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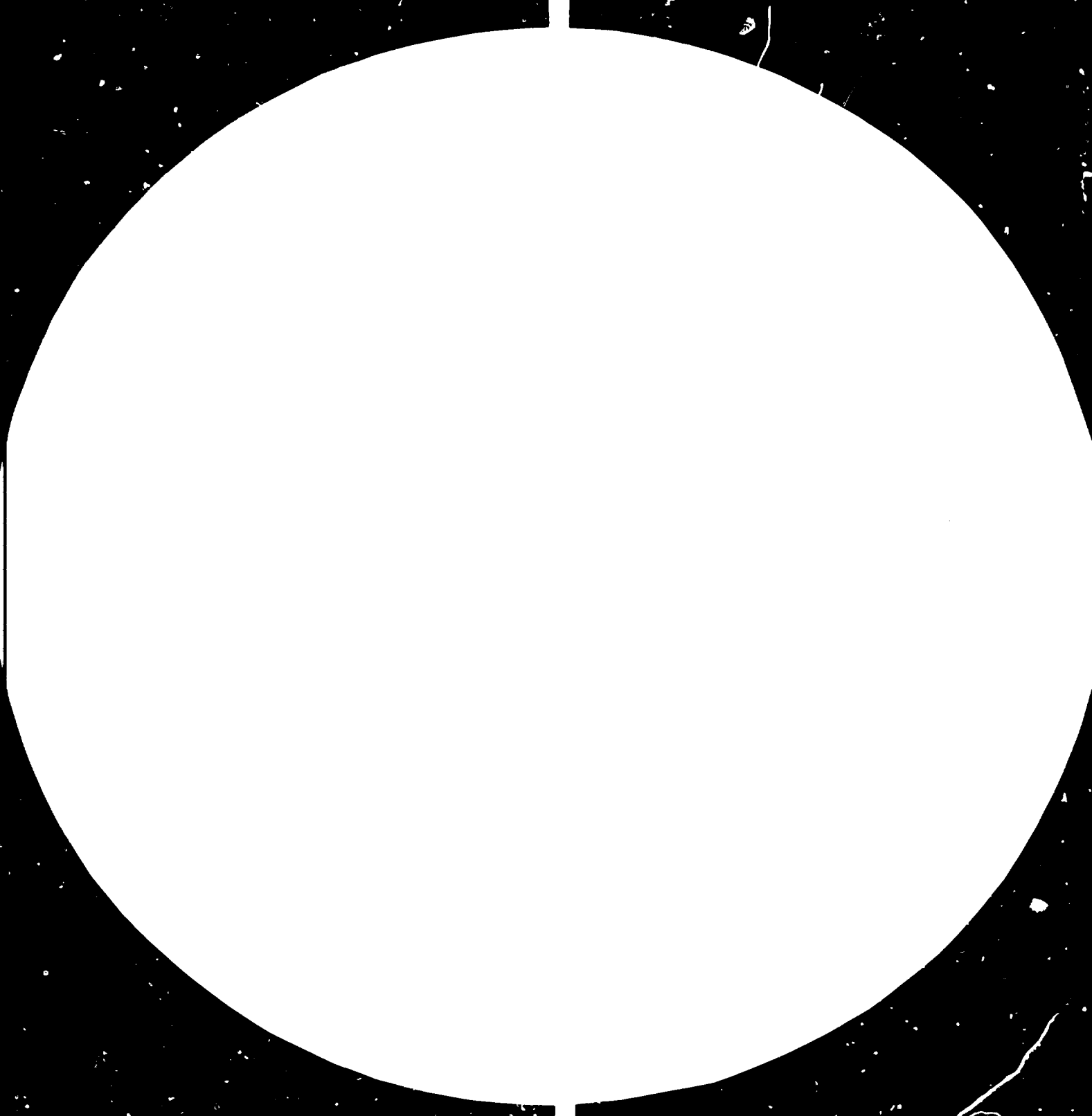
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**GROUP TRAINING IN PRODUCTION  
OF ALUMINA  
VOLUME 6**

**TECHNOLOGICAL INVESTIGATIONS OF BAUXITES AND  
RED MUDS**

**ALUTERV-FKI**

**BUDAPEST, JULY 1970**

## VOLUME 6

### TECHNOLOGICAL INVESTIGATIONS OF BAUXITES AND RED MUDDS BY

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## GENERAL ASPECTS OF BAUXITE EVALUATION FROM THE POINT OF VIEW OF ALUMINA PRODUCTION

Though the literature dealing with alumina production is voluminous, publications related to the grading of bauxites from the point of view of alumina production are almost totally missing, because the relevant information forms a part of the knowledge, jealously hidden as "know how" at big alumina corporation.

By evaluation of bauxites from the point of view of alumina production, the determination of technological parameters depending on the quality of the bauxite and having a principal effect on the cost of production is meant. Those technological conditions have to be determined primarily which can minimize caustic soda and bauxite consumption. Therefore, the most important task at the technological evaluation of bauxites is the determination of extractable alumina, of the parameters of digestion and of the composition and separability of the obtained red mud /1/.

### DEVELOPMENT OF BAUXITE GRADING FROM THE POINT OF VIEW OF ALUMINA PRODUCTION

Very few publications dealing with grading of bauxites from the point of view of alumina production are available. In case the bauxite is produced for sale the analysis is confined mostly to the main components as regards performing the quality requirements fixed in conditions of delivery. Delivery contracts are based even nowadays mainly on the basis number,  $B = Al_2O_3 \% - 2SiO_2 \%$  where  $Al_2O_3$  and  $SiO_2$  content of bauxite is presented in percentages.

Another commonly used practice applied in the Soviet Union and Hungary too is to classify bauxites by their module. The module is the ratio of the  $\text{Al}_2\text{O}_3$  and  $\text{SiO}_2$  content of bauxite  $/\text{Al}_2\text{O}_3 \text{ \%}/\text{SiO}_2 \text{ \%}/$ .

#### THE MOST IMPORTANT FACTORS OF THE INDUSTRIAL VALUE OF BAUXITE

The aim was to ascertain the industrial value of bauxite in a manner that ensures reasonable profit in alumina production.

Corresponding to this statement the value of bauxite can be expressed with the following formula

$$V_{\text{bx}} = \frac{S_{\text{alumina}} - Pr_{\text{alumina}} - C_{\text{constant}} - C_{\text{proportional}}}{G_{\text{bauxite}}}$$

where:  $V_{\text{bx}}$  = value of bauxite / ton dry bauxite  
 $S_{\text{alumina}}$  = selling price of alumina / t  
 $Pr_{\text{alumina}}$  = profit of the alumina production / t  
 $C_{\text{constant}}$  = operating costs independent of the grade of bauxite / t alumina  
 $C_{\text{prop.}}$  = operating costs depending on bauxite quality / t alumina  
 $G_{\text{bauxite}}$  = dry bauxite consumption t / t alumina

The costs of process operations independent of the quality of bauxite - like precipitation, calcination, evaporation - can be counted among constant costs. The settling costs may also be included insofar as the cost difference corresponding to the changes in the amount and characteristics of red mud can be accounted for in changes of caustic losses.

The proportional costs  $/C_{prop.}/$  are expressed by the following formula

$$C_{prop.} = C_{Na_2O} + C_{dressing} + C_{water}$$

where:  $C_{Na_2O}$  = costs of making up the  $Na_2O$  losses / t alumina

$C_{dressing}$  = dressing costs of bauxite / t alumina

$C_{water}$  = evaporation costs of the water introduced with bauxite / t alumina

The most significant among these costs is the cost of  $Na_2O$  losses which varies sharply with bauxite grade while  $C_{dressing}$  and  $C_{water}$  can be considered constant costs at the first approach.

Using that negligence:

$$C_{prop.} = C_{Na_2O} + C$$

where:  $C$  = constant

Put the value of  $C_{prop.}$  into the formula calculating the value of bauxite:

$$V_{bauxite} = \frac{S_{alumina} - Pr_{alumina} - C_{constant} - C_{Na_2O} - C}{G_{bauxite}}$$

The selling price of alumina and the reasonable profit of production can be considered as constant, so they can be

drawn together with and  $C$  in a new constant:  $C'_c$

$$V_{\text{bauxite}} = \frac{C'_c - C_{\text{Na}_2\text{O}}}{G_{\text{bauxite}}}$$

So the value of bauxite is determined in the main by the costs of making up the caustic losses and by the consumed amount of dry bauxite. The magnitude of  $G_{\text{bauxite}}$  depends on its 'soluble'  $\text{Al}_2\text{O}_3$  content determined besides its reactive  $\text{SiO}_2$  content by the undigested and operating losses.

#### TECHNOLOGICAL GRADING SYSTEM OF BAUXITES IN HUNGARY

A dynamic and scientifically well-established system is to be developed which reflects the effect of bauxite quality produced on operating costs of alumina and satisfies the requirements of technical development and economic optimization. The first two aims /dynamic character and scientific establishment/ can be met by petrological and mineralogical evaluation, the latter two /technological development and economic optimum/ by technological investigations.

The setting up of the grading system is summarized in Table 6.1. We have to underline the mineralogical analysis serving as basis of planning the technological investigations.

The theoretical establishment of the grading system and its role in promoting the technological development can be ensured only by applying these investigations and developing these methods ceaselessly.

The earlier evaluating system was confined only to technological investigations. Evaluation of bauxites was limited



Technological Grading System of Bauxites in ALUTERV-FKI

Table 6.1

Group	Petrological and Mineralogical Analysis	Technological Investigations
Purpose	Scientific Establishment, Dynamic Feature	Economic Optimum, Technological Devt.
Methodology	1. Determination of the petrological texture /with help of micro section, electronic microscope, electronic microprobe/	1. Grindability
	2. Quantitative phase analysis of bauxites /X-ray diffractometry, IR, thermal methods/	2. Digestibility, parameters
	3. Quantitative phase analysis of red mud	3. Soda losses
	4. Physico-chemical investigations /measuring of the specific surface area, pore distribution and heat of immersion/	4. Red mud separation and washing
	5. Introduction of new analytical methods	5. Technological consequences of impurities in bauxite
Testing		
Application	1. Processing and evaluation of bauxites of high calcite and dolomite content	
	2. Evaluation of bauxites of goethite content from the point of view of alumina production	
	3. Evaluation of 'low grade' bauxites	
	4. Evaluation of bauxites of high siderite content	

to digestible alumina /bauxite consumption per ton alumina/ and to NaOH consumption. At evaluation of bauxites from the point of view of alumina production also these two factors were minimized by well chosen technological parameters.

The nature of bauxites during processing is determined first of all by its mineralogical composition but the petrological composition and the morphology are important, too. In most cases the role of these two factors can not be separated from each other. At a first approach the mineralogical composition determines the equilibrium and pseudo-equilibrium conditions, while the texture influences the kinetics firstly.

The fine dispersed structure and large specific surface area of bauxites promotes e.g. the quick extraction of alumina minerals but parallelly renders the separation and washing of produced red mud more difficult and increases the so-called dissolved caustic losses. These statements are valid only as long as certain phases do not suffer any phase transformation by the technological treatment, first of all by digestion. In most cases such transformations take place - at least partly - and this underlines the significance of the usage of mansided petrological and physico-chemical investigation methodology in the technological grading and evaluation system.

Quartz and rutile are harmful only in case, when they seem to react with the digesting liquor. Different types of chamosites are known. If they do not react during digestion, neither their  $\text{SiO}_2$  nor their  $\text{Al}_2\text{O}_3$  content get dissolved, and do not cause caustic losses. Goethite and hematite increases the undigested  $\text{Al}_2\text{O}_3$  losses proportionally to the amount of Al atoms built in the crystal lattice. The hematite improves, however, the goethite deteriorates the settling and the washing of the mud. The carbonate content of calcite and dolomite increases the  $\text{Na}_2\text{CO}_3$  content of the liquor sharply but decreases the caustic losses bound to the mud.

It is evident that the result of many effects has to be determined. The behaviour of certain components can change by the interaction of different mineralogical phases. So the complicated task of evaluating the bauxites from the point of view of alumina production can be solved only on the basis of wide range of investigations and detection of causal relations.

#### MODELLING OF TECHNOLOGICAL PROCESSES; COMPARISON OF LABORATORY AND PILOT PLANT INVESTIGATIONS

A model is a materially realized or speculatively synthesized system which substitutes the object of the research in the process of cognition, so that it is analogous to the research-object. In consequence, studying the model and carrying out processes with it enables gaining of informations about the real object of research.

Principally the models can be classified in two groups:

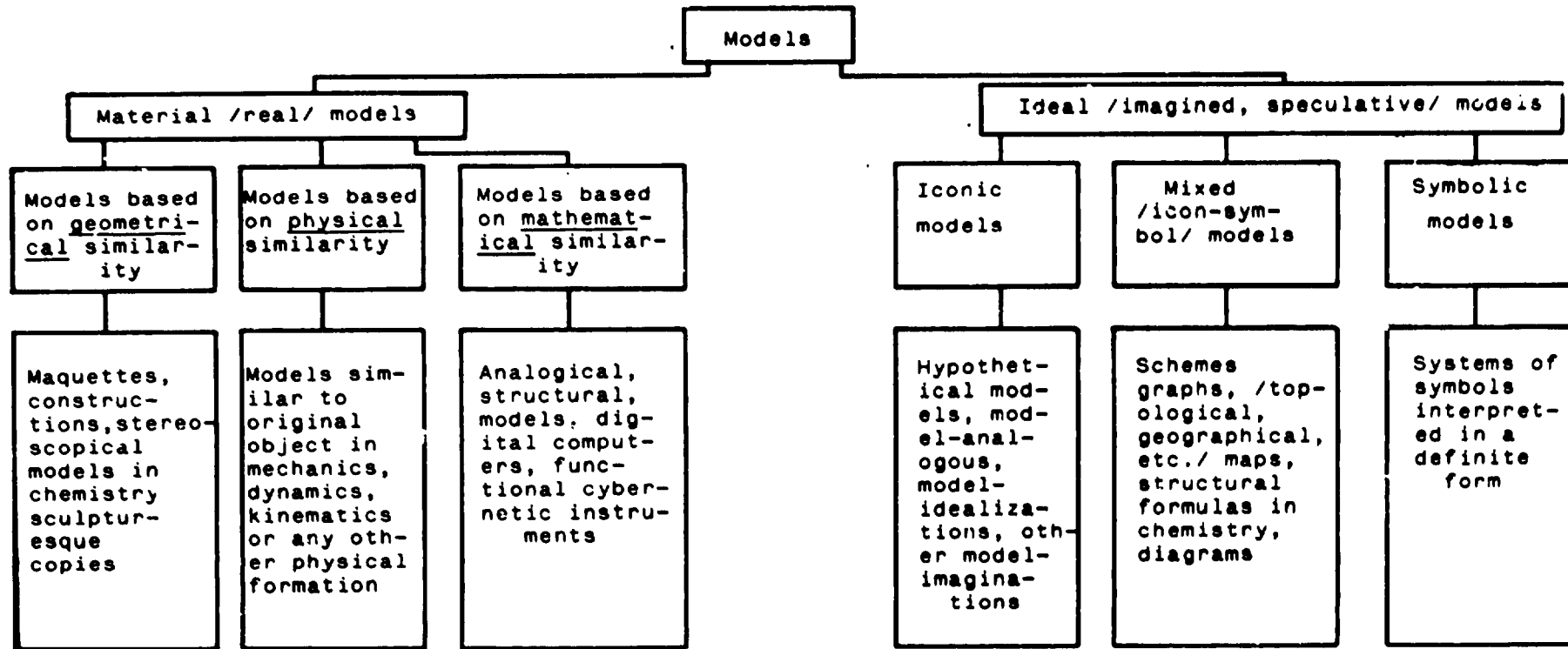
1. material /real/
2. ideal /imagined, speculative/ models.

Both of the groups can be divided in more subgroups corresponding to Table 6.2 /2/.

Our models are material ones, based on geometrical, physical and mathematical analogy. They reflect the substance /all the elements the system is built of/, the structure /all the connections among the elements/ and the function /behaviour of the system as unit/ of the original object. One of the theoretical principles of modelling /3/ is the theory of similarity. It provides the laws of modelling where the 'physical' nature of the object and that of the model is the same or nearly the same. This means that the model and the original object belong to the same form of motion /most frequently to

Classification of Models

Table 6.2



the same mechanical motion/. Our technological investigations belong to this category.

In mathematical modelling the basic principle of conformity between the model and the original object is a generalization of the theory of similarity. It takes into consideration the differences in the quality of the model and of the object and the fact that they belong to different motion forms of the material.

Though certain units of the Bayer process can be modelled fairly well, the significance of pilot plant investigations is increasing. In new plants of higher and higher capacity parts of the process line must not be under- or oversized. On the other hand, by using new deposits, new types of ores have to be processed without former plant experiences with them.

These difficulties can be reduced by applying pilot plants. Experiences gained in pilot plants are also suitable for designing the start-up of a new plant for training the new staff, saving thereby considerable expenses. E.g. Swiss Aluminium Ltd. designed an alumina plant of 1 million ton per year capacity, located on the bauxite deposit at Gove, on the experiences of a pilot plant set up partly for that purpose. The scale between the pilot plant and the real plant was about 1:100.

However, as building and operating a pilot plant needs considerable expenses very frequently in critical cases, to save time, the parameters of the plant are determined on the basis of laboratory investigations and excess capacities of machinery and equipment are designed at critical places.

Model tests are carried out on laboratory scale. Results of some investigations can be directly used at the design work and at the operation of even 1000 times greater plant

machinery and equipment. Investigations of processes like fluidization, crushing, grinding, digestion and so on can be listed in this group. However, in many cases the switch-over from laboratory to pilot plant scale raises problems, even more considerable when transforming the result to plant scale. In these cases laboratory results can be evaluated only as a tendency of phenomena and they can not be considered as absolute figures. Only after appropriate practice laboratory and the pilot plant results data can be transferred to plant conditions.

On the basis of adequate laboratory investigations, mathematical models can be generated, which follow properly the changes of technological parameters, and are suitable for process control in a plant or for determining the optimum parameters of a new project.

In Fig. 6.1 the scheme of elaboration of optimum technology by means of the former and up-to-date methods are shown /4/. In part a/ of the figure the test material is submitted to laboratory and pilot plant investigations for further developing the process technology or for determining the optimum technology in a new plant to be designed. Part b/ of the figure shows that pilot plant tests and large scale experiments can be omitted by applying /mathematical/ modelling of processes in many cases. Application of modelling the deviations of results of laboratory, pilot plant and large scale experiments can be eliminated.

The digestion and soft gamma ray absorption settling tests described below give a good example of this statement.

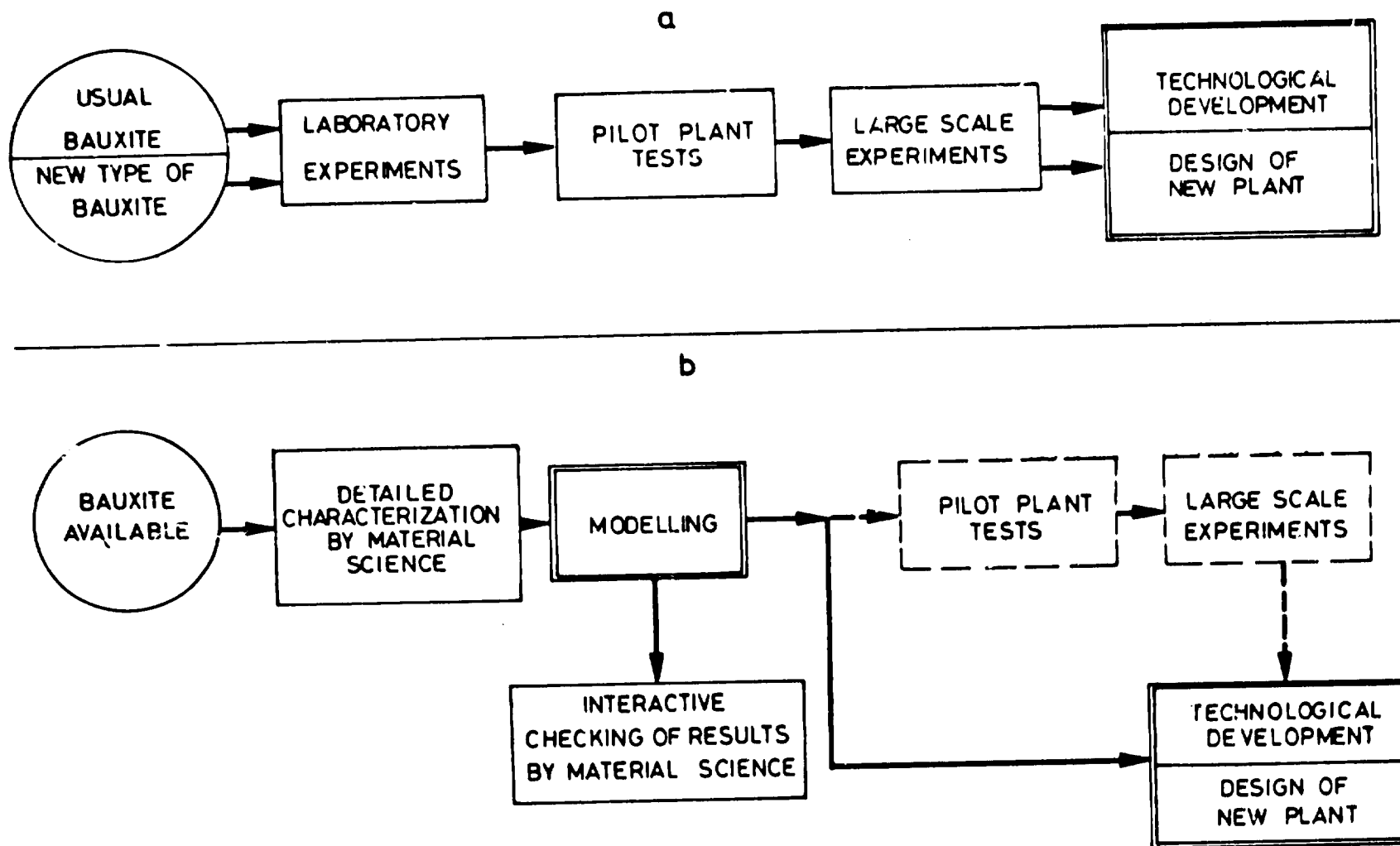


Fig. 6.1  
 THE SCHEME OF ELABORATION OF OPTIMUM TECHNOLOGY BY MEANS  
 OF FORMER (a) AND UP-TO-DATE (b) METHODS, RESPECTIVELY

TECHNOLOGICAL INVESTIGATION ON THE CHARACTERISTICS  
OF BAUXITE AND RED MUD

CRUSHING AND GRINDING TESTS

The grain-size of bauxites arriving from mines to alumina plants is generally too high to extract its  $Al_2O_3$  content quickly and with high efficiency. Therefore, the ore is to be comminuted, namely crushed and ground before introducing it into the Bayer process. The efficiency of equipment generally used is very low. So, it is necessary to determine, how 'hard' is the ore, namely what amount of energy is needed to its comminution.

The hardness and density of some alumina minerals are shown in Table 6.3 corresponding to Table 5.8 in volume 5 page 5-59. The hardest alumina mineral is corundum, followed by diasporite, boehmite and gibbsite. Diasporic bauxites are usually harder than the others /5/.

The hardness of bauxites depends not only on their phases but on their age, too. Older bauxites are generally harder, but some of the bauxites of the same geological age exposed to higher tectonic pressure became harder /6/.

The petrological texture of the ore has a significant role as regards comminution of bauxites. Diameters of bauxite grains of spherical structure - so-called oolite bauxites - can vary from 100  $\mu m$  up to 10 mm.

Frequently, their core is a bigger homogeneous mineral grain, as a boehmite grain in Yugoslavian bauxite Fig. 6.2 /7/. This oolite grain is difficult to be dissolved by caustic liquor so it can be digested only following proper grinding of the ore.



Density and Hardness of Alumina Minerals

Table 6.3

Name and Chemical Composition	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	Na <sub>2</sub> O + R <sub>2</sub> O	Density gpccm	Hardness by Mohs- scale
	content in per cent				
Corundum Al <sub>2</sub> O <sub>3</sub>	100	-	-	4.0 -4.1	9
Boehmite Al <sub>2</sub> O <sub>3</sub> .H <sub>2</sub> O	85	-	-	3.01-3.06	3.5-4
Diaspore Al <sub>2</sub> O <sub>3</sub> .H <sub>2</sub> O	85	-	-	3.3 -3.5	6.5-7
Hydrargillite /Gibbsite/ Al <sub>2</sub> O <sub>3</sub> .H <sub>2</sub> O	65.4	-	-	2.35-2.42	2.5-3.5
Chianite Al <sub>2</sub> O <sub>3</sub> .SiO <sub>2</sub>	63.0	37.0	-	3.56-3.68	4.5-7
Andalusite Al <sub>2</sub> O <sub>3</sub> .SiO <sub>2</sub>	63.0	37.0	-	3.15	7.5
Sillimanite Al <sub>2</sub> O <sub>3</sub> .SiO <sub>2</sub>	63.0	37.0	-	3.23-3.25	7
Nephelite /Na,K/ $\frac{1}{2}$ O.Al <sub>2</sub> O <sub>3</sub> .2SiO <sub>2</sub>	32.3	38.0	19.6	2.63	5.5-6
	36.0	42.3	21.0		
Caliofillite K <sub>2</sub> O.Al <sub>2</sub> O <sub>3</sub> .2SiO <sub>2</sub>	32.3	38.0	29.5	2.4 -2.6	6
Feldspars	18.4	65.5	11.0	-	-
/Na,K/ $\frac{1}{2}$ O.Al <sub>2</sub> O <sub>3</sub> .6SiO <sub>2</sub>	19.3	69.3	11.2		
Muscovite	38.5	45.2	9.7	-	2
K <sub>2</sub> O.3Al <sub>2</sub> O <sub>3</sub> .6SiO <sub>2</sub> .2H <sub>2</sub> O					
Leucite	23.5	55.1	21.5	2.45-2.5	5 - 6
K <sub>2</sub> O.Al <sub>2</sub> O <sub>3</sub> .4SiO <sub>2</sub>					
Albite	19.4	68.8	11.8	2.61	6 -6.5
Na <sub>2</sub> O.Al <sub>2</sub> O <sub>3</sub> .6SiO <sub>2</sub>					
Analcite	23.2	54.5	14.0	2.25	5
Na <sub>2</sub> O.Al <sub>2</sub> O <sub>3</sub> .6SiO <sub>2</sub> .2H <sub>2</sub> O					
Sericite	39.5	46.4	-	2.58-2.6	1
K <sub>2</sub> O.Al <sub>2</sub> O <sub>3</sub> .6SiO <sub>2</sub> .2H <sub>2</sub> O					
Alunite	37.0	-	11.3	2.60-2.80	3.5-4.0
K <sub>2</sub> SO <sub>4</sub> .Al <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> .4Al(OH) <sub>3</sub>					
Cryolite 3NaF.AlF <sub>3</sub>	24.3	-	-	2.95-3.01	2 - 3



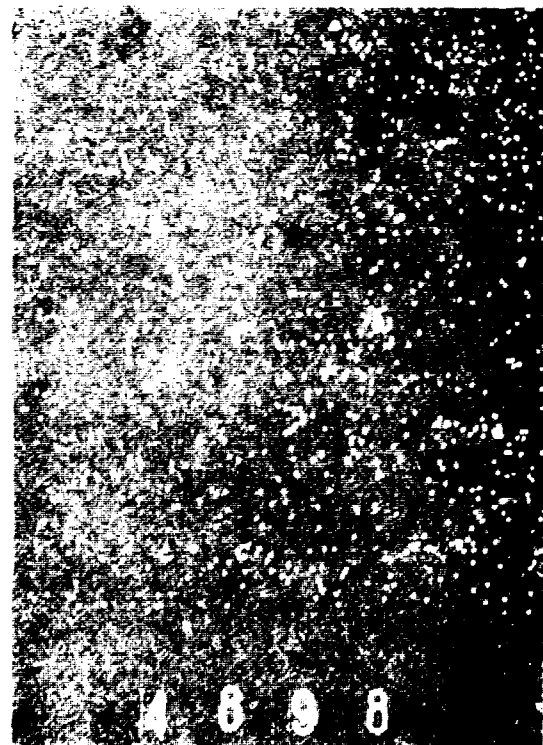
Picture of composition



Al - distribution



Fe - distribution



Si - distribution

Fig. 6.2  
DISTRIBUTION OF CERTAIN ELEMENTS IN AN  
YUGOSLAVIAN BAUXITE SAMPLE

Actually, comminution of bauxites is a preparatory process of digestion in Bayer process. Lateritic bauxites are soft, their comminution is rather a crumbling; Ores of boehmite and diasporic type are hard. Nevertheless, selection of an appropriate comminution equipment serves not only for a proper digestion, but has a significant influence on several other processes, too. Considering Fig. 5.6 in Volume 5, the amount of extractable  $Al_2O_3$  increases by the grinding fineness /8/. Overgrinding of the ore leads to deterioration of red mud settling and washing and the amount of the soda losses adherent to the mud increases.

Consequently, the optimum grinding technology is to be elaborated for each bauxite, taking into consideration all aspects mentioned above /digestion, settling, washing abilities, adhesive soda losses/.

In the practice of alumina production bauxites are not graded according to the Mohs-scale but to their comminutibility.

There are several theories for the determination of energy requirement of the comminution /9/. On the basis of these principles some indices /Zeisel-, or Hardgrove-number, Bond index/ can be worked out for the comparison of grindability of different ores and for the selection of the most appropriate grinding equipment.

However, it is to be underlined, that none of these procedures is modelling reliably the wet grinding technology applied in most plants. So, when elaborating an optimum grinding technology it is expedient to carry out grinding and settling experiments in pilot plant size.

## INVESTIGATION ON PREDESILICATION KINETICS; INFLUENCE OF THE MINERALOGY AND MORPHOLOGY OF THE BAUXITE

This procedure is not applied in every alumina plant but the security of plant operation processing bauxites of high silica content necessitates developing this process stage, too.

When treating bauxites by aluminate liquor  $\text{SiO}_2$  minerals and the soda content of the liquor get into reaction and a dissoluble compound, Na-Al-hydrosilicate /Linde zeolite/ is formed, causing breakdowns in the plant by forming scales on the tubes. In order to avoid these troubles pre-desilication process is interposed into the Bayer process. Non-reactive  $\text{SiO}_2$ , as quartz and its cryptocrystalline modifications /quartzite, calcedon, opal/ is present in bauxites. Reactive  $\text{SiO}_2$  is found in the form of different aluminium-hydrosilicates /kaolinite, illite, dickite, halloysite, pyrophyllite, allophane, muscovite, hydromuscovite, chamosite, etc./.

During the pre-desilication process non reactive  $\text{SiO}_2$  does not dissolve normally at 95-100 °C, or only a small quantity of it - fine particles - goes into solution.

Out of the minerals containing silica, chamosite does not dissolve normally, while kaolinite, halloysite, pyrophyllite, allophane dissolve very well under pre-desilication conditions /9/.

During pre-desilication, the reaction between the reactive silica content of a given bauxite and aluminate liquor of known composition should be followed in function of time. The changing of silica content has to be measured both in the liquid and solid phases. A conceptual curve is shown in Fig. 6.3.

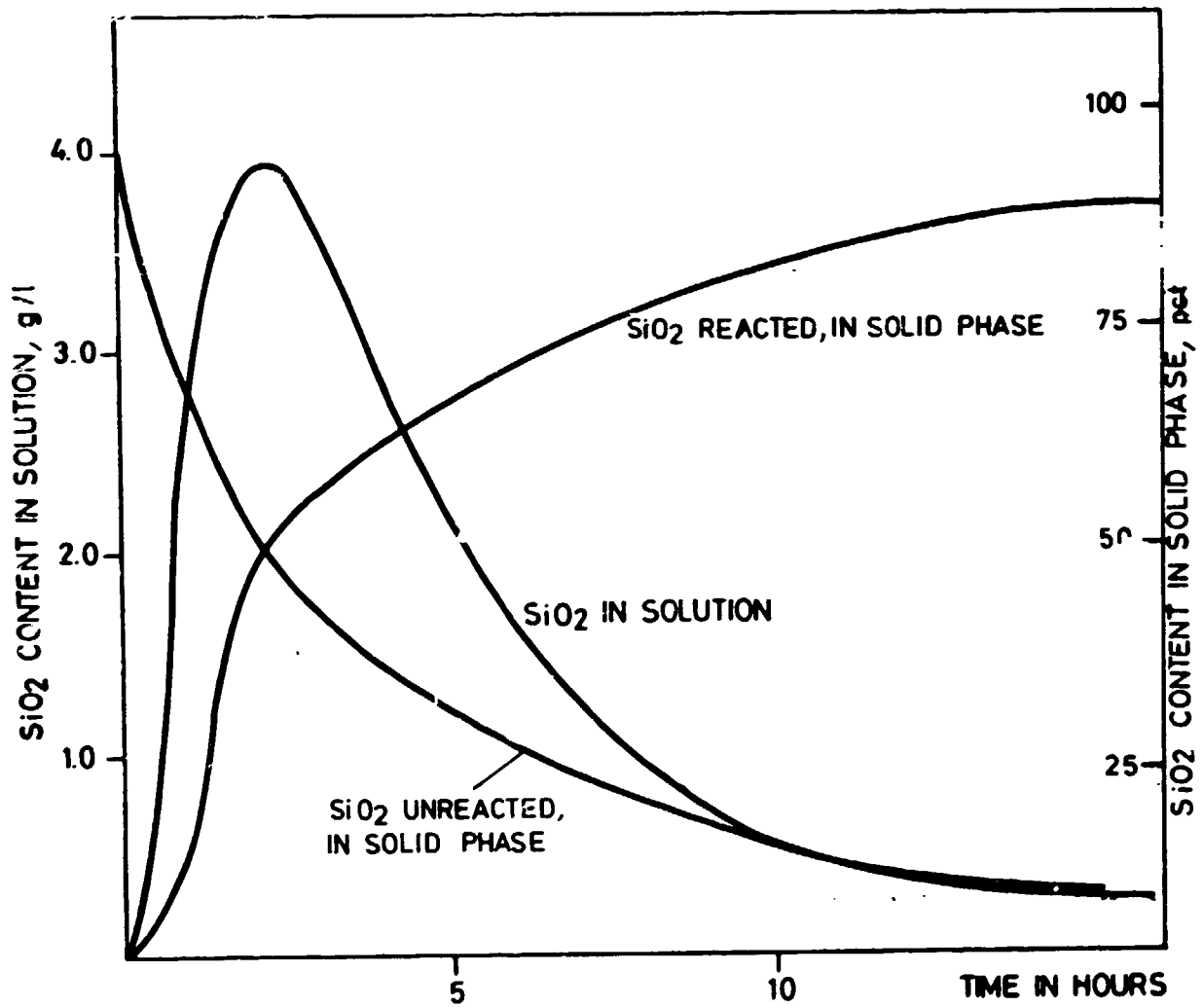


Fig. 6.3  
CHANGING OF SiO<sub>2</sub> CONTENT IN SOLUTION AND IN  
SOLID PHASE vs. TIME

The dissolution of  $\text{SiO}_2$  is completed practically during the heating up period, but its precipitation starts immediately as well. The shaping of the curve is influenced by the temperature, liquor concentration and molar ratio, so they have to be taken into consideration in determining the optimum.

Examples of the pre-desilication in case of kaolinite and some bauxites are shown in Figures 6.4 and 6.5, resp.

#### DETERMINATION OF OPTIMUM PARAMETERS OF DIGESTION; BEHAVIOUR OF BAUXITE MINERALS DURING DIGESTION

The basic chemical process of the Bayer technology is digestion, determining essentially both the  $\text{Al}_2\text{O}_3$  recovery and the loss of caustic soda.

Digestion technology, requires the optimization of the following parameters:

1. maximum  $\text{Al}_2\text{O}_3$  recovery
2. minimum NaOH loss
3. optimum washing, separation and handling of red mud

Digestion is one of the processes of the alumina technology well suited for modelling. Though the plant digesters operating in continuous series have to be substituted by batch digester models in laboratory, the material flow can be studied by radioisotope tracers in plant scale /12, 13, 14/ and approached by mathematical functions. The influence of the scale effect on the rate of reaction is not significant. The time of heating up and desilication can be adjusted easily according to the actual plant conditions.

#### Phase Analysis, the Foundation of Technological Grading

Knowledge of the mineralogical composition of bauxite and red mud obtained during digestion is indispensable for

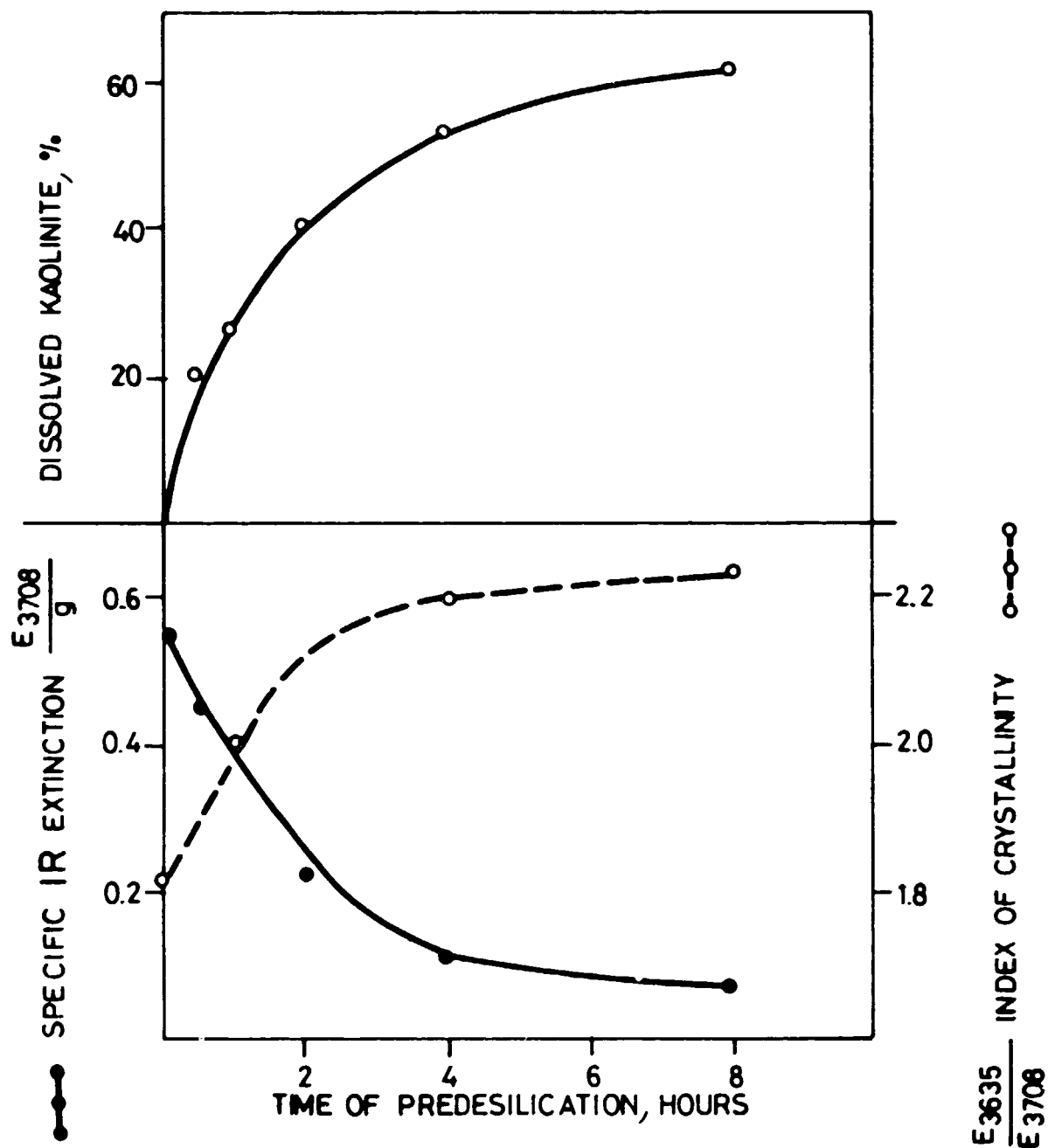


Fig. 6. 4

DESILICATION OF A KAOLINITE FROM ZETTLITZ AT 95°C  
 $\text{Na}_2\text{O}_{\text{CAUST}} = 200 \text{ g/l}$

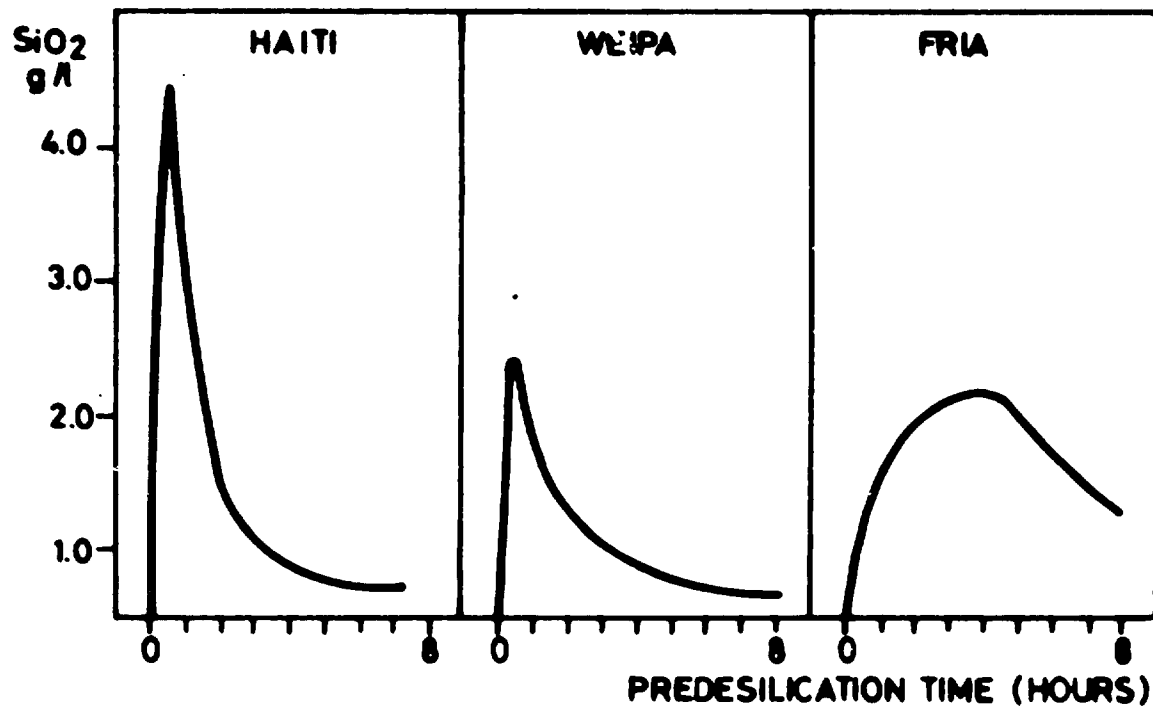


Fig.6. 5

SiO<sub>2</sub> CONTENT OF ALUMINATE LIQUOR IN FUNCTION OF PREDESILICATION TIME FOR HAITI, WEIPA, AND FRIA BAUXITES. ORIGINAL CONCENTRATION OF THE CAUSTIC LIQUOR: Na<sub>2</sub>O 194.8 g/l; Al<sub>2</sub>O<sub>3</sub> 78.0 g/l; MOLAR RATIO 4.11.



the distribution of undigested losses among individual mineralogical constituents. Petrological and mineralogical evaluation of a representative bauxite sample allows its preliminary technological grading and planning of detailed technological investigations without performing technological tests.

The nature of aluminium minerals defines the type of the bauxite /gibbsitic, gibbsitic with quartz content, boehmitic, diasporic, goethitic etc./. The non-reactive fraction of  $\text{SiO}_2$  /well-crystallized quartz/ does not react with the digesting liquor, does not cause caustic losses, in case of low temperature digestion. Aluminium incorporated in the lattice of iron minerals /goethite, hematite/ and detectable by means of X-ray diffractometry, can not be digested in the traditional Bayer technology. Also, diasporic occurring in most bauxites in percentages of about 1 % or more, remains unextracted in the Bayer cycle unless  $\text{CaO}$  is added. These direct information concerning alumina recovery to be expected may be obtained from quantitative phase analysis. Phase analysis of red muds reveals the efficiency of digestion, and allows to determine the distribution of alumina losses among the constituents.

If no instruments are at disposal, conclusions on the amounts of individual aluminium can be obtained from the results of digestion tests carried out under standard conditions at different temperatures. These tests simultaneously give information on available alumina, and may, therefore, be considered as technological tests also /12/. The parameters applied in these tests are usually as follows: temperature 120, 180 and 230-240 °C; digestion time 1 to 3 hours; caustic concentration 200 g/litre, so that the alumina percentage in the liquor after digestion should not reach the molar ratio corresponding to equilibrium. Gibbsite is digested at 120 °C, gibbsite and boehmite at 180-240 °C. Diasporic, however, becomes soluble only at 240 °C, only in presence of 3 to 4 per cent of  $\text{CaO}$ .

The value of loss on ignition makes possible already to estimate the type of the bauxite. An exact determination of the mineralogical composition, however, can be carried out only by applying up-to-date physico-chemical methods, namely thermal analysis, X-ray diffractometry and IR spectrometry.

The nomogram used for the determination of alumina incorporated in the lattice of goethite is shown in Fig. 6.6 /15/. The  $\text{Al}_2\text{O}_3$  substitution in the lattice of aluminohematite can be measured by X-ray diffraction by means of the calibration curves plotted in Fig. 6.7 /15/.

The calibration curve for the determination of Al-substitution in aluminogoethite is presented in Volume 4. Fig. 4.32.

The addition of CaO to bauxite or to aluminate liquor results in the formation of a phase having the composition of  $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 6\text{H}_2\text{O}$ . If the aluminate liquor contains dissolved  $\text{SiO}_2$  also, hydrogarnets with the composition  $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot k\text{SiO}_2 \cdot (6-2k)/\text{H}_2\text{O}$  will be formed.

Proceeding bauxite digestion at conditions close to those of desilication / $\text{Na}_2\text{O}_{\text{caust}} = 150-200 \text{ gpl}$ ,  $\alpha_{\text{caust.}} = 1.65$ ,  $T = 90^\circ\text{C}$ / the maximum value of  $k$  is 0.3. In the experiments carried out under digestion conditions / $\text{Na}_2\text{O}_{\text{caust}} = 220 \text{ gpl}$ ,  $\alpha_{\text{caust.}} = 1.68$ ,  $T = 210^\circ\text{C}$ ,  $t = 1 \text{ hr}$ / a value of  $k = 0.77$  was found.

A higher  $k$  value was only obtained by causticization of sodium aluminium hydrosilicates in low alumina containing caustic slurries. At these experiments the value of  $k$  approached 2.0 /16/.

The calibration curves for the determination of value  $k$  / $\text{SiO}_2/\text{Al}_2\text{O}_3$  molar ratio/ which has a determining importance in the phase analysis of red muds with CaO content is demonstrated in Fig. 6.8 /17/.

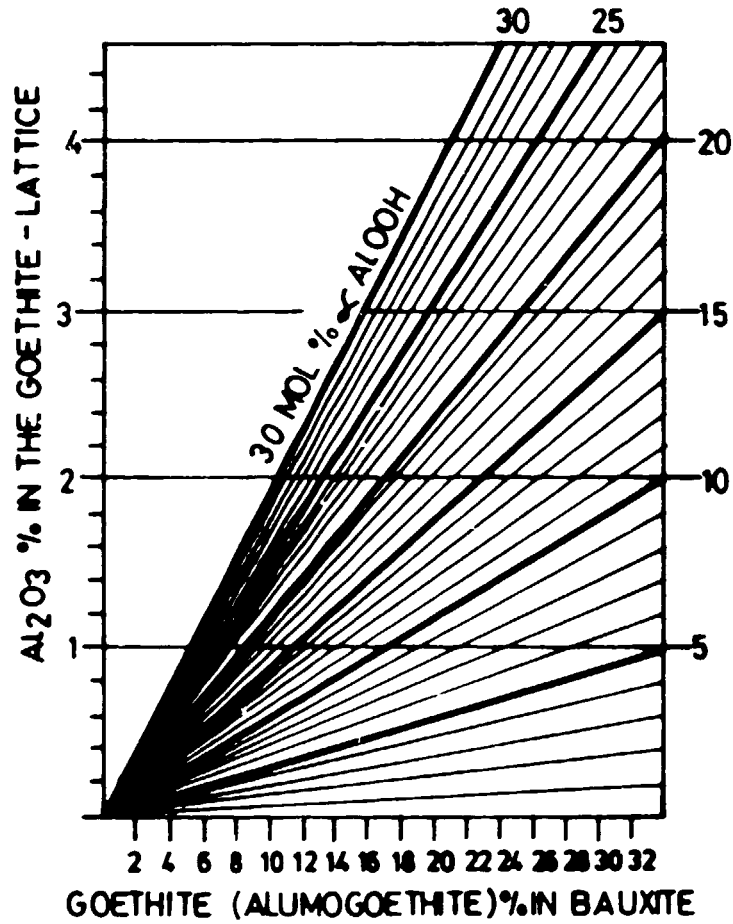


Fig. 6.6

NOMOGRAM FOR THE DETERMINATION OF  $Al_2O_3$   
INCORPORATED IN THE GOETHITE-LATTICE

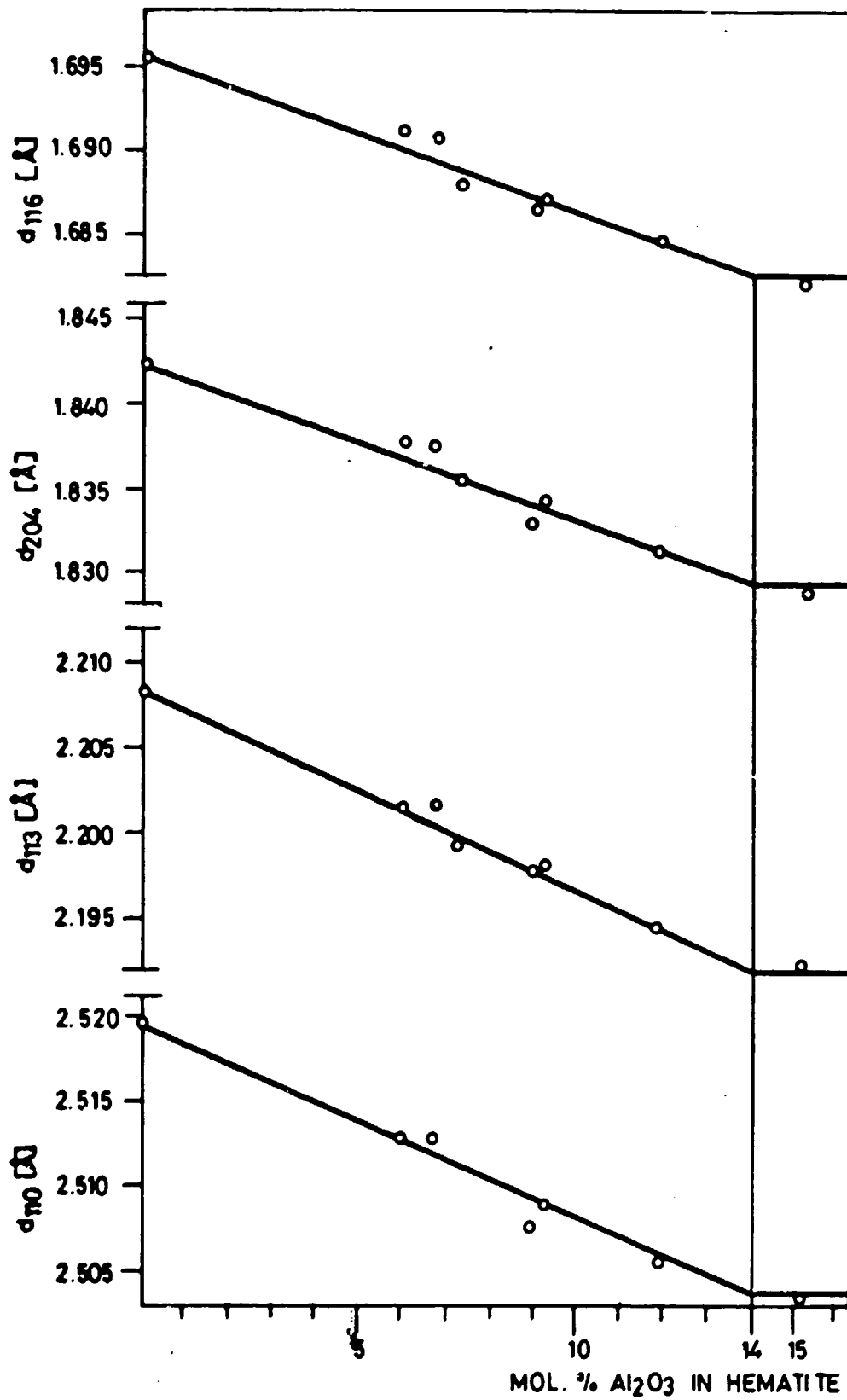


Fig. 6.7

CALIBRATION CURVES FOR DETERMINATION OF Al<sub>2</sub>O<sub>3</sub>  
IN ALUMOHEMATITE BY X-RAY

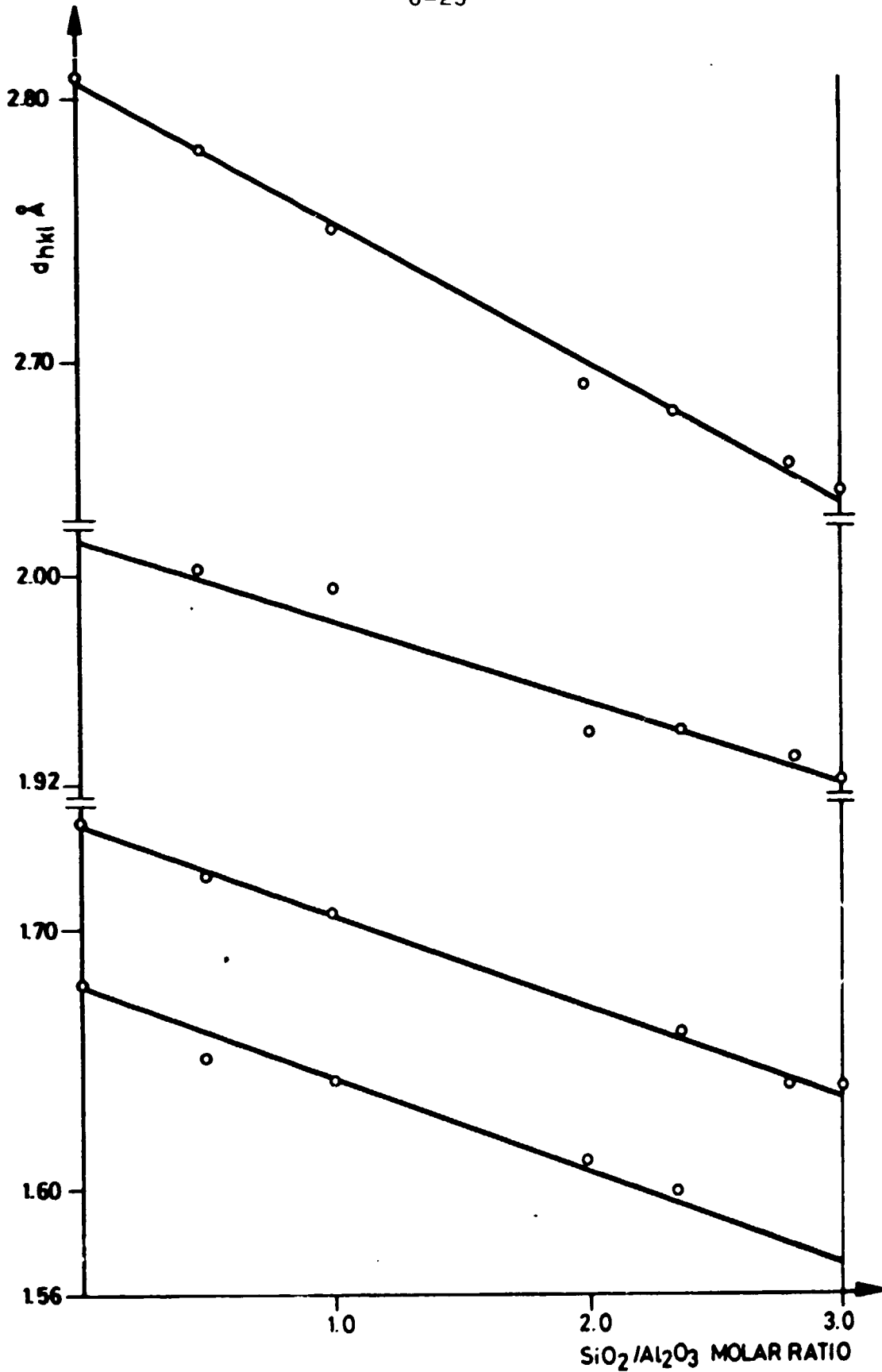


Fig. 6.8

CALIBRATION CURVES FOR THE DETERMINATION OF  
K VALUE ( $\text{SiO}_2/\text{Al}_2\text{O}_3$  MOLAR RATIO)

Determination of the Expected Alumina Recovery from the  
Results of Phase Analysis

Theoretical alumina recovery is usually calculated by means of the formula:

$$\eta_{\text{theor.}} = \frac{\text{Al}_2\text{O}_3\text{total} - 0.85\text{SiO}_2}{\text{Al}_2\text{O}_3\text{total}} \cdot 100 \%$$

In this formula only reactive  $\text{SiO}_2$  has to be taken into consideration.

For bauxites of the gibbsitic and boehmitic type /if no  $\text{CaO}$  is added in digestion/, the following formula is suggested:

$$\eta_{\text{mod.}} = \frac{\text{Al}_2\text{O}_3\text{total} - K_1 \cdot 0.85\text{SiO}_2\text{react.} - K_2 \cdot \text{Al}_2\text{O}_3\text{diasp.} - K_3 \cdot \text{Al}_2\text{O}_3\text{goeth.}}{\text{Al}_2\text{O}_3\text{total}}$$

$$- \frac{\text{Al}_2\text{O}_3\text{hem.}}{\text{Al}_2\text{O}_3\text{total}} \cdot 100$$

where:  $K_1 \approx 1.00$  number of  $\text{Al}_2\text{O}_3$  mols related to 2 moles of  $\text{SiO}_2$  in sodium aluminium hydrosilicate,

$K_2 \approx 1.00$  fraction of diasporé that remains undigested,

$K_3 \approx 1.00$  undigested fraction of alumina incorporated in the goethite lattice.

In practice,  $K_1 = 1.00$  to  $1.10$ ;  $K_2 = 0.80$  to  $1.00$  /part of the diasporé is recoverable, due to its disperse nature/;  $K_3 = 1.00$ , and finally, aluminium substitution in hematite is

usually negligible /2 to 3 per cent/. Therefore, the formula may assume a simplified form:

$$\eta_{\text{mod}}^* = \frac{\text{Al}_2\text{O}_3\text{total} - 0.85\text{SiO}_2\text{react.} - \text{Al}_2\text{O}_3\text{diasp.} - \text{Al}_2\text{O}_3\text{goeth.}}{\text{Al}_2\text{O}_3\text{total}} \cdot 100$$

It should be noted that if other constituents unextractable or only partly extractable by the given technology are present in the bauxite, their amount must be taken into account similar to that of diasporite and alumogothite. Chamosite, e.g. in Iranian bauxites, is such an 'unextractable' constituent.

To characterize the effectiveness of digestion, the introduction of the concept 'efficiency of digestion' is suggested. This term expresses the sum of the losses arising from undigested constituents and from hydrolysis. It is obtained by dividing the practical recovery  $\eta_{\text{pract}}$ , calculated from the composition of red mud, by the modified theoretical recovery  $\eta_{\text{mod}}^*$  /15/.

$$\eta' = \frac{\eta_{\text{pract}}}{\eta_{\text{mod}}^*}$$

By means of proper technology the diasporite and goethite content can be dissolved as well. /Addition of CaO and catalysts for the transformation of goethite into hematite./ Other factors to be considered are non-reactive  $\text{SiO}_2$  that increases the grade of the bauxite and impurities e.g. carbonates, sulfates, organic matter, pyrite etc. causing caustic losses or difficulties in processing and thereby reducing the value of the bauxite. In recent years it has become customary to include the value of some of these constituents

/e.g. the carbonate content of diasporic bauxites/ in commercial contracts.

The above-discussed method allows a pre-estimation of the alumina recovery to be expected, i.e. a preliminary technological grading of the ore.

The problem of physical and chemical enrichment shall also be mentioned. The feasibility of physical enrichment can be judged e.g. by the microprobe study demonstrating the distribution of Si and Al, without the necessity of performing technological tests. This feasibility, on the other hand, may substantially affect the technological evaluation, and the value of the bauxite. All these factors have to be taken into account in the evaluation and grading of the so-called low 'grade' bauxites.

#### Main Parameters of Digestion

The main technological parameters of digestion are:

- the equilibrium molar ratio
- the parameters influencing the kinetics /retention time,  $t$ ; temperature,  $K$ ; surface area of bauxite,  $S$ ;  $\text{OH}^-$  concentration of digesting liquor,  $\text{Al}/\text{OH}/4$  concentration of digesting liquor.

The molar ratio of the aluminate liquor can be expressed by means of different terms as follows:

$$\begin{array}{l} \text{caustic molar ratio:} \\ \text{/European practice/} \end{array} \quad \frac{\text{Na}_2\text{O}_{\text{caust}} \text{ moles}}{\text{Al}_2\text{O}_3 \text{ moles}}$$

$$\begin{array}{l} \text{caustic weight ratio:} \\ \text{/French practice/} \end{array} \quad \frac{\text{Al}_2\text{O}_3 \text{ gpl}}{\text{Na}_2\text{O} \text{ gpl}}$$



A/C /alumina/caustic/  
 ratio:  $\frac{\text{Al}_2\text{O}_3 \text{ gpl}}{\text{Na}_2\text{O gpl}}$  /expressed as  
 /oversea's practice/  $\text{Na}_2\text{CO}_3/$

All of these expressions are used in the technical literature. Often the following abbreviations are used, too:

CS = caustic soda / $\text{Na}_2\text{O}_{\text{caustic}}$  expressed as  $\text{Na}_2\text{CO}_3/$ ,  
 gpl

S = soda /inorganic  $\text{CO}_2$  as  $\text{Na}_2\text{CO}_3/$ , gpl

TS = total soda /CS + S/, gpl

C/S = caustic /total soda/CS/TS/, causticity

TAA = total available alumina / $\text{?}$  /

During digestion the equilibrium molar ratio of the aluminate solution is the most important factor determining considerably the optimum technological parameters of the digestion. The equilibrium molar ratio of aluminate liquor digesting different types of bauxite was determined by Soviet authors. Their results are shown in Fig. 6.9 /8/.

The data plotted for comparison were obtained with minerals prepared by Rasses, Bernstein etc. The difference between the solubility of synthetized minerals and that of the processed bauxites can be explained by the feature of mineral individualism and heterotypism. Data given in Fig. 6.9, serve as a basis of selecting the main technological parameters during bauxite digestion /temperature of digestion, concentration, final caustic molar ratio/. The diasporic bauxite samples were digested with lime addition /3 % on dry bauxite.

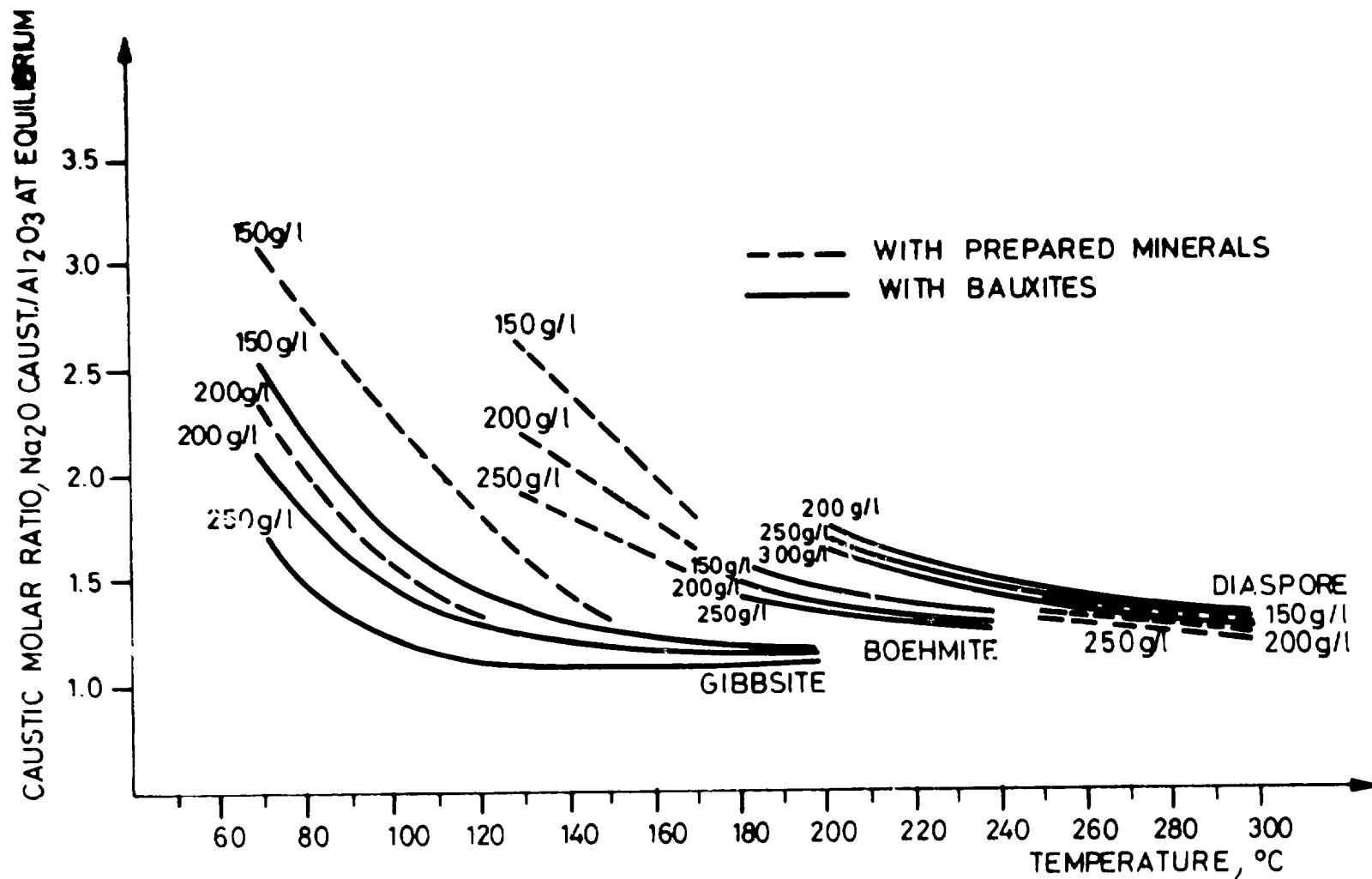


Fig. 6.9  
EQUILIBRIUM CAUSTIC MOLAR RATIO DURING DIGESTION OF BAUXITES  
OF DIFFERENT MINERALOGICAL TYPES

The kinetics of digestion of different mineralogical types of bauxite is given in Fig. 6.10 /18/.

As seen, the digestion of gibbsitic bauxites is finished in the first ten minutes and the further retention time is required by the desilication of the solution progressing rather slowly at 105 °C.

In contrary, the digestion of boehmitic and diasporic bauxites will be completed during longer retention time only, even at higher temperature while the desilication is intensified by the higher temperature. In this case the digestibility of alumina containing minerals is a function of the holding time.

The chemical reaction can be characterized by the following equation:

$$\frac{d \text{ Al/OH/}_4}{dt} = k f_1 / S / f_2 / \text{OH}^- / f_3 / \text{Al/OH/}_4 \quad f_4 / T /$$

t = reaction time

s = surface of solid phase

k = reaction velocity constant

T = absolute temperature, K

#### Main Specific Material Consumptions

The alumina and silica contents of bauxite determine directly the consumption of bauxite and caustic soda. The other consumption values depend considerably on the perfection of the adapted technological flow sheet, equipment, and the plant capacity as well.

The bauxite consumption is determined by the expression /18/:

$$Q_{\text{bauxite}} = \frac{9900}{A \eta_{\text{actual}}}$$

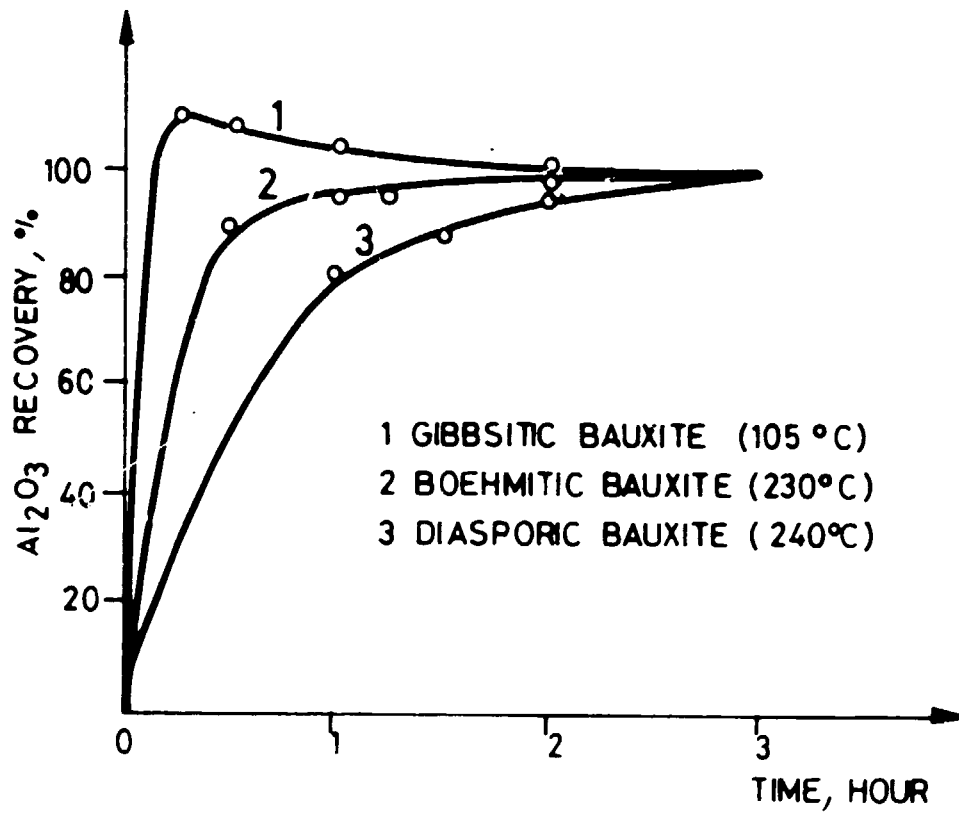


Fig. 6.10

KINETICS OF DIGESTION

where:  $Q_{\text{bauxite}}$  = bauxite consumption, t/t

A =  $\text{Al}_2\text{O}_3$  content in bauxite, %

$\eta_{\text{actual}}$  = actual recovery of alumina, %

/The  $\text{Al}_2\text{O}_3$  content of the produced alumina is supposed to be 99 per cent./

The bauxite consumption in the function of the total available alumina content /TAA/ is demonstrated in Fig. 6.11.

The consumption of caustic soda can be calculated from the sodium content of the red mud obtained by the following expression /18/:

$$Q_{\text{caustic}} = \frac{990 \cdot h}{M \cdot \eta_{\text{actual}}} \cdot 100$$

where:  $Q_{\text{caustic}}$  = caustic soda consumption,  $\text{Na}_2\text{O}$ , kgpt

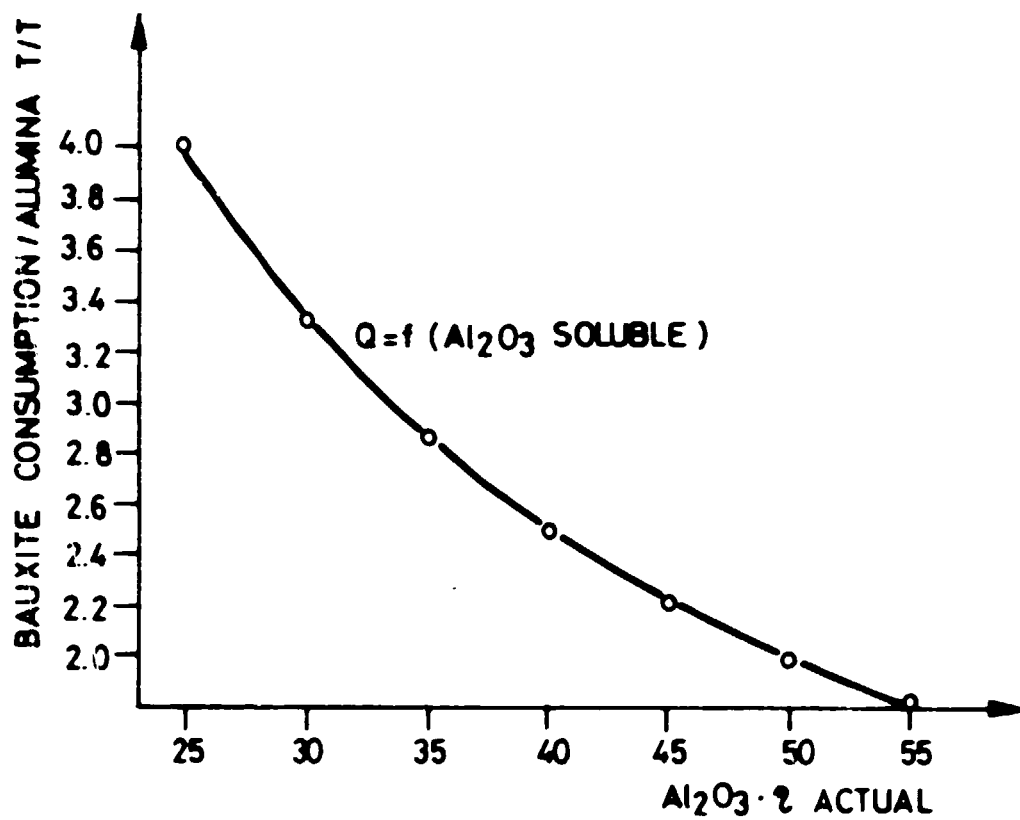
h = weight ratio  $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$  in red mud determined experimentally

M = silica module:  $\text{Al}_2\text{O}_3/\text{SiO}_2$  of bauxite

The caustic soda consumption depending on the silica module of bauxite at different weight ratios of  $\text{Na}_2\text{O}/\text{SiO}_2$  in mud after digestion can be seen in Fig. 6.12.

#### Determination of the Digestion Parameters

The simplest determination method for extractable alumina is the treatment of the bauxite sample with aluminate liquor under standard conditions. However, this method provides no information on the parameters of digestion /optimum tempera-



$\text{Al}_2\text{O}_3 = \text{Al}_2\text{O}_3$  CONTENT IN BAUXITE, WPCT

$\% \text{ ACTUAL} = \text{Al}_2\text{O}_3$  RECOVERY, %

Fig. 6. 11

BAUXITE CONSUMPTION

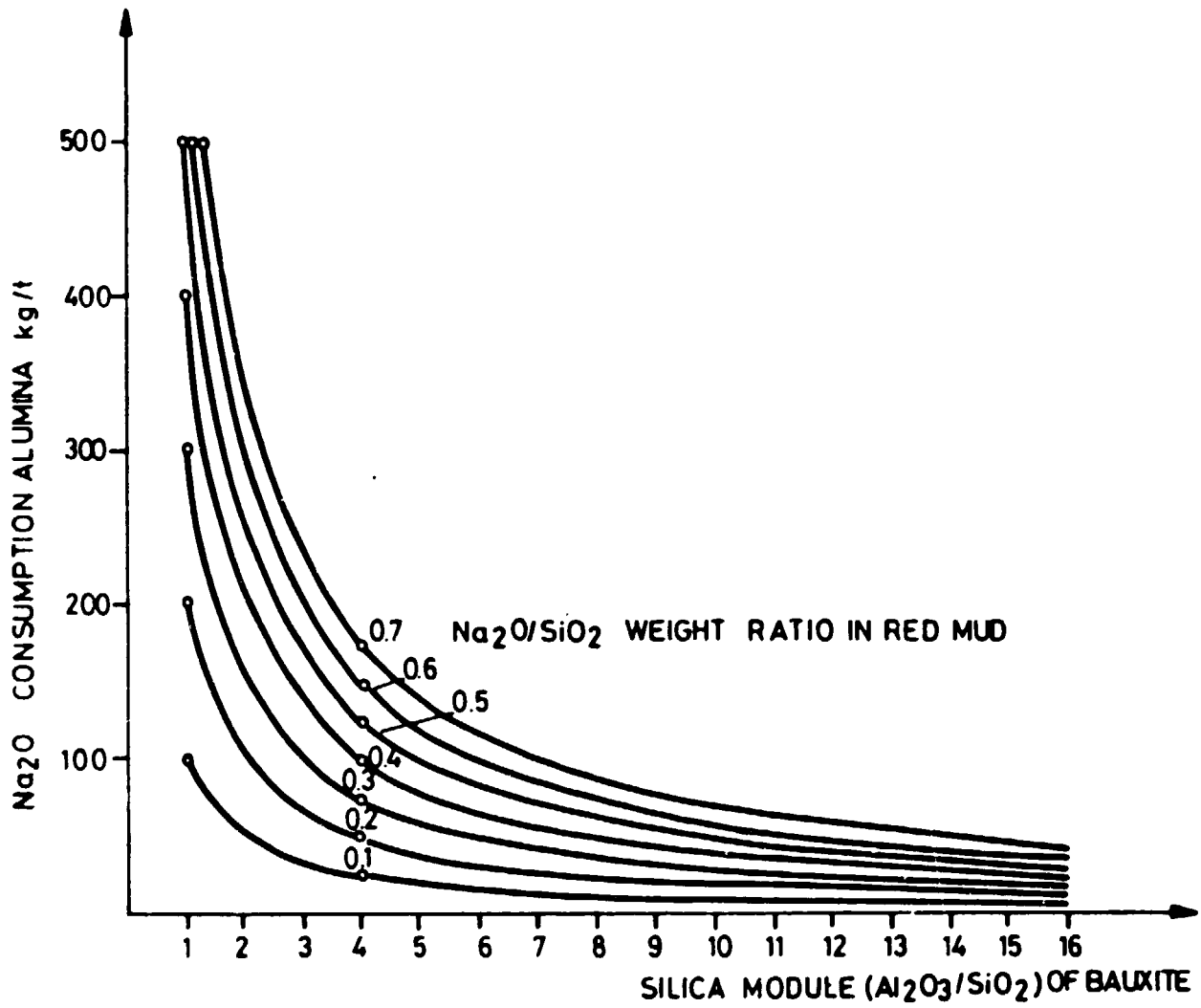


Fig. 6.12

CAUSTIC SODA LOSS BOUND IN MUD

ture, attainable molar ratio etc./ . Therefore, the digestion process is modelled by determining digestion characteristics under laboratory conditions.

Bauxite samples with different bauxite to digesting liquor ratios are digested at the temperature selected on the basis of phase analysis for a defined period of time, so that to reach with some samples to the equilibrium molar ratio /saturation of the solution with  $\text{Al}_2\text{O}_3$ /, after the digestion while with the other samples to reach to a higher final molar ratio, i.e. maximum alumina extraction attainable in practice. Digestion is carried out in steel autoclaves rotated in electrically heated, thermostated, temperature controlled oil, salt or air baths. Digestion characteristics are plotted from the results of 6 to 10 simultaneous tests. The working volume of the further experimental tasks. Digestion tests are complemented with preliminary and subsequent desilication. After digestion, aluminate liquor is separated from red mud by centrifuge. Two or three-stages washing of the mud with a solution containing 2 g  $\text{Na}_2\text{O}$  per litre or with distilled water, and centrifuging between stages are carried out.

Characteristic digestion diagrams of two Hungarian bauxite samples are shown in Fig. 6.13 a, b.

The distribution of the main components of bauxites in individual mineralogical phases are given in Table 6.4.

The results of the digestion tests show that the sample No. 11. can be digested at a temperature 200 °C with high recovery but the optimum parameters will be influenced by the values of caustic molar ratio at different temperatures. The sample No. 12. has only 1.9 %  $\text{Al}_2\text{O}_3$ , in form of boehmite, consequently a low temperature digestion can be suggested.



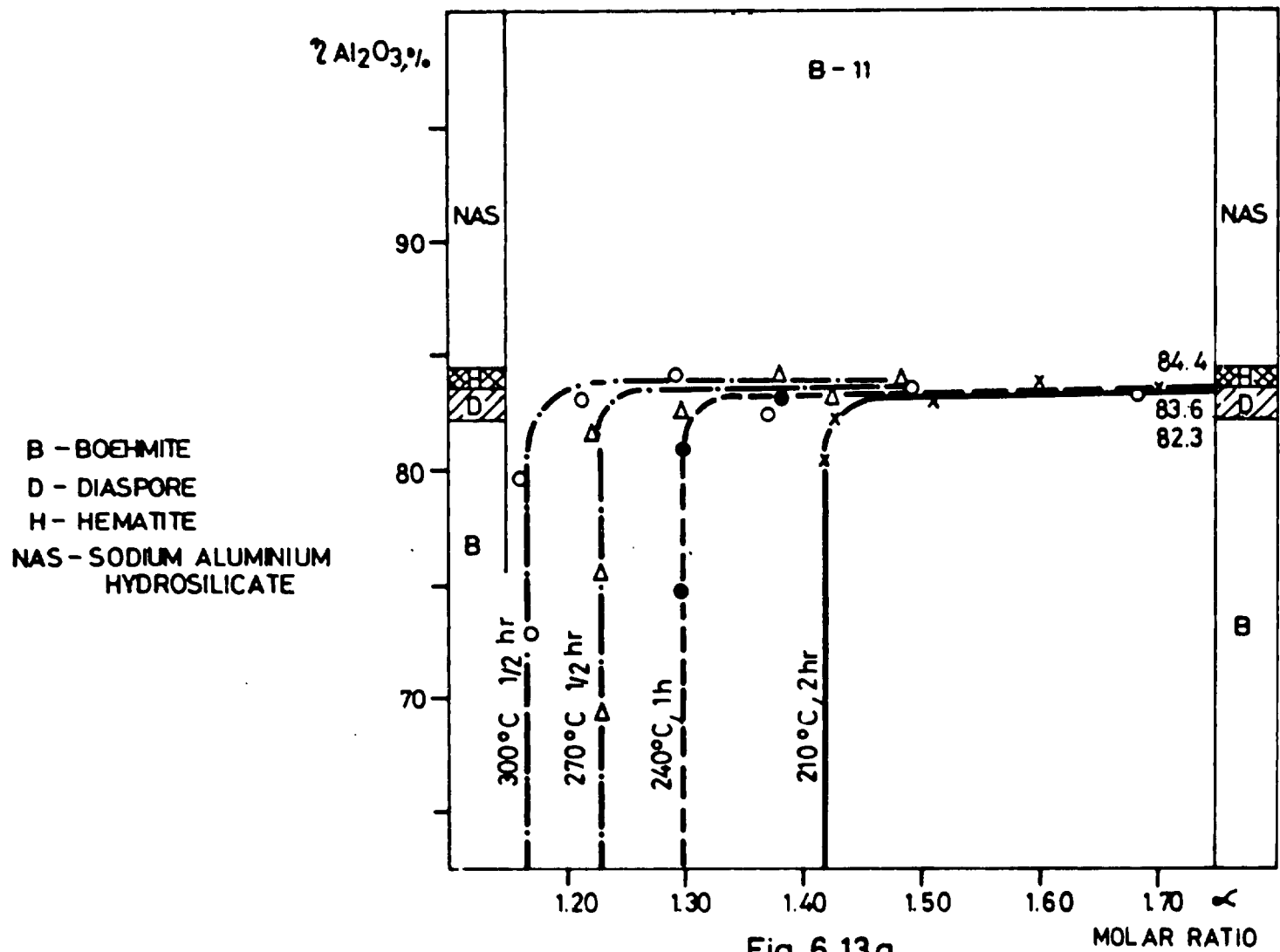


Fig. 6.13a  
 CHARACTERISTIC DIGESTION DIAGRAM OF BAUXITE E-11

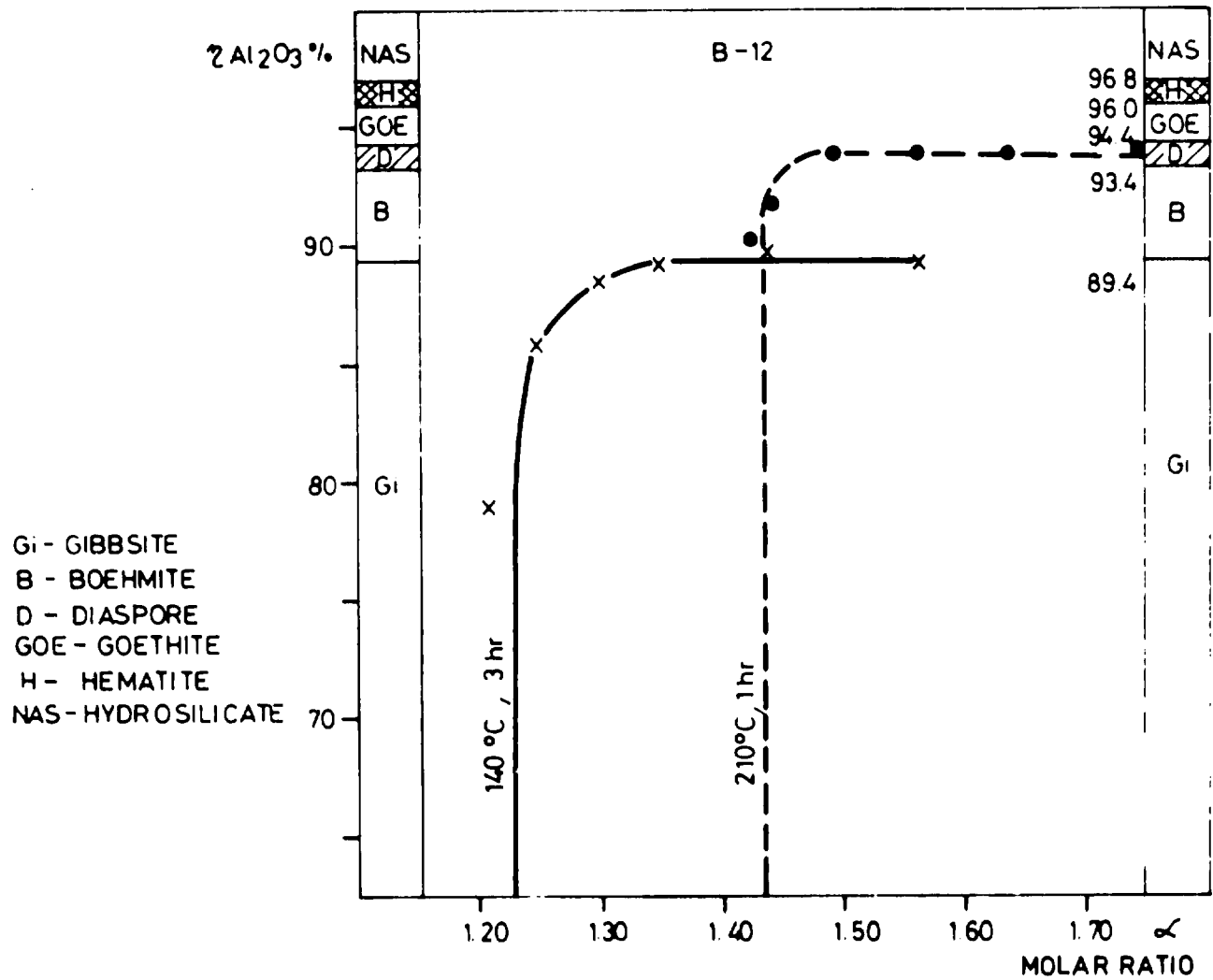


Fig.13 b

CHARACTERISTIC DIGESTION DIAGRAM OF BAUXITE B-12

Phase Composition of Hungarian Bauxites

Table 6.4.

Constituents	S a m p l e s	
	No. 11	No. 12
Al <sub>2</sub> O <sub>3</sub> in gibbsite in boehmite in diaspore in kaolinite in goethite in hematite	6.1	42.1
	35.6	1.9
	0.7	0.5
	7.9	1.5
	-	0.7
	0.4	0.4
Total	50.7	47.1
Fe <sub>2</sub> O <sub>3</sub> in hematite in goethite	21.3	16.3
	1.0	6.1
Total	22.3	22.4
SiO <sub>2</sub> in kaolinite	9.3	1.8
TiO <sub>2</sub> in anatase in nickite	1.7	1.3
	0.5	0.6
Total	2.2	1.9

The gibbsitic and boehmitic bauxites are processed usually without additives, the processing of diasporic and goethitic bauxites requires the addition of lime and other catalysts.

In this case some other parameters have great importance as well:

- determination of the optimal quantity of lime affecting the  $\text{Al}_2\text{O}_3$  recovery and NaOH consumption;
- kinetics of dissolution of  $\text{Al}_2\text{O}_3$  minerals;
- kinetics of goethite-hematite transformation in the presence of different quantity of different additives.

The traditional parameters /temperature, holding time, caustic concentration and equilibrium molar ratio of the liquor/ are to be determined in all cases as well.

To demonstrate some correlations, the following figures are shown: Fig. 6.14 demonstrates the effect of lime addition on the  $\text{Al}_2\text{O}_3$  recovery by processing diasporic bauxites /19/. The alumina recovery as a function of caustic weight ratio  $\text{Al}_2\text{O}_3$  gpl /  $\text{Na}_2\text{O}$  gpl at different digesting temperatures can be seen in Fig. 6.15 /19/.

The  $\text{Na}_2\text{O}/\text{SiO}_2$  weight ratio in red mud in the function of CaO addition /related to  $\text{SiO}_2$  in bauxite/ is shown in Fig. 6.16 measured by French authors /19/.

The digestion properties of a goethitic bauxite are demonstrated in Fig. 6.17 /20/ and Fig. 6.18 /21/. The same data are plotted in both figures but with different parameters /caustic molar ratio and A/C ratio/. The compositions of characteristic red muds are given in Table 6.5.

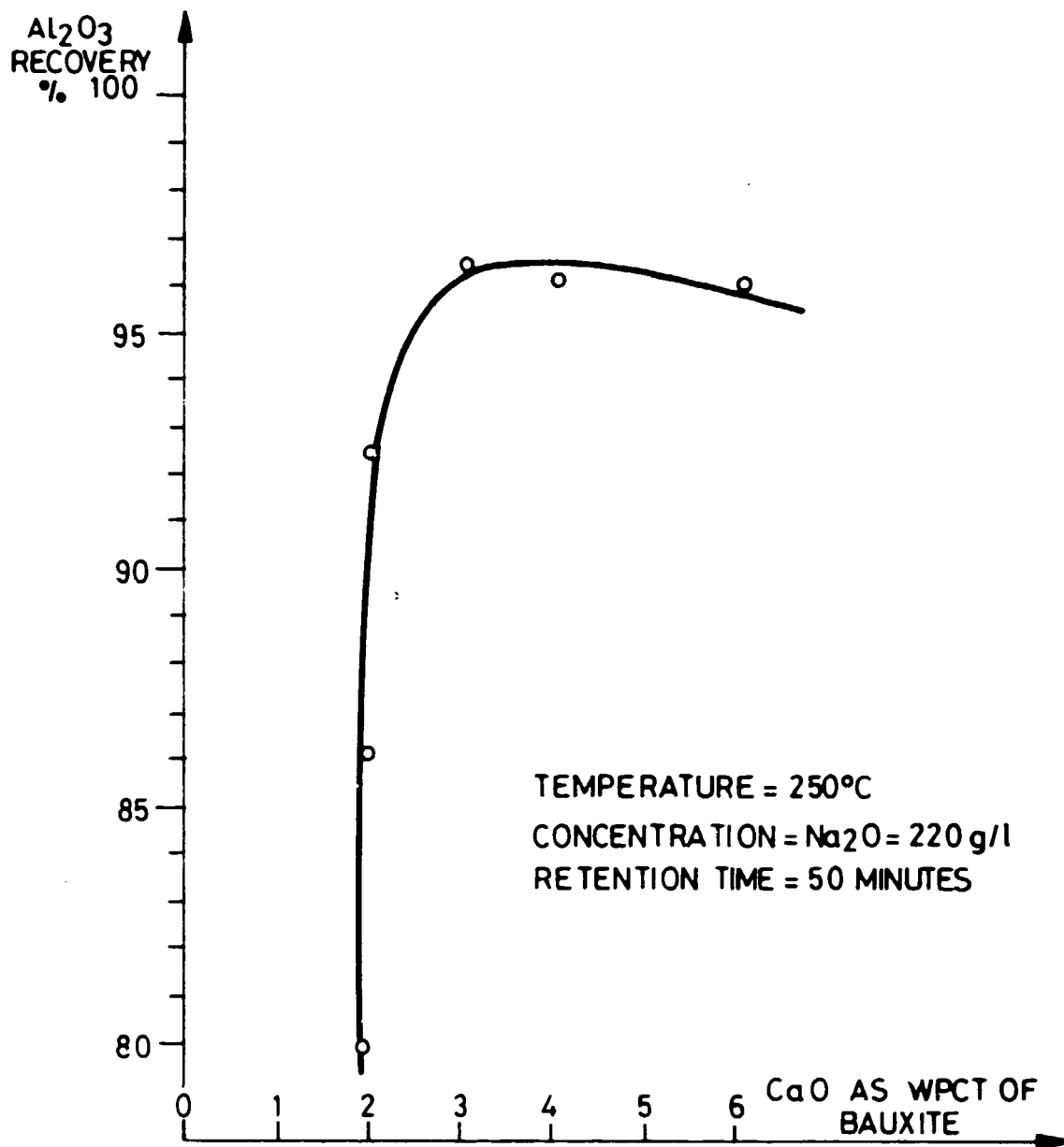


Fig. 6.14

EFFECT OF THE LIME ADDITION

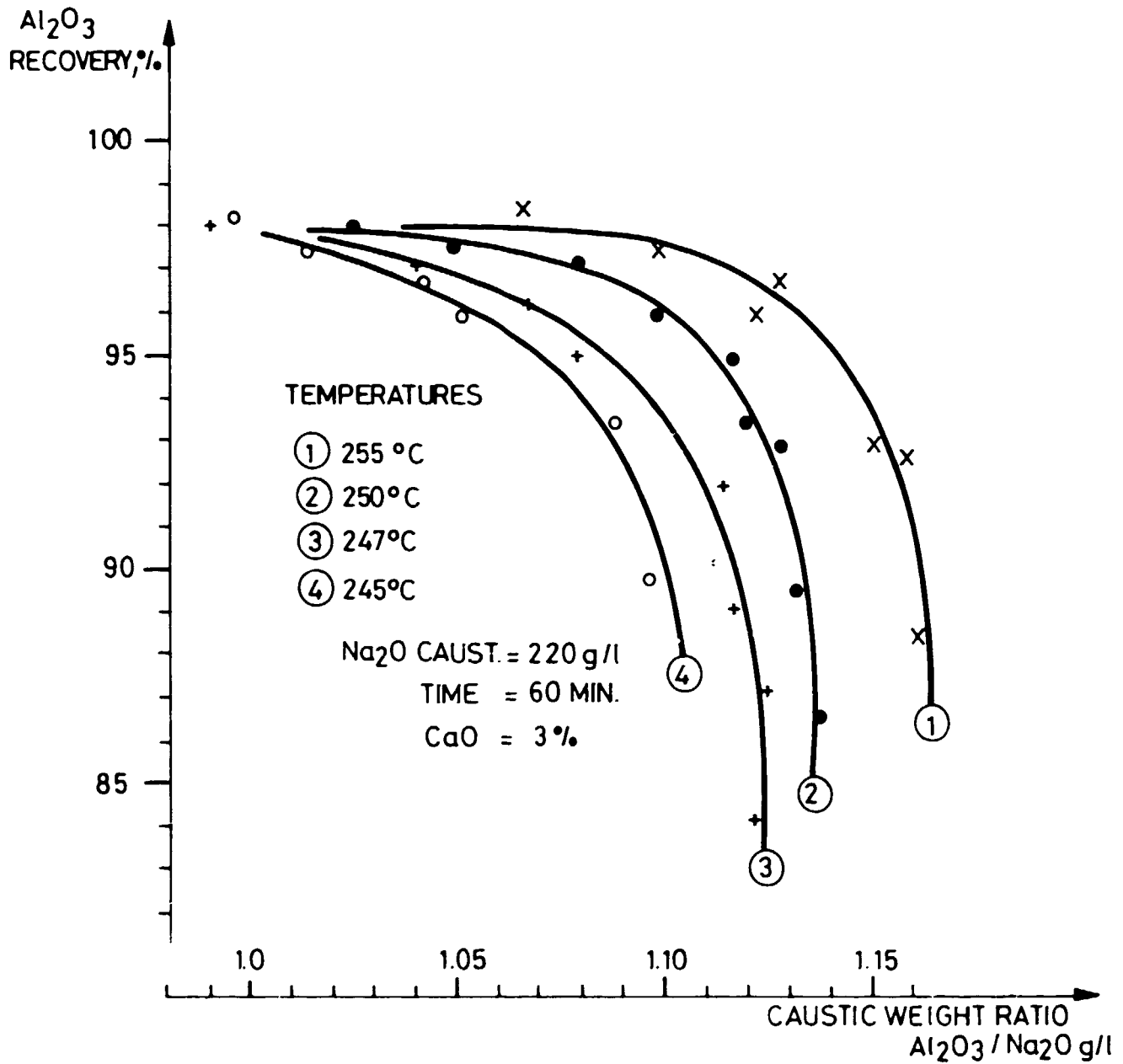


Fig. 6.15

Al<sub>2</sub>O<sub>3</sub> RECOVERY vs. CAUSTIC WEIGHT RATIO OF  
 THE LIQUOR AFTER DIGESTION

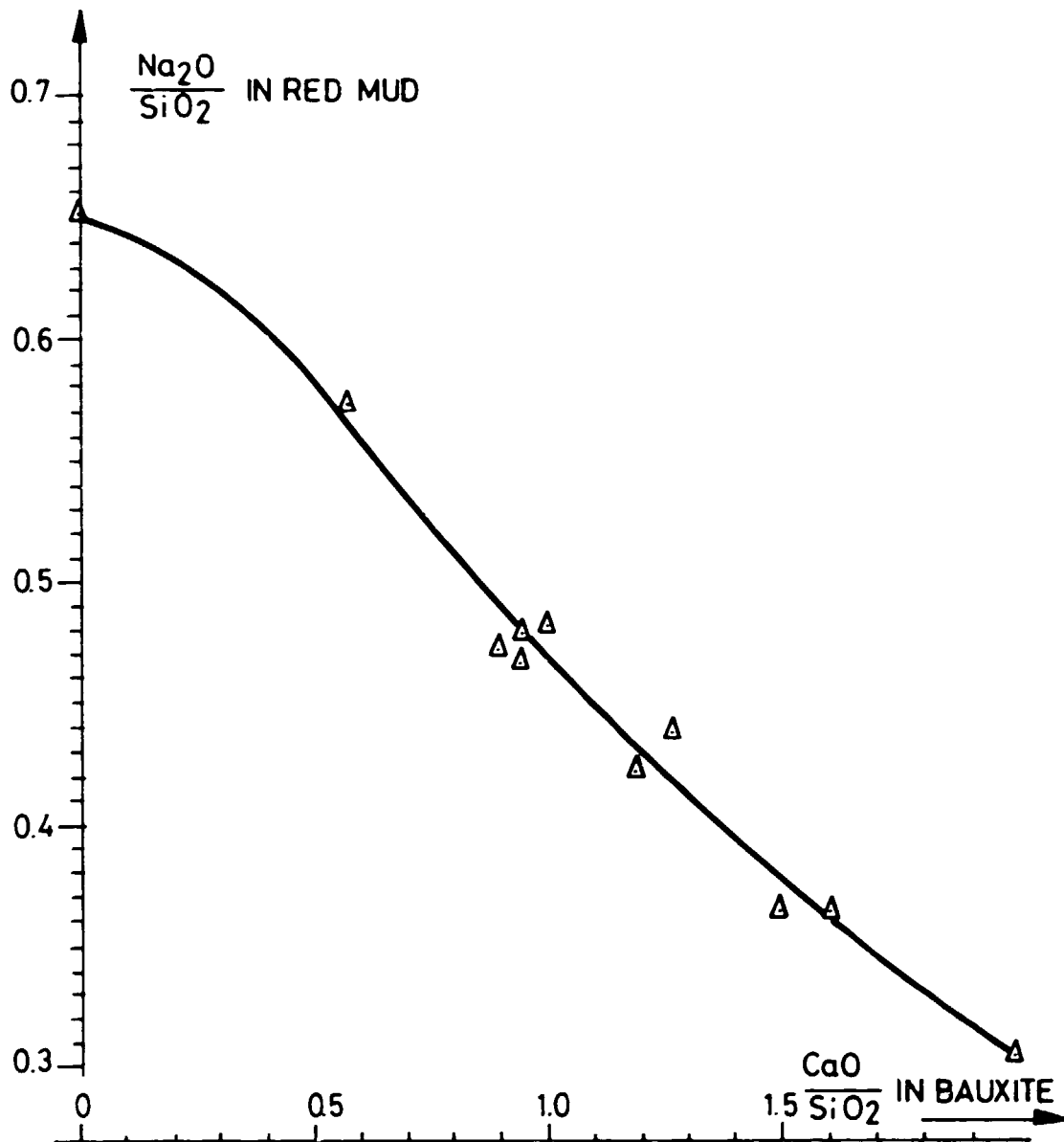


Fig. 6.16

$\text{Na}_2\text{O}/\text{SiO}_2$  WEIGHT RATIO IN RED MUD vs.  
ADDED  $\text{CaO}/\text{SiO}_2$  RATIO

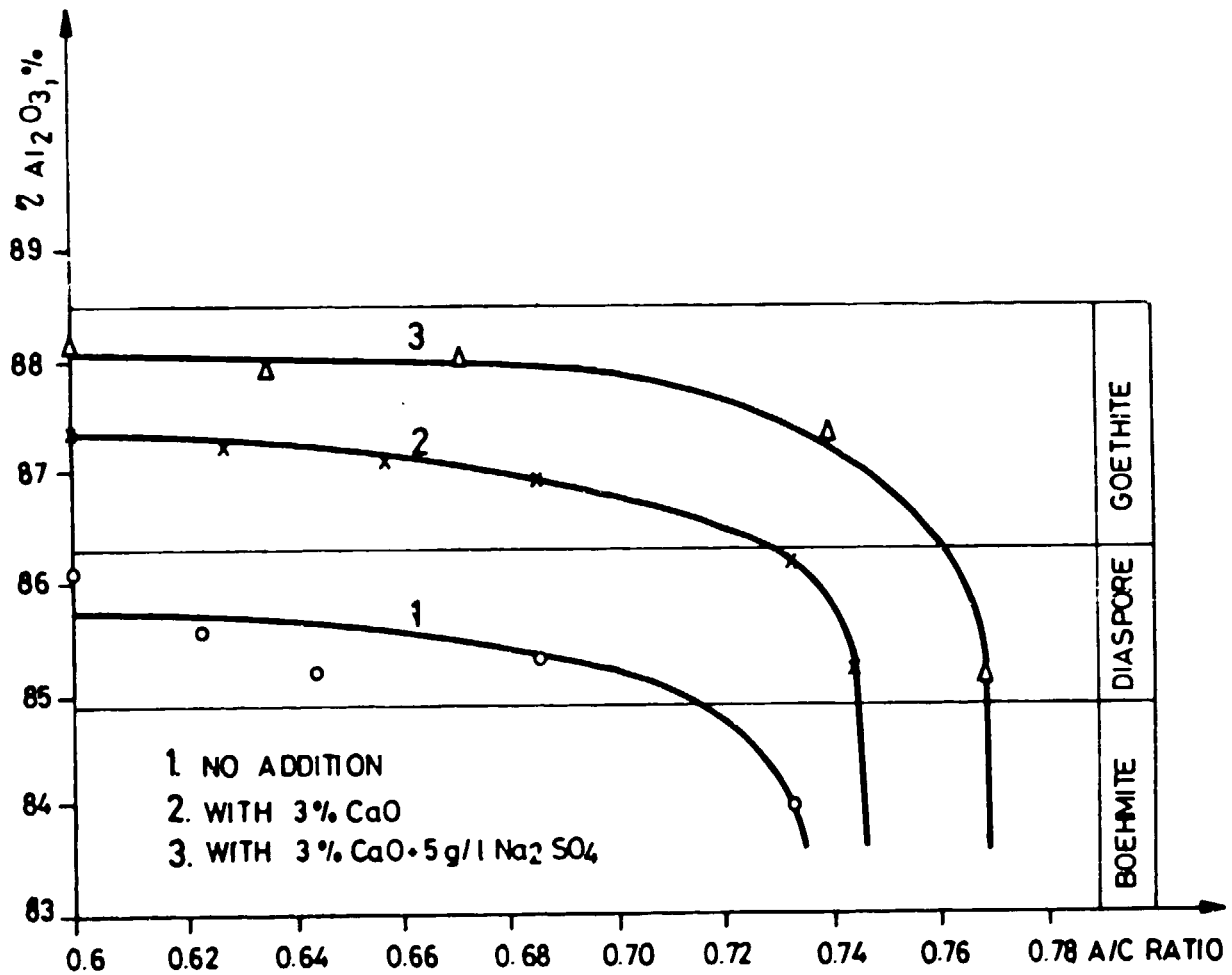
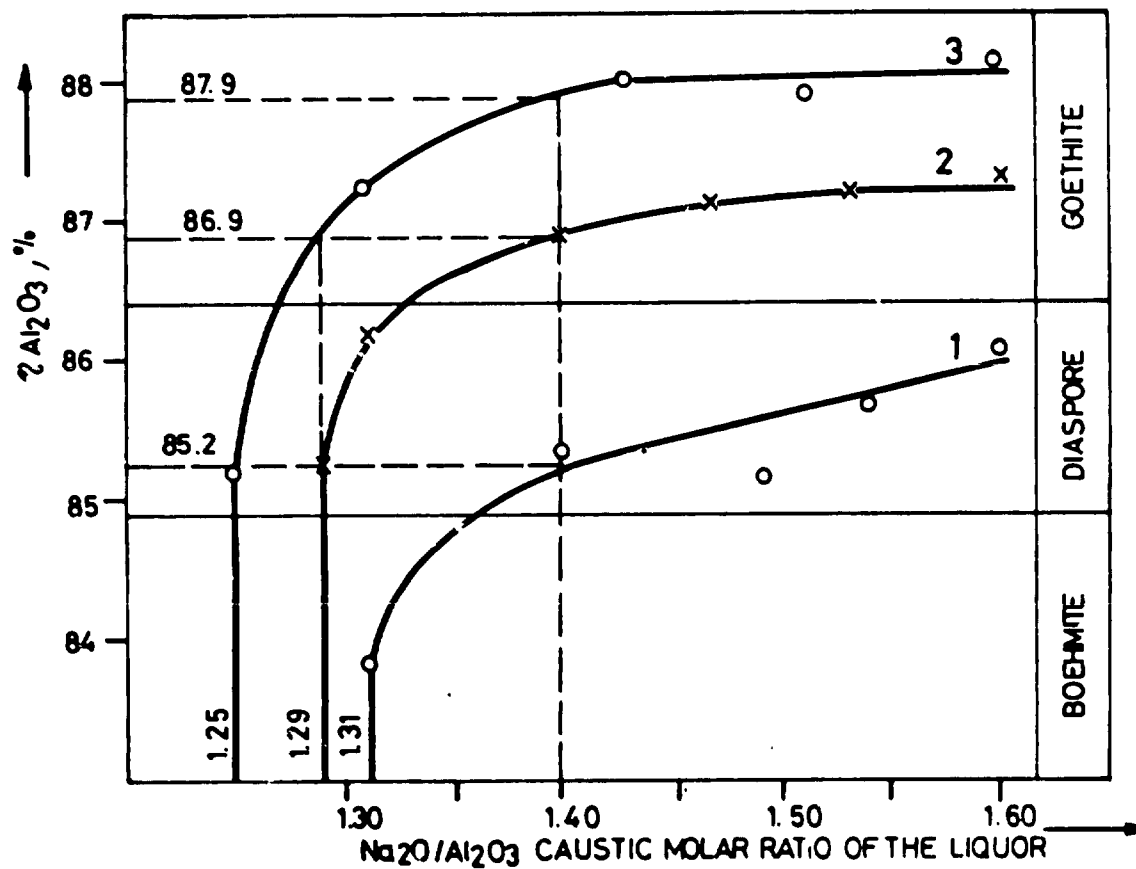


Fig.6.17

Al<sub>2</sub>O<sub>3</sub> RECOVERY PLOTTED vs. A/C RATIO OF DIGESTED LIQUOR





DIGESTION: 240°C, 60 MIN, Na<sub>2</sub>O<sub>CAUSTIC</sub> = 200 g/l

- 1. WITHOUT ADDITIVE
- 2. WITH 3% CaO
- 3. WITH 3% CaO + 5g/l Na<sub>2</sub>SO<sub>4</sub>

Fig. 6.18

VARIATION OF Al<sub>2</sub>O<sub>3</sub> RECOVERY vs. MOLAR RATIO OF LIQUOR (AFTER DIGESTION) IN LABORATORY PROCESSING OF GOETHITIC BAUXITE

Experiments for Transformation of Goethite into Hematite  
on Laboratory Scale. Digestion at 240 °C, 60 Minutes.  
Mineralogical Composition of the Formed Red Muds.

Table 6.5.

Constituent in phase	Digesting conditions		
	with 3 % CaO	with- out CaO	with 3 % CaO + 5 g/l Na <sub>2</sub> SO <sub>4</sub>
Al <sub>2</sub> O <sub>3</sub> %			
Boehmite	0	0.3	—
Gibbsite	0.3	0.5	0.2
3CaO · Al <sub>2</sub> O <sub>3</sub> · kSiO <sub>2</sub> · (6-2k)H <sub>2</sub> O	1.9	0.3	1.8
Sodalite	2.2	4.9	1.8
Cancrinite	4.0	6.7	4.3
Na <sub>2</sub> O · 8CaO · 3Al <sub>2</sub> O <sub>3</sub> · 10SiO <sub>2</sub>	1.8	trace	2.4
Ca(Mg, Al) — titanate	0.2	0.2	0.3
Goethite	—	3.2	—
CaO · 2Al <sub>2</sub> O <sub>3</sub> · 2SiO <sub>2</sub> · H <sub>2</sub> O	1.8	trace	1.7
Mg <sub>3</sub> Al <sub>2</sub> (OH) <sub>6</sub> · 4H <sub>2</sub> O	0.1	0.1	trace
Mg <sub>3</sub> Al <sub>2</sub> (Al <sub>2</sub> SiO <sub>5</sub> ) <sub>2</sub> (OH) <sub>6</sub>	0.2	0.1	0.1
Hematite	1.0	0.4	1.0
Total:	13.5	16.7	13.6
SiO <sub>2</sub> %			
3CaO · Al <sub>2</sub> O <sub>3</sub> · kSiO <sub>2</sub> · (6-2k)H <sub>2</sub> O	0.5	0.1	0.5
Na <sub>2</sub> O · 8CaO · 3Al <sub>2</sub> O <sub>3</sub> · 10SiO <sub>2</sub>	3.6	—	4.8
Sodalite	2.5	5.4	2.0
CaO · 2Al <sub>2</sub> O <sub>3</sub> · 2SiO <sub>2</sub> · H <sub>2</sub> O	1.1	—	1.0
Cancrinite	4.3	7.3	4.5
Total:	12.0	12.8	12.8
Fe <sub>2</sub> O <sub>3</sub> %			
Goethite	1.8	18.2	1.8
Hematite	37.6	23.9	41.0
Maghemite	—	2.1	0
Total:	39.4	44.2	42.8
TiO <sub>2</sub> %			
Na <sub>2</sub> O · 3TiO <sub>2</sub>	3.2	4.4	3.8
Na <sub>2</sub> O · TiO <sub>2</sub>	1.6	1.0	0.8
CaTiO <sub>3</sub>	0.6	0.3	0.8
Ca(Mg, Al) — titanate	0.2	0.2	0.3
CaO %			
CaTiO <sub>3</sub>	0.4	0.2	0.6
Na <sub>2</sub> O · 8CaO · 3Al <sub>2</sub> O <sub>3</sub> · 10SiO <sub>2</sub>	2.9	—	3.6
Dolomite	—	—	—
CaO · 2Al <sub>2</sub> O <sub>3</sub> · 2SiO <sub>2</sub> · H <sub>2</sub> O	0.3	—	0.3
Calcite	0.4	0.1	—
3CaO · Al <sub>2</sub> O <sub>3</sub> · kSiO <sub>2</sub> · (6-2k)H <sub>2</sub> O	3.1	0.5	2.9
Ca(Mg, Al) titanate	0.1	0.1	0.2
Total:	7.2	0.9	7.6
η Al <sub>2</sub> O <sub>3</sub> % (Alumina yield)	87.2	85.9	88.1
Na <sub>2</sub> O %	6.5	7.9	7.2
L. O. I. %	8.1	7.9	4.9
Na <sub>2</sub> O/SiO <sub>2</sub> molar ratio	0.52	0.60	0.53

The kinetics of the goethite-hematite transformation in the presence of  $Mn^{2+}$  catalyst during digestion of Hungarian Iszkaszentgyörgy bauxite is shown in Fig. 6.19 /22/.

The phase compositions of red muds obtained from Yugoslav Obrovac bauxite and Hungarian Iszkaszentgyörgy bauxite after digestion without and with catalysts are demonstrated in Fig. 6.20 /22/.

#### Processing of Low-Grade Bauxites by Bayer Technology

Sometimes it is possible to find a proper modification of the Bayer technology suitable for processing low-grade bauxites /23/ containing silica in a non-reactive form under certain digestion parameters.

Fig. 6.21 shows digestion graphs of a tropical bauxite, containing 40.1 per cent  $Al_2O_3$ , 6.9 per cent  $SiO_2$  and 27.8 per cent  $Fe_2O_3$ , digested with a liquor containing 150 gpl  $Na_2O$ , at 150 and 240 °C, respectively. Out of the 6.9 %  $SiO_2$  content 5.1 % was in quartz form and 1.8 % in kaolinite. Digesting thus bauxite at 150 °C, the  $Na_2O$  content of red mud was not higher than 2.4 per cent and the  $Al_2O_3$  recovery was about 92 %. Consequently, this ore is suitable for processing by the American Bayer technology. However, digesting this bauxite at 240 °C quartz becomes reactive; this bauxite is practically worthless for a plant using high temperature digestion.

Fig. 6.22 shows digestion curves of a bauxite containing chamosite, digested at 240 °C. This bauxite contains 46.3 per cent alumina /of which 0.8 per cent is in hoehmite, 35.2 per cent in diaspore, 2.8 per cent in kaolinite, 0.4 per cent in illite and 7 per cent in chamosite/, 8.2 per cent  $SiO_2$  /of which 3.3 per cent in kaolinite, 0.8 per cent in illite, and 4.1 per cent in chamosite/, and 25.7 per cent  $Fe_2O_3$  /16.8 per cent in hematite, 9.8 per cent in chamosite/. Chamosite did not react with the digestion liquor, so that soda loss was

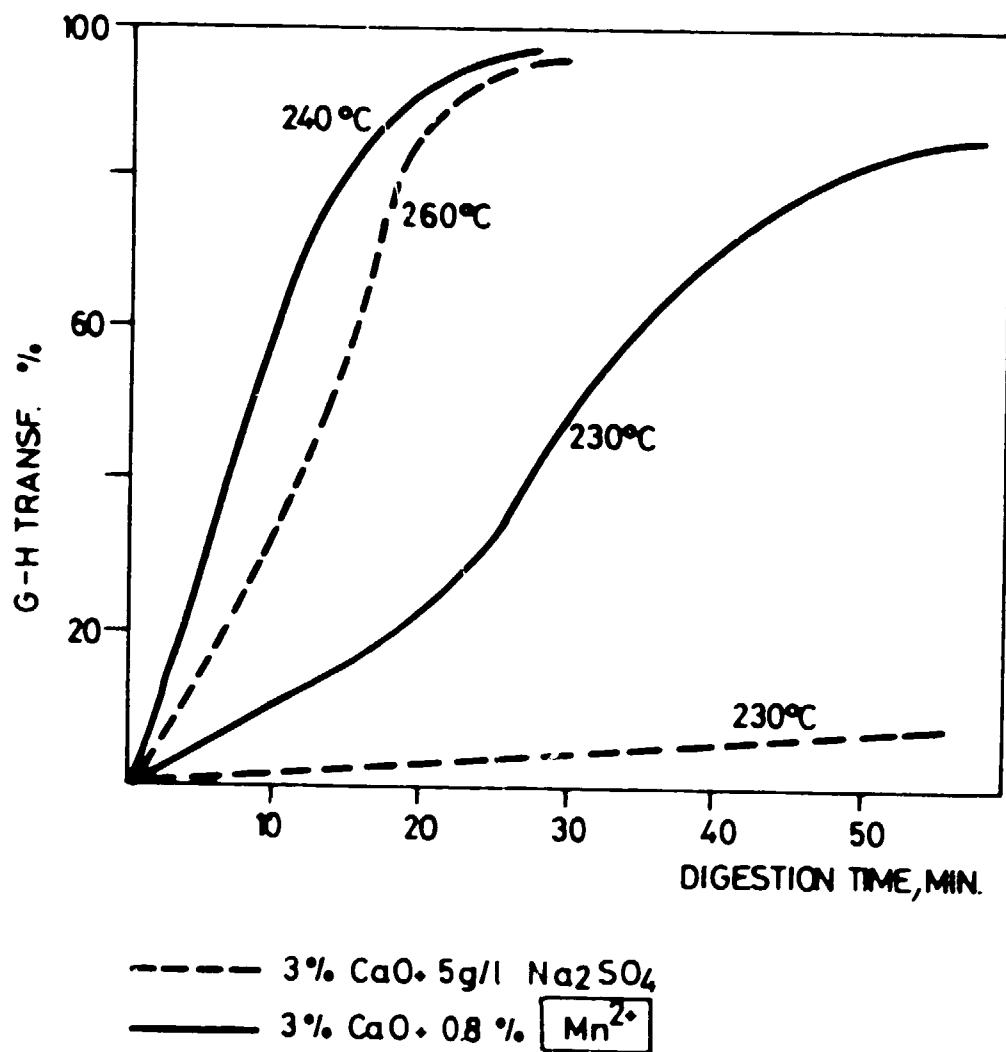
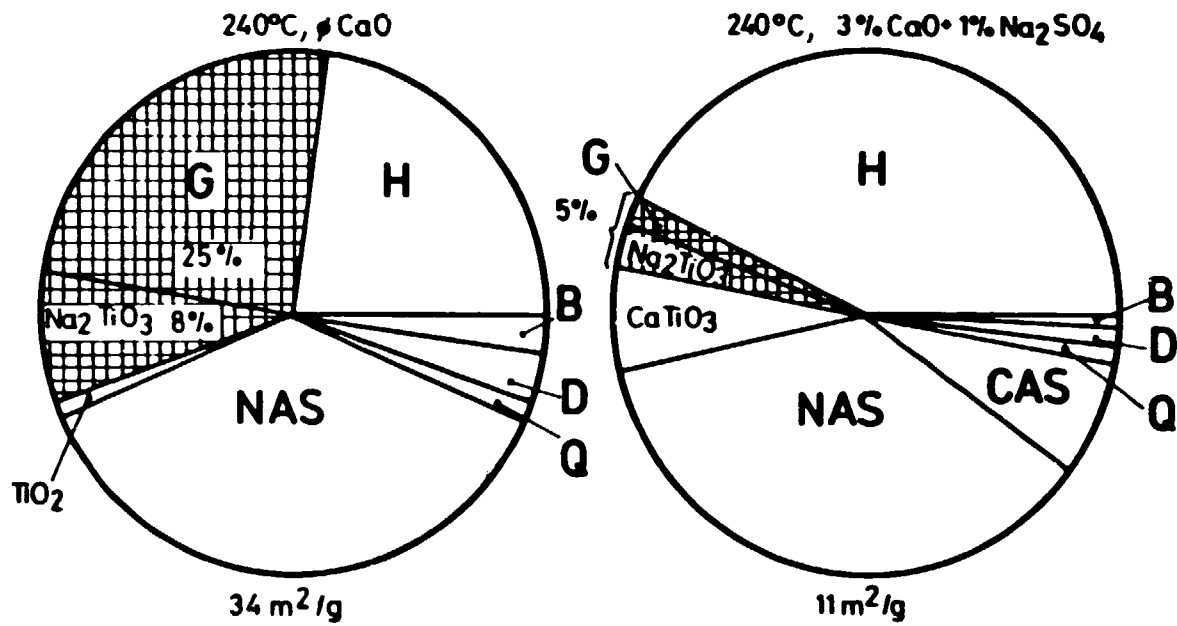


Fig. 6.19

GOETHITE-HEMATITE TRANSFORMATION vs.  
DIGESTION TIME USING CATALYSTS

YUGOSLAVIAN BAUXITE, OBROVAC



HUNGARIAN BAUXITE, ISZKASZENTGYÖRGY

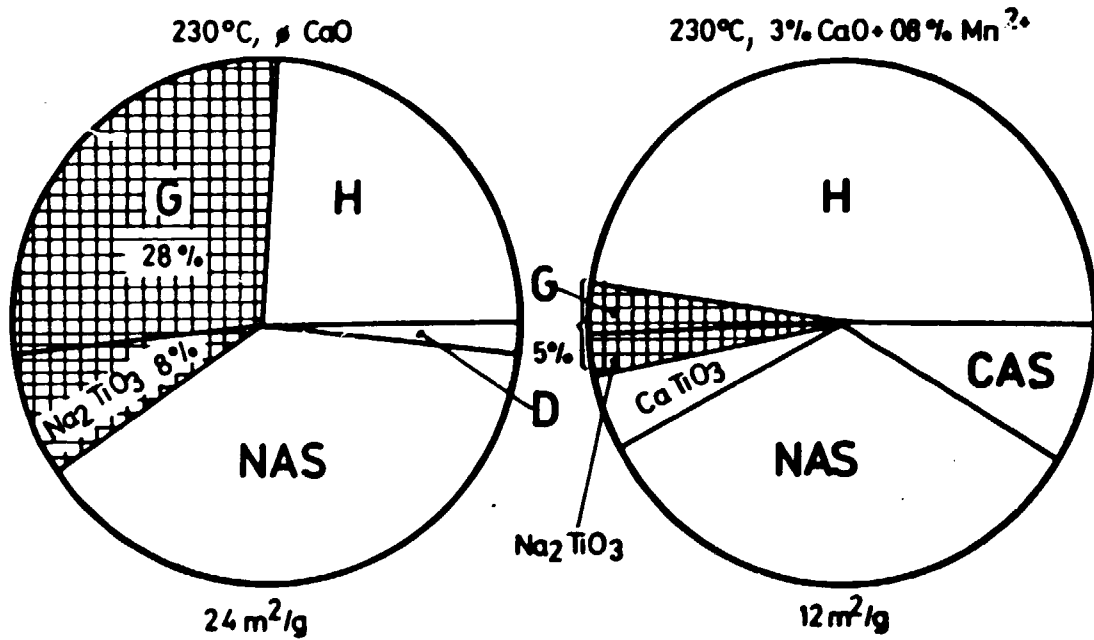


Fig. 6. 20

PHASE COMPOSITION OF RED MUDS AFTER DIGESTION WITHOUT AND WITH CATALYSTS

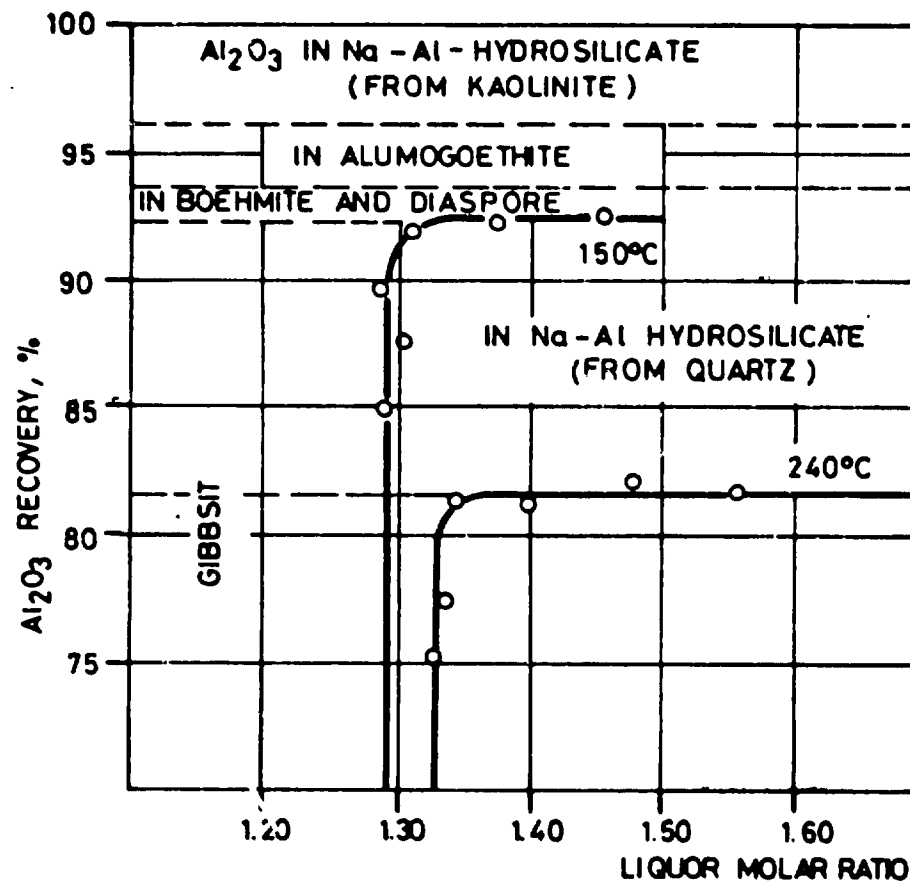


Fig. 6.21

DIGESTION GRAPHS OF A GIBBSITIC TROPICAL BAUXITE CONTAINING QUARTZ AT TEMPERATURES OF 150 AND 240°C. COMPOSITION OF DIGESTION LIQUOR: Na<sub>2</sub>O<sub>c</sub> = 150gpl; MOLAR RATIO: 4

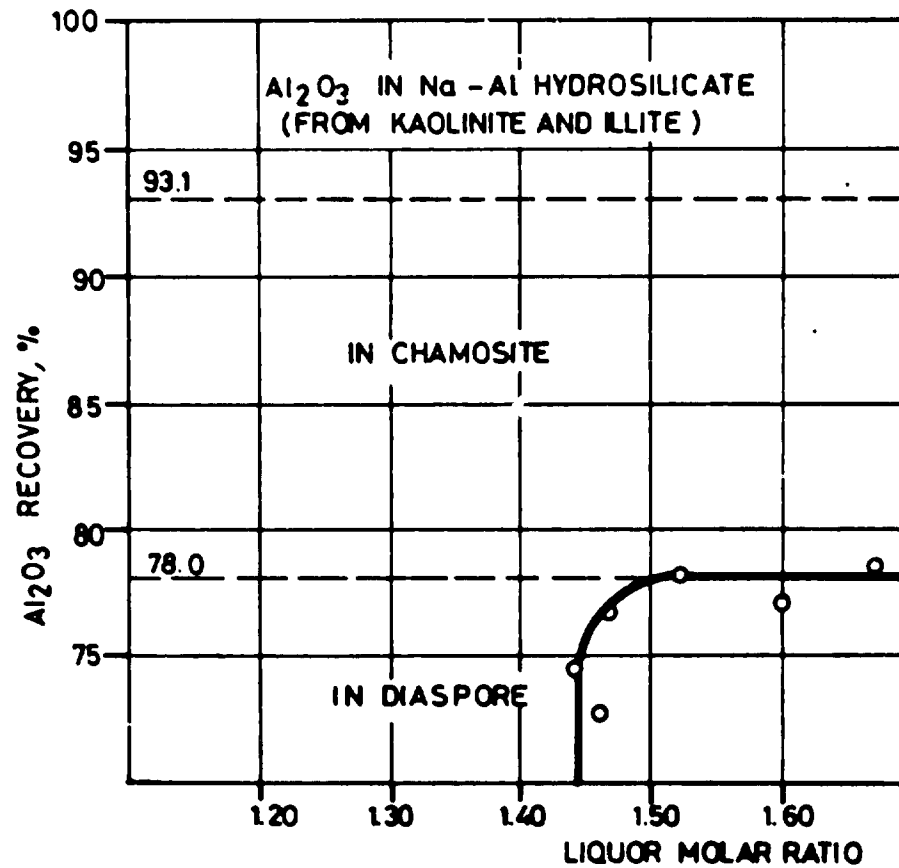


Fig.6.22

DIGESTION CHARACTERISTIC OF CHAMOSITIC, DIASPORIC BAUXITE AT 240°C  
 DIGESTION LIQUOR: Na<sub>2</sub>O<sub>c</sub> = 240 gpl, MOLAR RATIO: 4, CaO ADDITION: 3%

much lower than expected. Consequently an  $\text{Al}_2\text{O}_3$  recovery of 78 per cent associated with a low soda consumption makes this bauxite acceptable for Bayer plant feed. /4.9 %  $\text{Na}_2\text{O}_c$  and 12.2 %  $\text{SiO}_2$  was found in red mud/

Determination of the Recommended Technological Parameters of Digestion

Summarizing the results of the digestion tests and taking into account the varying grade of bauxite to be processed the parameters to be recommended will be determined. Sometimes some variants of digestion technology are given to the designers to optimize the parameters by means of computer programs. The following data are necessary for a routine calculation:

Digestion liquor: caust.  $\text{Na}_2\text{O}$  gpl  
 Digestion temperature,  $^{\circ}\text{C}$   
 Holding time, hours  
 Caustic molar ratio after digestion  
 CaO addition /referred to dry bauxite/  
 Expected alumina recovery  
 Expected  $\text{Na}_2\text{O}/\text{SiO}_2$  ratio in red mud  
 Grade of goethite-hematite transformation, %  
 Quality, quantity and method of the preparation of the catalyst to accelerate the goethite-hematite conversion  
 Digestibility of various contaminants of bauxite and methods for eliminating their detrimental effect

DETERMINATION OF THE SETTLING PROPERTIES OF RED MUD;  
 USE OF FLOCCULANTS

Besides the liquid phase composition the settling properties of red mud depend principally on the size and shape of the red mud particles.

Instead of the original small particles the solid phase of a red mud slurry after digestion contains much bigger ag-



gregates of particles stuck together. In order to determine the elementary particle size the slurry is to be decanted, and the particles stuck together have to be comminuted in a mortar. The particle size can be measured with an optical or electron-microscope. Investigations show that besides insoluble quartz particles retaining the particle size of the original ground bauxite the solid phase of red mud consists of particles sized below 1  $\mu\text{m}$  in diameter.

Building of aggregates proceeds already during the digestion because of the frequent interaction among particles in the thick suspension. The relatively strong adhesion of the particles in the aggregate is ensured by the free electrical charge of crystal surface of the particles, and by the chemical adhesive power. Building of aggregates results in a poly-dispersive suspension of about 10  $\mu\text{m}$  average particle size.

The problem of determining the size of aggregates is that their size may decrease during the preparation. The real size of aggregates can be deduced from the settling properties observed in the original solution.

The aggregates break in pieces if exposed to a force greater than the adhesive force among the particles. This happens during the flashing and as an effect of viscous shear forces brought about by intensive stirring or adjusting of slurry by pumping. In case these effects come to an end the aggregates will build up again after a short or long induction period.

The aggregates contain liquor, sealed between the primary particles of red mud or adhered in form of a solvate cover to the surface of particles. Consequently the specific weight of aggregates is smaller than that of the solid phase.

Natural and synthetic flocculants are used to accelerate settling by joining together the aggregates of primary grains of the red mud to flocs of much greater size.

Electrochemical effects, ionic bonds between the interfaces of solid phase and the macromolecules of flocculant and the hydrogen bond are playing role in the formation of flocs. The density of ions on the surface of the solvated phase and the sign of these ions is determined principally by the chemical composition of the liquid phase. Consequently, the desired degree of flocculation can be realized most economically in different cases, like in different stages of the washer series, by macromolecules of different ionic charges.

The average diameter of the relatively loose flocs is 10-20 times bigger their solvation 2-3 times thicker, than that of the aggregates. So the specific weight of flocs is even less than that of the aggregates, nevertheless their settling velocity is higher owing to their larger size.

The bond in the flocs is weaker than in the aggregates, so they are more sensitive to shearing effects. The size of the fragments originated from the flocs by shearing, approximately corresponds to the size of the aggregates the flocs were built of. This type of transformation in the floc-structure results in the release of a part of the liquid bound to the floc. Breaking up of the flocs into fragments during the settling promotes the compression of the mud.

It can be observed at repeated settling tests with repulped red muds obtained after settling with flocculant addition, that their settling velocity is considerably less than that of the freshly flocculated slurries. The reason is the irreversible breakage of flocs. The natural and synthetic longchained macromolecules can not be regenerated fully after breakage. This effect is particularly considerable in case of synthetic flocculants.

### Settling Test Methods

The solid and liquid phases in settlers are separated based on the difference between their specific weights under the effect of gravitation. Besides the problem of the scale the laboratory modelling of the continuously operating settlers encounters other difficulties, too. Aiming at continuous operation of the settler, the red mud would have to be produced by a continuously operating laboratory digester, the flocculant would have to be added at constant rate, and a steady underflow of the settler would have to be ensured in order to reach the slowly settling equilibrium state of operation.

Transfer of data of a continuously operating small scale model to a plant size settler would have to be solved mathematically, too.

Therefore, the settling tests have been carried out in a static settling tube. The homogenized slurry is poured into the tube at the start of the test and the settling and compression behaviour of the solid phase of slurry is measured without further feed or discharge.

It can be calculated theoretically and proved with tests that in very thin suspensions where the solid species are located far enough from each-other and so exert no interaction, they sink with different speeds according to their size, shape and specific weight. The settling velocity of the ball-shaped particles can be described with the Stokes' formula:

$$u_{St} = \frac{10^4 d^2 (\rho_s - \rho_l) g}{18 \eta}$$

where:  $d$  = diameter of the ball, m  
 $\rho_s$  = specific density of the solid phase,  $\text{kg/m}^3$   
 $\rho_l$  = specific density of the liquid phase,  $\text{kg/m}^3$   
 $\eta$  = viscosity of liquid, Pa s  
 $U_{St}$  = sinking velocity of particle relatively to liquid  
 /Stokes velocity/, m/s  
 $g$  = gravitational factor,  $\text{m/s}^2$

In case of flocs, instead of the particle diameter  $d$ , the average Stokes diameter  $D$  of flocs considered to have ball shape has to be used and instead of the solid phase density  $\rho_s$ , the effective density of flocs ought to be used. Therefore, a solvation factor  $\alpha$  is introduced:

$$\alpha = \frac{V}{v}$$

where:  $V$  = the volume of the floc  
 $v$  = the total volume of the solid particles in the floc

The Stokes' velocity formula will read in that case /24/ as follows:

$$U_{St} = \frac{10^4 D^2 / \rho_s - \rho_l / g}{18 \alpha \cdot \eta}$$

The velocity of falling of particles in a thick suspension is considerably less than the Stokes velocity because of the frequent interaction between the particles /hindered settling/. If the solid concentration is high enough, segregation will

also cease, and the small particles are drifted downwards, together with the big ones. That is called collective settling.

A characteristic feature of the collective settling is that a very thin transition layer can be observed between the clear zone and the settling mud zone, i.e. the mud level is sharp. In case of red mud slurries collective settling is observed above 30 gpl solids content already, with a settling velocity depending on the solids concentration.

#### *Settling Test Using Settling Series*

This is the traditional method of settling tests. The slurry to be tested is poured into a settling cylinder of 30-40 cm height and 2-3 cm diameter.

Slurries of different solids concentration or treated by different flocculants are poured into each tube and allowed to settle by gravitation.

The sinking velocity of the mud level in the cylinders can be determined in the function of time by reading the mud level regularly off. Plotting the data in a settling diagram /H-t diagram/ three characteristic types of curves can generally be obtained, which are shown in Figures 6.23 a, b, c /25/.

The settling diagram shown in Fig. 6.23 a. is the most common, in which the starting period of settling is described by a line with a slope depending on the starting solids concentration and from a point P a curve is obtained with a slope decreasing first quickly and then slowly until it becomes horizontal in the fully settled state.

The slope of the tangent of the H-t diagram gives the settling velocity at any time. So in case of curves type 1.a, the velocity is constant till a certain period /till point P in our figure/ and then it decreases continuously.

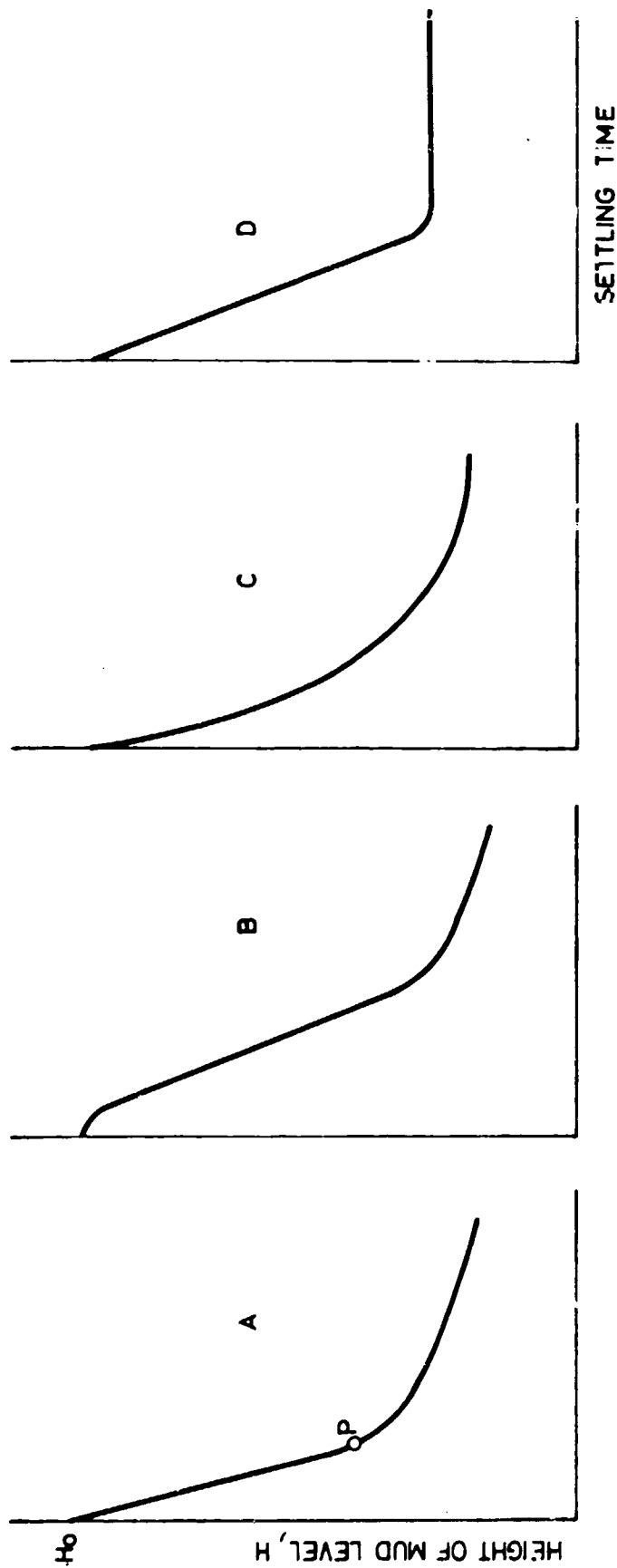


Fig. 6. 23  
SETTLING OF RED MUDDS OF DIFFERENT TYPE BAUXITES

The initial settling velocity  $/u_0/$  depends on the initial solids concentration of the homogeneous slurry filled in. The relationship can be determined by carrying out measurements with series of tubes filled with slurries of different solids concentration. It is characteristic that the thicker the suspension the smaller is the settling velocity. The part of the curve in Fig. 6.23 1.a after the point P showing a decreasing velocity indicates that as the settling proceeds even the solids concentration in the mud level becomes much higher than the initial solids concentration  $/c_0/$ .

The initial settling velocity  $/u_0/$  is the slope of the starting period of the curve /see Fig. 6.23 a./

$$U_0 = \frac{H_0 - H_1}{t_1}$$

It is convenient to characterize the compressibility of different red mud samples by the average mud compression - i.e. the ratio of the total solids content of the mud phase and the volume of the mud zone - developed during a selected  $t'$  period /e.g. 30 or 60 min./. The average compression attained in  $t'$  minutes can be expressed by the initial  $c_0$  and  $H_0$  and b., the decreased height of the mud level  $H'$  in  $t'$  minutes, as follows:

$$c_{H'} = c_0 \frac{H_0}{H'}$$

Curves plotted in Fig. 6.23 b. differ from the previous ones in that the linear period of the H-t diagram starts only after a certain induction period  $/t_i/$ . Flocs characteristic of the given suspension are formed in the initial slowly settling mud during the induction period. The settling velocity, calcu-

lated from the slope of the linear period, can not be ordered unequivocally to the known initial solids concentration because there is no evidence about, that there was no compression in the mud level zone during the induction period or that no thinning due to segregation took place.

No initial linear period is found in the diagram Fig. 6.23 c. indicating that the settling velocity of the mud level is different ~~is~~ every moment. That unequivocally shows that the slurry is compressed continuously in its whole volume, so there are no separate settling and compression zones. Curves of that type belong usually to properly flocculated muds in case their initial solid concentration is high enough.

The H-t diagram in Fig. 6.23 d. having first a linear slope and, after a short transition, a horizontal section, is a characteristic settling curve of ideal suspensions containing solid particles of the same size. The unique particles fall on the bottom of the settling cylinder or on the layer of the earlier arrived particles with a constant velocity depending on the initial solids concentration and there they stop moving. In the bottom of the cylinder immediately a layer of such a solids concentration is formed in which the space filling is optimum, contrary to the real suspensions in which the compression takes place slowly during releasing liquor.

#### *Soft Gamma-Ray Absorption Model-Settler /SAM/*

##### Testing Apparatus

As compared to the simple settling cylinder tests far more information can be obtained from the tests where the distribution of local solids concentration of a compacting zone and its changes vs. time can be determined. The local solids concentration can be measured without disturbing the settling process by determining the absorption of a ray of great penetration.



The SAM-test is based on that the intensity of the ray transmitted through a slurry-column of a settling tube radiated through by a narrow ray-beam is the exponential function of the solids concentration, supposing that the composition and temperature of the liquor phase is constant. The law of the radiation is the following:

$$I = I_0 \exp[-a.c/]$$

where:  $I_0$  = intensity of radiation after transition of the clear liquor phase, pps

$I$  = intensity of radiation after transition of the slurry of  $c$  /gpl/ solids concentration, pps

$a$  = absorption coefficient of radiation, lpg

The test slurry is poured into a thermostated vertical settling tube situated between a beam source and a detector mounted on a lifting equipment. During the time-programmed rising of the lift a narrow beam scans over the slurry column.

The frequency of the detected signals depends on the solids concentration found at the given height. It is measured and registered in function of the vertical position of the lift by an electronic measurement system. The SAM test is drawing concentration profiles in the above way during 24 hours' or longer settling time.

Fig. 6.24 shows a characteristic series of such concentration profiles.

Curve 1. was registered at the start of settling ( $t_1=0$ ). The vertical line from the 0 level to  $H_1$  shows that the initial solids concentration was  $c_0$  in the whole cylinder. At  $H_1$  mud level the solids concentration shows a sudden decrease to zero.

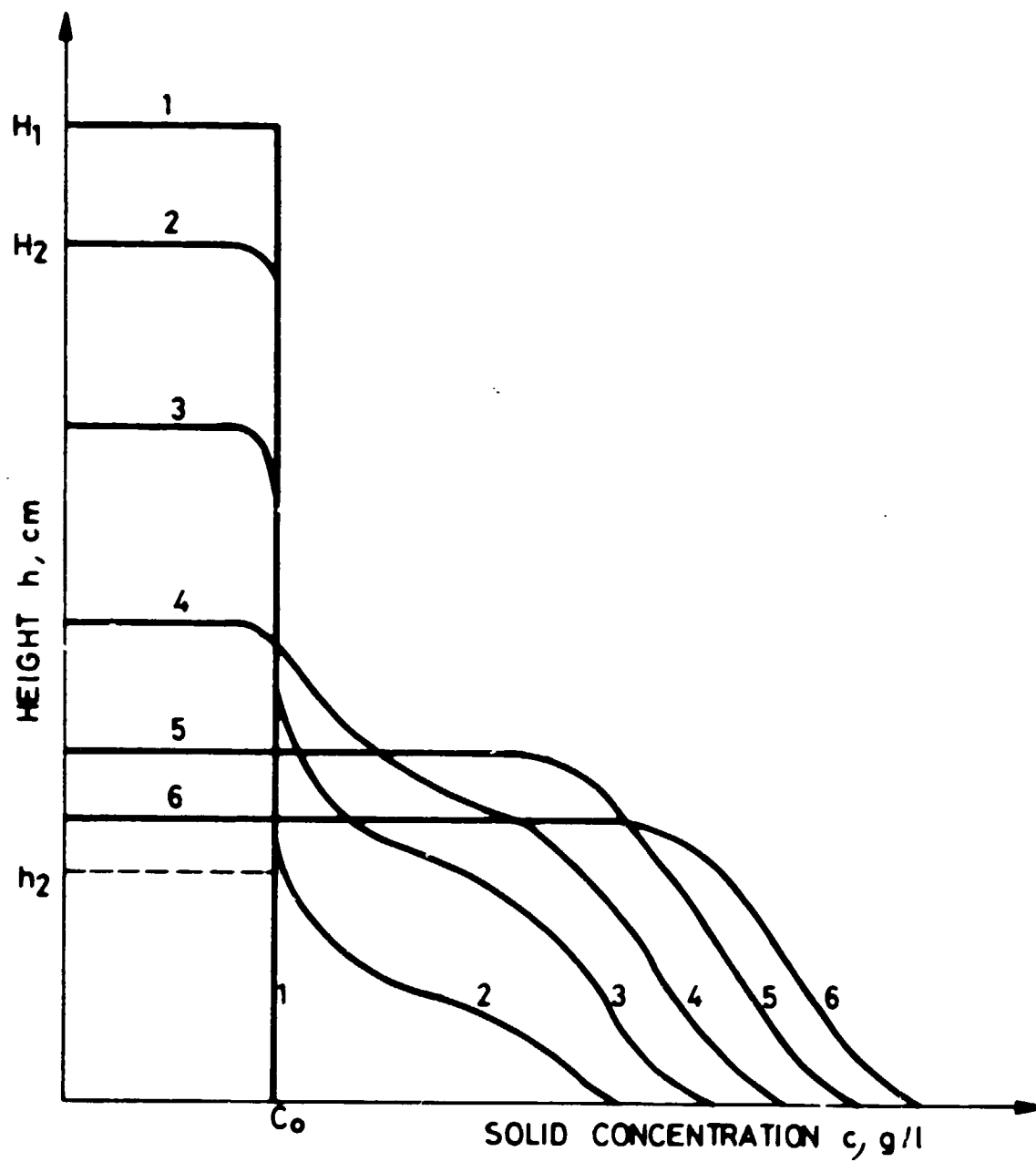


Fig. 6.24

CONCENTRATION PROFILES OF RED MUD SETTLING

Curve 2, registered at  $t_2$  illustrates the developing of the compression zone. The solids concentration at the bottom of the cylinder is much higher at that time already than the initial  $c_0$  value and it reaches the  $c_0$  value after a gradual decrease to  $h_2$ . From this point up to  $H_2$  the initial  $c_0$  is found. At  $H_2$  the concentration decreases suddenly to 0. From the bottom to  $h_2$  is the compression zone. From  $h_2$  to  $H_2$  is the interval of hindered settling and above  $H_2$  is the zone of the clear liquor.

As the settling advances, the compressibility of the mud in the bottom of the cylinder and the height of the compression zone increases, the zone of hindered settling decreases. The height of the clear liquid zone above the mud level increases.

Later the hindered settling zone disappears /profile curve 4./, and as there is no further mud supply to the compression zone it is compressed further.

The H-t diagram shown in Fig. 6.24 can be plotted from the series of concentration profile curves of the gamma-ray absorption the same way the simple test tube investigations, because the point of sudden drop of the profile curves to zero solids concentration gives the height of the mud level at any time. Not only the current height of the mud zone can be determined from the profile curves but the distribution of their solids concentration, too. Settling velocity of the red mud particles can be determined for any solids concentration with the help of computer analysis of the curves and, in addition, the diameter and other parameters of the aggregates and flocs formed during settling can also be determined.

The tests show definitely that in a well flocculated red mud suspension the diameter of the flocs is about the same and the flocs settle in the same way, in spite of the fact that the elementary particles have a polydisperse size distribution.

During the compression of the mud the structure of the flocs changes and a considerable part of the liquor closed in the flocs is released. Owing to this a higher compression rate can be attained in settlers than it would be enabled by the close fitting of flocs containing much liquor.

A characteristic function of settling of solids is the flux-diagram which gives the quantity of the solids settled on a unit area in unit time in function of the concentration /26/. The solid flux /S/ is a product of the settling velocity and the solids concentration

$$S = c \cdot u \quad \frac{\text{kg}}{\text{m}^2\text{h}}$$

A characteristic flux-diagram calculated from concentration profiles is shown in Fig. 6.25. The flux-diagrams are always similar to that one. The values corresponding to the given concentrations depend on the type of digested bauxite, on the way of digestion and on the flocculant. Settling ability depends on the specific surface of the solid phase of the mud.

The results of model settling tests can also be transmitted to the continuously operating settlers, by the use of computer simulation. The settling capacity of the continuously operating plant settlers is determined on the basis of small sample laboratory tests /27, 28, 29/.

The model tests are suitable to determine the effects of technological modifications - e.g. additives to digestion - on the settling and compression properties of the red mud, and the optimal dosage of new types of flocculants /30/.

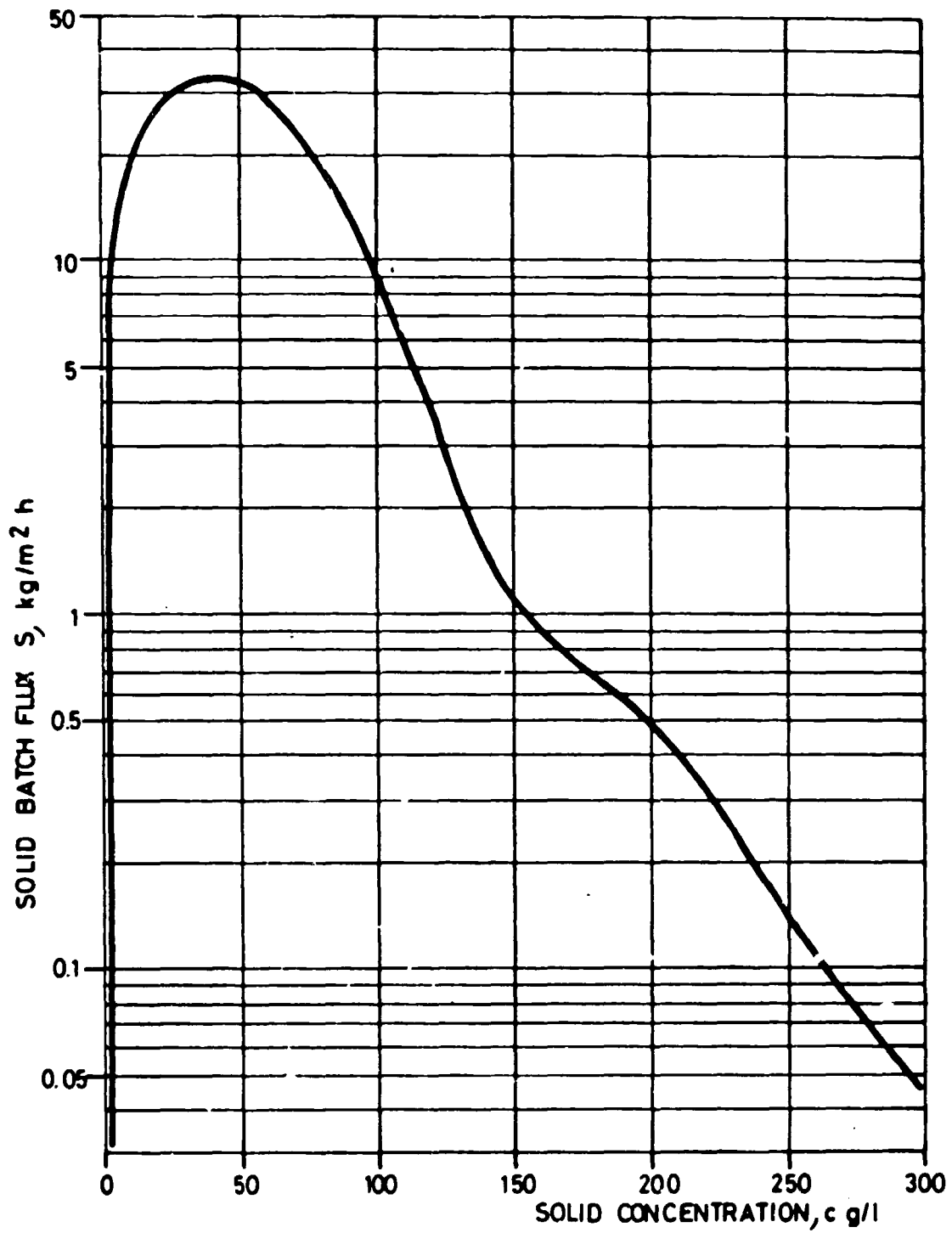


Fig.6.25

SOLID BATCH FLUX vs. SOLID CONCENTRATION

## DETERMINATION OF RED MUD WASHABILITY AND FILTERABILITY

The washing in counter-current series and the subsequent filtration /if any/ serve for the removal of the adherent liquor of red mud and partly of the caustic soda and alumina bound in the solid phase.

The subsequent stages of the counter-current washer line are fed by the thickened slurry of the previous stage diluted by the thinner liquor of the next stage. In the last stage of the series water or a very thin washing liquor is used as diluting liquid. This liquor progressing forward in the washing series dilutes the residue liquors of increasingly higher concentration, consequently its caustic soda and alumina concentration increases. On the other hand the mud progressing to the end of the washer line becomes more and more poor in caustic and alumina: it's liquid phase gets gradually diluted.

During dilution bound losses are decreasing too as caustic soda and alumina are leached out of the solid phase.

The rate of dilution in the washer line can be calculated with a fairly good approximation for each stage.

Separation of the liquid phase from the mud by filtration results in considerably less residual liquor in the mud than in the washing series, therefore, the washing also needs less water.

Generally the work of a washing line or a filter can not accurately be copied in the laboratory model tests made for determining the efficiency of washing or filtration. This tests serve, first of all for comparing the characteristics of the mud samples.

### Washing Tests

For the accurate determination of  $\text{Na}_2\text{O}$  and  $\text{Al}_2\text{O}_3$  content released from the mud during washing, the concentrations of the liquor have to be determined. The liquor is separated from the mud by a centrifuge and is analysed. Analysis requires high sensitivity and a selective measuring method. It is often carried out with radioisotope tracing concentration measurements based on radiation.

Since the  $\text{Na}_2\text{O}$  and  $\text{Al}_2\text{O}_3$  losses are showing up due to the formation of sodalites and sodium aluminates, it is expedient to produce these materials in pure form, and carry out the washing tests on these model materials /29/.

Fig. 6.25 shows the  $\text{Na}_2\text{O}$  concentration of the liquid phase, developing during the washing. It is characteristic that, plotting the  $\text{Na}_2\text{O}$  concentrations on a logarithmic scale the resulting curve is decreasing linearly at the beginning, at the end they are situated above the line.

The decreasing line shows the regular dilution of the adherent liquor, and the deviation from the line indicates the simultaneously partial dissolution of the bound  $\text{Na}_2\text{O}$ .

A characteristic example of dissolution of model materials by distilled water is shown in Fig. 6.27 where the  $\text{Na}_2\text{O}$ ,  $\text{Al}_2\text{O}_3$  and  $\text{SiO}_2$  liquid concentrations relative to the OH-sodalite are plotted vs. time. Similar curves can be plotted for the other sodalites, too.

The  $\text{Na}_2\text{O}$  concentration of the solution increases steeply at the beginning of the dissolution, then it transforms into a line of well defined slope. Contrary to that, the  $\text{Al}_2\text{O}_3$  and  $\text{SiO}_2$  concentration decreases nearly linearly after a quick increase. The ratio of the components in the solution is not

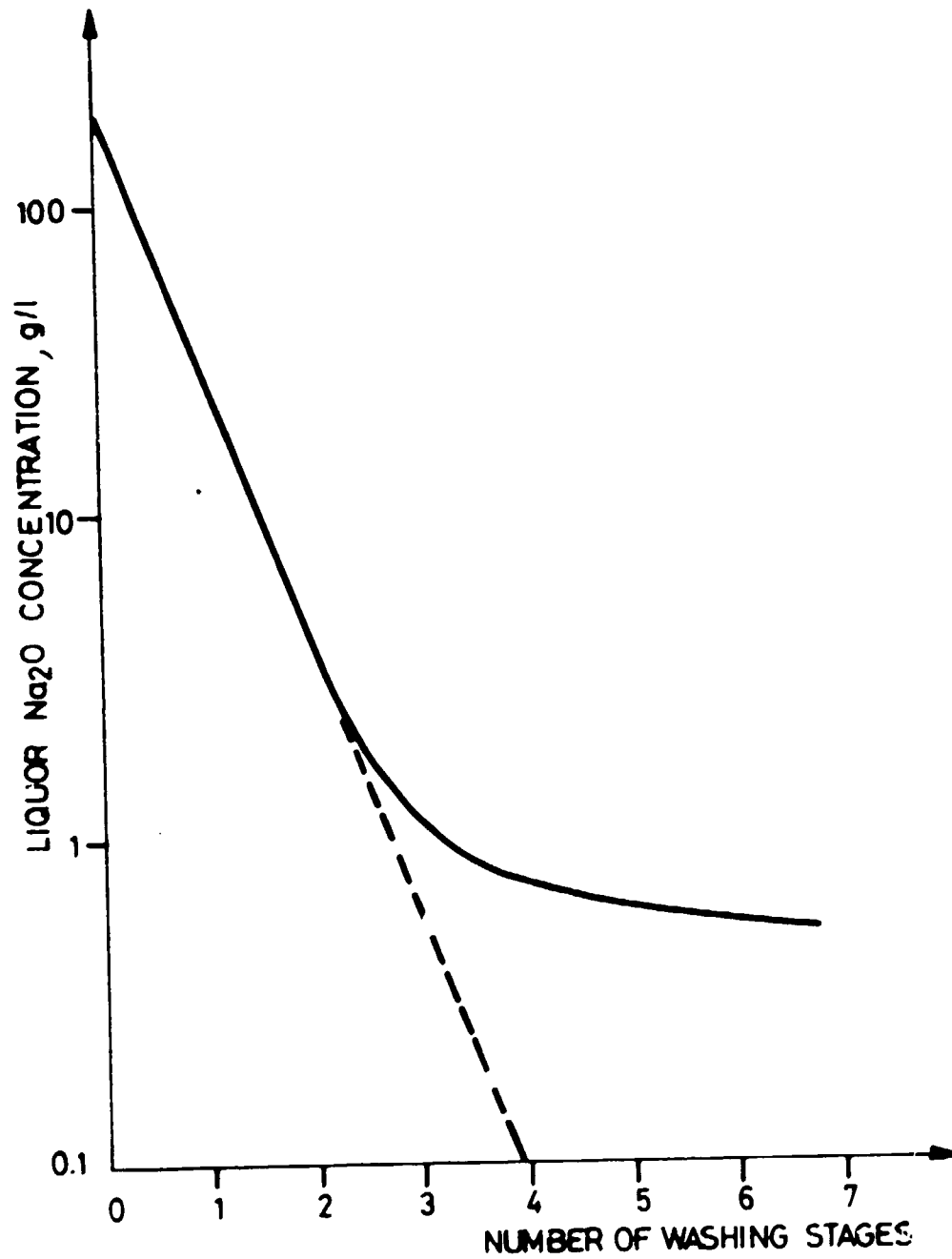


Fig. 6. 26

$\text{Na}_2\text{O}$  CONCENTRATION IN LIQUOR vs. NUMBER  
OF WASHING STAGES



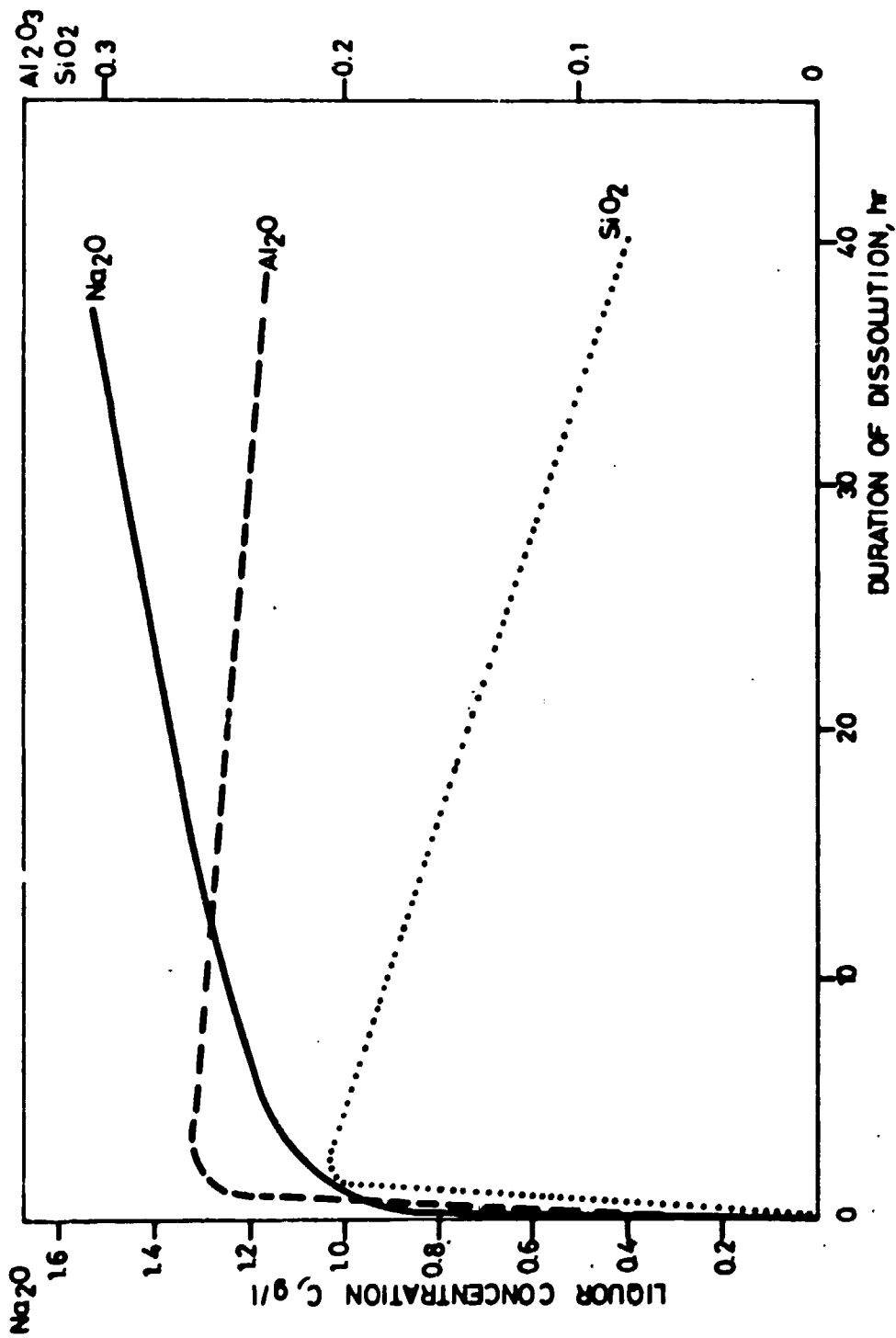


Fig. 6.27

DISSOLUTION OF MODEL MATERIALS IN DISTILLED WATER

constant and it is not equal to the ratio of the components in the solid phase, consequently, a simple stoichiometric dissolution is out of question.

The following processes are taking place during the dissolution and partly during the washing:

1. partial dissolution /in the beginning the concentration of all the three components increases in the solution/
2. hydrolysis /the  $\text{Na}_2\text{O}$  concentration of the solutions is higher than that corresponding to the stoichiometric ratio of solid phase/
3. phase transformations /partial transformation of the crystal structure, formation of new phases/

The amount of  $\text{Na}_2\text{O}$  dissolved from the solid phase depends equally on the solid concentration of the slurry and on the dissolution time.

No characteristic solubility values were found at the dissolution of titanite compounds resulted in the digestion process. Dissolution, hydrolysis and phase transformations determine collectively the amount of  $\text{Na}_2\text{O}$  recoverable from the solid phase.

#### Filtration Tests

Laboratory filtration tests of red mud are carried out by small filters at moderate vacuum. If constant conditions are ensured, comparable tests can be carried out on different muds /31/.

The period required to the liquor phase of a red mud sample of given volume through a selected paper or textile filter is measured. The residue liquor of about 10-12 weight per cent

after vacuum filtration would cause considerable losses, hence washing is also applied at the filtration.

## CONTROL FILTRATION AND PRECIPITATION MODEL TESTS

### Removal of Contaminants from the Aluminate Liquor

Technological evaluation of bauxites covers the model tests depending considerably on bauxite quality /grinding, predesilication, digestion, settling/. In certain cases, when the contaminants of the aluminate liquor obtained from the settling process are higher than the admissible values, the technological investigations have also to be extended to purification tests of the aluminate liquor. Suspended  $\text{Fe}_2\text{O}_3$ , dissolved Zn and  $\text{P}_2\text{O}_5$  content of the aluminate liquor must be mentioned among contaminants having a detrimental effect on the quality of the produced alumina.

High suspended  $\text{Fe}_2\text{O}_3$  content in the aluminate liquor has to be reckoned with when it originates from a bauxite containing poorly settling iron minerals /goethite, pyrite, siderite, chlorite/.

In case of digesting certain bauxites with Zn content the latter dissolves into the liquor to such an extent that it has to be removed continuously in order to reach the required alumina quality.

In processing bauxite of high phosphorus content,  $\text{P}_2\text{O}_5$  as a detrimental contaminant of alumina has to be taken into consideration. An appropriate  $\text{P}_2\text{O}_5$  level of the circuit liquor can be attained by adding lime to digestion or by intensive salt removal.

In the case the aluminate liquor contains high suspended quantities of  $\text{Fe}_2\text{O}_3$  its required purity is ensured by control filtration with different filtering aids.

The laboratory control filtration tests can not model the filtration technology in the plant they can only qualify the filtering aid and examine its effect on control filtration. Lime-hydrate and paper precoat are the most frequently applied filtering aids. On the basis of laboratory pressure or vacuum filter tests data can be furnished on the quality, optimal thickness and efficiency of the filtering aid.

When the Zn content of the investigated bauxite is high the quantity of dissolved Zn is to be measured and the efficiency of the applied Zn removal technology by  $\text{Na}_2\text{S}$  is to be checked. On the basis of this test a proposal can be made to the technological designer on the optimal dosage of  $\text{Na}_2\text{S}$ . It is very important, that the determined optimal dosage of  $\text{Na}_2\text{S}$  must not be exceeded during the operation because excess  $\text{Na}_2\text{S}$  causes corrosion and settling troubles.

#### Laboratory Precipitation Tests

The precipitation process of alumina hydrate does not depend strictly on the quality of the processed bauxite, so the laboratory precipitation tests do not belong to the technological evaluation of bauxites. The basic operating parameters of precipitation are standardized in both the American and European Bayer processes. The laboratory precipitation tests are usually directed to a certain plant operational problem or aimed at studying the kinetics and mechanism of the precipitation from scientific point of view.

Precipitation is the most difficult modellable part of the Bayer process. The greatest problem of laboratory modelling is that it is very difficult to reproduce the hydrodynamic conditions of plant precipitation which have a considerable influence on the crystallization. The reproduction of the temperature profile of the natural cooling in the plant faces the same difficulties. An additional problem is that the alumina hydrate seed stored in the laboratory can grow old and become inactive.

Taking into consideration the difficulties outlined above, a perfect reproduction of plant conditions can not be aimed at laboratory model tests of precipitation. However, comparative tests are to be made at well defined conditions varying some parameters. These comparative tests are frequently made under isothermal conditions.

Seed and product alumina hydrate can be qualified on the basis of laboratory precipitation tests. The most important properties of the hydrate are as follows: grain-size distribution, specific surface area, and the morphological properties of the hydrate. For determining the size distribution screen analysis and sedimentation are the most frequently applied methods.

The specific surface area is measured by BET method. An approximative value can be calculated from the granulometry. Further information can be gained relating to the surface area of the hydrate by measuring the heat effect of immersion. Electron microscopic investigations have a particular role in cognition of processes of precipitation. By using electron microscope phenomena, like crystal growth and agglomeration can be studied.

A principal aim of the precipitation process is to produce alumina hydrate of suitable granulometry and physical properties at as high effectiveness as possible. Recently, the up-to-date alumina smelters and the environment protection rises more and more strict requirements on physical properties of alumina /appropriate granulometry, dust loss, degasing capacity/.

Laboratory tests serve to determine the optimum parameters to attain the aims described above. In the following, the effect of some significant parameters with a few examples taken from literature or our own experiments is illustrated.

The alumina recovery is plotted versus time and temperature at 150 gpl seed charge in Fig. 6.28 /32/.

The alumina recovery is plotted versus time and seed charge at 65.5 °C in pure aluminate solution in Fig. 6.29 /32/.

The evolution of crystal size distribution is plotted vs. time at low seed charge in Fig. 6.30 /33/.

The evaluation of crystal size distribution versus time at higher seed charge is shown in Fig. 6.31 /33/.

Average grain-size of the product vs.  $Fe_2O_3$  content /Fig. 6.32/ and oxalate content /Fig. 6.33/ of the pregnant liquor are plotted on the basis of experiments made at ALUTERV-FKI.

#### LABORATORY TESTS FOR REGENERATION OF CAUSTIC SODA

##### Distribution of Caustic Soda Losses in Bayer Process

One of the most important cost factors of the Bayer process is the cost of make up caustic soda. The amount of the caustic soda loss per 1 ton of alumina produced is a sum of several elements. Based on data of Hungarian alumina plants the distribution of caustic soda losses is given below:

	NaOH loss, %
Chemical losses:	83.5
Dissolved losses with the red mud:	3.3
Losses with the alumina:	2.3
Carbonate and mechanical losses:	9.6
Scales and other losses:	1.3

It is seen from the above distribution that the majority of the losses are the chemical losses leaving the circuit together with the red mud in form of sodium-aluminium-silicates

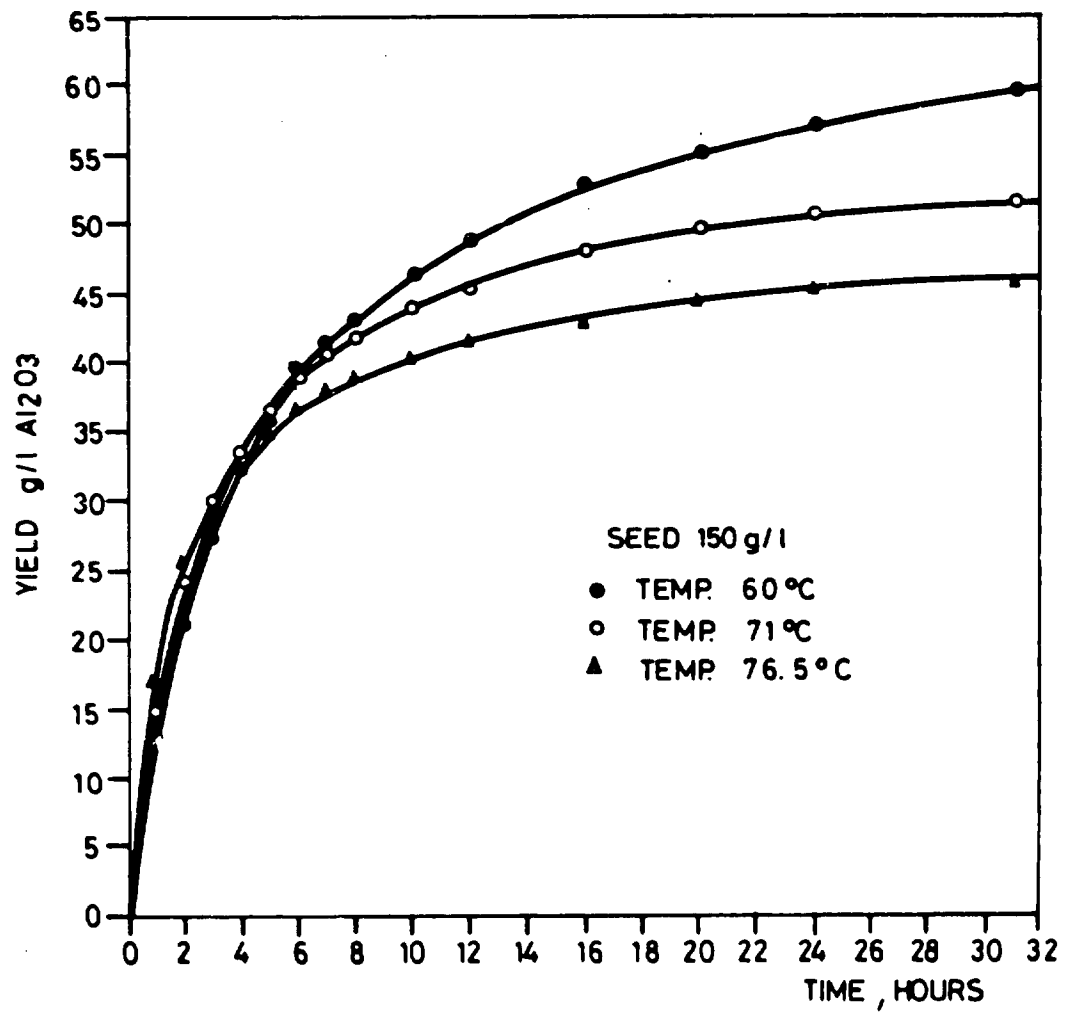


Fig. 6.28

ALUMINA YIELD vs. TIME AND TEMPERATURE AT 150 g/l SEED CHARGE

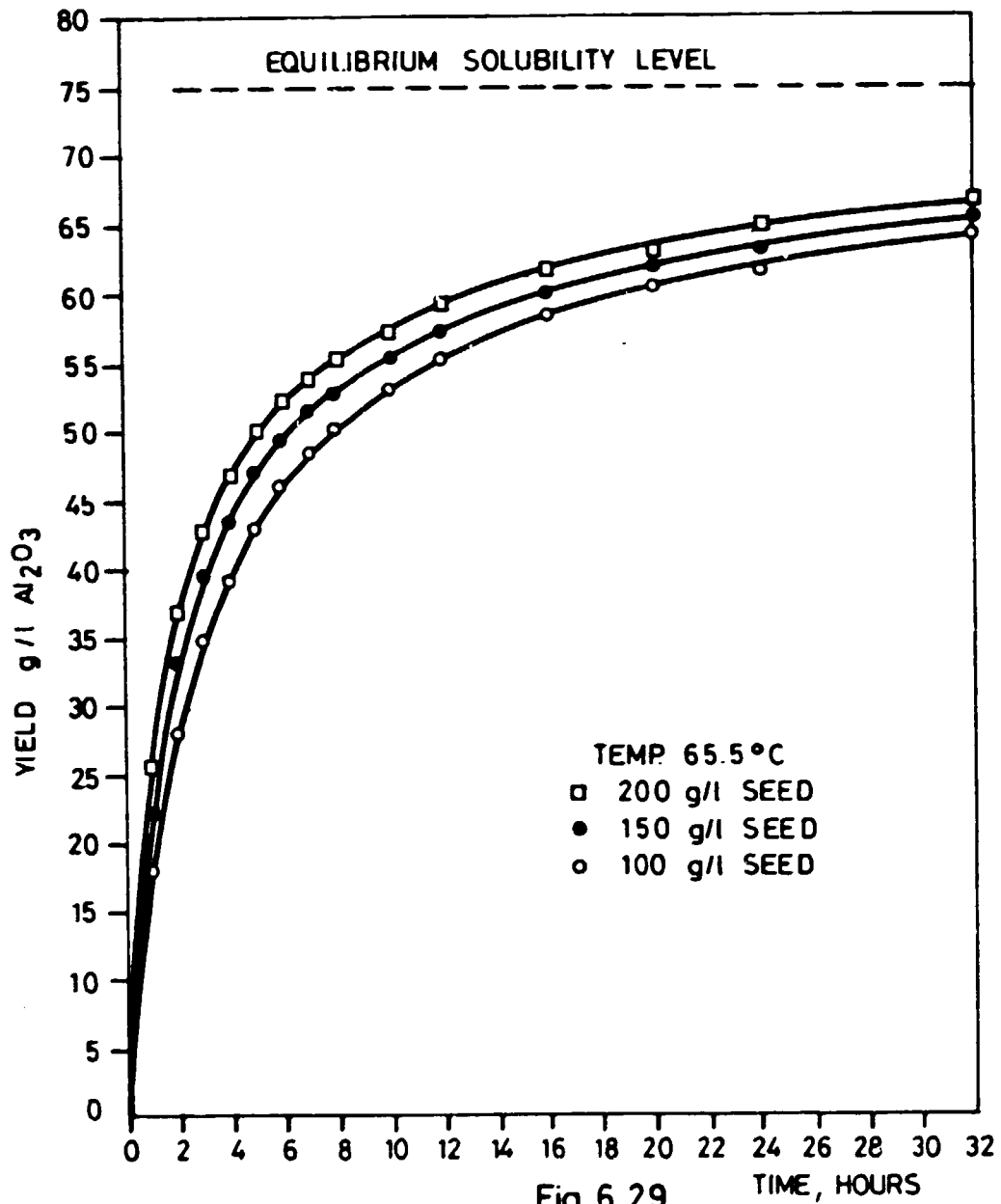


Fig. 6.29 ALUMINA YIELD vs. TIME AND SEED CHARGE AT 65.5°C



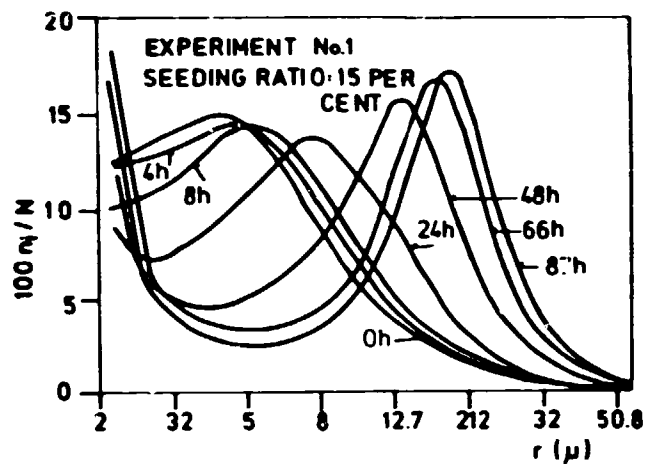


Fig. 6.30

EVOLUTION OF CSD WITH TIME, IN A BATCH CRYSTALLIZER-LOW SEED CHARGE

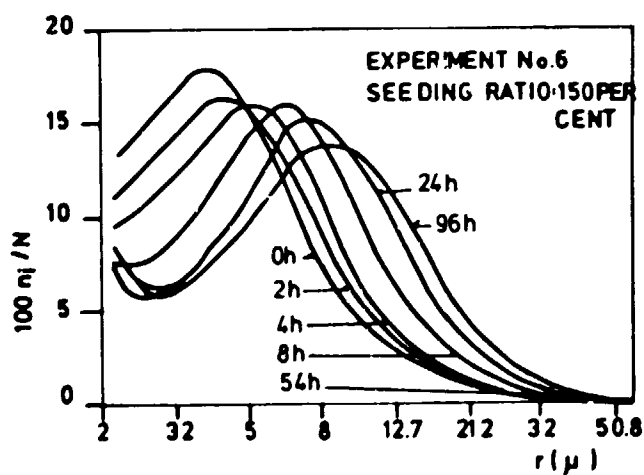


Fig. 6.31

EVOLUTION OF CSD WITH TIME, IN A BATCH CRYSTALLIZER-HIGH SEED CHARGE

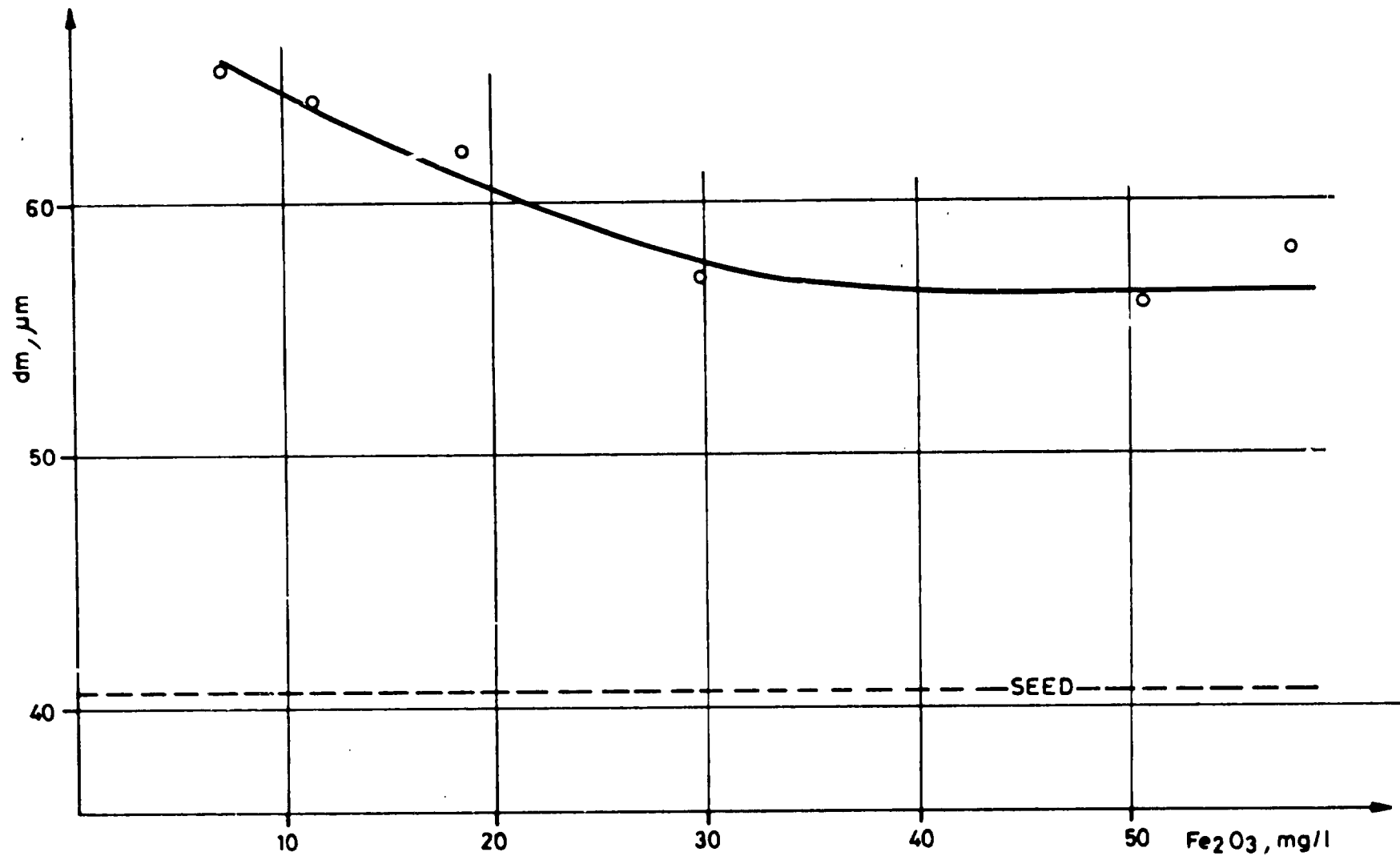


Fig. 6.32  
 AVERAGE GRAIN-SIZE OF THE PRODUCT vs. Fe<sub>2</sub>O<sub>3</sub>  
 CONTENT OF PREGNANT LIQUOR

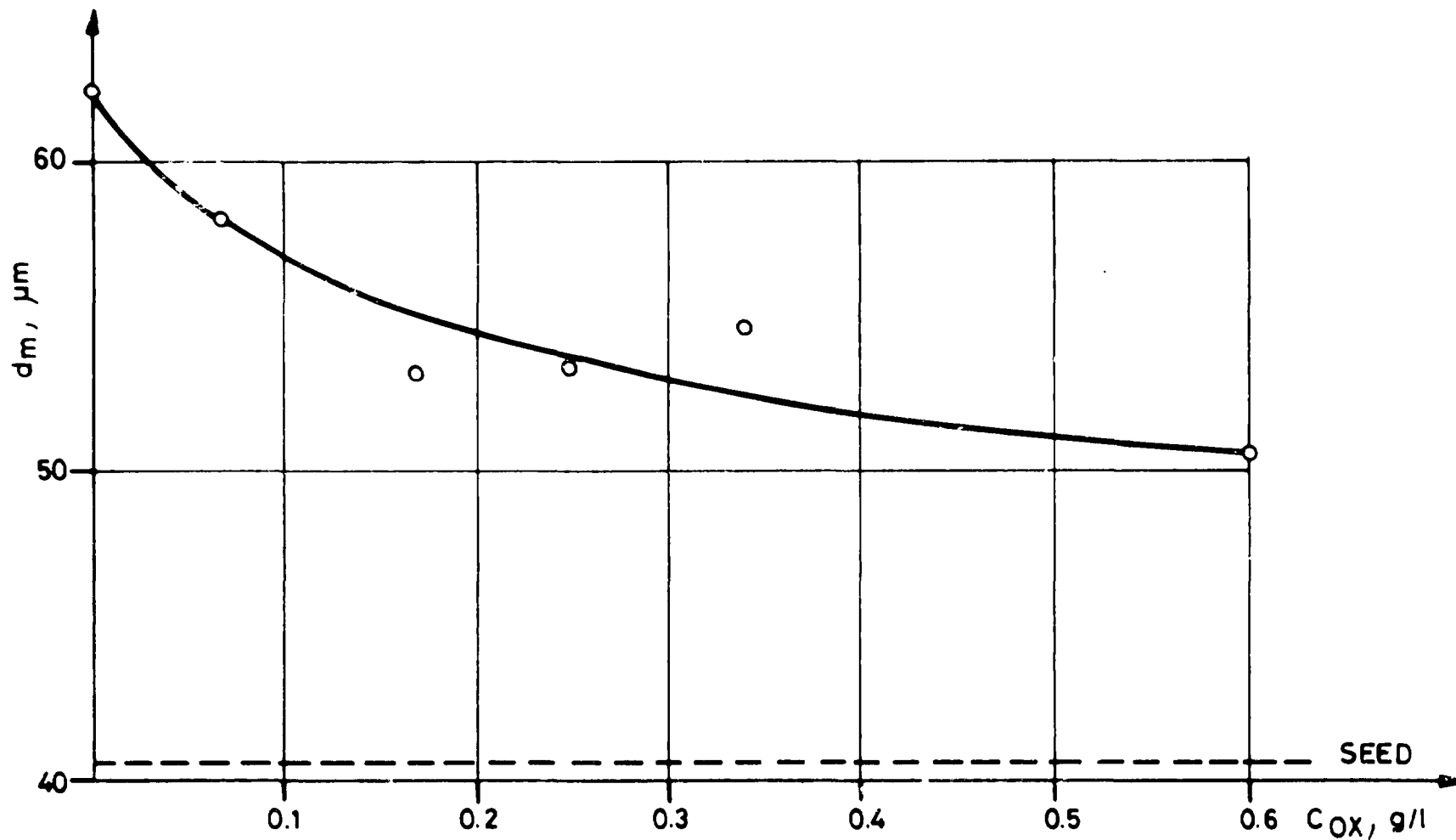
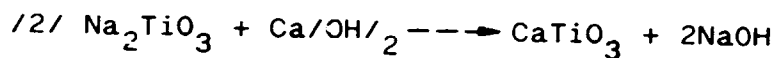
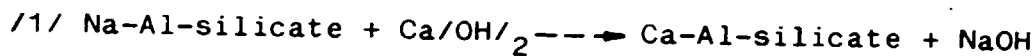


Fig. 6.33  
AVERAGE GRAIN-SIZE OF THE PRODUCT vs. OXALATE CONTENT  
OF PREGNANT LIQUOR

and sodium-titanates, and the carbonate losses. These losses can be reduced by regeneration process inside the circuit or joined to it.

Caustic Soda Regeneration by Causticization of Red Mud  
Laboratory Modelling of Causticization Process

In the course of causticization process of red mud the NaOH content bound in form of sodium-aluminium-silicates and sodium-titanate is set free by lime addition corresponding to the reactions given below:



The product of reaction /1/ is a hydrogarnet type compound, at  $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot k\text{SiO}_2 \cdot /6-2/k\text{H}_2\text{O}$  where the value of k varies between 0.8 and 2.2 /34/.

There are different theories concerning the mechanism of the reaction, e.g.: ion exchange, reactions taking place in dissolved, phase on interface etc. Juhász and his collaborators on the basis of their experiments postulate ion exchange in the initial period of the causticization and a total conversion of silicate phase in the further course of the reaction /35/.

The recovery and the kinetics of causticization differ very much for different Na-Al-silicates. Na-titanates can be causticized practically with 100 per cent recovery. The causticizability of a few synthetically produced silicate phases of

$3/\text{Na}_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2/\text{Na}_2\text{X}$  composition.

- where X means different anions as  $\text{AlO}_2^-$ ,  $\text{CO}_3^{--}$ ,  $\text{SO}_4^{--}$ ,  $\text{Cl}^-$ , etc. - is compared in Fig. 6.34.

Causticization was carried out under the same conditions in each case, namely at  $90^\circ\text{C}$  and addition of 3.5 mole  $\text{CaO}$ /mole  $\text{Na}_2\text{O}$  /36/.

In a plant a combination of various Na-Al-silicates of different composition and crystal structure is formed, depending on the digestion parameters and the level of salt contaminants of sodium-aluminate solutions /see Vol. 2. p. 44./. Taking these into consideration the optimum parameters of causticization are to be determined by laboratory tests, using always the red mud sample of the tested plant.

The causticization process can be modelled very well under laboratory conditions using 500-1000 ml volumes. Corresponding to the plant technology, the slurry sample used to laboratory tests has to be taken from the underflow of the last stage of the washer line. The efficiency of causticization of a given red mud is affected by the following factors:

- a/ dosage of  $\text{CaO}$  / $\text{CaO}/\text{Na}_2\text{O}$  molar ratio/
- b/ temperature of causticization
- c/ solids concentration of the red mud slurry
- d/  $\text{Na}_2\text{O}$  concentration of the liquor phase.

The optimum values of these parameters are to be determined on the basis of laboratory tests with a view to the possibility of realization in the plant.

The effect of the  $\text{CaO}$  dosage and the run of reaction vs. time at optimum lime dosage are demonstrated in Fig. 6.35, using the data of laboratory causticization tests with red mud from Almásfüzitő alumina plant. The effect of the causticization temperature and solids concentration are demonstrated in Figures 36 and 37. The initial  $\text{Na}_2\text{O}$  concentration of the liquor

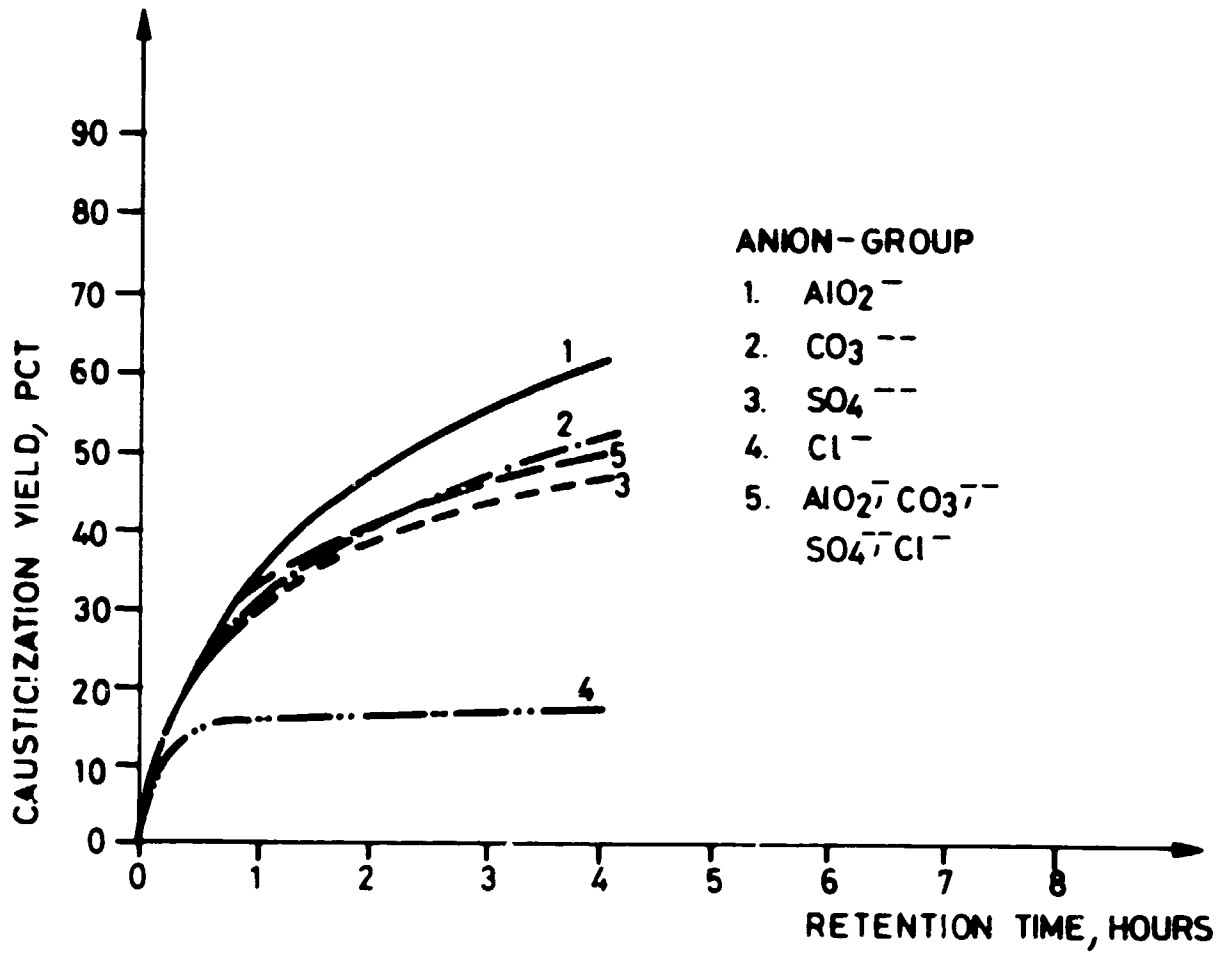


Fig.6.34

CAUSTICIZATION CURVES OF DIFFERENT  
Na - Al - SILICATES

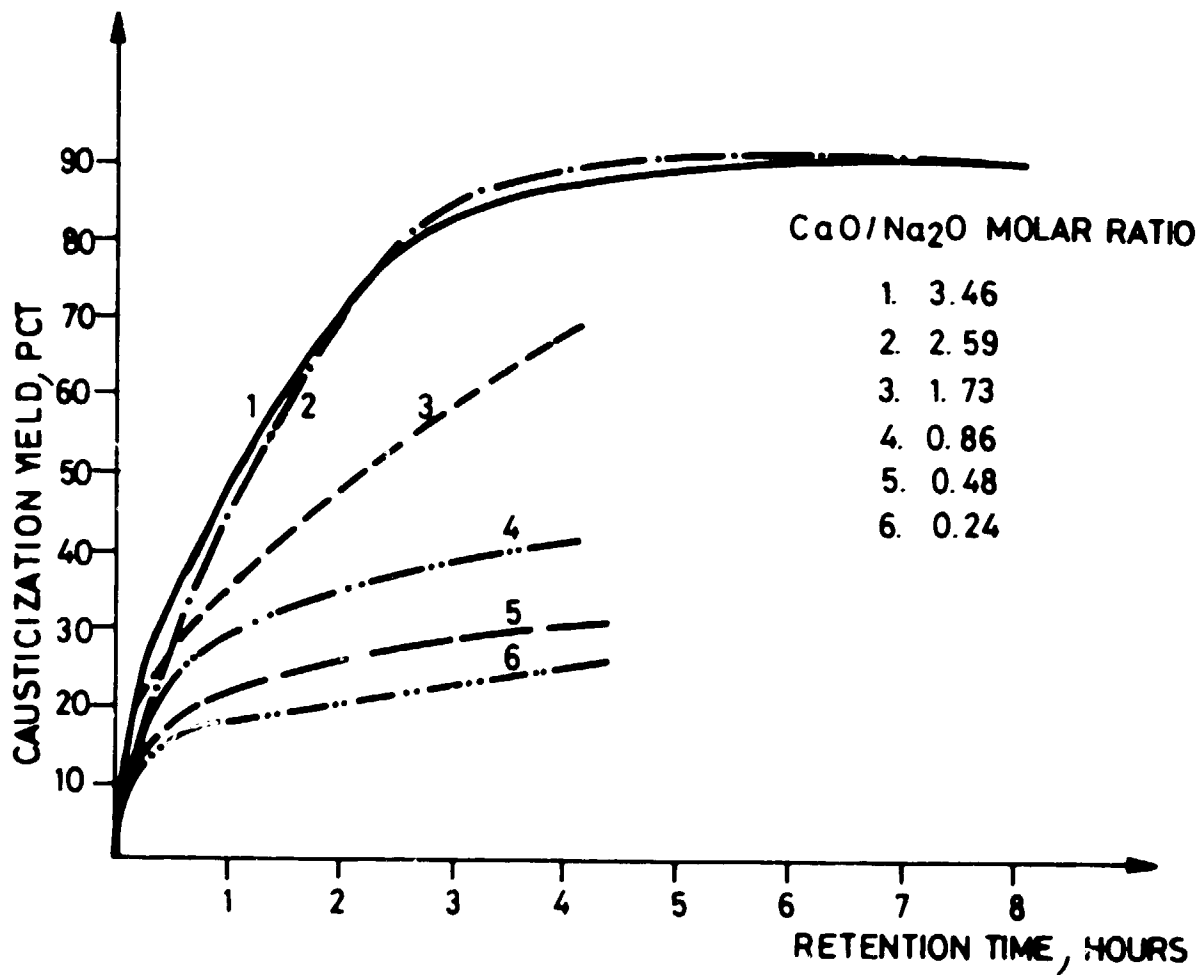


Fig.6.35

CAUSTICIZATION EFFICIENCY vs. CaO ADDITION

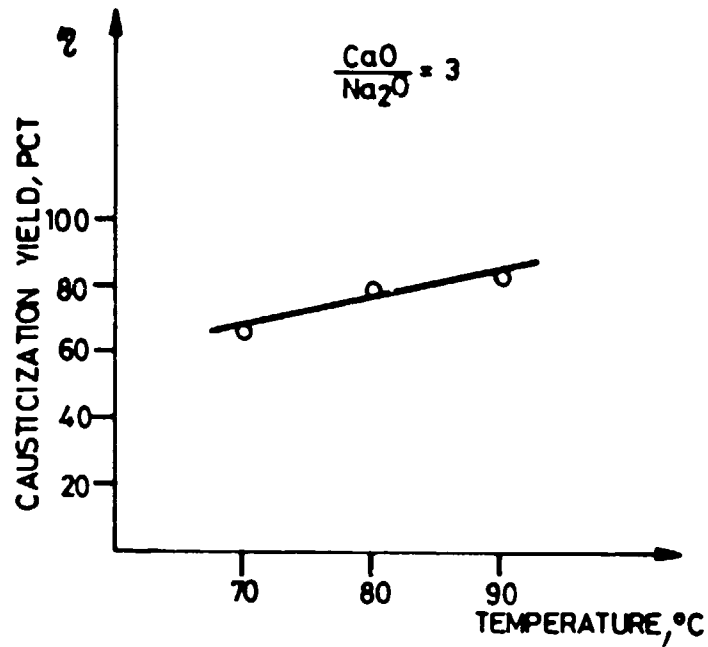


Fig. 6.36

CAUSTICIZATION EFFICIENCY vs. TEMPERATURE

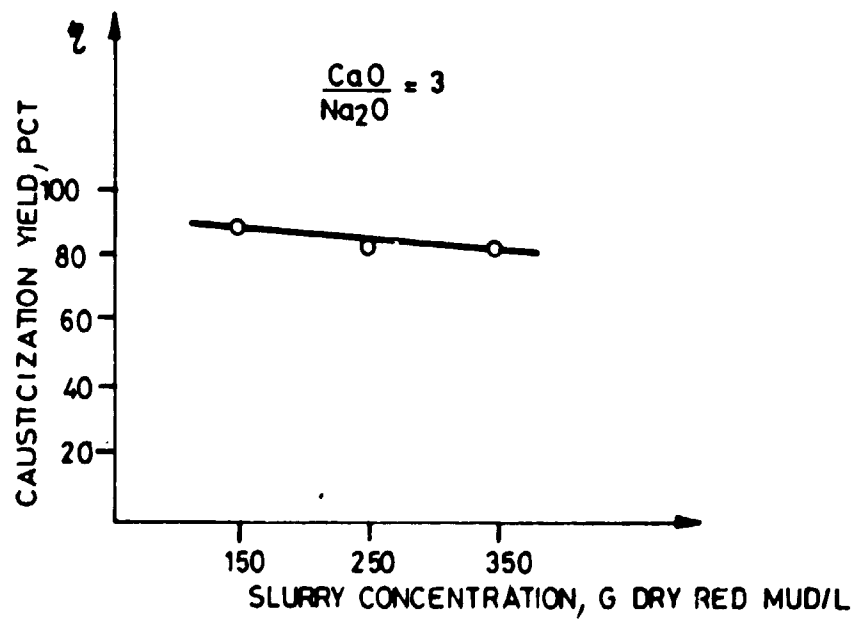


Fig. 6.37

CAUSTICIZATION EFFICIENCY vs. SOLID CONCENTRATION  
OF THE SLURRY



phase is given by the concentration of the last washing stage. The lower this value the more advantageous it is from the view point of causticization.

Even 70 to 90 per cent efficiency of causticization can be attained under laboratory conditions provided the causticized red mud is washed carefully.

#### Problems of Realization of Red Mud Causticization in Plant

The fairly high regeneration efficiency achieved in laboratory can hardly be realized in a plant. The main reason is that the separation of red mud from the slurry leaving the last washer and being causticized is carried out in a one step filtration process. The filtrate is put into the washer stage of similar caustic concentration that of, while the mud is disposed to the red mud pond. The filtered red mud contains in general 50 wpcnt adhesive liquor, thus a considerable amount of caustic converted to liquor phase by causticization is disposed to the red mud pond.

Causticization at one of the preceding washer stages is out of question because of inadmissible  $Al_2O_3$  losses due to the formation of  $3CaO \cdot Al_2O_3 \cdot 6H_2O$  with the higher alumina content of the liquor.

Provided the causticization technology is controlled precisely, 40-50 per cent caustic recovery can be achieved in plant practice.

#### Regeneration of Soda Salt Content of Bayer Circle

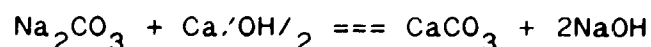
Carbonate impurities of bauxite /siderite, dolomite, calcite/ cause considerable decausticization, soda losses, and technological troubles. Soda salt originated from decomposition

of carbonate minerals accumulates in the liquor circuit to the limit of solubility, so its removal or continuous regeneration is to be provided /see Vol. 2. p. 156./.

The solubility of  $\text{Na}_2\text{CO}_3$  depend firstly on the concentration of the aluminate liquor so, in plants operating by different technology the admissible level of  $\text{Na}_2\text{CO}_3$  level is different. In plant operating with thin liquors of the American Bayer technology the carbonate soda level can be as high as 20 to 30 per cent while in plant operating with the more concentrated liquors of the European Bayer process the carbonate soda level is to be held at 12 to 14 per cent in the interest of preventing troubles in the technology.

Suitable ways for regeneration of carbonate soda are either causticization of 60 to 80 gpl, solution of the soda salt removed from the strong /appr. 260 gpl  $\text{Na}_2\text{O}_c$ / liquor, at a temperature of 80 to 90 °C with addition of lime slurry or regeneration of the carbonate soda inside the Bayer circuit. Both methods are described in Vol. 2. p. 161 to 162. Separate causticization of soda salt is a very simple process not needing any separate laboratory tests.

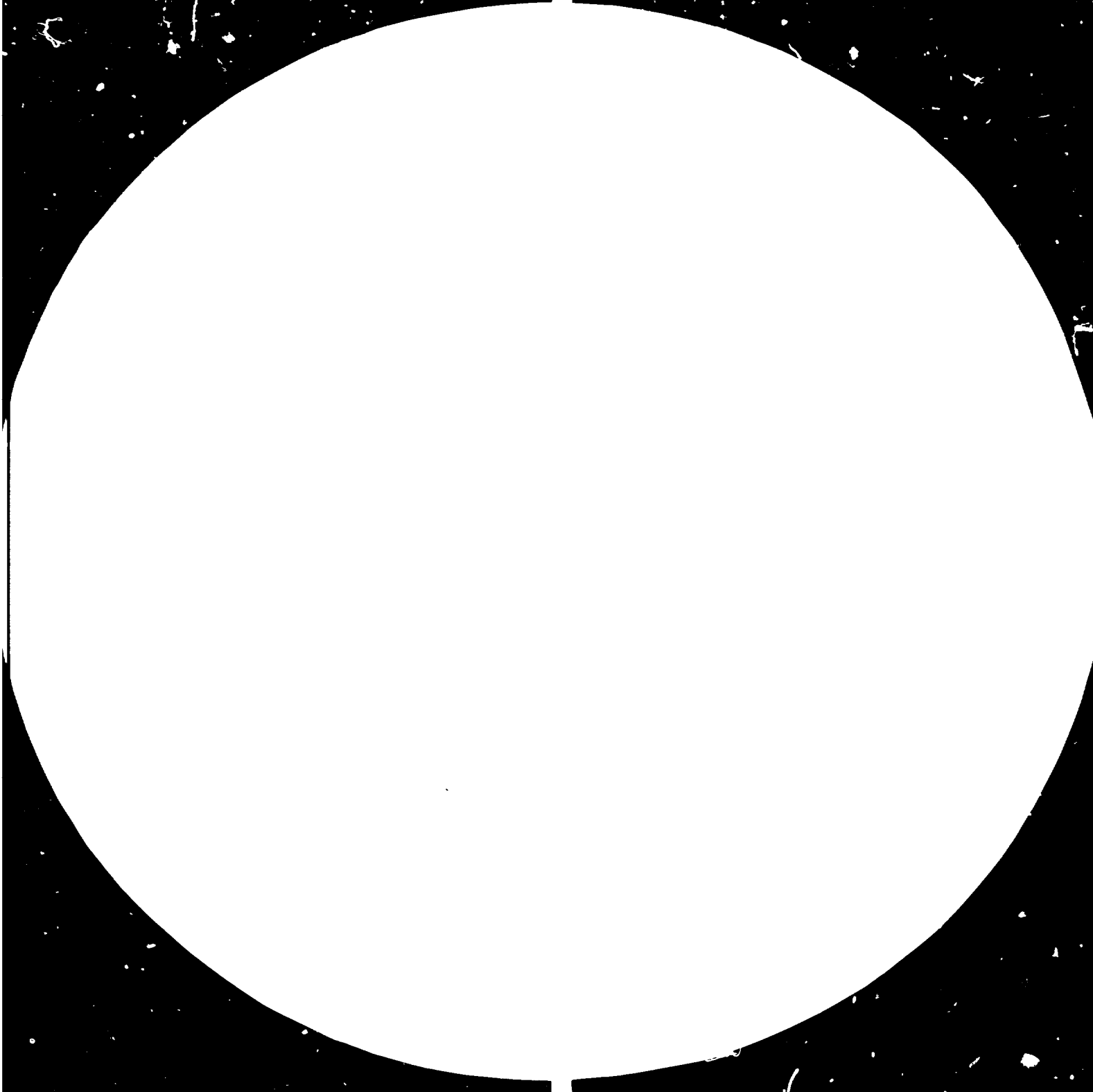
In the regeneration of  $\text{Na}_2\text{CO}_3$  inside the Bayer circuit the equilibrium conditions of  $\text{Na}_2\text{O}-\text{CaO}-\text{CO}_2-\text{Al}_2\text{O}_3-\text{H}_2\text{O}$  system are to be taken into consideration. The regeneration of soda can be realized only at the very point of the circuit where the equilibrium phase is  $\text{CaCO}_3$ , so that the introduced  $\text{Ca}/\text{OH}/_2$  is consumed by the reaction:



and losses in the form of  $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 6\text{H}_2\text{O}$  are at minimum. The

810113







3.6

4.5



MEASUREMENTS OF RESOLUTION TEST TARGET

RESOLUTION TEST TARGET

equilibrium diagram is demonstrated in Fig. 6.38 the area of  $\text{CaCO}_3$  composition is shown in Fig. 6.39.

It can be seen in the diagrams that the causticization of soda salts inside the system can be realized at concentrations of the washing series. The ideal territory of formation of  $\text{CaCO}_3$  is at the end of the washing line, but because of the low concentration only a small amount of carbonate soda can be causticized at this point. It is expedient to determine by laboratory experiments that concentration - that point of the washing series - where considerable amount of  $\text{Na}_2\text{CO}_3$  can be regenerated besides comparatively low  $\text{Al}_2\text{O}_3$  losses.

Data of some laboratory tests carried out for optimization of the causticization process in the washing line of Ajka Alumina Plant are shown as an example in Figures 6.40, 6.41 and 6.42. The efficiency given in the figure is calculated in percentages of the total  $\text{Na}_2\text{CO}_3$  content of the original liquor.

On the basis of laboratory tests dosage of 0.5 mole  $\text{Ca}/\text{OH}/_2$  per mole  $\text{Na}_2\text{CO}_3$  at 40 gpl  $\text{Na}_2\text{O}$  and at  $90^\circ\text{C}$  were suggested. Applying these parameters the Ajka Plant is recovering 30 to 40 per cent the  $\text{Na}_2\text{CO}_3$  formed in the circuit. A further advantage of the method is that  $\text{NaOH}$  delivered by the causticization reaction increases the caustic molar ratio in the washing line and decreases the  $\text{Al}_2\text{O}_3$  losses caused by hydrolysis.

In a further developed variant of causticization the soda salt extracted from the concentrated liquor is treated also in the causticization system built for the washing line.

#### INVESTIGATIONS OF THE TECHNOLOGICAL EFFECTS OF BAUXITE CONTAMINANTS

The technological evaluation of bauxites is to be extended to investigations of the contaminating components of bauxite.

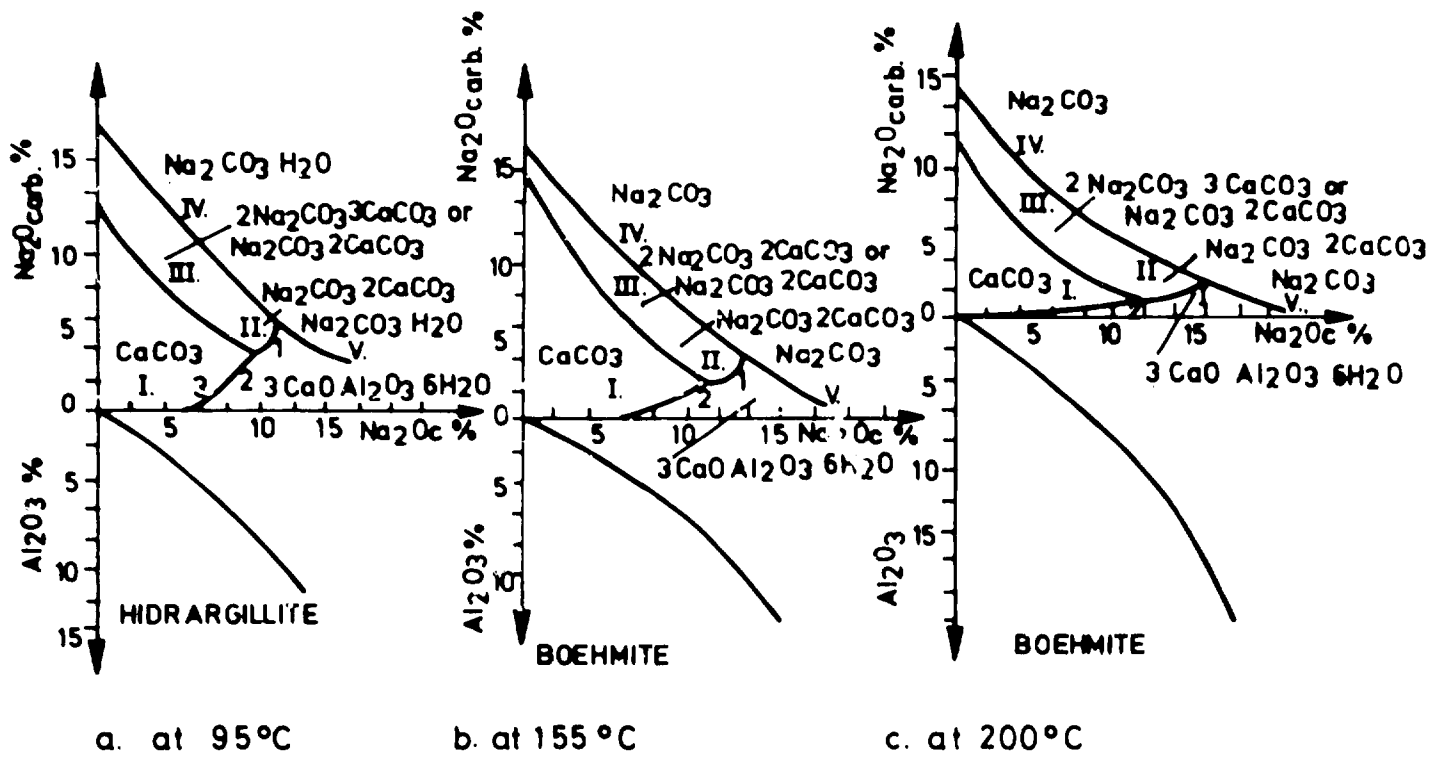
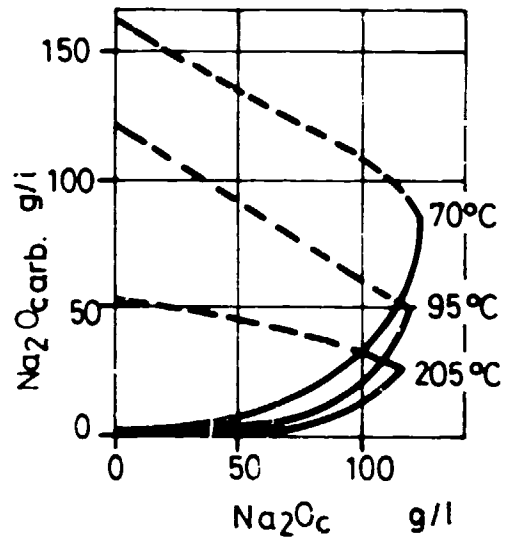


Fig. 6.38

ISOTHERMS OF  $\text{Na}_2\text{O}-\text{CaO}-\text{CO}_2-\text{Al}_2\text{O}_3-\text{H}_2\text{O}$  SYSTEM



LIMITED BY THE PHASES

---  $\text{CaCO}_3 \cdot a\text{Na}_2\text{CO}_3 \cdot b\text{H}_2\text{O}$

—  $\text{CaO} \cdot n\text{Al}_2\text{O}_3 \cdot m\text{H}_2\text{O}$

Fig. 6.39

AREAS OF  $\text{CaCO}_3$  COMPOSITION



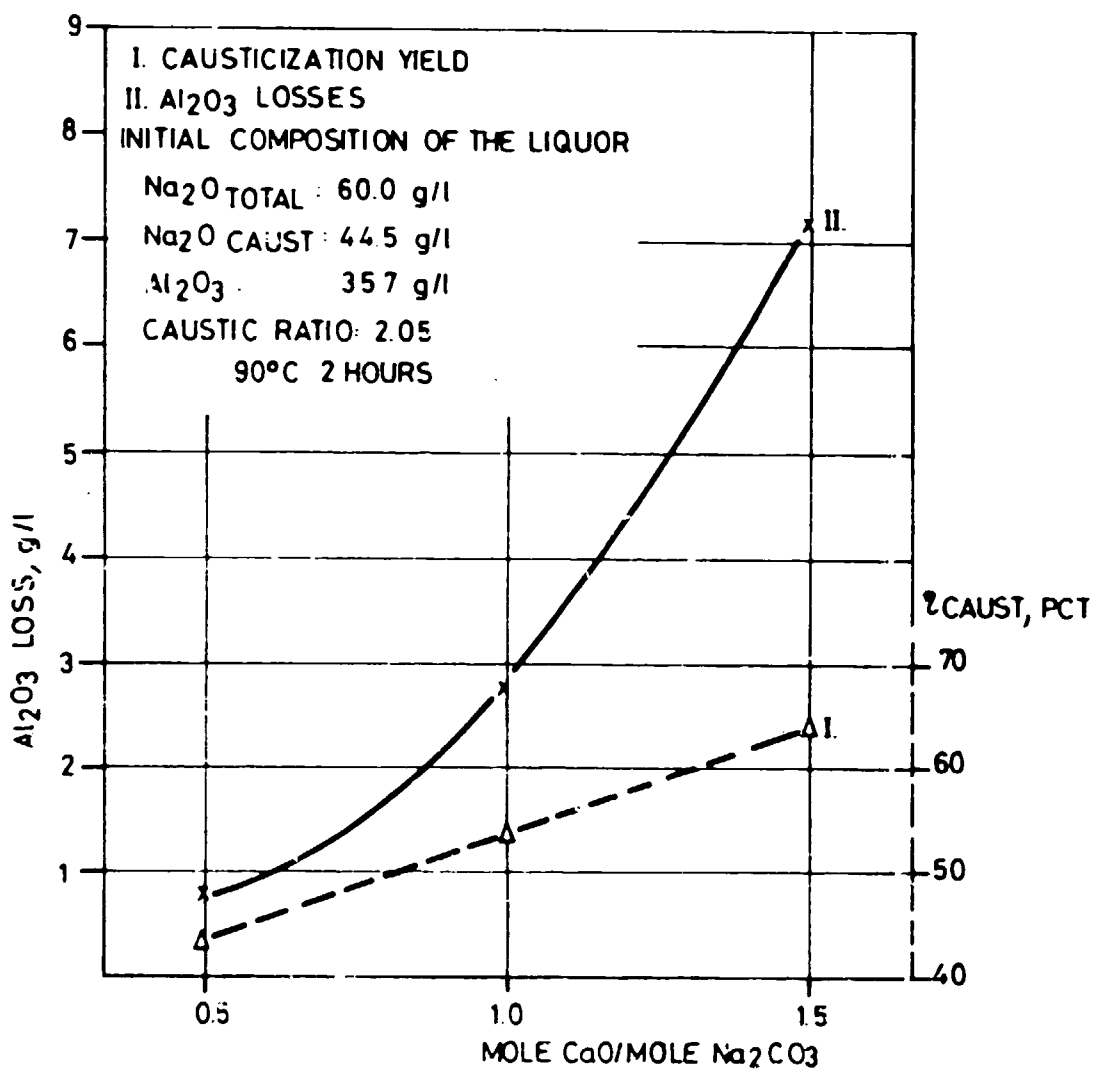


Fig. 6.40

CAUSTICIZATION EFFICIENCY AND  $\text{Al}_2\text{O}_3$  LOSSES AT  
 DIFFERENT LIME DOSAGES

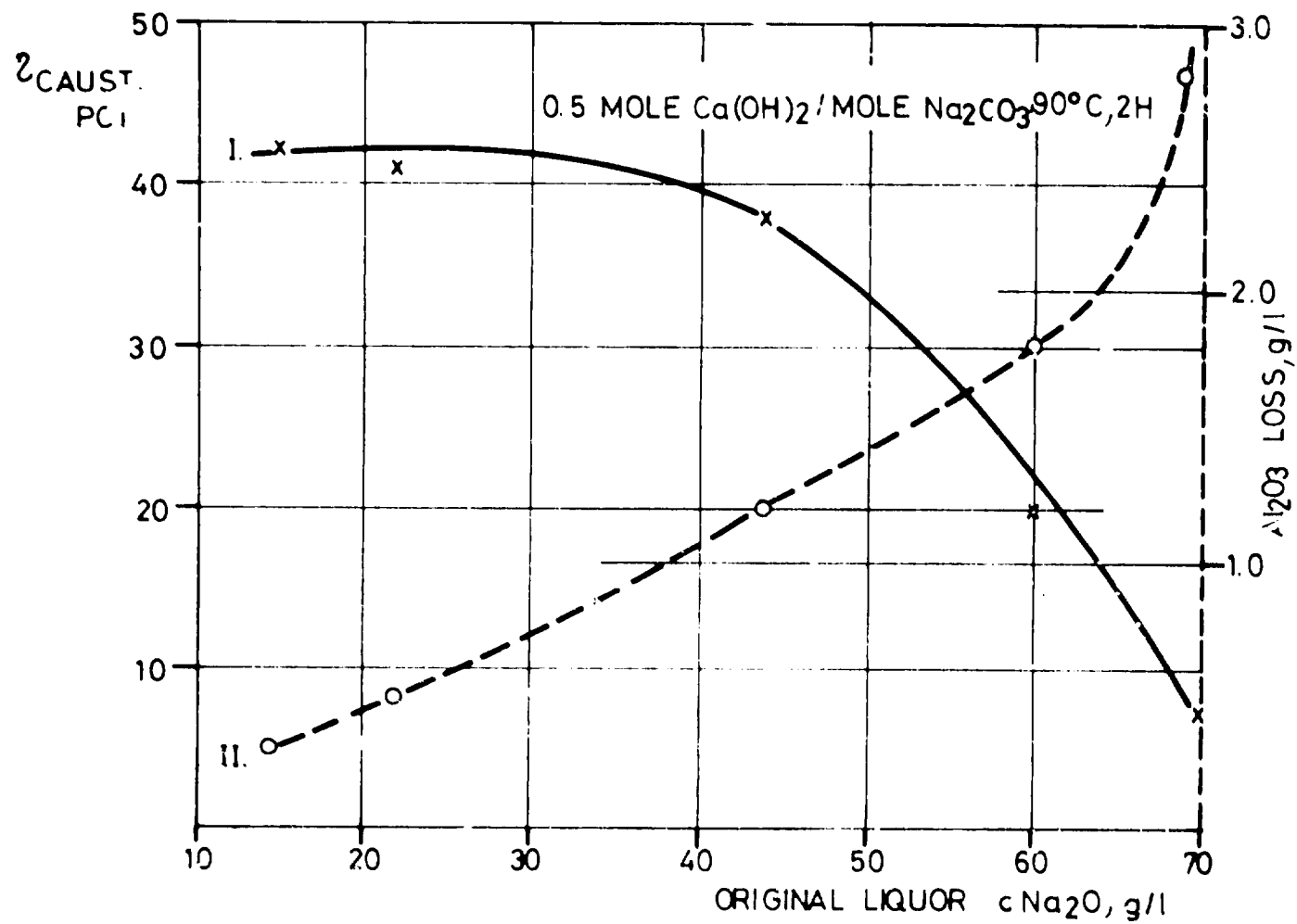


Fig.6.41

CAUSTICIZATION EFFICIENCY (I.) AND  $\text{Al}_2\text{O}_3$  LOSSES (II.) AT DIFFERENT CONCENTRATIONS OF THE WASHING LINE

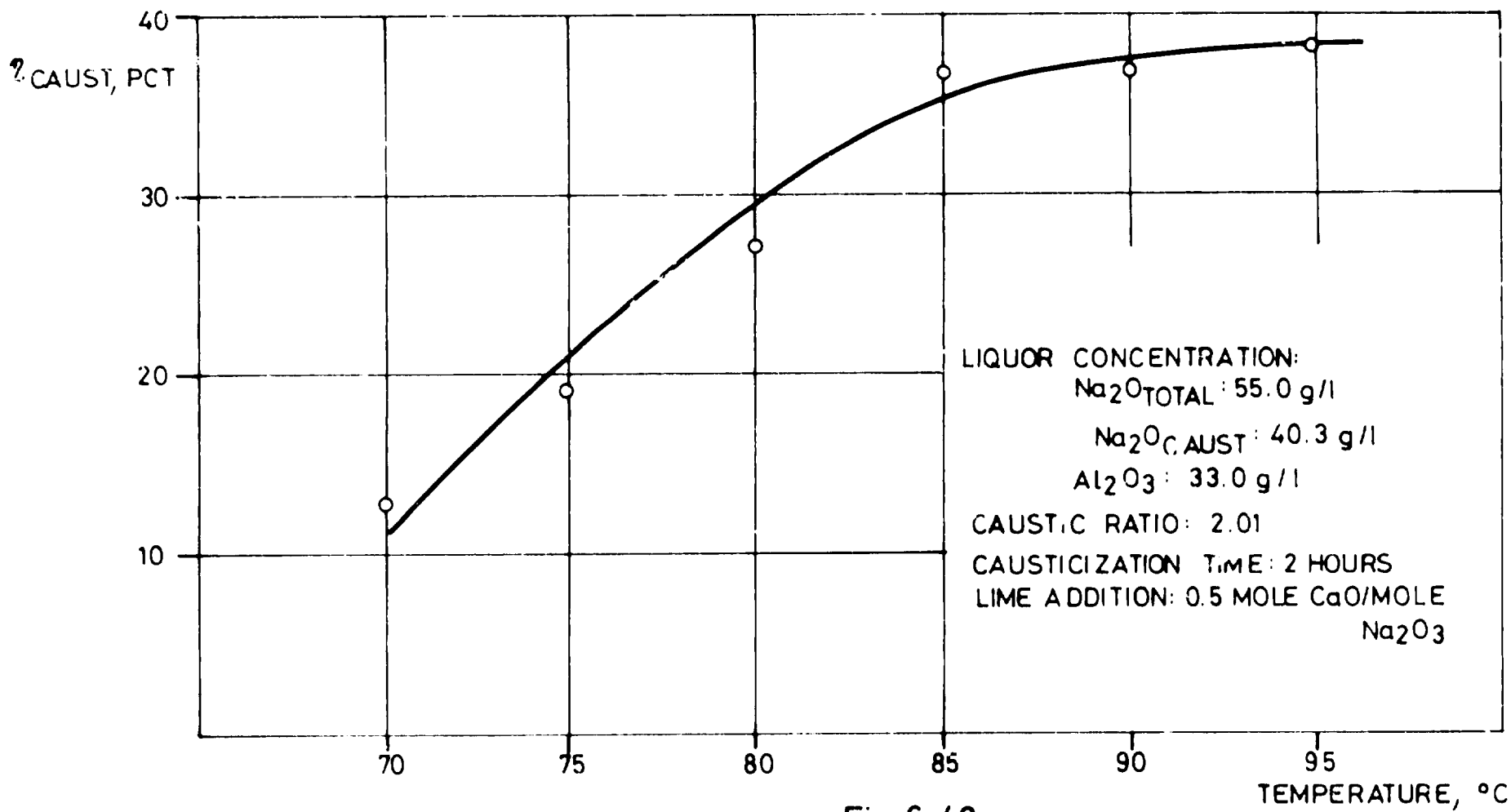


Fig.6.42

CAUSTICIZATION EFFICIENCY vs. TEMPERATURE

These can be classified into three groups on the basis of their technological effect:

- a/ Valuable by-products recovered from the circuit, e.g. vanadate, gallium.
- b/ Contaminants appearing in the alumina and directly affecting its quality, e.g. zinc, phosphorus.
- c/ Contaminants having detrimental effect on different phases of the process, e.g. calcite, dolomite, siderite, pyrite, organic compounds.

The laboratory investigations on contaminants have the following phases:

- quantitative analysis
- dissolving and accumulation tests
- analysis of the effect of contaminant's in certain parts of the process
- elaboration of methods for recovering or removal

#### Quantitative Analysis

The quantitative analysis of contaminants is an integral part of grading of bauxites, however, in case, the contaminant content of the bauxite is low there is no need for further investigations.

#### Dissolution and Accumulation Tests

In case, a certain contaminant occurs in the analysed bauxite in such a large amount, that technological measures have to be taken to remove or to eliminate its effect, the rate of dissolution of the contaminant has to be determined.

Dissolution tests are performed connected to the digestion tests at suggested optimal digestion parameters. On the basis of dissolution tests the technological designer can take

the required technological measures worked out corresponding to the practice into consideration, e.g. designing of soda salt removal and regeneration, calculation of lime addition for  $P_2O_5$  control etc.

In certain cases, the parameters of digestion are to be modified, too, to eliminate or reduce the detrimental effect of a contaminant, e.g. at lower liquor concentration the rate of dissolution is lower or at higher temperature such phase transformations can be realized by which the detrimental effect of the contaminant can be eliminated etc.

The investigation of accumulation of contaminants is a very difficult technical task because the contaminants influence each-other's solubility. Investigations on accumulation can not be carried out in a usual bauxite evaluating laboratory equipped only for batch system investigations, but in pilot plants modelling the whole Bayer circuit. Useful experiences can be gained on contaminant accumulation at starting up of new alumina plants.

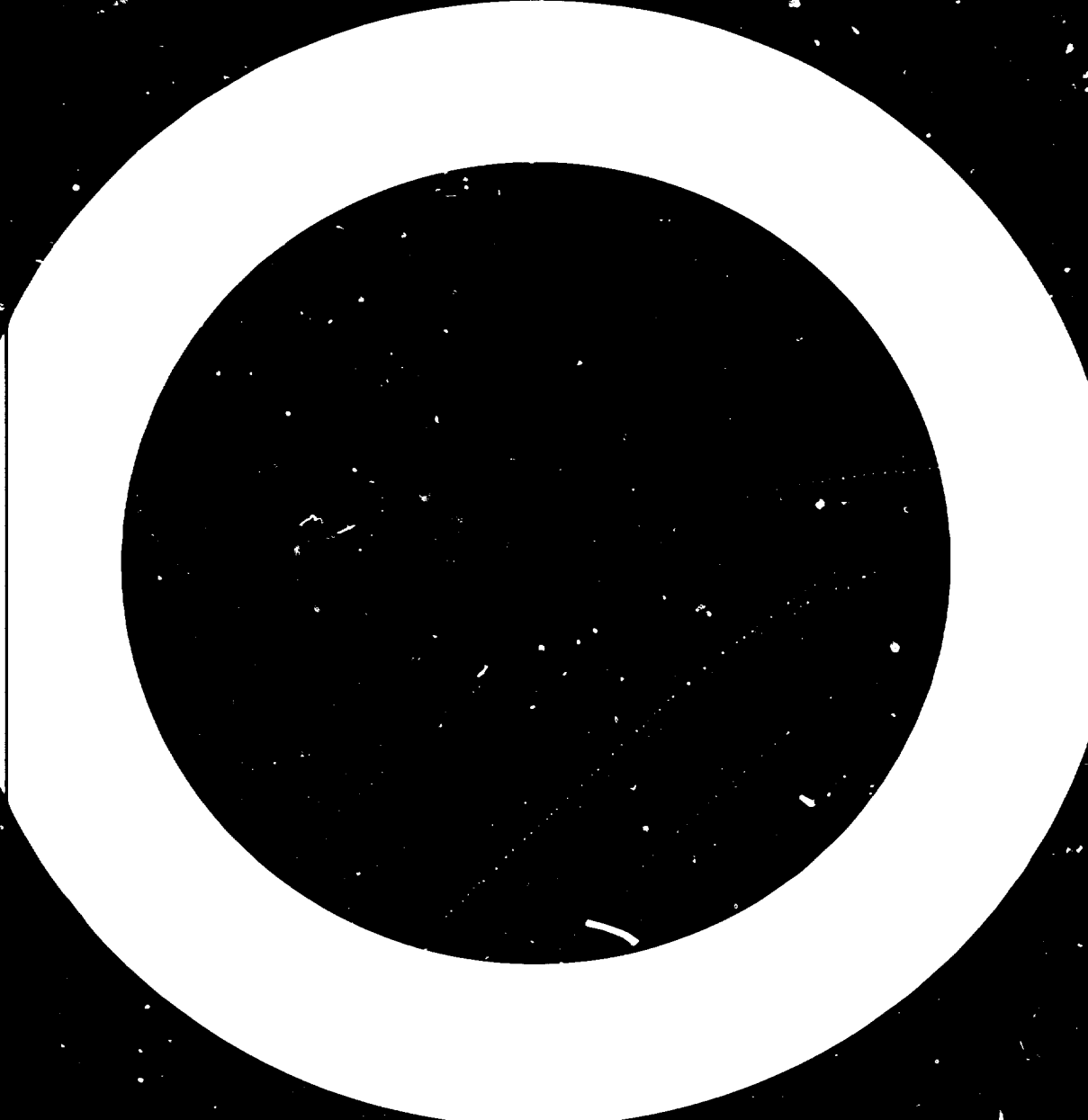
#### Investigation of the Effect of Contaminants in Certain Stages of the Process

The investigated bauxite can have such kinds of contaminants that make the process difficult or, in certain cases, impossible to operate /e.g. high pyrite content spoils settling etc./. In such cases testing the mechanism of the effect of the contaminant, the determination of its acceptable maximum level and the elimination method of its detrimental effect, e.g. previous removal, neutralization inside the process require separate research work.

Studying the Possibilities of Recovery or Removal  
of Contaminants

The recovery of useful components accumulated in the circuit liquor and the removal of contaminants form a separate part of research. For recovering certain contaminants plant technologies are worked out which have to be adapted to the certain plant conditions e.g. recovery of vanadate, soda salt removal, oxalate removal etc.

Though the removal of detrimental organic content is one of the most widely tested problems there is not a single process worked out which could be applied generally in plants. Promising investigations are in progress.



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