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STUDY OF THE REACTIVITY OF THE "WASHED METLAOUI" PHOSPHATE  
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## I. INTRODUCTION

The usual tendency for the classification of a phosphate is based on the  $P_2O_5$  content and the contents of different impurities ( $MgO$ ,  $Al_2O_3$ ,  $Fe_2O_3$  etc.).

However, we have observed that this basis for the classification of phosphate is not always unquestionable. In fact, our plant has been the seat of interferences during the conversion of the Metlaoui washed phosphate originating from the Kef Schfaier deposit (consisting of eight phosphate layers). These interferences manifest themselves by a drop in the filtration and treatment yield because of the different behaviours of the mixture of certain layers during the conversion.

Attempts at interpretation based on a comparison of the chemical composition were inconclusive because the contents of the main elements were nearly the same.

The present paper attempts to determine the origin of these interferences by means of a study consisting of:

1. chemical, mineralogical and thermal analysis of the eight layers taken separately;
2. drawing up a reactivity scale for the different layers;
3. studying the behaviour of the eight layers with regard to the sulphuric acid treatment.

This study, carried out jointly with the Compagnie des Phosphates de GAFSA (C.P.G.) and the Ecole National d'Ingénieurs de GABES (ENIG), was aimed at determining the optimum mixture of these different layers required for obtaining a better conversion and to develop a method for defining a better exploitation of the phosphate deposits, based on reactivity.

## II. ANALYTICAL STUDY

SAEPA is supplied with phosphate essentially

coming from the Kef Schfaier deposit near Metlaoui. This deposit consists of eight phosphate layers with sterile layers (clay, limestone etc.) in between.

This phosphate undergoes a first homogenization operation before being enriched by washing. SAEPA converts this washed phosphate into phosphoric acid by the wet process according to the Tunisian "SIAPE" process.

Interferences in operation have been recorded during the last few years, although the chemical composition was maintained substantially the same.

These difficulties have led to a drop in the filtration and treatment yields in our plants and to a decrease in our phosphoric acid production capacity essentially due to the increase in the recycling rates. In order to determine the origin of these interferences, we have first carried out an analytical study consisting of:

- the chemical analysis of the eight layers taken separately,
- the mineralogical analysis of the eight layers taken separately and
- the thermal analysis of the eight layers taken separately.

1. The results of the chemical analyses of enriched phosphate from the eight layers of the Kef Schfaier deposit are given in the table below:

Layer No.		A	B	C	D	E	F	G	H
Component									
P <sub>2</sub> O <sub>5</sub>	(%)	28.55	30.20	27.52	28.29	28.48	30.22	28.49	30.12
CaO	(%)	48.22	49.79	46.63	47.39	47.11	49.80	47.62	50.36
SO <sub>3</sub>	(%)	4.32	4.34	5.96	5.66	4.01	4.48	5.61	5.12
MgO	(%)	0.44	0.47	0.65	0.54	0.54	0.48	0.59	0.38
Al <sub>2</sub> O <sub>3</sub>	(%)	0.58	0.38	1.01	0.83	0.62	0.48	0.65	0.48
Fe <sub>2</sub> O <sub>3</sub>	(%)	0.23	0.19	0.48	0.35	0.34	0.20	0.27	0.10
F	(%)	3.33	3.39	2.98	3.08	3.62	3.49	3.03	3.59
C	(%)	0.34	0.42	0.36	0.37	0.33	0.27	0.37	0.45
SiO <sub>2</sub>	(%)	3.12	3.34	6.25	4.35	3.38	3.66	5.98	3.94
CO <sub>2</sub>	(%)	6.43	6.50	5.50	5.91	6.38	6.35	5.94	6.69

In order to demonstrate the variation in the chemical composition of the different layers, the mean chemical compositions of Metlaoui washed phosphate for the years: 1981, 1982, 1983, 1984 and 1985 are taken as reference.

Component :	P <sub>2</sub> O <sub>5</sub>	CaO	SO <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	F	SiO <sub>2</sub>
Year	%	%	%	%	%	%	%	%
1981	28.88	48.59	4.07	0.53	0.49	0.25	3.46	2.92
1982	29.03	48.52	4.34	0.54	0.53	0.29	3.57	2.86
1983	28.57	48.40	4.51	0.51	0.48	0.32	3.53	3.38
1984	28.53	48.33	4.44	0.52	0.62	0.31	3.29	3.69
1985	28.76	48.88	4.56	0.47	0.50	0.28	3.23	3.34
Mean	28.75	48.54	4.38	0.51	0.52	0.29	3.41	3.24

It is thus noticed that:

- the P<sub>2</sub>O<sub>5</sub> contents of the different layers (excepting layer "c", which has a content of 27.52), are identical to (if not better than) those for the reference (28.75);
- the MgO contents are practically the same as those of the reference;
- the Al<sub>2</sub>O<sub>3</sub> contents of layers C and D are relatively high, this could be due to a high clay content. In fact, it is noticed that the SiO<sub>2</sub> and Fe<sub>2</sub>O<sub>3</sub> contents are relatively high for these two layers.

## 2. Mineralogical analysis:

The X-ray diffraction diagrams for the different samples of the eight phosphate layers show that all the solids have a hexagonal apatite structure. The mean values of the parameters of the apatite crystal lattice are:  $a = 9.326$  (Å) and  $c = 6.898$  (Å). The differences between the interlattice distances of this apatite phase are too insignificant for the crystal parameters  $a$  and  $c$  of the lattice to appear sufficiently different.

However, it should be pointed out that the parameters found for the eight layers are well within the range of variation of the crystal parameters of a series of samples of synthetic fluoroapatite with carbonate

containing variable levels of carbonate ion (reference).

Moreover, some additional lines corresponding to one or more non-apatite crystalline phases are revealed essentially in the diagrams for layers C, D and G.

### 3. Thermal analysis:

The thermogravimetric analysis curves for the same samples show three weight losses occurring at different temperature regions. However, it should be noted that the differences observed between the different layers are not significant to be able to distinguish them or to classify them.

## III. STUDY OF REACTIVITY

### A. Introduction:

The present study attempts to draw up a scale of reactivity for the different layers.

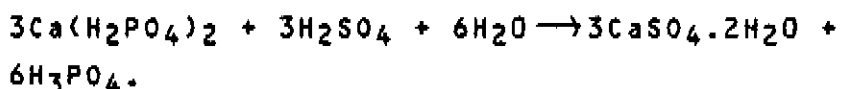
The sulphuric acid treatment of the phosphate is carried out in two stages:

1. Dissolution of the ore in phosphoric acid



i.e. the conversion of tricalcium phosphate into monocalcium phosphate.

2. Treatment of the monocalcium phosphate with sulphuric acid.



The first stage is known as the slow phase of the overall reaction. Usually, the reactivity of a phosphate is defined as the rate of dissolution of the ore in the phosphoric acid: (the rate at which the maximum solubility of tricalcium phosphate in phosphoric acid is obtained).

It should be noted that this characteristic of reactivity may often be sought in the parameters involved in the equation for the treatment kinetics: rate constant (k) and activation energy (Ea).

The use of the reactivity can, in practice, be deduced from the change in the dissolution rate with time. This rate can be monitored by three methods:

- a. variation in the mass introduced (determination of the solubilization rate);
  - b. variation in the  $P_2O_5$  content of the filtrate (changes in %  $P_2O_5$  with time);
  - c. variation in the CaO content of the filtrate (changes in % CaO with time).
- B. Choice of the method to use for the determination of reactivity:

Two constraints must be taken into consideration during the study of reactivity:

performance of the trials in a constant concentration medium;

performance of the solubilization trials under conditions which ensure that the reaction medium does not reach supersaturation.

1. Method for monitoring the reactivity by weighing the insoluble mass

This is not significant because, by increasing the (sterile) insolubles, the conversion rate which does not actually represent reactivity (in this case) is decreased.

2. Method for monitoring the reactivity by the variation in the  $P_2O_5$  content of the filtrate

The quantity of  $P_2O_5$  contributed by the ore in the filtrate is very low (the mass of phosphate introduced is low). The analysis of  $P_2O_5$  becomes inaccurate because the variation in this content is at present lower than the error in the method of analysis.

3. Method for monitoring the reactivity by the variation in the CaO content of the filtrate

The variation in the CaO content of the filtrate is significant so that it can be determined more accurately.

Thus, we have chosen the CaO method for studying reactivity. Graph No. 1 illustrates the results of the three methods tried.

- C. Operating conditions:

From studies carried out on the parameters: acid concentration (C), S/L ratio and temperature (T), the following conclusions were drawn:

a. Acid concentration (graph No. 2):

As seen in graph No. 2, the aim is to study the reactivity of the different phosphates (with reactivities close to one another). In order to distinguish it clearly, it is essential to operate with a low  $P_2O_5$  content in the reaction medium.

In order to operate under conditions which enable the dissolution before saturation to be studied satisfactorily, the solubilization phosphoric acid concentration was fixed at 5%  $P_2O_5$ .

b. S/L ratio (graph No. 3):

The results presented on graph No. 3 show that it is preferable to adopt an  $S/L = 2/100$ .

c. Temperature (graph No. 4):

For convenience and for following the changes in the dissolution with time more satisfactorily, the trials were carried out at the ambient temperature.

Thus, for trials on the study of the reactivity of different phosphates, the following operating conditions are adopted:

$P_2O_5$  content of phosphoric acid: 5%

S/L ratio = 2/100

ambient temperature

identical stirring for all trials

D. Scale of reactivity for the eight layers:

Considering the impossibility of having industrially treated samples layer by layer, a laboratory treatment which gives results close to industrial conditions was used.

1. Study on the effect of the reaction medium (acid)

With a view to investigating the effect of the reaction medium on reactivity, the reactivity of layer A in three different acids was studied:

In the acid obtained from the sulphuric acid treatment of the phosphate from this same layer.

In the acid from the phosphoric acid plant (obtained industrially from a mixture of the eight layers of Kef Schfaier).

In pure acid (orthophosphoric).

The results for the reactivity of layer A in the different acids are presented on graph No. 5. This graph shows that:

The reactivity of this layer has not changed very much with the acid from the plant and with its own acid. This low variation may be due to the CaO content of the acid.

The presence of impurities in the acid has a large influence on the reactivity (compared with pure acid).

## 2. Drawing up the scale of reactivity for the eight layers

The reactivity of the different layers of Kef Schfaier in the production acid was then studied.

This study was carried out in a discontinuous fashion with the operating conditions mentioned above.

The curves giving the degrees of progress of the dissolution reaction of all the phosphates are presented as a function of time in graph No. 6.

From these curves, the two variables characteristic of each phosphate can be determined:

Solubilization maximum: reactivity index ( $\alpha_{max}$ ).

Time ( $t^*$ ) required for reaching this maximum.

Layer	A	B	C	D	E	F	G	H
max (%)	76	75.2	82.5	84.3	87	77	78	83
$t^*$ (mn)	14	15	26	25	14	14	24	16

It can be seen that there are two types of layers which behave differently:

- Layers No. E, H, F, A and B which reach the solubilization maximum in a mean time  $t^* = 15$  min.
- Layers No. C, D and G: for which the solubilization maximum is reached after  $t^* = 25$  min.

Layers of each type have reactivities (dissolution

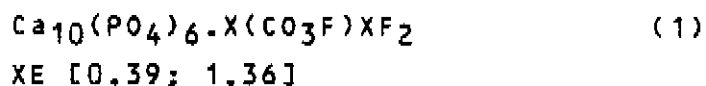


rate) which are close to one another. They are higher for the first type. The latter is therefore considered as the more reactive type.

### 3. Interpretation of results

The most commonly occurring apatite in Tunisia is of sedimentary origin,  $\text{Ca}_5(\text{PO}_4)_3(\text{F}, \text{Cl})$ . This formula is only theoretical; many substitutions may occur, modifying this structure.

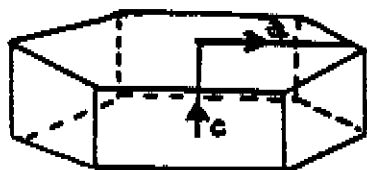
The most noted substitution in sedimentary apatites is that of  $\text{PO}_4^{3-}$  with  $\text{CO}_3^{2-}$ . This enables a more general formula to be defined, under the name carbonate-fluorapatite or frankolite:



Several investigations have led to the definition of the basic unit of the fluorapatite lattice which is a prism of rather complicated structure (2).

The structure tends to become more fragile when the substitution of  $\text{PO}_4^{3-}$  with  $\text{CO}_3^{2-}$  is accentuated. Thus, the reactivity of the corresponding phosphate tends to increase (3).

This fragility may be explained in simple terms by the expansion or stretching of two geometrical parameters ( $a$ ,  $c$ ) of the basic unit during any substitution: the following figure shows the basic unit of pure fluorapatite (3):



for pure fluorapatite  $a = 9.39$   
 $c = 6.88$

When the  $\text{CO}_3^{2-}$  ions take the place of  $\text{PO}_4^{3-}$  ions, two events take place:

1. An imbalance in the electrical neutrality of the crystal followed by the introduction of additional  $\text{F}^-$  ions in order to establish the balance.
2. A shift in the dimensions ( $a$ ,  $c$ ) which leads to deformations of the basic unit (source of fragility of the crystal) caused by the difference in size between the  $\text{PO}_4^{3-}$  and  $\text{CO}_3^{2-}$  ions.

Thus, the reactivity happens to be strongly linked to the degree of substitution of  $\text{PO}_4^{3-}$  ions by  $\text{CO}_3^{=}$  ions, and as a result, to the  $\text{CO}_2$  content (endogangue) of the ore.

From the results of the radiocrystallographic and thermogravimetric analyses, it is not possible to define a reactivity scale based on the crystal parameters and the degrees of substitution possible (because the differences between the eight layers are not significant). In fact, the eight layers constitute the same deposit and, on average, they have the same grade of fluorapatite with slight differences.

On the other hand, from the chemical analyses, it is possible to draw up the following table:

Layer	A	B	C	D	E	F	G	H
$\text{P}_2\text{O}_5$	28.55	30.20	27.52	28.29	28.48	30.22	28.49	30.12
F	3.33	3.39	2.98	3.08	3.62	3.49	3.03	3.59
$\text{CO}_3$	8.18	8.27	7.00	7.52	8.12	8.08	7.56	8.51
F/ $\text{P}_2\text{O}_5$ %	11.66	11.22	10.83	10.89	12.71	11.55	10.63	11.92
$\text{CO}_3 / \text{P}_2\text{O}_5$ %	28.65	27.40	25.43	26.58	28.51	28.36	25.01	28.35
RI	5.43	5.01	3.16	3.37	6.21	5.5	3.25	5.19

It is observed in this table that the F/ $\text{P}_2\text{O}_5$  and  $\text{CO}_3/\text{P}_2\text{O}_5$  ratios confirm the presence of two types of phosphate in the Kef Schfaier deposit:

1st type consisting of layers A, B, E, F and H which have

$$\text{F}/\text{P}_2\text{O}_5 > 11$$

$$\text{CO}_3/\text{P}_2\text{O}_5 > 27$$

2nd type consisting of layers C, D and G which have

$$\text{F}/\text{P}_2\text{O}_5 < 11$$

$$\text{CO}_3/\text{P}_2\text{O}_5 < 27$$

It is also observed that the reactivity index varies in the same direction as the F/ $\text{P}_2\text{O}_5$  and  $\text{CO}_3/\text{P}_2\text{O}_5$

ratios (graphs No. 7 and 8).

Thus, it is noted that the reactivity depends on the following two parameters:

$F/P_2O_5$  which could be an index of substitution of  $PO_4^{3-}$  by the  $F^-$  ion

$CO_3/P_2O_5$  which could be directly related to the degree of  $CO_3=$  substituting  $PO_4^{3-}$ .

#### VI. IMPACT OF REACTIVITY ON SULPHURIC ACID TREATMENT

A series of trials on the conversion was carried out under the usual conditions of sulphuric acid treatment, with the eight phosphate layers, in an identical manner. These trials led to the following results:

Layer No.	A	B	C	D	E	F	G	H
g: gypsum factor	4.70	4.64	4.91	4.87	4.86	4.85	5.18	4.98
$P_2O_5$ g/kg U	2.32	2.68	11.17	15.2	2.85	1.82	11.47	1.34
$P_2O_5$ g/kg C+U	16.15	14.32	19.57	19.8	12.5	14.1	20.05	14.1
Treatment yield Ty (%)	92.40	93.36	90.39	90.36	93.94	93.16	89.61	92.98
$H_2SO_4$ (g/l)	- 19	- 29	+ 17	+ 10	- 28	- 27	+ 17	- 18
$P_2O_5$ (g/l)	315	350	280	282	342	3.04	309	328

It is observed in this table that these results confirm once again the presence of the two types of phosphate in the test Kef Schfaier deposit: in effect,

The layers A, B, E, F and H behave in the same way during the sulphuric acid treatment.

The layers C, D and G have the same behaviour during the treatment (excess of  $H_2SO_4 \rightleftharpoons$  treatment stoppage).

#### V. CONCLUSION

From the study carried out on the eight phosphate layers of the Kef Schfaier deposit, the following can be ascertained:

The deposit is made up of 2 types, of which:

a first consists of the layers A, B, E, F and H which have a dissolution rate (reactivity index) of 5 to 6%/min and

a second which consists of the layers C, D and G which have a dissolution rate of 3.2 to 3.4%/min.

This result was confirmed using the results of chemical analyses. In effect, the calculation of the  $F/P_2O_5$  and  $CO_3/P_2O_5$  ratios (which could be the indices of the " $F^- + CO_3$ " group substituting  $PO_4^{3-}$ ) reveals the following:

The layers A, B, E, F and H have a  $F/P_2O_5$  ratio  $> 11$  and a  $CO_3/P_2O_5$  ratio  $> 27$ .

The layers C, D and G have an  $F/P_2O_5$  ratio  $< 11$  and a  $CO_3/P_2O_5$  ratio  $< 27$ .

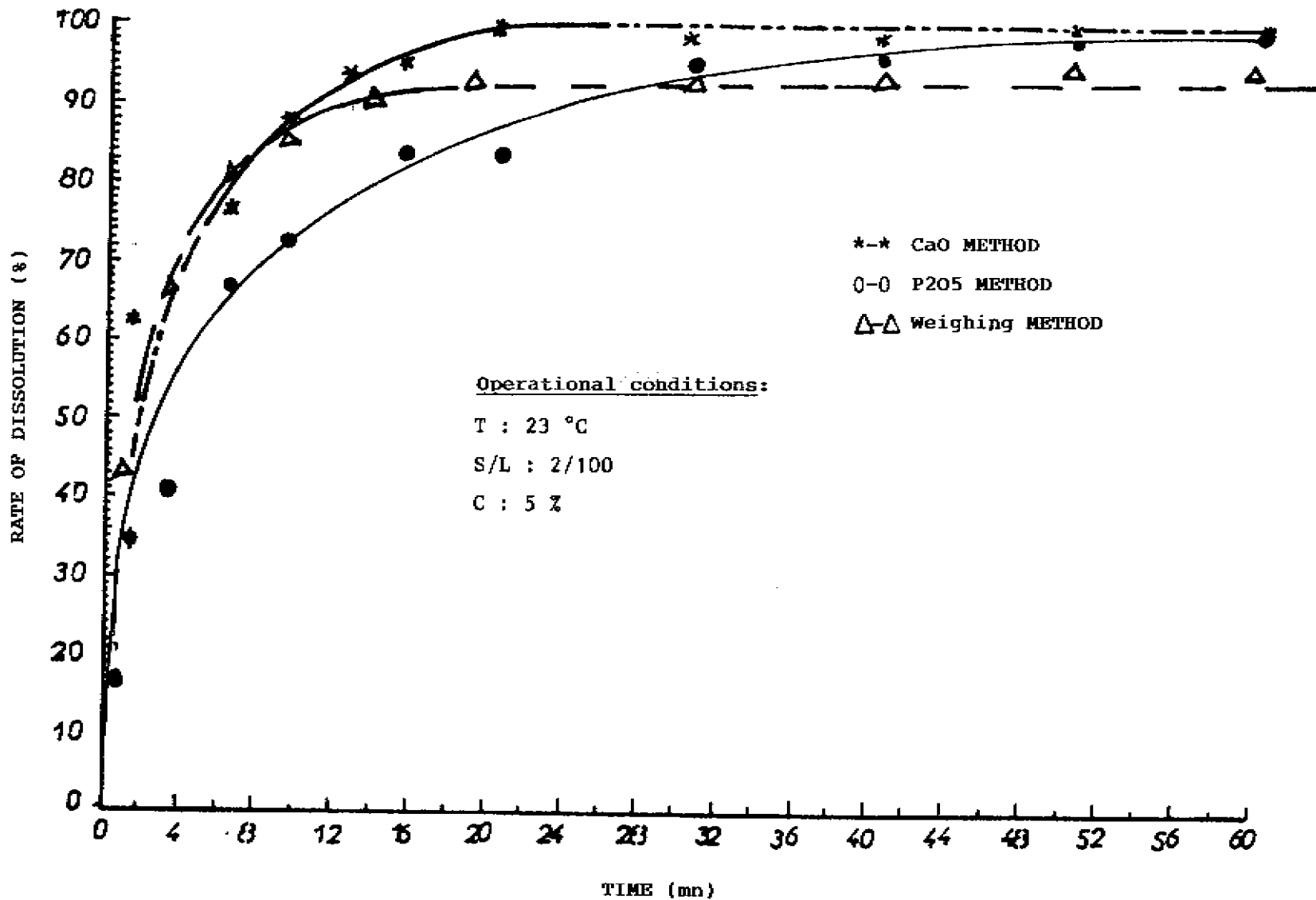
It should also be noted that mineralogical and thermal analyses were carried out on the eight layers, but it would appear, at the current stage of the study, that the results obtained from the reactivity tests cannot be confirmed by the radiocrystallography and the thermogravimetric study.

#### REFERENCES

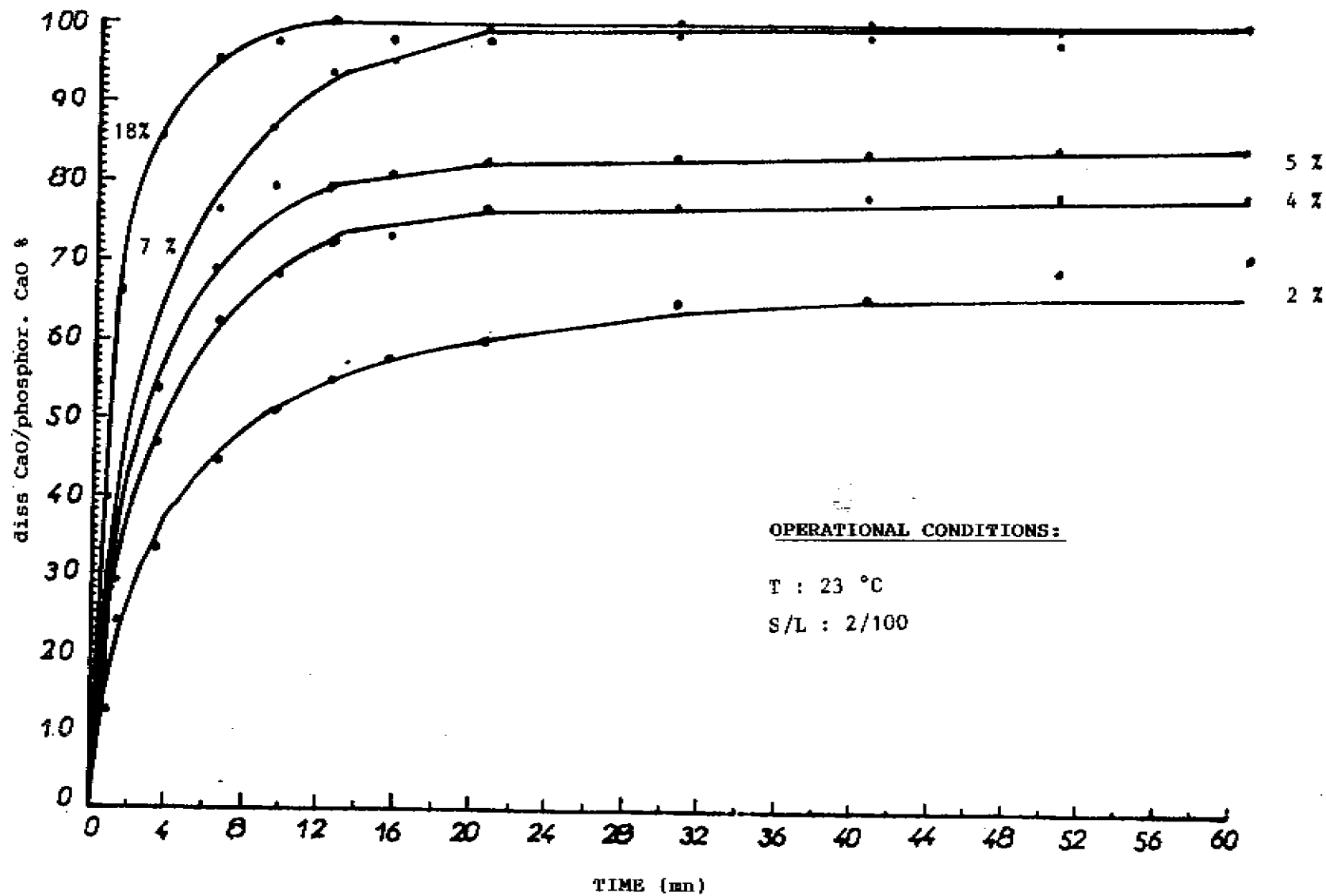
- (1) R. Kremf & A. Savouret, Cerphos No. 3448 of 18/6/1976, Study of the reactivity of phosphates.
- (2) Petro Parlovsky & Mrchedlichvili, Précis de chimie industrielle, 2nd part, Industries d'engrais minéraux, ENIG (GABES) 1982.
- (3) Phosphorus and Potassium No. 98, November/December 1978.

## SYMBOLS

T	: temperature (in °C)	
S/L	: solid:liquid mass ratio	$\frac{\text{mass of phosphate}}{\text{mass of the corresponding acid}}$
C	: P <sub>2</sub> O <sub>5</sub> concentration of phosphoric acid	
t	: residence time (in min)	
α	: phosphate solubilization rate (in %)	
RI	: reactivity index (phosphate solubilization rate per unit time)	
t*	: time required to reach maximum solubilization	
(P <sub>2</sub> O <sub>5</sub> ) <sub>U</sub>	: untreated P <sub>2</sub> O <sub>5</sub> present in the gypsum, having the untreated phosphate (g/kg of dry gypsum)	
(P <sub>2</sub> O <sub>5</sub> ) <sub>C</sub>	: cocrystallized P <sub>2</sub> O <sub>5</sub> present in the gypsum (g/kg of dry gypsum)	
g	: gypsum factor: (kg of dry gypsum/kg of P <sub>2</sub> O <sub>5</sub> )	
Ty	: treatment yield	

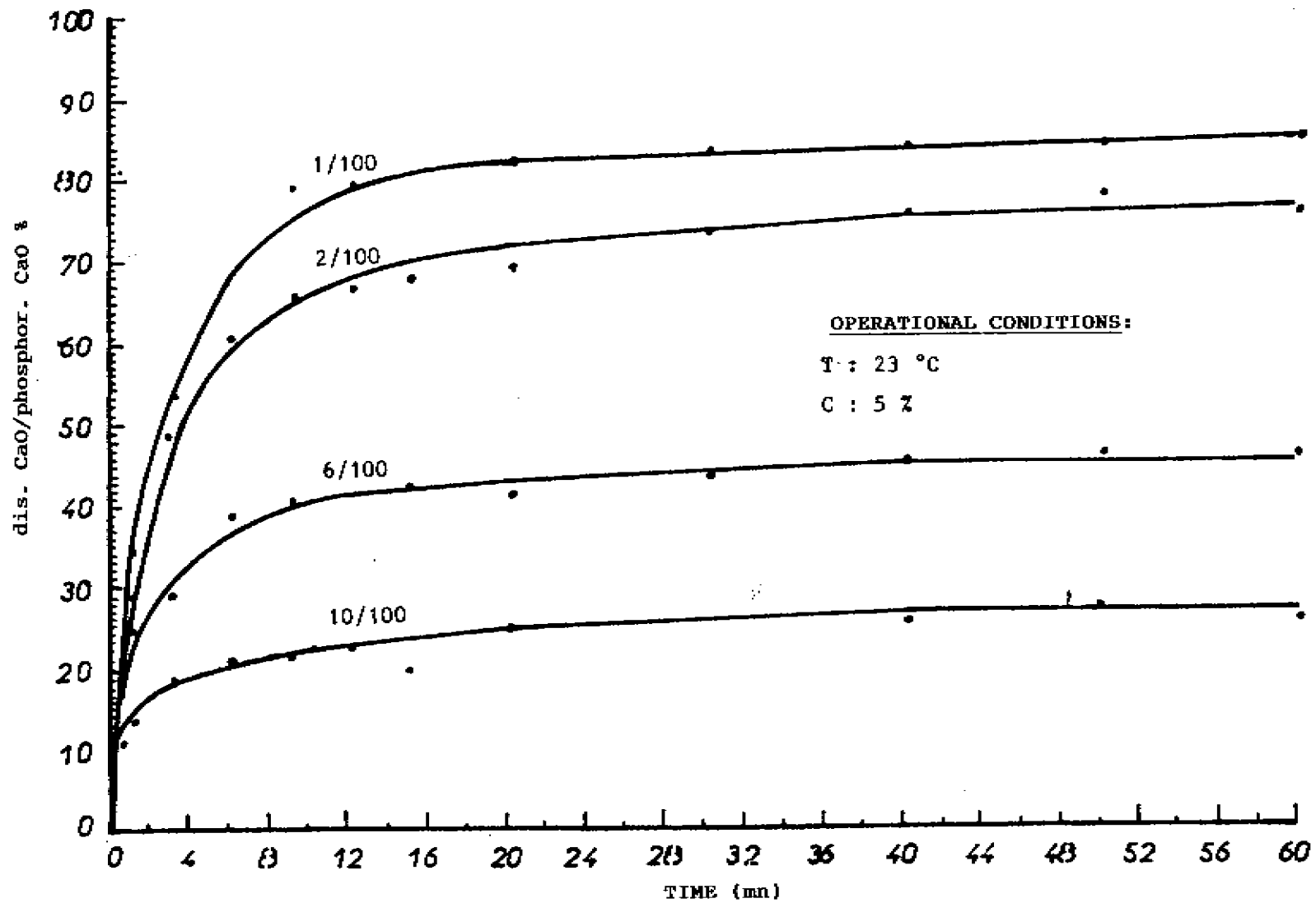


DIFFERENT METHODS TO EVALUATE REACTIVITY  
 FIGURE 1



VARIATION IN PHOSPHORIC ACID CONCENTRATION

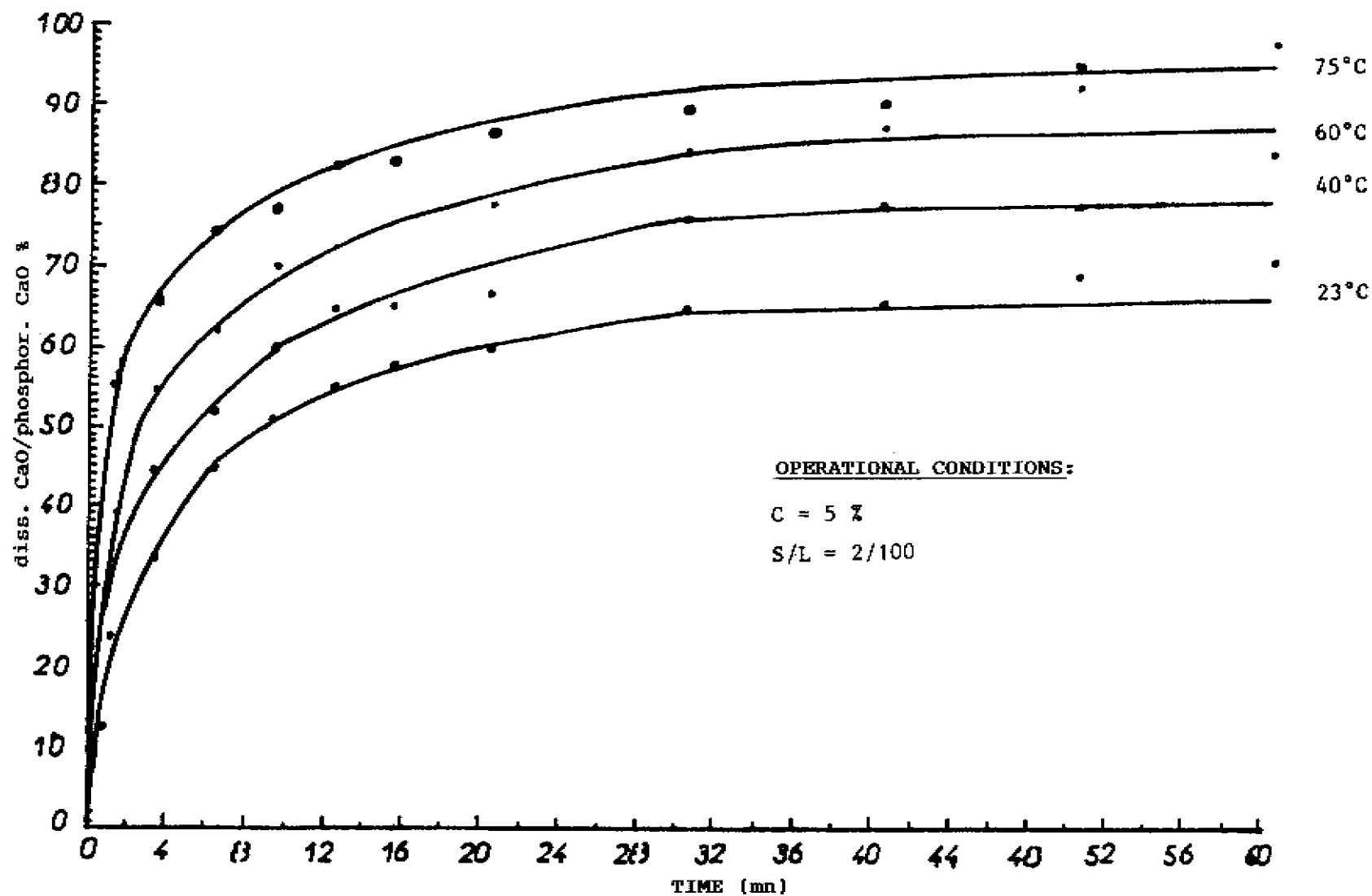
FIGURE 2



VARIATION OF THE S/L RATIO

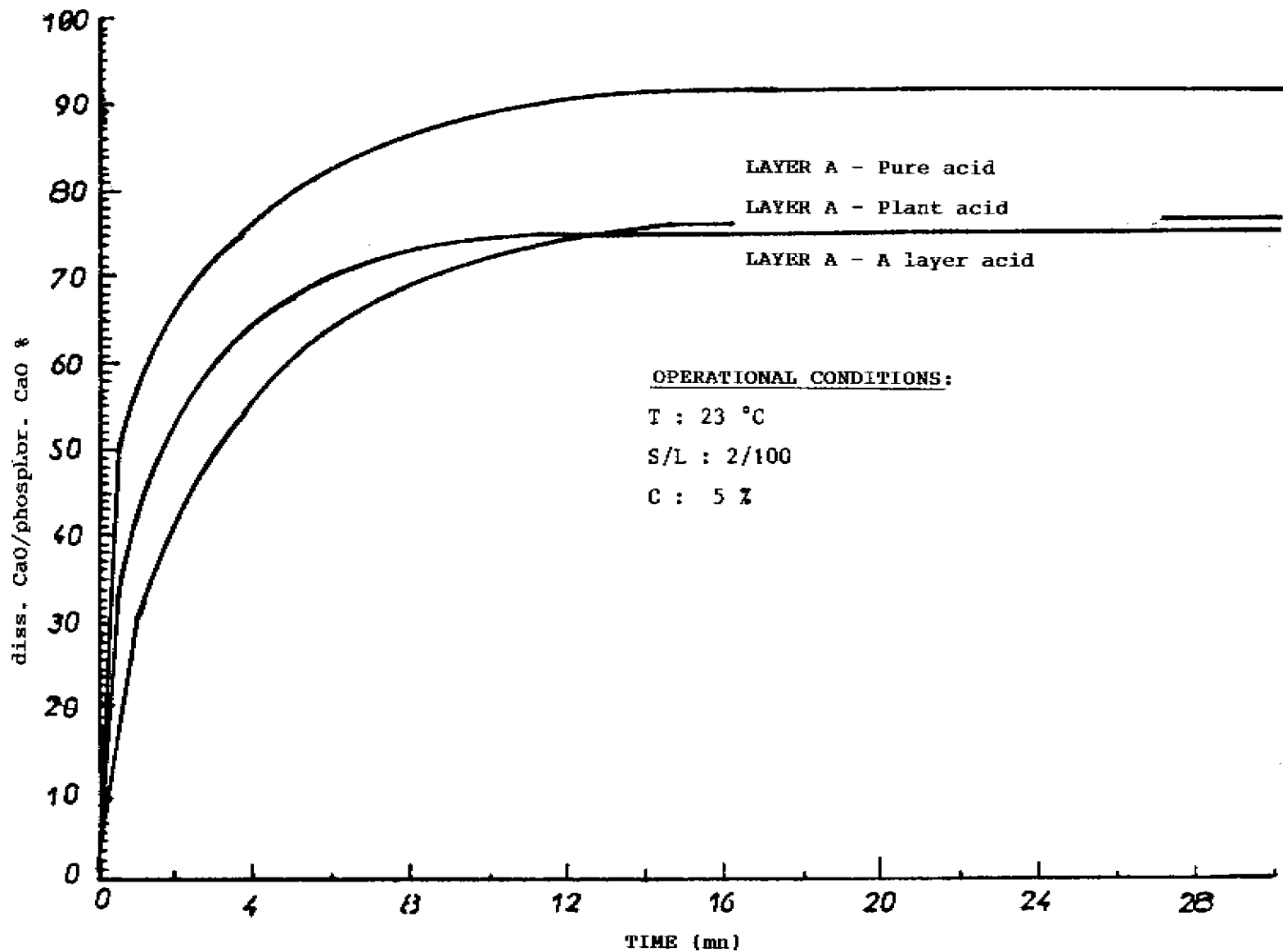
FIGURE 3





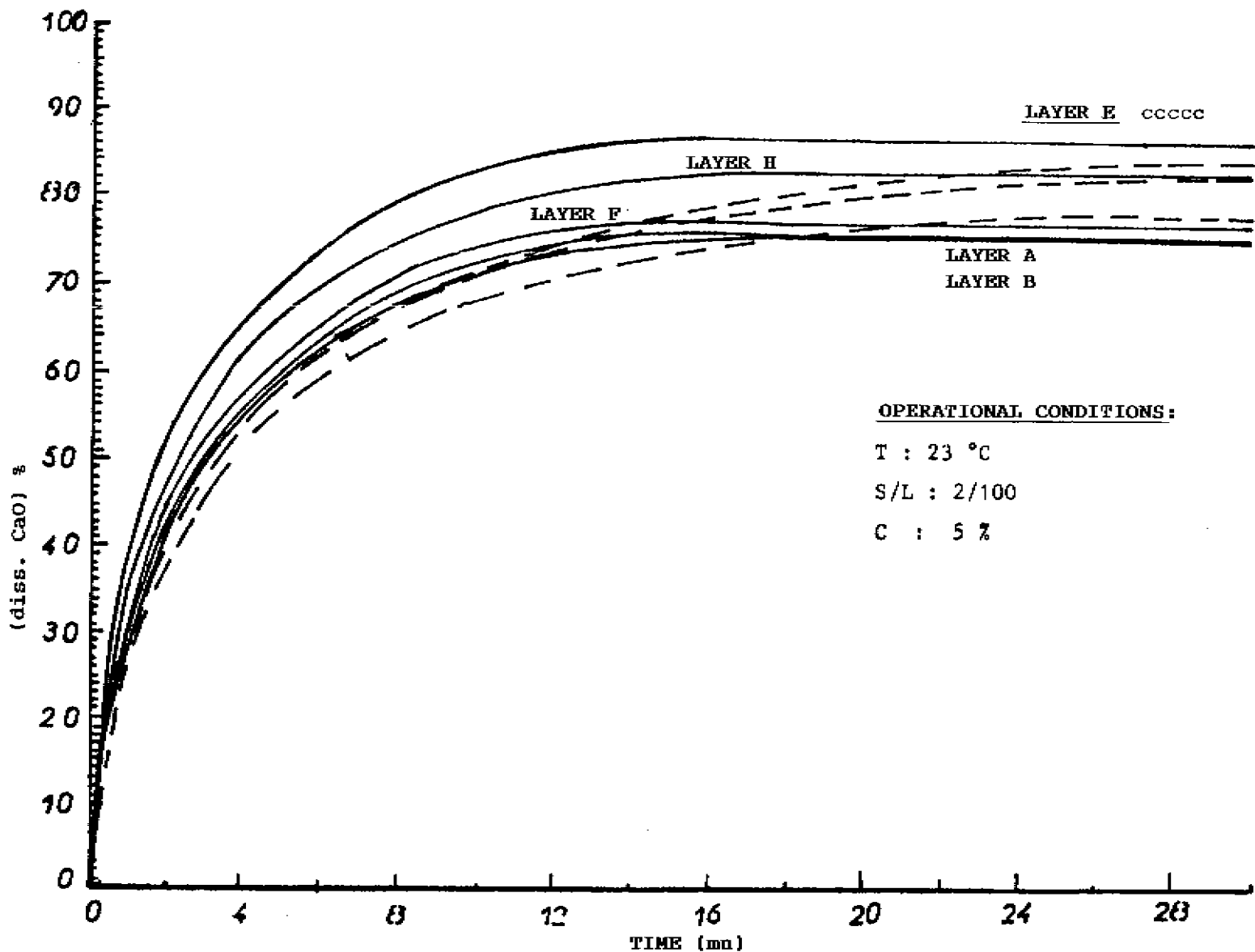
VARIATION OF THE TEMPERATURE

FIGURE 4



INFLUENCE OF THE REACTION MEDIUM

FIGURE 5



REACTIVITY OF THE VARIOUS PHOSPHATE LAYERS

FIGURE 6



TA/86/1 Study of the reactivity of the washed Metlaoui phosphate by  
A. Charfy & M.L. Rahoui, ICM, Tunisia

DISCUSSION : (Rapporteurs Messrs M. Barloy, SCPA, France & M. HAMDI,  
SAEPA, Tunisia)

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

a) The objective was to investigate the reactivity of rock for phosphoric acid. The use of 5% P<sub>2</sub>O<sub>5</sub> medium seems unreal. Rock dissolution is sensitive to SO<sub>4</sub> concentration (inhibition). What work has been done on testing reactivity at different SO<sub>4</sub> level?

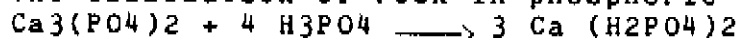
b) Referring to table on page 9 the choice of F/P<sub>2</sub>O<sub>5</sub> (=11) and CO<sub>3</sub>/P<sub>2</sub>O<sub>5</sub> (=27) is based on different results from earlier stage which showed C - D and G layers differing from other layers. What scientific and crystallographic explanation is there for the numbers 11 and 27?

A - a) Although, in practice, phosphate rock is dissolved in a medium containing about 28% P<sub>2</sub>O<sub>5</sub>, the reactivity test was carried out in a phosphoric acid solution with 5% P<sub>2</sub>O<sub>5</sub>. This choice was decided as a result of the survey on the variation of dissolution with time when the concentration of the reaction medium is modified. Indeed, it was found that the dissolution of phosphate rock in a 28% P<sub>2</sub>O<sub>5</sub> phosphoric acid solution was immediate so that the progress of phosphate dissolution in a concentrated medium could not be followed.

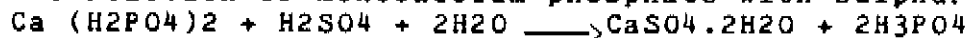
The objective of this investigation was to work out a reactivity scale of the various layers (that is a classification of the layers). We feel that the dissolution in a 5% P<sub>2</sub>O<sub>5</sub> solution can produce that reactivity scale; the results were confirmed later by reacting these layers with sulphuric acid in the laboratory under industrial conditions.

Phosphate is solubilized in two stages:

1. the dissolution of rock in phosphoric acid



2. the reaction of monocalcium phosphate with sulphuric acid



The kinetics of the overall reaction is determined by the kinetics of the slower reaction.

In that case the first phase is the slowest. In addition the presence of SO<sub>4</sub><sup>--</sup> in the reaction medium could induce the two following facts:

- Increased speed and rate of dissolution as a result of the reaction of SO<sub>4</sub><sup>--</sup> ions with monocalcium phosphate releasing H<sub>3</sub>PO<sub>4</sub> (difficult to follow).

- Slow down of the dissolution owing to the coating of phosphate particles with the gypsum produced (reaction stopped).

For these reasons the investigation was limited to phosphate dissolution in a phosphoric acid medium containing no free  $SO_4^{--}$ .

- b) The classification of layers was based on the reactivity index:  $Ir = \frac{\alpha_{max.}}{t^*}$

In fact the ABEFH layers have a reactivity index between 5 and 6.2 and the C, D and G layers an index between 3.1 and 3.4. The difference between these two categories of layers is quite striking.

The F/P205 and CO3/P205 ratios show that:

- Layers ABEFH:  

F/P205 > 11	CO3/P205 > 27
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- Layers CDG:  

F/P205 < 11	CO3/P205 < 27
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The values 11 and 27 were not chosen in advance, but they were found for the layers under study.

Q - Mr. A. EL-SAYED, CMRDI, Egypt

Are the ratios of CO3/P205 and F/P205 considered a measure of reactivity for all sedimentary phosphate rocks in the world?

A - The study on the eight layers showed that the reactivity depends on the CO3/P205 and F/P205 ratios, which could be explained by the substitution of  $PO_4(3-)$  ions by the  $CO_3^{--}$  and F- ions. These results were confirmed by the study of two other mines in the Gafsa basin.

The variation of these ratios confirms the classification established on the basis of the reactivity index  $Ir = \frac{\alpha_{max.}}{t^*}$

(for the phosphates under study). However, it is impossible to state categorically that these results are applicable to all phosphate ores in the world.

Mr. P. BECKER, COFAZ, France added that the results of this study are valid for all phosphates except those with much calcium carbonate in the outside matrix. However, the F/P205 ratio follows fairly regularly the phosphate reactivity.

Q - Mr. P. BECKER, COFAZ, France

On which granule size basis were the reactivity tests carried out?

- a - Limited fraction
- b - Run of pile
- c - After grinding

This test seems better than those presently used; however, the operational procedure and the equipment used should be specified: agitation, etc...

- A - The tests were carried out on samples taken in the mine and upgraded in the laboratory according to industrial conditions.

Solubilization tests were done (according to the operational conditions mentioned in the paper) in beakers located above magnetic agitators operating at constant revolving speed in all the tests.

The particle size of the samples was the same as merchant grade phosphate, namely:

- R.T. 1 mm            5 to 12%
- R.T. 0.125 mm      80 to 88%
- R.T. 0.08 mm       94 to 99%

Mr. BECKER added that he preferred the method used in the tests described in the paper to the one commonly used involving the dissolution of phosphate in 2% citric acid and 2% formic acid.

- Q - Mr. A. BARBERA, Agrimont, Italy

I should like to know if in your study you have considered the following points:

- 1 - Sieve size of phosphate rock
- 2 - Crystallite dimensions of phosphate rock
- 3 - Carbonate content of apatite crystals

If you have considered them, can you give details on the results related to reactivity?

Further on I did not understand how you selected conditions of tests. For example, why did you select S/L ratio 2/100 instead of 1/100 or 3/100.

- A - We do not investigate the variation of the reactivity according to particle size, crystalline characteristics of the phosphate and the carbonate content of the apatite crystal. Our selection of operational conditions was based on the following criteria:

- The rate of dissolution can only be followed if it is not instantaneous
- The dissolution must be achieved far from the supersaturation zone.

A  $\frac{\text{solid}}{\text{liquida}}$  ratio of  $\frac{2}{100}$  suits these two criteria best.

Q - Mr. M. BARLOY, SCPA, France

- a - What is the granule size of phosphates used in the reactivity tests?
- b - What is the influence of granule size on reactivity?
- c - What is the correlation with the industrial retention time?

A - a - The granule size of phosphates used in the reactivity tests was as follows:

- R.T. 1 mm : 4 to 9%
- R.T. 0.4 mm : 7 to 23%
- R.T. 0.125 mm : 81 to 87%
- R.T. 0.08 mm : 97 to 99%

The granule size varies in those ranges from one sample to another according to the layer used.

b - We did not study the influence of granule size on reactivity. To our knowledge there is no direct relationship between reactivity and particle size (for Tunisian phosphate). It is worth mentioning that Tunisian phosphate is very reactive and that it is not ground before dissolution.

c - The industrial retention time is the time necessary to complete the following stages:

- phosphate solubilization
- the reaction of monocalcium phosphate with H<sub>2</sub>SO<sub>4</sub>
- maturation of the slurry (growth of gypsum crystals)

In that connection the dissolution time is only a minimal fraction of the total retention time.

Q - Mr. P. ORPHANIDES, Duetag, France

Do you intend to continue your reactivity work on other ores?

A - We intend to continue the research mainly in the following directions:

- Thermogravimetric studies
- Radiocrystallographic studies

The idea is to determine the crystalline parameters and the possible substitution rates to confirm the established reactivity scale. This research work will be extended to other ores in Tunisia.

Q - Mr. L.C.J. THIREZ, BASF, Belgium

Why do you mix the layers of the ore under study and you do not process them separately?

A - The mining of the ore does not enable to separate the layers.



