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Second Interregional Fertilizer Symposium

Kiev, USSR, 21 September - 1 October 1971
New Delhi, India, 2 - 13 October 1971

Agenda item [illegible]

[illegible][1]

by

[illegible]
[illegible]

[illegible]
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United Nations Industrial Development Organization

[illegible] Interregional Fertilizer Symposium [illegible]

New Delhi, India [illegible]

Agenda item [illegible]

[illegible]

[illegible]

by

[illegible]

[illegible]

[illegible]
[illegible] Finland

To make the phosphorus of phosphate rock soluble in water is the task of the industry producing phosphate fertilizers. This is conventionally done by breaking the mineral structure and transforming tricalcium phosphate into a soluble form by one of the following methods:

1) Phosphoric acid is added
2) Calcium is converted to another compound
3) Calcium is partially removed as sulphate, nitrate or chloride.

In the solvent extraction process, in contrast, the phosphorus itself is extracted as phosphoric acid and calcium together with other unwanted components remain unseparated. This in principle enables the production of a NPK-fertilizer with high nutrient content and high water solubility.

[illegible] solvent extraction [illegible] nitrophosphate process [illegible] the phosphate rock in nitric acid. [illegible], after [illegible] of the [illegible] is extracted [illegible] solvent, [illegible] which phosphoric acid and a [illegible] are transferred. The extraction is carried out in a new type

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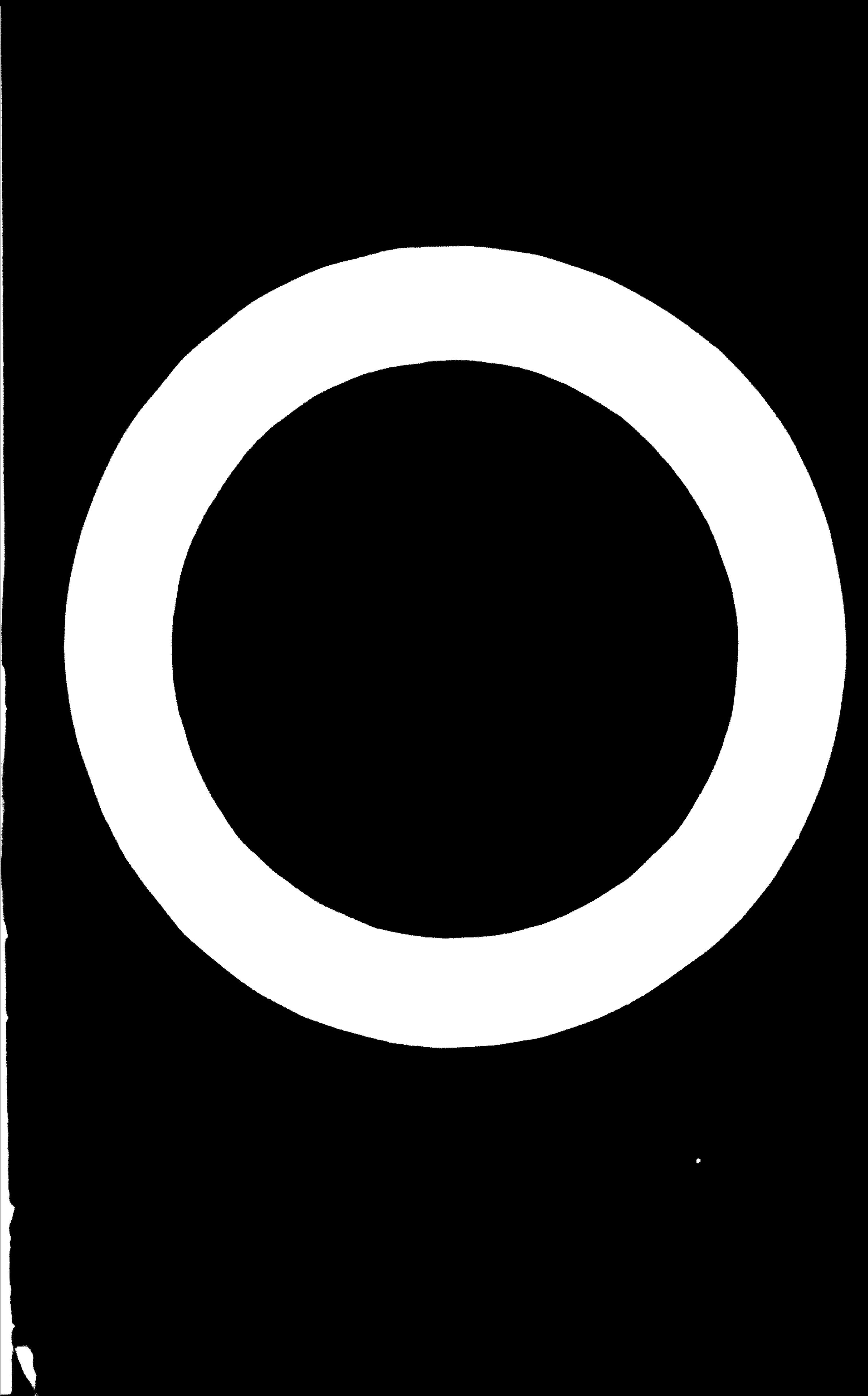

[illegible] pure form.

The important characteristics of this process are the high nutrient content of the products, the high water solubility of the phosphorus ([illegible] per cent), high recoveries of phosphorus and nitrogen and byproducts which are easily marketed. Economically it has been calculated to be competitive with other fertilizer processes giving the same quality of products.

This process has until now been used on pilot-plant scale with Kola-apatite as raw material. Other raw materials are under investigation. Consumption figures have been calculated and are given in the paper.

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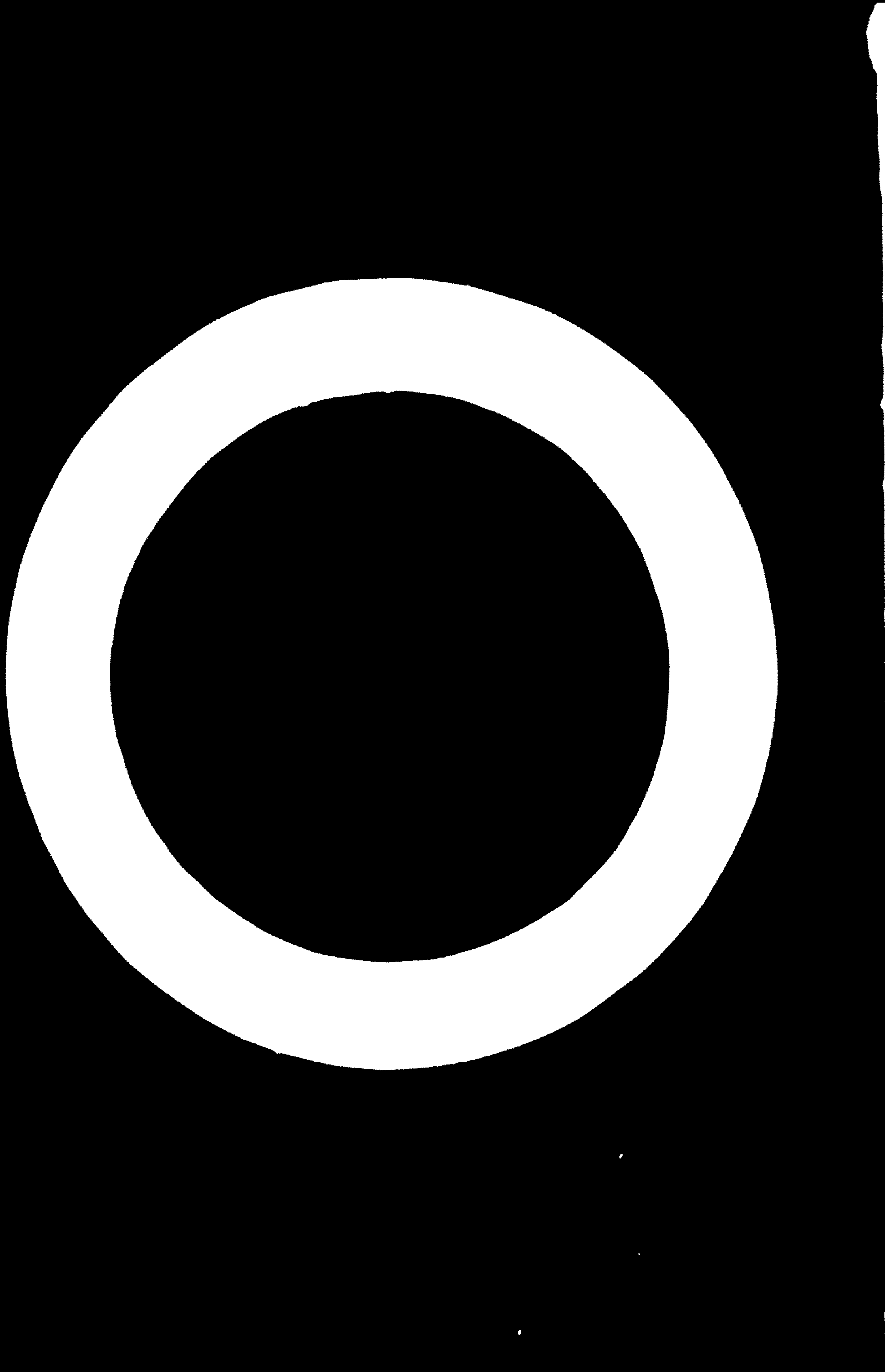


[illegible]

[illegible]

[illegible]

[illegible]

[illegible]

[illegible]

[illegible]

[illegible] and therefore only [illegible] can be given here. To take a [illegible] iron (III) chloride, ferric chloride [illegible] percentage of [illegible] (and some other compounds). [illegible] If we shake [illegible] containing ions of ferric iron and [illegible] manganese [illegible] separate into layers [illegible] ether [illegible] greater part of the iron is in the ether layer, whereas the manganese [illegible] in the water layer. The ether layer [illegible] separated [illegible], the iron is transferred to the water [illegible] and the ether can again be used to extract iron. [illegible] iron and manganese [illegible] operation can be repeated to give [illegible] separation [illegible].

Figure 1. The separation of the iron and the manganese by means of solvent extraction. This diagram illustrates the different stages of the separation of iron and manganese by means of solvent extraction as described.

The choosing of a suitable solvent for a given separation is, of course, [illegible] the component to be separated. It [illegible] insoluble in the water phase, that [illegible] from that of the water phase, that it does not react with [illegible], if it is to be used on an [illegible] scale, it must be readily available and cheap.

Solvent extraction as a separation method on a really large scale was first used for the [illegible], when methods were developed for [illegible] fuel. Nuclear fuel processes still are one of the [illegible] of solvent extraction. [illegible] separated by solvent extraction on an industrial scale are [illegible], copper, nickel, [illegible] and [illegible]. In the [illegible] of organic chemicals solvent extraction has [illegible] important applications.

[illegible] solvent extraction as a separation [illegible]. Only a few large scale [illegible] to persuade the [illegible]. [illegible] as long as [illegible]

[illegible]

16. [illegible] for [illegible] of [illegible] and [illegible] phosphate [illegible] Figure 2. [illegible] the Typpi [illegible] process.

[illegible] OF PROCESS

17. In the solvent extraction method developed by Typpi Oy, phosphate rock is dissolved in nitric acid and the insoluble matter is separated. The solution thus obtained containing calcium nitrate, phosphoric acid and nitric acid is fed to the [illegible] stage of the extractor, from which it passes through the extraction section of the extractor. Phosphoric acid and a part of nitric acid transfer into the solvent. The solvent coming from the extraction section of the apparatus still contains some calcium and other components from the phosphate rock and is fed into the washing section. The solvent is washed with ammonium nitrate solution obtained from the evaporation. From the washing section the washed organic phase containing phosphoric acid and nitric acid is obtained and also an aqueous phase containing ammonium nitrate which is combined with the aqueous phase of the extraction section, which then contains calcium and ammonium nitrates and the main part of the impurities of the phosphate rock.

18. The organic phase obtained from the extractor is neutralized with ammonia whereby a mixture of organic solvent, water and crystals of ammonium phosphate and of ammonium nitrate is formed. After settling, an upper layer of nearly pure organic solvent and a lower layer of ammonium phosphate and ammonium nitrate slurry (NP-slurry) are obtained. After the separation of the phases the solvent is recycled to the extraction section. From the NP-slurry a minor amount of solvent is recovered by [illegible] and recycled. The NP-slurry is transferred to the evaporation unit and from there to the production of fertilizers. The nutrient content of an NP-fertilizer produced by the Typpi Oy solvent extraction method is [illegible] and for the corresponding NPK-fertilizer the nutrient content is [illegible] and the water solubility of the phosphorus is about 95 %.

19. The aqueous phase obtained from the extraction is neutralized with ammonia and the solvent dissolved in this phase is recovered by distillation. The calcium nitrate solution can then be converted to ammonium nitrate by carbon dioxide and ammonia. The ammonium nitrate solution can be used for the production of ammonium nitrate [illegible].

20. The extraction [illegible] is [illegible] a [illegible] extractor, which [illegible]

[illegible] Typpi Oy [illegible] Typpi Oy
[illegible]
[illegible] from different solvent
[illegible]
[illegible] has been sold to
other companies for their own extraction processes.

15. The choice of organic solvent is a most important decision when developing new solvent extraction processes for nitrophosphates. The solvents mostly used for extracting the phosphoric acid from nitric acid solution have been n-butanol and [illegible]. These alcohols, however, react rather violently with nitric acid if the alcohol and nitric acid are mixed together as concentrated solutions or at too high temperature. Typpi Oy has investigated these alcohols but has decided to use tertiary amylalcohol, because it decomposes only very slowly under the conditions prevailing in the extraction process.

16. Solvent losses have a strong influence on the process economy and therefore Typpi Oy has carried out a very thorough investigation as regards the reasons and amount of solvent losses. In the solvent extraction process of Typpi Oy the solvent losses have been minimized by optimizing the process conditions. One essential factor in reaching good process economy has been the fact that almost [illegible] % of the cycling solvent is separated by mechanical means and only a little part is recovered by distillation.

The quality of fertilizers produced by the Typpi Oy solvent extraction process

17. The main part of the research and development work has been carried out using Kola-apatite. It has, however, been verified that this process can also be used for low-grade raw materials. Using Kola-apatite as raw material a product is obtained the analytical data of which are given in Table I.

18. Table I. The analytical composition of the products obtained by the Typpi Oy extraction process

		Kola-apatite	NP [illegible]	Ammonium nitrate
Tot N	%		26,9	34,0
NH_4-N	%		16,1	17,0
NO_3-N	%		9,9	17,0
Tot P_2O_5	%	39,[illegible]	26,0	0,0[illegible]
WS P_2O_5	%		25,6	
CaO	%	5[illegible],6	3,[illegible]	0,[illegible]

		[illegible]	[illegible]	[illegible]
P		[illegible]	[illegible]	0,075
SiO_2	%	[illegible]	[illegible]	
Fe_2O_3	%	0,[illegible]	0,[illegible]	[illegible]
Al_2O_3	%	[illegible]	0,[illegible]	0,[illegible]
MgO	%	0,[illegible]	0,[illegible]	0,[illegible]
Na_2O	%	0,[illegible]	0,[illegible]	0,[illegible]
K_2O	%	0,[illegible]	0,[illegible]	0,[illegible]
SO_3	%	0,[illegible]		
[illegible]	%	0,10		

19. The fertilizers produced consequently have a very high nutrient content and also the water solubility of phosphorus is high, about 90 %. In the NP-product the ratio nitrogen-phosphorus pentoxide is [illegible] or larger, which is [illegible] proportions. When producing an NPK-fertilizer potassium can be added as potassium chloride and a fertilizer with the composition NPK 10-10-10 is obtained.

[illegible]

20. The overall costs of the extraction method have been thoroughly compared with the costs of other nitrophosphate processes.

21. **Table IIa** Typical consumption figures for a production of 1 ton NPK + [illegible] ton [illegible] % ammonium nitrate solution, calculated as 100 %.

Rock phosphate ([illegible] % P_2O_5)	0,660 ton
Nitric acid 100 %	0,[illegible] "
Ammonia	0,207 "
Potash (60 % K_2O)	0,180 "
Carbon dioxide	0,180 "
Steam [illegible] atü	0,225 "
Steam [illegible] atü	0,217 "
Electricity	50 kWh
Fuel	30 kg
Cooling water	30 m^3
Process water	0,2 m^3
[illegible]	< 2 kg

22. The investment for a plant with the production figures mentioned below is [illegible] Mill. US Doll.

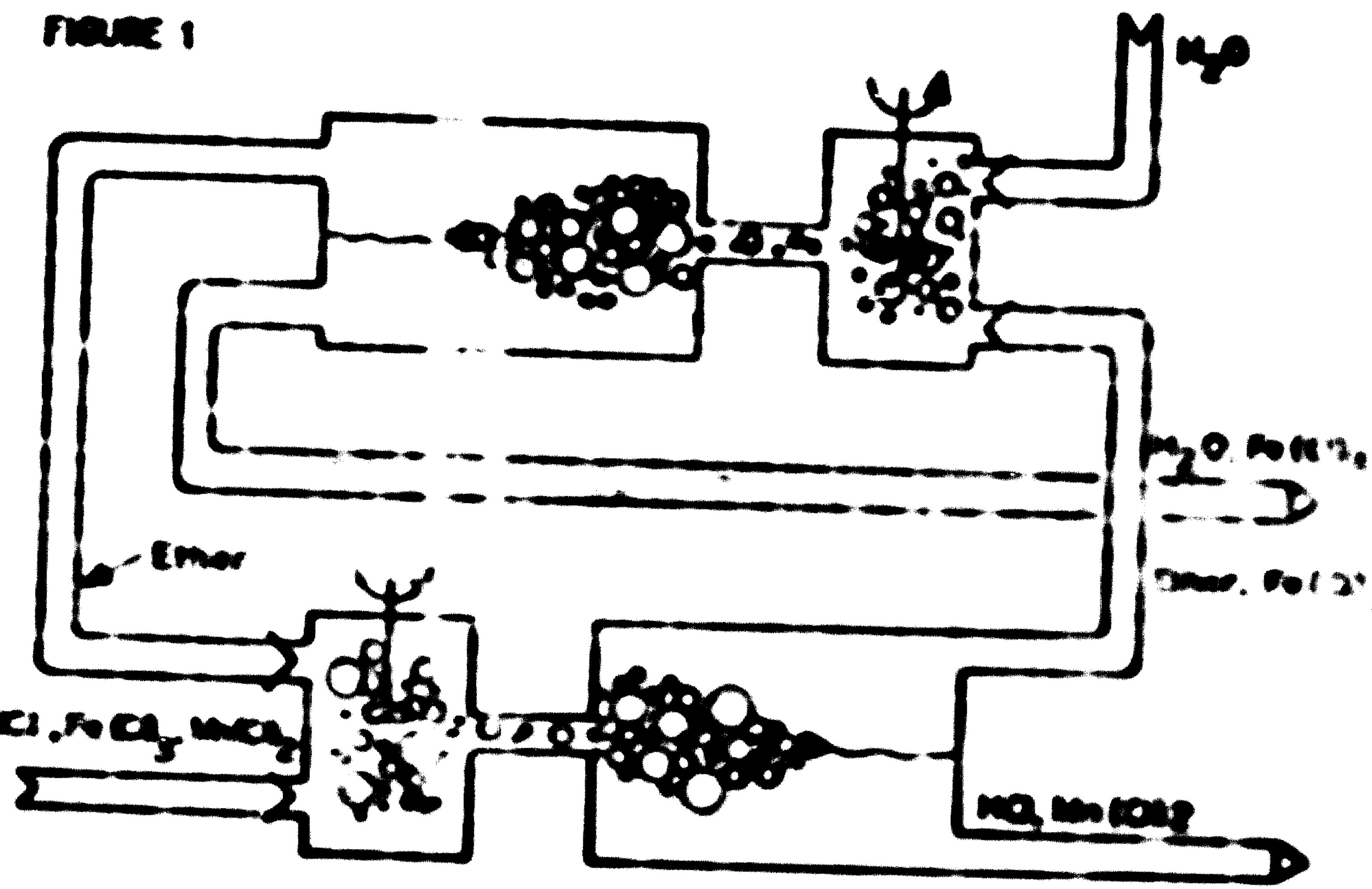
23. The plant [illegible] tons P_2O_5 as a fertilizer, [illegible] ratio [illegible] as byproduct [illegible] tons/a pure [illegible] ammonium nitrate solution are obtained. Daily production of this facility is:

[illegible] tons NPK [illegible] and

[illegible] tons 60 % ammonium nitrate solution, calculated as 100 %

24. This process has as its main advantages the high quality of the products, [illegible] recovery of the components phosphorus and nitrogen and also byproducts such as fluorine and the absence of wastes. Also when nitric acid is more readily available than sulphuric acid this process is preferred. The possible use of low-grade phosphate rock may also be one of the main advantages of this process. Solvent extraction being a new technology for fertilizer production it offers many new promises for the future, which until now have not been fully exploited.

FIGURE 1



SEPARATION OF IRON AND MANGANESE BY SOLVENT EXTRACTION

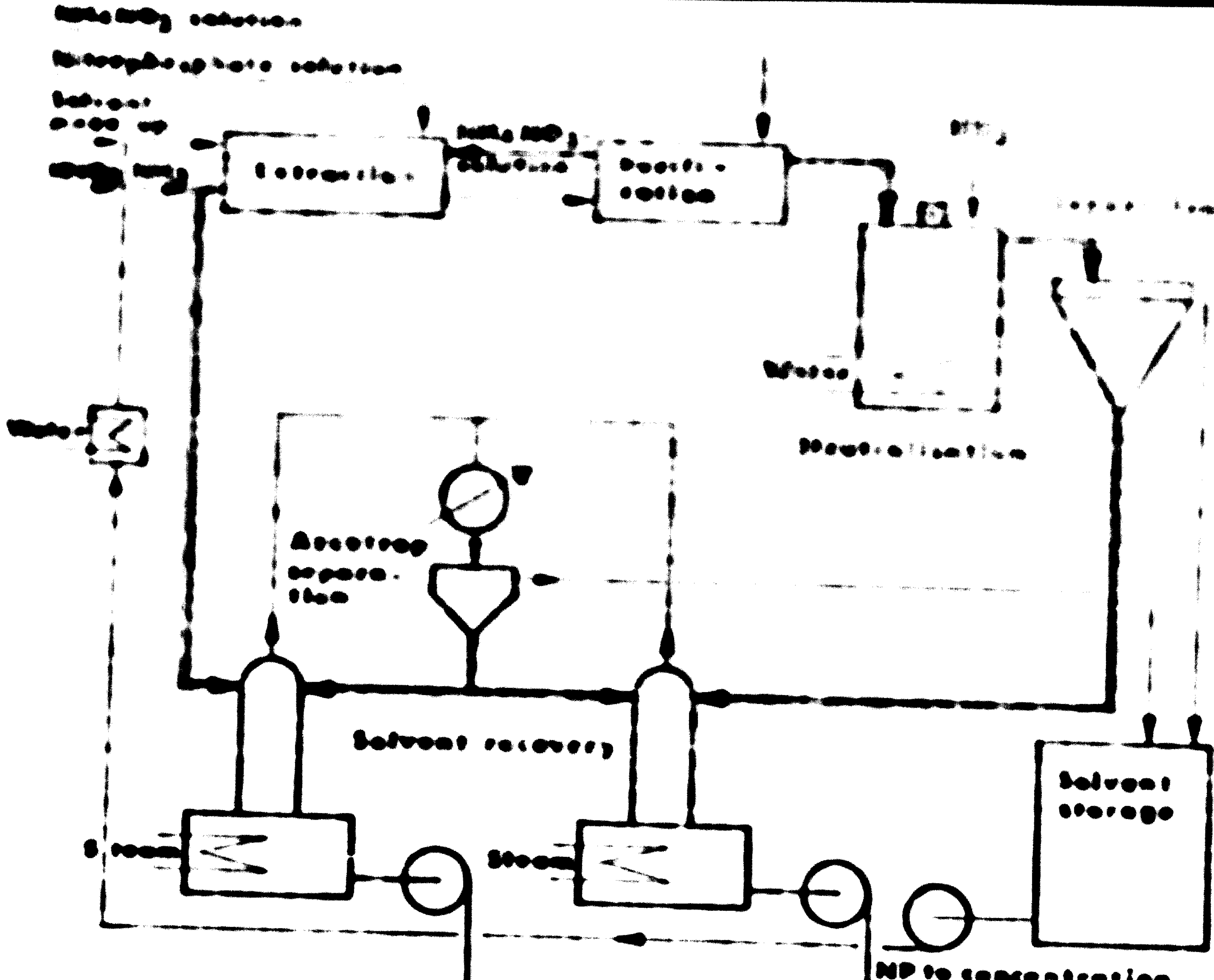


FIGURE 2. TYPE OF SOLVENT EXTRACTION PROCESS FOR THE PRODUCTION OF COMPOUND FERTILIZERS.

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