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THE AUTOMATIC CONTROL OF A GRANULATION PLANT

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The objective of the fertiliser manufacturer is to produce a granular product conforming to:-

- (i) strict legal requirements in respect of N.P.K. analysis with limits of tolerance governing the permitted deviation of this analysis from that declared by the manufacturer. These tolerances differ from country to country and may be tolerances about the mean as in the United Kingdom, or in some countries minimal analyses may have to be guaranteed. In some instances specification of the forms in which the main plant nutrients, and particularly nitrogen, may be required, again with individual limits of tolerance;
- (ii) limitations in size range which may be dictated by legal requirements or by the commercial considerations of competition which oblige the manufacturer to produce fertilisers of consistent physical quality;
- (iii) limits on moisture content which have a strong bearing on the storage properties of each product, in a market where storage for periods of up to twelve months has become customary and where material may be stored out of doors in plastic sacks under extremes of temperature and weather conditions.

Clearly then, current manufacturing practice must be carried out under tight control, and it is of interest to note the changes which have taken place in the past twenty years which have necessitated this control. The sale of plant nutrients has risen steadily and the concentration of nutrients in fertilisers has increased sharply. At the same time, heavy expenditure on research and development, coupled with capital investment on a large scale, have led to the establishment of large manufacturing units with a small number of plant operators so that it is now commonplace to run a granulation plant producing 30 tons per hour with three or four men per shift and there can be little doubt that this trend will continue.

It is relevant to consider the ultimate possibility of automatic plant control and the potential advantages which may

accrue. It is suggested that these may include:-

- (i) reduction in manufacturing costs;
- (ii) improvement in consistency of chemical and physical quality of products;
- (iii) increased plant utilisation and production rate;
- (iv) improved utilisation of services e.g. steam, water, power;
- (v) reduction in fuel costs for drying;
- (vi) elimination of human error and fatigue in sampling, analysis and particularly plant control;
- (vii) rapid detection and correction of process disturbances;
- (viii) provision of more accurate information on raw material usage and accountancy information;
- (ix) reduction in load on process control laboratory;
- (x) more detailed understanding of the manufacturing process and the fluctuations occurring.

Against these advantages must be set:-

- (i) cost of development of suitable and reliable systems of control;
- (ii) cost of installation of the control system;
- (iii) operating and maintenance cost of the control system;
- (iv) difficulty of forecasting accurately the benefits to be derived from automatic control.

How may automatic control be achieved?

The three main properties of the product outlined above must clearly be monitored, namely:-

- (i) chemical analysis for N.P.K. and possibly other additives, e.g. boron, magnesium, trace metals etc;
- (ii) physical quality i.e. size grading;
- (iii) moisture content.

If suitable automatic methods can be developed then it follows that monitoring of raw materials and process streams can

be achieved, and the development of a system of computer control can be envisaged. However, before such a procedure can be developed, the methods to be adopted for on-line analysis must be chosen. It is considered unlikely that analysis for the plant nutrients N.P.K. will be possible without recourse to sampling as methods of analysis which could perhaps be developed for continuous analysis of material on a conveyor belt, e.g. activation analysis by neutron bombardment would probably be very cumbersome to instal and expensive. Hence, the more traditional methods of analysis must be employed and this raises the question of sampling.

Sampling and Sample Preparation

The difficulties in obtaining representative samples of any material are widely known and appreciated, but these problems are multiplied many times when it is necessary to sample process streams of solids, to carry out sub-sampling and to prepare these solids for on-line analysis by mechanical means.

In the field of on-line gas analysis, where sampling problems are perhaps most easily overcome, techniques have been developed for automatic sampling and the samples may be piped perhaps several hundred feet before analysis by gas chromatography, infra-red polarography etc. The flexibility thus afforded in housing analytical instruments is obviously invaluable. The technique of sampling liquid streams is also progressing rapidly, but the problems to be surmounted in sampling liquid slurries and solids are relatively intractable, for instance:-

- (i) the sample taken must be representative of the main material stream, or very nearly so;
- (ii) sub-sampling will probably be necessary;
- (iii) dissolution of the sub-sample to give a solution of known concentration may be required;
- (iv) the sample or sample solution must be delivered to the analytical instrument without sensible segregation or contamination.

Thus the problem of transportation alone must require a great deal of ingenuity and thought, particularly if the provision of on-line analysers for each process stream to be monitored is to be avoided.

Baldwin (ref 1) has described a simple device for sampling a free falling stream of granular fertiliser, e.g. at a belt changeover using a pneumatically operated diversion flap controlled by a timer and feeding a sample splitter. This system has been found to work well and rejected material can be readily returned to the process stream by a small worm conveyor. Sampling devices based on a travelling hopper which traverses

the granule stream and takes representative samples, are also available commercially. Passage of the sample through sample dividers or series of riffles can be arranged to give a sub-sample of almost any size but at this stage account must be taken of any variation in chemical analysis with granule size, and this will dictate the minimum size of the sub-sample. Such variations will depend on the type of manufacturing process used; it may be anticipated that the effect will be less important as the recycle of the process rises, and variation in a slurry granulation process with a recycle ratio of 10 parts recycled material/unit output would be appreciably less than in an aggregation process operated at a recycle ratio of, say, one. Again, the physical form and variation in chemical analysis of the raw materials will be additional factors to be taken into account. Work by Bowen (ref 2) on dissolution of granular fertilisers made by an agglomeration process at a recycle ratio of about 1:1 has shown the need to take a minimum of 30 grams unground granular product if bias is to be avoided. Assuming that the sample should be dissolved in 50 times its own weight of water, then a minimum quantity of about 1.5 litres of sample solution should be prepared. It may be preferable to split the original sample to a convenient size to weigh the whole sub-sample automatically and dissolve it in a volume of water proportional to the weight of sample. Alternatively, grinding of the sample followed by further sub-division prior to dissolution could be considered but it is often found that the larger particles in a ground sample tend to vary in composition from the finer particles and the grinding operation may, in itself, tend to introduce sample bias and error.

The preparation of a sample solution must usually be followed by removal of suspended matter e.g. by dialysis, or perhaps even by a straightforward filtration technique if a large bulk of solution is available to enable adequate washing of the filter medium; a second alternative may be the development of a small scale continuous centrifuge.

Given that the sample solution has been prepared, it is possible that the transference of an aliquot to the analysis instruments may be accomplished by injecting the aliquot into a plastic tube which is subsequently heat sealed and "posted". Sampling of effluents is already being done by this means and it is suggested that this may be a way to avoid duplication of expensive analytical equipment, and also an important consideration, to give some choice in siting the instruments. Alternatively, it may be preferable to package the solid sample with a central dissolution apparatus.

Whatever system of sampling and sample preparation may be developed, it is clear that there are a great many individual problems to be resolved, indeed the development of mechanical means to accomplish the presentation of the sample, either in solid or solution form to the appropriate analytical system, may well prove as problematical as the subsequent analysis and plant control.

N.P.K. Analysis

Docherty (ref 3) has described a system of continuous analysis applied to fertiliser samples based on the use of the peristaltic pump as a metering and pumping device and applied to colorimetric and flame photometric techniques, the results of the analyses being logged on automatic recorders. Such equipment can be set up to give the N.P.K. analyses which may be required on process streams, for instance, methods have been developed for estimation of:-

Ammoniacal N
 Nitrate N
 Urea N
 Total N by Kjeldahl
 Water Soluble P_2O_5
 Total P_2O_5
 K_2O

and other elements such as magnesium, calcium, manganese etc., may also be determined. The accuracy of the results obtained is at least as good as that in manual, routine analysis and the door is open, in theory at least, to on-line, automated analysis and hence to automatic plant control.

Equipment of this type is in use for on-line monitoring and is linked with computer control on plants in the plastics and pharmaceutical industries. As far as is known, there are no direct on-line applications in granular fertiliser manufacture, but an application in the continuous monitoring of phosphate-rock out-loaded to ship is known. The system is also being used in monitoring phosphoric acid made by the wet-process.

It is interesting to speculate on the possible alternatives to this system of analysis which may be developed for automated analysis. It seems improbable that physical methods will be suitable, and this must then mean the employment of chemical reactions. It is perhaps, true to say that colorimetric and photometric methods lend themselves to continuous methods of analysis and that methods which are unsuitable for manual use may be employed on automatic systems, indeed, it may be that one relies on reactions reaching the same stage repeatedly, under identical conditions, rather than going to completion.

There has been considerable growth of interest recently in thermometric titrimetry and the development of reliable thermistors raises the hope that adaptation of these methods to automated analysis may be realised. The availability of accurate metering and constant flow equipment which would obviously be an essential part of this system of analysis also suggests the possibility that some of the classical titrimetric methods may be used, particularly where end-points may be sensed electrically or photoelectrically.

Determination of Size Grading

If the usual practice of screening the dried material into oversize, product and fines fractions is followed, it is possible that metering of the flow rates of three material streams would obviate the need for monitoring the physical quality of the product. This, however, pre-supposes the availability of a plant scale system of segregating the granular stream into size fractions which would maintain a sensibly constant efficiency. The need for a means of monitoring size distribution is therefore not certain, but should this be required, Sharples and Dodds (ref 4) have described a small scale rotary screen and a small double deck vibrating screen, either of which may be adaptable for this duty. Provided the sample presented to the screen can be separated into the appropriate fractions with the required accuracy, the weight of sample would not be important as the proportional weights of the various fractions could then be determined.

Air classification methods have been examined but have not proved successful as the density and shape of the granules can influence the degree of separation.

Moisture Content

The strict control of moisture content is essential in the manufacture of high analysis granular fertilisers, yet there is no precise rapid method available to estimate moisture content. Vacuum drying at about 50°C gives reasonable accuracy but tends to be time consuming, and is therefore not very suitable for control purposes. Drying by infra-red lamps may be used for speed but at the expense of accuracy, and the usual compromise is to oven dry a small sample at atmospheric pressure at a constant temperature around 100°C, and within the range 80°C to 120°C. This last method has the merit of simplicity but care must be taken to avoid any risk of segregation within the sample and the method is subject to other errors such as temperature variation within the oven. Furthermore, the test is more accurately a determination of volatile loss at the particular temperature of test, and may therefore be dependent on the composition of the fertiliser.

Sharples and Dodds (ref 4) have described a relatively simple mechanical oven designed with the intention of standardising the heating and cooling cycle, but there is no time saving and the adaptation of any such system of heat drying for automatic control would present a great many problems.

An examination of the Karl Fischer method as applied to fertilisers has also been made. The results are influenced by water of hydration of monocalcium phosphate and calcium sulphate if it is present. Furthermore, the adaptation of this method to an automatic system would be particularly troublesome.

Physical methods based on determination of dielectric

effects have proved unsuccessful, but interest is now being shown in the use of microwave attenuation which varies according to moisture content. Watson, in 1957, published a paper (ref. 5) on the non-destructive determination of moisture content of bricks by this method and equipment is now commercially available. It has been found that calibration of the instrument is necessary for each mixing formulation and variation in bulk density between grades also contributes to this need for calibration. In order to get sufficient alternation, a rather large sample must be taken, but in some respect this is an advantage in reducing the likelihood of sample bias. It is not suggested that the microwave moisture meter is currently suitable for use in routine moisture determination, but the results to date are encouraging and there is therefore the possibility that further development could permit the installation of on-line moisture measurement and hence plant control.

Automatic Plant Control

It has been shown above that the equipment and methods for automatic monitoring of the important properties of the fertiliser product have reached promising stages of development.

Farquhar (ref. 6) has discussed the metering and control of ingredients for mixed fertilisers and continuous proportioning of both solid and liquid raw material feeds is now standard practice on granulation plants. Hence it should be possible to link raw material proportioning with product analysis, but the amount of recycled material and the hold-up or retention in the plant would lead to continuous hunting in any system of direct linkage. Furthermore, other process variables would come into play such as the dependence of granulation characteristics upon the raw material formulation used, the influence of drier gas temperatures upon granulation etc. The vital feature of any automatic control system must be the prediction of possible disturbances to the process which would thereby permit concurrent corrective action. That is to say, a successful system must depend on feed-forward of information to the controller, perhaps with the use of feed-back information to trim the control.

The controller must also be so programmed that tolerance limits can be set before changes in plant operation are effected, and these tolerances must apply at all stages of the process. Time delays may be necessary to allow for retention times in the various stages of the process, and a detailed study of the interactions of these variables must be made, probably for each grade of fertiliser to be manufactured. Overall plant control of this nature can probably best be achieved by computer. Computer engineers would require to study all the factors affecting product quality and plant stability in order to establish the programme necessary for computer control of the process. This, in itself, may require the installation of much metering equipment not considered necessary for manual control,

and the complexity and capital cost of the plant could be greatly increased.

It is very unlikely that installation of a computer solely for control of a granulation plant could be justified. However, the trend is toward the establishment of large fertilizer complexes where many processes and plants are operated. In circumstances such as these, the installation of a computer to integrate plant control throughout the complex may make the economics of automation much more attractive. There can be no doubt that the development of an effective system of granulation plant control can only be achieved by slow and painstaking effort and it may be regarded as a mere pipedream. Equally, it must be accepted that change and innovation are inevitable and the issue may ultimately resolve into a race between the development of automation and the obsolescence of the process.

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DISCUSSION

Mr. R.I.O. PASSMORE (Wisons Fertilizers Ltd., U.K.): Not unnaturally, manufacturers are reluctant to talk about their own product quality, and, in any case, legal tolerances vary widely from country to country. However, I have attempted in my paper to consider some of the implications of automatic control of a granulation plant, where perhaps the most intractable problem is also the problem of longest standing, i.e. that of handling solid material in process streams. I suspect that considerable ingenuity will still be required before we can surmount the difficulties. Given a solution to this particular problem, I think progress should then be straightforward.

Mr. W. MAK (Windmill Fertilizers, Holland): The automatic control of the granulating process is the ultimate dream of plant managers, but when one studies the subject one soon encounters such difficult problems that one tends to lose heart and to discount the dream as a vision not to be realised. We know from experience that an effective system of granulation plant control can be achieved only by slow and painstaking efforts. Hence, Mr. Passmore is to be congratulated on his courage in offering us a paper on such a subject. He reviews the present possibilities for modern automatic control and obliges us to re-examine the basic problems. However, I think the paper is more concerned with the automatic control of the end products of granulation than with the granulation process itself.

From our own experience, we know that sampling errors are often far greater than analytical errors. It is true that the higher the recycle ratio, the less is the variation in chemical composition, but then the process is less sensitive for deviations in the proportioning of raw materials. In this case, the analytical methods should be more accurate, if small but significant fluctuations in the NPK analysis are not to be overlooked over a long period. It was interesting to learn that with a recycle ratio of about 1 : 1 in an agglomeration process a minimum of 30 gm. granules should be taken to avoid bias. As far as I know, this is the first time such a figure has been published. I should like to ask whether such figures can be given for slurry granulation processes.

Did you study the possibilities of x-ray fluorescence? This method could possibly give results in solid material for total P_2O_5 and K_2O and particularly for trace elements. Water solubility could be analysed in a continuous stream cell. Only for nitrogen compounds could this method not yet be used.

With regard to size grading and moisture, further control of the granulation process immediately after granulation is very important, in view of the regulation of the recycle ratio and the determination of, for example, moisture content. Do you believe it is possible to mix the sticky material with some kind of powder, in a fixed weight-ratio, in order to make it more manageable for analysis? With regard to moisture, I believe the method of

continuous micro-wave attenuation is reliable. I agree that calibration is necessary for each mix and formulation. Also, variations in bulk density will affect results, but this would not be serious: all methods for moisture determination in fertilisers are to some extent a compromise between the determination of free water, combined water and volatile matter. Finally, I should be very careful in linking automatically the raw material proportioning with product analysis: this would only be possible if the sum of the errors in the results of analysis and sampling are less than legal tolerance limits.

Mr. PASSMORE:

Methods of handling and analysis of end products may well be applicable to handling solid raw material feeds, and for this reason I may have overemphasised their importance. There can be no doubt that if automatic plant control is to be achieved, monitoring the raw material feeds will be of greater importance than monitoring the product analysis. I think that the technique in this case would be that analysis of the forward-going process streams would be carried out and fed to a computer, and that, in effect, product analysis would only trim this information.

The question of minimum sample size must obviously be influenced by the legal tolerances within which one is working. In our case, we have found that 30 gms. is about the minimum we can use, and with slurry products, we would need to take a sample size of about 20 gms. to achieve equivalent accuracy.

We have the necessary equipment for studying x-ray fluorescence, and it is in use on a research basis. The inability to determine nitrogen by this method seems to be a particular drawback, especially when one is concerned to know not only the total nitrogen content but also the forms in which nitrogen is present. However, I understand that work is progressing on a suitable cell for using x-ray fluorescence on nitrogen. If this is successful, this method would no doubt represent a very elegant technique to use.

Your comment on the handling of material taken from the granulator is a very valid point, but I wonder whether the addition of an inert material would be sufficient to prevent the stickiness. I had thought rather of quenching the material in some sort of organic solvent or perhaps in thin oil.

With regard to analysis, provided that one can be fairly sure of the concentration of one of the plant foods, a proportional NPK determination would perhaps be adequate, rather than an absolute determination.

With regard to the microwave moisture meter, I agree with you that this is a valuable technique. One is concerned to provide a parameter for plant control and not with the absolute determination of the water content of any process stream. The microwave moisture meter will, of course, determine the free water in the material and will not take account of water which is bound as water of crystallisation. But there are certain points to watch out for: we have found in determining moisture

in a 13-13-20 formulation that the composition has a very marked influence on the attenuation which can be obtained. If one considers a formulation using mainly TSP as the P_2O_5 carrier, with a small proportion - say 5% - of MAP, the attenuation would be only about half that obtained with a formulation in which the P_2O_5 is derived almost entirely from MAP. We have also found that the temperature of the material has some effect on attenuation, as does the age of the sample, particularly in the case of material straight from the drier as compared with material which has stood at room temperature for, say, half an hour. Nevertheless, provided one takes account of these possible pitfalls, I think the microwave moisture meter offers a very useful technique. The equipment could presumably be installed on an industrial plant without undue trouble, and one of the manufacturers has also proposed that we should use an on-line method of analysis by having a solid-wave guide.

Finally, I agree that the link between raw material and product analysis would be absolutely essential.
