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SOME EXPERIENCES IN THE PRODUCTION OF NPK PRILLED FERTILISERS AT BARTON-ON-HUMBER, ENGLAND

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INTRODUCTION

The pattern of demand for fertilisers in the U.K.in the early 1960's indicated a growing demand for highly concentrated water soluble compound fertilisers, and for straight nitrogen in the form of ammonium nitrate.

At that time our Company operated a number of granulation plants strategically located in Northern England. To meet the anticipated future demands, plans were prepared for centralisation of production of N.P.K. fertilisers using ammonium nitrate as a major constituent.

Barton-on-Humber, Lincolnshire, was chosen as the site most conveniently located to supply our main sales areas.

Considerable time was spent in examining the various types of processes used throughout Europe and America, and finally the decision was made to build a plant based on the prilling process developed by the Stamicarbon Division of Dutch State Mines.

The main reasons for this decision were:-

1. Solid ammonium nitrate was also required, and the prilling process could be used to produce this, and also a range of N.P.K. compounds on one plant.
2. Capital requirements were lower than other possible alternative processes.
3. Operating costs were lower than the alternative processes.

4. Elimination of a drier greatly reduced hazards with products based on ammonium nitrate. In addition there are no problems with gaseous effluents.

PROCESS DESCRIPTION

Figure 1 shows a schematic flow diagram. It will be noted that the P_2O_5 is derived from 50% P_2O_5 wet process phosphoric acid. This is necessary to meet U.K. demand for 95% water soluble P_2O_5.

The nitric acid is produced at 57%-58% HNO_3.

Figure 2 shows a simplified flow diagram of the units which are used to produce concentrated ammonium nitrate, or ammonium nitrate/phosphate, solutions.

The ammonium nitrate neutraliser is a thermal circulatory system, working at about 4 atmospheres absolute, into which ammonia gas is fed at a pre-determined rate to react with nitric acid. The pH controller is set in the pH range 5-6 and controls the rate of addition of the nitric acid. The product is approx. 70%-74% ammonium nitrate which is concentrated to 90%-94% in the first stage vacuum evaporator, where the calandria is heated by exhaust steam from the neutraliser. Any ammonia carry-over is recovered from the condensed steam and recycled to the neutraliser. The subsequent routing can be by either of two ways, depending upon the desired end-product.

(a) Ammonium Nitrate Prills

For this the 90%-94% ammonium nitrate is pumped to a second stage vacuum evaporator operating at about 0.03 atmospheres absolute at the top of the prill tower where it is concentrated to 99.6%-99.8% solution. This is fed to a rotating perforated bucket from which droplets fall against a flow of air to the tower base where it is recovered as solid prilled product.

(b) N.P. or N.P.K. Compounds

The 90%-94% ammonium nitrate solution is mixed in a predetermined ratio with 50% P_2O_5 phosphoric acid and again neutralised with ammonia to give a mixed ammonium nitrate/phosphate solution. Again the pH is controlled to approximately 2.8 - 3.3.

This solution is concentrated in a thermal circulatory vacuum evaporator, by means of a steam heated calandria section to reduce the water content to 0.5%-1.0%. The concentrated N.P. melt is pumped to the top of the prill tower where the required amount of hot ground potash is added and mixed, prior to feeding to the prilling bucket.

Again the solidified droplets are collected at the base of the tower.

For either type of product the dry prills are cooled, screened, and given an anti-caking treatment before conveying to the bulk product store.

Figure 3 shows the flow of solutions at the top of the tower, and also the method of heating and transfer of the ground potash, and recovery of solid materials from the base of the tower.

The oversize material from the screens is crushed and recycled. The fine material is recycled to the NP solution tank where it is dissolved and pumped back to the potash mixing vessel at the top of the tower.

All products are packed in 1 cwt. (50 kg.) bags. The bags are of polythene of 8 thou thickness (200 micron).

The source of K_2O can be potassium chloride or potassium sulphate.

PRODUCTS RANGE

Development work is still proceeding to extend the range of N.P.K. products but the following have been successfully produced and sold.

Ammonium Nitrate - 34.5% N.

	%N.	%P_2O_5	%K_2O
N.P.K. Compounds	28	14	0
	25	9	9
	22	11	11
	20	14	14
	17	17	17
	15	15	19

Products between these ratios can be produced, but these meet the current general demand in the U.K.

Products containing higher proportions of P_2O_5 or low nitrogen are more conveniently produced on an adjacent more traditional granulation plant.

AGRICULTURAL CONSIDERATIONS

Prilled products have been well received by farmers who have had no difficulty in handling the slightly smaller particles (i.e. 1.4 to 3.4 mm, as compared with the usual 2mm - 4mm granular products).

Satisfactory distribution patterns have been obtained using combine drills and rotating disc or oscillating spout distributors.

TECHNICAL PROBLEMS

The prilling system using a centrifugal bucket as the basic prilling equipment has been developed and used by D.S.M. for making limed ammonium nitrate for many years. The same plant at Geleen in the Netherlands has also been used to make N.P. prilled products and, to a lesser extent, N.P.K. products based on the use of N.P. feed stock melts originating from nitric acid attack of phosphate rock and potash in the form of potassium sulphate only.

The extension of this process to enable it to be used with wet process phosphoric acid, ammonium nitrate solutions and potassium chloride was the subject of small scale pilot operations by the Stamicarbon Division of D.S.M. Full plant scale operation on the D.S.M. plant was not possible for various reasons and it was anticipated, therefore, that in starting up the Barton-on-Humber plant, technical problems would be experienced and we discuss some of these in detail.

Ammonium Nitrate Neutraliser

This equipment has always operated satisfactorily processwise.

However, some problems arose due to the original type of construction and also from corrosion of some components.

The neutraliser is constructed from a number of sections bolted together. Originally, tafted flanges were used but soon, during the commissioning of the plant, the tafts cracked through where they flexed over the edges of the carbon steel backing flanges and serious leakage took place. Also corrosion of the taft faces took place. Fig 4a/b.

After continuing trouble with these tafted joints they were removed and replaced by welded-on solid stainless steel flanges and this action with the use of fluon wrapped compressed asbestos joints has resulted in a very great improvement.

Corrosion of certain sections of the neutraliser has also been a cause for concern and modification. The ammonia sparger, in particular, had a very short life. Various types of stainless steel have been used for the more seriously attacked components but even the most suitable steel require repair and replacement at fairly frequent intervals. An experimental ammonia sparger made from special material has now been in continuous use for nine months and is still in very good condition. This appears to be a satisfactory solution to the problem.

Ammonium Phosphate Neutralisation

The 92% ammonium nitrate solution is premixed under ratio control with phosphoric acid and the combined "mixed acid" is fed to the N.P. neutraliser. Some corrosion problems have been associated with this mixed acid such as, for example, the severe attack which takes place in the fume extraction vent pipes made from 347 stainless steel. In the mixed acid buffer tank itself fairly rapid weld decay has taken place but this is observed to be restricted to the vapour space in the upper part of the tank, and etching of the plate has been quite mild.

This neutraliser is a vessel about 10 cubic metre size in which the mixed ammonium nitrate-phosphoric acid, fed in by a centrally placed dip pipe is neutralised to pH 2.7-3.0 with ammonia vapour fed in by four pipes. The pH is checked regularly by diluting a sample with an equal volume of water and measuring the pH on a bench meter. A pH control system based on continuous measurement of the pH by a flow cell incorporating an antimony block and remote calomel reference electrodes was originally installed on the overflow line from the neutraliser. This latter system had been found satisfactory on similar material in other plants but failed to give reliable results in this installation. The failure is thought to be due to the rapid build-up of a thick antimony oxide layer, the temperature and concentration of the melt being higher than on previous applications.

The heat of reaction is sufficient to concentrate the solution from about 85% to 94-95%, the exact figures being dependent on the composition. The boiling point is very close to the temperature at which crystallisation commences. This is indicated in Figure 5, which is for a melt of total N/P_2O_5 ratio of 1.0, the worst case in our range of compounds.

Severe "foaming" was experienced in early commissioning and an anti-foaming agent was added with the mixed acid via a metering pump with suitable controls to avoid the possible hazards associated with the introduction of organic materials into the system.

Ammonium Nitrate/Phosphate Evaporator

The evaporator performance was not satisfactory initially. The design throughput could only be achieved with a higher moisture in the product melt than was acceptable to obtain a satisfactory non-caking product. Alternatively, the required concentration could be achieved but only at the expense of serious reduction of throughput - about 60% of design. By reducing the hydrostatic pressure on the calandria we were able to obtain throughputs of 100%-125% of design rate at the required moisture of 0.7%-0.2%, depending on type of product.

During the early commissioning period it was observed that scaling of the calandria tubes was quite rapid and although boiling with a diluted solution of nitric acid restored the heat transfer rates it was feared that the frequency of descaling may limit total production rates. However, it has been found that, since making the above changes, runs of 3 to 4 weeks at design throughputs can be achieved between descaling and that the latter operation can be completed in 8-12 hours. It is quite clear to us that scaling only becomes a serious problem during discontinuous operation and is probably due to the dilution which takes place while maintaining circulation and temperature by using direct steam injection when the plant is stopped.

Potash Addition

The potassium salt, chloride or sulphate, is required in a hot, finely divided form. The general principles of the preparation of the potassium salt are indicated in Figure 3. This gives an aerated material which, as might be expected, is difficult to meter. The present volumetric control, based on screw feeder, is reasonably satisfactory but we hope shortly to develop an improved technique to give better control of potash addition. Frequent potash analyses are carried out on the product, using a rapid method for determining potash, which we have developed, using a gamma-ray absorption meter.

The raw potash is first screened to remove foreign matter, such as wood, which could cause blockage in the prill bucket. The ground potassium chloride which we have used satisfactorily is in the particle size range 45% greater than 100 microns and 85% greater than 45 microns. It is fed at a temperature of 140° C-175° C.

The Prilling Operation

In order for a prilling operation to be successful, certain requirements have to be met. These are :

1. The material to be prilled must be of a homogeneous consistency so that it will flow freely through the holes of the prilling device without blockage of these holes.
2. Any increase in viscosity which takes place after mixing, as a consequence of chemical reaction, must be taken into consideration in the design of the prilling device.
3. The height of the prilling tower and the possible

3. cooling which can take place during the free fall of the prills must be correctly related to the freezing temperature of the prilled melt and the maximum size of the prill droplets.

It was in the prilling operation that we overcame some of our more difficult problems in establishing the above requirements.

The product from the prill tower initially was not composed entirely of free flowing prills but included quantities of prills cemented together into agglomerates by material which had obviously reached the base of the tower in a molten condition. The magnitude of this is illustrated by the fact that although the proportion of agglomerates was very variable it could reach 25% of throughput.

Much of the initial trouble was due to intermittent operation of the plant, which allowed the temperatures of the melt and potash to vary outside the required limits. The resulting mixtures had variable physical properties and intermittent overflow of the prill bucket took place.

Subsequently we were able also to relate bucket overflow to poor dispersion of the potash in the melt. The resulting lumps of potash restricted the feed pipe causing wide fluctuations in flows to the bucket.

Much improved dispersion of the potash has been obtained using a new type of mixer. This, together with better control, and continuous operation, has elimminated bucket overflow problems.

Study of Melt Characteristics in Prilling

The following is a resumé of the work which has been done to try and establish the reasons for melt arriving at the base of the tower in a molten condition, a phenomenon which we call "splatter".

"Splatter" has been collected separately and photographs of it are shown (Figure 6). Some droplets are almost completely molten but sometimes there is a solid shell which breaks on impact on the floor. Comparing the mass of "splatter" specimens and single prills, it was found that there were very few prills larger than 3.0 mm diameter and very few "splatter" pieces of mass less than a prill of 2.9 mm diameter. The median prill diameter is usually 2.1 mm with a standard deviation of 0.5 and since the size distribution appears to be a normal one, we can expect a small amount of material greater than 3.0 mm.

The threshold size for droplets which will not solidify can vary with a number of operating conditions such as melt temperature, concentration and composition, throughput rate, ambient temperature, air flow characteristics in the tower, and prill distribution profile across the tower. The last item has been measured for different throughputs and bucket speeds and the results obtained are indicated in Figure 7. At the higher bucket speed, the throughput distribution is less even, but there is much less material in the 2.4 - 3.4 mm size at the higher speed. See Figures 8 and 9.

Changes in operating conditions probably account for variations in the range 0%-5% in the amount of agglomerate greater than 4 mm size, but for a given set of operating conditions, we have found wider variations in agglomeration with different raw materials. We have operated successfully with phosphoric acid produced from Moroccan, Kola and Florida rock and also with thermal acid, but we have had inferior prilling when there was an excessive amount of sludge in the acid. Potassium chloride from different sources has given widely different results even when the particle size distribution was the same. Potassium chloride which was easy to disperse gave better prilling, and there is evidence that the level of organic impurity is a large factor in this. The efficiency of dispersion and the reactivity of the potassium chloride can affect the operation in various ways. Poorly dispersed potassium chloride in the form of minute lumps act in the same way as large single particles in affecting the flow through the prill bucket holes. The amount of potassium chloride which has reacted affects the viscosity of the melt, and therefore the droplet size, and also the crystallisation temperature. The change in crystallisation temperature with the amount of potassium chloride added to a melt total N/P_2O_5, ratio 2:1, is shown in Figure 10. In laboratory experiments, adding the full amount of potassium chloride quickly and cooling rapidly, crystallisation temperatures in the range 112 - 116°C were obtained. X-ray diffraction analysis of rapidly cooled N.P.K. melt indicated that practically all the K was present as the double salt $KNO_3xNH_4NO_3$ and in all respects, the composition was similar to that of well aged plant samples. Our results indicate that in the 22:11:11 system the potassium chloride reacts very rapidly. The crystallisation temperature indicated above, and calculations of the expected temperature at the tower floor of various sizes of drops of melt confirm that the threshold prill size is about 3.0 mm diameter.

Our studies of the various chemical and physical variables which occur in mixing KCl into N.P. melts, in the operation of the prill bucket, and in the crystallisation in the prill tower, have been complex and extensive. This work continues in order that we can completely understand and optimise all of the factors involved.

Recycle of Fines

Fines from the screen and the dust extraction equipment are recycled to the N.P. melt buffer tank which is fitted with a heating and cooling coil and agitator.

After some initial difficulties this operation has worked smoothly, providing that the following conditions are met:-

1. The pH of the melt is maintained in the range pH 3.5 - 4.0 (10% solution) by the addition of gaseous ammonia.

2. The temperature of the melt is maintained at 170 - 172°C by the use of steam and water in the coils.

3. A belt feeder is provided to control the rate of recycle of fines to a preset rate of maximum 10% of throughput. In practice, recycle is much less than this.

4. Certain operational procedures are strictly adhered to, so that fines concentrations and residence times remain within pre-set safe limits at all times.

5. The provision of equipment so that all vessels containing recycled materials can be rapidly quenched with water if the temperature rises above 180°C.

Product Quality

In early days, the product was produced with a moisture content about 0.6% and even after "anti-caking" treatment, problems of caking developed during storage. Progressive reduction of the moisture conten and increasing the pH were significant in reducing the caking tendency and now with a moisture content from the plant below 0.4% and pH 3.5 - 4.0 (10% solution) we have a very satisfactory non-caking product.

Long chain amines dispersed in oil, plus a fine clay, is the standard anti-caking treatment for all our N.P.K. fertilisers.

These products have been readily accepted in markets where larger particle size "granular" products were normally used.

They remain free-flowing, are dust-free, and are spread uniformly by a wide range of fertiliser distributors.

Future Developments

Albright & Wilson Ltd., A.C.C. Division and Stamicarbon N.V. have worked, and are continuing to work, jointly on the development of the process.

From our experiences to date we are confident that we can further improve the process to give higher outputs with further economies.

We anticipate also that we can further increase the flexibility of the process to give a wider range of products, although the limiting factor will always be that of melt viscosity at low total $N:P_2O_5$ ratios, particularly using KCl as the source of potash.

Acknowledgements

The authors wish to thank the Directors of Stamicarbon N.V. and of Albright & Wilson Ltd., Associated Chemical Companies Division, for permission to publish this paper.

DISCUSSION

Mr. T.A. MITCHELL (Albright & Wilson, U.K.) : In choosing the title for our paper we have tried to summarise our intentions. The scope for the paper is a wide one and we have tried to condense 3 years of experience by outlining some of the main problems.

Many of the delegates present have seen our plant and we hope that in this paper we have augmented the knowledge they already had.

We realise that further developments and improved technical knowledge are still to be achieved, and we hope that the later discussion will stimulate our own thoughts for the future.

The U.K. pattern of demand for fertilisers required us to produce straight nitrogen and high nitrogen NPK compounds. Our plant has this flexibility and in addition produces the necessary high water soluble P_2O_5 in the compounds.

Capital and operating costs are relatively low because of the efficient recovery of heat, and low recycle rates which permit the use of small plant items with low power demand.

There is no dryer and hence potential hazards in handling ammonium nitrate are reduced and gaseous effluents are minimised.

Figures 1, 2 and 3 show the process outlines.

Products and Raw Materials

The range of products made to date is listed on p. 3. These are made using 50% K_2O potassium chloride, with the exception of 20-14-14 and 17-17-17 which require about 55% K_2O grade of KCl.

Since the original paper was prepared our Sales Department has requested that we supply an $N:P_2O_5:K_2O$ of 17-17-16 instead of the 17-17-17 given on p. 3 of our paper.

The former can be obtained using 54% K_2O grade potassium chloride.

These formulations are possible because of a polyphosphate formation equivalent to 15 - 20% of the total P_2O_5 at the postulated water contents.

We have not yet attempted to make compounds with $N:P_2O_5$ ratios less than 1.0, because of possible viscosity and crystallisation problems.

The phosphoric acid used has been mainly wet process acid based on Morocco rock, although successful extended runs have been made using acid based on Florida and Kola rocks.

We have made short runs using thermal phosphoric acid without plant problems.

In this latter trial we found that the product (22-11-11 in this case) showed less satisfactory storage properties than when using wet process acid.

We would be interested to hear if any of the delegates have observed similar behaviour.

Corrosion

There have been no serious problems, and those mentioned in our paper have been due mainly to small design errors, or incorrect choice of materials.

pH Control of Neutralisions

We have had no problems in pH control of the ammonium nitrate neutraliser which uses a glass - calomel electrode system on a continuous cooled sampling system for the 72% AN solution.

In the Nitrate Phosphate neutraliser the original antimony-block - calomel electrode system was unreliable although it had been successful on similar applications elsewhere. In practice this was not a major problem although we are currently experimenting with a continuous dilution sampling system which looks promising using glass - calomel electrode system.

Phosphate Evaporator

On p. 6, first paragraph, next to last line there is an error. This should read "0.7%± 0.2%* as the required moisture level (i.e. moisture range 0.5 to 0.9%).

Prilling Operation

This is based on Stamicarbon NV knowledge and an excellent summary has been given by Messrs STEENWINKEL and HOOGENDONCK of Dutch State Mines in a paper to the Fertiliser Society - Proceedings No. 9, May 1969.

Potash Addition

The addition of KCl to ammonium nitrate - phosphate melt has been the subject of much effort by the designers and ourselves.

A very efficient mixer has been devised and together with detailed studies of the various qualities of potash, has resulted in most satisfactory operation.

"Splatter"

On p. 7 we have attempted to define this phenomenon, and the illustrations in Fig. 6 show this very well.

The phenomenon is observed mainly at the periphery of the tower, confirming that it is related to large drop formation.

This material is the major source of oversize recycle and we have been very cautious when returning such solids containing chlorides to the NP melt system, particularly when large proportions of ammonium nitrate are present.

We limit recycle to 8% of throughput after extended laboratory and controlled full scale experiments. This is somewhat arbitrary but biased towards safe operation.

When making 22-11-11, which shows the greatest tendancy to form "splatter", our present recycle rate is 5%.

Conclusion

Although we have met, and overcome, many problems to obtain what we consider to be a very satisfactory process for our purposes, we are aware that a number of questions remain for which we have not yet found full explanations, and we hope that the discussion to follow will help us to answer some of these questions.

Mr. KELETI (Girdler) : We have an excellent paper given by Mr. MITCHELL and co-authored by Messrs FOTHERGILL and KELLY of Albright & Wilson. It gives a success story. I for one appreciate the British understatement used in the title of the paper when it stressed "some experiences" in production of NPK prilled fertilisers at Barton-on-Humber. I do not think I am exaggerating when I say that in recent years success stories have become rather rare in the Western European fertiliser business. The prilling process in the fertiliser industry used to be an art and is now a well defined technique and adopted for ammonium nitrate production. Today, as long as the criteria are well defined for the prilling of ammonium nitrate, plants can be standardised and designs processed with the aid of a computer. Ten years ago most of the plants built used spray headers in the prilling tower, today spin bucket or prill pots are taking over. The necessity of diversification in product formulations, essentially utilising the same production facilities, led to the development of NPKs as prilled products.

After reading the paper, we can see at Barton-on-Humber solutions to a number of problems that existed before. One such problem is foaming after mixing ammonium nitrate with wet process phosphoric acid containing impurities including organic matter. The second stage ammoniation also at times results in foaming by additives. These problems have been apparently solved in production. The operating criteria, such as viscosity, pH and temperature, for melts of different composition, have also been defined for formulations tried so far and I am sure they are going to be defined as the market requires changes in the formulations. A number of corrosion problems, associated with maintenance headaches and causing production interruptions, have also been solved. It is stressed by Mr. MITCHELL in his paper that it is very important to maintain a continuous operation and to reduce interruptions to a minimum.

In order to have Mr. MITCHELL give us some additional information, I should like to start the discussion by asking the following questions :

1. Recycle was mentioned in the ten minutes talk by Mr. MITCHELL and what I should like to know is : the corrected figures are now 8%, what is the % distribution between oversize and fines ? Does it vary with different formulations and the quality of KCl used ?

2. What happens to the oversize or splatter lumps ? Are they returned to the melt or dissolved in reverting solution before further concentration ?

3. Could Mr. MITCHELL amplify on the anti-caking treatment and the use of long chain amines in dispersed oil ?

4. On p. 5, paragraph 1 you refer to special material used for the ammonium sparger employed and you have given to understand that it has been 9 months in operation without changing. Can you tell us the alloy composition for this material ?

I am sure this paper will stimulate a number of questions from the floor. The special techniques and at times costly experience that go into the word "know-how" are evidently very firmly in hand with the Albright & Wilson production people.

Mr. MITCHELL : Your first question concerned the recycle. It was also linked with your second question where you asked about oversize recovery. These two must be linked together. The recycle material consists generally of something like two thirds fine small prills, which are satellite prills from the main prilling operation, and the remainder is the oversize created by the splatter problem. In effect I think you will see from Fig. 3 in our paper that the oversize from the screens which, as I say, is mainly the splatter, is crushed and returned back over the screens, so that ultimately the recycle is all recovered as fines. This is then fed back to the melt storage vessel which pumps it back to the top of the tower into the mixer.

You asked about the effects of different formulations. There is a slight variation and the figure I quoted is for 22-11-11. This is probably the highest recycle, depending on what we are making. Generally one can say that the recycle is reduced with the nitrogen content of the product so that if we have, say 5% with 22-11-11, we would expect about 4% with a 17-17-16.

Your next question was the question of anticaking treatment. I imagine we are using a treatment which is fairly common throughout the whole of Europe of using these long chain amines, which are proprietary materials. The level of application which we apply is something of the order of 0.25% of the total weight of product and to this we add 1 to 1½% ground kaolin.

Your last question was the one that caused me some embarrassment. The reference in the paper to the special material of construction was phrased in that way at the request of the designers, who specifically have asked that we should not say what this material is, and I am very sorry that I cannot answer.

Mr. P. MORAILLON (Péchiney-Saint Gobain) : At the foot of the first page, the author writes that there are no gaseous effluent problems in a prilling process. However, there is a considerable volume of air which can entrain dust and fume at the exit of the tower.

Second question : in the vacuum evaporator for the NP solution, would not forced circulation be preferable to thermo-syphon circulation ?

Third question : does not the pumping of almost anhydrous concentrated NP solution pose safety and corrosion problems, taking into account the presence of potassium chloride carried by recycled fines ?

Fourth question : why is a mixture of 92% ammonium nitrate and 50% phosphoric acid used, which necessitates, according to the plan, a vessel with an agitator, a reserve vessel on the ground and a vessel at the top high up in the factory ? Why are the two liquids not introduced directly into the NP neutraliser ?

Mr. MITCHELL : Your first question concerned the reference on p. 1 to the waste effluent gas treatment and the relationship to the dust and the fume from the top of the tower. In practice the fume from the top of the tower does not contain any dust. This has been checked by the U.K. Alkali Inspectorate and they are quite happy about this. So dust at the top of the tower is no problem. Concerning the gases at the top of the tower, these are very few and they are certainly within the Alkali limits and again the Alkali Inspectorate is quite happy that we do not need any scrubbing treatment in this connection. The only areas where we have to scrub the gases are of course those associated with the phosphoric acid side of the system, where there are the fluorides, in which case there is a very small scrubber which is perhaps the size of this desk.

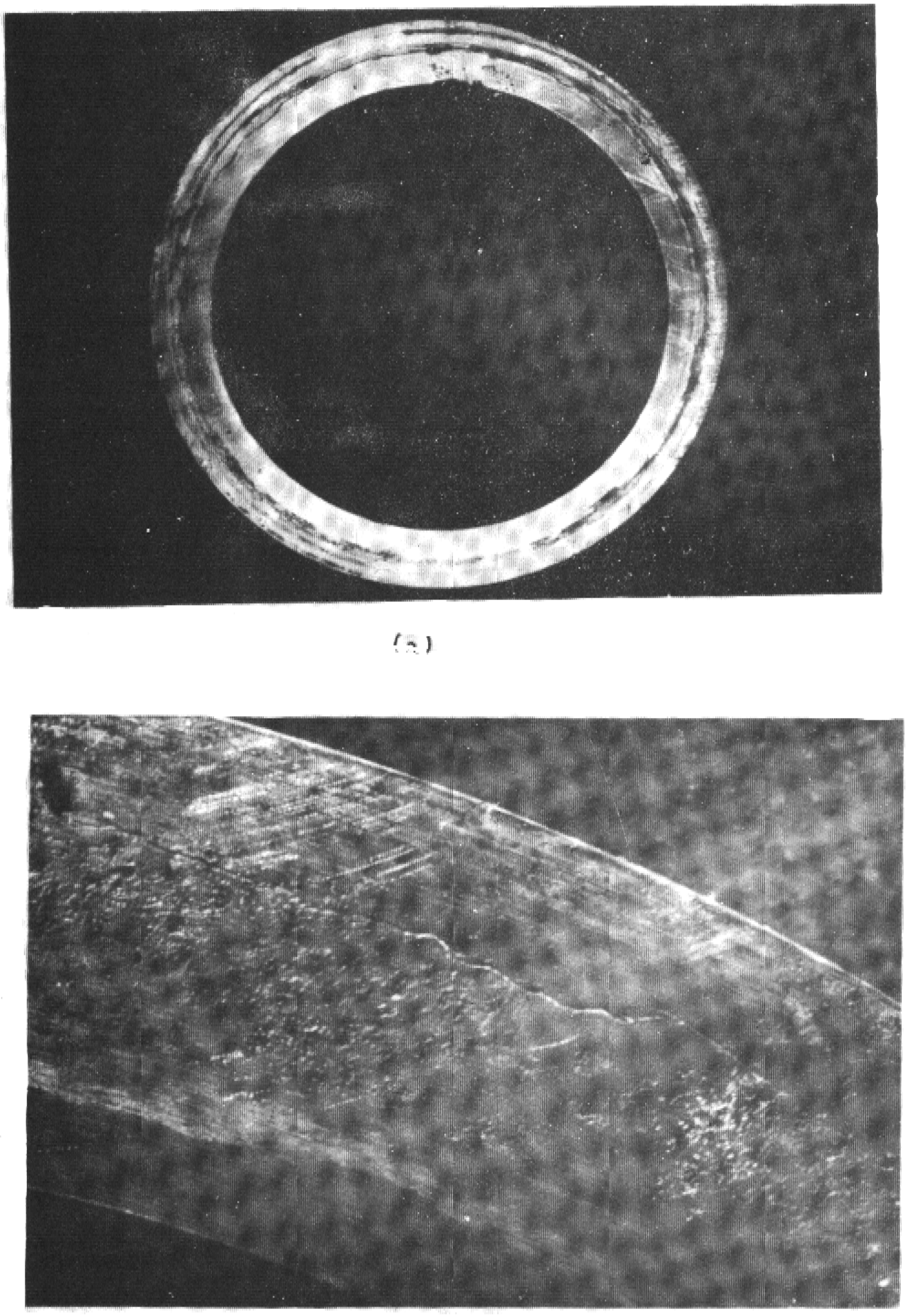
Your next question concerned the vacuum evaporator for the NP melt, and whether forced circulation would be preferable. This is a factor that we did look at very closely in the early days when we were having troubles, which we referred to in the paper, in getting the evaporator to perform at a satisfactory rate, in terms of designed capacity. We found that by raising the calendria section to the highest possible position in the circulating system, the disengagement of the bubbles in the calendria caused sufficient air lift in effect to give us a very effective circulation and, indeed, we are now running it at 125% of designed capacity. We prefer to avoid having yet another pump to handle these lumps, which then takes us to the next question.

The pumping of NP melts and safety problems with the presence of chlorides. We have carried out a long series of tests to establish what the safe limit is for the KCl or the chloride content, and we have set a figure of 8%, a rather arbitrary figure perhaps, but well on the safe side. We have tried very hard to create a condition where we get decomposition of these melts with the chlorides in, and we have found that even if we take the temperature to as high as 200° C in an extended trial, decreasing the residence time by a matter of an hour or two, we were unable to get a condition where the heat of reaction was beginning to rise very rapidly and the temperature was beginning to rise as a result of the heat of decomposition. To do this we actually used steam heat in the coils to raise the temperature artificially and we found that, as soon as we took off the steam heating, the temperature immediately fell back down again. So it was not self sustaining at these concentrations.

Your fourth question was why do we not mix the nitric and phosphoric acids and do it as a single stage operation. If we had only been producing NPK compounds, certainly this is what we should have done. But for flexibility, we wish to have a plant where we can produce solid ammonium nitrate on the same plant, which meant that we had to separate the ammonium nitrate stream. This is the only reason.

Mr. NOIRBENT (Kaltenbach, France) : Why is the spin bucket technique used at the top of the tower ? Evidently it is very advantageous when one has slurries with a high viscosity, but it leads to a dimensioning of the tower which is less favourable compared with more classical prilling of the ammonium nitrate type with sprinklers.

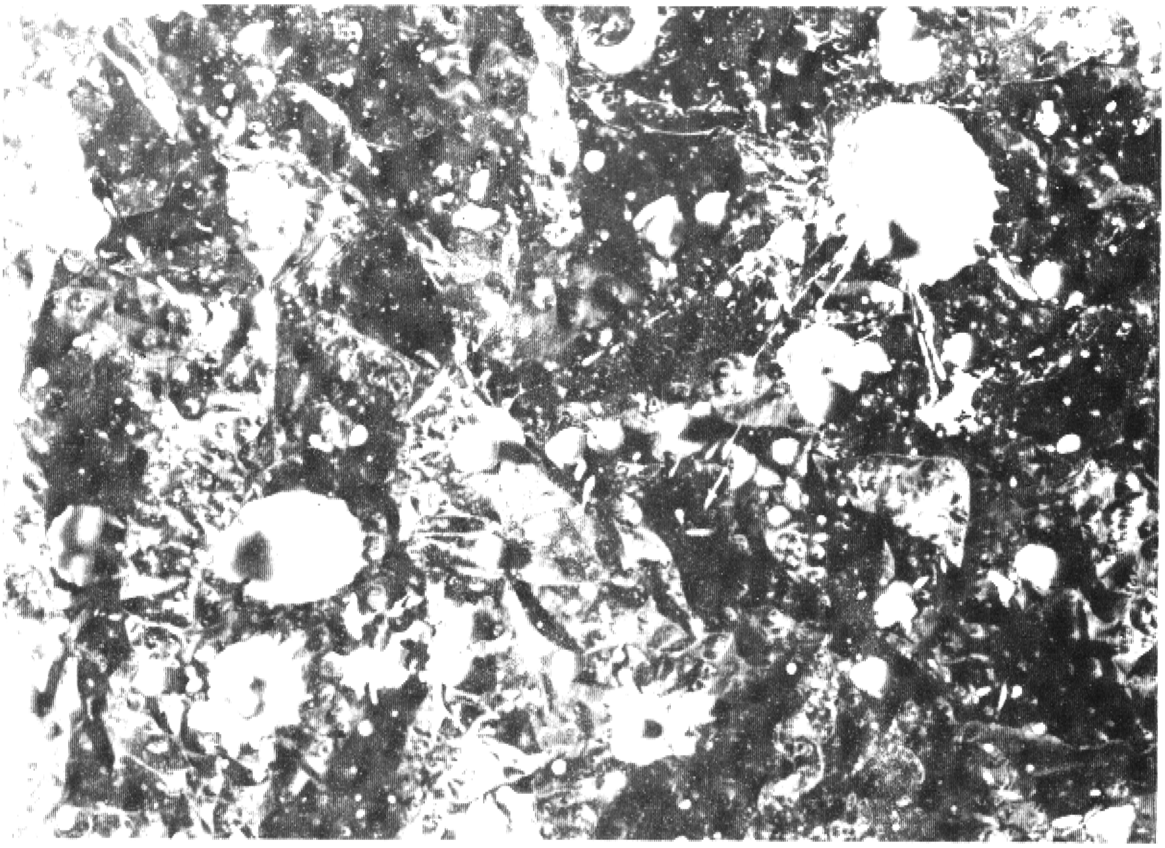
Mr. MITCHELL : Indeed you are correct in that, because we use the spinning bucket system, we do need a taller tower than with the traditional sprinklers. The problem basically is that with some of these NPK mixtures, the resultant slurry or melt has thixotropic properties. The design of the bucket, of the spin bucket that we are using for the NPK melts, incorporates a scraper system inside. Initially the scrapers were designed and installed to ensure that the holes in the bucket were kept clear. The scrapers rotate in the same direction as the bucket, but at a faster speed and the ratio of the speed of the bucket to the speed of the scrapers became a very important factor, not for cleaning the holes but for giving mechanical agitation so that these thixotropic materials could be readily dispersed through the bucket.



(a)

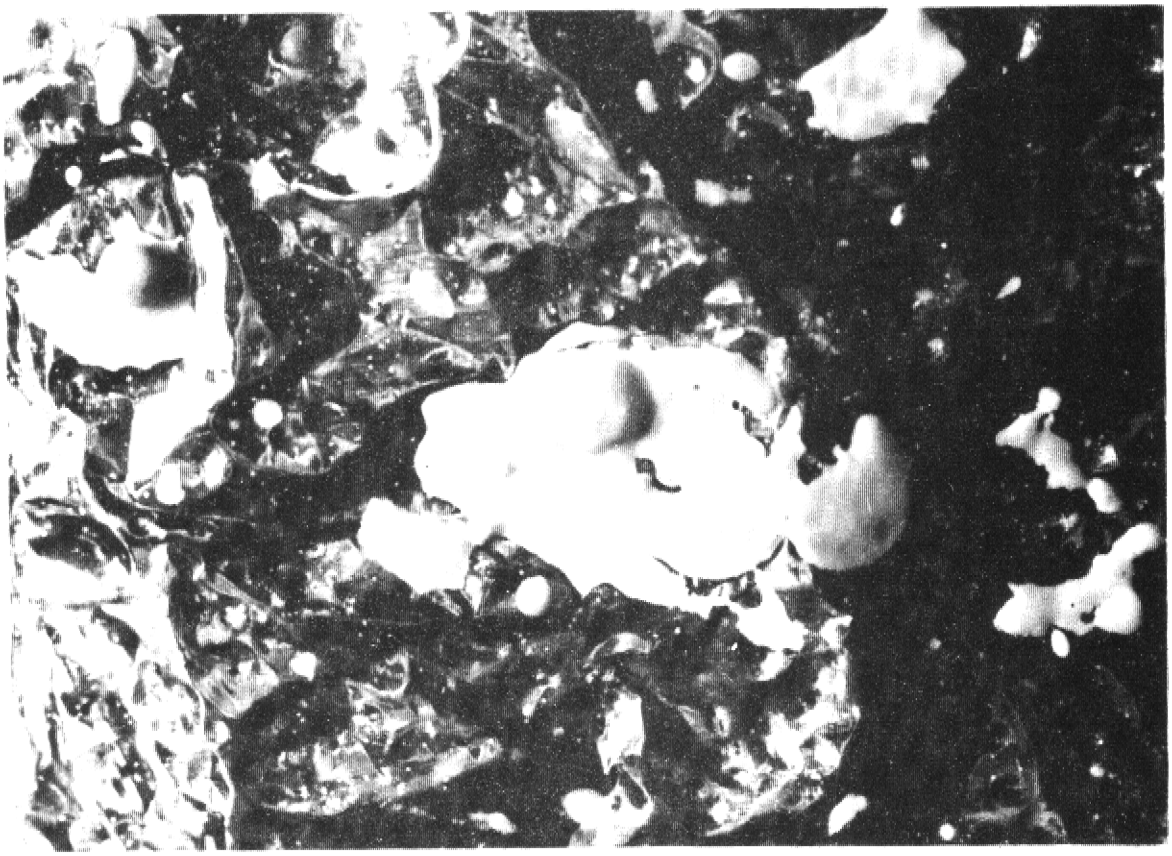
(b)

FIGURE 4. A.N. Neutraliser. Flange corrosion.
Neutraliseur nitrate d'amm. - Corrosion du collet



About 1½ times actual size.

Grossissement env. 1,5



About 3 times actual size.

Grossissement envi. 3

FIGURE 6. "Splatter"

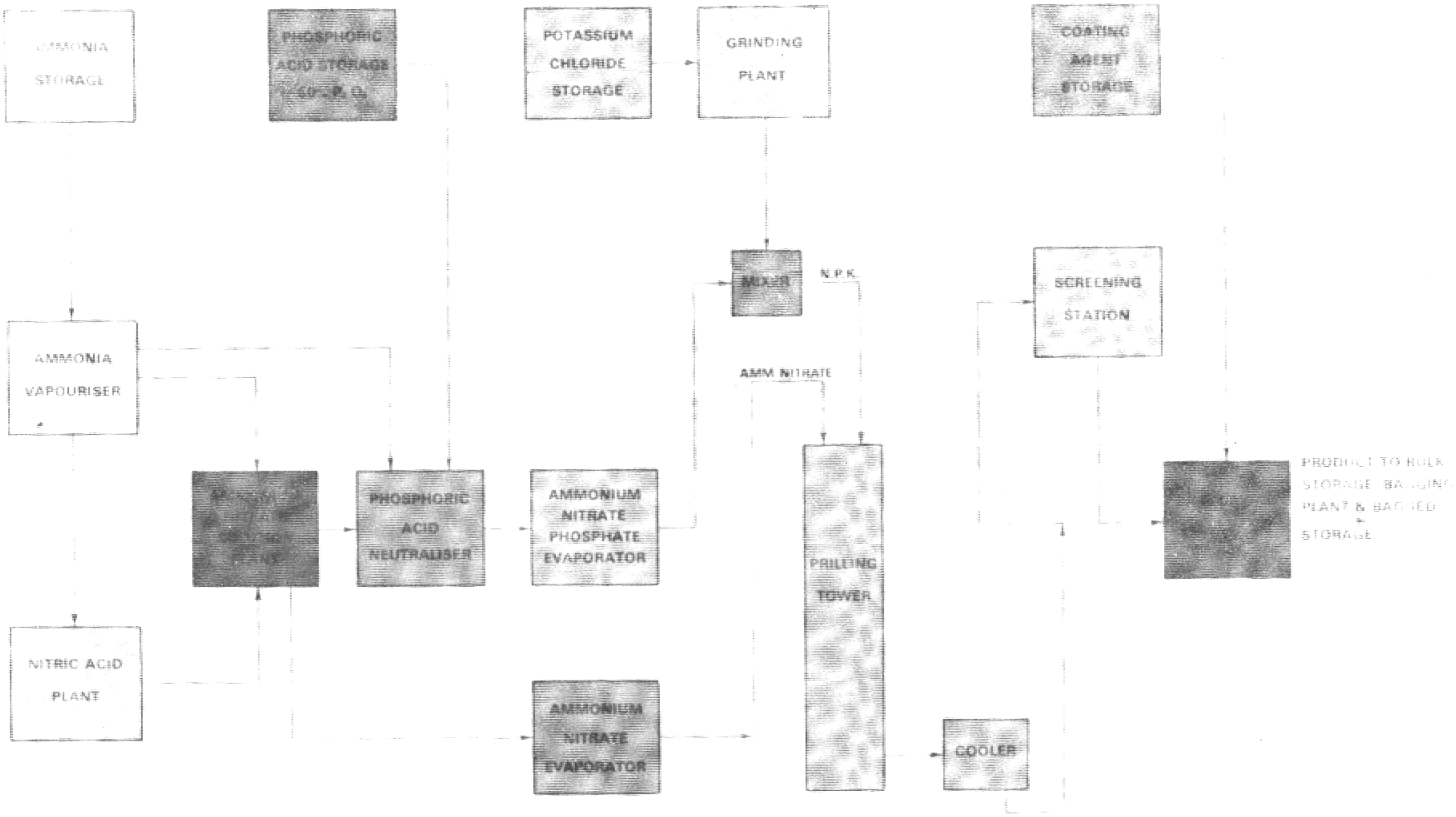


FIGURE 1

FLOW DIAGRAM OF PRILLING PLANT FOR PRODUCTION OF N.P.K. COMPOUND AND AMMONIUM NITRATE FERTILISERS

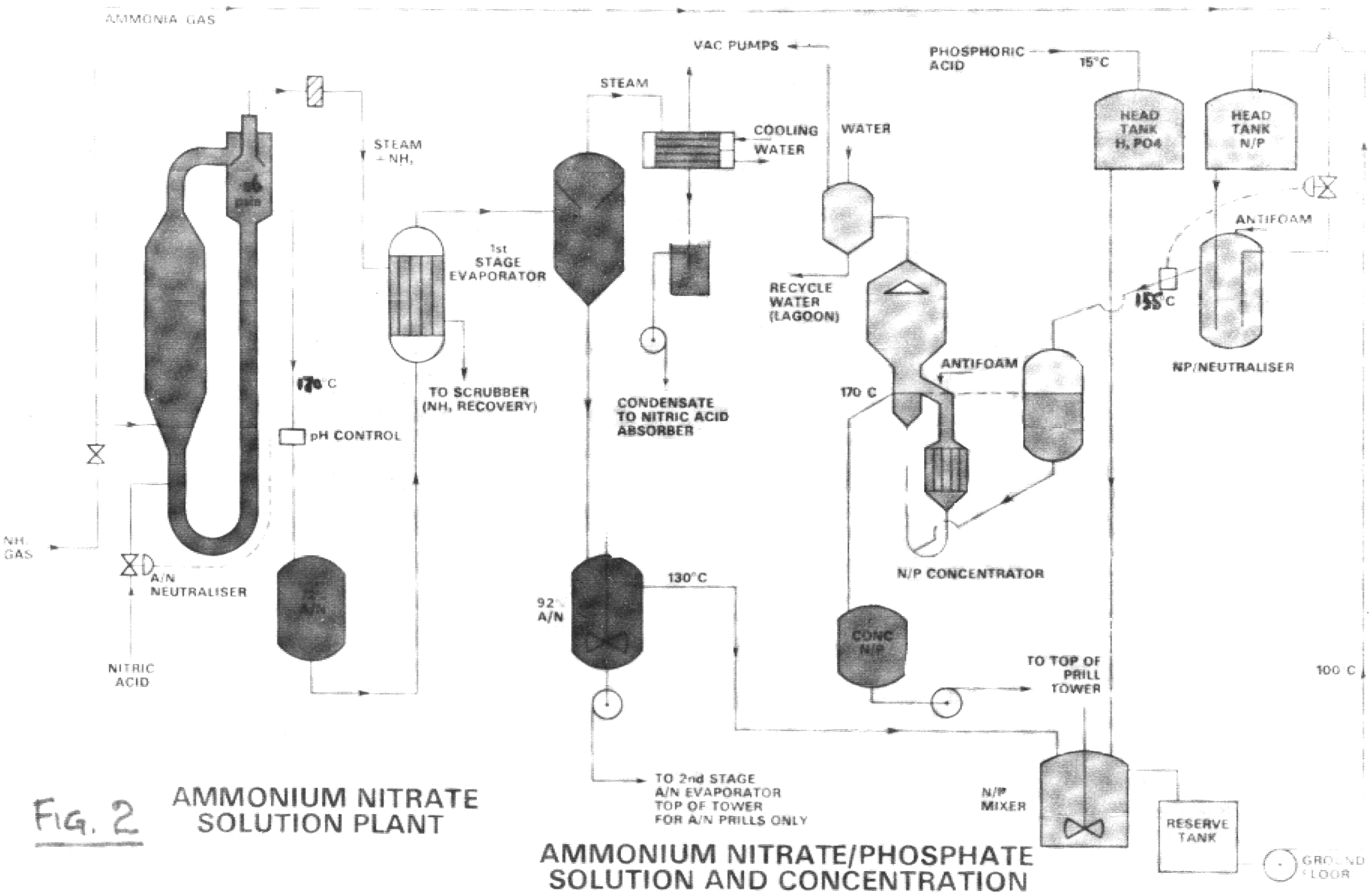
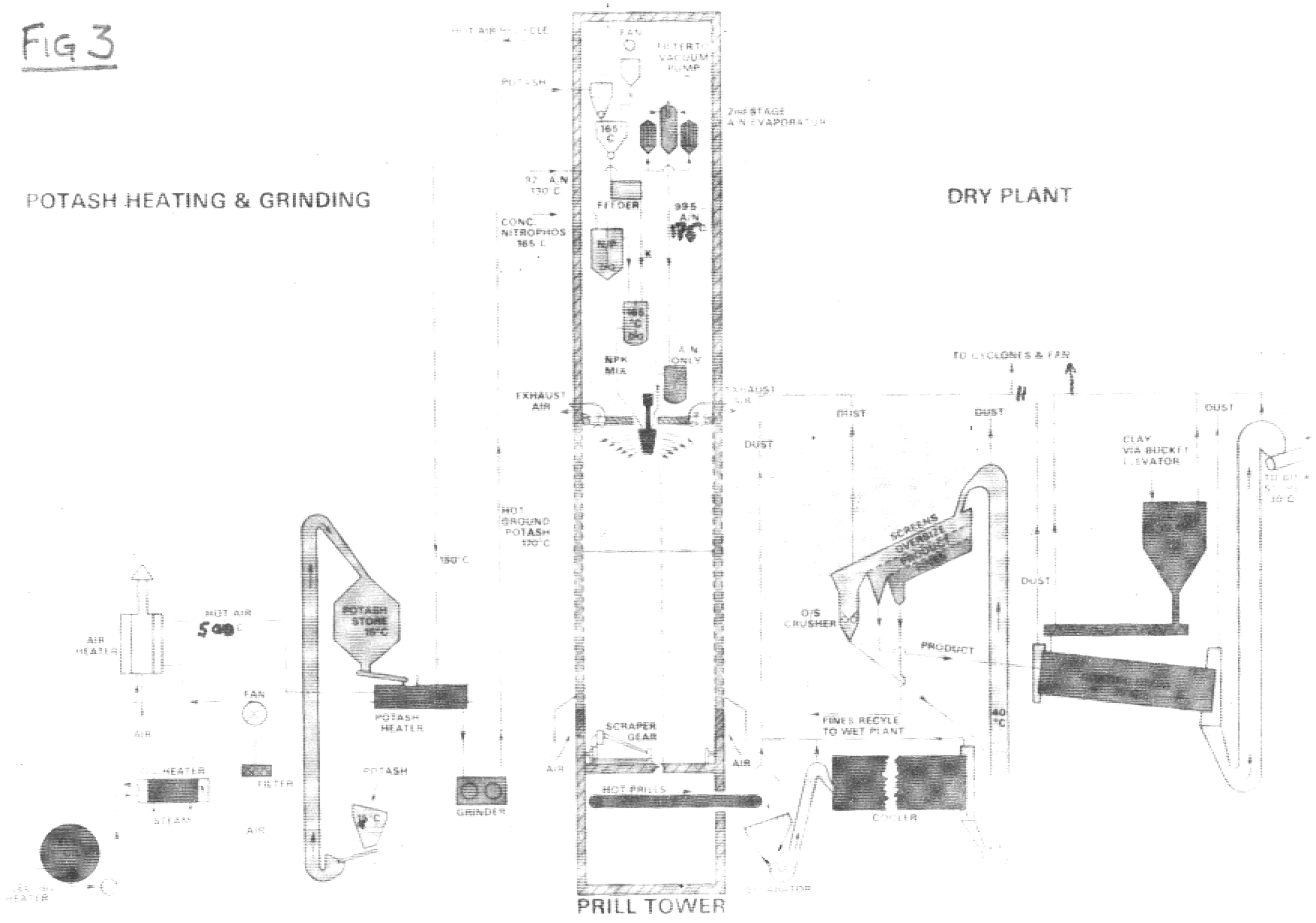


Fig. 2

AMMONIUM NITRATE SOLUTION PLANT

AMMONIUM NITRATE/PHOSPHATE SOLUTION AND CONCENTRATION PLANT

FIG 3


POTASH HEATING & GRINDING
DRY PLANT
PRILL TOWER
HOT AIR RECYCLE
FAN
FILTER TO VACUUM PUMP
POTASH
165 C
2nd STAGE A/N EVAPORATOR
92 A/N 130 C
FEEDER
99.5 A/N 175 C
CONC. NITROPHOS 165 C
N/P
K
165 C
NPK MIX
A/N ONLY
EXHAUST AIR
EXHAUST AIR
DUST
DUST
DUST
DUST
DUST
TO CYCLONES & FAN
CLAY VIA BUCKET ELEVATOR
TO BULK STORE 30°C
SCREENS
OVERSIZE
PRODUCT
FINES
O/S CRUSHER
PRODUCT
40 °C
FINES RECYLE TO WET PLANT
COOLER
SEPARATOR
HOT GROUND POTASH 170°C
150°C
HOT AIR 500 C
AIR HEATER
POTASH STORE 15°C
FAN
POTASH HEATER
AIR
OIL HEATER
FILTER
STEAM
AIR
POTASH
15°C
GRINDER
ELECTRIC HEATER
SCRAPER GEAR
AIR
AIR
HOT PRILLS

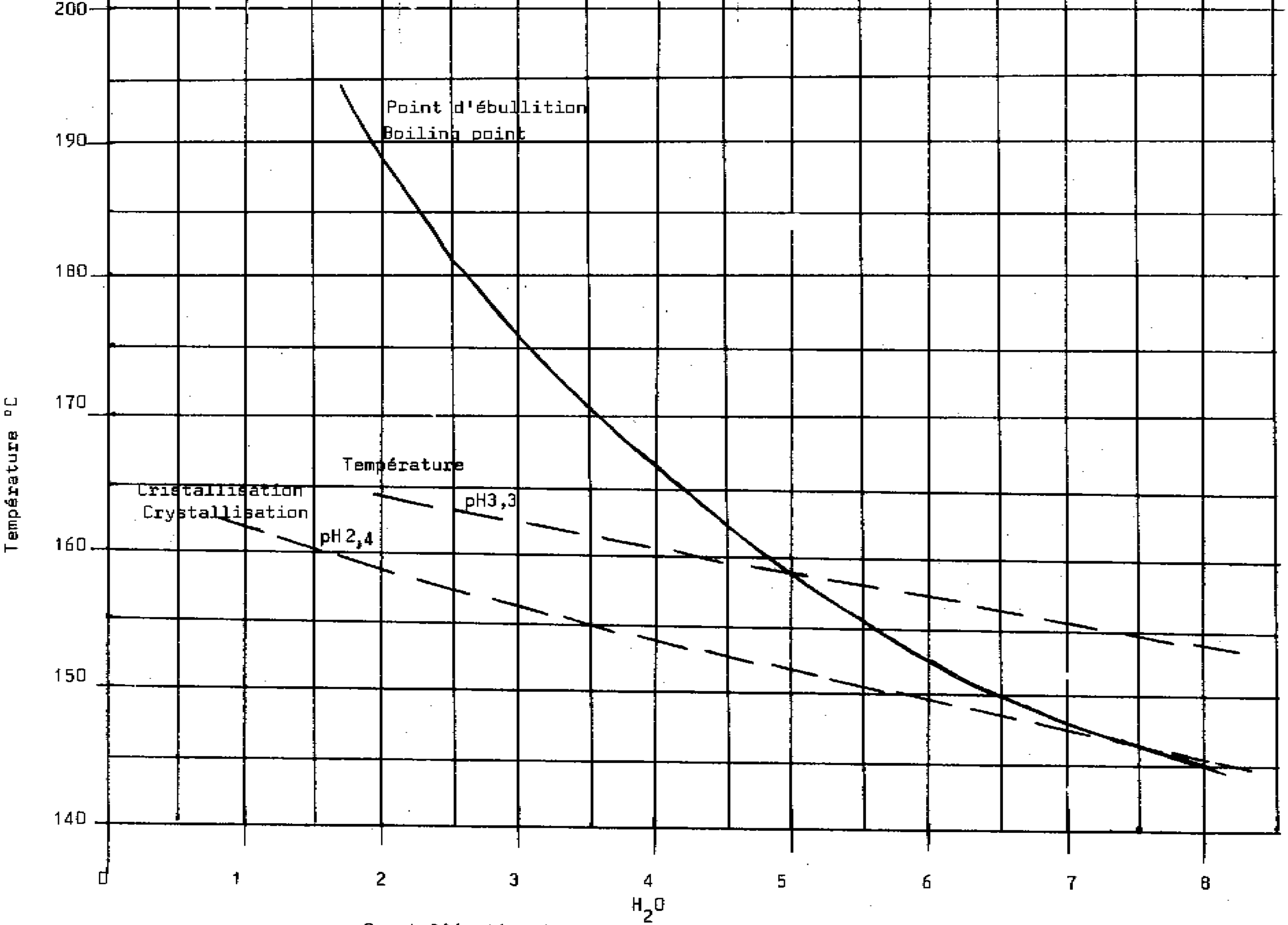


FIG. 5

Crystallisation temperature and boiling point at 1 at.a. - Ammonium nitrate - ammonium phosphate solution, Total N/P_2O_5 = 1.0

Température de cristallisation et point d'ébullition à 1 atm.
Solution nitrate am.-phosphate am., N/P_2O_5 total = 1.0

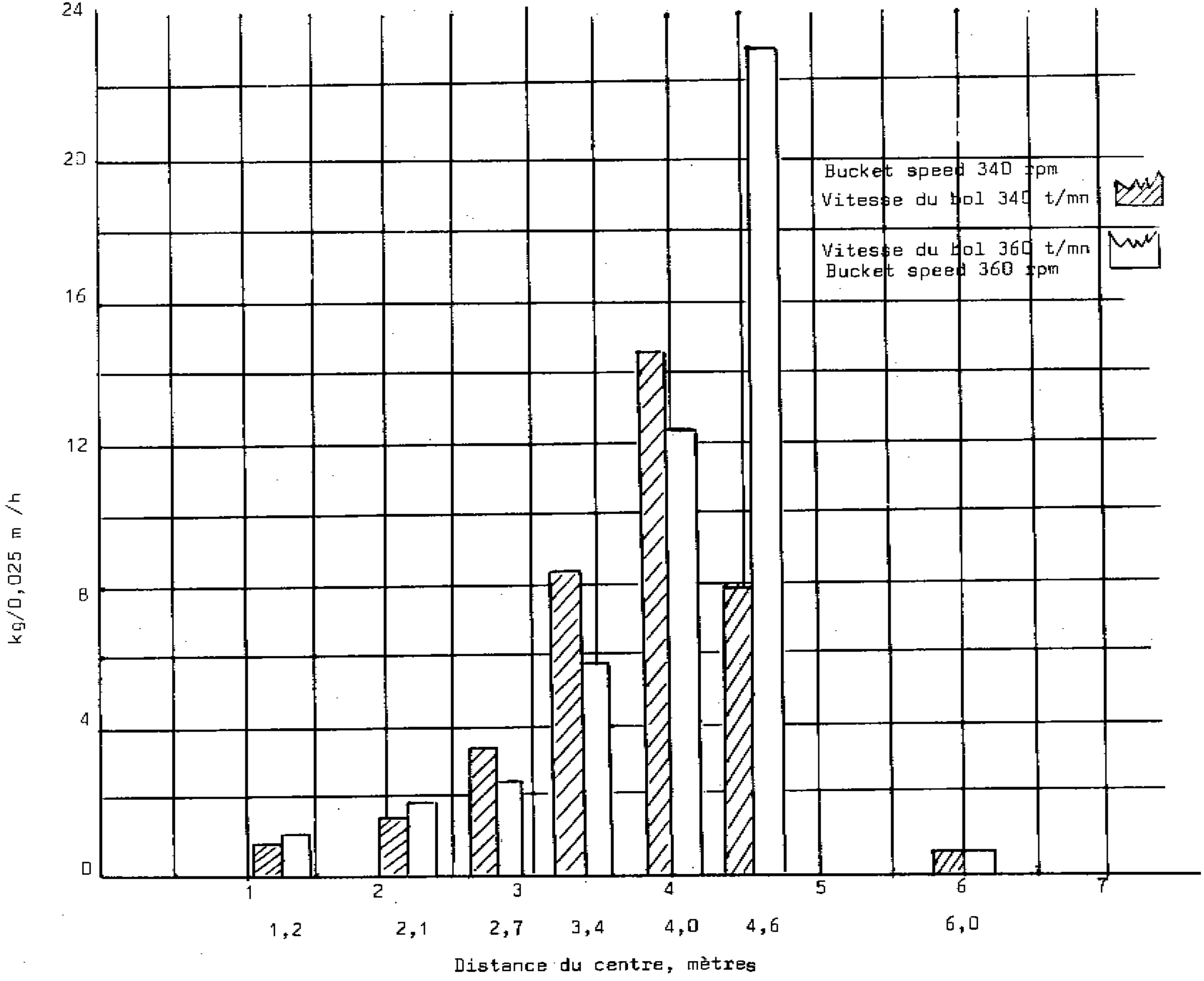
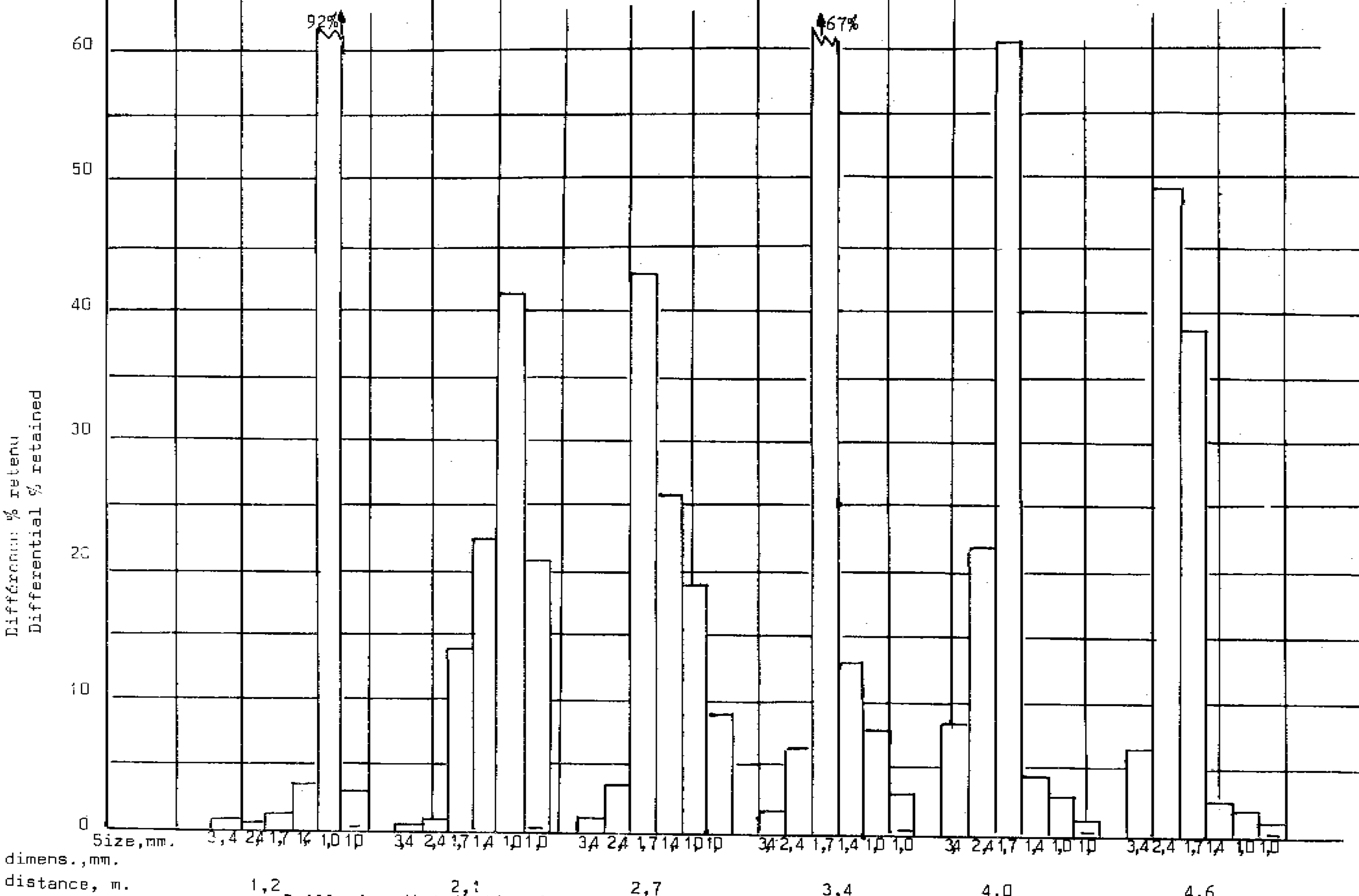
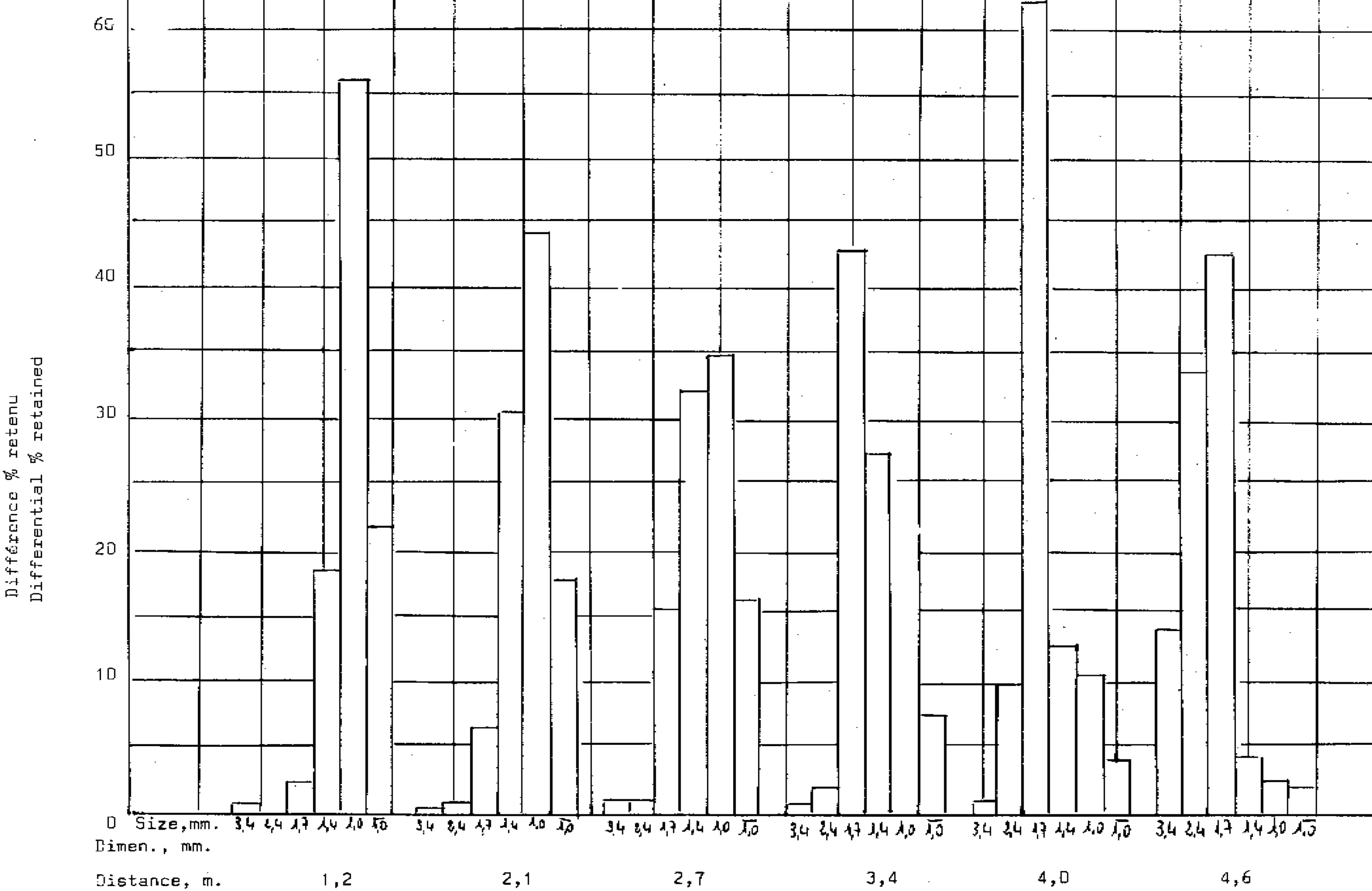


FIG. 7 Profil de distribution en bas de la tour
Throughput distribution on prill tower floor



Prill size distribution in samples at various distances from the centre of the floor
Bucket speed. 340 rpm, Product. 22-11-11

Figure 8 Distribution de la granulométrie d'échantillon à différentes distances du centre de la base de la tour - Vitesse du bol : 340 t/m, formule 22-11-11



Prill size distribution in samples at various distances from the centre of the floor.
Bucket speed. 365 rpm, Product. 22-11-11

Figure 9 Distribution de la granulométrie d'échantillon à différentes distances du centre de la base de la tour - Vitesse du bol : 365 t/m, formule 22-11-11

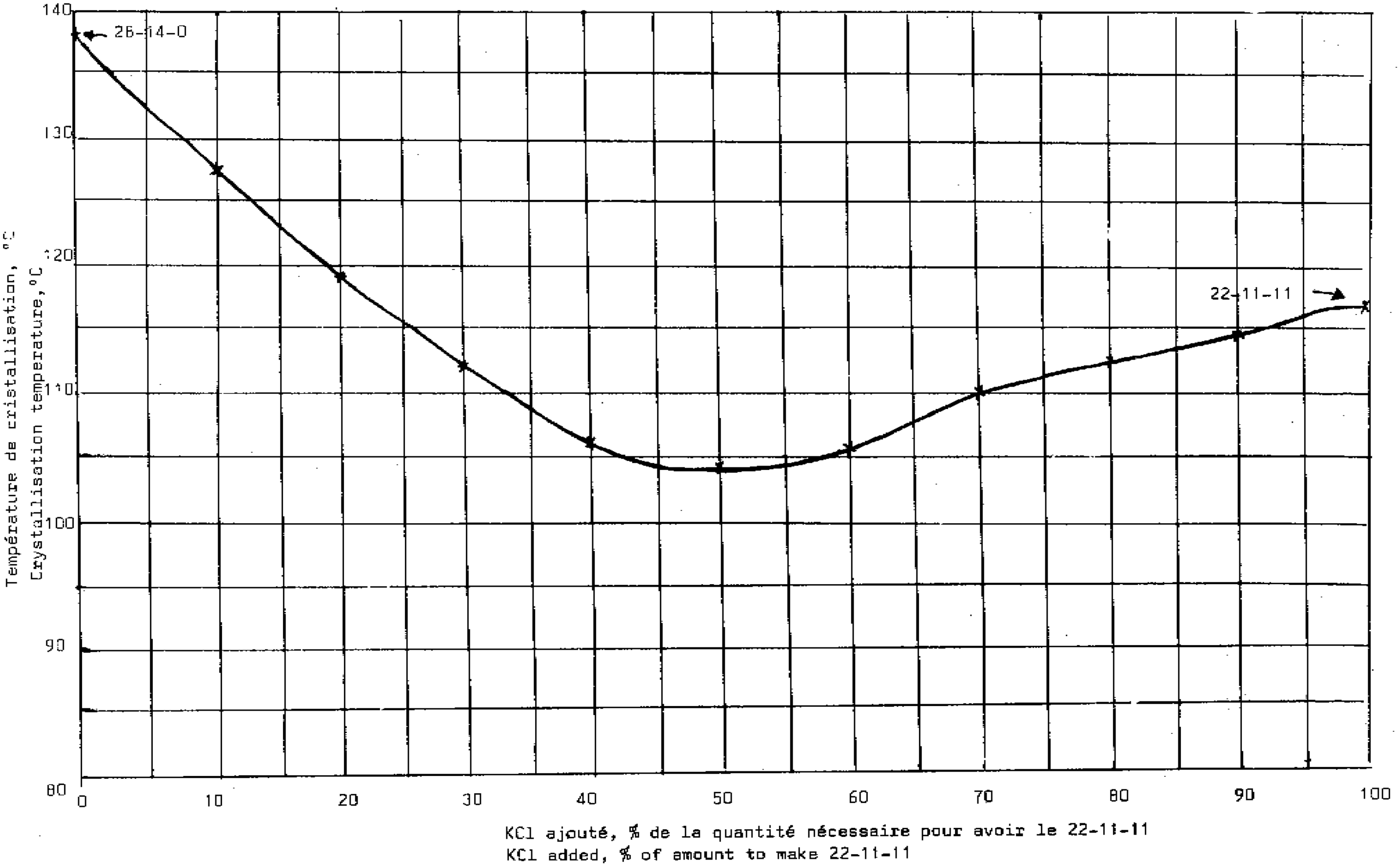


FIGURE 10 Température de cristallisation de la bouillie de NPK, N/P_2O_5 total = 2

Crystallisation temperature of NPK melt, Total N/P_2O_5 = 2.0