

IFA Technical Conference

**Beijing, China
20-23 April 2004**

EXPERIENCES IN THE PRODUCTION OF NP; NK AND NPK GRANULAR FERTILIZERS USING UREA WITH AMMONIUM PHOSPHATES AND / OR SUPERPHOSPHATES

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1. SUMMARY

In this paper the author reviews his personal experiences in using urea in the production of granular NP; NK and NPK fertilisers in many different locations.

In the conclusions there is a presentation of the suggested flow sheets for the different processes.

2. HISTORY

In Europe and North America until the late 1960's early 1970's the preferred sources of nitrogen in the granular NP; NK and NPK fertilisers were, ammonium nitrate, ammonium sulphate and ammonia. It was during this period there were many advances in the technologies used to produce ammonia and urea. These advances were in the scale of operations, the reliability of the plants, on-stream factor and their consumption of energy during the production of the urea.

The majority of the existing granulation plants at that time had been designed to operate with a cold recycle. They were mainly using a hot concentrated solution/melt of ammonium nitrate and the cooled recycle helped to control the recycle ratio and as a consequence the output of the unit.(Ref. 1 and 2). As the production of urea increased during the 1970's and since its cost per unit of "N" was lower than that of the ammonium nitrate there was an incentive to use the urea. The cold recycle plants were at a definite disadvantage when using solid urea since granulation temperatures in the range of 80 c were and are required to melt the prills to incorporate them in the granules. This still holds true today in recent tests in Brazil when testing a new formulation with 20% solid urea the test was successful on a unit with hot fines recycle but much more difficult on a cold recycle plant.

My first experiences with urea in NPK's was the production of 20 -10-10 on cold recycle plants. This would have been in 1973 in the United Kingdom which at that time guaranteed P2O5 in terms of water solubility and total, with a minimum water solubility of 88% which basically eliminated the use of deep bed ammoniation.

Received 10 January 2004 for presentation at the 2004 IFA Technical Conference, 20-23 April, Beijing, China
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The first tests did not achieve the required temperatures in the granulator and trying to heat the granulate in the dryer, apart for causing high ammonia losses, also caused blockages.

One final test was programmed with a novel approach to generating high temperatures in the granulator. After carefully cleaning the granulator drum 3 direct fire propane gas nozzles were installed and when the feeds were started the nozzles were fired up. The results were better than before but the temperatures in the granulator were still low ~ 70 C. That was until one of the nozzles fell off and we had an open ended pipe, and greeny blue flames licking out of the drum. The temperatures reached the desired values but discretion and the nearly empty gas supply dictated the end of the test. It was never repeated.

It was at this time various urea-based NPK plants were built in Europe, the most published of these being in Spain (Ref. 3, 4).

These plants were of relatively small capacities and were used mainly to produce NPK's. Reference 3 was a critical paper of the time, and was presented in slightly different forms on several occasions, besides the cited conference it had been given at the Regional ISMA Technical Conference , New Delhi, India, September 1971.

This paper became an internal reference for all the subsequent plants of this type. It gave details on both the USP(Urea - Superphosphate) and the UAP (Urea - Ammonium Phosphate), systems. The grades discussed were all or nearly all NPK's :-

USP: 11 -11-11, 15 -15 -15, 18 -9 -9, and 20 -10 -10.

UAP: 19 -19-19, 26 -13-13 , 15 -30-15, and 28 -28-0.

None of the presentations gave any operating data on the only NP grade cited, the 28 - 28 -00 except to say that it would require 47% urea in the formulation.

The basic raw materials were , crushed urea prills, ROP SSP, powder MAP, KCl, NH₃ and in some cases a small amount of H₂SO₄. This was reacted with the NH₃. to raise the temperature in the granulator. In the case of the USP limestone, CaCO₃, was also used for pH control. It should be noted that the industrial production of these grades was very limited. Future experiences showed that the rate of reaction between the CaCO₃ and the free acid in the SSP was relatively slow which made accurate pH control difficult.

In the case of the USP formulations good pH control is essential both in the granulation stage and in the drying. For example at pH 4.1 in the granulator the was rapid granulation, but there was an excessive formation of oversize in the dryer.

At pH 4.5 there was still good granulation, but the dryer suffered from a steady build up throughout the production run. At pH 4.6 the granulation was good and there was little or no build up in the dryer.

In the case of the UAP formulations, the products Mole Ratio was controlled in the range of 1.1 - 1.3: 1.0. This allowed for good granulation and trouble free drying. The products all showed a reduction in water soluble P_2O_5 , which in some cases fell to as low as 35% of the total P_2O_5 while the APA P_2O_5 maintained levels of 93 - 98%. Due to legislation in the UK at that time this made these types of formulations impossible to operate.

The next three experiences were very different, the first and second of these were in Brazil. The first on a small modularised granulation unit which was marketed as "Granpack". This was the No. 2 size with a throughput of 35 /40 tons per hour. This had a very simple layout with a hot recycle, and hot crushing, the other unusual features was that it used a single fan for both the dryer and cooler.

The products that were tested both had low levels of P_2O_5 , and were for the coffee and sugar cane crops :- 20 - 05 - 20 and 18 - 06 - 18. The raw materials being prilled urea, powder MAP, KCl, SAM, the heat in the granulator being provided by the reaction between NH_3 and H_2SO_4 .

The granulation was reasonable, since the urea had not been precrushed and since the granulation temperature was a maximum of 75 C not all the prills were dissolved and fully incorporated. This may have contributed to the major problem which was as follows. The product left the dryer with a moisture content of ~ 1.5 - 1.8% and a good physical appearance, it screened without any problems, there were a few problems with the hot crushing, but since it was not possible to reduce the cooler airflow to very low level without affecting the dryer airflow with a single fan, the product picked up excessive amount of water in the cooler: leaving the cooler with moisture levels of 3.0+%!

This happened in Brazil in late summer with an ambient temperatures of 30+C and 80% relative humidity, the production had to be abandoned. This helped to reinforce the horror stories that circulate in Brazil about it being impossible to use more than 5 - 8% of urea in their NPK granulation plants. All this happened in 1976.

The following year on a bigger plant , a nominal 90 tph throughput, this time with a cold recycle, the client requested the following product :- 15 - 09 - 18, using the formulation, per ton basis:

KCl 300kg/t, Powder MAP 194kg/t, SAM 347kg, Urea 125kg, $CaCO_3$ 50kg

The following conditions were used:-

Recycle ratio 0.52 :1.0

Granulation aide LP Steam 15kg/t, no water.

Temperatures:- Ex Gran. 44 C

Ex Dryer 73 C (Air in 350 air out 75 C).

Two special precautions were taken:

The cooler fan damper was closed to minimise the airflow and to avoid moisture pick up in the cooler.

This raw materials were fed onto a collector conveyor belt, and the urea was added at the closest point to the elevator which fed the granulator, being sandwiched between the SAM and the fines. The granulation was surprisingly good, and was done in the dryer and as long as the temperatures did not exceed 80°C the build up was not serious, once again not all the urea was incorporated. This product was made at the peak of the application period in Brazil - "The Safra", so it would have been used within the week.

In many ways this production is still a mystery - cold recycle, no heat of reaction and production started at 35 tph and was increased to 57tph during the run which lasted 55hours and produced 2360tons. (Confirmed by the despatches from the bagging plant). The third plant was a dedicated plant for UAP formulations specifically two NP grades 23 - 23 - 00, and 20 -20-00, using prilled urea, powder MAP, NH₃, H₂SO₄ and an inert - sand. Other grades had been included in the design specification such as :- 18 - 23 - 00 and 15 - 15 - 15. The design rate was for 40tph at a recycle ratio of 2:1.

This unit was located in Turkey, with the powder MAP being made onsite. The unit had many mechanical problems which did not have serious effects on the process other than not allowing long periods of continuous operation.

The main problem was that the main grade 20 - 20 -00 did not granulate very well when using the design conditions in the granulator and the dryer. These conditions were as follows:-

Granulator	pH	4.6 - 4.9	
	Temperature	75 -80 C	
	Moisture	2.1 - 2.3%	
Dryer	Product Outlet		80 C
Air	Inlet	135 -140 C	
Air	Outlet	80 - 85 C	

Even when using crushed urea the granulation was poor, attempts were made to "heat granulate" the product in the dryer, by raising the outlet temperature to 115 C! Apart from some ammonia losses nothing happened, wet sand went in and dry sand came out. Showing at this pH at least the NP's do not soften or granulate just with TEMPERATURE. This is not the case with the NPK's.

The granulation problems were finally solved by changing the sequence of the addition of the liquid feeds in the granulator. The sulphuric acid addition is initiated before the ammonia creating a low pH zone which starts granulation. This is then neutralised and heated up by the addition of ammonia and steam.

My understanding of the UAP Systems came during the commissioning of a plant in the Middle East in 1990. The unit was designed to make NP's and NPK's.

15 - 15 - 15; 18 - 18 - 18; 23 - 23 - 00 and 27 - 27 - 00. The design rate was 48tph at a recycle ratio of 3: 1, with a size specification of a minimum of 85% between (+4.00mm and -1.5mm).

The unit was equipped with a urea crusher, which was greatly over dimensioned and on the few occasions it was tested filled the plant with a fine white powder - the effect was like being in a snow storm! The plant could also use "Hot" (directly) produced powder MAP or "Cold " ex store product. Since the client had almost 50,000 tons of cold product this was the normal feed.

Note the "Cold" MAP had caked badly and was a constant source of not being able to maintain the design rate for lack of free flowing feed material. This was a project using Norsk Hydro Agri technology , the then Davy McKEE engineering and was built in Iraq, for a brief but vitally important period I had the help of one of the other authors at this conference. This period did not last for more than a few weeks, but it was during this time it became possible to define the operating conditions for the production of UAP based NP's and NPK 's formulations, using solid urea and solid powder MAP.

It is important to stress these conditions as the amount of free water required in the granulator to generate the liquid phase for good granulation is very small and in the units which use urea solutions, as opposed to urea melts, and or ammonium phosphate solutions from pipe reactors generate not only higher temperatures and the need for even lower free moisture contents and higher recycle ratios. The work on this plant showed two different granulation conditions for the NPK's and the NP's formulations.

The conditions for the NPK's were as follows:-

Granulator:-

pH :5.2 - 5.6
Temperature : Minimum 80 C, Normal 82 C

Dryer

Air Inlet 90 - 120 C
Air Outlet 80 - 85 C
Product Outlet 80 - 82 C

The conditions for the NP's were as follows :-

Granulator:-

pH : 5.6 - 6.0
Temperature: Minimum 80 C, Normal 82 C

Dryer Air Inlet 100 - 120 C
 Air Outlet 82 - 85 C
 Product Outlet 80 - 82 C

In both cases the granulation temperature was generated by the use of NH₃ and H₂SO₄ and steam typical values being :-

Kg/t	NPK	NP
NH ₃	34 - 43	42 - 48
H ₂ SO ₄	45 - 55	35 - 40
LP Steam	20 -28*	29 -38*

The lower value when using "hot" MAP, the higher for "cold" MAP. Fuel consumption was typically 2 - 4 kg/t.

3. OBSERVATIONS ON FLOWSHEETS.

These observations apply to units using solid raw materials, such as urea, ammonium phosphates, potassium chloride, potassium sulphate, ammonia, sulphuric acid, fillers, ammonium sulphate, ammonium chloride and superphosphates.

1. When using solid granular urea a raw material crusher is required if all the solid urea is to be incorporated.

Note: This not necessary when using prilled urea.

2. Sufficient ammonia and sulphuric acid must be available to increase the granulator temperature to a minimum of 80 C.

3. A hot recycle of both fines and crushed oversize is an advantage.

4. A long residence time dryer, and an oversize dilution fan so that it is possible to use very low air inlet temperatures. An indirect steam heat exchanger is a possible alternative.

5. Conditioned air for a rotary or fluid bed Cooler is advisable to avoid moisture pick up during this operation.

6. Although not specific to this process single deck screens and "split" fines cloths are advisable because this makes the recycle control and control of the products SGN much easier.

3.1 Basic Case (Figure 1)

Mainly solid feeds, a crusher for urea, this is optional if prills are used, but essential for granular urea. All the solid raw material and recycle, both fines and crushed oversize, are fed to a collector conveyor, then by an elevator to the rotary granulator. Here the desired process conditions are obtained by the addition of NH_3 , H_2SO_4 , steam and scrubber liquor.

The granulator discharges into the rotary dryer which is operated in the co-current mode, using hot air from a fired furnace. The dryer has a long residence time since low air temperatures are used to avoid ammonia losses, further granulation and build-up in the drum.

The dryer discharges on to a small belt conveyor which is equipped with a weigher indicator recorder. This is a very useful process control parameter. The belt discharges into an elevator which feeds a single deck oversize screens. The oversize goes to a crusher. (either double chain or Trio- roller type), the fines and product are fed to a second single deck screen. This uses two different mesh sizes with a control flap underneath which is linked to the product weigher, which can send the small product fraction to either the recycle or to the product cooler thus maintaining recycle control and equilibrium of the system. The product goes to the counter current rotary cooler and the fines to the recycle. The product ex cooler is weighed before being oiled and coated in the coating device and then out to the storage building. The emission control systems are as follows :-

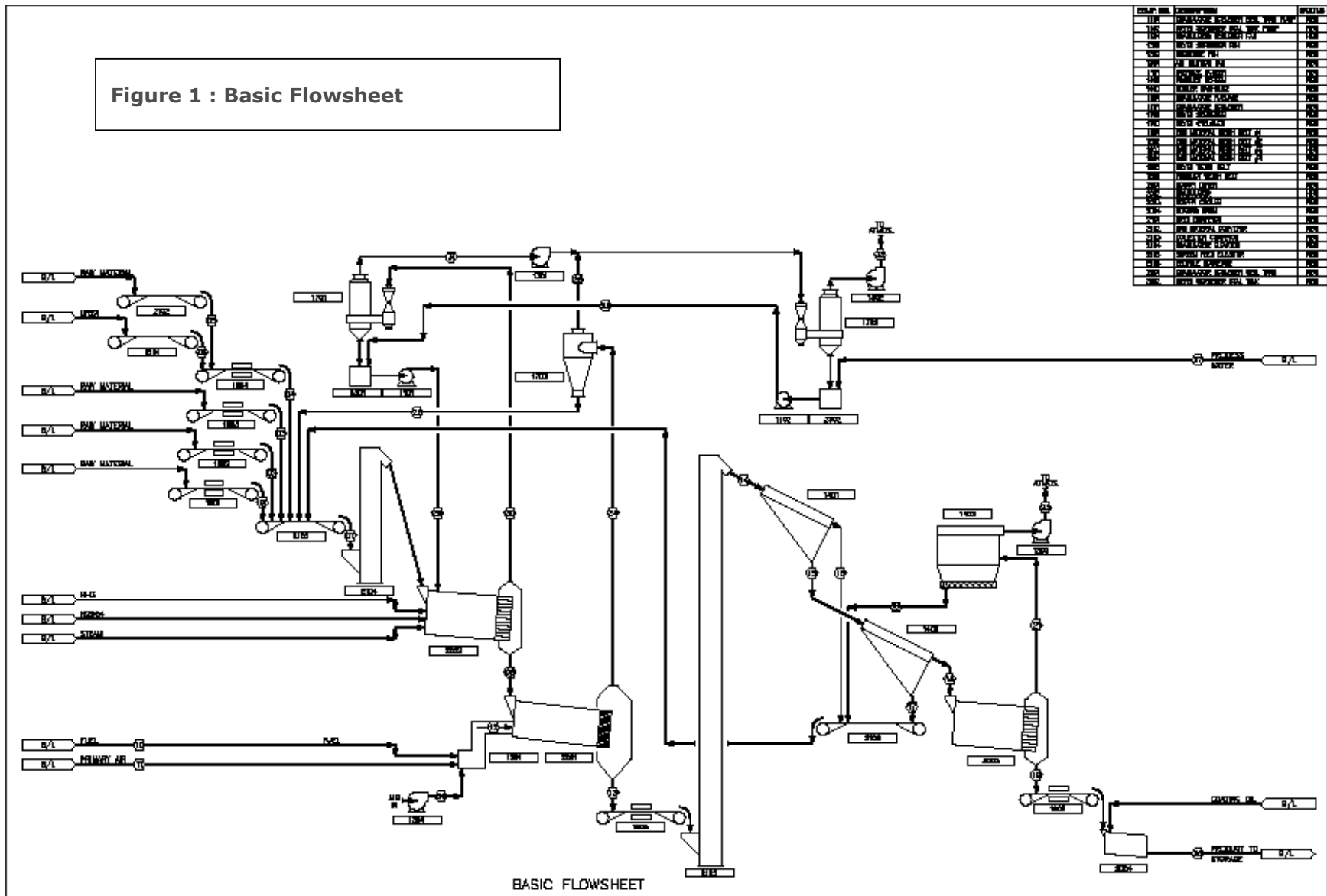
Granulator: A wet scrubber which receives the make up fluid from the dryer scrubber, the liquid from this scrubber is used if required in the granulator.

Dryer: The gases are first treated with cyclones before going to the wet scrubber. The make up liquid is either process water or a very dilute solution of sulphuric acid. The level in this scrubber is controlled by the level in the granulator scrubber or by the solids concentration. Then the liquid is bled off to the ETEL (effluent treatment station).

Cooler: The gases are treated in a bag filter with the solids being returned to the solid recycle.

Note: Depending on the local conditions the air being fed to the cooler may have to be climatized (cooled and dehumidified). This type of unit is more suitable for medium to small sized plants in countries which use many different NPK products and there are problems with transport and logistics, for example, the People's Republic of China.

Figure 1 : Basic Flowsheet



3.2 Flowsheet Option B (Figure 2)

This is basically the same as the first, but as an option the fuel fired furnace is replaced by an indirect steam air heater. This guarantees low temperatures in the dryer and as another advantage it enables the wet scrubber to be replaced by a bag filter after the cyclones. This bag filter will need an external heating source so as to avoid problems of condensation and blockages of the filter media.

3.3 Flowsheet Option C (Figure 3)

The main change from the base model is that the rotary cooler is substituted by a Fluid Bed Cooler, which would have the final section using chilled dehumidified air. This air can be linked to an ammonia vaporiser. The ammonia gas being used in the granulator.

Figure 2 : Option B Flowsheet

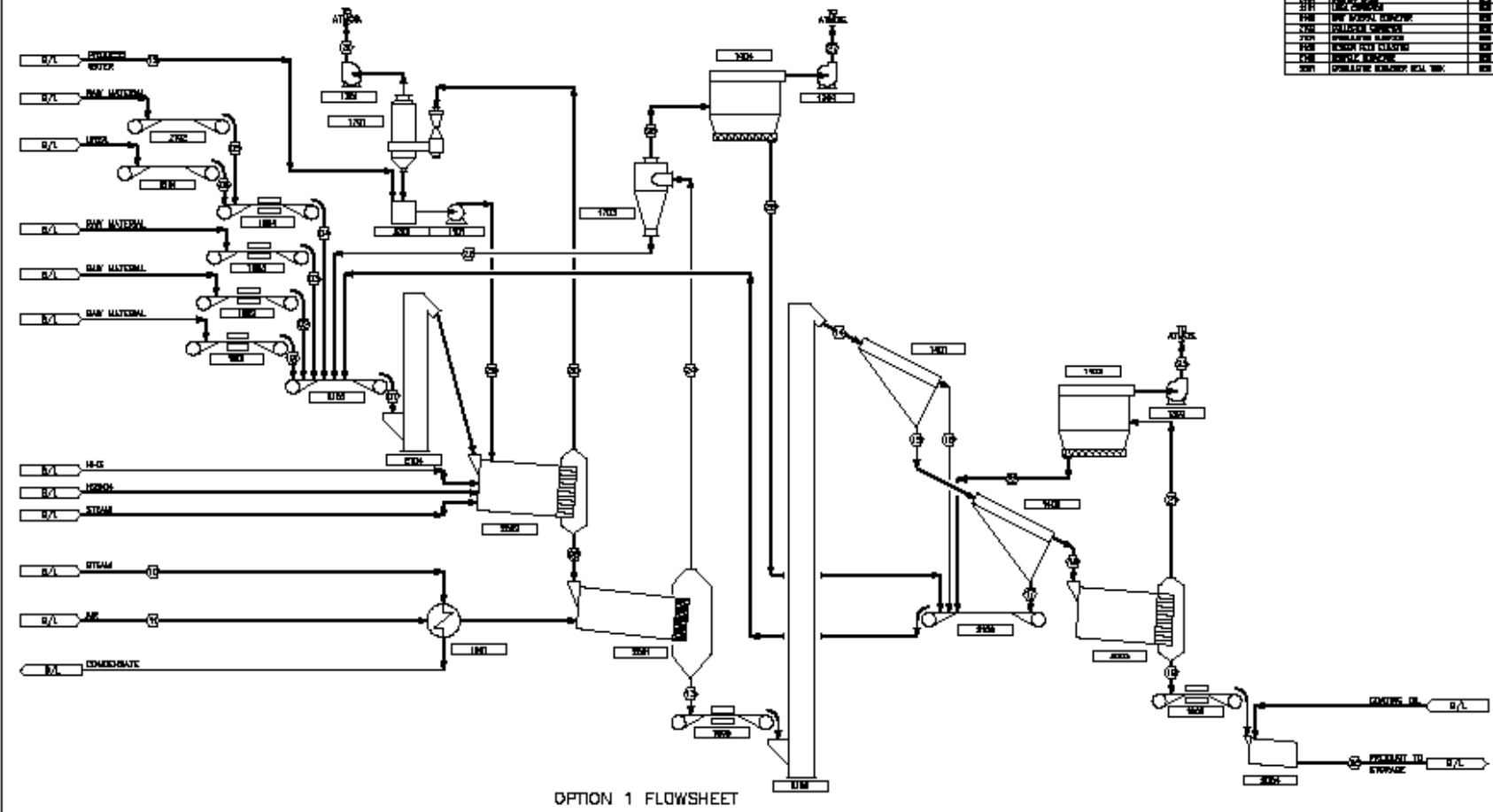
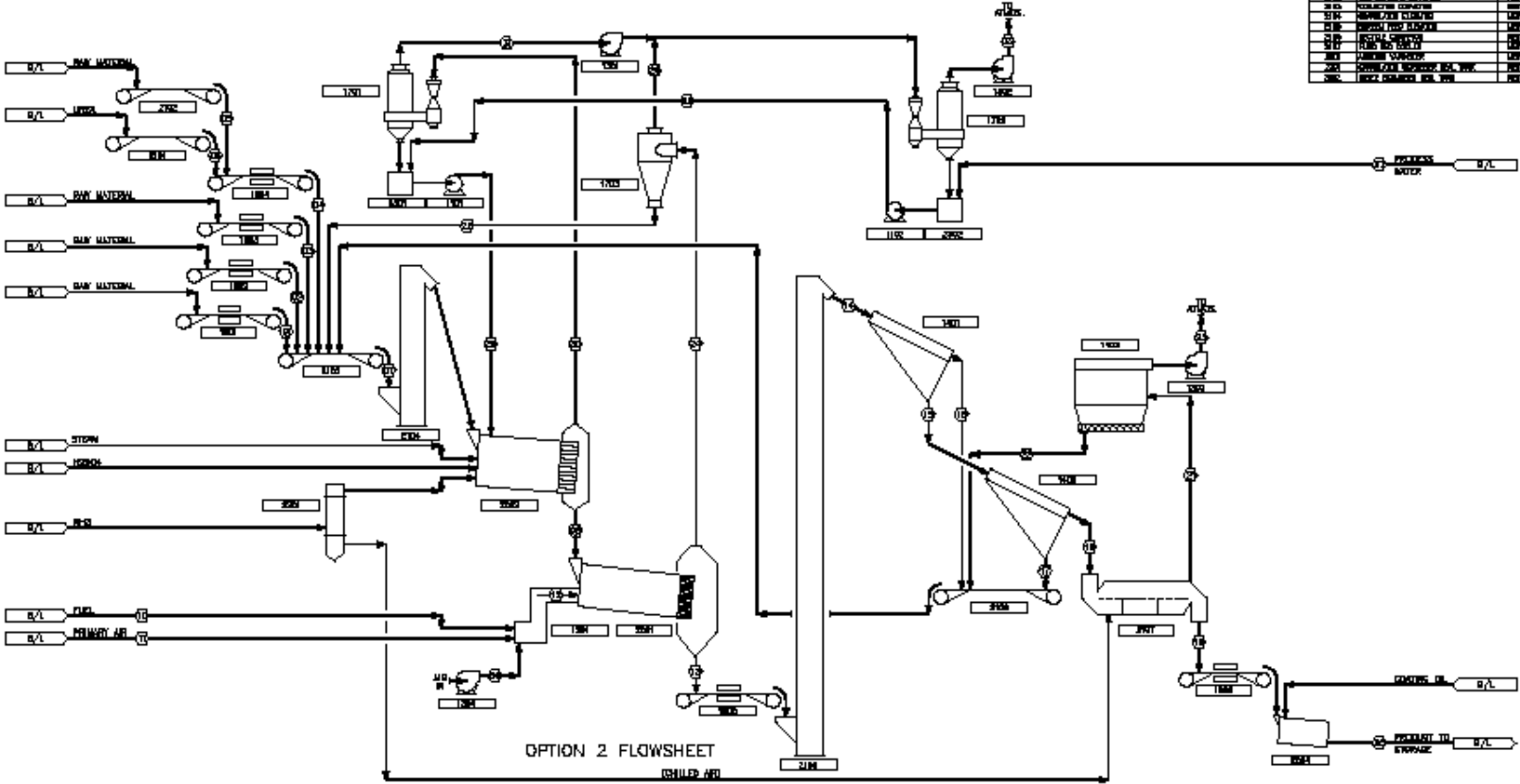


Figure 3 : Option C Flowsheet



UNIT NO.	DESCRIPTION	TYPE
1100	RAW MATERIAL STORAGE	STORAGE
1110	RAW MATERIAL STORAGE	STORAGE
1120	RAW MATERIAL STORAGE	STORAGE
1130	RAW MATERIAL STORAGE	STORAGE
1140	RAW MATERIAL STORAGE	STORAGE
1150	RAW MATERIAL STORAGE	STORAGE
1200	GRINDER	GRINDER
1300	GRINDER	GRINDER
1400	GRINDER	GRINDER
1500	GRINDER	GRINDER
1600	GRINDER	GRINDER
1700	GRINDER	GRINDER
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4000	GRINDER	GRINDER
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4200	GRINDER	GRINDER
4300	GRINDER	GRINDER
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4600	GRINDER	GRINDER
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9500	GRINDER	GRINDER
9600	GRINDER	GRINDER
9700	GRINDER	GRINDER
9800	GRINDER	GRINDER
9900	GRINDER	GRINDER
10000	GRINDER	GRINDER

4. FINAL COMMENTS

At the end of 2003, I carried out a new consultancy contract in China, on two NPK plants, making medium concentration products such as 15 - 15 - 15; 16 - 16 - 16; 15 - 15 - 10; and 15 - 10 - 15, with plans to produce grades with higher nitrogen contents. The main raw materials were powder MAP; KCl; NH₄Cl (ammonium chloride); NH₄HCO₃, (ammonium bicarbonate) ; Urea and a filler, using steam as the granulation aid. The quality of the NH₄Cl was very poor basically in was supplied in 50kg blocks! The percentage of urea that could be used was limited because of the problems of control on the dryer gases (the dilution - secondary fans did not have sufficient capacity to reduce the gas temperatures). The main novelty to me after years of experience was to use the decomposition of the NH₄HCO₃ - as the source of ammonia to raise the Mole Ratio of the MAP high enough to obtain a very good granulation even when using up to 10% of uncrushed prilled urea.

5. REFERENCES

- 1.The Application of Cooled Fines Recycle in Conventional Granulation Process. J.D.C. Hemsley, W.F. Sheldrick, Bu -Craa Symposium, Jan. 1971.
2. Low - Recycle Granulation Processes for Ammonium Nitrate- Based NPK Fertilizers. J. L.Hawksley et al, FAI-ISMA Seminar, New Delhi, Dec. 1975.
3. The Manufacturing of Granular Compound Fertilizers Based on Urea as the Principal Source of Nitrogen. J.D.C. Hemsley and F. Roig, ISMA , Seville, 1972.
4. Low Investment and Optimisation in the Production Costs of a Urea MAP - NPK Fertiliser Complex. F. G. Membrillera, J.L Thorat and F. Codina. ISMA The Hague, 1976.