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                    ACIDULATION OF EGYPTIAN
SIBAIYA PHOSPHATES - SOME FACTORS AFFECTING THE EQUILIBRIUM:
    "Moisture - Free Phosphoric Acid - Water Soluble P_2O_5 -
                    Total P_2O_5".

    by A.F. Sabry, B.Sc., M.Sc., Chemist, Sté.Financière et
                                Industrielle d'Egypte S.A.E.
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INTRODUCTION TO EGYPTIAN SIBAIYA PHOSPHATES.

          A several years' practice in the acidulation of Egyptian
Sibaiya phosphates indicates that in the same district, Sibaiya,
exist two types of the mineral phosphorite, which belong to two
different mines. These two types, although not so distant from
each other, possess different physical and chemical character-
istics. For the present investigation a ground sample of each
type has been prepared, the degree of fineness of which being
lower than the usual practice. Portions of these samples were
removed separately and more finely ground so that from the two
types designated as A and B, samples A1 and B1 with coarser
average particle size and samples A2 and B2 with reduced particle
size were prepared. The samples from the two types have been
chosen from the same grade which is used in acidulation in Egypt.
The range of particle size of each sample has been determined by
screening a weighed portion through a suitable series of standard
sieves of known aperture sizes. Results of screening are found
in tables 1 and 2.

          Analysis has been carried out on the two types for the
contents of phosphate and other important ingredients. Results
are found in table 3.

                        TABLE 1.

Maximum particle size - microns	Percent of sample A1	Percent of sample B1
More than 330	5.4	4.1
330	12.2	9.4
180	8.7	7.7
142	26.6	27.4
75	47.0	51.4
TOTAL	99.9	100.0

TABLE 2.

Maximum particle size – microns	Percent of sample A2	Percent of sample B2
More than 180	3.7	2.1
180	5.2	4.2
142	5.1	4.2
118	16.7	17.5
75	69.3	71.9
TOTAL	100.0	99.9

TABLE 3.

INGREDIENTS	% In Phosphate A.	% In Phosphate B.
Water soluble portion		
Moisture	1.31	0.96
Chloride as NaCl	0.45	0.07
Sulphate as $CaSO_4$	1.67	0.33
Acid soluble portion		
Tricalcium phosphate	64.97	65.98
Calcium Carbonate	11.26	8.53
Calcium fluoride	2.77	3.12
Fe and Al oxides	2.18	3.74
Insoluble portion	6.56	10.76

It can be seen that, although the two types are of about the same phosphate content, yet they differ as regards all other ingredients, the difference being so considerable that a single ingredient may be sufficient to differentiate between the two types.

In the water soluble portion, the chloride determination has been carried out by Mohr's method, the sulphate by the ordinary barium sulphate method (13). Considering the acid soluble portion the phosphate has been determined by the recently developed method of Kassner, Kummer and Ozlor (5), the carbonate by the commonly used method of acid decomposition and absorption of CO_2 evolved with soda lime (12), the calcium fluoride by the method of Berzelino, improved by J.I. Hoffman and G.E.F. Lundell (4) and the iron and aluminium as oxides after previous precipitation as phosphates and removal of phosphate.

The insoluble portion has been determined by the method mentioned by Barton (2) as used by most Florida phosphate chemists.

ACIDULATION OF SIMPLE SUPERPHOSPHATE

The complicated reactions taking place in the process of acidulation, which may be thought a simple mixing operation, are numerous and can neither be assumed to take place to completion nor to a certain extent under the various conditions of different plants and countries.

Following recent studies (7) separate processes represented by chemical equations can be assumed to represent, approximately, those

taking place in the acidulation process. An attempt to relate the superphosphate produced to the phosphate rock used is found in Schucht's results (3), who calculated the weight of superphosphate produced by multiplying the weight of phosphate rock used by the ratio. :

Total P_2O_5 in phosphate / Total P_2O_5 in superphosphate.

Thus $w_1/w = x/x_1$ where w = weight of phosphate rock, x = % P_2O_5 in rock, w_1 = weight of superphosphate produced, and x_1 = % total P_2O_5 in superphosphate. This general formula can be applied under all conditions.

It can finally be stated that from the mixing process, which is always accompanied with loss in weight due to evolution of gases and water vapour, results an equilibrium mixture between different phases and ingredients. A liquid phase is represented by a solution of phosphoric acid in water saturated with monocalcium phosphate and containing other soluble minor ingredients. The solid phase mainly includes calcium sulphate, different forms of calcium phosphates, insoluble material and other minor ingredients.

Equilibrium: Moisture - Free Phosphoric Acid - Water soluble P_2O_5 - Total P_2O_5.

From the industrial point of view, it may be more convenient to consider among the above mentioned ingredients the water and phosphatic material. Thus, a superphosphate sample may be regarded as a system including the equilibrium: Moisture - Free phosphoric acid-Water soluble P_2O_5 - Total P_2O_5. The factors affecting this equilibrium are classified by the author as:

1. Factors belonging to phosphate rock.
2. Factors belonging to sulphuric acid.
3. Factors due to external conditions.

Factors belonging to phosphate rock are: the type of rock and the fineness of grinding. The type of rock includes its composition, hardness, etc., etc. For example, the iron and aluminium oxides are considered as objectionable ingredients if present in high proportions in the rock, while some authorities claim to obtain good results using rocks with high iron and aluminium contents (17). The fine grinding of rock is generally considered a good practice in superphosphate manufacture (15).

Factors belonging to sulphuric acid include its concentration and temperature, which are considered as separate problems for each individual plant, depending on its specific conditions. The quantity of acid applied to a certain quantity of rock, comes in the most important place among other factors. The bases on which the quantity of acid to be applied has to be predetermined for a certain rock, are not so accurate as to be applied to all types of rock (6), (10).

Recently Shoeld, Wight and Sauchelli (11) made an important contribution to the study of "Rock-acid ratio in superphosphate manufacture". They studied the effect of quantity of acid of certain concentration applied to a fixed quantity of phosphate rock, upon the percentage conversion to available P_2O_5. The nomographs, developed by the authors, illustrate this relation and the relation between the percentage conversion and the available and total P_2O_5 contents in superphosphate, both in combination with rock composition in terms of its P_2O_5 and CO_2 contents only. As the citrate solubility is the measure of availability in the United States, the authors' study has been confined to the "available" P_2O_5. They found that there exists an optimum rock-acid ratio at which the material cost per unit available P_2O_5 is minimum.

To the external factors belong the ageing and weather conditions during ageing. Some phenomena due to the ageing factor have been cited (8) and (16). Considering the equilibrium under study, it can be stated that this equilibrium is unstable in fresh super-

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phosphate, and that important changes take place in its ingredients on ageing, which were found to be affected by the prevailing weather conditions during the ageing period.

FACTORS STUDIED IN THE PRESENT WORK.

Study on the acidulation of phosphate A began by first fixing the factors belonging to phosphate rock, i.e., by using the sample A_1. The concentration of acid was also kept constant throughout and so was its temperature which agreed with the room temperature.

Concentration of acid H_2SO_4	68.94 %
Specific gravity at 20° C	1.6020

The acidulation involves the preparation of laboratory superphosphate samples, using for each sample 200 grams of the ground rock and the desired weight of the above acid. After well mixing, the thickened mass was kept in the electric oven at 105°C for one hour, after which it was quantitatively discharged, kept for 3 days, weighed, sampled and analysed for moisture, free acidity, water soluble P_2O_5 and total P_2O_5. The principal factor, which is the quantity of acid per 200 grams rock, has been studied by the preparation of superphosphate samples containing increasing quantities of acid, carrying out their analysis for the figures of the equilibrium after three days, the room temperature during this period being recorded in daytime. The equilibrium curves have been developed from figures calculated on a dry basis in order that samples be compared on the same basis of moisture 0.0%.

The effect of ageing on the steady state equilibrium has been studied by leaving the samples in their containers for 30 days and re-analysing them for moisture, free acidity and water soluble P_2O_5.

Study of the effect of temperature during ageing has been made by carrying out for each rock sample a series of acidulations in the winter season and another series during the summer season, the room temperature being recorded in each case.

The effect of reduction of the particle size has been studied by making the same series of summer and winter acidulations, for samples A_1 and A_2 with coarser and reduced particles respectively.

ANALYSIS OF SUPERPHOSPHATE

The free acidity determination has been carried out by titration of the first hydrogen of phosphoric acid in the water soluble solution with standard alkali, using methyl orange as indicator. Considering the water soluble and total P_2O_5, they have been determined by the application of the recently developed method by Kaesner, Kramer and Ozier (5). In the determination of water soluble P_2O_5 several procedures are mentioned in literature (1), (19), (20), (21). An important phenomenon has been met with by the author in the preparation of water soluble solution. It is a general belief that the turbidity which appears in this solution after filtration represents a hydrolytic decomposition of monocalcium phosphate. The author, however, treating superphosphate samples, containing increasing amounts of free acidity, found that the amount of turbidity increases with the increase in the free acidity of superphosphate. At the same time, the time after which the turbidity appeared, was found to be inversely proportional to the free acid content. These phenomena appeared so markedly that in case of high acid containing samples, turbidity took place spontaneously, while in the clear filtrate obtained from low acid containing samples, a slight turbidity appeared after several hours. In cases where this turbidity represents a hydrolytic decomposition it may be expected that an increase in the free acidity of superphosphate will decrease the amount of turbidity, i.e., retard the hydrolysis. As the reverse has been practiced, this led us to believe that this turbidity may represent something other than hydrolysis,

which may be a peptising action of phosphoric acid upon dicalcium phosphate.   (See Appendix - Graph No. 1.)

ACIDULATION OF SAMPLE A₁

Results of the effect of quantity of acid upon the time of thickening of the mass are found in Table 4 and are graphically represented in Graph No. 1.   The approximate time of thickening has been recorded from the beginning of mixing till the mass assumed a state which was thick enough to prevent further frothing.

TABLE 4.

Sample No.	Quantity of acid grs.	Approx. time of thickening in minutes Sample A₁ @15°C. Sample A₂ @ 14°C	
1	150	3	1.5
2	160	4.5	2.5
3	170	6	3.5
4	180	7	5
5	190	8.5	6.5

The effect of reduction of particle size upon the curve of time of thickening can be clearly seen, the curve of sample A₂ being at a lower position than that of A₁.

The results of acidulation of sample A₁ at 14-15.5°C room temperature are found in Table 5.   The effect of the quantity of acid upon the percentage moisture retained by superphosphate is illustrated in Graph No.2, which represents the series of moisture curves of sample A₁.   It can be seen that the moisture content of superphosphate increases with an increase in the quantity of acid with a change in the rate of increase at a certain point, the break point in the curve.   (See Appendix - Graph No. 2.)

Table 5.
ACIDULATION TABLE SAMPLE A₁
Room Temp. 14-15°C.

Sample No.		1	2	3	4	5
Wt.of Acid, Grs.		150	160	170	180	190
Wt.of Super."		319.3	329.7	341.2	354.2	365.0

Analysis 3 days after preparation.

Moisture	%	9.81	10.69	12.42	14.39	16.24
Free Acid P_2O_5	%	2.33	2.83	3.83	5.57	7.15
"on dry "	%	2.58	3.17	4.37	6.51	8.54
Water Sol. "	%	14.85	15.20	15.16	15.20	14.76
"on dry "	%	16.46	17.02	17.31	17.75	17.62
Total "	%	18.75	18.15	17.55	16.81	16.25
"on dry "	%	20.78	20.32	20.04	19.63	19.40
Recovery	%	79.2	83.7	86.4	90.4	90.8

Analysis after 30 days ageing.

Moisture	%	9.95	10.65	12.35	14.66	16.60
Free Acid P_2O_5	%	1.83	2.33	3.08	4.83	6.66
" on dry "	%	2.03	2.61	3.51	5.66	7.99
Water Sol. "	%	14.50	15.27	15.55	15.42	14.95
" on dry "	%	16.10	17.09	17.74	18.07	17.92
Recovery	%	77.5	84.1	88.5	92.5	92.4

The equilibrium curves (See Appendix - Graph No.3) have been developed by combination of the free acidity, water soluble and total P_2O_5 curves, all calculated on a dry basis. The free acidity line, three days after preparation, shows an increase in the free phosphoric acid content with an increase in the quantity of acid applied in a way similar to the moisture curve with the same break point. The water soluble P_2O_5 line indicates an increase in the water soluble value on a dry basis with an increase in the quantity of the acid till a certain maximum value is attained beyondwhich a further increase in the quantity of acid causes a decrease in the water soluble value. Considering the total P_2O_5 curve, it shows a straight line decrease with an increase in the quantity of acid. If it is assumed that the conversion of phosphate to water soluble P_2O_5 takes place to the extent of 100%, the water soluble P_2O_5 line should meet the total P_2O_5 line, something which never happens in practice. The combination of the three lines gives rise to three regions of existence of the phases entering in the equilibrium : the phosphoric acid region which exists below the free acidity line, the water soluble phosphate region which represents mainly mono-calcium phosphate and exists between the free acidity and the water soluble P_2O_5 lines, and the water insoluble phosphate region existing between the water soluble and the total P_2O_5 lines. This latter region includes the citrate soluble and the insoluble phosphates. In the equilibrium figure the composition of a superphosphate sample can be represented by a perpendicular line to the (X) axis.

The effect of the quantity of acid upon the percentage re-covery to water soluble P_2O_5 is illustrated in Graph 4 (See appendix).

The percentage recovery increases with the increase in the quantity of acid till a certain maximum value is attained above which a slight increase in recovery takes place with an increase in the quantity of acid.

EFFECT OF AGEING ON THE STEADY EQUILIBRIUM

The effect of ageing on the moisture curve is illustrated in Graph No. 2 (see appendix) where it is found that the changes in the moisture content are very slight with a small increase in the case of high acid containing samples. A few other cases of increase in moisture after the ageing period have been met with in cases of high acid containing samples. Considering the effect of ageing on the equilibrium curves (see appendix - Graph No.3) it can be summarised as follows:

The free acidity line, after the 30 days' ageing period, is found to assume a lower position than the original line, indicating a drop in the free acid contents of all samples.

The water soluble P_2O_5 line, after ageing, sets itself in such a position that a decrease in the water soluble value takes place in low acidity samples and an increase in the same value takes place in high acidity samples. The result is that the two lines intersect at a certain point, represent-ing no change in the water soluble P_2O_5 content on a dry basis on ageing.
The total P_2O_5 is supposed to follow the moisture content, and*
The effect of ageing on the recovery curve is illustrated in Graph No.4 (see appendix). Decrease takes place in the per-centage recovery in low acidity samples, while increase takes place in the same in high acidity samples. There is an intersecting point between the curves before and after ageing which represents no change in the percentage recovery on ageing.

EFFECT OF TEMPERATURE DURING AGEING

Acidulation of sample A_1 has been carried out in summer at a room temperature of 28 - 29°C. Results of this acidulation are found in Table 6.

* this it is assumed that no change takes place in the total P_2O_5 calculated in dry samples on ageing.

TABLE 6

ACIDULATION OF SAMPLE A₁
Room Temp. 28-29°C.

Sample No		1	2	3	4	5
Wt.of Acid	Grs.	150	160	170	180	190
Wt.of Super.	"	301.7	311.8	324.3	336.9	350.1

Analysis 3 days after preparation.

Moisture	%	4.60	5.75	7.36	9.87	11.67
Free acid P₂O₅	%	2.45	3.21	4.15	6.42	8.50
" on dry "	%	2.57	3.40	4.48	7.12	9.62
Water Sol. "	%	15.90	16.28	16.51	15.90	15.51
" on dry "	%	16.67	17.27	17.82	17.64	17.56
Total "	%	19.77	19.20	18.50	17.74	17.08
" on dry "	%	20.72	20.37	19.97	19.68	19.34
Recovery	%	80.4	84.8	89.2	89.6	90.8

Analysis after 30 days ageing.

Moisture	%	3.96	4.84	6.93	9.18	11.25
Free acid P₂O₅	%	1.13	1.89	3.02	5.10	7.17
" on dry "	%	1.18	1.99	3.24	5.61	8.08
Water Sol. "	%	15.33	15.98	16.45	16.36	15.68
" on dry "	%	15.96	16.79	17.67	18.01	17.67
Recovery	%	77.0	82.4	88.5	91.5	91.4

The marked effect of seasonal variation of room temperature and weather conditions on the moisture curves is illustrated in Graph No.2 (see appendix). The curves at 28-29°C lie considerably below those at 14-15.5°C. The effect of ageing on the summer moisture curve indicates loss in the moisture contents of both low and high acidity samples. Graph No. 5 represents the equilibrium curves of sample A₁ at 28-29°C. The shape of equilibrium curves in general is not affected by a rise in room temperature. However, we find that the effect of a rise in room temperature in case of the free acidity line appears in the ageing period. It can be seen that the free acidity curves before and after ageing are more distant than in the case of the winter curves. This indicates that the rise in room temperature accelerates the drop in the free acidity in the ageing period.

Recovery curves are found in Graph No.6 (see appendix) where it can be seen that the same shape of curve is obtained before and after ageing with the intersecting point representing no change on ageing.

EFFECT OF REDUCTION OF PARTICLE SIZE

ACIDULATION OF SAMPLE A₂

Acidulation of sample A₂ with a reduced range of particle size has been carried out at different room temperatures of winter and summer in the same way as before. Results of winter acidulation at 13-14°C room temperature are found in Table 7.

Graph No. 7 (see appendix) includes the moisture curves of sample A₂. The markedly high moisture content of samples prepared at such a relatively low room temperature can be noticed. For example, sample No.1 contains 11.42% moisture in comparison with 9.81% in the corresponding sample with the same acid content in the case of sample A₁ at a room temperature with a difference of 1°C. The effect of ageing on the winter moisture curve shows a

decrease in all the moisture contents on ageing. Considerable difficultios have been encountered in the sampling of high moisture containing samples. As mentioned in the table, only 300 grams could be sampled from sample No. 5.

Equilibrium curves are found in Graph No. 8 (see appendix) which reveal the following factors:

The same shape of free acidity curve is found 3 days after preparation with the same break point at 170 grams acid. The curve after 30 days ageing indicates the usual loss in all samples, the rate of loss decreasing with the increase in the quantity of acid.

Reduction of the particle size has been found to have no effect upon the water soluble P_2O_5 in low acidity samples. For example, sample No. 1 in Table 7 contains 16.48% water soluble P_2O_5 on a dry basis in comparison to 16.46% in the corresponding sample No.1 in Table 5. On the other hand, it is found that the maximum water-soluble value three days after preparation, in case of sample A1 is 17.75% in sample No.4 Table 5, while the corresponding sample in case of A2, Table 7, contains 18.18%. The former value had undergone an increase from 17.75 to 18.07 during the ageing period, while the latter has increased from 18.18 to 18.29%. It can be concluded that the reduction of the particle size has no effect upon the water soluble values of low acid containing samples, while, in the case of high acid containing samples, a more stable equilibrium and a higher maximum water soluble value are obtained after three days. Although a greater increase took place in the maximum water soluble value of sample A1 on ageing than in that of sample A2, the former still remains lower than the latter. The stability can be seen in the water soluble P_2O_5 curves (before and after ageing) which are closer to each other than in the case of sample A1.

The same straight line decrease in the total P_2O_5 with an increase in the quantity of acid is found in the equilibrium curves.

TABLE 7

ACIDULATION OF SAMPLE A2
Room Temp. 13-14°C.

Sample No.		1	2	3	4	5 (+)
Wt.of acid, grs.		150	160	170	180	190
Wt.of Super "		325.5	336.1	346.0	357.4	372.8
Analysis 3 days after preparation						
Moisture	%	11.42	12.60	13.65	15.22	17.56
Free acid P_2O_5	%	2.50	2.83	3.66	5.49	7.32
" on dry "	%	2.82	3.24	4.24	6.47	8.88
Water Sol. "	%	14.60	15.06	15.34	15.41	15.06
" on dry "	%	16.48	17.23	17.76	18.18	18.27
Total "	%	18.36	17.86	17.10	16.60	15.94
" on dry "	%	20.73	20.43	19.80	19.58	19.33
Recovery	%	79.5	84.3	89.7	92.8	94.5
Analysis after 30 days ageing.						
Moisture	%	10.82	11.61	12.55	14.77	16.90
Free acid P_2O_5	%	1.83	2.33	2.99	5.16	7.32
" on dry "	%	2.05	2.64	3.42	6.05	8.81
Water Sol. "	%	14.32	14.95	15.52	15.59	15.13
" on dry "	%	16.06	16.91	17.75	18.29	18.21
Recovery	%	77.5	82.8	89.6	93.4	94.2

(+) Only 300 grs. could be sampled.

Graph No. 9 (see Appendix)represents the recovery curves of sample A2 at 13-14°C. The reduction in the size of the particle has no effect upon the percentage recovery in the case of low acidity samples. For example, the recovery in sample No.1, Table 7, is 79.5%, which drops to 77.5% after ageing in comparison with 79.2% and 77.5% respectively in Table 5. On the other hand, the maximum percentage recovery in samples No. 5 is 94.5% and 94.2% before and after ageing in the case of sample A2, and 90.8% and 92.4% in the case of A1. The result is that, although the reduction of particle size increases the maximum percentage recovery before and after ageing, it has no effect upon this value in the case of low acid containing samples. At the same time a more stable recovery curve is obtained after three days, as may be seen from the closeness of curves before and after ageing.

EFFECT OF TEMPERATURE DURING AGEING

Results of summer acidulation of sample A2 are found in Table 8. In the acidulation of sample A2 at 28-29°C it has been found that a sample with suitable physical conditions could be prepared containing 200 grams acid. The range of the quantities of acid has been changed from 150-190 to 160-200 grams. The effect of a rise in the room temperature upon the moisture curve is illustrated in Graph No.7, where the curves at 28-29°C room temperature assume a considerably lower position than those at 13-14°C. The break point is not affected in the curves before or after ageing.

In the equilibrium curves, Graph No. 10 (see Appendix) the effect of a rise in the room temperature upon the decrease in free acidity after ageing is illustrated. The free acidity line after 30 days ageing assumes a lower position than the corresponding line in the winter curves. The rise in the room temperature increases the drop in the free acidity during the ageing period. No appreciable effect of the room temperature during ageing is found with regard to the other curves of the equilibrium before or after ageing.

The recovery curves of sample A2 at 28-29°C are found in Graph No.11 (see Appendix)which have the usual shape of recovery curves with the intersection point representing no change on ageing.
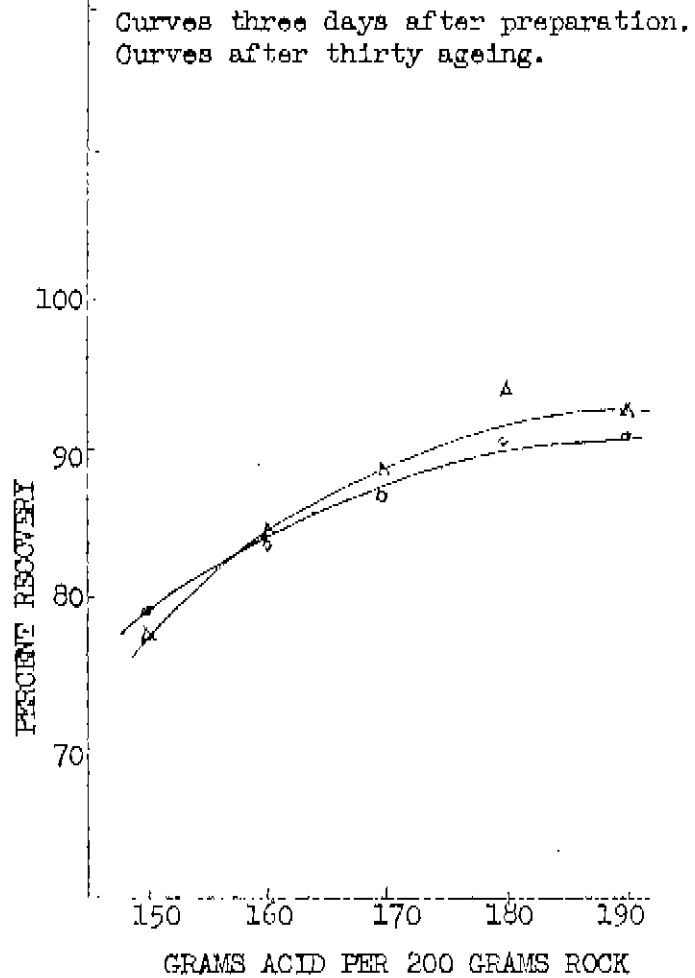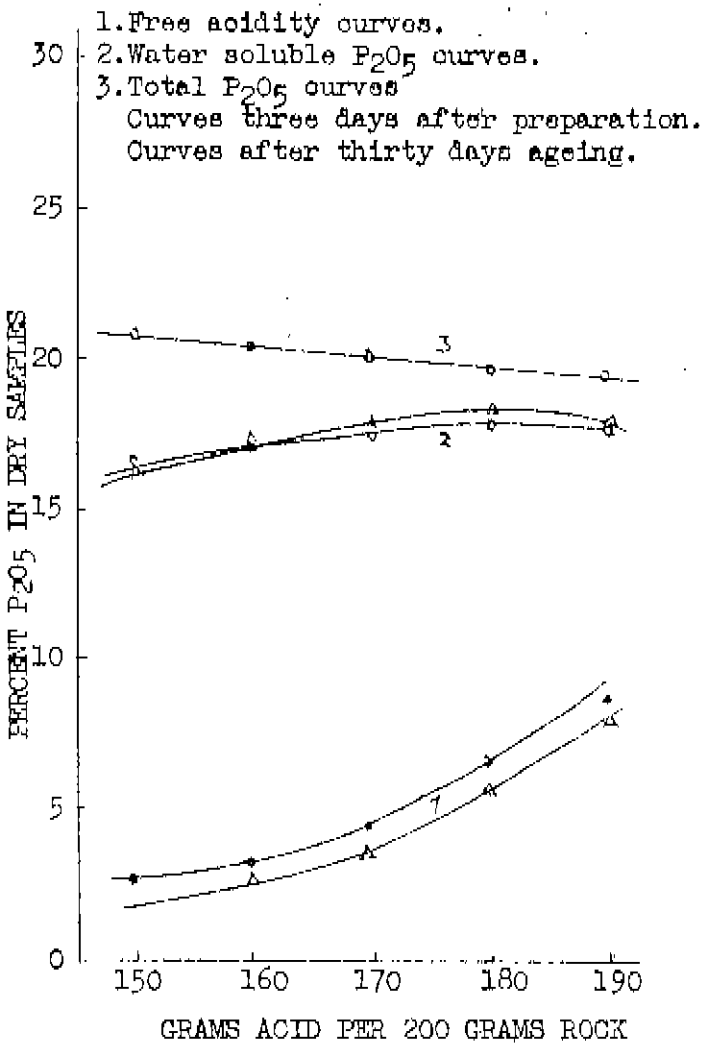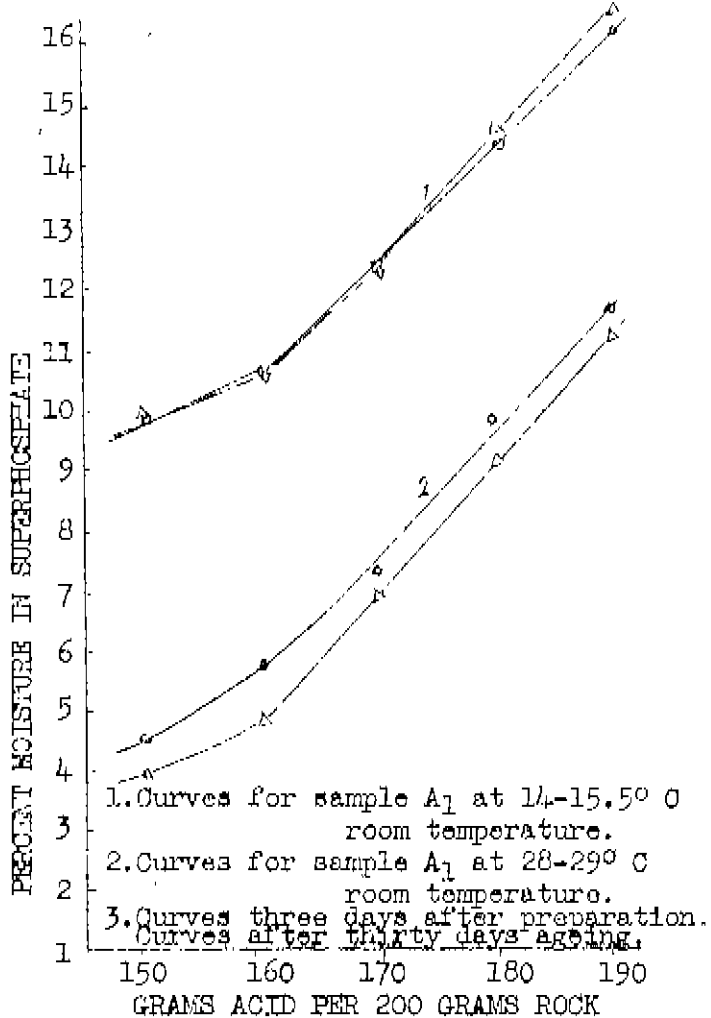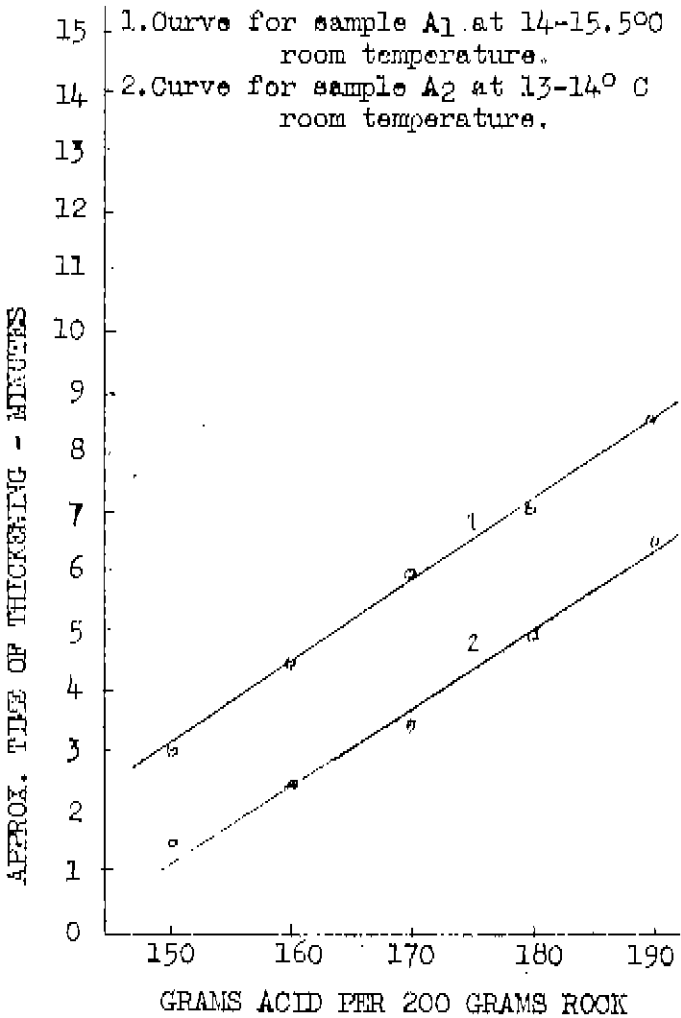
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TABLE 8
ACIDULATION OF SAMPLE A2
Room Temp. 28-29°C.

Sample No.		1	2	3	4	5
Wt.of acid, grs.		160	170	180	190	200
Wt.of Super. "		308.8	318.5	329.8	342.8	356.0
Analysis 3 days after preparation						
Moisture	%	4.5	5.71	7.65	9.69	11.65
Free acid P2O5	%	3.21	4.15	6.14	8.31	10.38
" on dry "	%	3.36	4.40	6.65	9.20	11.75
Water Sol. "	%	16.38	16.66	16.81	16.05	15.58
" on dry "	%	17.15	17.67	18.20	17.77	17.63
Total "	%	19.48	18.83	18.14	17.46	16.81
" on dry "	%	20.40	19.97	19.64	19.33	19.03
Recovery	%	84.1	88.5	92.7	91.9	92.7
Analysis after 30 days ageing						
Moisture	%	3.45	4.97	7.28	9.55	11.83
Free acid P2O5	%	1.42	2.74	4.91	7.17	9.45
" on dry "	%	1.47	2.88	5.29	7.93	10.72
Water Sol. "	%	16.51	16.90	16.98	16.33	15.72
" on dry "	%	17.10	17.78	18.31	18.05	17.83
Recovery "	%	83.8	89.0	93.2	93.4	93.7

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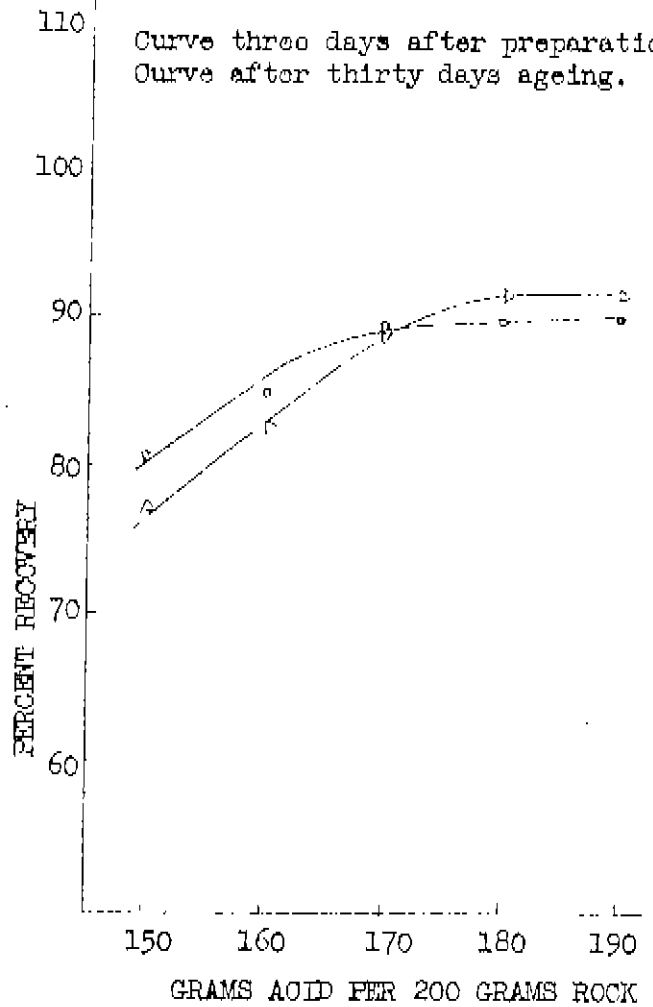21. Op.Cit.   P.224.

1

1. Curve for sample A₁ at 14-15.5°C
   room temperature.
2. Curve for sample A₂ at 13-14° C
   room temperature.

APPROX. TIME OF THICKENING - MINUTES

GRAMS ACID PER 200 GRAMS ROCK

2

PERCENT MOISTURE IN SUPERPHOSPHATE

1. Curves for sample A₁ at 14-15.5° C
   room temperature.
2. Curves for sample A₁ at 28-29° C
   room temperature.
3. Curves three days after preparation.
   Curves after thirty days ageing.

GRAMS ACID PER 200 GRAMS ROCK

3

1. Free acidity curves.
2. Water soluble P₂O₅ curves.
3. Total P₂O₅ curves
   Curves three days after preparation.
   Curves after thirty days ageing.

PERCENT P₂O₅ IN DRY SAMPLES

GRAMS ACID PER 200 GRAMS ROCK

4

Curves three days after preparation.
Curves after thirty ageing.

PERCENT RECOVERY

GRAMS ACID PER 200 GRAMS ROCK

No. 5

1. Free acidity curves.
2. Water soluble P_2O_5 curves.
3. Total P_2O_5 curves.
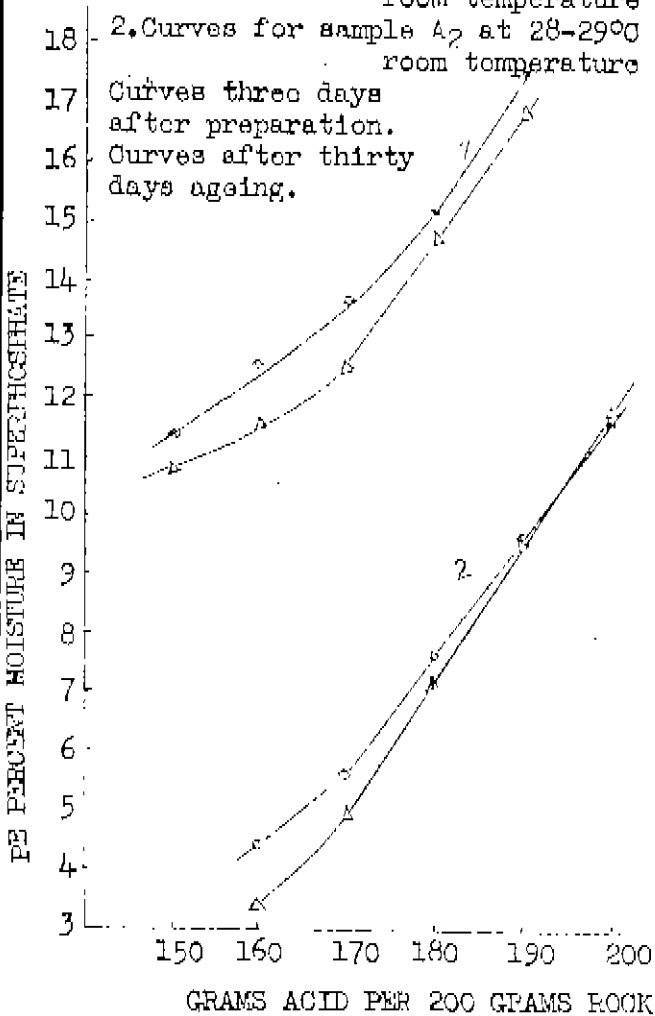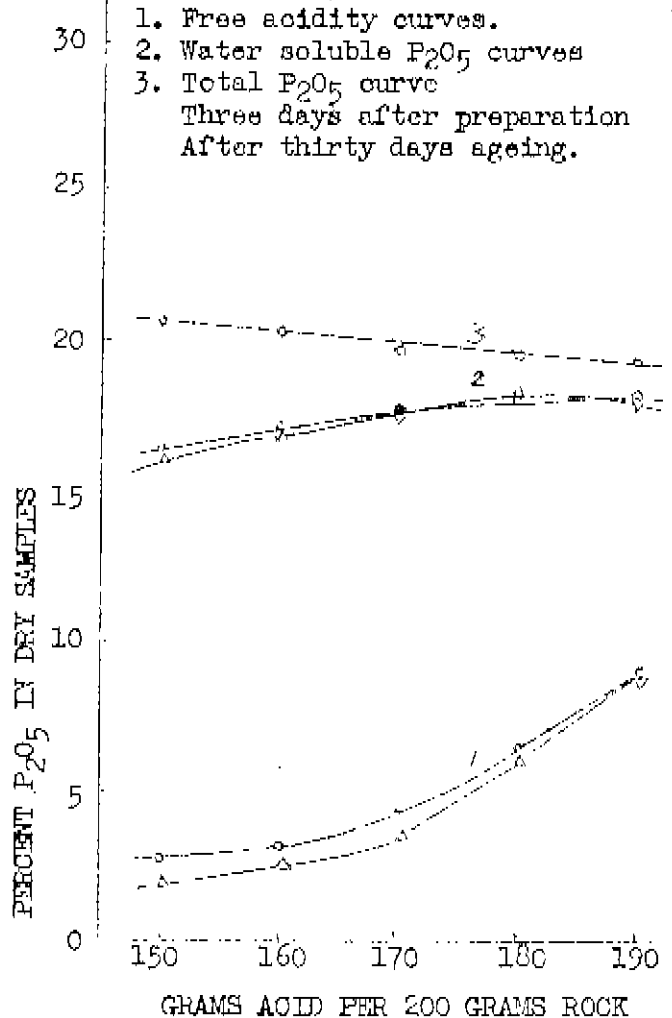   Three days after preparation
   After thirty days ageing.

PERCENT P_2O_5 IN DRY SAMPLES

GRAMS ACID PER 200 GRAMS ROCK
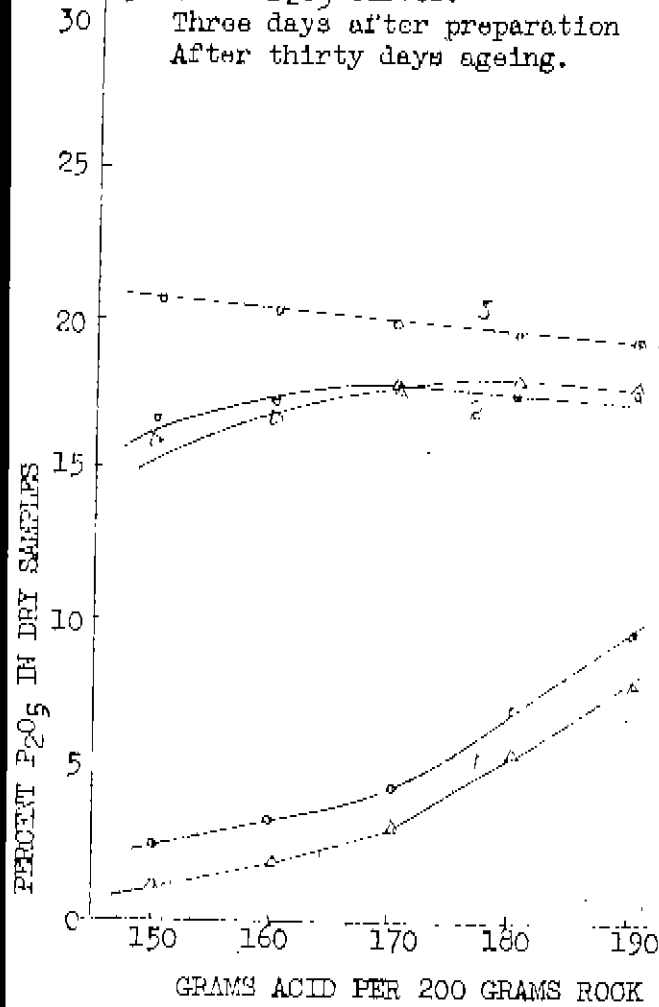
No. 6

A_1 28 - 29°C

Curve three days after preparation
Curve after thirty days ageing.

PERCENT RECOVERY

GRAMS ACID PER 200 GRAMS ROCK

No. 7

1. Curves for sample A_2 at 13-14°C
   room temperature
2. Curves for sample A_2 at 28-29°C
   room temperature

Curves three days
after preparation.
Curves after thirty
days ageing.

P3 PERCENT MOISTURE IN SUPERPHOSPHATE
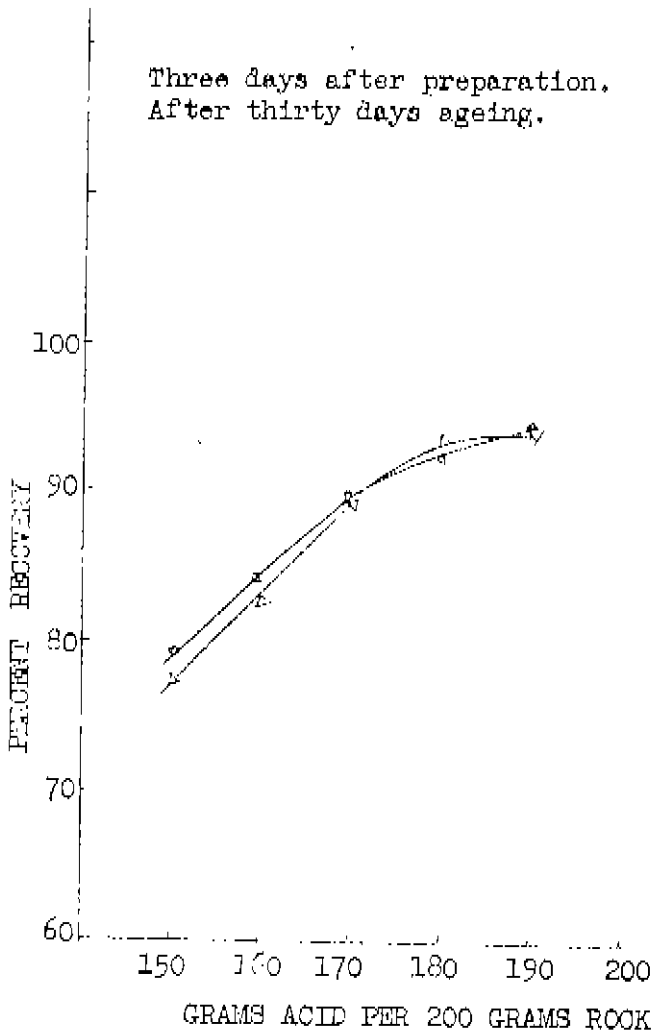
GRAMS ACID PER 200 GRAMS ROCK

No. 8

13 - 14°C

1. Free acidity curves.
2. Water soluble P_2O_5 curves
3. Total P_2O_5 curve
   Three days after preparation
   After thirty days ageing.

PERCENT P_2O_5 IN DRY SAMPLES

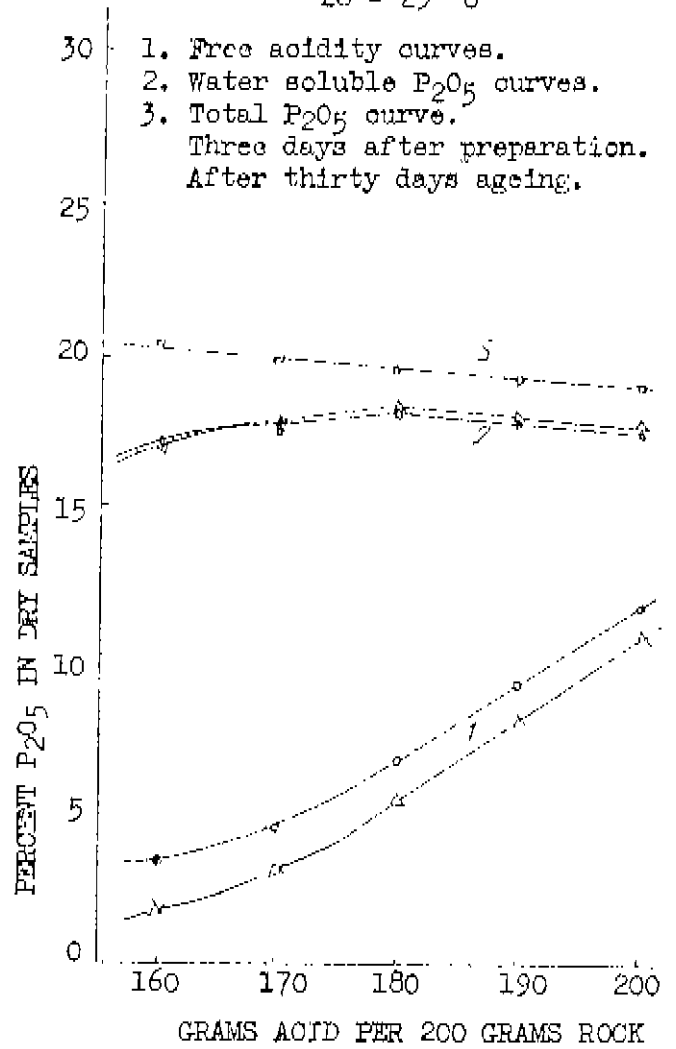GRAMS ACID PER 200 GRAMS ROCK

No. 9

A 13 - 14 °C

Three days after preparation.
After thirty days ageing.

No. 10

28 - 29 °C

1. Free acidity curves.
2. Water soluble P_2O_5 curves.
3. Total P_2O_5 curve.
   Three days after preparation.
   After thirty days ageing.

No. 11

A 28 - 29°C

Three days after preparation.
After thirty days ageing.