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COMPUTER CONTROL OF GRANULATION PLANT NPK AUTOANALYSERS

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DESCRIPTION OF GRANULATION PLANTS

The ICI Production Site at Billingham has been established for over 50 years and is currently occupied by several Manufacturing Divisions. Agricultural Division is the major producer on the Site and has its Headquarters at Billingham.

On the main Site the major activity is the production of ammonia. Four plants, using natural gas as feedstock, produce 4,500 tes of ammonia per day and associated byproduct carbon dioxide. In the same manufacturing group are two Methanol Plants and a Urea Plant. Alongside the Ammonia Group Plants are the plants producing fertiliser intermediates. There are six Nitric Acid Plants producing up to 3,000 tes HNO_3/day and a Phosphoric Acid Plant. Sulphuric Acid is produced on two 500 tes/day double absorption plants.

Fertiliser production is at the Portrack Site, an area approximately 1km from the main Site. The area was established in 1957 when the Granulation Plant was built. Prilled ammonium nitrate plants were built in 1969 and 1979 together with associated packing facilities. Total capacity for prilled ammonium nitrate is well in excess of 1,000,000 tes/year.

The Granulation Plant originally comprised two identical Dorr-Oliver units producing a single fertiliser formulation 12:12:18 based on Ammonium Sulphate, Ammonium Phosphate and Potash. In 1964 the plant was extensively modified to change to ammonium nitrate based fertilisers. An ammonium nitrate neutraliser was constructed with associated nitric acid storage fed by pipeline from the main Site. Each granulation unit was equipped with an ammonium phosphate reactor to allow on-Site production of ammonium phosphate slurry previously transferred by tanker from the main Site. Product treatment sections were added to each unit to cool and coat the product before transfer to the bulk silos. A diagram of the plant is shown in Fig 1. With the new plants the fertiliser range was expanded to meet the changing needs of the farmers and approximately 12 formulations are now produced. The present capacity of the plant is over 400,000 tes/year.

DEVELOPMENT OF NPK AUTOANALYSERS

The expanded range of formulations produced on the granulation units had to meet the legal requirements of UK regulations which were later superceded by the EEC Directive. There was also an obvious need to avoid any over-formulation since this represented a significant loss of raw materials. Analysis control was first achieved by taking fertiliser samples every 2 hours from each granulation unit. These samples were then transferred to the main Site laboratory for analysis and the results were then communicated back to the granulation plant staff. This lengthy process together with the normal residence time effects of the granulation loop made analysis control extremely difficult and the inevitable consequence was overformulation with its associated cost.

In the late 1960's work commenced on the development of plant-based autoanalysers for the NPK analysis. By 1970 the installation of equipment was complete and operating successfully on the two granulation units.

There were four distinct operations:-

a) The sampling unit in which a representative sample of the fertiliser is taken every eight minutes from a falling stream of granules.

b) The solution preparation unit for preparing a solution of 50 gms of fertiliser per litre.

c) The analysis unit for the determination of N, P and K by spectrophotometric methods of analysis using Technicon AutoAnalyzer equipment.

d) The recorder units which display the results both in the analyser house and in the Granulation Plant control room.

a) Sampler - The sample of fertiliser was taken from the duct feeding product from the cooler to the secondary screen. The sample was taken by automatic operation of a pneumatic actuator which uncovered an aperture over which the granular fertiliser was falling. Since 50 gms of material are required for the sample preparation unit the timer was adjusted to give approximately 100 gms. This also allowed for variations in plant production rates. The collected sample passed down a pipe to feed the sample preparation unit. Baffles were positioned in the pipe to slow down the material preventing spillage and dust.

b) Sample Preparation Unit - Fig 2 shows a diagram of the unit. The material from the sampler comes to rest in the vibrofeeder trough. This then vibrates at a moderate amplitude feeding material into the weigh pan until the shutter vane interrupts the photocell beam which causes the feeder to slow to a trickle. The weigh beam is then checked near the balance position by picking up the ballast weight. When the weight of sample in the pan reaches the correct value the ballast weight is lifted, the photocell is uncovered and the vibrofeeder stopped. Meanwhile the automatic pipette has delivered 1,000 ml of water to the dissolving vessel. The weighed sample is now also tipped into the dissolving vessel via a chute by rotating the weight pan through 180°. The mixture is then stirred by an air driven motor. The made up solution is finally discharged by opening the ball valve into the filter reservoir unit so that the solution is filtered before analysis. This reservoir retains sufficient solution to supply the analyser for one cycle before being displaced by the succeeding batch of solution. During this process the vibrofeeder trough has been rotated to a position above a discharge funnel and any remaining fertiliser sample is discharged. The trough is then returned to its position above the weigh pan ready for the next cycle.

c) Analysis Unit - The methods used are as follows:-

i) Determination of Total Nitrogen

The method involves reduction of nitrate ion to ammonium ion with hypovanadous sulphate solution followed by determination of the ammoniacal nitrogen formed, together with any originally present by the indophenol blue colorimetric procedure. The intensity of the blue colour measured at 625 nm is proportional to the total nitrogen present.

ii) Determination of Phosphorous

This is a simple colorimetric procedure involving the addition of a vanadate - molybdate reagent which reacts with the phosphate in the fertiliser to form a yellow vanado - phosphomolybdate complex. The intensity of the yellow colour at a wavelength of 420 nm is proportional to the amount of P_2O_5 present.

iii) Determination of Potassium

The method of determination of potassium involves precipitation of potassium tetraphenylborate by the addition of a solution of sodium tetraphenylborate (STPB). The precipitate is filtered off and the excess STPB determined by measuring the absorbance of the filtrate at a wavelength of 254 nm.

Proportioning pumps are used to feed the various reagents and solution at the required rates. Mixing is achieved by passing the stream of reagent and sample, segmented by air bubbles, through 3 mm glass mixing coils in a horizontal axis. Time delay to achieve adequate colour development is achieved by passing the mixed stream through a glass coil of the required length.

The Technicon colorimeters used are simple double-beam filter instruments. The light from a tungsten filament lamp is divided to give two beams. One beam passes through the sample to a photocell while the other beam goes to an identical reference photocell. Two colorimeters are used, one for the total Nitrogen stream and one for the Phosphorous stream.

A LKB Ultraviolet Absorptionmeter ('Uvicord') is used for the potassium determination. With the LKB 'Uvicord', ultraviolet light from a mercury vapour lamp passes through an adjustable slit. A liquid filter (nickle sulphate solution) on the other side of the slit, absorbs most of the unwanted radiation and light which is eventually monochromatic then passes through the measuring cell. The amount of light absorbed in the cell depends on the nature of the solution flowing through it. Unabsorbed ultraviolet light continues through a black glass filter and on to a phototube detector. The black glass filter absorbs any visible light coming from the mercury vapour lamp or from external sources. A quartz cell with a 1 mm light path and 0.05 ml volume is used in the LKB 'Uvicord'. Owing to the extremely small volume of the cell it is necessary to ensure that the solution flowing through it is thoroughly mixed. A small magnetic stirrer encased in a Pyrex glass chamber is used. The volume of the vessel is 2 ml. A similar mixing vessel is used in the total nitrogen stream to ensure thorough mixing.

Continuous filtration is used to remove the precipitation by allowing the suspension to drip on to a moving roll of filter paper. A small hole in the guide plate positioned under the paper allows filtrate to be drawn into the LKB 'Uvicord'. A coarse sintered glass filter is placed immediately after the continuous filter to remove any small fibres carried over from the filter paper.

In the STPB method for potassium, the potassium tetraphenylborate precipitate has a tendency to build up on the inside walls and at the joints of the mixing coils, resulting in blocked tubes. In order to overcome this problem, the mixing coils immediately after the point where the STPB is added are made as a single unit and immersed in an ultrasonic bath. The ultrasonic vibrations ensure that the precipitate is kept moving thus preventing any build up.

d) Recorder Units

The basic measurement techniques used in the equipment give a non-linear output. This creates problems with calibration of the instrument and transmission of the output to the recorders in the plant control room. The calibration of the equipment was set up using a standard fertiliser of the exact analysis of the fertiliser to be produced. This use of solid fertiliser for calibration did give recurring problems with analysis variation with the standard sample but this was overcome by careful selection and preparation.

To overcome the non-linear output from the measurement equipment electronic equipment was fitted to produce a reasonable linear signal. This converted signal was transmitted to conventional chart recorders in the plant control room.

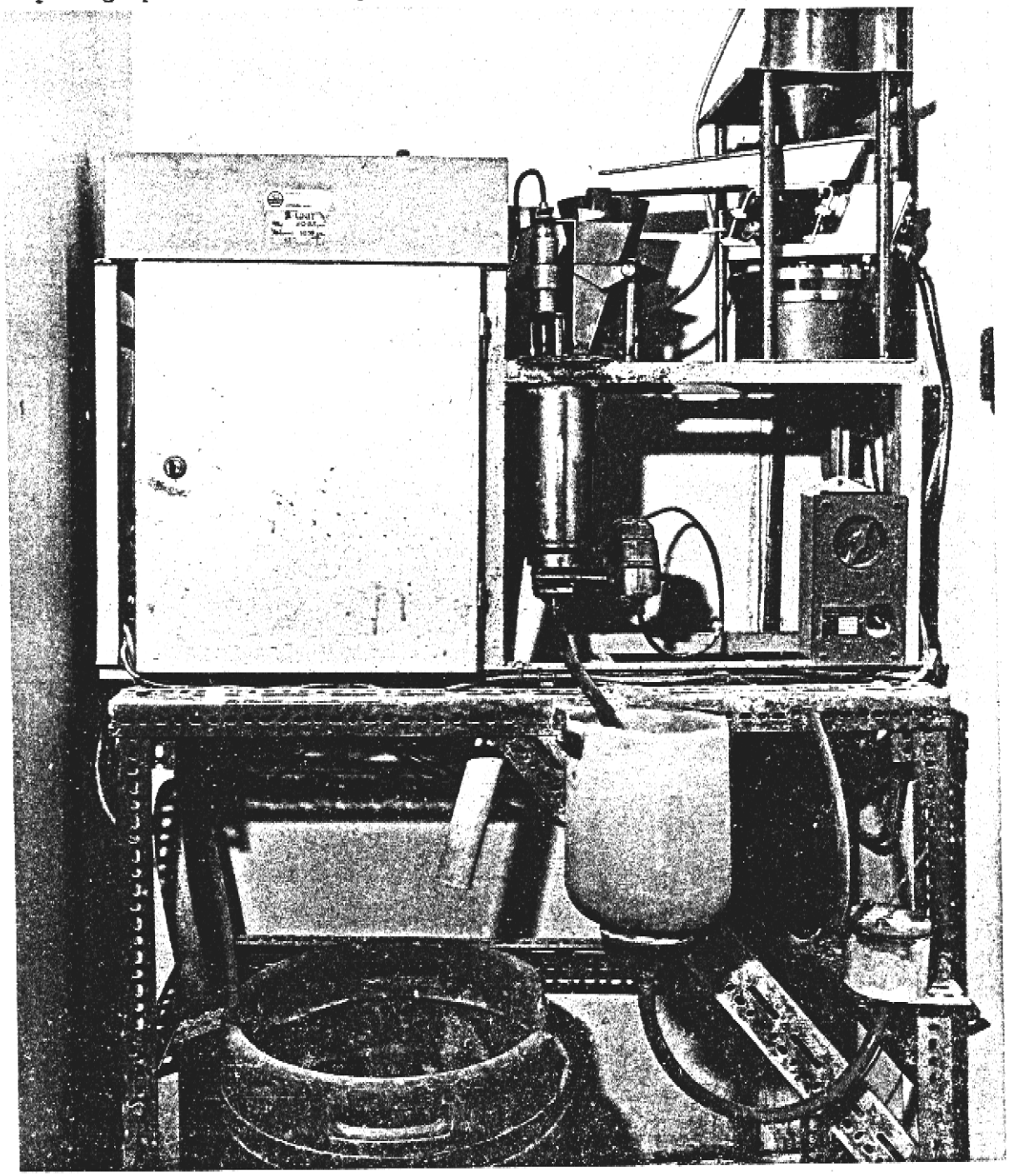
INTERIM PERFORMANCE

The equipment described operated up to 1975, at which time it was decided to improve certain aspects of the equipment hardware and to introduce computer controlled standardisation to improve the absolute accuracy of the measurements. During this early period of operation the plant autoanalysers were used by the operators as a guide to the fertiliser analysis but plant control was carried out on the basis of 4 hourly samples sent to the Works Central Laboratories for analysis. To maintain the equipment a full-time instrument technician was employed and this was supplemented from time to time by an Instrument Development Officer.

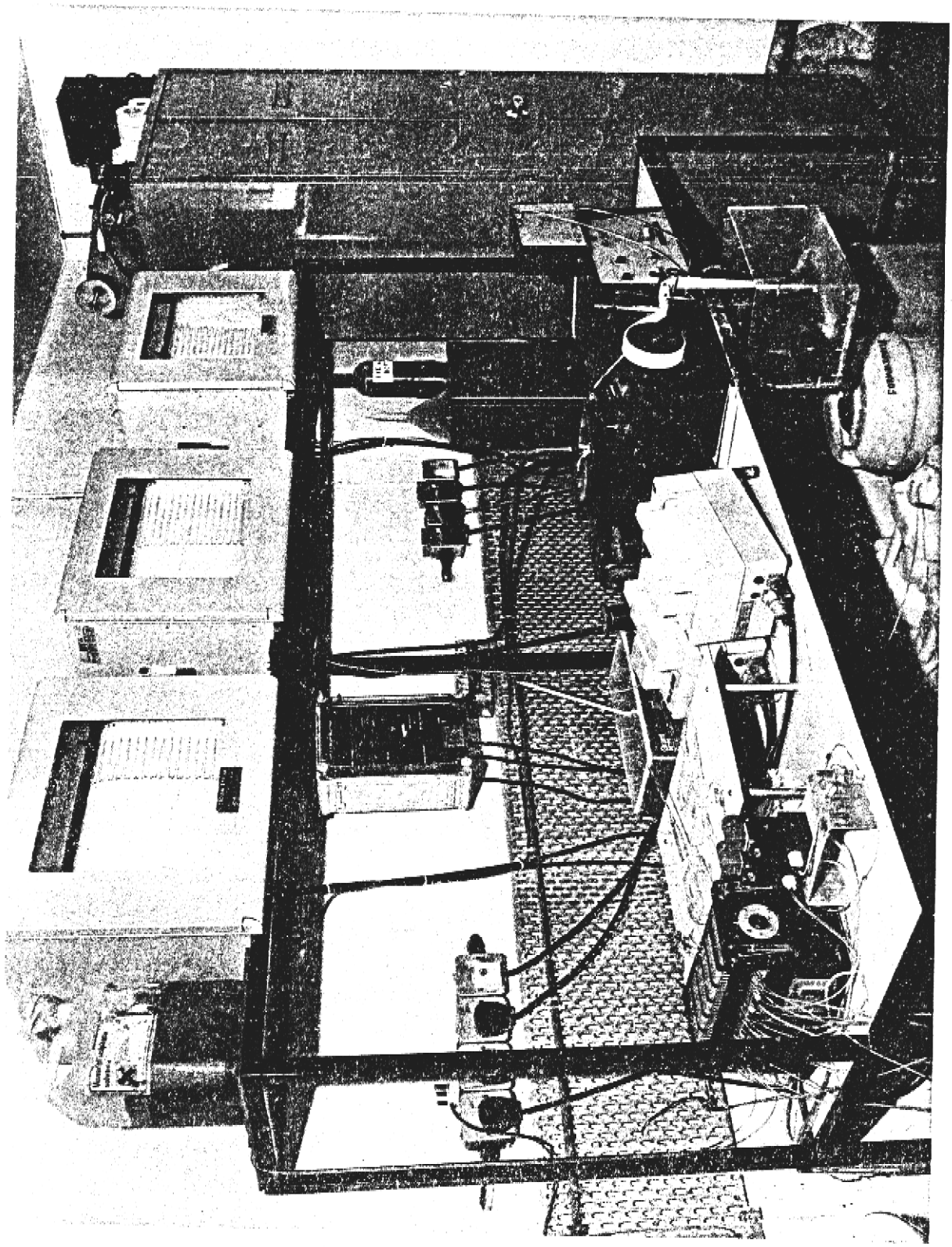
EQUIPMENT IMPROVEMENTS

One of the major problems with the installation was the analyser building used to house the equipment. In its original position, taking a sample of fertiliser from between the cooler and the secondary screen, a cheap wooden building was used. The building offered poor resistance to ingress of dust from the plant environment and this was aggravated by having the Sample Preparation Unit adjacent to the analysis equipment. Conditions inside the Analyser building were far removed from laboratory conditions and this contributed to poor operation and accuracy from the equipment. A further problem was that the building was poorly insulated and although heating was provided the temperature variation inside the building was sufficient to affect the measurement accuracy.

In 1975 arrangements were made to provide a new purpose built analyser house for the equipment. The house contained 2 rooms, one for the sample preparation unit and one for the analysis equipment. The construction materials used produced a sealed, well insulated building with filtered ventilation. A heating/control system was fitted to maintain a steady temperature within the building. The new house was positioned to receive a sample of material taken from the conveyor feeding the cooler by siting the building directly under the sample point. The following photographs show the sample preparation room and the analysis room.

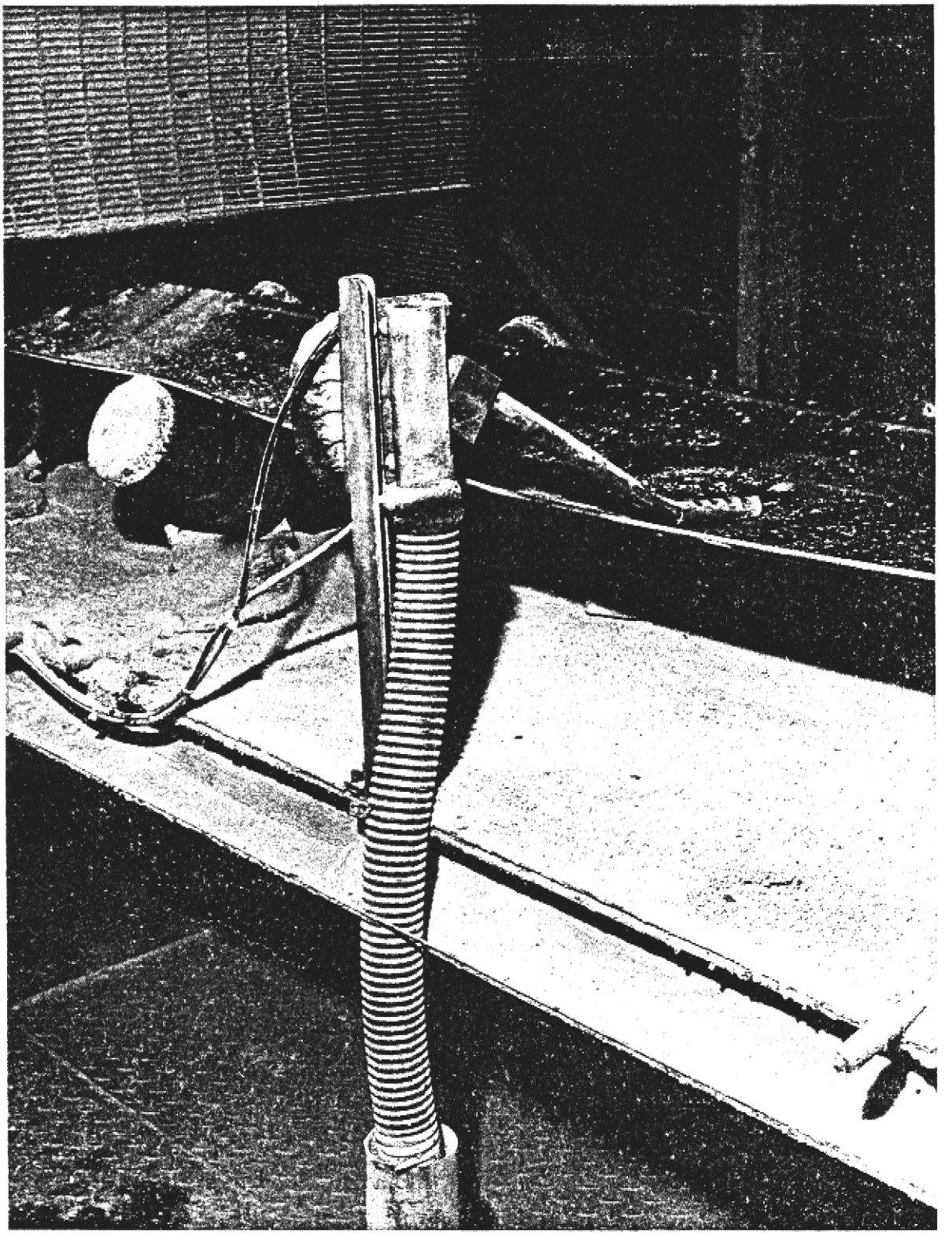


SAMPLE PREPARATION ROOM



ANALYSIS ROOM

At the same time as the analyser house was resited a new sampler was developed to take a representative sample of fertiliser from an open conveyor band. This "hockey stick" sampler is shown in the following photograph.



The device consists of a hollow pipe with an angled end. Slots are cut into the end section so that when the sampler is in the horizontal position and immersed in the fertiliser on the conveyor band, the granules fall through the slots and into the hollow tube. When the sampler returns to the vertical position the granules in the tube fall down the tube and are fed directly to the analyser house.

COMPUTERISATION AND AUTO-STANDARDISATION

One of the main shortcomings of the colorimetric analysis system as described was drift in the characteristics of the analyser itself. An example of this was the phosphate channel normally regarded as the most satisfactory measurement, where a progressive drift in the output optical density from 0.50 to 0.56 on a constant standard sample over a 24 hour period would not be unusual. To overcome this problem the equipment was standardised every eight hours but this created other difficulties in that it disturbed the record, made it difficult to see trends, was laborious and was difficult to arrange outside normal working hours and at weekends. Even with these difficulties the equipment produced acceptable although not ideal results. In order to reduce this source of error it would be necessary to standardise at a frequency of less than one hour. On a manual basis the level of attention required would make this impossible but on an automatic basis using a computer to process the data this could be done satisfactorily. It was therefore proposed that a system be installed using a small computer in which the span and the zero of the analyser are checked before each process sample is measured using a liquid standard. The absolute standardisation would still be done manually, on a daily basis, by inhibiting the plant sample and substituting a fertiliser standard of known composition, near that of the plant make.

Since the analyser would be operated on a batch basis, scanning a number of samples, it is important that the response should be as rapid as possible. The "block and bleed" method would have to be used to switch the input samples which would be pumped continuously. This was achieved using two small solenoid valves along with changes to the piping system on the analysers.

A sequencing controller was needed to signal the computer as to which sample was being measured at any particular time and to control the switching of the process sample and standard. Synchronisation was tried, but this proved difficult. The sample is now taken at a high frequency compared to the analysis frequency. When the equipment is ready for the next analysis it uses the most recent sample, which is never more than 4 minutes old. Fig 3 shows a block diagram for the new systems.

DATA PROCESSING

The starting point for the data processing is the analyser output signal from the amplifier. Although this is a 4 - 20 mA signal, the machine regards it numerically as the percentage of full scale within this range. The 4 - 20 mA output signal is converted by the computer to a percentage of full scale.

The computer monitors the signal (x%) and normally identifies and measures the zero peak. It then measures the signal at the other 3 quarter points during the cycle and tags them as corresponding to the process, standard and process samples. Fig 4 shows a typical chart from the local recorder with the 3 peaks identified.

Each of the values must then be converted to concentration in proportional terms or "linearised". This is because the colorimeter output follows the Beer-Lambert law: $x = \exp(-n)$. Account is then taken of the fact that the signals have not had time to reach their steady state levels when they are measured and therefore depend to some extent on the previous value, and this is an inevitable consequence of batch analysis techniques. In practice the signal reaches about 90% of the way to its new equilibrium value.

Compensation for this 'memory' effect involves subtracting from each measurement a fixed fraction 'K' of the previous reading. In fact these compensated values are all low by a factor of (1 - K) compared with the actual steady state readings but this is of no consequence as ratios are taken in the last stage.

Allowance is finally made for the fact that the material is analysed before coating when the analysis after coating is actually required. The calculated analysis results are reduced by a factor of approximately 0.98. A facility is retained to alter this factor depending on the material being produced.

PRESENTATION

There are two forms of presentation in the plant control room.

1) The most recent determination is displayed on digital indicators.

2) The same values are displayed on chart recorders. The 'peak' effect is removed to give a continuous trace and this provides the operators with a trend for the 3 analysis measurements.

PERFORMANCE

The equipment has now been in operation for over 5 years and requires little attention apart from a daily check, daily/weekly cleaning to remove deposits from tubes etc and absolute standardisation. The analysis scheme used follows the standard laboratory method and an acceptable standard of accuracy is obtained from the system. Plant control now depends on the autoanalyser results which are used as the basis for changes to flow-rates on the plant. An occasional check is carried out at the Main Works Laboratory particularly at the start of a production run when the formulation has been changed.

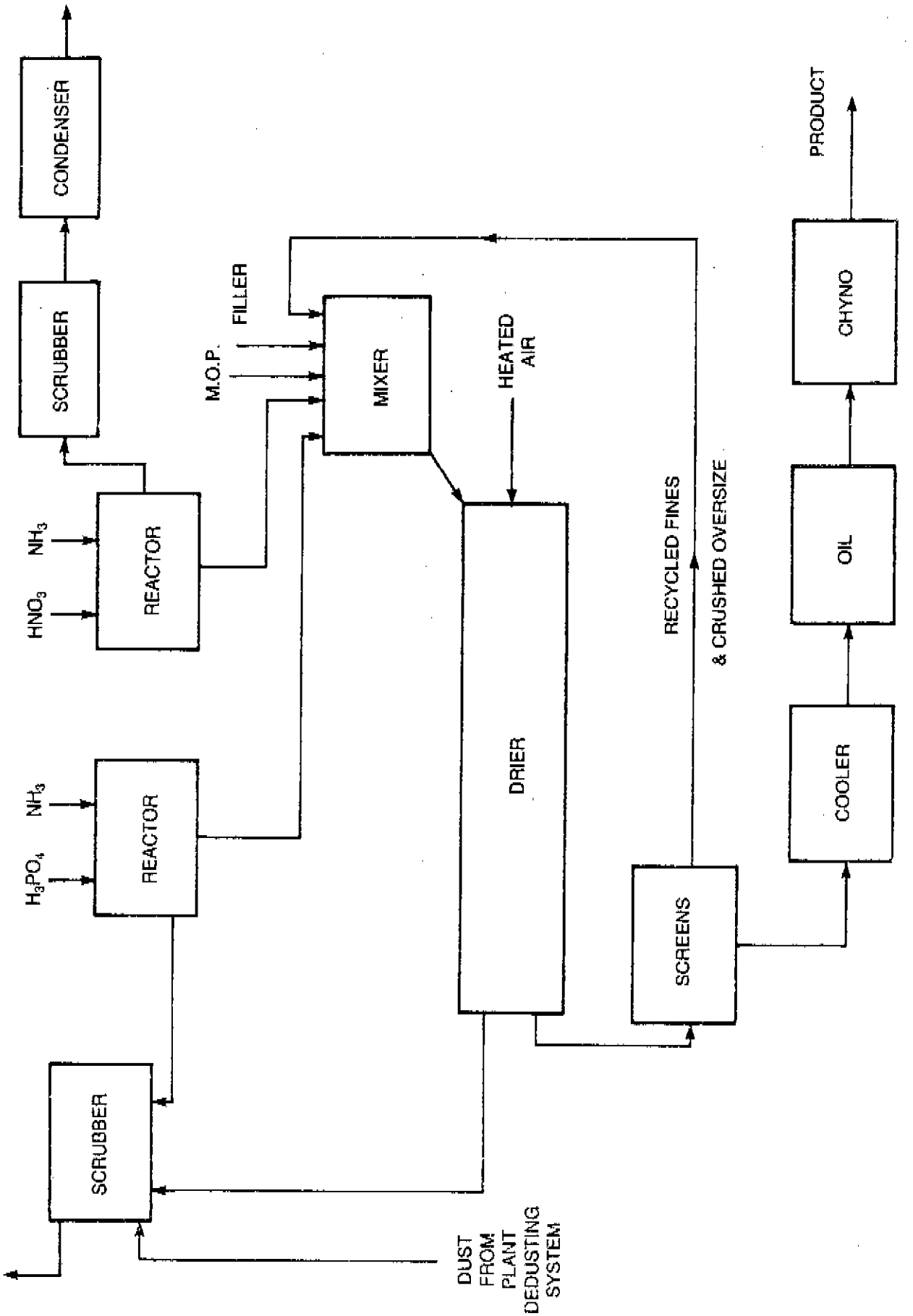


FIG. 1 GRANULATION PLANT FLOWSHEET

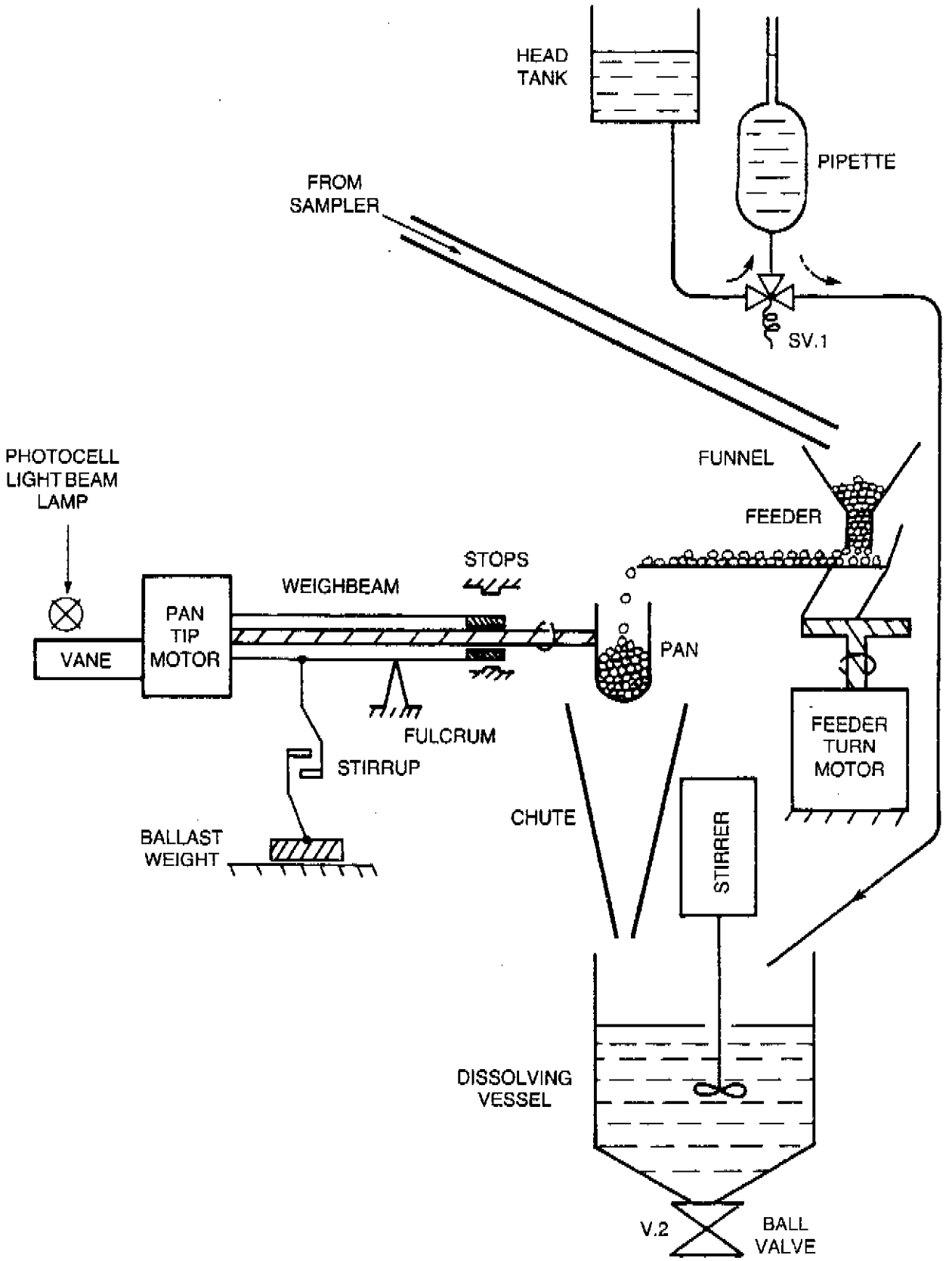


FIG. 2 SAMPLE PREPARATION UNIT

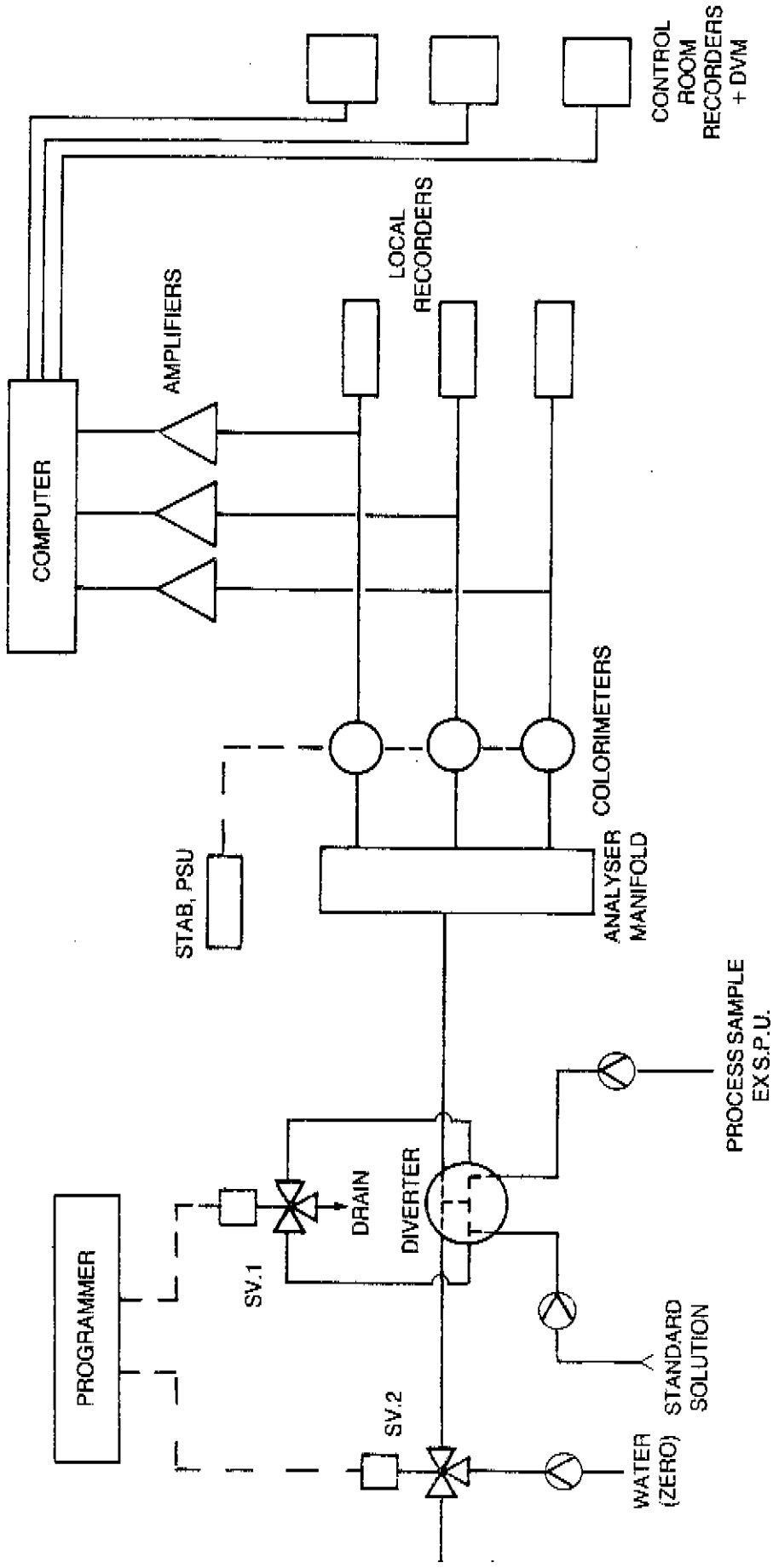


FIG. 3 COMPUTERISED SYSTEM BLOCK DIAGRAM

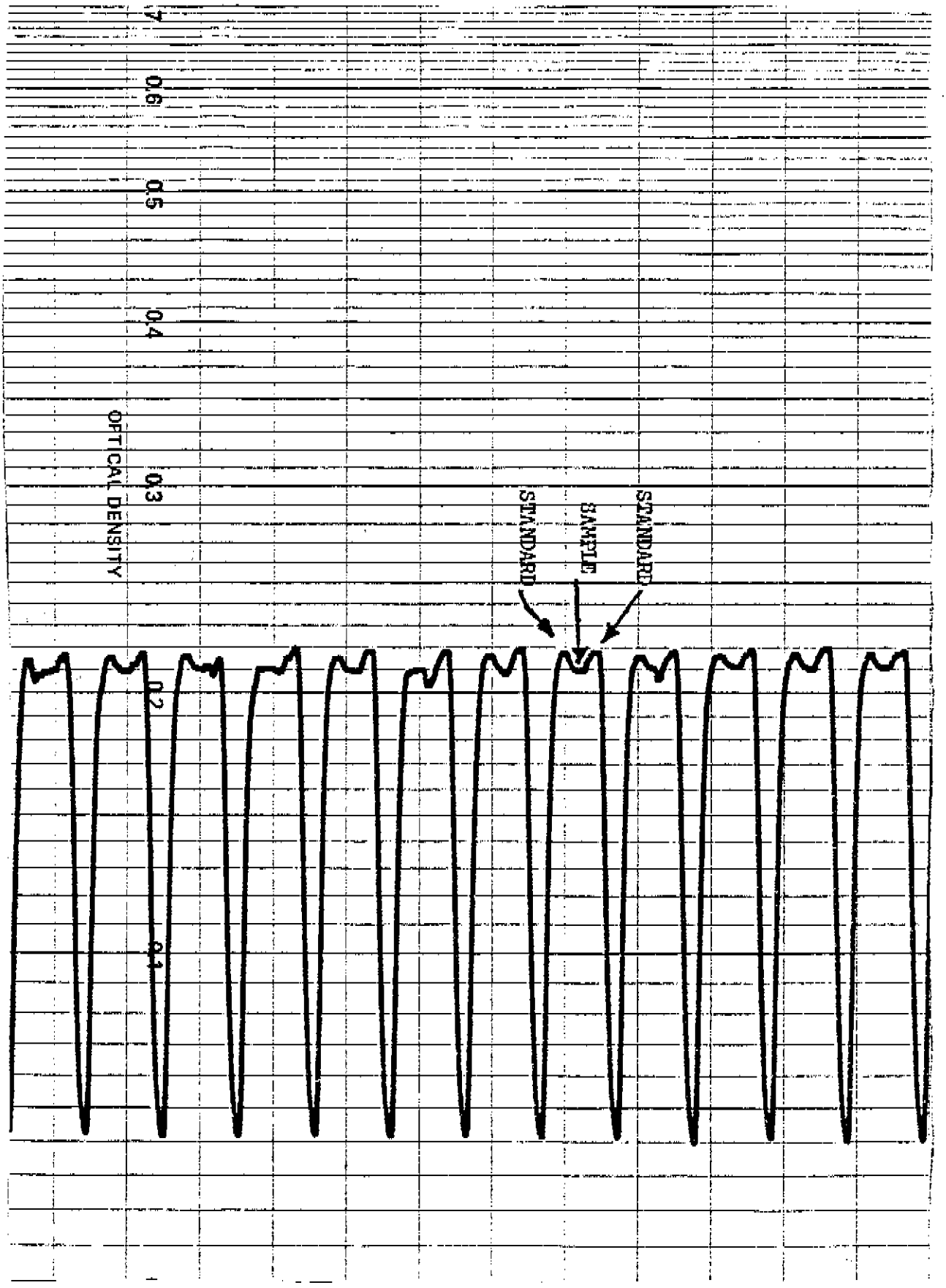


FIG 4 CHART FROM LOCAL RECORDER

TA/84/21 Computer control of granulation plant NPK autoanalysers by J.H. Markham, ICI PLC, United Kingdom

DISCUSSION: Rapporteurs L.K. RASMUSSEN, Superfos AS, Denmark and B. PERSSON, SUPRA AB, Sweden

Q - Mr. A. SINTE MAARTENSDIJK, AMFERT, Netherlands

Although the author refers to the EEC regulations, the P205 determination is done as water-soluble and not as neutral ammonium citrate P205. In our experience the process-control on basis of water-soluble P205, when selling on basis of n.a.c. P205, does (at least when using superphosphates) not give the same results. I would like to hear your opinion on this.

A - I have an easy reply to this as far as we are concerned at ICI. We use Senegal phosphate rock as our basic phosphate supply. This rock produces fertilizers with a relatively high water-soluble P205 content and presents no problems to us in the determination of the phosphate content of our fertilizers.

Q - Could you give a rough estimate of the cost involved in a system as described and did you make an evaluation of the cost and profits after some years of operation with this equipment?

A - I knew that this would be an inevitable question but I haven't prepared a cost estimate for a modern system to the standard that we have installed at Billingham. From the description I have given, delegates will realize that we have developed this system over a number of years. We have added to and modified the original equipment and this makes it difficult to put a composite cost to the present installation. The equipment used is, in the main, relatively simple and easily obtained. Some of the key items, particularly the sample preparation unit, are produced in our own workshops at Billingham. I have discussed the situation with my colleagues and we believe that Pounds 50,000 might be needed to provide an installation to an ICI standard.

Q - In normal laboratory-analysis the sample is ground before determination of the nutrients, in your equipment it is not; did you investigate whether you got deviations in the analysis due to this simplification?

A - The only problem we have ever experienced in this area has been the determination of total P205 content of our fertilizers in our main laboratories. It was found necessary to grind the fertilizer down to a very fine size grading to give an accurate measurement of total P205. This problem was never experienced in the water soluble P205 determination on the auto-analyzer which provides the information needed for plant analysis control.

Q - Mr. J.D. CRERAR, Norsk Hydro Fertilizers Ltd, United Kingdom

Sampling of a stream of moving granules is difficult to achieve without segregation. How much work did ICI do to evaluate the performance of the hockey stick sampler with respect to segregation?

A - The hockey stick sampler which I have described is a very simple device and represents an easy solution to the problem of obtaining a representative sample from a moving stream of granules on a conveyor. When we were developing the sampler the major concern was the problem of segregation. Plant operating staff in the audience will be very familiar with this situation where the fine material is predominantly at the base of the granular fertilizer while the large size particles run along at the top. We carried out a series of tests by installing a "GECO" sampler which takes a sweep across the flow of granules at the discharge of the conveyor. The results from the "GECO" were compared with those from the hockey stick sampler and remarkable agreement was found. We are very confident in the representative nature of the sample taken by the hockey stick sampler.

Q - Would you use the same device for sampling for size analysis?

A - We have installed hockey stick samplers on the product conveyors from the two granulation units and they are used to take the routine samples for size grading and quality checks along with the occasional samples taken for check analysis. I believe that this demonstrates our confidence in this device.

Q - Is there segregation in the vibrating feeder to the dissolving stage?

A - There is no segregation because the vibrating feeder is effectively choke-fed. The granules fall down onto the base of the vibrating trough and in effect the granules then pass along the trough in the order that they are presented to the trough itself.

Q - Mr. R. MONALDI, Fertimont SpA, Italy

Considering that also the NPK plants of Fertimont are controlled by full automated systems developed using the technique of thermometric analysis, I should like to get some information on these points, if possible:

Coefficient of variation % for each determination performed by your system?

A - We all use different terms to describe the accuracy of analysis control. At Billingham we use the term standard deviation. If I use 17 nutrient units as an average nutrient level we would expect to achieve a standard deviation of 0.35 unit. I believe that is approximately 2% in accuracy terms.

Q - What is the accuracy level with respect to the same determinations manually performed?

A - If you are referring to a manual determination in a laboratory then I would not claim that our on-plant analyses give the same accuracy. In the laboratory determination a lot more time is taken to allow the sample and standards to reach the steady state situation which reduces the scope for significant errors. We do achieve, on a continuous basis, a very high degree of correlation with laboratory analysis and I wouldn't want to leave anyone with

the wrong impression on this point. Our results from the on-plant analyzers are very close to the results obtained by laboratory determination.

Q - What is the actual time (on yearly basis) required to maintain the system in good performance?

A - We now allow our instrument technician half an hour a day to do a daily standard check and check over the tubes etc... That is two and a half hours a week for this installation.

Q - Mr. S. ORMBERG, Norsk Hydro, Norway

With the auto-analyzer you are determining the total nitrogen content. Is it possible also to get separate values for NH4-N and nitrate-N?

A - The development of this system has opened up the opportunity to use the automatic equipment on other measurement duties. We have already installed analyzers on the effluent discharge from our ammonium nitrate plants to continuously monitor NH4-N and NO3-N. It is our eventual aim to use this data to achieve automatic control of certain parts of the plant.

Q - What is the accuracy of the N, P and K analysis and have you managed to reduce the manpower in the laboratory after introducing the auto-analyzer?

A - I have dealt with the question of accuracy. In terms of the manpower used on the installation we used to have one and a half instrument technicians full-time plus additional resources when needed and this has now been reduced to one man working half an hour per day. In addition we are now doing more than 95% of our analysis on the plant and this has reduced the resources needed in the central laboratory The experience that we have gained by developing the on-plant system has been transferred to the central laboratory situation where many of the systems have now been automated giving further manpower reductions.

Q - Mr. J. POUKARI, Kemira Oy, Finland

Your NPK-analysis is from year 1975. Have you made development after 1975 and in which parts of the analyzer?

A - We have changed very little in terms of the installation in the last 5 years. I think the 1975 you refer to is mentioned in the paper as being the break-point between phase one of the development which was developing the basic system and phase two of the development which was developing the computerisation, etc... It took a little while to establish the computerisation, the hockey stick sampler, the new house and the new equipment, but for the last five years we have made no significant change to the equipment at all. I know that a number of people in the audience have visited the installation at Billingham and actually seen it operating during that time period.

Q - What is the maximum time for operation without maintenance?

A - It's something we have never tested. When I say that we use half an hour a day of an instrument technician's time I mean Monday to Friday. We do very little over the weekend. We have a shift instrument technician who checks the equipment over during the weekend. I suppose really that relates to two days of continuous operation in that situation, but we have never tested that because we do feel that it is important to go in on a daily basis, doing an absolute determination, checking over the tubes and making sure there are no problems.

Q - Is the weighing procedure precise enough?

A - This is the most accurate weigher of this type that I have come across. For a plant-based piece of equipment it is a very sophisticated piece of precise engineering, which has been developed for this particular activity.

Q - Mr. E. SEUNA, Kemira Oy, Finland

You daily feed a manual standard, which is near the grade being produced. How many different standards of this kind do you need?

A - There are 8 standard solutions used to cover the complete range of NPK formulations produced on the plant.