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USE OF UREA IN COMPLEX FERTILIZERS:
RESULTS OBTAINED IN RECENT YEARS IN THE ERT FACTORIES

1. INTRODUCTION

Over forty years ago the manufacture of compound fertilizers began in Spain, being based on a mixture of simple fertilizers and on empirical formulae which were prepared in accordance with the requirements of the farmers themselves.

Since 1960, the mixtures have been produced by automatic processes with proportioners of raw materials and a granulation process.

Nevertheless, the true increase in the consumption of complex fertilizers began in 1964 with the first production plants employing the chemical reaction of the raw materials. Thus national consumption has risen from 185,000 metric tons in 1964 to about 1,500,000 metric tons in 1971.

Parallel with this increased consumption there has been an increase in the production of fertilizing units in complex fertilizers, as is proved by the change from 4-11-5 and 7-7-7 to 12-24-12 or 15-15-15.

This increased content led to the need to use raw materials with a higher content of nutrient elements, such as triple superphosphate, ammonium superphosphates and urea, in particular.

In the following pages a description is given of the tests and industrial results obtained in various factories producing complex fertilizers, belonging to the Union Explosivos Rio Tinto S.A., which were not designed to use urea as raw material.

2. USE OF UREA IN THE ERT GROUP

As we have already stated, the growing demand for increasingly high-quality fertilizers forced ERT as manufacturers of fertilizers to use urea and other high-yield products in the proportioning of the various products.

Since all ERT granulation plants using urea were built before the appearance of highly-concentrated NPK granulated fertilizers, it was necessary in every one of the agricultural areas where these plants were

situated to make intensive tests of the use of urea, until products were obtained which could compete on the market and provide sufficient guarantee of quality. All the tests were carried out without making any great changes in the installations since the normal production programmes had to be carried on at the same time.

The agricultural districts of Spain where the demand for increasingly concentrated fertilizers was and still is most marked, and where ERT supplies the markets, are Andalusia-Estremadura, the Eastern area and Castile, and Catalonia. The greater demand for N_2 is to be found in the second and third areas, the first area mentioned displaying the greatest demand for potash. P_2O_5 may be said to have increased equally in all three.

At the same time it must be borne in mind that each of these plants had certain facilities for obtaining raw materials, apart from differences in design and operation.

It may be inferred from the above that the efficient production of urea fertilizers with different types of installations and different classes of raw materials, has provided various data and experiences which we are sure will be a help in the development of these fertilizers, both within our group and elsewhere.

Industrial plants

ERT have three granulation plants used for testing and producing urea fertilizers and we shall see in the following that they are very different in design and operation. Henceforth we shall call these plants 1-C, 2-C and 1-A. There are also two other plants which, within their own limitations, are at present experimenting with the use of urea in the granulation of fertilizers.

Appendices 1a, b and c give diagrams of the three plants concerned, and the following differences may be mentioned:

	<u>1-C</u>	<u>2-C</u>	<u>1-A</u>
Granulation	Drum	Plate	Drum
Drying	Combustion-fuel oil	Reaction	Combustion-fuel oil
Cooling	Atmospheric air	Evap. NH_3	Atmospheric air

	<u>1-C</u>	<u>2-C</u>	<u>1-A</u>
Classification	Cold	Cold	Hot
Dust removal	Scrubber- cyclones	Cyclones	Cyclones- filter- hoses
Recycling fines	Granulator & drier	Granulator & drier	To the granulator
Recovery	Yes	No	Yes

In the three plants the raw materials are proportioned by means of discontinuous weighing by the tonne, being mixed before being continuously fed to the granulator. The fines are fed in a continuous and regulable manner by weighing in all three cases.

The liquid raw materials are fed to the granulator in a continuous and regulable manner by rotameter.

The three plants also have a finished product scale (discontinuous, by the tonne) and also automatic samplers.

The rated production capacities attained with urea fertilizers of various titres are as follows: 1-C: 12 tonnes/hour; 2-C: 10 tonnes/hour, and 1-A: 22 tonnes/hour.

The most noteworthy differences in the design of the equipment are shown in Appendix 2. Some aspects of the information it contains may be mentioned here.

The control of proportioning and of the finished product is sufficiently good and efficient for the evaluation and repetition of tests in the three plants under consideration.

As regards the granulating equipment, the three plants differ essentially: even the two which have drums. The perfection of the liquid-distribution system in 1-A (sparger), in conjunction with hot recycling, means that this granulation system offers ample facilities for controlling the efficiency of the operation. Consequently, the ammonia proportion can be accurately calculated in relation to materials of an acid nature (H_2SO_4 , P_2O_5 of the superphosphate, MAP etc.).

This does not apply to the similar plant 1-C which, recycling cold and operating with a shorter residence time and less satisfactory distribution of ammonia in the mass, cannot ensure efficient operation except by

reducing the water in the system as much as possible and increasing the temperature by reaction of the sulphuric acid and ammonia. Ammonium is still lost, notwithstanding the better atomisation of the acid. For this reason the plant has a subsequent washing tower, the effluent from which is utilised to moisten the granulator when appropriate.

The plate plant suffers to a greater degree from the disadvantage already mentioned, since it is unthinkable that any portion of NH_3 would react with the MAP or with the monocalcium phosphate of the superphosphate. On the contrary, it is necessary to add excess acid in order not to lose the NH_3 , which will later be neutralised in the dryer by reaction with more ammonia. Since at the same time recycling is cold, it is advisable to add a little water and raise the temperature as much as possible.

The granulator of plant 2-C undoubtedly achieves greater hydrolysis of the urea than does the apparatus of plant 1-A which does not bring about this phenomenon to any appreciable extent.

It is well known in connection with the drying operation that urea fertilizers must be dried to the optimum value, by means of considerable air flow at a moderate temperature. Consequently it is from this aspect that the three plants we are investigating differ most.

Since plant 1-C can only regulate drying by means of the output temperature of the air, it suffers from problems of excessive melting of the product in the top of the drum with the resultant build-ups on the walls, and losses of water-soluble N_2 and P_2O_5 . It must be borne in mind that in these highly concentrated formulae, the first part of this apparatus (helical blade zone) acts as an extension of the granulator, otherwise the circuit would be turned to fines.

Plant 2-C has the least perfect drying system. Although it uses very little water for granulation, the NH_3 -sulphuric acid reaction is very sharp and the heat released is sufficient to evaporate the water and at the same time to prevent the separation of the more insoluble or more abundant materials. The ammonium added to the bed at the top of this apparatus and in the mass of product may be gaseous or liquid according to whether it has or has not been evaporated in the cooler housing. This is the only temperature-regulating system since the temperature of the fines from the coarse grains entering this apparatus is consequently self-regulated by the operating temperature.

The cooling devices are similar in all three plants

investigated and fairly proportional to the amount of product handled, at least where the basic dimensions are concerned. Plant 2-C differs inasmuch as the housing of the apparatus is used to evaporate part of the liquid ammonia consumed. The principal difference lies in the relative humidity of the air handled which renders the operations.

NPK Balance and Formulations Tested and Manufactured

The three plants investigated were geographically situated in agricultural areas where the crops are generally extensive and varied, so that the demand for concentrated fertilizers has increased for many types of NPK balances, nearly always justified by one crop in particular. Thus, for example, in addition to 1:1:1 which is widely used, the olive groves in Andalusia have preferred 1:2:3, rice 2:1:0, brewing barley 2:2:1 and the Eastern fruit growers 4-2-1 and 2-1-1, sometimes taking into account some nutrient of a secondary kind (Mg or Boron).

As a result of the above, the range of balances and formulations tested and manufactured by plants 1-C, 2-C and 1-A is considerable and is shown in the following table:

<u>Balance</u> NPK	<u>Units</u> 1-C	<u>Totals</u> 2-C	<u>N⁺P₂O₅⁺K₂O</u> 1-A
1 - 1 - 1	48	45	45
	39-S	42	30
1 - 1 - 2	-	-	48
2 - 1 - 1	40	-	-
	40-S		
1 - 2 - 1	44	39	48
		48	
2 - 2 - 1	36-S-Mg	35	-
		36-S-Mg	
1 - 2 - 3	42-S-Mg	-	48-B
4 - 2 - 1	35-S-Mg	35-S-Mg	-
2 - 1 - 0	42	-	-

Note: S - no chloride Mg - with magnesium
B - with boron

In accordance with the tonnages produced and without taking into account those intended for tests and samples, the average NPK titration in each of the plants is as follows:

	<u>1-C</u>	<u>2-C</u>	<u>1-A</u>
Average mixture rates			
N ₂ %	18	13	10
P ₂ O ₅ %	15	18	16
K ₂ O %	7	12	22
Total richness	30	43	48
Average mixture rate of urea N ₂ %	13	5	3
Nitrogen of non-urea origin % of total	30	60	70

3. EXPERIMENTAL SECTION

Reasons for investigation

As we have already stated the three plants differ considerably in their characteristics. This, coupled with the need to market urea fertilizers of competitive quality has led us to make maximum use of the facilities of the plants (which at first appeared to be inadequate in design), and to obtain results which would be satisfactory in all respects.

Logically, each plant, by reason of its situation and the accessibility to certain raw materials in particular, attempted to produce minimum-cost formulations, basing its calculations on the comparative price of the nutrients (particularly N₂) contained in the various raw materials.

Since ammonium nitrate and urea are hygroscopically incompatible, the source of the N₂ tends to follow the following order of priority: Ammonia-Urea-DAP-MAP and the source of P₂O₅ is always preferred in the order: Superphosphates-MAP-DAP.

Where potash (K₂O) is concerned, ClK is always preferred as being cheapest, but chlorine has agricultural limitations and some incompatibilities with urea and ammonium salts.

It will be seen that we had to make a selection of important proportioning parameters to provide a basis

for the operating practice which would lead us to interesting results and conclusions in the three plants.

Choice of important proportioning parameters

1. Maximum quantity of ammonia to be used in liquid or gaseous form = AMMONIA
2. Maximum quantity of urea to be used in bead or crystalline form = UREA
3. Ratio in weight Urea/P₂O₅ from the superphosphate = Ru
4. Method of adding the potassium (as chloride or sulphide) = POTASSIUM
5. HUMIDITY in the granulator

The AMMONIA parameter is related to the economy of formulation and to the heat contributed to granulation since its reaction with sulphuric acid must be utilised to the full. It is of the greatest operational importance in plant 2-C where, in addition to acting as a granulation regulator, it is to some extent a drying regulator.

The UREA parameter is logically one of the most important, since after ammonia it is the cheapest source of N₂ and it is also very much related to the quantity of MAP or DAP used if at the same time it is desired to consume as much superphosphate as possible which is supplied by P₂O₅ which is itself cheaper.

The ratio Urea/P₂O₅ referred to the superphosphate alone is an important parameter, owing to the formation of the compound Ca(H₂PO₄)₂.4 CO(NH₂)₂ with the release of water and the formation of Ca(SO₄)₂.4 CO(NH₂)₂ which also has a markedly hygroscopic character.

The addition of potassium to the formula in the form of chloride or sulphate is also very important owing to the formation of compounds with a basis of urea and ammonium chloride with a marked deliquescent character.

Finally, the amount of water present in the system affects granulation, drying, and the losses of NH₃ by hydrolysis of the urea, particularly if excess acid is present.

Quality of raw materials used

Appendix 3 gives this information, showing chemical

composition and particle sizes.

Evolution of the important parameters and proportioning, and operation criteria until optimum results were obtained in each plant

Plant 1-C

- The maximum quantity of ammonia which could be used was 45 kg/tonne without great loss, the optimum being fixed at 40 with yields higher than 98%. The plant has acid gas-washing facilities.
- It has been established that in the granulator the DAP gives off some ammonia (1 kg NH₃ per 100 kg of DAP) for which reason it is necessary to proportion the corresponding additional sulphuric acid. The fact that under these conditions losses of N₂ by hydrolysis of the urea were not detected seems to show that the NH₃ given off is fixed in the monocalcium phosphate of the superphosphate.
- Various formulations have been tried out with DAP or MAP alternately, and no differences were noted in the efficiency of the granulator, which is further evidence that over-ammoniation of MAP in the granulator does not occur in practice.
- With the criterion of using the largest possible quantity of superphosphate and ammonia, various formulations have been tried with increasing amounts of urea, it being found by experiment that the optimum amounts to be used for the different formulations correspond to the following Urea/P₂O₅ ratios of the superphosphate.

<u>Formula</u>	<u>Proportion of Urea %</u>	<u>Ru</u>	<u>% P₂O₅ from the ammonium phosphate</u>
16-16-16	25	8	70
12-12-15-S	15	3	60
20-10-10	35	8	55
20-10-10-S	35	8	55
12-24-8	10	2	80
15-15-6-S-Mg	20	4	65
6-12-24-S-Mg	5	1	75
20-10-5-S-Mg	35	8	50
26-16-0	45	10	70

These optimum values are denoted by their good (that is, efficient) granulation properties, i.e. minimum segregation of urea and minimum quantity of water necessary to granulate with the maximum reaction heat. It will be seen that the amount of P_2O_5 coming from the ammonium phosphate is considerable and that urea does not form a compound with it. The urea gives rise to problems when added in excess of 25% owing to segregations in the granulator, for which reason it is desirable to force the NH_3 -acid reaction in the granulator and to continue the operation in the first part of the dryer where the urea melts and consolidates granulation. In formulae such as 26-16-0, the granulation phenomenon is detected as far as the cooler.

- With regard to the method of adding the potassium we found that in every case granulation is better when no chloride ion is present. Otherwise the process may be regarded as satisfactory for K_2O contents of less than 18%, since from that point onwards it is almost impossible to obtain a product without separated potassium.

This parameter is of great importance to the storage properties of the products. Fertilizers with chloride and urea produced by us show a marked tendency to form lumps. If a network of intergranular bridges occurs and the surface of the granules turns white, it is certainly a case of the compound $NH_4Cl.CO(NH_2)_2$.

- The tests carried out with magnesium sulphate have once more shown that if this salt is included in small quantities (5% and less) it reacts with the urea and produces compounds which are difficult to dry and whose specific gravity varies suddenly with the temperature.
- The optimum granulation conditions were determined by us in the following way:

Recycling proportion 1:1

Temperature: maximum attainable ($50^{\circ}C$)

Water present 4.0 to 4.5% (of which 20% is contributed by the fines)

Concentration of sulphuric acid

- The optimum drying conditions have been established, taking into account the fact that the only way to regulate the process is by regulating the temperature of the output gases, which automatically regulates the fuel-oil fed to the heating furnace. At the same time attempts were made to avoid hydrolysis of

the urea and particularly to ensure that the plasticity of the materials in the dryer top was such that granulation would proceed without causing building-up on the walls.

Using these criteria, we have come to the conclusion after carrying out many tests, that optimum operating conditions exist when:

- Maximum temperature of output gases = 70°C
- Maximum temperature of input gases = 200°C
- Air flow = maximum (30,000 m³/h)
- Maximum load of dryer = 25 tonnes/h
- Maximum quantity of lumps = 20%

We have found that in these conditions the product at the output of the dryer has a temperature of 60°C and a humidity of between 1.5 and 2.5%, with a content of fines (2 mm) not higher than 60%.

- The cooling system of this plant has no means of regulation so that it was not possible to optimise its operation.

It is only necessary to deal with the distribution of sizes in the product coming from the dryer, but if this is kept within the optimum limits the cooling apparatus should make a product at less than 40°C, which is not very high for fertilizers without nitrogen in NO₃ form.

Plant 2-C

- The maximum quantity of Ammonia which can be used is of the order of 80 kg/tonne without appreciable losses, taking into account the possibility of adding it in liquid form to the granulation plate and in gaseous or liquid form to the Dryer-Ammoniator. In fertilizers with urea it is more convenient to add it in liquid form at the two points and in the ratio 1:1 in nearly every case, since if the drying capacity is low it is necessary to obtain efficient granulation based on temperature, and the only means is the NH₃+H₂SO₄ reaction.

The optimum operation seems to be to add 40% of NH₃ to the plate and 60% to the drier, putting all the acid (98%) in the first apparatus.

- The addition of the acid is stoichiometric with

5% excess quantity which we found sufficient to absorb the NH_3 losses (including those of hydrolysis of the urea and decomposition of the DAP).

- We were unable to prove that in this type of process NH_3 is attached to the MAP or to the P_2O_5 of the superphosphate.
- We have concluded from the tests carried out with MAP or DAP for the same formulation that the operation succeeds better with MAP, certainly because of a phenomenon of internal ammonisation arising from the hydrolysis of the urea which we were investigating.
- Having determined the NH_3 and the sulphuric acid, we followed a criterion of using the maximum amount of superphosphate as the source of P_2O_5 . A large number of proportions were tried, preference being given to the practicability of obtaining a good-quality product at maximum production capacity and minimum cost, the following optimum values being found:

<u>Formula</u>	<u>Proportion or Urea %</u>	<u>Ru</u>	<u>% P_2O_5 from the ammonium phosphate</u>
10-22-7	4	0.7	70
12-24-12	44	0.7	85
14-14-7	14	2.0	55
14-14-14	13	4.7	80
15-15-15	13	6.5	80
15-15-6 S-Mg	13	3.2	70
20-10-5 S-Mg	25	6.2	60

These optimum values are characterised by the good condition of the product on leaving the dryer, since this is the final stage of granulation in this type of plant. In every case it will be seen that the amount of ammonium phosphates is large, and causes no granulation problems.

The tests carried out with increasing amounts of potassium have shown that with this type of plant it is not possible to include in the granule a higher concentration than 16% of K_2O added as chloride.

It was also possible to confirm that when

potassium is added as sulphate, even the difficulties involved in concentrations of over 20% of K_2O practically disappear.

- In view of the experience of plant 1-C, the interaction of magnesium sulphate with the urea was not tested. Nevertheless, it was established that the addition of magnesium in oxide form involves no problems for the formulations tried out and manufactured.

The granulator operating conditions which we regard as optimum may be defined as follows:

<u>Formula</u>	<u>Temperature</u>	<u>Recycling</u>	<u>Humidity %</u>
10-22-7	78	2 : 1)	3% in all cases
)	
12-24-12	80	2 : 1)	
)	
14-14-7	76	3 : 1)	
)	
14-14-14	73	3 : 1)	
)	
15-15-15	80	4 : 1)	
)	
15-15-6 S-4	83	4 : 1)	
)	
20-10-5 S-3	82	4 : 1)	
)	

If we consider in all cases recycling with approximately 2% of H_2O , it will be seen that, with the exception of the first two formulae, which yield a good deal of superphosphate (340 and 300 kg/tonne), water must be added to the granulator. Moreover, the sulphuric acid has to be 98% (max 300 litres/hour) if efficient operating conditions are to be obtained.

- Optimum operation conditions in drying were established as a function of the fact that granulation proceeds in this apparatus until the fines are reduced to a lower value than the ratio applied in the top granulator. The ratio of lumps to the product passing into the dryer must certainly also be kept at 1 : 2.

The only means of regulating the operation amounts to introducing a greater or lesser amount of liquid ammonia into the reactor-dryer so that reaction with sulphuric acid causes the temperature to be raised as much as possible (never below $95^{\circ}C$) in order to obtain a humidity of about 1.5% which we regard as the maximum permissible for acceptable

storage properties.

- Cooling of the product with the available equipment is efficient, provided the fines in the operation do not exceed 20% of the product in the feed.

Plant 1-A

- The characteristics of the granulator of this plant have made it possible to obtain quite good knowledge of the operation. On the other hand, the "sparger" or distributor of ammonia and acids makes possible almost perfect mixing of the materials in the granulation bed, and on the other hand the residence time is sufficient for a high degree of homogeneity to be obtained in the composition of the granules. Furthermore, the utilisation of the NH_3 is such as to enable it to be added as a function of the quantity of all the acid materials present.

After many tests we found that the rate of ammonia used must be based upon the following points (with losses of less than 2%):

Maximum quantity NH_3 per tonne of product	65 Kg
Quantity of NH_3 established per 100 Kg of P_2O_5 from standard superphosphate	30 Kg
Ditto, Triple Superphosphate	17 Kg
Ditto, MAP	10 Kg
Ditto per 100 Kg of H_2SO_4 100%	30 Kg
Quantity of NH_3 released per 100 Kg of DAP	5 Kg

In calculating the proportions, account is also taken of the fact that at operating temperature, 4 Kg of NH_3 are released for every 100 Kg of urea used.

We also found that the granulation of urea fertilizers is more efficient when MAP is used, which is certainly due to the over-ammonisation of this product in the granulator.

- With regard to the optimisation of proportions, we have concluded after a number of tests that optimum financial and operational results are obtained by using the following parameters:

<u>Formula</u>	<u>Proportion of Urea %</u>	<u>Ru</u>	<u>% P₂O₅ from the ammonium phosphate</u>
8-16-24-2B	3	0.7	80
12-12-24	13	4.0	80
12-24-12	7	4.0	85
15-15-15	20	4.5	75
10-10-10	8	0.7	-

Ru being the ratio Urea/P₂O₅ from the superphosphate.

The input of potassium alone has been tested by means of chloride, but we found that above 20% K₂O the outputs are low owing to chimney losses, particularly in the drier.

- We found no problems in connection with the proportioning of borate in fertilizers with urea.
- The following are the optimum operating conditions in the granulator:

Recycling proportion	1:1
Temperature °C	90/95
Water present %	5
% concentration sulphuric acid	78

- The optimum operating conditions in dryer and cooler are:

	<u>Dryer</u>	<u>Cooler</u>
Air flow, M ³ /hour	58,000	51,000
Temperature of solid at outlet of apparatus in °C	85	30
Product in operation, tonnes/hour	35	17
Maximum quantity of lumps %	5	-

4. CHARACTERISTICS OF THE PRODUCTS

The nutrient element content of all the above-mentioned products manufactured with urea, complied with Spanish

statutory regulations and with maximum tolerances of $\pm 0.2\%$, normally being positive in N and P_2O_5 and negative in K_2O , particularly with high mixture rates. In order to obtain these results we sometimes had to work with low yields, particularly in potassium. It should be noted that Spanish regulations require the assimilable P_2O_5 to be the sum of that soluble in water and that soluble in ammonium citrate.

The granulometry of the products differs in each of the three plants investigated, according to the screening characteristics (Appendix 1), in every case 85% of the product being between 1 and 4 m/m the fractions higher or lower than this limit not exceeding 10%.

As regards the hygroscopic nature of these products, we concluded after numerous tests that there are no great differences between makes and products, the critical relative humidity always being between 50% and 60%.

The same may be said of the melting temperature, the apparent specific gravity of the granules, and the angle of repose, which have always been between $140^\circ/100^\circ C$, 1.2 - 1.3 gr/cc and 35° and 40° respectively.

The tendency to compact varies remarkably from one product to another. They usually behave well if the storage time does not exceed 45 days and the stacking depth does not exceed 4 metres. It should be noted that when there is high relative humidity in the stores at plant 1-C, special precautions must be taken, there being a considerable difference in the penetration of humidity which reaches 5 cm in the stack measured over one month.

5. CONCLUSIONS

As this paper shows, it was possible to obtain high-quality compound granulated fertilizers by using urea in plants which were not originally designed for such use. ERT was able to produce these products industrially in these plants so as to comply with statutory quality requirements.

At the same time, we gained some experimental knowledge in the test stages which corroborated the theories appearing in the relevant literature with regard to the formation of burnt matter, hydrolysis of urea, internal ammoniation of MAP, ammonia balance in the granulator and losses of water-soluble P_2O_5 by ammoniation and drying.

DISCUSSION

Mr. J. OLIVARES (Union Explosivos Rio Tinto, Spain): The manufacture in Spain of compound fertilisers based on a mixture of simple fertilisers started in about 1930, but the first NPK granulation plants did not begin operating until 1961; the Union Espanola de Explosivos (predecessor of E.R.T.) was one of the principal producers in Spain, with very advanced technology in nitrogen, phosphorus and potassium-based fertilisers. It was at this time that complex granulated fertilisers, produced from chemical reaction of the raw materials, first appeared in Spain.

However, it was from 1964 onwards that a large annual increase in the consumption of these fertilisers began to make itself felt in Spain, the figures rising from 185,000 metric tonnes to a figure eight times as great in 1971.

Alongside this increase in consumption an appreciable increase in the nutrient units contained in each actual tonne of fertiliser took place, almost trebling in many cases. For this reason it was necessary to provide increasingly enriched raw materials - particularly in N_2 or P_2O_5 or both. Urea is one of these raw materials in NPK granulated fertilisers and as such has been in use in various ERT plants since 1968, although industrial scale tests were carried out before that date.

The ERT group's production of fertilisers with urea in 1968 amounted to only 10,000 metric tonnes; by 1971 193,000 metric tonnes had been produced and by the end of the current year a total of 300,000 tonnes, reckoned as from 1968, is expected to have been exceeded. This production has been achieved in industrial plants which were not designed for the purpose, despite the difficulties involved in the use of urea.

The design of the plants which ERT operates for the manufacture of fertilisers with urea did not, as we have said, anticipate this use, and it has therefore been necessary to carry out intensive research and experiment in order to manufacture products which would be competitive on the market and carry adequate guarantees of quality, on occasion sacrificing production to meet the demand for increasingly rich fertilisers with increasing agricultural requirements.

In addition to the operation and design characteristics of the plants operated by ERT, importance had to be attached to the comparative price of the raw materials, which involves very different possibilities

for each installation. In any case, the basis of preference in the use of raw material was: ammonia-urea-DAP-MAP for nitrogen, and superphosphates-MAP-DAP for P_2O_5 .

As regards K_2O , chloride is cheaper than sulphate, but has agricultural limitations; certain incompatibilities with urea, and gives poor yields. These criteria were modified on some occasions in favour of more efficient plant operation and optimisation of quality and use of products.

Another important point is that the experience acquired by ERT in the manufacture of fertilisers with urea, in view of the individual characteristics of each plant, which mean that they differ, and the fact that they were not designed for the purpose, has provided valuable information which has enabled us to obtain these production figures in the existing installations and to think of future applications.

In this paper, we are showing ISMA the scope attained by our manufacturing programme for fertilisers with urea, as regards diversity of formulations, richness, modus operandi etc., in each of the three industrial plants (1-C, 2-C and 1-A) which served as the basis for these industrial-scale experiments described in the report, together with the operational parameters.

The three installations differ appreciably:

- 1-C. Consists of conventional granulating, drying, cooling and coating drums with cold recycling, gas scrubbing in the granulator and an average production capacity of urea fertilisers of 12 metric tonnes per hour.
- 2-C. This is an installation with a plate granulator without a conventional dryer and a cooling drum without a coating unit. Recycling is cold and drying takes place in an ammoniator drum which uses the heat of reaction of $NH_3 + H_2SO_4$ to eliminate the water. The fines are recycled to the plate and the ground coarse grains to the ammoniator drum.

The average production capacity achieved with this urea fertiliser plant is 10 mt/h.

- 1-A. This is a plant similar to 1-C, but different from it in that the recycling is a hot process and the granulator has a sparger of special design which has enabled us to obtain a fairly

accurate idea of the NH_3 balance in the granulator.

Both these differences, and other more individual differences between the various units are set out in the report.

Depending on the demand for nutrients in each of the agricultural zones in which the three plants are located, we have succeeded in making some 20 different NPK formulations with balances of 1-1-1, 1-1-2, 2-1-1, 1-2-1, 2-2-1, 1-2-3, 4-2-1 and 2-1-0, achieving a maximum nutrient content of 48 units in the three plants, as set out in the report.

The average percentage of N_2 of urea origin in the fertilisers manufactured commercially was 70% in plant 1-C, 40% in 2-C and 30% in 1-A.

As regards the parameters we considered of interest in the manufacture of this series of products, we would mention the following:

Quantity of liquid or gaseous ammonia to use

Minimum quantity of urea

Urea/ P_2O_5 ratio of superphosphate

Form of addition of potassium

Humidity in the granulator

The report describes the evolution of these parameters in each of the three plants, and the optimum dosage for each of the products. The granulation and drying conditions are also given, with an indication of the recycling operations.

In general we may say that all these formulations with more than 15% urea content present problems if enough care is not taken over the drying and the storage period not kept down to a maximum of 45 days. The use of potassium sulphate eliminates many of these problems, and gives better yields in K_2O rich formulations. Above 25% urea the operation must be carried out with sufficiently high granulating and drying temperatures without causing losses by hydrolysis which must be checked by the MAP and P_2O_5 of the superphosphate.

The grades which offered most difficulty in drying and storage were those with a molar ratio urea/ P_2O_5 of between 2 and 6.

Although operating in almost all the formulations

with an excess of acid, we did not detect any major losses of ammonia through hydrolysis of the urea, no doubt as a result of internal phenomena of ammoniation in the granulator on the MAP and superphosphate. Of course the 2-C plate installation is the least perfect in this sense, and although sufficient sulphuric acid is used both in the dryer and the granulator the ammonia losses occasionally reach 5%.

However, in the granulator fitted with a sparger we managed to work out the ammonia balance quite accurately, as set out in the report, and the losses are therefore minimal.

The quality of the products with urea which ERT has put on the market has been good, and within the statutory Spanish specifications, although it is necessary to point out that our production programmes for these urea fertilisers have on occasion been affected by the tendency of some grades to agglomerate or compact during storage.

Mr. K. SHARPLES (Seabright Chemicals, United Kingdom):
It is my privilege to open the discussion on the paper by Olivares and Oliva on "Use of Urea in Complex Fertilisers: Results obtained in Recent Years in ERT Factories."

My qualification for this task is that some seven years ago I was responsible for developing the use of Urea on conventional granulation plants for a consortium of 14 small companies in the U.K. which were designed Fertiliser Development Ltd. Today, I am representing Seabright Chemicals Ltd., as their Technical Advisor.

The paper is complex, in that it seeks to compare and contrast three different plants. This is always difficult - it is like comparing apples, oranges and pears and assessing the results in bananas.

Plant 1-C produces at 12 t.p.h. in a system based on TVA without a pre-reactor.

The range of formulation indicates much higher concentrations and very high proportions of Urea. In the formulations on page 9 - 8 I presume I am right in assuming you use 35% Urea, that is 16 units N in 20 - 10-10. All experience in the U.K. tends to use only 20% Urea (9.2 units of N.)

My experience agrees with that of the authors that this type of plant is difficult to control.

I am most surprised that a re-cycle of 1:1 can be

achieved.

Plant 2-C produces 10 t.p.h. of somewhat less concentration of compounds. I have no experience of plate granulators. My first reaction to the circuit and description is to question why such a complicated system has been maintained. All results from true TVA ammoniation reactions have in my experience been most satisfactory. It is our job as technicians to design out troublesome equipment. For such a low throughput then the more standard methods would appear to me to be more acceptable.

The limitation on 16% K_2O added as KCl is surely a constraint on the concentration of compounds that can be made.

I am intrigued to know why fines are screened off first, rather than over-size. On small plants it is generally easier to screen out over-size to make a better job of screening out the fines from the product.

Perhaps the authors would comment on this?

Plant 1-A is a large plant giving 22 t.p.h. The obvious success of this plant is that the range of compounds made is comparatively simple with respect to concentration. The very large screening section prompts a question. Are the two circuits always in use, or does one tend to be a stand-by unit whilst parts are out of action for cleaning or screen cloth replacement?

Turning to the more general points of the paper, it is obviously economically important to the company to maximize use of superphosphate and ammonia in terms of cheapness of the materials. This was a strong phase in the U.K. and I wonder how valid it is.

So many times the cost of raw materials is taken as a criterion without reference to rate and quality of production. Cheap raw materials can often be expensive when used. In these days, excellent forms of solid ammonium phosphates are available and granulation and drier control can be so rigid that the plugging of driers is a very rare event. Even plants with ammoniation vessels eliminate superphosphate as much as possible. Formulations tend to maximise the in situ ammonium phosphate and urea and if necessary balance the formula with a good quality sand to give a much harder granule.

I fully agree with the authors that the complex calcium phosphate-urea intermediates are detrimental

I should like to thank Mr. Sharples and all those present for their attention, and hope that all the questions have been answered.

Mr. J.D. RIBEIRO MARCAL (Companhia União Fabril, Portugal):

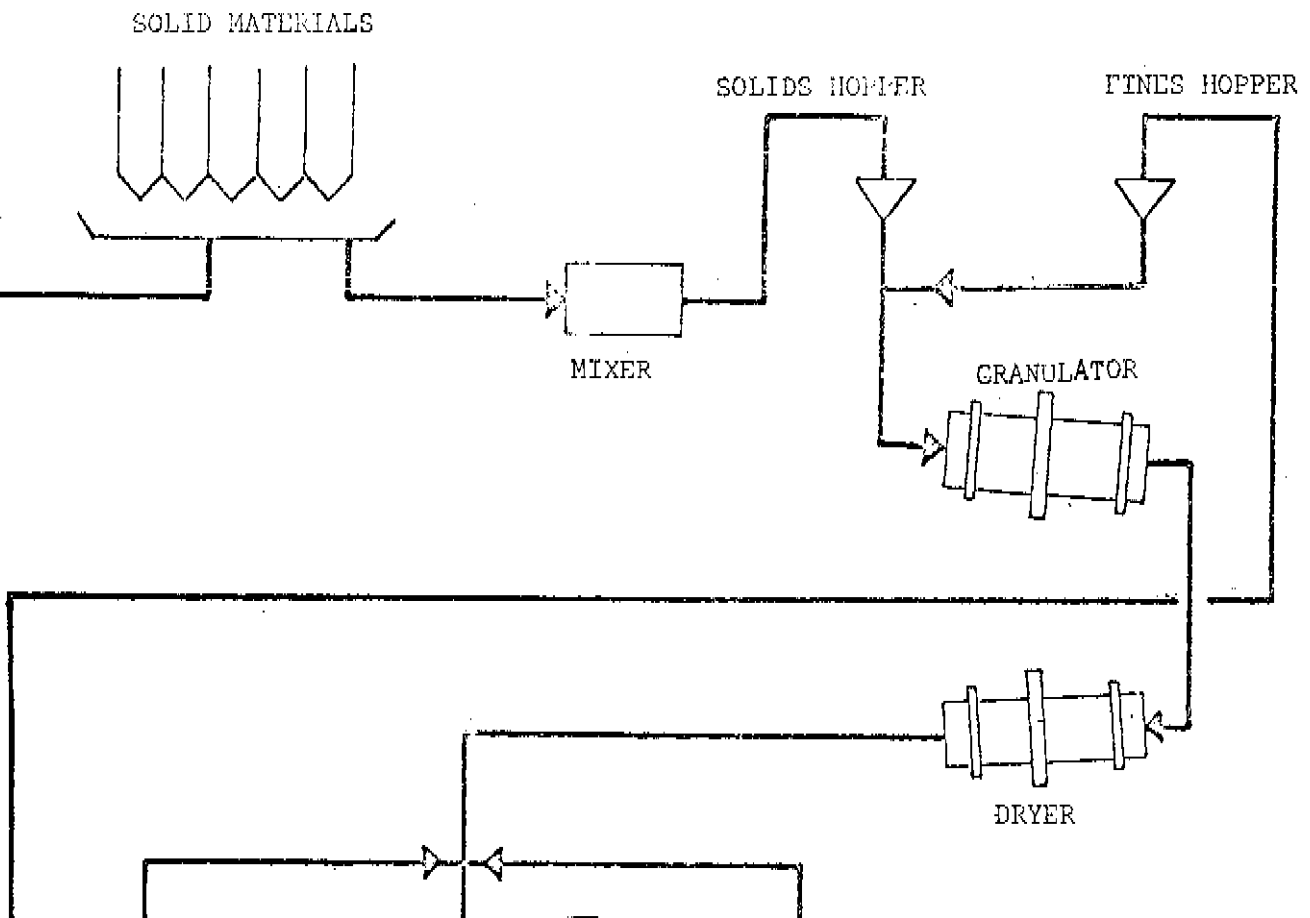
1. Have you noticed any degradation in quality, particularly loss of nitrogen after storage in bags for a few months.
2. If you use the dryer, do you use it with a large amount of fuel or do you use only the monohydrate sulphuric acid to evaporate the water as is necessary. Sulphuric acid reacts with ammonia to give sufficient heat to evaporate water.
3. If, as is the case in Portugal, you may only have 25% of your nitrogen content in the form of ureic nitrogen as opposed to nitrogen based on nitric acid, what reactions are there from your agronomic services?

Mr. OLIVARES:

1. As to the first question, on losses of nitrogen in the products to which we referred, we watch closely the composition of products after manufacture and on dispatch; deterioration is not marked. However, the problems which do tend to occur are those of agglomeration or compacting. As we have screening facilities prior to dispatch, the rejected fines may represent a deterioration of the grade.
2. As regards the driers we use, despite the considerable differences between the three installations, the same fundamental system is used in all cases. The fact is that a granulated product containing urea has to be dried with a large quantity of air, at a low temperature; then the reaction between the ammonia and the sulphuric acid within the granulating mass, at a temperature of 80/90°C, provides, with a large amount of air, optimum drying conditions. In addition, plants 2-C and 1-A have an aerothermal unit and fuel oil burner respectively. I repeat that the temperature must in all cases be as low as possible, and achieved within the granulating mass.
3. As for the Portuguese standard limiting the content of ureic N₂ in fertilizers, I believe this to be a purely agricultural matter, on which I am not qualified to speak, and at all events the question of substituting

ammonical or ureic nitrogen for nitric nitrogen is possibly one of the trickiest problems in nitrogenated fertilizers at the present time. I therefore refrain from answering this question.

APPENDIX 4B



to quality. So far in our work the chloride ion has not been detected as responsible for quality deterioration. The difference is that with total solid systems the water available for ionisation reactions is absent so that ammonium chloride is not formed.

The paper has a lot of data relevant to those with similar plants and I thoroughly admire the style and thoroughness of the information. Maybe I am over-critical in saying that you are solving problems on these plants which progress has eliminated. We still have problems with urea and high analysis granular compounds but they are different. The principal difference in the U.K. and many other countries is that we believe single superphosphate is a dying commodity and are facing up to this. Perhaps the authors would like to comment on this?

Mr. OLIVARES: I am grateful for Mr. Sharples' words and for the interest he has shown in this work by making some points which I shall endeavour to answer as best I can.

First, this report states that the capacity of our plants for producing urea fertilisers is 12, 10 and 22 tonnes per hour respectively, but we also say that these figures have been obtained as averages, with various grades. There are in fact formulations in which we use 35% urea and a 1:1 recycling ratio in plant 1-C. Specifically, the proportioning of the 20.10.10 and 20.10.10-S (with potassium sulphate) involves the introduction of 350 kg urea per tonne, and there is a considerable difference between the two products, which are differentiated by the form of addition of the K_2O . That is, 35% urea is introduced in both forms, but the "S" formulation can be made more easily and with fewer problems in the drier and, generally, with dust in the installation.

It is important to consider, also, that this plant 1-C, because it operates at a very low granulator temperature, which may perhaps be surprising, completes the process in the first section of the drier. Again, there are formulations, such as the 26.16.0 with 45% urea, where granulation can be observed until after the drier stage, a feature which can moreover be noted in many plants. I do not think plant 2-C is a complicated plant - quite the reverse, since it has no drier or coater. However, I suppose the fact that the recycling of fines takes place both to the drier and the reactor/ammoniator represents an apparent complication, but this to some extent makes good granulation more readily

obtainable, with better possibilities of regulation. It should not be forgotten that this is a plant which has been used to make urea fertilizers, but was not designed for the purpose. As for the maximum quantity of K_2O , added as chloride, we find not more than 16% advisable, and K_2SO_4 should be used if richer fertilizers are required. We did this, as we mention in the report, to keep up the quality and operation of the plant.

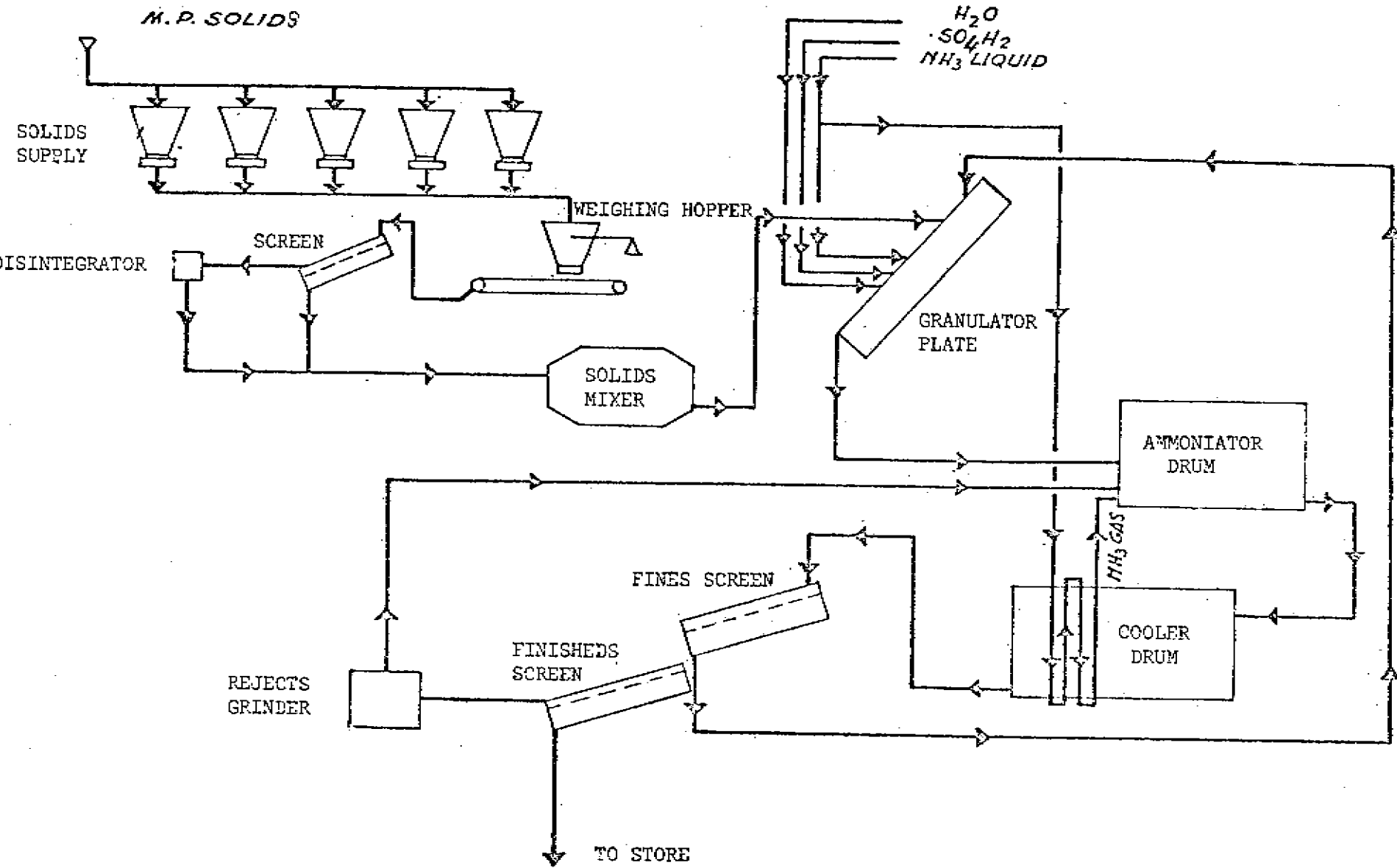
As to the reason for separating the fines before the coarser grains, we are in complete agreement, and this is one of the modifications we had thought of introducing, although it does not prevent us from thanking Mr. Sharples for the comment.

In fact the screening design of plant 1-A is quite adequate and indeed can operate on occasions with a single line of screens. It should be borne in mind that it is an installation designed to make fertilizers other than those we are now dealing with. Specifically, when handling ureic fertilizers, this plant can operate with a single line of screens, while the other is being cleaned.

Finally, we agree that we should not sacrifice the quality of the products to the cost of the raw materials, but in the case of our country, with a very strong sulphuric acid and superphosphate industry, we thought it worthwhile to continue using the latter provided the quality of the product did not suffer. There are also agricultural reasons, such as the contribution of sulphur to the NPK fertilizers. And finally there are economic reasons, since most of the superphosphate we use is produced in our own plants, with the consequent saving in transport of P_2O_5 . Apart from the above, as we said in the report, the P_2O_5 in our formulations derives mainly from ammonium phosphate.

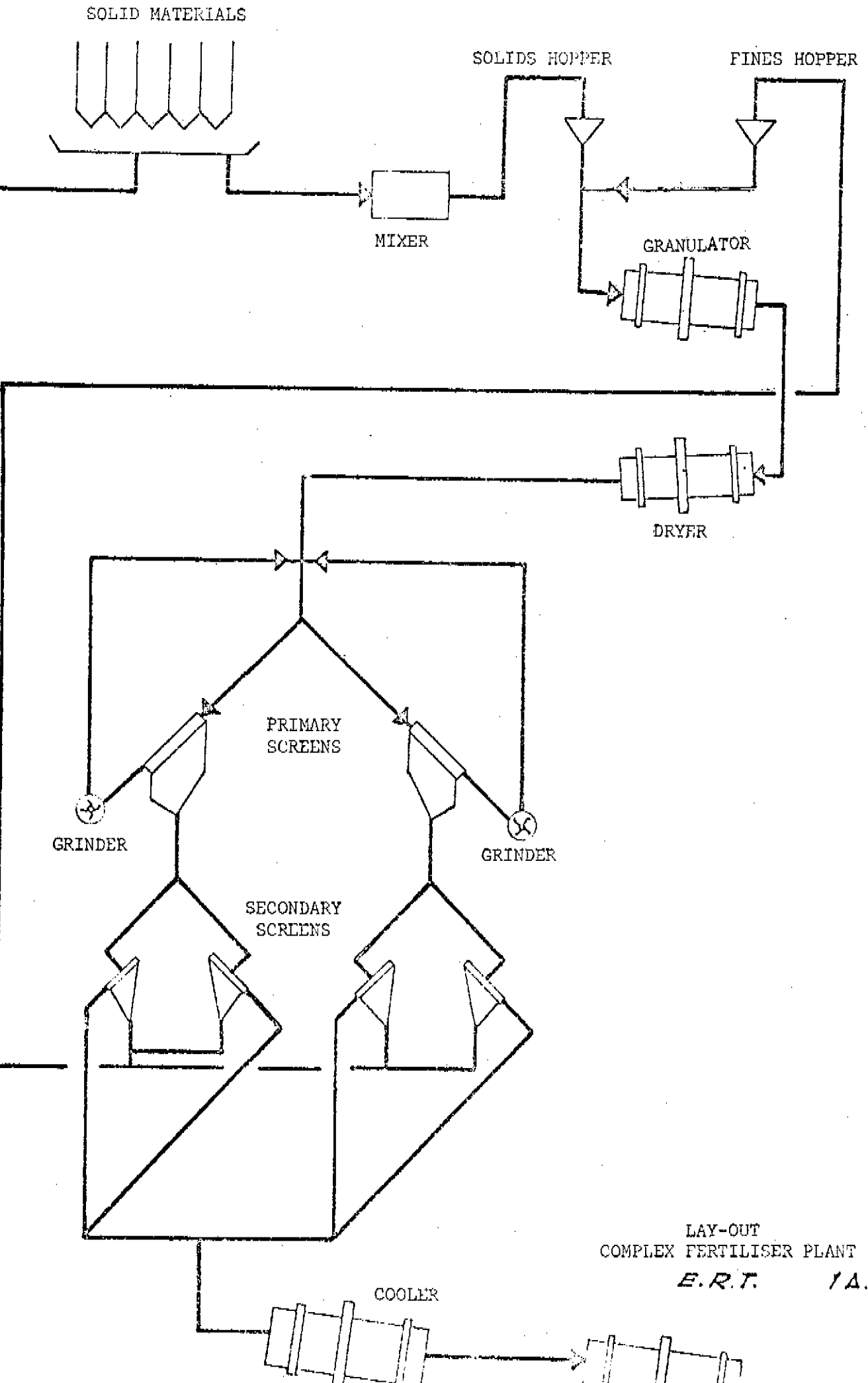
With reference to the influence of chlorides on the quality of the fertilizers we make, clear indications of compacting have been detected; this disappears to the extent of 80-90% with the use of potassium sulphate.

We must admit that plants 1-C and 2-C are obsolete, since granulated urea fertilizers appeared after they were designed, but the essence of our work has in fact been to use them successfully. Our plant 1-A is that of most recent construction and technology; the only change is that the operating manual did not provide for the manufacture of urea fertilizers, a development which we have achieved separately.

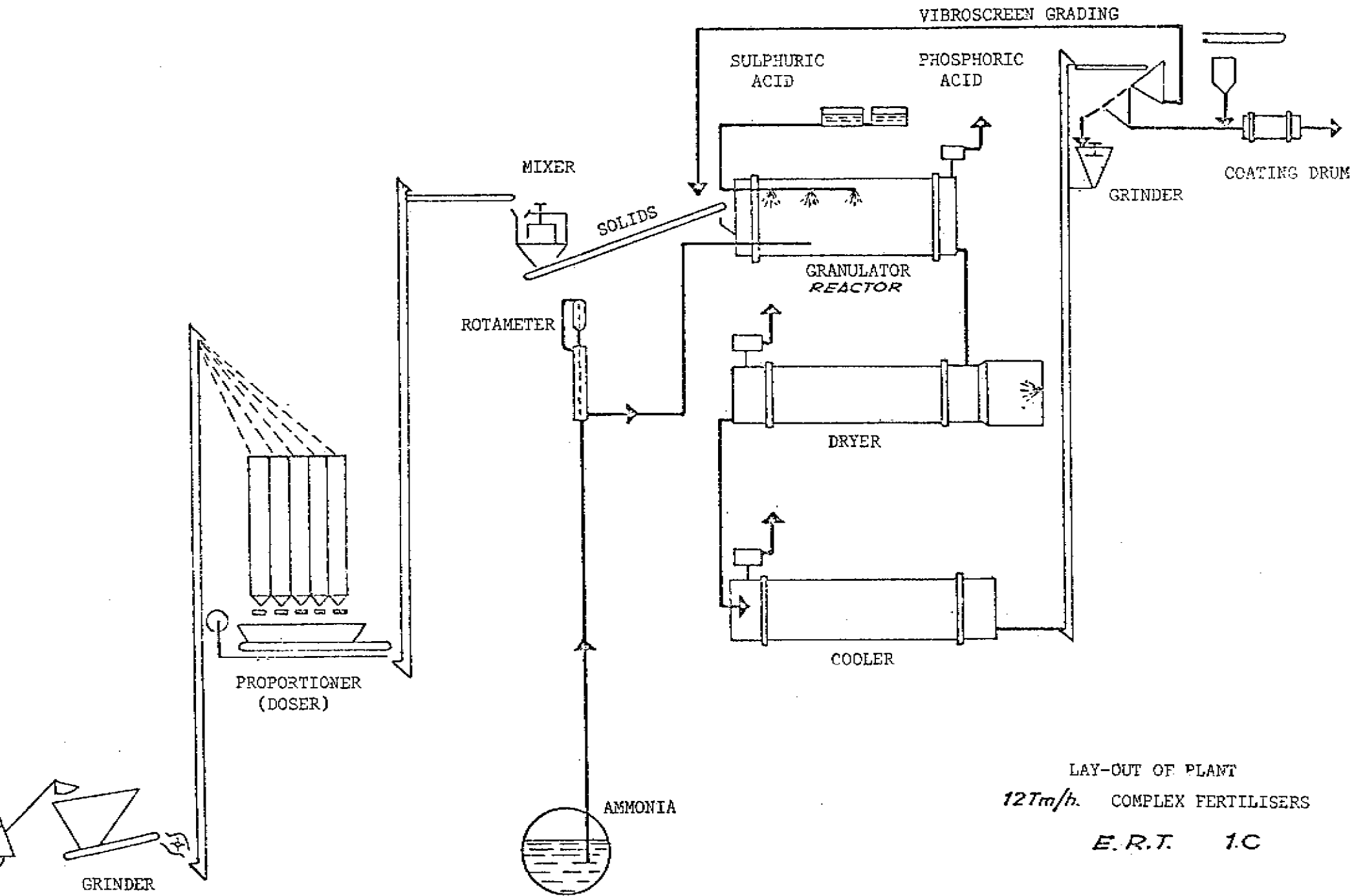


APPENDIX 1A

MANUFACTURING LAY-OUT
COMPLEX FERTILISERS



LAY-OUT
COMPLEX FERTILISER PLANT
E.R.T. 1A.



LAY-OUT OF PLANT
 12T/h. COMPLEX FERTILISERS
 E.R.T. 1.C