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(54) **PROCEDE DE PURIFICATION D'ACIDE PHOSPHORIQUE PAR
VOIE HUMIDE**

(54) **WET-PROCESS TECHNIQUE FOR REFINING PHOSPHORIC
ACID**

(57) La présente invention concerne un procédé de purification d'acide phosphorique par voie humide, consistant à extraire les impuretés en plusieurs étapes en utilisant un mélange solvant, qui est distillé et cyclé. Ce procédé permet de fabriquer de manière avantageuse deux qualités d'acide phosphorique avec un rendement et un degré de pureté élevés.

(57) The present invention relates to a wet-process technique for refining phosphoric acid, consisting in multi-step removal of impurities by means of a solvent mixture, which is distilled and cycled. The inventive method permits the economic production of two phosphoric acid qualities with a high yield and purity grade.



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(54) Title: WET-PROCESS TECHNIQUE FOR REFINING PHOSPHORIC ACID (54) Bezeichnung: VERFAHREN ZUR REINIGUNG VON NASSVERFAHRENSPHOSPHORSÄURE (57) Abstract The present invention relates to a wet-process technique for refining phosphoric acid, consisting in multi-step removal of impurities by means of a solvent mixture, which is distilled and cycled. The inventive method permits the economic production of two phosphoric acid qualities with a high yield and purity grade. (57) Zusammenfassung Die vorliegende Erfindung betrifft ein Verfahren zur Reinigung von Naßverfahrensphosphorsäure durch eine Mehrstufen-Extraktion mit einem Lösungsmittelgemisch, wobei das Lösungsmittelgemisch destilliert und im Kreislauf geführt wird. Besonders vorteilhaft können durch dieses Verfahren zwei Phosphorsäure-Qualitäten in großer Ausbeute und Reinheit hergestellt werden.				

Process for the purification of wet-process phosphoric acid

5 The present invention relates to a process for the purification by an organic solvent mixture of an aqueous phosphoric acid which was obtained by the wet process. The process is carried out by the counter-current extraction principle in a plurality of stages.

10 Wet-process phosphoric acid, in the present invention, is taken to mean an aqueous phosphoric acid which has been prepared by decomposition of crude phosphate using acid, preferably sulphuric acid, and which has a P_2O_5 concentration between 40 and 62% by weight. An acid of
15 this type contains a number of inorganic impurities which interfere with the further use of this acid and must be removed.

To produce pure phosphoric acid by extraction with
20 organic solvents which are water-immiscible or only partially miscible with water, a large number of processes have already been proposed.

In the processes which have been disclosed, solvents such as alcohols, ketones, ethers and esters of
25 phosphoric acid are used.

However, there are only a few proven processes in practical use. All processes have the disadvantage that the solvents used are not able to take up the water
30 present in the wet-process phosphoric acid. This means that the degree of extraction, i.e. the yield of phosphoric acid that can be achieved, is determined by the decreased water uptake capacity of the solvent. For this reason, the circulated partially water-miscible
35 organic solvent, in particular the alcohols, must be completely or partially dehydrated. Processes of this type are described, for example, in DE-C 23 21 751, DE-C 23 34 019, DE-C 21 27 141 and DE-C 26 57 189. The degree of extraction is known to be dependent on the

- 2 -

P₂O₅ concentration of the crude acid, on the number of extraction stages, on the solvent to acid ratio and on the amount of recycled wash water.

5 In EP-A1 0 111 802, the partition coefficient when anhydrous alcohol is used is comparatively greater than when water-saturated alcohols are used. The P₂O₅ yield is here only 50 to 60% by weight. Nevertheless, this makes little difference to the fact that to achieve
10 maximum yields, a high number of extraction stages are required and, at the same time, a high solvent to acid ratio of 8 : 1 to 20 : 1 is required.

Wet-process phosphoric acid may also be extracted by
15 the so-called splitting process. This process is based on the fact that only a proportion of the P₂O₅, approximately 40 - 60% by weight, is extracted from the crude acid.

A typical procedure is described in US-A 3,318,661.
20 According to this, the crude acid containing approximately 48 - 62% by weight of P₂O₅ is extracted with isopropyl ether in a weight ratio of 1:0.5 to 1.5, about 40% of the P₂O₅ passing into the organic phase. The purified phosphoric acid is produced from the
25 isopropyl ether extract by adding sufficient water for an aqueous phosphoric acid solution having a P₂O₅ content of 48% by weight to be separated out from the organic phase. It is a disadvantage in this process that the isopropyl ether extract cannot be washed and
30 therefore only a low-quality phosphoric acid can be prepared. It is not possible to set a higher acid to solvent ratio to achieve a higher selectivity, since a third phase is then formed.

A further disadvantage of this process is that the
35 crude acid must be concentrated from approximately 48% by weight of P₂O₅ at least to 57% by weight of P₂O₅, in order to achieve a P₂O₅ yield of 60%.

Improvement in selectivity has been described in

- 3 -

US-A-3,903,247, according to which the crude acid is extracted with a solvent mixture consisting of 75 - 95% of a water-immiscible C2-C5 ether and 5 - 25% of a lower C3 - C8 alcohol. A particular disadvantage of this process is the poor yield and also, especially, the fact that an excessive amount of acid remains in the raffinate. The quality of the acid that is prepared must be classified principally only as "technical grade", i.e. it is not possible to prepare a higher quality without great expenditure on equipment.

Further extraction processes are described in DE-A 23 20 877 and DE-A 35 56 739, in which dialkyl ketones are used as solvent in a weight ratio to the acid of 0.5 to 2.0:1. An advantage of this process is the fact that the ketone extract, in contrast to the ether extract, can be purified by washing with water or phosphoric acid. The quality of the purified phosphoric acid is moderate, for which reason a further more complex purification step by concentration and crystallization of the phosphoric acid must also be carried out.

The object was therefore to eliminate the disadvantages occurring in the known extraction processes, in particular to prepare a phosphoric acid in high purity and at high yield. Such high-purity phosphoric acids are required in the food industry, for example.

The object set was surprisingly achieved according to the present process, which is characterized in that a prepurified phosphoric acid is extracted with recirculated solvent mixture in an extraction comprising one or more stages, which solvent mixture comprises a water-immiscible solvent or only partially water-miscible solvent and a fully water-miscible solvent, the mixing ratio being selected in such a manner that the mixture can take up the free water from the phosphoric acid. The prepurified phosphoric acid is

- 4 -

mixed with the solvent mixture and this phase is then washed with a solution containing alkali metal and/or ammonium ions in a multistage countercurrent treatment and the wash solution is then recycled back to the feed
5 phosphoric acid or to the extraction stage.

The process according to the invention comprises the following process steps, the numbers and letters listed here relating to Figures 1 and 2:

10

A(5) The prepurified phosphoric acid, possibly also containing alkali metal ions, is mixed with the solvent mixture and subjected to a counter-current multistage extraction comprising 3-5 mixer-settler extractor
15 units. This produces a bottom phase and a top phase, also termed extract. The resulting bottom phase is further treated in 1 to 3 extractor units with the fully water-miscible solvent, depending on whether sulphuric acid is added to the bottom phase.

20

A(4) Here, the bottom phase from A(5) is mixed with the water-soluble and water-insoluble solvent and extracted again. The bottom phase principally contains water, free acid and cations such as aluminium, magnesium,
25 iron etc. in the form of their phosphate salts.

A(2)-A(3) The bottom phase arriving from A(4) is mixed with the extract of the stages A(2) and A(1) in order to remove a portion of the sulphate ions from the
30 organic (solvent-containing) phase of the stages A(2) and A(3).

A(1) The aqueous phase from A(2) and A(3) is admixed with sulphuric acid and again extracted with the water-soluble solvent.

35 If preparation of a substantially sulphate-free phosphoric acid is desired, it is advisable not to add sulphuric acid to the extraction stage. In this case, 3 extractor units in total are sufficient, with the consequence that the P_2O_5 loss in the raffinate is

- 5 -

between 8 and 15% by weight, depending on the cation and sulphate content in the phosphoric acid used. In order to be able to decrease the P_2O_5 loss in the raffinate to 2 - 4% by weight, the sulphuric acid must
5 be added to the bottom phase. It is advisable to add the sulphate ions in the form of sulphuric acid in an amount equivalent to the cations in the feed acid. In this case, up to 5 extractor units are used. The raffinate produced at this point, which still contains
10 small amounts of solvent (approximately 10 to 15 g/l), is distilled and the residual raffinate is conducted away as waste.

A(5) The extract produced here, which is a phase
15 containing solvent mixture/phosphoric acid/water, is washed with an acidic alkali metal phosphate solution in B in a battery of 7 mixer-settler extractor units operated in counter-current. The volumetric ratio of wash solution to extract is between 0.02 and 0.03 : 1.
20 The number of extraction stages can be varied depending on the desired purity. The more stages through which the phosphoric acid passes, the higher the degree of purity.

25 C. The washed phase containing solvent mixture/phosphoric acid/water is passed into a continuous distillation apparatus (C) in order to separate off the lower-boiling fully water-miscible solvent and, after it is cooled to approximately 30°C, to pass it into the
30 multistage extraction stage A(4). In this operation, a two-phase system is achieved, since the organic solvent is supersaturated with respect to the water.

D The water-insoluble solvent mixture which remains
35 after the distillation from stage C and which still contains acid, is washed with the condensate from stage F. A dilute acid thus accumulates in the aqueous phase. In general, the water is not sufficient for reextracting the acid. Since water is taken off from

- 6 -

the system together with the raffinate, a small amount of water must additionally be added.

5 F. The phosphoric acid produced from D is concentrated in an evaporator to a P_2O_5 content of 45%, and can be separated off in F.

10 E. The water-immiscible or partially water-miscible solvent phase, after the reextraction stage, still contains about 5 to 8% by weight of P_2O_5 , based on the feed P_2O_5 , which phase is treated in the neutralization stage E with a 50% strength alkali metal hydroxide solution to pH 2 in a mixer-settler extractor unit, an acidic alkali metal phosphate solution being produced
15 which is recycled to the wash stage B. The water-saturated solvent is passed to the extraction stage A(4).

20 As water-immiscible solvents or only partially water-miscible solvents, use can be made of aliphatic or cyclic C_4 - C_{10} ketones or ethers or C_4 - C_8 alcohols. Such solvents are, for example, methyl isopropyl ketone, diisopropyl ketone, methyl i-butyl ketone, diisobutyl ketone, cyclohexanone, diisopropyl ether, diisobutyl
25 ether, i-amyl alcohol, hexanol.

As fully water-miscible solvents, use can be made of C_1 - C_3 alcohols, e.g. methanol, ethanol and i-propanol and n-propanol or acetone.

30 It is of importance in the process according to the invention that, in the solvent mixture, the fully water-miscible solvent has a lower boiling point than the water-immiscible solvent or only partially water-miscible solvent.
35

The suitable mixing ratio of the two solvents shall be selected in such a manner that the solvent mixture has the water uptake capacity required.

- 7 -

The commercially available wet-process phosphoric acids have a P_2O_5 content of 50 - 55% by weight, depending on cation and sulphate content. The following solvent mixtures, for example, are suitable for these phosphoric acids:

- i-amyl alcohol/isopropanol in a volumetric ratio of 90 : 10 to 60 : 40,
- methyl i-butyl ketone/isopropanol in a volumetric ratio of 80 : 20 to 50 : 50,
- diisobutyl ether/isopropanol in a volumetric ratio of 70 : 30 to 50 : 50.

15

The volumetric ratio of phosphoric acid to organic solvent mixture is 1 : 1 to 1 : 10, preferably 1 : 2 to 1 : 6.

If ether is used as solvent, the reextraction or washing stage is unnecessary, since ethers have a negative temperature gradient in relation to the dissolution of phosphoric acid and, after distilling off the fully water-miscible solvent at temperatures between 50 and 80°C, the dissolved phosphoric acid separates out with the water. This phosphoric acid has a P_2O_5 content of approximately 50% by weight, if the water which is formed on concentration of the phosphoric acid to 55% by weight of P_2O_5 is passed to the distillation. The temperature during the overall extraction can be between 5 and 90°C, preferably it is 30 - 80°C.

The alkali metal content, calculated as oxide, should be 0.1 to 3% by weight, preferably 0.1 to 1% by weight, based on the phosphoric acid, in the extraction and/or prepurification stage of the wet-process phosphoric acid by the organic solvent mixture. The alkali metal content in the phosphoric acid is adjusted by mixing with the recycled wash solution either in the prepurification or during the extraction. Depending on

- 8 -

the origin of the phosphoric acid, it contains greater or lesser amounts of impurities in the form of Al_2O_3 , MgO , etc. These cations form stable hydrates in the form of water complexes and thus bind the water in the crude acid.

If the Al_2O_3 content of the acid is above 0.6% by weight, e.g. in the case of acid from Morocco, it is advisable to add the alkali metal ions during the prepurification. If the Al_2O_3 content of the acid is above 1% by weight, e.g. in the case of acid originating from North Carolina/USA or from Israel, the wash solution containing the alkali metal ions should be admixed with water-miscible solvent during the extraction.

Prepurification of the wet-process phosphoric acid for the extraction is always necessary.

The prepurification can comprise not only precipitation reactions with CaCO_3 or crude phosphate, sodium sulphide and active silicic acid, in order to remove the dissolved arsenic and the fluoride and sulphate ions, but also adsorption processes using activated carbon in order to eliminate the organic impurities.

The subject-matter of the invention is described in more detail with reference to drawings 1 and 2 which represent the process according to the invention in a flow diagram, and by examples.

Figure 2 shows a further variant according to the invention of the process. By means of this variant it is possible to obtain the isolated phosphoric acid in two quality grades, in a technical and a high-purity quality grade. In this case, the process is carried out as described in Figure 1, but with the difference that the number of extractors in the wash stage B can be reduced from 7 to 4. The aqueous phosphoric acid separated out, which is formed after the evaporation of the fully water-miscible solvent in the distillation apparatus, is separated off and concentrated (in g) in the evaporator to a technical quality grade up to a P_2O_5

- 9 -

content of 55% by weight. The resulting water can be fully or partially recycled to the distillation apparatus, in order to control the amount of the aqueous phosphoric acid.

5

Example 1

A wet-process phosphoric acid produced from Morocco phosphate is mixed with the wash solution from the purification stage B, Fig. 1, and treated in a manner known per se with silicic acid, crude phosphate, sodium sulphide and activated carbon.

The phosphoric acid thus prepurified has the following contents:

15

P ₂ O ₅	53% by weight	MgO	0.59% by weight
SO ₄ ²⁻	1.5% by weight	Na ₂ O	0.60% by weight
Fe ₂ O ₃	0.43% by weight	As	< 1 ppm
Al ₂ O ₃	0.63% by weight		

In this example, a solvent mixture consisting of 70% by volume of i-amyl alcohol and 30% by volume of isopropanol was used. The volumetric ratio of phosphoric acid to alcohol mixture was 1 : 5.

20

10 l/h of this phosphoric acid are treated at a temperature of 25°C with approximately 51 l/h of a phase from stage A(4) containing alcohol mixture/phosphoric acid/water. 3.4 l/h of an aqueous bottom phase are produced continuously in the separation vessel. This bottom phase is again intimately mixed in stage A(4) with 35 l/h of circulated water-saturated i-amyl alcohol and 16 l/h of a phase from stage A(3) comprising isopropanol/phosphoric acid/sulphuric acid/water. The aqueous bottom phase from stage A(2) is mixed with 0.28 l/h of a 94% strength sulphuric acid and extracted in counter-current in the stages A(1) to A(3) with 15 l/h of isopropanol from the distillation stage.

25
30

- 10 -

In this treatment, 1.8 l/h of an aqueous bottom phase having a P_2O_5 content of 9% by weight are produced, which corresponds to a P_2O_5 loss of 2.9% by weight based on the feed P_2O_5 . It can be used in fertilizer
5 production.

The top phase from the extraction stage A(5) comprising alcohol mixture/phosphoric acid/water is washed in counter-current in a battery (B) of 7 mixer-settler vessels with wash solution which comprises alkali metal
10 ions and phosphoric acid and is formed by addition of sodium hydroxide solution to the neutralization stage (E) at the end of the process. After this treatment, 1.5 l/h of wash solution are produced which are passed to the prepurification stage of the wet-process
15 phosphoric acid.

The washed phase comprising alcohol mixture/phosphoric acid/water is passed to a continuous distillation apparatus and 15 l/h of isopropanol are evaporated at a temperature of 80-82°C. The isopropanol is passed to
20 the extraction stage A(1).

In this operation, a two-phase system is achieved, since the i-amyl alcohol is supersaturated with respect to water.

The phosphoric acid is reextracted from the two-phase
25 system with 6.8 l/h of water in counter-current in a battery of 4 mixer-settler vessels. This produces 14.7 l/h of a purified phosphoric acid having a P_2O_5 content of 40% by weight, which is concentrated in an evaporator to a concentration of 55% by weight of P_2O_5 .
30 5.5 l/h of condensate water are produced, which are used in the washing stage. The yield of P_2O_5 is 97.2%.

The water-saturated i-amyl alcohol phase, which still contains some phosphoric acid, is neutralized to pH 2 with 0.18 l/h of a 50% strength sodium hydroxide
35 solution. 1.2 l/h of a sodium phosphate solution are produced in the separation vessel. This solution is used in the purification stage. The water-saturated i-amyl alcohol is passed to the extraction stage A(4). The analytical data show the degree of purity of the

- 11 -

purified phosphoric acid in ppm, based on % P_2O_5 :

SO_4^{2-}	< 600	Ni	< 1
CaO	< 5	Cr	< 1
MgO	< 10	Cu	< 1
Al_2O_3	< 10	Zn	< 2
Fe_2O_3	< 5	Cd	< 1
		V	< 1

Example 2:

5

In this experiment, the same alcohol mixture as described in Example 1 is used. The volumetric ratio alcohol mixture : phosphoric acid is 4 : 1, however. A commercially available prepurified green phosphoric acid which was prepared by digesting calcined Israeli phosphate with sulphuric acid is used. It has the following chemical composition:

P_2O_5	54% by weight	Al_2O_3	1.92% by weight
SO_4^{2-}	2.5% by weight	Cd	6 ppm
MgO	0.51% by weight	As	< 1 ppm
CaO	0.08% by weight	Na_2O	0.09% by weight
Fe_2O_3	0.5% by weight		

15 10 l/h of this phosphoric acid are passed through in a similar manner to the process described in Example 1, but with the difference that the extraction is only performed in one battery of a total of 3 mixer-settler vessels in such a manner that the green phosphoric acid
 20 is extracted in stage A(3) with 41 l/h of the phase from stage A(2) which comprises alcohol mixture/phosphoric acid/water. The bottom phase produced in this case is treated once more in stage A(2) with 28 l/h of water-saturated and circulated i-amyl alcohol

- 12 -

and 13 l/h of phase from extraction stage A(1) comprising isopropanol/phosphoric acid/water. The bottom phase from stage A(2) is continuously mixed with the wash solution from the purification battery (Fig. 1, B) and, without sulphuric acid addition, is extracted in stage A(1) with 12 l/h of circulated isopropanol, 2 l/h of an aqueous raffinate having a P_2O_5 content of 22% by weight being produced, which corresponds to a P_2O_5 loss of 8% by weight, based on the feed P_2O_5 .

Further action is carried out as described in Example 1.

In the reextraction of the purified phosphoric acid with water, 13 l/h of pure phosphoric acid having a P_2O_5 content of 43% by weight are produced, which corresponds to a yield of P_2O_5 of 92%.

The analytical data of the phosphoric acid produced are summarized in the listing below in ppm based on % P_2O_5 :

SO_4^{2-}	< 100	Fe_2O_3	< 5	Zn	< 1
MgO	< 5	Ni	< 1	V	< 1
CaO	< 5	Cr	< 1	Cd	< 1
Al_2O_3	< 10	Cu	< 1	As	< 1

Example 3

In this example, a solvent mixture consisting of 60% by volume of diisobutyl ether and 40% by volume of isopropanol was used. The volumetric ratio of phosphoric acid to solvent mixture was 1 : 4.

10 l/h of green phosphoric acid used in Example 2 are extracted with 40 l/h of solvent mixture as described in Example 1. The difference from Example 1 is that the bottom phase from stage A(4) is continuously mixed with the wash solution from the battery B (Fig. 1, B) and extracted with 16 l/h of phase from stage A(2) comprising isopropanol/phosphoric acid/sulphuric acid/water. The bottom phase from stage A(2) is mixed with 0.2 l/h of a 94% strength sulphuric acid and extracted

- 13 -

in counter-current in the stages A(1) to A(3) with 16 l/h of isopropanol from the distillation stage. 1.7 l/h of raffinate having a P_2O_5 content of 10.5% by weight are produced in stage A(1), which corresponds to
 5 a P_2O_5 loss of 2.99%, based on the feed P_2O_5 .

After the solvent mixture/phosphoric acid/water phase is purified by washing, it is admixed with 2.5 l/h of water (from the phosphoric acid evaporation unit), and the isopropanol is distilled off as azeotrope in a
 10 continuous distillation apparatus at a temperature between 80 and 83°C.

The residue, consisting of ether, phosphoric acid and water, separates into two phases at this temperature, the aqueous phase containing virtually all of the
 15 phosphoric acid and having a P_2O_5 content of 50% by weight. 10.7 l/h of an aqueous phosphoric acid are produced. The yield of P_2O_5 is 97%, based on the feed P_2O_5 . The phosphoric acid thus produced is concentrated in an evaporator to a P_2O_5 content of 55% by weight. The
 20 resulting condensate, approximately 2.5 l/h, is passed to the distillation apparatus.

The ether phase still contains some phosphoric acid, which is washed out with 1.0 l/h of a 10% strength sodium hydroxide solution.

25 This alkaline phosphate-containing solution is used as wash solution in the purification stage.

The composition of the pure acid in ppm, based on %
 30 P_2O_5 :

SO_4^{2-}	< 600	Ni	< 1	Cd	< 1
Al_2O_3	< 10	Cr	< 1	As	< 1
Fe_2O_3	< 5	Cu	< 1		
MgO	< 10	Zn	< 1		
CaO	< 5	V	< 1		

Example 4

- 14 -

In this example, a solvent mixture consisting of 60% by volume of methyl isobutyl ketone and 40% by volume of isopropanol is used; the volumetric ratio of solvent mixture to phosphoric acid is 4 : 1.

10 l/h of green prepurified phosphoric acid are extracted with 40 l/h of solvent mixture as described in Example 3. 1.5 l/h of an aqueous raffinate having a P_2O_5 content of 11% by weight are produced, which corresponds to a P_2O_5 loss of 3%, based on the feed P_2O_5 . In the reextraction stage, 13.5 l/h of a purified phosphoric acid having a P_2O_5 content of 40% by weight are isolated using 6 l/h of water. The yield is 97%.

The analytical data of the pure acid are summarized in the listing in ppm based on % P_2O_5 :

SO_4^{2-}	< 600	Ni	< 1	Cd	< 1
Al_2O_3	< 10	Cr	< 1	As	< 1
Fe_2O_3	< 5	Cu	< 1		
MgO	< 10	Zn	< 1		
CaO	< 5	V	< 1		

Example 5 (Figure 2)

In this example, the process variant shown in Figure 2 is carried out. The Moroccan phosphoric acid is pretreated as described in Example 1. The pretreated phosphoric acid has a P_2O_5 content of 53% by weight and an SO_4^{2-} content of 3% by weight. 10 l/h of this acid are extracted with 50 l/h of alcohol mixture in a similar manner to Example 1, 1.9 l/h of an aqueous raffinate having a P_2O_5 content of 10% by weight being produced. The P_2O_5 loss is 3.4%.

The phase from extraction stage A(5) comprising alcohol mixture/phosphoric acid/water is washed with the wash solution in a battery of 5 mixer-settler vessels, 1.8 l/h of wash solution being produced.

- 15 -

The washed alcohol mixture extract is admixed with 1 l/h of condensate water and the isopropanol is evaporated as azeotrope in the distillation apparatus C. Two phases form. 5.9 l/h of aqueous bottom phase having a P_2O_5 content of 46.9% by weight are continuously produced in the separation vessel. The aqueous phosphoric acid of technical-grade quality is concentrated in the evaporator G to a concentration of 55% by weight of P_2O_5 . In this case, 1.4 l/h of condensate water are produced, of which 1 l/h is used in the distillation apparatus and 0.4 l/h is used in the reextraction stage.

The residual phosphoric acid is reextracted with 5.3 l/h from the i-amyl alcohol extract in a battery of 4 extractor units. 8.7 l/h of an aqueous phosphoric acid having a P_2O_5 content of 36% by weight are produced. This corresponds to a P_2O_5 yield of 49.7%.

Further action is performed as described in Example 1. The analytical data of the phosphoric acid thus produced in two quality grades are summarized in the listing in ppm based on % P_2O_5 :

	Technical-grade quality	Pure quality
SO_4^{2-}	1500	< 50
Al_2O_3	< 60	< 1
Fe_2O_3	< 50	< 1
MgO	< 100	< 1
CaO	< 10	< 1
Ni	< 2	< 1
Cr	< 2	< 1
Cu	< 2	< 1
Zn	< 50	< 1
V	< 10	< 1
Cd	< 1	< 1
As	< 1	< 1

15a

Explanation of the meaning of the numerals in Figures 1 and 2:

1. Entry of crude phosphoric acid
2. Entry of sulphuric acid
3. Exit of raffinate
4. Entry of sodium hydroxide solution
5. Entry of water
6. Exit of concentrated phosphoric acid (product) of technical-quality grade
7. Exit of concentrated phosphoric acid (product) of pure quality grade

- 16 -

Patent claims

1. Process for the purification of a wet-process phosphoric acid in a multistage extraction, the acid being prepared from the wet digestion of crude phosphates by sulphuric acid, containing alkali metal ions and being prepurified and being extracted with recirculated solvent mixture, characterized in that the solvent mixture consists of a water-immiscible solvent or only partially water-miscible solvent and a fully water-miscible solvent, the volumetric ratio of phosphoric acid to organic solvent mixture is 1 : 1 to 1 : 10, preferably 1 : 2 to 1 : 6, and the fully water-miscible solvent is separated off by distillation before the washing and recycled to stage A(1) of the extraction and the temperature during the overall extraction is between 5 and 90°C, preferably between 30 and 80°C.
2. Process according to Claim 1, characterized in that in the solvent mixture, the fully water-miscible solvent has a lower boiling point than the water-immiscible or only partly water-miscible solvent.
3. Process according to Claims 1 or 2, characterized in that, as solvents which are water-immiscible or only partially water-miscible, use is made of the aliphatic or cyclic C₄-C₁₀ ketones or ethers or C₄-C₈ alcohols, but preferably methyl isopropyl ketone, diisopropyl ketone, methyl i-butyl ketone, diisobutyl ketone, cyclohexanone, diisopropyl ether, diisobutyl ether, i-amyl alcohol and hexanol.
4. Process according to Claims 1 to 3, characterized in that, as fully water-miscible solvents, use is made of the C₁-C₃ alcohols, preferably methanol,

- 17 -

ethanol, i-propanol, n-propanol or acetone.

5. Process according to Claims 1 to 4, characterized in that the alkali metal content of the phosphoric acid, calculated as oxide, in the extraction and/or prepurification stage with the organic solvent mixture is 0.1 to 3% by weight, preferably 0.4 to 1% by weight, based on the phosphoric acid.
6. Process according to Claims 1 to 5, characterized in that the alkali metal content in the phosphoric acid is adjusted by mixing with the recycled wash solution either in the prepurification or prior to the extraction.
7. Process according to Claims 1 to 6, characterized in that the solvent phase which forms after the washing and still contains small amounts of phosphoric acid is neutralized to pH 2 with aqueous alkali metal hydroxide.
8. Process according to Claims 1 to 7, characterized in that the prepurification of the crude acid comprises not only precipitation reactions with CaCO_3 or crude phosphate, sodium sulphide and active silicic acid, but also adsorption processes using activated carbon.

-15-

Fig. 1

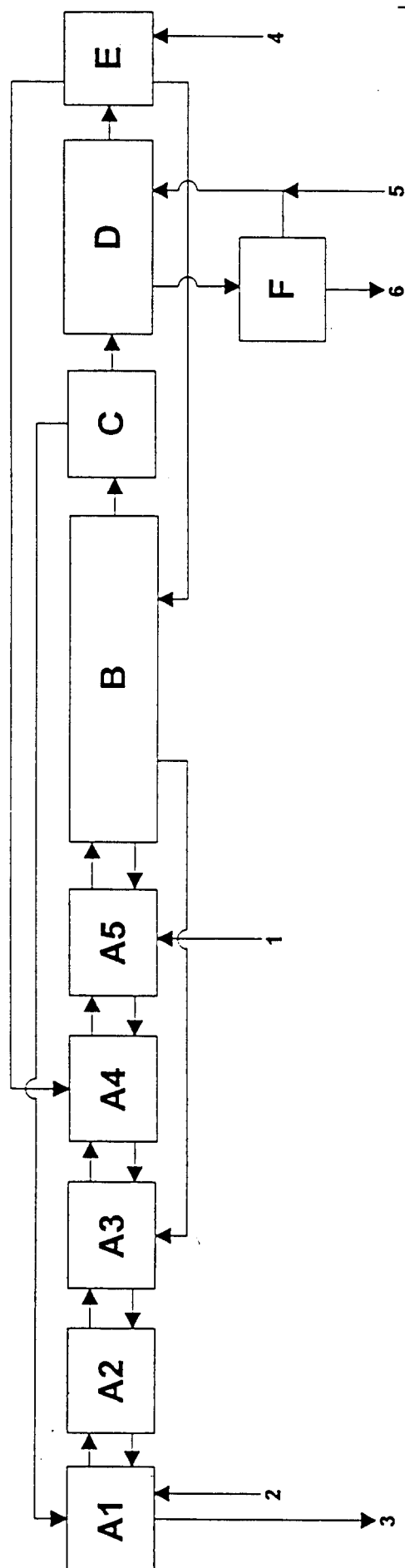


Fig. 2

