foszfát előállítására használják.

3. A 2. igénypont szerinti eljárás, **azzal jellemezve, hogy** a mono-ammónium foszfát említett telített vizes oldatának pH-ját 2-6 pH értékre szabályozzák, vagy a di-ammónium-foszfát említett telített vizes oldatának pH-ját 6-10 pH értékre szabályozzák, hogy a kémiai reakciókat az adott mono-ammónium-foszfát vagy di-ammónium-foszfát adott vegyületeinek termelése céljából irányítsák, ahol a mono-ammónium-foszfát említett telített vizes oldata pH-jának említett szabályozása magában foglalja foszforsav vagy ammónia hozzáadását és a di-ammónium-foszfát említett telített vizes oldata pH-jának említett szabályozása magában foglalja ammónia hozzáadását.

4. Az 1-3. igénypontok bármelyike szerinti eljárás, **azzal jellemezve, hogy**
a foszforral terhelt, vízzel nem elegyedő folyékony fázis (14) előállításának említett lépése (210) magában foglalja azt a lépést, melynek során foszfort adszorbeáltatnak egy foszfortartalmú vizes oldatból egy folyékony megkötő anyagba, amely a foszfor iránt affinitást mutat, melynek során az említett, foszforral terhelt megkötő anyag az említett foszforral terhelt, vízzel nem elegyedő folyékony fázist alkotja(14); valamint azon további lépést amelynek során újrahasználják az említett, regenerált megkötő anyagot, amelyet az említett extrakciós lépés során nyertek, (218) a foszfor további adszorbeálására.

5. Az 1-4. igénypontok bármelyike szerinti eljárás, **azzal jellemezve, hogy** az említett hozzáadási (212) lépés magában foglalja az említett, vízzel nem elegyedő folyékony fázis vezetőképességének vagy pH-jának megfigyelését (14) valamint a hozzáadott vízmentes ammónia mennyiségének szabályozását (18) az említett, megfigyelt vezetőképesség vagy pH alapján.

6. Az 1-5. igénypontok bármelyike szerinti módszer **azzal jellemezve, hogy** a hőmérséklet szabályozásának (216) említett lépése magában foglalja a hőelvonást az említett vízzel nem elegyedő folyékony fázisból az említett hozzáadás (212) és lecsapatás (214) lépései előtt, alatt és/vagy után.

7. Ammónium-foszfátok előállítására szolgáló berendezés (10) amely az alábbiakat foglalja magában:
egy keverő térfogati berendezés (20);
az említett keverő térfogati berendezés (20) egy bemenettel rendelkezik (22) a foszforral terhelt vízzel nem elegyedő folyékony fázishoz (14);
az említett keverő térfogati berendezés (20) egy bemenettel rendelkezik a vízmentes ammóniának (18) az említett vízzel nem elegyedő folyékony fázishoz (14) való hozzáadásához;
egy hőcserélő (30), amely úgy van elrendezve hogy termikus érintkezésben legyen az említett, vízzel nem elegyedő folyékony fázissal (15);
egy vezérlő berendezés (34) amely az említett hőcserélő működtetésére van berendezve (30) hogy az említett vízzel nem elegyedő folyékony fázist (14) az említett keverő térfogati berendezésben (20) egy előre meghatározott hőmérséklet-intervallumon belül tartsa;
egy csapadékeltávolító (40) amely úgy van berendezve, hogy a lecsapatott mono-ammónium-foszfát és di-ammónium-foszfát közül legalább az egyik kristályait eltávolítsa az említett keverő térfogati berendezésből (20)

egy mosóberendezés (52, 52') amely az említett csapadékeltávolítóhoz csatlakozik (40) és az említett kristályok mosására van berendezve, és

egy szárító (54) az említeti mosóhoz csatlakoztatva (52, 52') és az említett, mosott kristályok szárítására berendezve,

a következőkkel jellemezve:

egy szeparátor (60) amely az említett mosóhoz csatlakozik (52, 52') és az említett kristályokról lemosott maradék vízzel nem elegyedő folyékony fázis (17) elválasztására van berendezve;

az említett szeparátor (60) az említett bemenethez csatlakozik az említett extrakciós szakasznál (12) a foszforból kinyert vízzel nem elegyedő folyékony fázishoz (17) az említett vízzel nem elegyedő maradék folyékony fázis újrafelhasználására (17) foszfor további adszorbeálása céljára az említett extrakciós szakaszban (12);

emellett, az említett szeparátor (60) úgy van elrendezve, hogy lehetővé tegye a maradék, vízzel nem elegyedő folyékony fázisból kinyert mosófolyadék (59) újrafelhasználását kristályok mosásra az említett mosóban (52, 52');

az említett mosó (52) az említett kristályok ammónium-foszfát telített vizes oldatával való mosására van berendezve; és

az említett szeparátor (60) egy fázisszeparátort foglal magában (58) amely az említett, vízzel nem elegyedő folyékony fázis (17) és az ammónium-foszfát említett telített vizes oldatának elválasztására van berendezve.

8. A 7. igénypont szerinti berendezés, **azzal jellemezve, hogy**
az említett mosó (52) a mono-ammónium-foszfát vagy di-ammónium-foszfát telített vizes oldatából álló mosóoldat alkalmazására van berendezve mono-ammónium-foszfát illetve di-ammónium-foszfát előállítására.

9. A 8. igénypont szerinti berendezés, **azzal jellemezve, hogy**
az említett mosó (52) a mono-ammónium-foszfát és/vagy di-ammónium-foszfát említett telített vizes oldata pH-jának szabályozására van berendezve.

10. A 9. igénypont szerinti berendezés, **azzal jellemezve, hogy**
az említett mosó (52) foszforsav vagy ammónia hozzáadására van berendezve az a mono-ammónium-foszfát említett, telített vizes oldatához vagy az ammónia hozzáadására di-ammónium-foszfát említett telített vizes oldatához.

11. A 7-10. igénypontok bármelyike, a **következőkkel jellemezve**:

egy extrakciós szakasz (12), amely foszfor adszorbeálására van berendezve egy foszfor-tartalmú vizes oldatból egy folyékony megkötő anyagba, amely a foszforra vonatkozóan affinitást mutat;

egy kimenet a nevezett extrakciós szakaszból (12) amely a foszforral terhelt megkötő anyag céljára csatlakozik az említett bemenethez (22) az említett keverő térfogati berendezés foszforral terhelt, vízzel nem elegyedő folyékony fázisához ahol az említett, foszforral terhelt megkötőanyag képezi az említett, foszforral terhelt

vízzel nem elegyedő folyékony fázist (14);

egy bemenet az említett extrakciós szakaszba (12) a foszforból kinyert megkötő anyaghoz (16) amely az említett keverő térfogati berendezéshez csatlakozik (20) és úgy van berendezve, hogy újrahasználja az említett keverő térfogati berendezésben képződő említett megkötő anyagot (20) az említett csapadékeltávolító működtetésével (40) foszfor további adszorbeálása céljára az említett extrakciós szakaszban (12).

12. A 7-11. igénypontok bármelyike szerinti berendezés, **a következőkkel jellemezve**:
az alábbiak egyike:

egy érzékelő (26), amelynek célja az említett, vízzel nem elegyedő folyékony fázis vezetőképességének megfigyelése (14); és

egy érzékelő (26), amelynek célja az említett, vízzel nem elegyedő folyékony fázis pH-jának megfigyelése (14); és

egy adagolóvezérlő egység (28), amely az említett érzékelőhöz csatlakozik (26) és a hozzáadott vízmentes ammónia mennyiségének szabályozására van berendezve (18) az említett vezetőképesség vagy pH megfigyelt értéke szerint.

13. 13. A 7-12. igénypontok bármelyike szerinti berendezés , **azzal jellemezve, hogy** az említett hőcserélő (30) működésének célja hogy hőt vonjon el az említett, foszforral terhelt, vízzel nem elegyedő folyékony fázisból, (14) amely belép az említett keverő térfogati berendezésbe (20), az említett, vízzel nem elegyedő folyékony fázisból az említett keverő térfogati berendezésen belül (20) és/vagy a foszfor-depletált vízzel nem elegyedő folyékony fázisból (17) amely az említett keverő térfogati berendezésből kilép (20).

14. A 13. igénypont szerinti berendezés, **a következőkkel jellemezve**:

egy folyékony ammónia-forrás (19);

egy hűtőegység (23) amely az említett folyékony ammónia-forráshoz csatlakozik (19) valamint az említett hőcserélőhöz csatlakozik vagy azzal össze van építve (30), az említett hűtőegység (23) úgy van berendezve, hogy az említett hőcserélőben (30) kivont hő legalább egy részét gáz halmazállapotú ammónia előállítására használják;

az említett bemenet (24) amelynek célja vízmentes ammónia hozzáadása (18) az említett keverő térfogati berendezésből, amely az említett gáz halmazállapotú ammóniának az említett folyékony ammóniaforrásból történő kivonása céljából van csatlakoztatva (19).