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THE AUTOMATIC DETERMINATION OF PHOSPHATE AND SULPHATE IN  
WET PROCESS PHOSPHORIC ACID

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INTRODUCTION

Several methods have been proposed for plant control, using gravimetric, volumetric and turbidimetric techniques for the determination of sulphate and phosphate in wet process phosphoric acid. In almost all cases the methods have been designed to give highest accuracy consistent with ease of manipulation and minimum time required for a determination.

In 1963 a method was proposed by Baumann et al (1) for the automatic determination of sulphate ion in wet process phosphoric acid, using a turbidimetric method and the Technicon Autoanalyser. In this paper, the authors referred to the adherence of precipitate to the cell walls as the main drawback to continuous measurement, and consequently aimed at intermittent readings with continuous sampling.

Using the normal turbidimetric procedures, the adherence of precipitate to cell walls had been confirmed in our laboratory. However, the use of Nonex (Methoxy-poly-ethylene glycol laurate) previously reported (2) as a stabiliser for the barium sulphate precipitate, enabled continuous determinations to be made by means of the Technicon Autoanalyser, for periods of up to 2 hours before adherence of precipitate to cell walls seriously affected the accuracy of the results. It was in an effort to overcome the necessity for frequent cleaning of the cell and transmission lines that further work was carried out.

Experience with the spectrophotometric vanadomolybdo phosphate method for the determination of phosphate in fertilisers, using the Autoanalyser enabled the necessary pump tube sizes to be selected for application to phosphoric acid analysis. Unfortunately, the need for several dilution steps in the automatic procedure reduces the accuracy of a determination. Because of this, it was felt necessary to adopt a partial differential method to this determination.

PART 1.THE DETERMINATION OF SULPHATE IN PHOSPHORIC ACID.

For a number of years a turbidimetric method has been used for the determination of sulphur trioxide in contact-plant exit gases. In the procedure the sulphur trioxide and sulphur dioxide are absorbed in a mixture of water and isopropanol, which was shown by Flint (3) to inhibit the oxidation of sulphur dioxide to sulphur trioxide. In his method, Flint determined the sulphate gravimetrically, however, to speed up the determination a turbidimetric finish has been used. In this method it has been observed that the barium sulphate, precipitated in the presence of isopropanol, does not readily adhere to the surface of flasks or cells. The method using isopropanol was therefore examined more fully on the Technicon Autoanalyser and the reagent concentrations and Autoanalyser conditions were optimised to give an acceptable sensitivity, reproducibility and linearity.

Details of the Technicon Autoanalyser, the function and use of the various components, have been described in several publications, 1,4,5,6, during recent years, and it is not proposed to describe the individual units in any detail in this paper.

Preliminary experiments enabled the size of pump lines to be fixed to give a useful range of 0-6.5g  $H_2SO_4/100$  ml, over 100%-30% transmission or 0-0.5 optical density<sup>4</sup>. Later work indicated that above a level of 7.0g  $H_2SO_4/100$  ml, marked deposition of barium sulphate occurred<sup>2</sup> in the "colorimeter" cell, however, for all practical purposes, an upper level of 6.5g  $H_2SO_4/100$  ml, was considered satisfactory. It was also found necessary to express concentration of sulphate and phosphate on a weight/volume basis, since a variation in specific gravity which occurred with a variation in either phosphoric acid or sulphuric acid concentration affected the results. This is due to the fact that the sample line, in conjunction with the peristaltic pump unit, abstracts samples at a constant volume, not constant weight. The possible effect of using a weight/weight relationship as opposed to a weight/volume relationship can be seen from Table 1. below, which gives details of the calibration standards used in this work.

TABLE 1.

Composition and specific gravity of calibration standards.

<u>H<sub>2</sub>SO<sub>4</sub> g/100 ml.</u>	<u>P<sub>2</sub>O<sub>5</sub> g/100 ml.</u>	<u>Specific Gravity</u>
0.40	42.3	1.292
1.02	40.0	1.279
2.28	48.0	1.339
3.76	34.5	1.254
4.93	45.7	1.333
6.16	41.1	1.311
7.40	37.5	1.297

The effect of isopropanol and barium chloride concentrations.

Preliminary experiments indicated that maximum sensitivity coupled with a linear relationship between sulphuric concentration and optical density depended upon the relative concentrations of isopropanol and barium chloride. \* The optimum reagent concentrations were established by a further series of experiments which showed that an isopropanol concentration of 45% v/v and a barium chloride concentration of 6g/litre were required.

The Autoanalyser layout finally adopted is shown diagrammatically in Figure 1.

A typical record of calibration standards is shown in Figure 2.

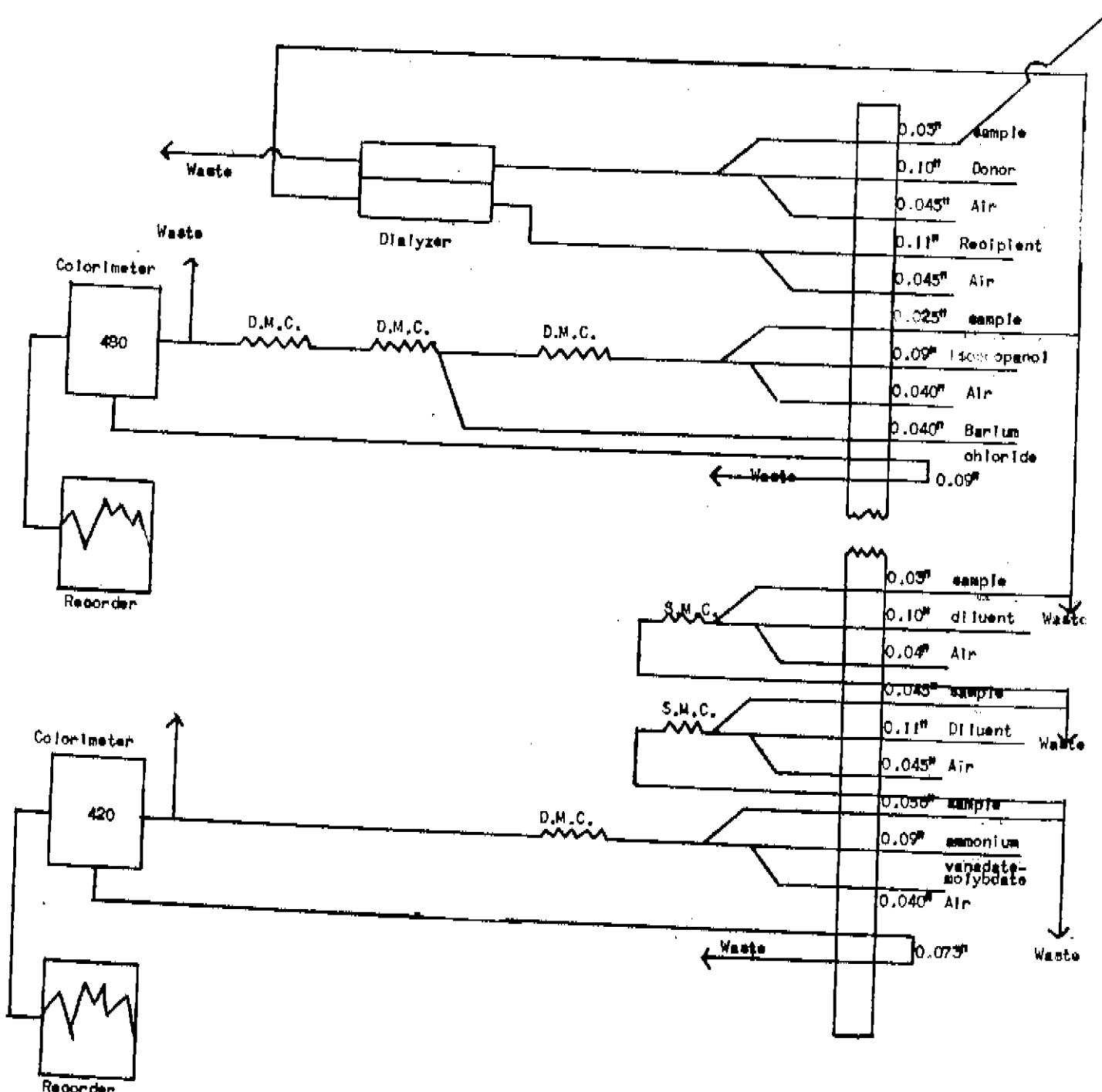
Techniques of analysis.

Two techniques have been examined for the determination of sulphuric acid in phosphoric acid. These employ intermittent sampling and continuous sampling. In both cases the sample fed to the Autoanalyser consisted of phosphoric acid filtrate. For continuous sampling, plant conditions after filtration of the sample were simulated in the laboratory.

Intermittent sampling.

In this technique separate samples are presented to the Autoanalyser, utilising the sampling plate device. The samples may be analysed at a rate of either twenty or forty an hour. At twenty analyses an hour, both sulphate and phosphate are satisfactory and no additional wash is required between samples. At forty samples an hour, sulphate determinations are satisfactory, but phosphate peaks do not reach the maximum peak height. It is recommended that a rate of twenty samples per hour are analysed on intermittent sampling.

FIGURE 1



S.M.C. = Single mixing coil  
 D.M.C. = Double mixing coil

TECHNICON RECORD OF A TYPICAL SERIES OF STANDARD SAMPLES

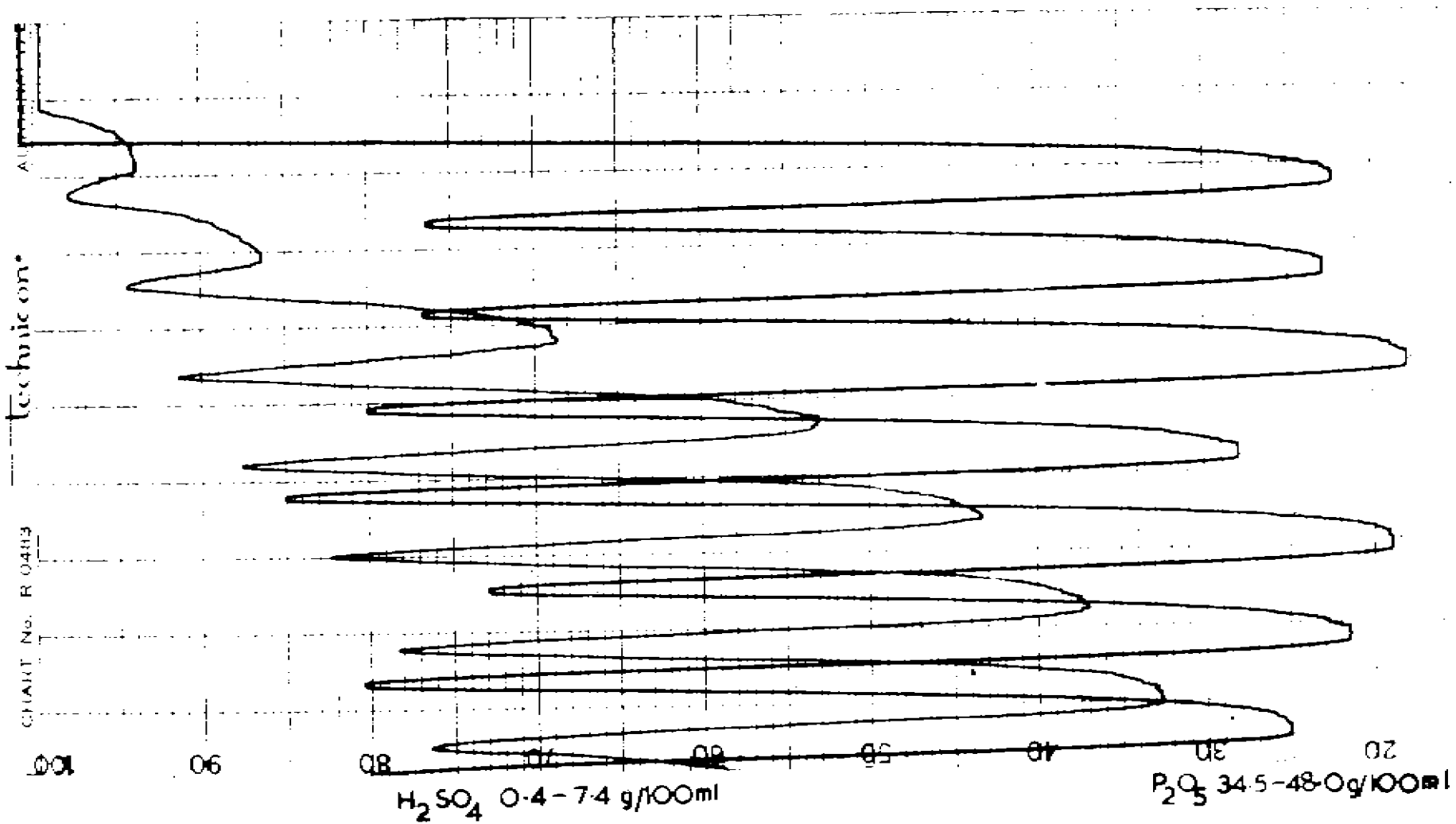
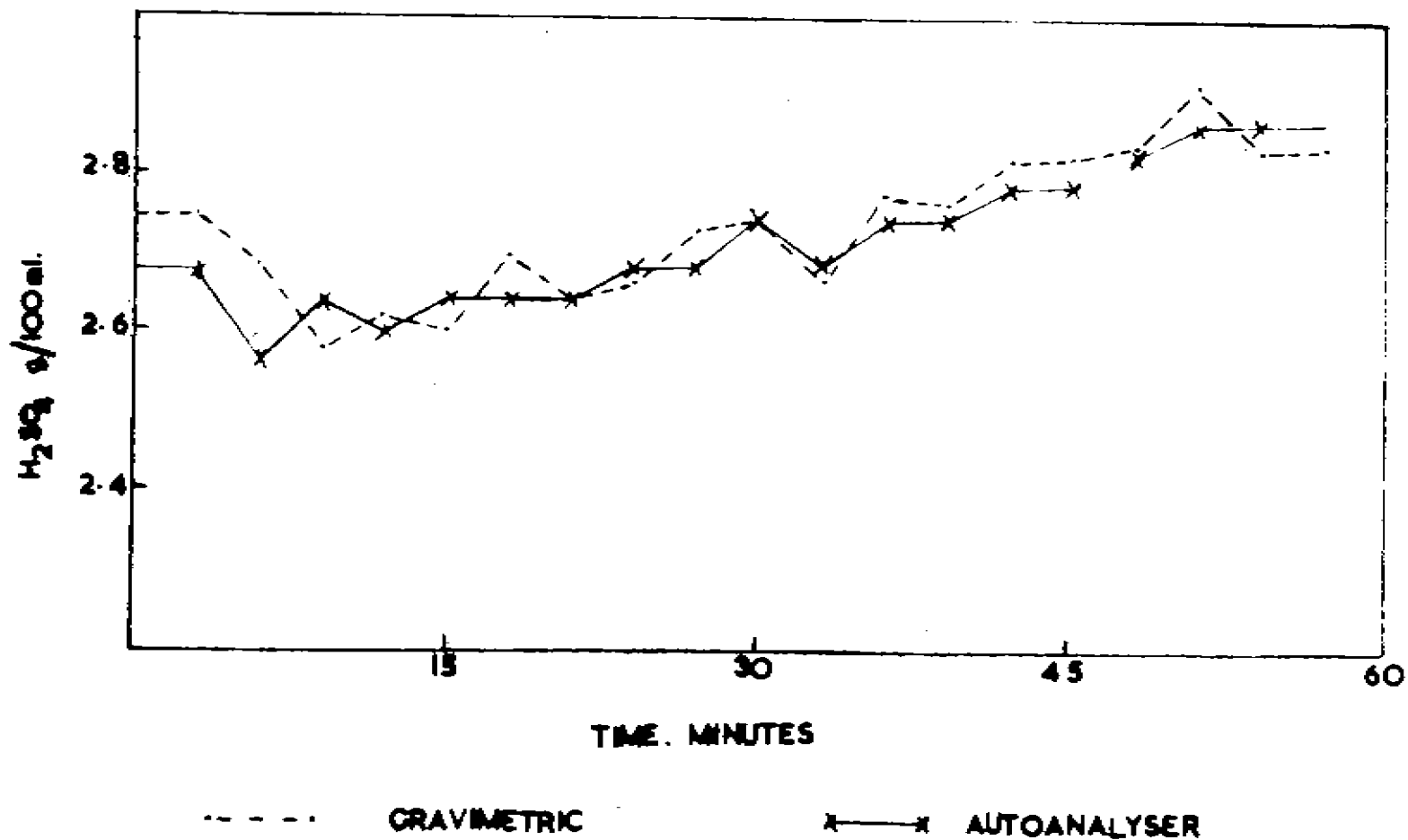


FIGURE 2

THE DETERMINATION OF SULPHURIC ACID IN PHOSPHORIC ACID.

A COMPARISON OF GRAVIMETRIC AND AUTOANALYSER RESULTS.

INTERMITTENT SAMPLING.



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FIGURE 3.

The results obtained on a series of twenty samples obtained at 3 minute intervals from the production unit during steady operating conditions and analysed by the gravimetric and Autoanalyser methods are shown graphically in Figure 3. A statistical comparison of these results gave a t value of 0.084, with a t critical value (19 D.F.) of 2.86 at the 1% probability level. This confirms the satisfactory agreement between gravimetric and Autoanalyser methods.

The reproducibility of the method during a period of thirty minutes was determined by carrying out replicate analyses of two samples containing 3.51g and 4.95g  $H_2SO_4/100$  ml.

TABLE 2.

<u>Intermittent Sampling</u>		<u>Statistical summary</u>	
<u>g <math>H_2SO_4/100</math> ml</u>	<u>No. of Analyses</u>	<u>Standard Deviation</u>	<u>Coefficient of Variation.</u>
3.51	10	0.035	1.00%
4.95	10	0.056	1.13%

With intermittent sampling it has been found necessary to adjust the baseline at intervals of eight hours. Precipitation in the cell is only slight after this time, and it is necessary to flush the system with a solution containing 50g diamino ethane tetra acetic acid disodium salt and 200 ml 0.880 ammonia solution per litre, for five minutes every twenty four hours. This wash completely cleans the system of any precipitated barium sulphate.

#### Continuous sampling.

A continuous record of the sulphate concentration is the ideal for plant control. This requires a continuous sample to be fed to the instrument from a suitable point in the plant, followed by a method which requires the minimum of attention from operators.

In this work the technique employed is similar to that used for intermittent sample analysis, and is as shown in Figure 1.

In the continuous sampling and analysis technique, the slow deposition of barium sulphate is more noticeable than with the intermittent sampling. In addition the long term instability of the analyser system must be taken into account.

Preliminary experiments indicated that the slow deposition of barium sulphate was of little consequence, providing that the baseline was adjusted to 100% transmission every four hours. During a period of four hours the barium sulphate deposited in the cell was found to be equivalent to an increase of 0.15g  $H_2SO_4/100$  ml and in 24 hours approximately 1.0g  $H_2SO_4/100$  ml if



the baseline was not adjusted. The increase in a four hourly period is of little importance with regard to plant control, but would be unacceptable over a 24 hour period.

Statistical data derived from a series of results taken at hourly intervals, for a period of twenty six hours, from a continuous analysis of a sample of constant composition is given in Table 3.

TABLE 3.

Continuous sampling. Determination of sulphuric acid.  
Statistical summary of 26 hourly results.  
Baseline adjusted at 4 hourly intervals.

<u>No. of Results</u>	<u>H<sub>2</sub>SO<sub>4</sub> content g/100 ml</u>		<u>Standard Deviation</u>	<u>Coefficient of Variation %</u>
	<u>Range</u>	<u>Average</u>		
26	2.86-3.24	2.98	0.132	4.43

An alternative procedure that is suggested employs a 5 minute wash every 4 hours using the diamino ethane tetra acetic acid disodium salt/ammonia solution previously described.

The ability of a continuous analysis system to respond to changes in the process is important, and this has been demonstrated. Samples were obtained at ten minute intervals from a vessel in which there was a continuously varying concentration of sulphuric acid in wet process phosphoric acid. The concentration of sulphuric acid was determined continuously by the Autoanalyser and on the samples taken at ten minute intervals by the Gravimetric method.

The results are given in Table 4. below.

TABLE 4.

Response of continuous sampling procedure to changes in sulphuric acid concentration.

<u>Time Mins.</u>	<u>Gravimetric.</u>	<u>H<sub>2</sub>SO<sub>4</sub> g/100 ml.</u>		<u>Difference.</u>
		<u>Autoanalyser.</u>		
0	3.86	4.10		- 0.24
10	5.19	5.15		+ 0.04
20	5.57	5.56		+ 0.01
30	6.29	6.10		+ 0.19
40	6.24	6.12		+ 0.12
50	5.16	5.26		- 0.10
60	4.33	4.40		- 0.07
70	3.11	3.16		- 0.05
80	2.49	2.58		- 0.09
90	2.34	2.40		- 0.06

PART 2.THE DETERMINATION OF PHOSPHORIC ACID IN WET PROCESS PHOSPHORIC ACID.

The layout of the Autoanalyser for the determination of phosphoric acid is shown in Figure 1. and is a modification of that used in the analysis of fertilisers. Due to limitations in the optical system of the colorimeter, a truly differential vanadomolybdo phosphate method could not be used, and the nearest alternative was to set the recorder baseline at 12 g  $P_2O_5/100$  ml. Even with this technique, full scale deflection of the recorder is equivalent to about 75g  $P_2O_5/100$  ml, thus a recorder error of  $\pm 1\%$  is equivalent to  $\pm 1.2g P_2O_5/100$  ml, at the normal level of concentration of 40g  $P_2O_5/100$  ml.

Techniques of analysis.

As for the determination of sulphuric acid, two forms of sampling were examined, namely intermittent and continuous.

Intermittent sampling.

Separate samples were presented to the Autoanalyser via the sampling plate at a rate of 20 samples per hour. These samples, preceded by a series of calibration standards, were analysed for  $P_2O_5$  content by the gravimetric modification of the quinolinium phosphomolybdate method described by Perrin (7) and by the Autoanalyser method.

A statistical comparison of these results gave a t value of 1.4 with a t critical value (10 D.F.) of 3.17 at the 1% probability level, indicating the satisfactory agreement between gravimetric and Autoanalyser methods.

The reproducibility of the method during a period of thirty minutes was determined by replicate analyses of two samples containing 42.4 and 47.9 g  $P_2O_5/100$  ml.

Results of the statistical tests are given in Table 5.

TABLE 5.

<u>Intermittent sampling.</u>		<u>Determination of phosphate.</u>	
<u>Statistical summary.</u>			
<u><math>P_2O_5</math> g/100 ml.</u>	<u>No. of Analyses.</u>	<u>Standard Deviation.</u>	<u>Coefficient of Variation.</u>
42.4	10	0.860	2.03%
47.9	10	0.445	0.93%

Continuous sampling.

The technique for continuous phosphate determination is the same as that used for intermittent analysis.

The main difficulty encountered in continuous analysis has been the stability of the colorimeter and recorder system, since any slight variation has a marked effect on the accuracy at the transmission level used.

It has also been shown that two additional dilutions required in the phosphate method to reduce the phosphate concentration to a suitable level for colorimetric determination affects the reproducibility. Thus the standard deviation of a determination in which the dilution was carried out manually was 0.245 with a coefficient of variation of 0.57% at the 43g P<sub>2</sub>O<sub>5</sub>/100 ml level.

Statistical data derived from a series of results taken at hourly intervals, for a period of twenty six hours, from a continuous analysis of a sample of constant composition is given in Table 6.

TABLE 6.

<u>No. of Results</u>	<u>Continuous analysis.</u>		<u>Determination of phosphate.</u>	
	<u>Statistical summary of 26 hourly results.</u>			
	<u>Baseline adjusted at 4 hourly intervals.</u>			
	<u>P<sub>2</sub>O<sub>5</sub> content g/100 ml.</u>		<u>Standard</u>	<u>Coefficient</u>
	<u>Range.</u>	<u>Average.</u>	<u>Deviation</u>	<u>of Variation</u>
26	42.4 - 46.8	44.4	1.32	2.98%

CONCLUSIONS AND DISCUSSIONS.

Sulphuric acid and phosphate can be determined simultaneously in wet process phosphoric acid streams by means of the Technicon Autoanalyser. The method developed enables the Autoanalyser system to operate for long periods with little operator attention. The difficulties of barium sulphate deposition usually encountered in the turbidimetric determination of sulphuric acid have been markedly reduced, and cleaning time of the system has been reduced to only five minutes every twenty four hours.

The technique used enables a result to be obtained within 10 minutes, when both sulphuric acid and phosphate are determined simultaneously. The sulphuric acid determination can be completed within seven minutes, while the phosphate determination requires ten minutes because of the need for two additional dilutions.

If normal calibration checks and standardisations are carried out every four hours, accurate and reproducible results can be obtained. An improvement in the accuracy of the determination of phosphate might be obtained by the use of the Technicon automatic standardisation technique, used more frequently than the presently suggested standardisation technique.

The automatic standardisation technique enables a standard sample to be injected into the system in place of the sample at any pre-determined time. The output from the colorimeter to recorder is then automatically adjusted, if necessary, to a pre-fixed value.

#### ACKNOWLEDGEMENTS.

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APPENDIXTHE DETERMINATION OF SULPHATE IN WET PROCESS PHOSPHORIC ACIDPRINCIPLE.

Sulphate is determined gravimetrically in the presence of excess hydrochloric acid to prevent co-precipitation.

REAGENTS.

Barium chloride.	10% solution.
Hydrochloric acid.	Concentrated.

PROCEDURE.

Accurately weigh about 2 g of phosphoric acid and transfer to a 500 ml beaker, dilute to approximately 200 ml with water and heat to boiling. Add sufficient hydrochloric acid to give a concentration equal to 1.0N, i.e. 10 ml concentrated hydrochloric acid per 100 ml solution. Add 10 ml of boiling barium chloride solution and allow to continue boiling for five minutes. Remove from heat and allow to stand for 1 - 2 hours. Filter using a tared No. 4. porosity silica crucible, wash well with hot water until free from chloride, and carefully ignite at 900°C for 10 minutes using a Meker burner or muffle furnace. Cool in a desiccator and reweigh.

CALCULATION.

$$\frac{\text{Weight Barium sulphate} \times 0.4202}{\text{Wt. of sample.}} = \% \text{H}_2\text{SO}_4$$

NOTE.

It is important to ensure that the hydrochloric acid concentration is 1.0N.

DISCUSSION

Mr. G. NELSON (West Norfolk Fertilisers Ltd., U.K.): A knowledge of the phosphate and sulphate concentration in reaction slurries in wet process phosphoric acid manufacture is required for process control. A continuous record of these parameters should enable complete manual or automatic control of a phosphoric acid plant. Several methods are in use for the determination of sulphate, but in our study of sulphate determination on the Technicon Auto-analyser, we decided to look at the turbidimetric method and to determine the various parameters required to use it.

The main difficulty encountered with continuous turbidimetric methods is the rapid deposition of precipitate on the cell walls. This leads to a marked increase in optical density and hence to unreliable results. Several techniques were tried, but we always found this rapid deposition on the cell walls. Finally, the precipitation of barium sulphate in an isopropanol medium was examined in more detail and was found to give rise to a precipitate which did not readily adhere to cell walls or to the transmission tubes used in the equipment. The concentration of isopropanol and barium chloride was examined to select the best conditions, using a randomised block experiment of 36 experiments, in which a series of 6 standards were analysed, using various levels of isopropanol and barium chloride. Of the 36 calibration curves obtained, only 2 gave any agreement with Beer's Law. These two were very closely related in concentration - 45% isopropanol and 6 and 8 gms. of barium chloride respectively. And so we chose one of these lines - the 6 gms. of barium chloride and 45% isopropanol - for our further work.

Having selected these optimum conditions, the method was examined both on the intermittent and on the continuous sampling techniques. For intermittent analysis, the sampling plate device was used to feed samples to the Auto-analyser. The preferred rate was 20 per hour, mainly because of difficulties with the phosphate determination, in which the peaks did not reach a maximum at the rate of 40 per hour.

Using this technique, the deposition of barium sulphate is only slight, and the base line adjustment is required after about 8 hours of operation. Every 24 hours we flush the system with EDTA ammonia solution for about 5 mins. This completely clears the cell and lines of any precipitated barium sulphate.

We find that with the Technicon Auto-analyser, the peaks obtained for sulphate determination are quite satisfactory, although for phosphate determination we obtain rather flat peaks.

The coefficient of variation for this method is about 1%, and the comparison of results by the Analyser and gravimetric method shows close agreement.

Continuous analysis was investigated by examining a wet process phosphoric acid of known sulphate concentration for long periods on the Auto-analyser. It was found that a slow deposition

of barium sulphate occurred which, over a period of 4 hours, was equivalent to about 0.15 gms sulphuric acid per 100 ml. We found it necessary to compensate for this by adjusting the base line every 4 hours or, alternatively, incorporating an automatic wash with EDTA and ammonia.

The response to changes in sulphate concentration is good, and the rate of response is limited only by the rate at which fresh samples can be presented to the Analyser. Typical of the response to a change in sulphate concentration are the figures shown in our Table 4, where samples were taken at intervals and examined by the gravimetric method and were found to give very good correlation.

The determination of phosphate follows the technique used for fertiliser analysis, which we have operated for about 2½ years. This makes use of the colorimetric vanado-molybdate method. In order to reduce the phosphate level to make it suitable for this colorimetric determination, we incorporate a dialysis step, followed by two dilution stages. This affects the reproducibility to some extent, as also does the additional effect of recorder instability. These effects can be seen by comparing the standard deviations and coefficients of variation used under different conditions.

Since writing the paper, we have made a number of improvements. We are hoping to improve the sulphate method, so that we shall be able to eliminate the need to clean the colorimeter cells, or at least not need to clean them for very long periods.

With regard to the phosphate method, the poor photo-cell response at 420 m $\mu$  is a serious drawback, when determining phosphate by this method, and we have now replaced the vanado-molybdate method with a molybdenum blue method. This improves the photo-cell response, since we measure the optical density at 630 m $\mu$ .

Two other additions which we think will also lead to an improvement are, firstly, the internal standardisation technique which Technicon has developed, and, secondly, the substitution of our present recorder for a zero-adjustable recorder with scale expansion. This should improve the accuracy and reproducibility of the phosphate method.

Mr. R. DEWALD (Chemische Werke Albert, Germany): In the manufacture of wet-process phosphoric acid, the determination of sulphate for the purpose of controlling the reaction is of particular importance. The sulphate content influences the formation of the gypsum crystals and hence the filterability of the gypsum slurry. To some extent, therefore, it also determines the economics of the whole plant.

Phosphate determination is also important for the control of filtration, particularly for the washing of the gypsum.

For practical plant control, volumetric methods of SO<sub>2</sub> determination, particularly the benzedine method, were introduced, but in recent years these have been hard pressed by the more accurate and above all more elegant turbidimetric method. As an indication of

the  $P_2O_5$  content, density determination is often sufficient.

As we have heard, an ingenious control apparatus has been successfully developed, which will indicate or record, and which operates according to the turbidimetric or colorimetric principle.

There is nothing to be said with regard to the chemistry and the principle of measurement in respect of either the  $SO_3$  or the  $P_2O_5$  determination, and questions in these respects seem unlikely. On the other hand, it seems to me desirable if the following points could be clarified:

1. Have long term experiments been made with the described apparatus both for  $SO_3$  determination and for  $P_2O_5$  determination?
2. A prerequisite of continuous indication, particularly of the  $SO_3$  content in a slurry, is perfect sampling and perfect operation of the sampling apparatus. Have you carried out experiments in this respect?
3. I think that the apparatus described, developed for industrial operation, is of great interest and value for all phosphoric acid producers and represents a genuine advance. I know from experience, however, that in the practical operation of a plant, preference is always given to the simpler apparatus requiring less attention. The method described in the paper requires a series of different though proved pieces of apparatus, and also necessitates the continual use of chemicals. A discussion of its future prospects in comparison with purely physical systems of control would certainly be most instructive. I am thinking in particular of the device developed by CUF and Dorr Oliver.

Mr. NELSON: With regard to long-term experiments, our equipment has been in use for periods of between 72 and 100 hours continuously. In these tests, we have presented continuous tests in the laboratory. We have had no difficulty and have run it for several hundred hours on this particular determination.

We are fully aware of the difficulties of sampling. So far, we have worked on samples taken from the plant and filtered manually. We are considering separating solids from liquids by a hydro-cyclone technique, or something similar, where the complete removal of solids is unnecessary, since the dialyser system acts as a filter as well as a dilution mechanism.

With regard to maintenance and the supply of chemicals, we have now been using the Technicon Autoanalyser for  $2\frac{1}{2}$  years and have found it to be very reliable. It does not require skilled operators. The preparation of chemicals is fairly simple, and since one can standardise at very frequent intervals, large batches of prepared chemicals will last for several weeks.

As far as other methods are concerned, I have no doubt that methods which do not involve the use of chemicals are highly



desirable for this particular determination; but I think it probable that, in this case, certain electrodes might be liable to scaling and might thus require maintenance. My experience of conductivity measurements in other systems suggests that scaling of platinum electrodes can be very rapid and can lead to erroneous results.

Mr. R. BAUWENS (Ets. Kuhlmann, France): Has the author considered a particular aspect, viz. the supersaturation of the phosphoric acid samples in calcium phosphate. In other words, is this degree of supersaturation always constant?

Mr. NELSON: We have examined the question of supersaturation, and in our particular plant over periods of many months we have been unable to detect large differences in degrees of supersaturation. They are roughly equivalent to about 0.15% of sulphate based on a calcium determination. I think that at the 2 - 3% sulphate level, at which our plant and probably many others are operated, this supersaturation is probably constant and could be possibly disregarded.

Mr. J. DU FOUR (Union Chimique-Chemische Bedrijven, Belgium): We have two Technicon units in service in our plant. We are trying to use them for the determination of  $SO_4$  ions in phosphoric acid manufacture.

On page 1 you speak of a solution of barium chloride and mention, in particular, the method proposed by Baumann. Now Mr. Baumann used a solution of neutral barium chloride. What is the composition of your solution of barium chloride?

What is the counter-dialysis liquid?

Your results have been obtained by laboratory work. Have you installed a Technicon unit in the factory, and do you have any results of a true industrial control?

How did you sample the acid? What device was used to reduce the delay in the analysis?

To what extent does your laboratory staff intervene for the maintenance and control of the apparatus?

Mr. NELSON: The composition of the barium chloride solution, as stated in the paper, was 6 gms. of barium chloride per litre of water, with no addition of acid. I believe Baumann uses 5% phosphoric acid, but we did not find this necessary.

We have not put a Technicon unit directly on our plant, but for plant use it would normally be built into a box which would be placed adjacent to the plant.

With regard to sampling, we plan to take samples from the

plant via a pump line from the reaction vessel, carrying out a very rough separation of the gypsum from the acid - taking no more than a few seconds - and then passing straight to the Technicon. The time involved will probably be less than half a minute, and the sulphate determination will take 6 - 7 minutes.

Extracting samples at the rate of 20 per hour, we adjust the recorder only every 8 - 12 hours. With continuous sampling, we think adjustment of the base line should be made every 4 hours.

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