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A NEW WAY TO PRODUCE UREA-SUPERPHOSPHATE FERTILIZERS THE AZF U.S.P. PROCESS

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RESUME

La GRANDE PAROISSE S.A. est l'une des principales sociétés d'engrais en Europe de l'Ouest. Le Groupe ARMINES/TRANSVALOR est l'opérateur de transfert et de recherche de technologie pour l'ÉCOLE DES MINES DE PARIS qui est l'une des plus fameuses grandes écoles françaises d'ingénieurs. Ils ont ensemble mis au point et breveté dans le monde entier un nouveau procédé de production d'engrais à base d'urée et de superphosphate appelé USP.

Le superphosphate et l'urée sont tous deux des engrais bien connus et ils sont fabriqués dans le monde entier. L'urée et le superphosphate ne peuvent être mélangés ensemble pour des raisons de qualité du produit fini car leur mélange devient humide, collant ou prend en masse. Ceci veut dire que d'autres sources de P_2O_5 comme MAP ou DAP doivent être mélangées à l'urée ; l'emploi de ce P_2O_5 est plus onéreux car la fabrication de ces produits implique des coûts élevés en capitaux (voie acide phosphorique).

Ce nouveau procédé AZF USP offre la possibilité d'obtenir directement à la sortie de l'unité un engrais NP pulvérulent à base d'urée de titre 20-10-0 avec une solubilité élevée du P_2O_5 dans l'eau et une très courte période de mûrissement. Les matières premières exigées sont l'urée, le phosphate naturel et l'acide sulfurique. Si l'on désire un USP granulé, alors le produit est granulé ; d'autres matières premières comme le chlorure de potassium (MOP) et même MAP ou DAP peuvent être ajoutées pendant le processus de granulation pour produire des formules comme le 14-14-14, 14-7-14, 8-16-26, etc...



PRESENT SITUATION

Urea is the most used nitrogen fertilizer in the world, the consumption has reached 35 million tons N in 1993. So the production is rapidly growing mostly in the areas where natural gas is available.

Combining nutrients, specially N and P, offers many advantages: reduction of distribution costs, homogeneous, simultaneous and uniform application in the fields, etc.

There would be a good advantage to combine urea with the most common well known and cheapest P_2O_5 source: the superphosphates but unfortunately mixing urea and superphosphates can give rise to problems.

Manufacturing NP or NPK fertilizers directly from urea and a large quantity of superphosphates is very difficult. The hydrated salts release bound water during granulation, producing an excess of liquid phase. So the recycle ratios are quite high, the energy consumption and air flow rate required for drying are high too. The quality of the product is not perfect. There is much risk of caking.

Blending urea and SSP has been described and the mixtures give some troubles too. Agglomeration, lumping, can be observed after several weeks of storage. Furthermore, the physical properties of the two materials are so different that segregation can occur when spreading them on the fields. This non-uniform application directly affects crop yields.

These difficulties have led fertilizer producers to use other P_2O_5 sources as MAP or DAP to be blended with urea. But, of course, this P_2O_5 is more expensive (huge capital investment, associated gypsum stacking treatment).

Moreover the phosphoric acid route implies that calcium and sulphur are discarded in phosphogypsum. A supplemental fertilization can be required to avoid deficiencies.

There are many locations where consumption patterns and logistics are not in favour of the large-scale MAP or DAP plants which are needed to justify the cost of making phosphoric acid.

GRANDE PAROISSE AZF together with ECOLE DES MINES DE PARIS has developed a method of making a fertilizer: USP (20-10-0) Urea super phosphate, incorporating urea, sulphuric acid and phosphate rock. The USP process makes compatible raw materials which were not previously.

This product with most of its P_2O_5 water soluble can be granulated alone or together with other fertilizing materials such as potassium salts (MOP), MAP, DAP, etc.

In the late 1980's the research laboratory of the Ecole des Mines de Paris, higher school for mining engineers, initiated a programme to minimize energy consumption in fertilizer production. In 1990, a cooperation agreement was concluded between this research center and GRANDE PAROISSE, which is a company well known for worldwide licensing and is the fertilizer arm of ELF ATOCHEM the chemical division of ELF AQUITAINE.

From the earliest tests, this cooperation successively produced: the establishment of the reactions at lab scale, the identification of the reaction products, the construction and operation of a pilot plant, the industrial manufacture of powdered and granular product in a GRANDE PAROISSE factory, the drawing up of a process book. Meanwhile crop trials on maize and rice were undertaken.

This work of research and development lasted three years and it allowed us to file an application for a common patent. It was sponsored by the French Agence de l'Environnement et de la Maitrise de l'Energie (ADEME) (Environment and Energy Saving Agency).

1. DEVELOPMENT OF THE USP PROCESS

1.1. Labscale tests

The studies carried out in the ARMINES (Ecole des Mines de Paris) and GRANDE PAROISSE laboratories enabled us to increase our knowledge of the chemical reactions involved and of the optimum operating conditions, and finally to identify the reaction products and determine their physicochemical properties.

Dalman [1] reported that urea and sulfuric acid form complexes: $[CO(NH_2)_2 \cdot H_2SO_4]$ and $\{[CO(NH_2)_2]_2 \cdot H_2SO_4\}$; there are two eutectics E1 and E2 (Figure 1) which respectively correspond to: 3.6 moles of urea to 1 mole of acid and 1.8 moles of urea to 1 mole of acid. While the melting point of urea is $132.7^\circ C$, both eutectics have a melting point of about $10^\circ C$.

The industrial processes for manufacturing fertilizers always have to deal with the management of water: i.e., in the case of USP, urea moisture, sulfuric acid dilution water, process water to be recycled from the gas scrubbing unit. Therefore, we did not study the binary diagram but we studied the ternary diagram: urea, sulfuric acid, water. The addition of water makes it possible to stabilize the mixture and avoid its decay during long storage at ambient temperature.

The preparation of the mixtures of urea, sulfuric acid and water according to the mole ratios 3.6:1:1 and 1.8:1:1 is exothermic in both cases. Evolution of heat with the first ratio is lower than with the second one. We have chosen the mole ratio 3.6:1:1 which enables us to prepare a reagent under stable and reliable conditions at an equilibrium temperature of $60-70^\circ C$. This level of temperature offers some interest to acidulate phosphate rock while requiring only usual, not sophisticated equipment and materials of construction such as 316 L stainless steel and polypropylene.

Reagent 3.6:1:1 is therefore a homogeneous liquid, stable for a long time within the temperature range of 30 to 50°C; and it can be pumped easily.

It must be noticed that we studied the stability and properties of this reagent with varying contents of each component and as far as the latter could vary in industrial plants. These variations had no effects on the reagent quality.

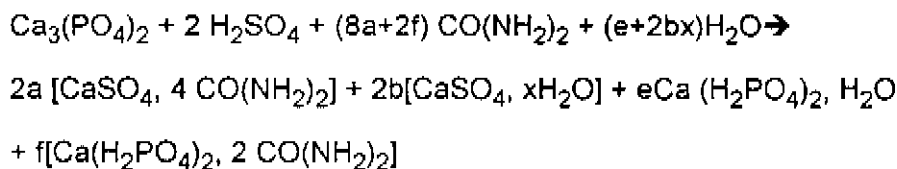
1.1.1. The acidulation of phosphate rock

The chemical reactions involved in the single superphosphate manufacture have been plentifully described. We will mention only the main reaction:



where x ranges from 0 to 2.

Different proportions of anhydrite, hemihydrate and dihydrate will be formed depending on operating conditions. In the manufacture of USP the reaction of the acidulation of phosphate rock with sulfuric acid may be written as follows:



with $a+b = 1$, and $e+f = 1$.

It will be noticed that urea is associated with calcium sulfate in place of water of hydration [2]. But the sulfuric acid to rock ratio has not changed.

To carry out our tests we applied the well known, time tested methodology used for superphosphates. It is based on the following principle [3]:

A molar ratio of 2.8 moles of sulfuric acid to 1 mole of rock P_2O_5 ensures the solubilization of more than 95% of the rock P_2O_5 . This ratio has to be adjusted as it depends on the specific impurities contained in the phosphate rock.

For a given reagent to rock ratio both materials are simultaneously fed into an agitating vessel. After 30 seconds the agitator is shutdown and the reaction is allowed to continue for 1 hour at a controlled temperature. Then USP is removed from the vessel. During the course of the test are recorded: the variation of temperature, the time when the mixture is no longer fluid, the time when the mixture sets and finally the time when the product can be handled. At these different steps, samples are taken to determine water soluble P_2O_5 , citrate soluble P_2O_5 and free acid contents.

The acidulation is promoted by the temperature of the reagent.

Therefore it will be advantageous to prepare the reagent and carry out the acidulation simultaneously. Several phosphate rocks coming from deposits located in PR of China, Israel, Morocco, Syria have been tested so far.

1.1.2. Identification of the Reaction Products

Samples of products have been submitted to X-ray analysis, and we have thus found (Figure 2):

- there is no more free urea
- there is a substantial amount of tetra urea calcium sulfate.
- P_2O_5 as monocalcium phosphate may be linked to 2 ureas.

1.1.3. Conclusions from the Laboratory Studies

These studies have enabled us to optimize the sulfo-urea reagent. Its preparation and storage conditions have been specified. We have developed a method to predict and determine the acidulating conditions of phosphate rocks. This method is derived from the conventional method used for superphosphates. Lastly the main reaction products have been identified.

As a result we could then undertake the production of USP in a pilot plant.

1.2. Production in a Pilot Plant

The trials include four steps:

- Production of the reagent,
- Production of powdered USP,
- Granulation of USP,
- Granulation of NPK grades in which USP is the principal raw material.

1.2.1. Production of the reagent

The production of the 3.6:1:1 reagent was carried out in compliance with the procedure developed at lab scale. The working capacity of the reactor was 55 litres. It was confirmed there was a thermal equilibrium at 70°C. The reactor enabled us to specify the agitator which ensured the dissolution of urea prills without previous crushing.

1.2.2. Production of powdered USP

The pilot unit for the acidulation of rock was composed of a horizontal mixer which was fed with ground rock and urea/sulfuric acid reagent; the slurry-like mixture discharged onto a 4.5 metre long curing belt where residence time could vary from 25 to 40 minutes. The production capacity was 100 kg/h USP. These pilot trials allowed us to become well acquainted with the residence times necessary for the different steps of the reaction, and to produce the powdered USP needed for the granulation pilot trials.

1.2.3. Granulation of USP

** Description of the granulation pilot plant:*

The granulation loop is composed of:

- a granulator: 1 metre diameter by 1.5 metre long
- a dryer: 1 metre diameter by 6.0 metre long
- a cooler: 0.7 metre diameter by 2.0 metre long
- a screen, a crusher, a gas scrubbing unit for the granulator and dryer exhaust gases
- 5 solids storage bins with the corresponding weigh feeders
- pipe reactors in the granulator and dryer (the neutralization of phosphoric and sulfuric acids with ammonia is possible)

** Granulation trials:*

In order to reduce USP to the usual feedable condition required for SSP and TSP, the lumps contained in powdered USP were coarsely pulverized so that 100% of the material passed through 2 millimetres.

The granulation was easy. Adding ammonia at the rate of 15 kilogrammes per tonne of USP and sparging steam beneath the granulator rolling bed of material ensured an output of 500 kg/h with a recycle ratio of 2.5:1. The material temperature in the granulation loop was approximately 80°C. The product characteristics were very satisfactory.

1.2.4. Granulation of USP-Based NPK Grades

USP was introduced into the formulation of NP or NPK grades. (Using nitrated salts such as ammonium nitrate is the only inadvisable thing to do in this case). In this way, we produced 14-14-14, 14-07-14, 9-17-24, 8-18-26, 8-15-22 grades.

The USP proportion varied from 15 to 50% according to grades. The suitability of the formulations for granulation was proved. Recycle ratios ranged from 2.5 to 4 depending on how critical the solid phase was. The other raw materials used were ammonia, phosphoric acid, potassium chloride. Various fillers were tested: sand, gypsum.

1.3. Plant Scale Production

All the tests and studies that had been carried out led us to think about an industrial production unit. As the USP manufacturing process was very similar to that of superphosphates, we decided to adapt the SSP/TSP plant in our Bordeaux factory to it.

This superphosphate plant had been designed for a production capacity of 700 t/d according to the AZF superphosphate process [4] and had been consequently equipped with a 2.73 metre diameter by 8 metre long drum den. We installed a 12 m³, 316 L stainless steel agitating tank ahead of the den. It was fed with 94% w/w sulfuric acid and urea (500 kilogramme bags of urea were emptied into it). Operating conditions were the same as those of the pilot-plant. The reagent was pumped to the drum den mixer to which the phosphate rock was fed. The residence time was about 1 hour. No evolution of gas was detected and this observation was confirmed by fluorine determinations. The fluorine contained in rock was entirely recovered in the powdered product. The production rate was limited to 15 t/h by the feed of solid urea and the capacity of the pump delivering the reagent to the mixer.

This production confirmed to us how the process was simple and the plant easy to operate.

After delumping, powdered USP was fed to a granulation loop of 120 tonne working capacity and comprising a 3 metre diameter by 6 metre long granulator and a 3 metre diameter by 31.5 metre long dryer. Moreover, the granulation plant was already fitted with the equipment required by the AZF dual pipe reactor process.

The results obtained at plant scale are:

Produced Grade:	8 - 18 - 26 (Zn, Cu)	
Raw Materials:		
powdered USP (20-10-0)	200	kg per tonne
phosphoric acid (P ₂ O ₅)	160	kg per tonne
sulphuric acid	18.6	kg per tonne
ammonia	48.6	kg per tonne
potassium chloride	434	kg per tonne
Filler (with Zn, Cu)	43.8	kg per tonne
molar ratio	1.05	
granulator material temperature	80°C	
dryer material temperature	65°C	
Production rate	30 T/h	
recycle ratio	3:1	
Product Quality		
Bulk density	1000 kg/m ³	
size distribution	d50 : 3.3 mm between 2.5 - 4.5 mm > 90%	
hardness	> 30 N	
<u>Conclusions</u>		
<ul style="list-style-type: none"> - granulation similar to that of the same grade without USP. - adding micronutrients during granulation causes no problem. - product of good quality. 		

2. DESCRIPTION OF THE INDUSTRIAL PROCESS

The industrial process can be divided into three steps:

- Preparation of the acidulating reagent
- Acidulation of phosphate rock with the reagent
- USP granulation only if required.

2.1. Preparation of the Reagent (Figure 3)

Urea in solid form, e.g. prilled or granulated urea, flows from a supply hopper and is delivered to a weigh feeder.

Sulfuric acid is pumped from its storage tank to the mixing tank.

Sulfuric acid is fed in proportion to the urea flow.

Some process water is added in proportion to the preceding flows, in order to achieve the mole ratios 3.6:1:1, i.e. 3.6 moles of urea to 1 mole of sulfuric acid and 1 mole of water.

The agitator of the mixing tank ensures the dispersion and dissolution of urea while maintaining uniform concentration and temperature throughout the mixture.

A coil which is fed with cooling water makes it possible to regulate the reaction temperature. The same coil when fed with hot water (condensate) makes it possible to reach normal operating conditions more quickly during the start-up operations.

The reaction temperature is stabilized between 60 and 70°C.

2.2. Acidulation of Phosphate Rock with the Reagent **(Figures 4 and 5)**

2.2.1. Mixer

Rock has to be ground to 80% through 80 micron mesh.

Ground rock flows from a storage bin and is delivered to a weigh feeder.

The reagent is continuously pumped from the mixing tank at a controlled flowrate which is determined and regulated in proportion to the rock flow.

The rock and reagent are fed into a horizontal paddle mixer. The orientation of its paddles makes it possible to adjust the residence time depending on the rock reactivity. The mixer outlet discharges the material directly into the drum den.

2.2.2. Drum Den

The drum den is a rotating, slightly inclined cylinder whose rotational speed can be adjusted (from 0.5 to 1.5 rpm). The material residence time depends on the processed phosphate rock.

The drum den is rubber-lined, which avoids the formation of material build-up. It will be noticed that no internal scraping device is required.

A fan sucks the mixer and drum den off-gases and the air draft makes it possible to regulate the moisture content and temperature of the product.

2.2.3. Effluent Treatment

The drum den exhaust-gases can be scrubbed in a unit made up of a Venturi scrubber and a cyclonic column.

Gases are passed through the Venturi scrubber where the scrubbing liquor is sprayed, and then discharged to the cyclonic column where they meet a spray of scrubbing liquor which flows countercurrently.

The liquid level is kept constant in the column by feeding fresh make-up water.

The scrubbing liquor density is regulated by recycling some scrubbing liquor back to the mixing tank where the reagent is prepared.

One unit or two with a Venturi scrubber and a cyclonic column apiece will be installed, depending on how prone to fluorine release the phosphate rock is.

2.2.4. Powdered USP

The product flowing from the drum den discharge end is conveyed to storage. It can be reclaimed as a powdered fertilizer, or granulated as 20-10-0, or used as a raw material in the production of NP and NPK fertilizers.

2.3. USP Granulation

Powdered USP can be granulated alone or together with other raw materials (KCl, K_2SO_4 , DAP, MAP, $(NH_4)_2SO_4$).

The granulation process is performed in a conventional granulation loop. Powdered USP is delivered to the granulator through the solid raw material feeding system. Steam injection supplies supplemental heat and a small feed of ammonia makes it possible to optimize the product quality.

In the dryer, the granules are dried by a stream of hot air flowing co-currently. The discharge from the dryer (which contains less than 1% moisture) is screened. The oversize is crushed and returned to the granulator together with fines. The on-size product (2-4 mm) is cooled, coated and conveyed to storage.

Other raw materials are delivered to the granulator (potassium sulfate or chloride, MAP, DAP) or available acids (phosphoric or sulfuric acid) can be neutralized with ammonia in a pipe reactor.

The dryer exhaust gases are dedusted in cyclones. Then the dryer and granulator exhaust gases are scrubbed in a gas scrubbing unit made up of Venturi scrubbers, a separator tank and a cyclonic column. That is the description of a conventional granulation unit where granular NPs, NPKs, TSP or SSP can be produced.

3. QUALITIES OF THE PRODUCT: 20-10-0 USP

3.1. Chemical Composition

USP simultaneously supplies urea nitrogen, phosphate with 95% of the citrate soluble phosphate being soluble in water, sulfur and calcium. A typical analysis of the granulated product is as follows:

Total nitrogen	20.90%
Urea nitrogen	19.30%
Ammoniacal nitrogen	1.60 %
Total P_2O_5	10.20%
Citrate soluble P_2O_5	9.70%
Water soluble P_2O_5	9.20%
SO_3	16.10%
H_2O	1.00%

During the granulation process it is possible to associate USP with other fertilizer nutrients and micronutrients.

3.2. Suitability to storage

USP can be used as produced, i.e. in pulverised form, or as a granular material. In this case, its physical properties are quite similar to those of urea-based NP and NPK grades:

The product can be easily stored. Its critical relative humidity (that humidity of the atmosphere above which the product will spontaneously absorb moisture and below which it will dry) is 65-70% at 20°C and consequently the product can be stored in bulk too.

3.3. Agronomic Interest

In order to know whether this new fertilizer, with the very special position of urea in the molecular structure, has the same agronomic properties as those typical of traditional fertilization, we have carried out agronomic tests with two crops: maize and rice.

3.3.1. Maize fertilization (Figure 6)

USP was tested in comparison with the same quantities of the elements nitrogen and phosphate supplied by DAP and urea. In both cases the same input of potassium was supplied by potassium chloride (KCl) which contained 60% K_2O .

Tests were carried out in five different combinations of clay, silt, sand and loam soils.

An increase in yield can be observed for the optimum nitrogen inputs. Thus, for a yield of 11,500 kg/ha of maize, the fertilization rate can be reduced by 40 kg/ha of nitrogen when applying USP.

3.3.2. Rice Fertilization (Figure 7)

USP of the 20-10-0 grade was associated with 60% K_2O potassium chloride and was compared with the mixture comprising ammonium sulfate, TSP and potassium chloride. Only one type of soil was used for this comparison: a loam soil made up of clay, sand and silt.

It would be noticed that, with the same fertilization rate of 140 kg/ha nitrogen, the yield was increased by 10%. Besides, the tests showed that splitting the application of nitrogen input, which was advisable in traditional fertilization, was no longer necessary with USP.

3.3.3. Conclusions drawn from these tests

The tests have shown that USP affects maize and rice fertilization very positively. At constant yield the consumption of nitrogen can be cut by at least 10%.

4. ADVANTAGES OF THE PROCESS

4.1. Zero liquid effluents, near zero fluorine emission

Scrubbing liquor recycle is integrated into the early stage of the process: the preparation of the reagent. As a result, there are no liquid effluents. With the phosphate rocks tested, the fluorine contained in rock is entirely recovered in USP. In order to meet the most stringent regulations concerning pollution control, we have added a single-stage scrubbing unit to the process. It will be noticed that a two-stage scrubbing unit is necessary for TSP production and that SSP production may require a three- or four-stage scrubbing unit.

4.2. The Phosphoric acid route is avoided

The USP process is one of the rare processes making it possible to produce a urea-based NP fertilizer without using the phosphoric acid route. The investment cost needed for the erection of a phosphoric acid plant is always very heavy mainly if it includes phosphogypsum stacking. With USP the consumption of sulfuric acid is reduced by 33% and sulfur can provide agronomic benefits. Besides, phosphate rocks unsuitable for the production of phosphoric acid can be used as a feedstock in the USP process (rocks of low P_2O_5 content, small ore deposits which can't justify the phosphoric acid investment).

4.3. The Process is fully tested

Irrespective of the many developing tests carried out, the process, as far as rock acidulation, gas scrubbing and possibly granulation are concerned, has the same features as the time-tested superphosphate process. Therefore the equipment, process control and materials of construction are well known.

4.4. Granulation plants can be retrofitted with the USP process

Powdered USP can be used as a raw material in the manufacture of urea-based NP, NPK grades. The conventional granulation unit can then operate as usual without any modification of its equipment.

A USP unit can be erected along or in an existing granulation plant where powdered USP will be either granulated alone or used as a raw material in urea-based NP-NPK grades.

In all cases the installation of a USP unit is highly advisable as far as ammonium nitrate is not the only nitrogen source in the granulation plant.

5. USP COST EFFICIENCY

5.1. Raw materials cost

When comparing the cost of the raw materials (urea, phosphate rock, sulfuric acid) needed for producing a 20-10-0 fertilizer with the USP process to that of the raw materials (urea, ammonium sulfate, phosphoric acid, ammonia) involved in a conventional process, the reduction of cost due to the USP process ranges from 10 to 30%:

minus 10% if most nitrogen is fed as ammoniacal nitrogen
 minus 30% if the whole of nitrogen used in the conventional process is urea nitrogen, i.e. it is very near the quantity of urea nitrogen used in the USP process.

5.2. Energy savings

The energy savings derived directly from the process are:

- the use of concentrated sulfuric acid : there is no need to dilute the acid to 70-75% as it is the case with SSP. The associated consumption of cooling water is thus avoided.
- consequently, the moisture content in the product is much lower: 3 to 4% water versus 10 to 12% in SSP production. If USP must be granulated, 40% of the drying energy is saved per unit of P_2O_5 .

5.3. The Process is simple and requires reduced capital cost

The production of powdered USP is a simple and fully tested process which requires only a limited number of pieces of equipment.

The capital cost for a 30 t/h USP plant, including rock grinding within the battery-limits, amounts approximately to US\$ 6 million on France basis.

(with rock grinding accounting for US\$ 2.5 million when required).

5.4. Easy and cheap revamping of a SSP or TSP plant

A plant which produces superphosphates with the process involving a drum den or a conveyor belt for curing [4] can be easily made suitable for the USP process. In most cases, it will only suffice to add the reagent preparing equipment to the existing plant.

6. BIBLIOGRAPHY

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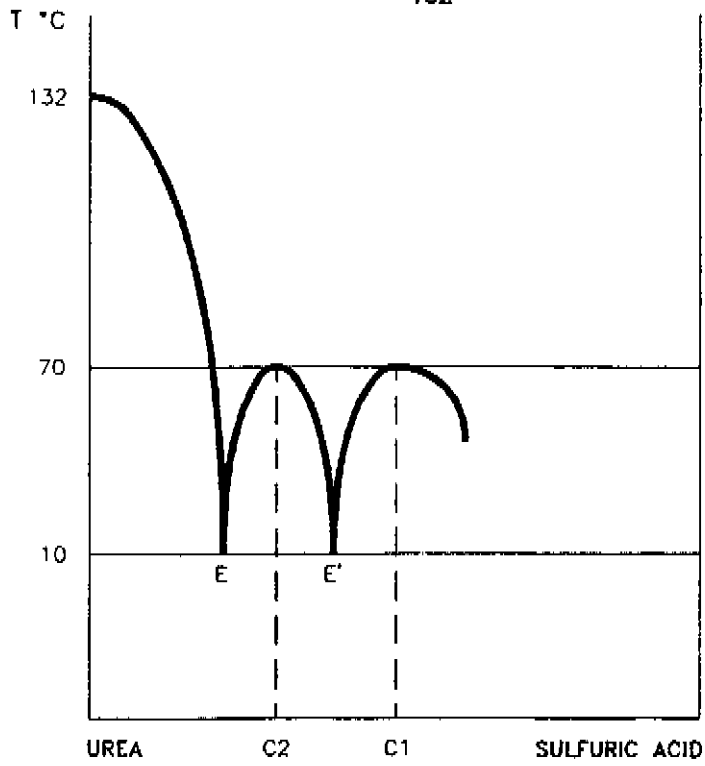


Fig. 1 : UREA/SULFURIC ACID EUTECTICS

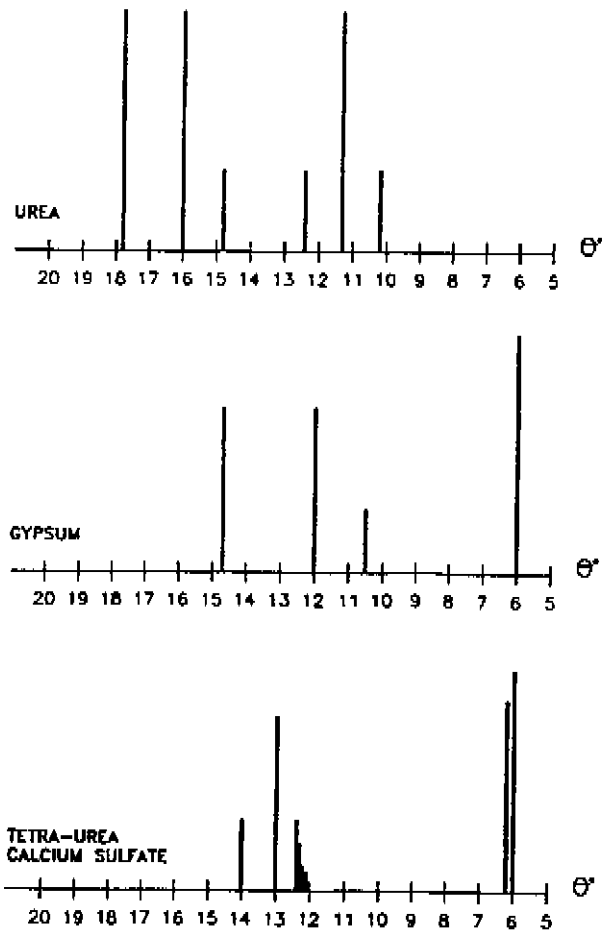


Fig. 2 : X-RAY ANALYSIS

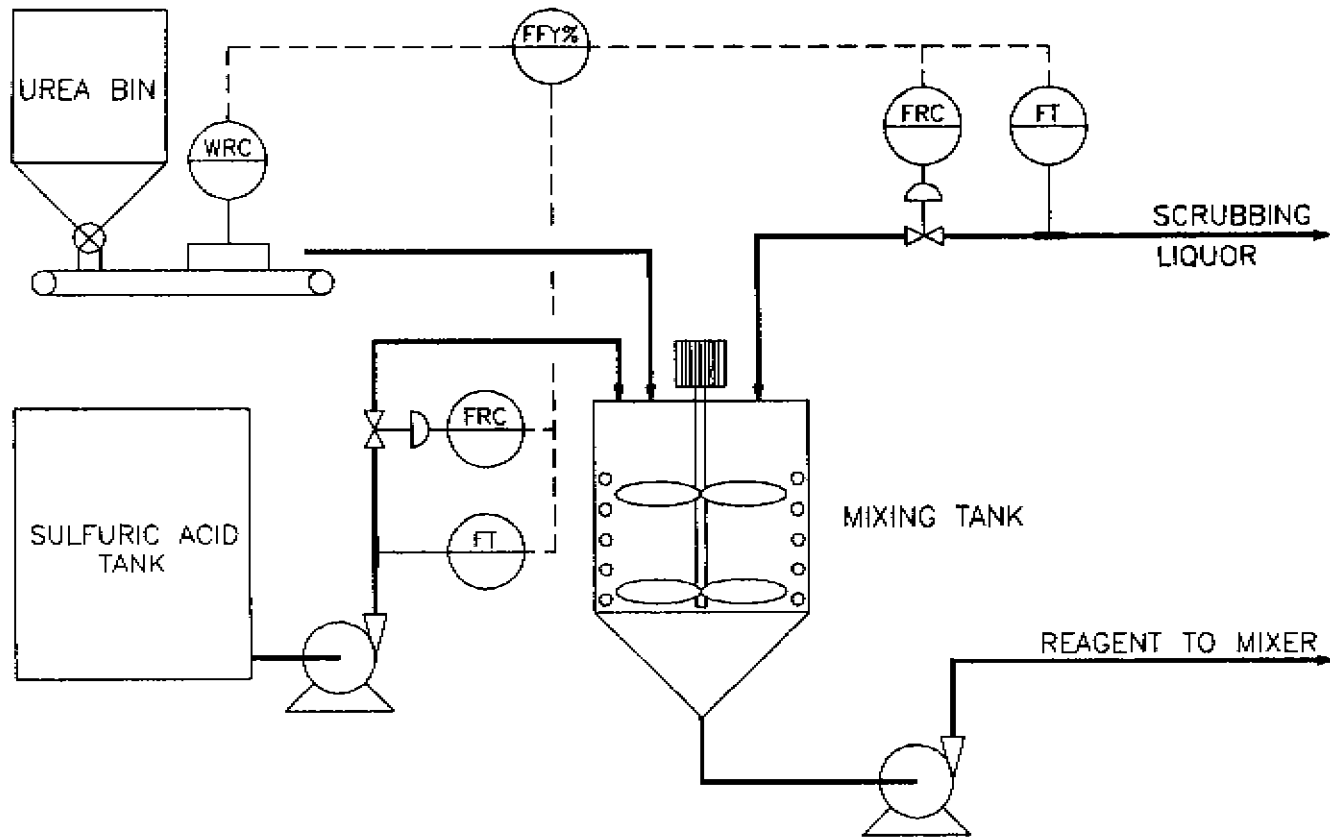


Fig. 3 : PREPARATION OF THE UREA/SULFURIC ACID REAGENT

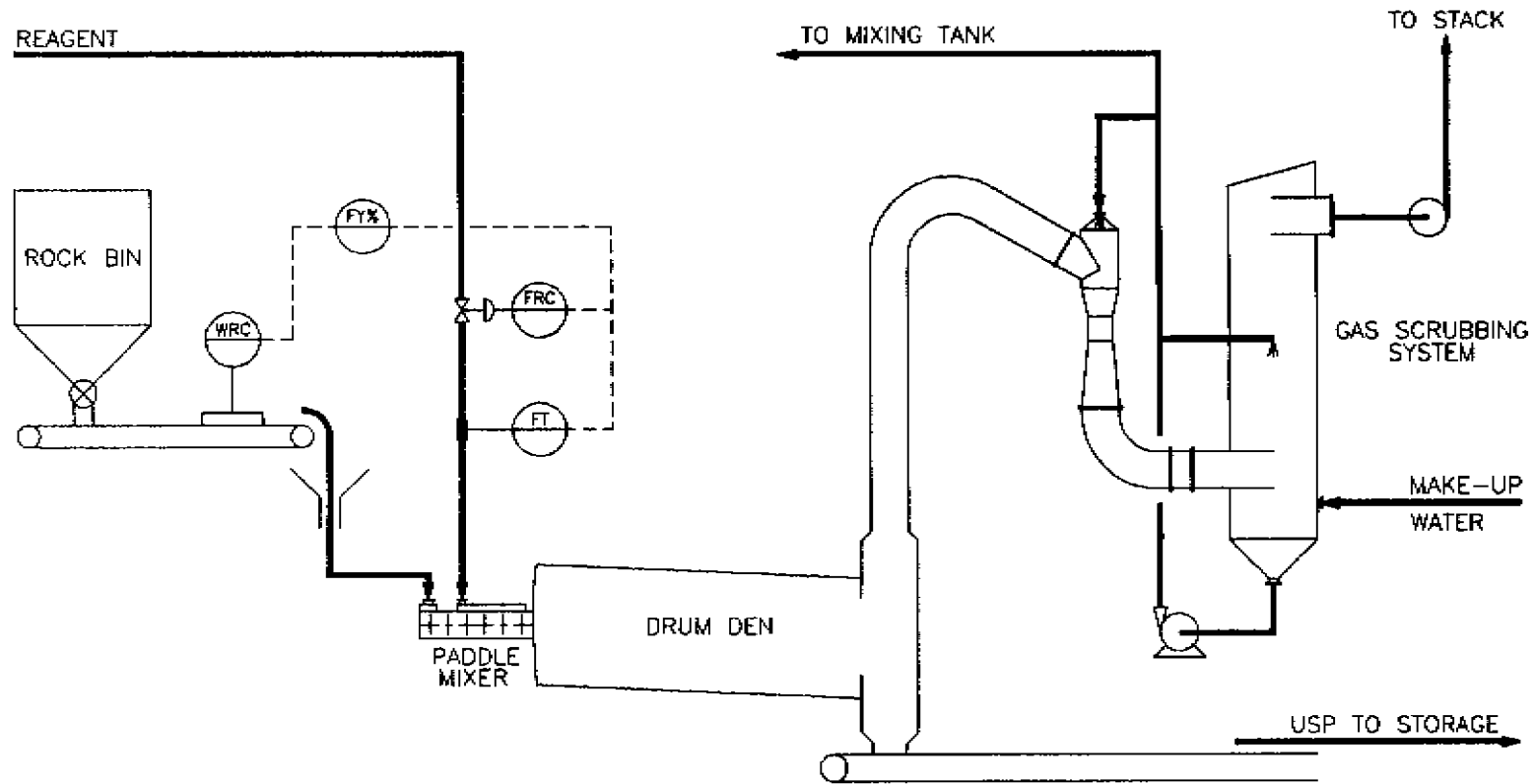


Fig. 4 : ACIDULATION OF ROCK WITH UREA/SULFURIC ACID REAGENT

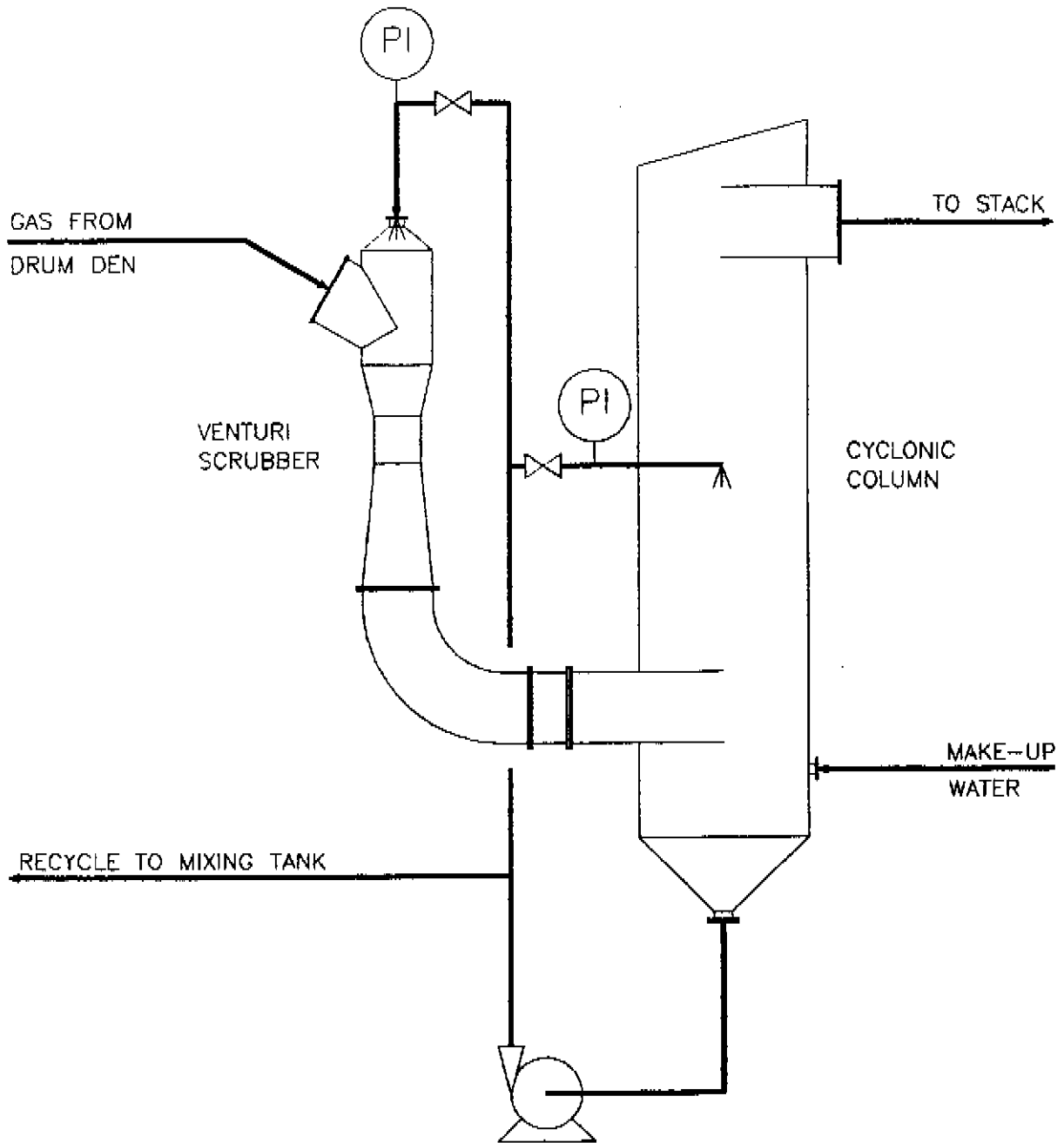


Fig. 5 : SINGLE-STAGE GAS SCRUBBING UNIT

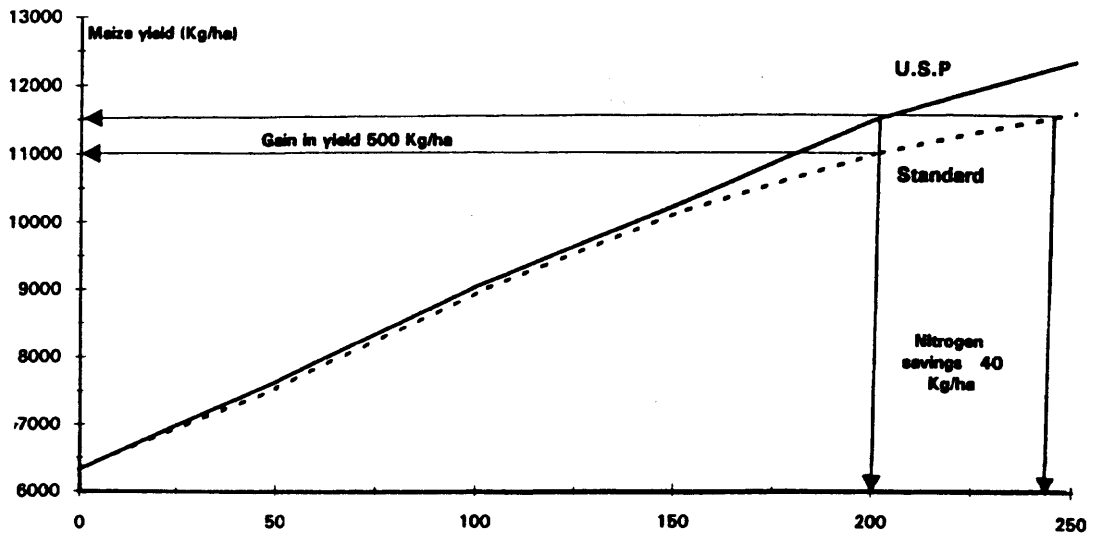


Fig.6: MAIZE FERTILIZATION TESTS ; Nitrogen applied (Kg/ha)

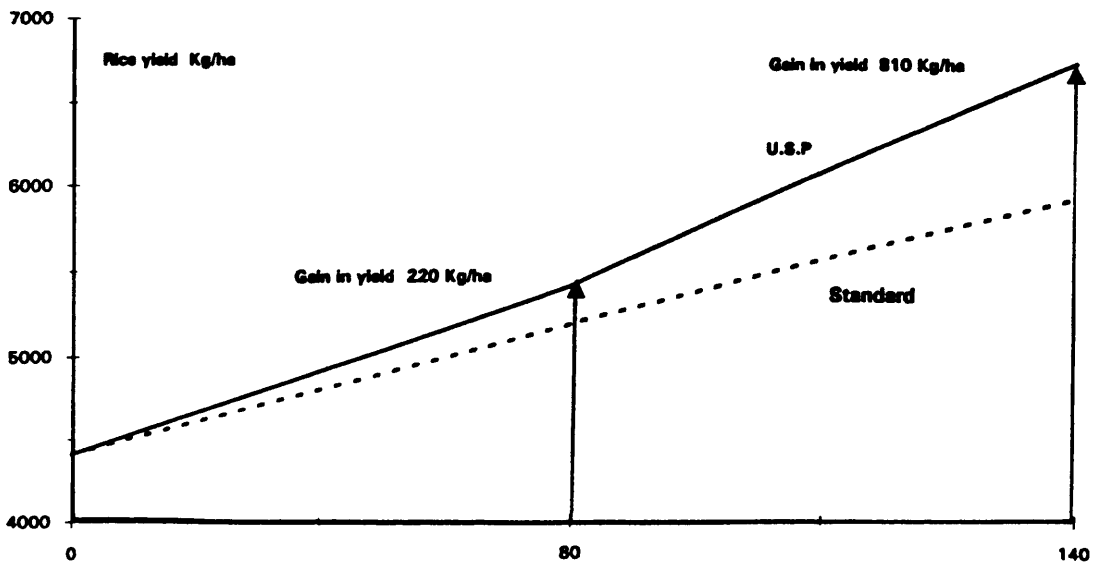


Fig.7: RICE FERTILIZATION TESTS ; Nitrogen applied (Kg/ha)