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PIPE REACTOR - AN INNOVATION FOR IMPROVEMENT OF GRANULATION PLANT PERFORMANCE

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1. INTRODUCTION

Following some earlier work by the Tennessee Valley Authority, S.A. Cros proceeded to develop its own pipe reactor technology in 1972. Pilot plant work was considered desirable so that basic parameters could be established to ensure that the product quality was as good or better than that of the product from existing slurry process and solid feed plants for which there was an established market. Because the potential advantages of the reactor were overwhelmingly evident, every effort was made to translate pilot data into commercial scale operation with minimum delay even though at times important links remained unresolved.

During the early stages of development work it became abundantly clear that the presence of pholyphosphate in the product did not offer any significant advantages. The formation of polyphosphate is due to a high rate of heat release and maintenance of high temperature in the reactor. This has to be accomplished by heat input through usually a combination of factors such as preheating the reagents, using a higher concentration of phosphoric acid, and the use of large amounts of sulphuric acid in the fertiliser formulation. Polyphosphate was also found to be slow to crystallize and it imparted plasticity to the product. Recycle requirements during granulation were high and the storage characteristics of the product not altogether satisfactory. These drawbacks were, in fact, later discovered by others (1).

A satisfactory technique for co-neutralisation of phosphoric and sulphuric acids was developed during the early work and was, soon after, translated to commercial scale production. Subsequent work covered neutralisation of sulphuric acid in the presence of urea solutions to produce special application nitrogen fertiliser containing sulphur.

More recent development work has been concerned with co-neutralisation of nitric and phosphoric acid.

The pipe reactor has been in regular operation in two of the S.A. Cros plants since 1975, to make over 300,000 tonnes annually of MAP, DAP and compound fertiliser. Care is taken to avoid the formation of polyphosphate. The heat which would otherwise be required for polymerisation is thus available for evaporation of water enabling the use of more dilute phosphoric acid. Depending on product formulation and the available concentration of feed acids (phosphoric and/or sulphuric), the reaction slurry discharged from the pipe reactor will have moisture level ranging between 1 to 4%. Other operating conditions of the reactor are as follows:-

Capacity	:	300-800 kg slurry/cm^2h
Heat Release	:	50,000-150,000 kcal/cm^2h or 165-190 kcal/kg slurry
Temperature	:	145-160^{o}C
Back Pressure	:	3.0-6.5 atmospheres

The capacity per unit cross-section is much higher and the heat generated per unit mass of slurry much lower than the corresponding figures published by other operators (2). These features result in a smooth flow pattern through the pipe reactor, free from surges and arrests, contributing to good granulation. The reactor discharges directly to the granulator. The distance travelled by the slurry is cut down to a minimum and the design and arrangement of other granulator accessories is such that the viscosity of the ammonium phosphate slurry is not critical.

2. PRODUCTION DATA

The following data relates to S.A. Cros plants equipped with a pipe reactor. They are in respect of well established industrial scale operation where economy in production is the prime objective.

2.1 MAP AND DAP

The NH_3 : H_3PO_4 molar ratio being around 1:1 for the production of MAP, ammonia loss from the reactor-granulation system is negligible.

Scrubbing of the granulator exhaust gases is thus unnecessary during normal operation. However, since the S.A. Cros plants are multi-product plants, as indeed most granulation plants are likely to be, they feature a granulator scrubber.

In diammonium phosphate production the molar ratio in the reactor is controlled at around 1.4. Additional ammoniation is carried out in the granulation drum where the molar ratio is boosted to above 1.8. Liquid ammonia is used and this is introduced through a plough-share device specially designed for the purpose by S.A. Cros. Ammonia escaping from the reactor-granulator system is recovered in the granulator scrubber using sulphuric acid (and/or phosphoric acid) and the recovered solution is sent back to the pipe-reactor.

In either case, MAP or DAP production, hot air drying of the product is not necessary. The products have good physical characteristics with moisture levels of about 2.5% for MAP and 1.5% for DAP. Whilst producing diammonium phosphate, Cros use the existing rotary drier as a counter-current recycle cooler. There is no fuel requirement for drying either product.

The products are hard with uniform and well-rounded granules. They remain free flowing after prolonged storage, and anti-caking treatment has not been found necessary.

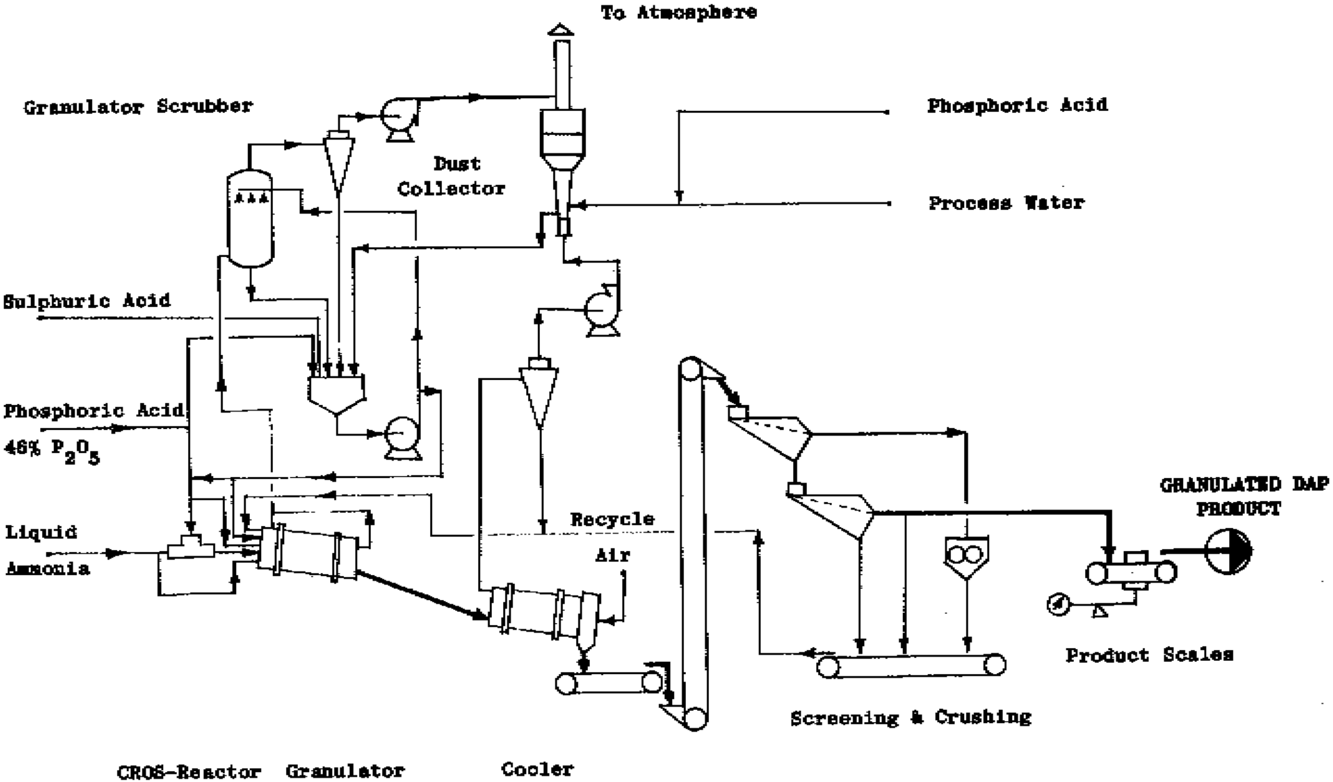
Table 1 below outlines operating conditions for MAP and DAP production. 11-52-0 is the standard analysis for MAP in Spain. This requires the use of a filler.

2.2 HIGH ANALYSIS COMPOUND FERTILISER

Table 2 gives operating data in respect of 20-10-10 and 17-17-17 which are the highest analysis 2:1:1 and 1:1:1 nutrient ratio compound fertiliser produced and marketed by S.A. Cros. The Cros pipe reactor process is capable of producing higher analysis 24-12-12 and 19-19-19. However, the company has established market for 20-10-10 and 17-17-17 and a very large part of its production consists of these two grades.

TABLE 1

	MAP (11-52-0)	DAP (18-46-0)
Pipe Reactor		
Ammonia (5°C) Kg.p.h. (liquid)	2274	2702
Phosphoric acid Kg.p.h. (50% P_2O_5)	21050	10700
Liquid from Scrubber. Kg.p.h.	-	8750
Slurry composition		
%N	11.2	12.6
%P_2O_5	54.0	47.0
%H_2O	4.0	4.5
Molar ratio (calc)	1.1	1.38
Temperature °C	145	156
Granulator Scrubber		
Sulphuric acid 98%. Kg.p.h.	-	1200
Phosphoric acid (52% P_2O_5) Kg.p.h.	-	6000
Water Kg.p.h.	-	1400
Granulator		
Ammonia (liquid) Kg.p.h.	-	1404
Inert (gypsum) Kg.p.h.	154	-
Recycle ratio. t/t product	1.4	3.5
Granulated product:		
Temperature °C	95	88-92
Moisture %	3.0	2.0
Product		
Rate t.p.h.	20	18.5
%N	11.1	17.7
%P_2O_5	52.1	46.3
%H_2O	2.5	1.5



S. A. CROS PROCESS FOR GRANULATED DAP

TABLE 1 (cont'd)

Screen analysis.%

+ 4 mm.	5	3
- 4 mm. + 1.5 mm.	93	94
- 1.5 mm	2	3

2.2 HIGH ANALYSIS COMPOUND FERTILISER (contd)

20-10-10 is produced by co-neutralisation of phosphoric and sulphuric acid. The large amount of heat release in the reactor permits the use of weeker acids. The overall water balance is adjusted to ensure that polyphosphate is not produced. This, coupled with the fact that the ammonium phosphate-sulphate slurry made in the reactor is highly concentrated, allows operation with the very low recycle ratio which is a feature of the Cros process. The reduction in water inventory means that no extraordinary problems are encountered even when comparatively high proportions of urea are used in any particular formulation.

Fuel consumption, at 40,400 k-cal per tonne of 20-10-10 and 30,000 K-cal per tonne of 17-17-17, is low and this considerable reduction in heat demand makes low temperature drying possible. Ammonia escaping from the pipe reactor/granulator system is recovered by absorption in sulphuric acid in the granulator scrubber. The scrubber solution is returned to the granulator.

It is worthy of note that production rates of 45 to 50 tonnes per hour are obtained from a 1½" diameter Cros reactor in the case of 20-10-10 and 2½" diameter reactor in the case of 17-17-17.

2.3 CO-NEUTRALISATION OF PHOSPHORIC ACID AND SULPHURIC ACIDS

S.A. Cros produce up to 20 tonnes per hour of 15-40-0 in a 2½" diameter pipe cross reactor.

All reactant raw materials are fed to the reactor and a concentrated ammonium phosphate-sulphate slurry is produced using comparatively dilute phosphoric acid (42% P_2O_5). The heat of neutralisation is used to best advantage in formulations such as 15-40-0 which, in spite of carrying a high P_2O_5 content, need no fuel for drying. Good granulation is achieved with a recycle ratio of about 1.4:1.

TABLE 2

		20-10-10	17-17-17
Pipe reactor			
Phosphoric acid, Kg.p.h.			
45% P_2O_5		-	14381
40% P_2O_5		5886	-
Ammonia (5°C) (liquid)	Kg.p.h.	1205	2256
Sulphuric acid (80%)	Kg.p.h.	1440	-
Slurry (calc)	Kg.p.h.	6020	12347
%H_2O		2.0	2.9
%N		16.1	14.4
%P_2O_5		38.8	52.4
Temperature °C		156	152
Granulator			
Ammonia (liq)	Kg.p.h.	1940	865
Urea (46%N)	Kg.p.h.	15350	11154
Sulphuric acid (80%)	Kg.p.h.	4050	1023
SS-18	Kg.p.h.	13860	6948
KCl (60% K_2O)	Kg.p.h.	8084	12736
Recycle ratio t/t product		1.5	1.2
Temperature °C		44	52
Granulated product			
% H_2O		2.5	2.2
temperature °C		89	84
Product			
%N		19.7	16.8
%P_2O_5		10.1	17.05
%K_2O		10.2	16.8
%H_2O		0.8	1.1
t.p.h.		48	45

TABLE 2 (contd')

Product Screen Analysis

+ 4 mm.	3.1	2.2
- 4 mm. + 1.5 mm.	94.4	95.2
- 1.5 mm.	2.5	2.6

Drying

Fuel consumption (K-cal/t)	40,400	30,000

2.3 CO-NEUTRALISATION OF PHOSPHORIC ACID AND SULPHURIC ACIDS (cont'd)

The dryer is used as a cooler, and this operation generally results in some drying. The granulator scrubber is in operation in order to recover ammonia escaping from the reactor-granulation system. The scrubber uses acids for this purpose and these acids are ultimately fed to the reactor.

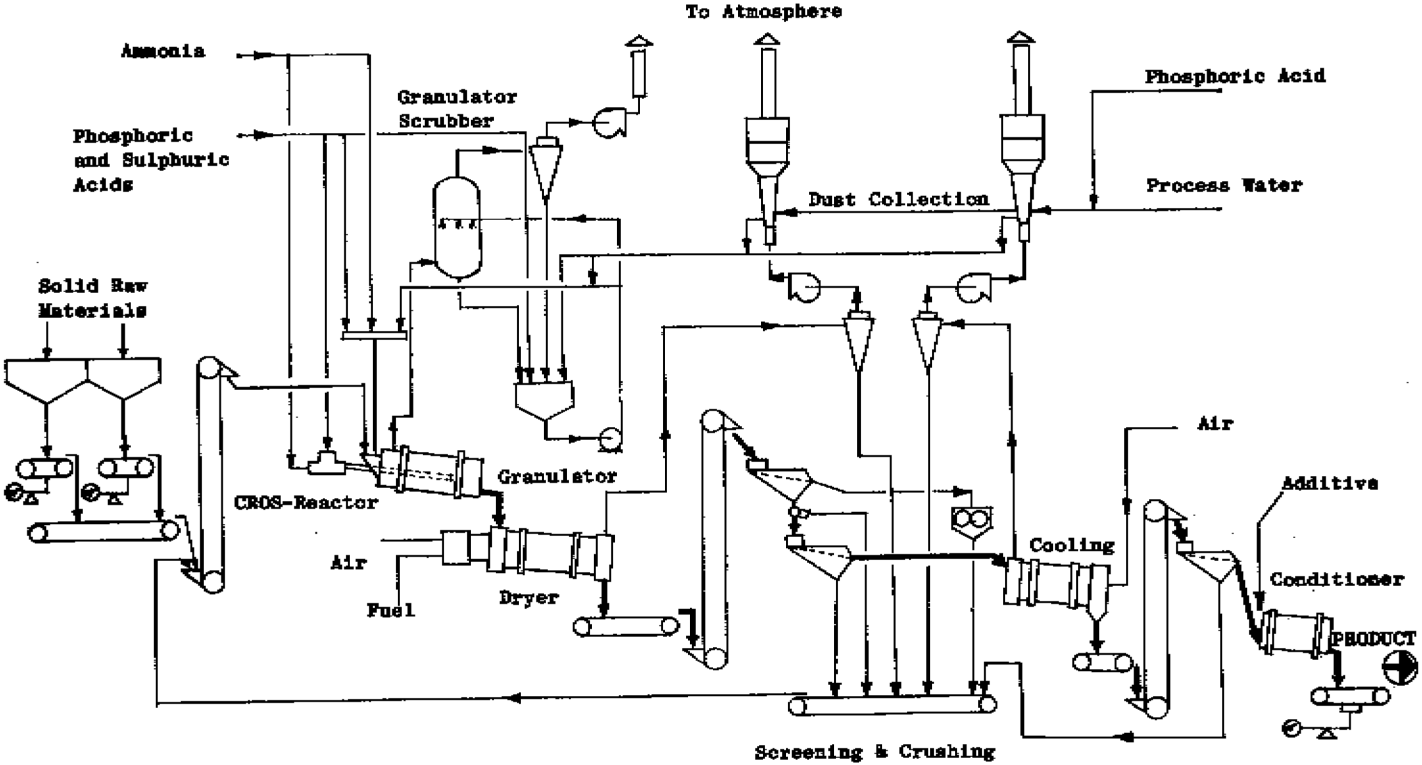
The 15-40-0 product usually has a moisture content of between 1.0-1.2%. It has excellent physical characteristics and stores well. No anti-caking treatment is required. Along with these attributes the product has high analysis and it contains a significant amount of sulphate. It is, therefore, a very useful material for bulk blending.

Table 3 below gives the operating conditions for the binary 15-40-0 fertiliser.

3. PRODUCTION COST SUMMARY

3.1 Compound Fertiliser

S.A. Cros have developed computer programmes which print out optimum formulation for any particular grade, taking into consideration the cost of raw materials and a required level of heat release in the Cros reactor. Table 4 below gives a comparison of the cost of producing the popular 20-10-10 and 17-17-17 from both solid MAP and wet process phosphoric acid. The cost unit used is the cost of 1 kg. of ammonia and all other costs are translated into this unit. Raw material costs ruling in Spain during the closing months of 1977 are taken into account.



S. A. CROS GRANULAR COMPOUND FERTILISER PROCESS

TABLE 3

	15-40-0
Pipe Reactor	
Ammonia (5°C) Kg.p.h. (liquid)	3740
Phosphoric acid Kg.p.h. (42% P_2O_5)	19033
Sulphuric acid Kg.p.h. (98%)	3907
Slurry Kg.p.h.	20362
% N	14.8
% P_2O_5	39.7
% H_2O	1.7
Temperature °C	157
Granulator	
Recycle t/t product	1.4
Temperature °C	51
Granulated product:	
% H_2O	1.5
Temperature °C	95
Production	
Product Rate t.p.h.	20
% N	14.8
% P_2O_5	39.8
% H_2O	1.2
Screen analysis - 4 mm + 1.5 mm %	96
Drying	
Fuel consumption	Nil

It was established that the use of the Cros type pipe reactor makes fertiliser production more economical when phosphoric acid is used instead of powder MAP. The savings in formulation costs is more pronounced for 17-17-17.

The use of the reactor reduces the extent of drying required and also the recycle requirements. It is estimated that the investment is about 70% of that required for a conventional slurry process plant. The Cros process, however, retains the fundamental advantages of the slurry process in its ability to use the cheaper basic raw materials.

Although less significant by comparision, the savings due to reduction in fuel oil and electrical energy consumption, and due to lower investment are nonetheless quite considerable. The saving in investments related costs is about 2.5 to 3 units (ammonia cost related) per tonne of product.

3.2 Diammonium Phosphate

The Cros pipe reactor process for DAP manufacture features ammoniation in the granulator. In basic concept it is, therefore, similar to the conventional slurry process and the efficiency in respect of raw material usage is about equal for the two processes. The introduction of the pipe reactor has, however, improved DAP production economics beyond doubt, in spite of the fact that there is no saving in formulation cost.

A conventional DAP plant uses recycle ratio of between 5:1 to 6:1 as opposed to the 3.5:1 for a plant equipped with a pipe reactor. The basic reason for this is the convetional plant's inability to produce a reaction slurry with acceptable fuidity at under 12% water content. This water requires to be removed from the product by drying, at considerable cost. The heat demand for this purpose is usually about 150,000 k-calories per tonne of DAP product. Electrical energy consumption is about 40 kWh per tonne of product for the conventional plant. The Cros reactor reduces this to about 20 kWh.

The reduction in investment is principally due to the following reasons:-

a) Substitution of an expensive preneutraliser, agitator, pumps, piping and the associated fume extraction and scrubbing equipment with a small, relatively cheap, pipe reactor designed for installation inside the drum granulator.

TABLE 4

20-10-10

	with MAP powder	with pipe-reactor	Difference	Cost Difference (NH_3 cost = 1)
		Kg / t		
Ammonia	62.5	65.3	+ 2.8	+ 2.8
Urea Prill	303.4	324	+ 20.6	+ 18.75
SS-18	246	288.8	+ 42.8	+ 16.26
MAP (10,5-51-0)	111.3	-	- 111.3	- 166.95
Phosphoric acid (40% P_2O_5)	-	122.6	+ 122.6	+ 116.1
Potassium Chloride	168.4	168.4	-	-
Sulphuric acid (98%)*	127.4	93.3	- 34.09	- 11.59
				- 24.63
Fuel oil (drying)	8.6	4.3	- 4.3	- 2.62
				- 27.25

17-17-17

	with MAP powder	with pipe-reactor	Difference	Cost Difference (NH_3 Cost = 1)
		Kg / t		
Ammonia	61.7	69.7	+ 8.	+ 8.
Urea Prill	189.9	250.3	+ 60.4	+ 54.96
SS-18	29.1	154.6	+ 125.5	+ 47.7
MAP (10.5-51.0)	326.4	-	- 326.4	- 489.6
Phosphoric acid (45% P_2O_5)	-	319.8	+ 391.8	+ 340.6
Potassium Chloride	286.2	286.2	-	-
Sulphuric acid (98%)*	118.8	18.5	- 100.3	- 34.1
				- 72.44
Fuel oil (drying)	7.6	3.6	- 4	- 2.44
				- 74.88

* In practice normally diluted to 80%. Consumption of fuel given includes the vaporization of this additional water.

(b) Elimination of the air heater for the dryer and the equipment associated with the air heater such as fuel storage, pumping and heating unit and combustion and secondary air fans, motors and switch gear.

(c) Elimination of ammonia vaporising equipment.

(d) Reduction in the size of the recycle handling and screening equipment.

(e) Substitution of the dryer and the dryer exhaust air handling equipment with a recycle cooler and a smaller air handling system.

The size of the granulation drum is, to a large extent, dependent on the ammoniation rate and the drum would probably be of similar size for both the trains, even though for the Cros reactor case the flow rate of solids through the drum is very much smaller.

An analysis of these differentials in equipment cost, based on mid-1978 prices in the United Kingdom revealed an estimate saving of \$570,000 in favour of the pipe reactor plant compared with a conventional plant, both plants being designed to produce 35 tonnes per hour of DAP. This difference would translate to about \$1,400,000 investment on erected basis. Assuming that the capital related charges amount to 30% of the investment per annum, the lower investment for the pipe reactor plant corresponds to a saving of about \$1.60 per tonne of product. The saving due to lower electric power consumption and from elimination of fuel cost for drying and steam demand for ammonia vaporization is estimated to total about \$2.80 per tonne of product. The overall saving is therefore expected to be over \$4.00 per tonne of product.

4. REVAMPING OF CONVENTIONAL PLANTS

From the fore-going it is evident that a Cros pipe reactor can be installed in existing plants to increase profitability through improved performance. The chief factors are the reduction in the cost of energy consumed and, in the case of a plant using P_2O_5 intermediates, also savings in the cost of raw materials through use of phosphoric acid.

The adaptation of a slurry process plant will more often than not involve the simple installation of the relatively cheap pipe reactor. Feeds to the reactor can be controlled by existing instruments. Equipment for the vaporization of ammonia, preneutralisation of acids and heating dryer air becomes redundant.

Although in itself the revamping of a plant designed to use solid feeds is equally simple, the changes required in off battery limit facilities may be somewhat elaborate. These may involve unloading and storage of ammonia and phosphoric acid.

Higher profitability can also be realized through an increase in plant capacity. In order to achieve this, debottlenecking and optimisation with respect to secondary equipment is likely to be required. For instance a DAP plant designed with 6:1 recycle ratio is potentially capable of giving 75% more output if it was operated with a recycle rate of 3:1 as the case would be if it was equipped with a Cros reactor. In order to do this however the raw material feed systems and the product handling system would have to be suitably modified. Other areas which would require appropriate changes would be the ammoniation capability of the granulator and the adaptation of a con-current rotary dryer to counter-current cooling.

There are several conventional granulation plants worldwide which are now either being modified to operate with a Cros type of reactor or which are under consideration for modification. The prime objective seems to be economy of energy utilisation. Interest also exists in lowering formulation costs through use of basic raw materials. There is no consistent pattern in the owner's attitude to increase in capacity. In certain instances there seems to be a complete lack of interest - obviously the supply/demand situation surrounding the particular owner is the deciding factor. Others have shown interest if the increase in capacity is coincidental and achieved at no extra cost. There are however one or two cases where existing operators are considering large expansion in production. These are currently under study. The Cros reactor and operating experience will undoubtedly contribute substantially towards improved performance of these plants regardless of whether the improvement is in energy use or in use of cheaper raw materials or in labour and finance related costs through increased capacity.

5. FURTHER DEVELOPMENTS

S. A. Cros are currently engaged in development work concerned with the manufacture of special applications concentrated urea based straight nitrogen fertiliser containing sulphur. Two major formulations are 40-0-0-5S and 26-0-0-15S. The work is in advanced stages of progress and several commercial scale production runs have been completed satisfactorily. Sulphuric acid, liquid ammonia and urea solution are fed to the reactor under controlled concentration conditions so that decomposition of urea is avoided. The operation retains all the advantages attributed to the pipe reactor.

Pilot plant scale tests have been carried out with co-neutralisation of phosphoric and nitric acid. Results show great promise. The rate of progress has reflected an overriding concern for safety and the decision to stay away from conditions which would lead to decomposition of ammonium nitrate. Highly concentrated solutions of ammonium nitrate-phosphate containing between 15 to 40% ammonium nitrate have been produced. The widening of this range is under investigation. The solutions granulate extremely well. The simple Cros reactor, in this case, replaces the conventional nitric acid neutralisation, ammonium nitrate evaporation and phosphoric acid neutralisation systems. Further development work is being carried out.

REFERENCES

(1) G. Hunter and J. L. Hawksley; FAI-ISMA Seminar: New Delhi, December, 1975

(2) R. D. Young and R. G. Lee; FAI-ISMA Seminar: New Delhi, December, 1975