The ERT/ESPINDESA Low-Recycle DAP Process

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by

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Introduction

Explosivos Rio Tinto (ERT) was founded more than 100 years ago. Today it is the largest chemical manufacturer in Spain and operates 14 granulation plants sized from 250 tpd to 1300 tpd and producing among others:

Ammonium Phosphates (MAP and DAP)
NPK Fertilizers
Granular Superphosphates
Ammonium Nitrates (26 and 33.5% N)

and the corresponding raw materials, such as Ammonia, Urea, Nitric Acid, Sulphuric Acid and Phosphoric Acid.

Total production of granular fertilizers amount to 1.3 million metric tons per year, of which about 10% is exported overseas.

ESPINDESA is a subsidiary of ERT and Tecnicas Reunidas (the largest Engineering Contractor in Spain) involved with the development and commercialization of technology in the nitric acid and fertilizers fields.

Development of the pipe-reactor in ERT

The ERT Huelva plant has manufactured powdered MAP since 1966. Part of this MAP is used in a neighboring NPK granulation plant, and the rest is exported by train, truck or ship to other ERT plants in Spain.

ERT has studied, since 1979, the use of the TVA Pressure Reactor for the manufacture of NPK fertilizers. Encouraging results were obtained and, by 1980, phosphoric acid substituted MAP as raw material in some plants for the manufacture of NPK fertilizers.

The next steps were the modification of the pipe-reactor design to enable the manufacture of powdered MAP (1981) and granular DAP (1983).

At present we are studying in the pilot plant the development of powdered DAP as an intermediate product for the manufacture of granular NPK and DAP extra-low recycle (below 1:1 recycle ratio).

Powdered MAP via pipe-reactor

Since 1981, the ERT plant at Huelva has produced 450 tpd of low moisture powder MAP. The process consists of a pipe reactor located at the top of a tower 17 meters high. The pipe reactor is made of stainless steel and is fed with phosphoric acid and ammonia, and produces a melt of MAP which solidifies when falling inside the tower (Fig. 1).

The moisture of the product is adjusted by means of the concentration of phosphoric acid fed to the pipe (see Fig. 2) in such a way that, for a certain P_2O_5 content in the phosphoric acid, and a molar ratio NH_3/H_3PO_4, a given moisture in the MAP is obtained.

The tower is a natural-draft type and the product is collected in the bottom as a cold powder which can be stored without caking or directly used as an intermediate product in the manufacture of NPK granules.

The cooling air from the tower is scrubbed with phosphoric acid or water, to recover the entrained MAP particles and the non-reacted ammonia. If phosphoric acid is used as a scrubbing liquor, the partially neutralized part of the water is used to dilute the feed acid.

Features of the system:

- Very simple plant design
- Easy operation
- Moisture of MAP can be adjusted from 2% to 12%
- Very high ammonia and P2O5 efficiencies
- Low energy consumption

The low-recycle DAP process

This process is based on the use of a special pipe reactor of proprietary design placed inside the granulator. What differentiates the ERT-ESPINDESA pipe reactor from other pipe reactors is that both reactions take place to completion in it, and it is thus the only pipe reactor system, to date, in which total reaction of ammonia and phosphoric acid to DAP is achieved in a single piece of equipment.

In the pipe reactor, ammonia and phosphoric acid are reacted under pressure in a very short time, in such a way that almost all the heat of reaction is evolved inside the pipe. Consequently most of the water is evaporated from the phosphoric acid solution. This yields a much more concentrated DAP melt than would be obtained in a conventional reaction vessel, which means that lower quantities of material need to be recycled to the granulator; the need for pumping slurries close to their solidification point is also avoided. The ammonia to phosphoric acid molar ratio reached 2.05:1.0; there is no need to add further ammonia to the granulator.

When compared to the conventional DAP granulation processes, the ERT-ESPINDESA low-recycle process offers a number of advantages for the manufacture of DAP. The process has a low recycle ratio - 2.5-3 (a conventional granulation process typically has a recycle ratio of 4-6). It is also easier to operate. For example: only one ammonia feed to the anti-foaming agents is required; and there is no need for an ammonia sparger in the granulator. Furthermore, P205 in the product has a high solubility in water (98% compared with 90-92% for conventional DAP); this seems to be due to the short residence time of the phosphoric acid and ammonia in the reaction equipment. These features mean that there is a lower operating cost. The investment required for a new plant is also smaller because of the smaller equipment size (due to the lower recycle ratio) and the simpler lay-out that is possible.

The process has several other advantages. Production rates in existing conventional DAP plants can be increased by up to 50% by retrofitting the ERT-ESPINDESA pipe reactor. Acids with a high impurity content can be employed as feed-stock; several phosphoric acids containing significant amounts of gypsum and agnesium have been tested and the quality of the DAP produced was excellent. It is also possible, in the same plant, to produce a wide range of NPK fertilizers. Finally, new plants are designed to meet strict environmental regulations.

Process description

An outline of the ERT-ESPINDESA low-recycle DAP process is shown in the Fig. 3. Initially, phosphoric acid with a concentration of 40% P2O5, or higher, is fed to a two-stage ammonia scrubber, where it absorbs most of the ammonia lost in the granulator. A pump is used to transfer the acid between the two sections of the scrubber. From the ammonia scrubber, the partially neutralized phosphoric acid, with an ammonia to phosphoric acid molar ratio of about 0.25, is pumped to the pipe reactor, where it reacts under pressure with ammonia to a final molar ratio of 1.9-2.05, depending on the impurities present in the acid feed. The reaction is instantaneous and exothermic, and part of the water content of the phosphoric acid evaporates to dissipate the heat of the reaction. A slight excess of ammonia (about 15% over the stoichiometric amount) is used in the pipe reactor, but NO AMMONIA IS ADDED DIRECTLY TO THE GRANULATOR. Excess ammonia is

recovered in the ammonia scrubber, as described. The mixture of air and water vapor removed from the ammonia scrubber is passed through a final gas scrubber before it is discharged to the atmosphere.

From the pipe reactor, the hot concentrated DAP melt is sprayed over recycled DAP in the granulator, which is of the rotary drum-type and lined with self-cleaning rubber panels. Due to the low water content of the hot DAP melt, the recycle ratio can be kept between 2.5 and 3.0. At the exit of the drum, the moisture content of the product is below 3.0%.

Granulated product falls directly into a rotary drier where it is dried to the desired moisture content. This drier is of a smaller size than those used in conventional processes. Product leaving the drier is sent, via a bucket elevator, to screens, where it is divided into three streams: oversize, product and fines. The coarse fraction (oversize) is passed to the mill whence the crushed material is discharged, together with the fines from the screen, into a storage bin. The output from this bin is regulated in such a way that a constant feed of material is recycled to the granulator. The on-size material from the screen is conveyed to storage.

Hot air from the drier is removed through cyclones to the same gas scrubber as the gases from the ammonia scrubber before being discharged to the atmosphere.

Several granulation plants are being retrofitted to utilize this new technology. The length of the pipe reactor is five meters and it is thus easily fitted inside the granulator. No other changes are required to the plants, as long as they are equipped with suitable systems for ammonia recovery.

Raw Materials and Utilities Consumptions for Producing DAP (18-46-0)

Raw materials consumptions, t/t

Ammonia (100% NH3)	0.221
Phosphoric acid (100% P2O5)	0.463

Efficiency of raw materials utilization, % (min)

Ammonia	99
Phosphoric acid	99.5

Utilities consumption

Steam, kg/t	1
Electricity, kWh/t	25
Fuel oil, kg/t	2

Pipe Reactor Description

Depending on the phosphoric acid impurities, the pipe-reactor can be made of 316 L SS or Uranus 20.

Pipe diameter and length are variable according to the design rate. For 30 t/h of DAP the diameter is 4 inches and the length 20 feet. Only 5 feet are placed inside the granulator.

The pipe-reactor consists of 3 zones: Raw-materials feed, reaction zone and the spray end.

The pipe-reactor is supported outside the granulator and therefore the retrofitting of it to an existing granulator is very simple.

DAP Quality

- Phosphoric Acid Feed Analysis, wt%:

P2O5	37.35
F	1.41
SO3	1.943
SiO2	0.47
CaO	0.79
Al2O3	0.434
Fe2O3	0.457
MgO	0.439
Na2O	0.164
K2O	0.017
Cl	0.033
Org. Mat.	0.02
Susp. Solids	2.44

- DAP Analysis obtained with that acid, wt%:

N	18.25
Water soluble P205	45.12
Citrate soluble P205	46.44
Total	46.57

$$\text{Ratio} \frac{\text{Water sol. P205}}{\text{Total}} = 96.9\%$$

$$\text{Ratio} \frac{\text{Citrate sol. P205}}{\text{Total}} = 99.7\%$$

Extra low-recycle DAP

As mentioned at the beginning of this paper, we have under pilot plant study the manufacture of powdered DAP in a similar way to the present manufacture of powdered MAP although using a shorter tower.

If the laboratory studies are confirmed in the pilot plant, we could, by adding a small tower to any existing DAP granulation plant, manufacture a granular DAP with a recycle ratio below 1:1.

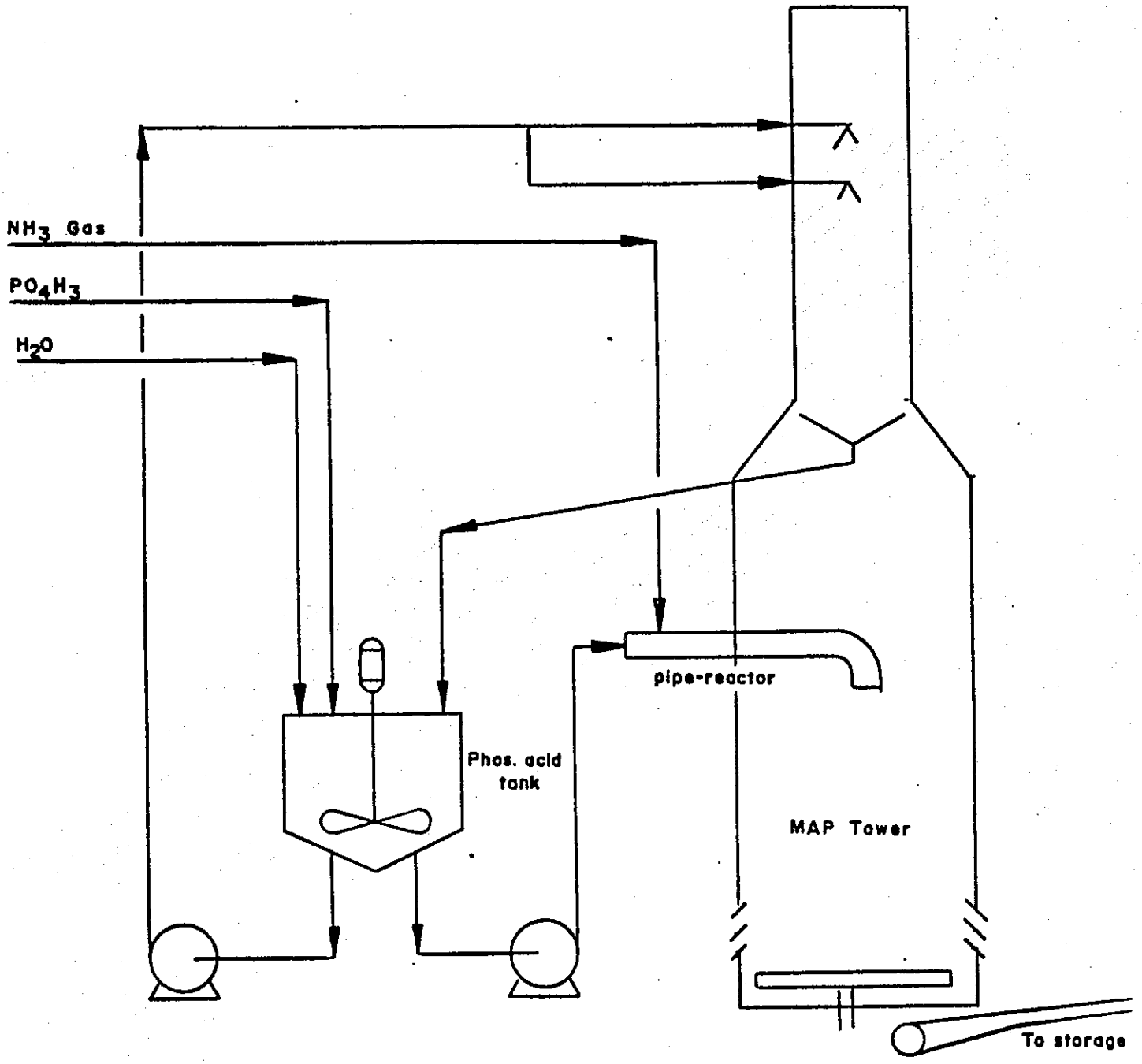

FIG. 1

ERT - ESPINDESA MAP Process

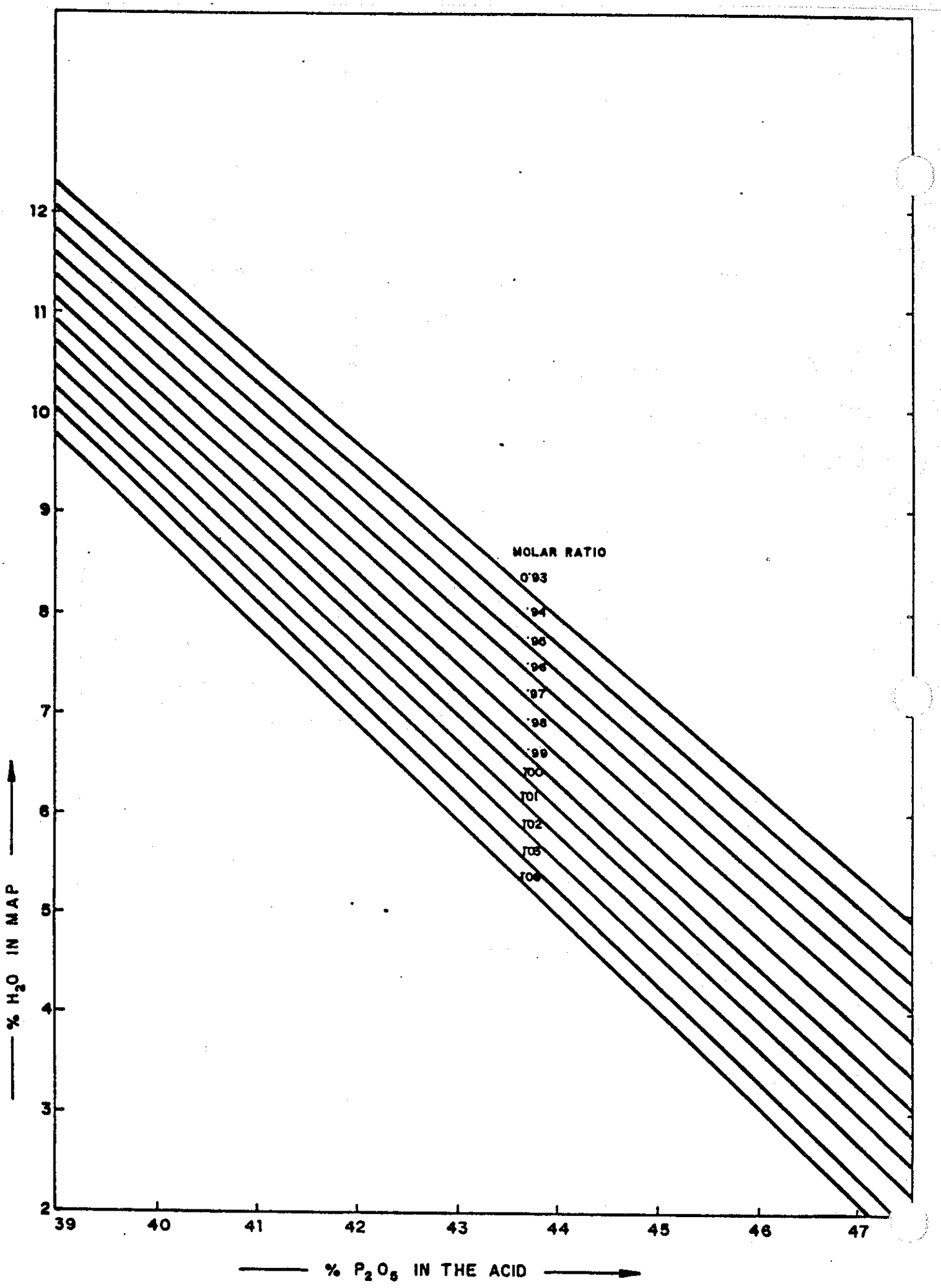

Fig.2 :-MOISTURE OF THE MAP VS P_2O_5 CONTENT OF THE ACID FEED.

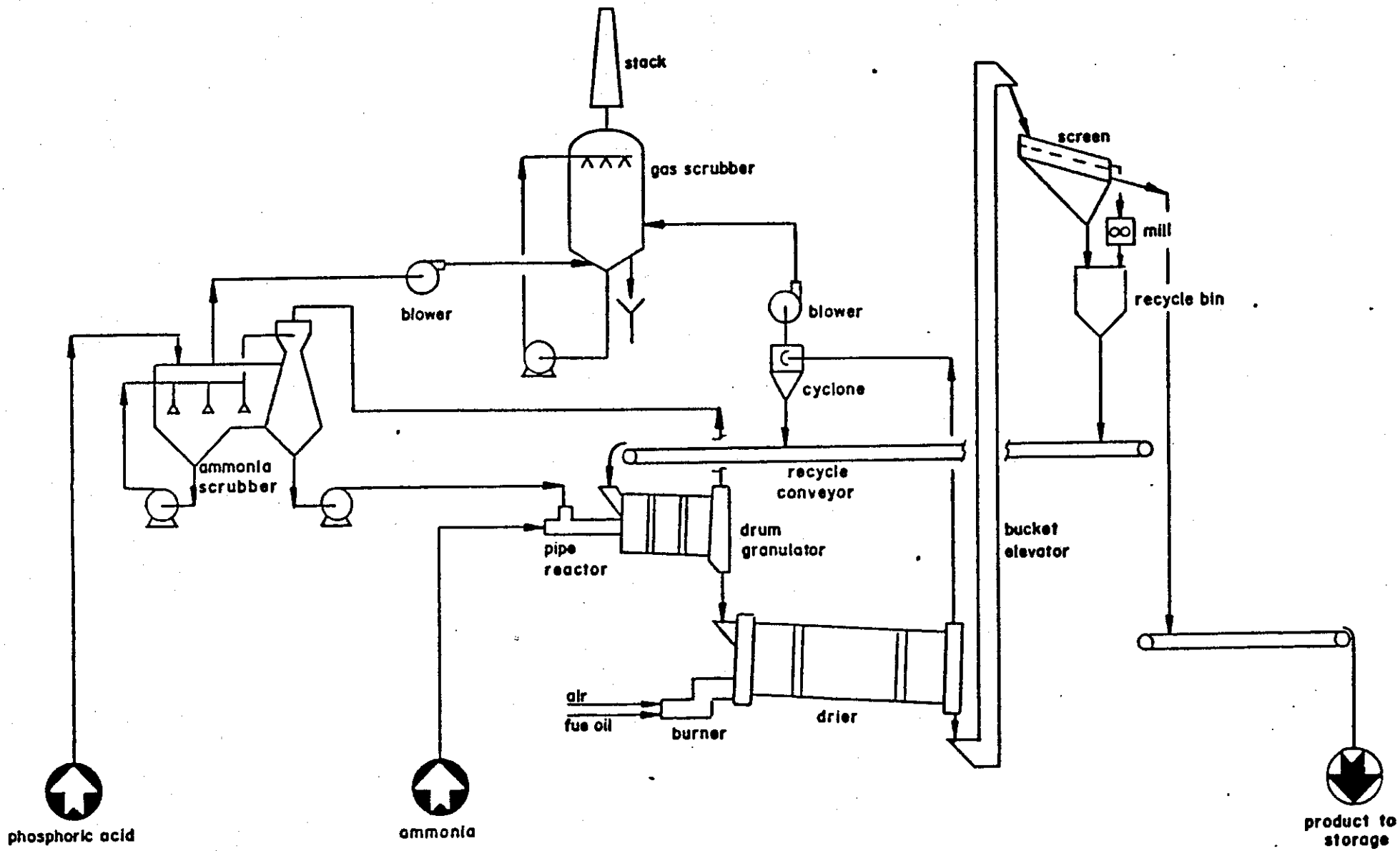

Fig. 3

The ERT – ESPINDESA Low Recycle DAP Process.