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(54) PURIFICATION OF PHOSPHORIC ACID

(71) We, ALBRIGHT & WILSON LIMITED, a British Company of P. O. Box No. 3, Oldbury, Warley, West Midlands (formerly Worcestershire), England, formerly of Oldbury near Birmingham, Warwickshire, England, do hereby declare the invention, for which we pray that a Patent may be granted to us and the method by which it is to be performed to be particularly described in and by the following statement:—

The present invention relates to the purification of phosphoric acid obtained by the wet process by reaction of sulphuric acid with phosphate rock. Such acid will hereafter be termed "wet process phosphoric acid". In particular the present invention relates to purification of such acid by solvent extraction techniques.

Proposals for solvent extraction purification of wet process phosphoric acid date back at least 40 years. Initially the solvents suggested (for example in U.S.P. 1968544) were water-miscible, for example alcohols and acetone, which required distillation in order to release the purified acid; alternatively there were suggested water immiscible short chain alcohols requiring many stages for effecting a satisfactory extraction.

In more recent years attempts to put into practice solvent extraction processes for wet process phosphoric acid have been made. However, these have largely been restricted to processes (such as those described in BP 085517 using butanol and BP 953378 using alkyl phosphates), in which either chloride ions are especially added to the acid or chloride ions are present as a result of using hydrochloric acid instead of sulphuric acid in the production of the phosphoric acid. The presence of chloride ions promotes a salting out effect which assists in promoting transfer of the phosphoric acid species to the organic phase.

A group of recent proposals is concerned with the use as solvents of certain ethers which extract the phosphoric acid present in aqueous

solutions at concentrations above a certain threshold value, quoted as 35% H₃PO₄, but do not extract any acid at concentrations below the threshold value. The first of these proposals is contained in BP 1,111,003 which, in addition to the ethers referred to above, also mentions a number of other possibilities including ketones. The method disclosed required a substantial temperature differential between extraction and release of the acid, the former being carried out at a relatively low temperature and the extract being heated, with or without the addition of a little water, to effect release of the acid. BP 1,240,285, which is a patent of addition to BP 1,112,003 describes the same process but using a mixture of solvents, one such mixture being an ether and a ketone, of which cyclohexanone is specifically mentioned. The third proposal in the group concerned with solvents having a threshold value quoted as 35% H₃PO₄ is USP 3,556,739 which describes the use of a wide range of aliphatic esters, aliphatic and cycloaliphatic ketones and glycol ethers. While the specific description is directed only to the same type of process as that of BP 1,112,003 in which the extract is heated in order to release the acid, reference is also made to the possibility of effecting release of the acid by an isothermal route involving one stage contact of the solvent extract with water. However no information is given as to which of the many solvents referred to in connection with the temperature-raising process may be employed or as to the temperatures appropriate to it.

A process has also been described in BP 1063248 for removing nitric acid from the aqueous phosphoric nitric acid mixture obtained by decomposing phosphate rock with nitric acid. In this process a ketone is used to extract the nitric acid, leaving the phosphoric acid in its impure form in the aqueous phase. Such acid could only be used for fertiliser production.

We have now found that if a ketone selected

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from a comparatively small group is used as the solvent it is possible to carry out an efficient purification of wet process acid at the commercially available concentrations, operating both the extraction and release at a temperature which is at or relatively near ambient temperature. This obviates the need either to refrigerate the acid for the extraction step or raise the temperature of the extract for the release step, and also ensures that the acid/solvent system is at a suitable viscosity for carrying out the process.

The present invention provides a process for purifying crude wet process phosphoric acid obtained from contact of sulphuric acid and phosphate rock which comprises contacting said crude wet process phosphoric acid with an unsubstituted acrylic dialkyl containing five or six carbon atoms, to give an organic extract containing at least some of the H_3PO_4 in the crude acid, and subsequently contacting the extract with water or an aqueous solution of a base to produce an aqueous phase consisting essentially of purified aqueous phosphoric acid or comprising a phosphate salt, the purified acid or salt being of increased purity having regard to the feed wet process acid and separating said aqueous phase from a ketone phase containing no more than 3% of the H_3PO_4 present in the extract, with the provisos that (a) when the ketone is a hexanone the acidity (as hereinbefore defined) of the crude acid is at least 65%, (b) when the ketone is methyl isobutyl ketone at least one of the contact of crude acid and ketone, and extract and water or base, is in more than 1 stage, and (c) when the ketone is a pentanone, the acidity of the crude acid is at least 50% and also, when the pentanone extract contacts water to produce an aqueous phase consisting essentially of aqueous purified phosphoric acid, the contact of extract and water is in more than one countercurrent stage. When employed herein the term "alkyl" means an unsubstituted saturated hydrocarbyl radical.

The preferred ketone is one unmentioned in USP 3556739 namely methyl isobutyl ketone. This is contrary to expectation, since this ketone is known to be useful for extracting from acid for analytical purposes the very metal impurities which remain in the aqueous phase in this solvent extraction process. We have found, however, that methyl isobutyl ketone has the advantage of being able to extract substantially all the H_3PO_4 values from a commercial wet process phosphoric acid of 70–85% acidity as defined below. Ketones of higher molecular weight require a higher concentration of phosphoric acid in the feed before they will extract this amount of H_3PO_4 , and it would therefore be necessary to institute a further difficult concentration stage of the acid prior to using such a ketone. Methyl isobutyl ketone further has the advantage of having a higher flash point as compared to

ketones of lower molecular weight but forming an azeotrope having a boiling point sufficiently low to enable easy solvent recovery by distillation. Furthermore methyl isobutyl ketone has low solubility (of the order of 1–2%) in aqueous H_3PO_4 , thus permitting the final product of the release stage to have only a low ketone content which may be removed with comparative ease as compared to other ketones as a result of the composition and boiling point of the azeotrope formed. It further permits the production of an aqueous raffinate having an H_3PO_4 content in the range 55–65% which after removal of the small amount of methyl isobutyl ketone present is suitable for use as such in fertiliser production, unlike the raffinate produced by using ketones of lower molecular weight.

The fact that phosphoric acid of normal merchant grade acidity may have substantially all of its H_3PO_4 content extracted into methyl isobutyl ketone at temperatures at which it is normally convenient and desirable to work (i.e. in the range 25–50°C, at which the system has a suitable viscosity) would normally require that a little water be added in order to produce a handlable raffinate. This may be accomplished either by adding water with or to the feed or more preferably by feeding aqueous phosphoric acid, which has been employed in a scrubbing operation to reduce the impurity content of extract, into the initial operation where wet process acid is contacted with the ketone.

Contrary to the teaching of USP 3556739, either or both of the extraction of H_3PO_4 from the wet process phosphoric acid into the five or six carbon atom-containing ketone and the release of purified acid, if carried out into water, is carried out in more than one stage, though in the case of the pentanones, at least the release into water to give purified acid is carried out in more than one countercurrent stage. The reason for this is that we have found a greater concentration of H_3PO_4 occurs in the raffinate for a given impurity content for a one stage extraction than for a two stage extraction. Thus less H_3PO_4 is lost in the raffinate if the extraction is carried out in two stages. The two stage extraction is conveniently accomplished by introducing the feed acid into one mixer unit of a two stage counter current mixer-settler extractor unit and the ketone into the other. If a scrubbing stage is employed, the recycled scrub liquor is fed into the same unit as the feed acid. The use of a multistage release permits an aqueous phosphoric acid of higher H_3PO_4 content to be obtained. Thus the initial extraction and/or release may be carried out in at least two (and preferably two) actual or theoretical countercurrent stages.

When an acid of lower concentration than merchant grade acid is available, e.g. acid of acidity in the range 50–70% eg 55–70% it may be preferred, rather than concentrating the acid, to employ a pentanone such as diethyl

ketone or methyl n-propyl ketone as the solvent, using both a two stage extraction into the ketone of H_3PO_4 and a 2 stage release into water, normally after a scrubbing operation has been effected. The concentration of phosphoric acid which is used to obtain most beneficial results according to the process of the invention will vary depending upon the ketone employed. If pentanones are employed an acid of an acidity as hereinafter defined of at least 50% normally up to 65% (preferably an acid of acidity from 50–55% as for example is obtained by the process described in BP 1209911) is employed. In such processes it is normal that instead of producing $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ as the by product of the reaction of H_2SO_4 on phosphate rock, $\text{CaSO}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$ is formed, either in one stage from the reaction or two stages, e.g. as a result of recrystallising the slurry obtained from a conventional process. If hexanones, such as methyl isobutyl ketone, are employed, the acid is of acidity of at least 65%, frequently in the range 70 to 85% preferably 74–79% (expressed as $\text{H}_3\text{PO}_4 + \text{H}_2\text{SO}_4$). The acidity of the acid is herein defined as the total weight percentage of H_3PO_4 and H_2SO_4 species present therein, this simple addition being possible as a result of H_3PO_4 and H_2SO_4 both having a molecular weight of 98. Thus an acid having an H_3PO_4 content of 74% and an H_2SO_4 content of 5% would have an acidity of 79%. All percentages in this specification are by weight unless otherwise indicated.

Use of such solvents will permit the production of purified acids of about 45% H_3PO_4 (by weight) if hexanones are used. The strengths of the acid obtainable can be increased if multi-stage releases are employed. For example an acid of concentration 55–58% H_3PO_4 can be obtained from an extract in methyl isobutyl ketone if such techniques are employed.

Phosphoric acid for purification according to the invention is obtained by filtration or other separation of calcium sulphate from the slurry obtained by the attack of sulphuric acid on phosphate rock. In such a state it contains a wide range of impurities, some of which such as fluorides and sulphate and dissolved organic material may if desired be removed from the acid prior to the solvent extraction of the present invention, other impurities such as iron, chromium, magnesium and other metallic cations, however, are not removable by such a pre-treatment.

Conventionally the acid from the attack stage is cooled as much as possible in order to induce post precipitation of the impurities present. The acid will normally be cooled to about 25–30°.

The temperature at which the solvent extraction is optimally carried out may vary according to the concentration of acid. However, we have found that a temperature equal to or below 50°C e.g. of 25 to 50°C is normally convenient for all concentrations of acid and

one in the range 35–40°C for methyl isobutyl ketone using an acid of an acidity in the range 65–85% is particularly useful.

The acid to be employed in the present process will either be obtained direct at the required concentration, or will be concentrated up to this level, e.g. by vacuum evaporation before purification. Acid of an acidity in the range 74–79% is the most commonly available commercial acid at the present time. Thus, for example, when methyl isobutyl ketone is employed it is common to employ an acid of about 79% acidity, for example 75% H_3PO_4 and 4% H_2SO_4 or 78% H_3PO_4 and 1% H_2SO_4 .

The wet process acid is contacted with a dialkyl ketone containing five or six carbon atoms, such as methyl n-propyl ketone, diethyl ketone, and methyl isobutyl ketone. The extracting ketone is preferably contacted with the aqueous wet process phosphoric acid in weight ratio of above 0.3:1, preferably in the range 0.5 to 2.0:1, more preferably in the case where the ketone is methyl isobutyl ketone in the range 1.0 to 1.5:1, optimally 1.2 to 1.4:1 for one stage extractions.

A ratio greater than 2.0:1 may be employed but would result in a requirement for larger vessels. If a 2 stage extraction is employed a slightly higher ratio e.g. 1.3 to 1.6:1 of ketone feed may be employed. We have found that aqueous phosphoric acid of the highest purity in a plant of the minimum size per unit H_3PO_4 throughput may be obtained if the ketone phase contains from 28 to 40% by weight of H_3PO_4 .

The contacting of the acid with the ketone is carried out in conventional equipment such as mixer-settlers and counter current packed columns. Normally an extractor with two or three actual or theoretical counter current stages, preferably two stages is employed for the reasons explained previously, although a one stage operation is possible.

Alternative apparatus for carrying out the contact such as sieved plate, rotating disc or pulsed column extractors of the desired number of theoretical stages may be employed.

If a phosphoric acid of high purity is required the extract containing the H_3PO_4 is then scrubbed by repeatedly contacting it with a small amount of scrub liquor which is preferably an aqueous solution of phosphoric acid of a high degree of purity in order to extract the cationic impurities into the aqueous phosphoric acid phase. Alternatively water can be used which re-extracts some H_3PO_4 in the first contact to give an aqueous phosphoric acid which may then be used as scrub liquor to further contact impure extract. The phosphoric acid used for scrubbing an impurity content sufficiently low to enable it to extract impurities from the ketone extract. It should also have an H_3PO_4 content of less than 45% for pentanones and less than 56% for hexanones in order to avoid transfer of H_3PO_4 from the scrub liquor

into the extract. It may either be acid obtained by the thermal process or be a wet process fulfilling the above conditions. Conveniently it is recycled purified acid from the release operation of the process. The amount of phosphoric acid used or produced by the partial extraction of H_3PO_4 into water and employed as the liquor to scrub the organic extract will normally be at least in amount of 10% of the amount of H_3PO_4 present in the organic phase. Preferably it is in the range 30–50%. That is to say the weight ratio of scrubbing liquor to extract is normally from 0.25 to 0.5:1 of the acid which has an H_3PO_4 content of about 56% if a hexanone such as methyl isobutyl ketone or about 45% if pentanones are employed.

After scrubbing the phosphoric acid is released from the organic phase by contacting it with water or with an aqueous solution of a base. This may be accomplished in one stage if desired. However, more commonly a multi-stage release, normally using 2 actual or theoretical stages is employed. The amount of water employed should be such that substantially all of the phosphoric acid passes into the aqueous phase from the organic. No more than 3% preferably no more than 1% of the H_3PO_4 values will be retained in the organic phase. The H_3PO_4 content of the aqueous phosphoric acid in a one stage release operation will be in the range 44 to 48% H_3PO_4 if the ketone employed is methyl isobutyl ketone depending upon the temperature employed; for example using water introduced at a temperature of 20°C an acid of H_3PO_4 content 45% may be obtained. Using a 2 counter current stage extraction higher concentrations e.g. in the range 55–59% may be obtained, for example 56% using water at 20°C. For pentanones such as diethyl ketone and methyl propyl ketone, a 2 stage release will provide an acid of about 43–48% H_3PO_4 . If desired, water at other temperatures for example condensates obtained from a heat exchanger such as used for vacuum concentrators may be employed. Typically temperatures in the range 20–40°C for the release step will be employed. For a 2 stage release the extract is fed to one mixer unit of a 2 stage counter current mixer settler unit and the water to the other mixer section. Although acid of these concentrations may be used direct for a number of applications it will normally be desirable to concentrate it if it is to be transported. When employing methyl isobutyl ketone as the solvent the amount of water employed for satisfactory one stage release is normally in the range 0.2 to 0.4:1 based on the weight of extract, whereas for 2 stage release an amount of water in the range 0.10 to 0.3:1 may be employed. For pentanones a ratio of 0.4 to 0.6:1 for a 1 stage and 0.05 to 0.3 for a 2 stage release suffices. It should be understood however that if a more dilute acid is desired more water may be employed. As

mentioned above instead of employing simply water for the release, it is also possible to employ an aqueous solution of a base. Suitable bases include caustic soda and caustic potash, ammonium hydroxide, water soluble amines and basic phosphates such as those of the formula M_2HPO_4 where M is a suitable cation so as to render the phosphate water soluble. The amount of base present is conventionally up to the amount required to neutralise all of the phosphoric acid in the extract to the dibasic form (e.g. MH_2PO_4) smaller amounts may be employed if sufficient water is employed to extract the remaining H_3PO_4 values from the ketone extract. Normally a one stage release is sufficient if an aqueous solution of base is employed for the release.

The final product obtained may contain a small amount of the ketone solvent employed. This can be released for example by distillation if desired. The process of the invention therefore normally produces two streams of phosphoric acid, one obtained via the solvent which contains approximately 50–70% of the H_3PO_4 present in the starting material and having an total metallic content less than 100 ppm relative to H_2PO_4 , and a less pure one containing phosphate not extracted into the organic phase which is suitable for use in fertilizer production. Alternatively a less pure purified stream containing e.g. 95% of the initial H_3PO_4 content may be produced by using a high concentration of feed acid and little or no scrubbing.

If desired the purified phosphoric acid may be further purified, (Serial No. 1436115) by raising its concentration and then crystallization as described in our Patent Application No. 44924/75.

The process of the invention is illustrated by the following examples:

In Examples 1 and 2 the feed wet process acid employed was of the following composition:

Specific gravity	1.66
H_3PO_4	77.4 %
H_2SO_4	1.6 %
Fe	0.23%
Mg	0.25%

In examples 1 and 2 the extracting solvent was methyl isobutyl ketone.

EXAMPLE 1

Is herein described with reference to the flow diagram Fig. 1. This illustrates a process in which the initial extraction of the H_3PO_4 into methyl isobutyl ketone is accomplished using a one stage mixer settler (1 in the Figure) and the final release into water is accomplished using 2 stage counter current mixer settlers (2 in the figure). The solvent extract is separated from the aqueous raffinate and scrubbed by being passed through a series of mixer settlers, 3, to extract cationic impurities from the solvent extract. The scrub liquor employed is the purified acid obtained from the release

mixer settler 2.

The feed acid is introduced to the first mixer settler at a temperature of about 25°C. The temperature rises as a result of the release of heat of extraction of the H_3PO_4 into the ketone.

The exact details of the conditions pertaining at each stage of the process are shown on the flow diagram.

Example 2 is illustrated by Fig. 2. In this example 2 counter current stage mixer settlers are employed both for the extraction of the H_3PO_4 into the ketone and for the release of H_3PO_4 into water. Figures for the conditions pertaining at various points and the compositions of the various streams are shown on the figure.

EXAMPLE 3

1000 cm³ wet process phosphoric acid derived from Moroccan phosphate rock having a specific gravity of 1.54, an H_3PO_4 content of 67% an SO_3 content of 1.6% and an iron content of 0.23% was fed into one mixer unit of a two stage counter-current mixer settler at 25°C where it was contacted with 425 cm³ of recycled scrub liquor of SG 1.34. Into the other mixer unit was fed 2000 cm³ of diethyl ketone of SG 0.82. 535 cm³ of a raffinate of S.G. 1.43 having an H_3PO_4 content of 53.5% an iron content of 0.46% and containing 6.5% of

ketone was taken from one settler unit. The ketone extract of the other settler unit was fed to a 3 stage counter-current mixer settler series in which it was scrubbed with 52 cm³ of water. The scrub liquor was recycled to the initial mixer settler of the extraction unit and the scrubbed organic phase passed on to a mixer in a 2 stage counter-current mixer-settler employed for the release. Into the other mixer of the 2 stage mixer-settler was fed 205 cm³ water. From one settler there was taken 1830 cm³ of solvent of SG 0.82 which was recycled. From the other 860 cm³ a product acid of specific gravity 1.34, or H_3PO_4 content of 54% a content of 7% and 125 ppm of iron was obtained.

It should be noted that a purer acid could be obtained by increasing the number of stages of the scrubbing operation.

EXAMPLE 4 and 5 COMPARATIVE EXPERIMENTS 1, 2 AND 3

In these examples and experiments the scrubbing operation was omitted. In each case 1000 cm³ wet process phosphoric acid as in Example 3 was extracted into a ketone and subsequently released into water in accordance with the conditions set out in table 1. The nature of the raffinate and the purified material is also set out in the table.

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TABLE 1

Example or Experiment No.	EXTRACTION				RAFFINATE			RELEASE			PRODUCT ACID		
	Solvent	Volume cm ³	No. of counter- current stages	Amount cm ³	H ₃ PO ₄ %	Fe%	S.G.	Volume of water cm ³	No. of counter- current stages	Amount cm ³	S.G.	H ₃ PO ₄ %	Fe%
4	diethyl ketone	1525	2	460c	52.5	0.39	1.42	235	2	995	1.34	52.5	67%
Compara- tive expt. 1	diethyl ketone	1525	2	460c	52.5	0.39	1.42	495	1	1185	1.29	45.5	67%
Compara- tive expt. 2	diethyl	1400	1	485	55.0	0.31	1.44	470	1	1110	1.29	45.5	63%
Example 5	methyl n-propyl ketone	1400	1	220	52.5	0.44	1.44	460	2	1385	1.31	48	0.09 84%
Compara- tive expt.	methyl n-propyl ketone	1400	1	220	52.5	0.44	1.44	810	1	1740	1.26	40	0.08 84%

**WHAT WE CLAIM IS:**

1. A process for purifying crude wet process phosphoric acid obtained from contact of sulphuric acid and phosphate rock which comprises contacting said crude wet process phosphoric acid with an unsubstituted acyclic dialkyl ketone containing five or six carbon atoms, to give an organic extract containing at least some of the H_3PO_4 in the crude acid, and subsequently contacting the extract with water or an aqueous solution of a base to produce an aqueous phase consisting essentially of purified aqueous phosphoric acid or comprising a phosphate salt, the purified acid or salt being of increased purity having regard to the feed wet process acid and separating said aqueous phase from a ketone phase containing no more than 3% of the H_3PO_4 present in the extract, with the provisos (a) that when the ketone is a hexanone the acidity (as hereinbefore defined) of the crude acid is at least 65%, (b) when the ketone is methyl isobutyl ketone at least one of the contact of crude acid and ketone, and extract and water of base, is in more than 1 stage, and (c) when the ketone is a pentanone, the acidity of the crude acid is at least 50% and also, when the pentanone extract contacts water to produce an aqueous phase consisting essentially of aqueous purified phosphoric acid, the contact of extract and water is in more than one countercurrent stage.
2. A process according to Claim 1 wherein the extraction of H_3PO_4 from the wet process acid into the ketone is carried out at from 25–50°C.
3. A process according to Claim 2 wherein the extraction is carried out at 35–40°C.
4. A process according to Claim 1 to 3 wherein the extract of phosphoric acid in the ketone is subjected to a scrubbing operation before it is contacted with the water or aqueous base.
5. A process according to Claim 4 wherein the scrubbing liquor after contact with the extract and containing impurities is fed to the first stage of the process in which the phosphoric acid is first contacted with the ketone in order to promote the formation of an aqueous raffinate.
6. A process according to either Claims 4 and 5 wherein the scrubbing liquor is phosphoric acid having an impurity content sufficiently low to extract impurities from the extract and is employed in an amount of H_3PO_4 of from 30–50% by weight of the amount of H_3PO_4 present in the organic extract.
7. A process according to any of the preceding claims wherein the initial extraction of phosphoric acid from the wet process phosphoric acid is effected in at least two actual or theoretical countercurrent stages.
8. A process according to Claim 7 wherein the extraction occurs in two actual or theoretical countercurrent stages.
9. A process according to any of the preceding claims wherein the H_3PO_4 from the organic extract is released by contact with water in at least two actual or theoretical countercurrent stages.
10. A process according to Claim 9 wherein the release is in two actual or theoretical countercurrent stages.
11. A process according to any of Claims 1 to 8 wherein the release is carried out in one stage by contacting the ketone extract with an aqueous solution of a base.
12. A process according to any one of Claims 1 - 11 wherein the contact of extract and water or base is at 20–40°C.
13. A process according to any one of Claims 1 - 8, 11 and 12 wherein the crude acid has an acidity (as herein before defined) of 65 to 85% and the ketone is a hexanone.
14. A process according to Claim 13 wherein the ketone is methyl isobutyl ketone.
15. A process according to Claim 13 or 14 wherein the crude acid has an acidity of from 70–85%.
16. A process according to Claim 15 wherein the crude acid has an acidity of from 74–79%.
17. A process according to any of Claims 13 to 16 wherein the ketone is contacted with the crude wet process phosphoric acid in a weight ratio of from 0.5 to 2.0:1.
18. A process according to Claim 17 wherein the ketone is contacted with the crude wet process phosphoric acid in a weight ratio of from 1.0 to 1.5:1.
19. A process according to any one of Claims 13–18 wherein the H_3PO_4 from the extract is released by contact with water in at least two actual or theoretical countercurrent stages.
20. A process according to Claim 19 wherein the contact of extract and water is in two actual or theoretical countercurrent stages, the weight ratio of the amount of water to extract being 0.1 to 0.3:1.
21. A process according to any of Claims 13–20 wherein the H_3PO_4 content of the extract is from 28–40% by weight.
22. A process according to any of Claims 13–21 wherein the initial extraction of phosphoric acid from the wet process phosphoric acid is effected in two actual or theoretical countercurrent stages, the weight ratio of ketone to crude acid being 1.3 to 1.6:1.
23. A process according to any one of Claims 1–10 and 12 wherein a pentanone contacts crude wet process acid of acidity (as herein before defined) at least 50% to give an organic extract containing 28–40% by weight of H_3PO_4 and the acid in the organic extract is released by contact with water in more than 1 actual or theoretical countercurrent stage, the weight ratio of water to extract being 0.5:1 to 0.3:1.
24. A process according to Claim 23 wherein the ketone is contacted with crude

acid in a weight ratio of 0.5:1 to 2:1.

25. A process according to Claim 23 or 24 wherein the ketone is contacted with crude acid of acidity 55–70% in two actual or theoretical countercurrent stages.

26. A process according to any of Claims 23–25 wherein the contact of acid and ketone occurs at 25–50°C and the contact of extract and water at 20–40°C.

27. A process according to Claim 5 or 7 wherein a hexanone contacts crude wet process acid of acidity (as herein before defined) 70–85% in an amount of 0.5 to 2.0:1 by weight to give an organic extract and an aqueous raffinate, the organic extract is scrubbed and then contacted with water to give the ketone phase and a purified aqueous phosphoric acid.

28. A process according to Claim 27 wherein the hexanone is methyl isobutyl ketone.

29. A process according to Claim 28 wherein the contact of water and extract is in two actual or theoretical countercurrent stages.

30. A process according to any one of Claims 27–29, wherein the crude acid acidity is as given in Claim 15 and/or the extraction temperature is as given in Claim 2 or 3 and/or the weight ratio of ketone to acid is as given in Claim 17.

31. A process according to Claim 5 or 7 wherein a pentanone contacts wet process acid of acidity (as herein before defined) of at least 50% in an amount of 0.5 to 2:1 by weight to give an organic extract and aqueous raffinate, the organic extract is scrubbed and then contacted with water in more than 1 countercurrent stage to give the ketone phase and a purified aqueous phosphoric acid.

32. A process according to Claim 31 wherein the extraction and release temperatures are as given in Claim 26 and/or the ketone is contacted with crude acid of acidity 55–70% in two actual or theoretical countercurrent stages.

33. A process as claimed in Claim 1 wherein methyl isobutyl ketone contacts crude wet process acid of acidity (as herein before defined) 74–79% in one stage at 25–50°C in a weight ratio of 1.2 to 1.4:1 to give an aqueous raffinate and an organic extract containing 28 to 40% by weight of H_3PO_4 , the extract is scrubbed with phosphoric acid having an impurity content sufficiently low to extract impurities from the extract, the scrub phosphoric acid containing an amount of H_3PO_4 of from 30–50% by weight of the amount of H_3PO_4 present in the extract, to give a scrubbed extract and used scrub liquor containing impurities which is fed to the first stage of the process in which the crude phosphoric acid is first contacted with the ketone, water then contacts the scrubbed extract in two actual or theoretical countercurrent stages at 20–40°C with a weight ratio

of water to extract of 0.1 to 0.3:1 to give a ketone phase containing less than 3% of the H_3PO_4 present in the extract, and a purified aqueous phosphoric acid and the ketone phase is recycled to the first stage for contacting the crude phosphoric acid.

34. A process as claimed in Claim 1 wherein methyl isobutyl ketone contacts crude wet process acid of acidity (as herein before defined) 74–79% in two actual or theoretical countercurrent stages at 25–50°C in a weight ratio of 1.3 to 1.6:1 to give an aqueous raffinate and an organic extract containing 28 to 40% by weight of H_3PO_4 , the extract is scrubbed with phosphoric acid having an impurity content sufficiently low to enable it to extract impurities from the extract, the scrub phosphoric acid containing an amount of H_3PO_4 of from 30–50% by weight of the amount of H_3PO_4 present in the extract, to give a scrubbed extract and used scrub liquor containing impurities which is fed to the first stage of the process in which the crude phosphoric acid is first contacted with the ketone, water then contacts the scrubbed extract in two actual or theoretical countercurrent stages at 20–40°C with a weight ratio of water to extract of 0.1 to 0.3:1 to give a ketone phase containing less than 3% of the H_3PO_4 present in the extract, and a purified aqueous phosphoric acid, and the ketone phase is recycled to the first stage for contacting the crude phosphoric acid.

35. A modification of the process as claimed in Claim 34 wherein the scrubbed extract is contacted with an aqueous solution of a base in one stage to give the ketone phase comprising a phosphate salt.

36. A process for preparing purified phosphoric acid substantially as herein before described in Example 1.

37. A process for preparing purified phosphoric acid substantially as herein before described in Example 2.

38. A process for preparing purified phosphoric acid substantially as herein before described in Example 3, 4 or 5.

39. A purified phosphoric acid prepared by a process according to any one of Claims 1–10, 12–34 and 36–38.

40. A phosphate salt prepared by a process according to any one of Claims 1–8, 11–18, 21, 22 and 35.

41. A purified phosphoric acid prepared by a process according to any one of Claims 27–30, 36 and 37.

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