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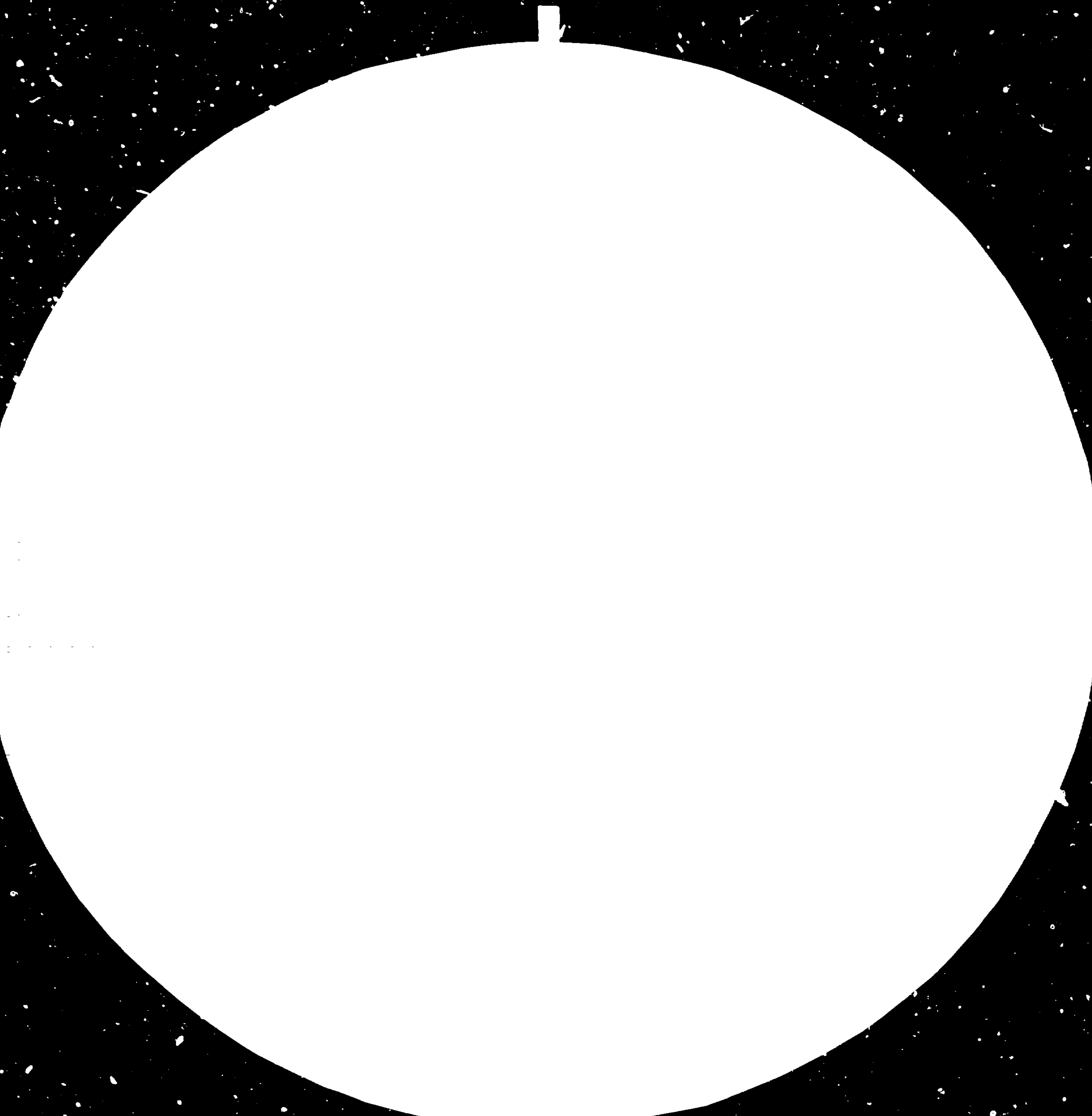

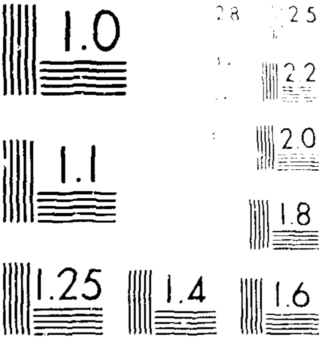
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PROFILE OF TRANSFERRING TECHNOLOGY IN TESTING, INVESTIGATION AND EVALUATION OF BAUXITE

Conducted by a working team of
ALUTERV-FKI, Budapest (Hungary)

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Budapest, July, 1981.

Dr. Károly Solymár

LIST OF ABBREVIATIONS

AAS	-	atomic absorption spectrometry
ABA BEA	-	automatic bauxite analyzer
AT-FKI	-	ALUTERV-FKI (Research, Engineering and Prime Contracting Centre of the Hungarian Aluminium Corporation)
CPU	-	central processor unit
CRT	-	cathode ray tube
DTG	-	differential thermogravimetry
EDAX	-	energy dispersive X-ray analysis
EDL	-	electrodeless discharge lamp
EDTA	-	ethylene diamine tetra acetic acid complex
EPMA	-	electron probe microanalysis
ETA	-	electrothermic atomizer
FS	-	flame spectrometry
HT	-	high tension
HUNGALU	-	Hungarian Aluminium Corporation
ICF	-	intensity conversion factor
IR	-	infra red spectrometry
LOI	-	loss on ignition
PTFE	-	platinium-steel lined
REL-DEV	-	relative deviation
RICF	-	relative intensity conversion factor
$	-	United States dollars
SEM	-	scanning electron microscopy
t	-	metric ton
TAA	-	total available alumina
TEM	-	transmission electron microscopy
TG	-	thermogravimetry
XRD	-	X-ray diffraction
XRF	-	X-ray fluorescence

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EXECUTIVE SUMMARY

0.1. BAUXITE RESERVES OF THE WORLD AND THEIR UTILIZATION

The world's bauxite resources are located mainly in the developing countries while the processing is concentrated in the developed ones. The developing countries aim to build up also processing facilities. Despite of the expected diminution of growth rate during the next decade, the aluminium industry will remain a potentially attractive area to build new capacities. For locating a new alumina plant, the following factors are to be considered:

(1) Shipping charges have become a more and more critical cost factor.

(2) The bauxite producing countries would benefit from significant shifts of the processing towards more valuable products.

(3) The developing countries should aim at a better control over mining and processing.

Consequently it might be expected that alumina plants will be located most frequently in close proximity of mining areas.

Bauxite testing laboratories have an important role in the economic utilizations of the resources.

0.2. THE FUNCTIONS OF BAUXITE TESTING LABORATORIES

There are different levels of bauxite testing laboratories according to their functions and complexities:

(1) The most simple service laboratory for geological exploration and mining (level A) performs bauxite analysis, in such a way that it enables the country to determine qualitatively and quantitatively the ore resources and promotes the economical marketing.

(2) The laboratory for technological evaluation of ores (level B) is a laboratory of level A, completed by the technological operations, i.e. modelling the recovery of alumina from bauxite.

This type of laboratories may be installed independently or connected to an alumina plant or an existing research laboratory.

(3) Complete research laboratory for bauxite testing and alumina technology (level C). This has the following further functions compared to the laboratory of level B: modelling of processing stages of the Bayer process on laboratory, large laboratory and pilot plant scale for the established alumina industry; preparation of feasibility studies for the future alumina plants; elaboration and optimization of different procedures and analytical methods.

0.3. ALTERNATIVES FOR SETTING UP BAUXITE TESTING LABORATORIES

The following conditions have to be considered: selection of the most reasonable methods and equipment, application of the "building block" system, estimation of the amount of required analytical work (inclusive the technological investigations), the justification or not of large scale laboratory and/or pilot plant investigations.

The main target of a pilot plant is to test the critical stages of the whole process and on the other hand to test equipment of new principles. Two types of pilot plants can be established, namely a pilot plant characterized by a high flexibility of parameters in each stage of the process, suitable for studying very different kinds of bauxite and another one where this flexibility is given for some selected processing steps only. Large scale laboratories and pilot plants are operated in a close circuit what is specially favourable to study the enrichment of contaminants, and the consequences thereof. Pilot plants should not be projected but individually according to specified conditions.

0.4. THE MAIN UNITS OF A BAUXITE TESTING LABORATORY

The activities of bauxite laboratories can be classified into three main groups independently of the level of their complexity:

- chemical analysis
- technological operations
- physical and physico-chemical investigations (including mineralogical analysis and texture analysis).

The following laboratory units are discussed in details referring to their functions, activities and requirements for equipment and human resources: analytical chemical units (sample preparation and storage, AAS and FS laboratory, wet chemical laboratory, laboratory for other instrumental methods); X-ray laboratory (XRF and XRD); laboratory for technological operations (sample preparation and tests of grindability, modelling of bauxite digestion and red mud settling - so-called "red laboratory"-, precipitation of $Al(OH)_3$ and its calcination to alumina - so-called "white laboratory") - laboratory for studying effects caused by bauxite contaminants; units for physical and physico-chemical investigations (laboratory for simple physical and physico-chemical investigations to determine density and volumetric density, grain-size distributions, specific surface area, porosity and pore size distribution etc. of bauxite, hydrate, red mud and alumina samples; laboratory for thermogravimetry (TG), laboratory for infrared spectrometry (IR), laboratory for textural investigations applying light microscopy, transmission electron microscopy (TEM), scanning electron microscopy (SEM) supplemented by energy dispersive X-ray analysis (EDAX) and electron probe microanalysis (EPMA)).

0.5. ACTIVITIES OF BAUXITE TESTING LABORATORIES

On the basis of the previous discussion the above-mentioned activities and functions of the bauxite testing laboratories are summarized for the typical levels and simultaneously the main equipment required with estimated costs and human resources are given.

0.6. REQUIREMENTS FOR EQUIPMENT, BUILDING AND HUMAN RESOURCES

The layouts and equipment lists are demonstrated for characteristic A level and B level laboratories, respectively. All levels of laboratories require similar infrastructure: water supply, electricity, canalization, gas supply, compressed air, vacuum and hot water networks. If the first three are missing no laboratory can be operated.

0.7. TRAINING REQUIREMENT

Supposing the availability of graduated specialists for the bauxite testing laboratories special training is required to introduce them into the organization and operation of such kind of a laboratory. Two kinds of training might be proposed:

- a general review and laboratory practices covering the whole activity
- special courses in a particular investigation technique.

Both kinds of training programs consist of two steps:

- firstly a few selected experts are trained (in form of group training) in the research center of the technology transferring institution
- in the second stage a course is organized in the installed new laboratory under on-site supervision of the experts invited.

0.8. COST ESTIMATIONS FOR SETTING UP ALTERNATIVE TYPES OF BAUXITE TESTING LABORATORIES

Informative figures for the costs of different levels of laboratories can be given as follows:

	x 1000 US$
A level laboratory	500- 1,000
B level laboratory	1,000- 3,000
C level laboratory	3,000- 5,000
Large scale laboratory with closed circuit	500- 1,000
Pilot plant with closed circuit	5,000-10,000

0.9. RECOMMENDATIONS

This profile serves as a guide to select the task and level of the bauxite testing laboratory in the given developing country. If the decision is passed, a detailed project and cost calculation must be prepared by a consulting organization. It seems desirable to set up a complete bauxite testing laboratory step by step corresponding to the required activity and financial possibilities.

Preferably, all activities connected with the setting up of the laboratory should be performed by the same organization.

APPENDIX

In the appendix there are given outlines for training programs and some examples for the technological evaluation of different kinds of bauxite by means of the discussed chemical, mineralogical, physical and technological investigations and a list of selected method of chemical analysis as well.

INTRODUCTION

The world's prospective bauxite resources are located mainly in the developing countries while the processing is concentrated in the developed ones. Thus, a claim for thorough knowledge of bauxite deposits as a prerequisite of establishing the national processing industries in developing countries is fully justified.

At the first meeting of the Expert Consulting Group on the Aluminium Industry organized by UNIDO in 1967 in Vienna [1], recommendations to UNIDO were accepted which include: "Organize in some interested developing countries installation of specialized laboratories dealing with bauxite analysis and laboratory scale technological tests, taking into view the possibility of utilization of the facilities of existing universities and institutions. (This may be of interest to a number of developing countries with bauxite deposits.)"

To realize these recommendations a study was prepared in the subject "Analytical methods for testing bauxite, alumina and intermediate products" [2] and published by UNIDO in 1973.

As a next step in this framework UNIDO organized a Group Training Concerning the Production of Alumina in 1979 in Budapest (in co-operation with the Hungarian Aluminium Corporation). This training cosisted of a theoretical part and of laboratory practice. The topics outlined by the lectures as well as a manual for the laboratory investigations were printed in 8 volumes, altogether [3].

The present profile is based on the above-mentioned publications as a continuation of this activity. However, the problems being discussed here, take into account the latest innovations and equipment for the chemical, mineralogical and technological investigations, and evaluation of bauxite in order to be made available to all developing countries possessing bauxite deposits being or planned to be mined and processed.

In broad terms the profile reviews the distribution of bauxite deposits in the world and the trends of their processing, the functions, performances and activities of bauxite testing laboratories, relevant cost estimations and requirements for training programs. In the Appendix a method for technological evaluation of bauxite, a list of the standard and suggested methods for chemical analysis and proposed topics for a training program can be found.

1. RESERVES AND UTILIZATION OF BAUXITE THROUGHOUT THE WORLD

1.1. DISTRIBUTION OF BAUXITE DEPOSITS IN THE WORLD

The bauxite production of the world in 1980 amounted to 81,4 million tons [4] while de Weisse estimated the total bauxite reserves at 8 billion tons in 1972 [5], and Lotze in 1978 stated already 38 billion tons [6], for the same. Lotze describes, however, that carrying out an overall examination of the specific bauxite costs in the alumina production, including the mining and transportation costs, merely 12 % of the total reserves fall into the lowest cost category presently processed. As much as 95 % of these is found in Jamaica, Guinea, Australia, Surinam and Greece.

In several cases the specific costs of the ore increased significantly as a consequence of the geographical location of the reserve, the poor infrastructure and the long transportation distances. The classification system for the world's bauxite resources according to J. Lotze is shown in Table 1. [6] The estimated bauxite resources classified for economic evaluation following this system are represented in Table 2. [6]

Africa forms a major bauxite region, however, the single major bauxite producing country is Guinea while the reserves/resources of Ghana, and Cameroon are also significant on a world scale.

In America Brazil, Surinam and Colombia have also significant resources beside the well known Jamaican ones.

According to the data and the prognostic prospecting developing countries have abundant bauxite resources and the aim to build up their own processing facilities seems to be justified.

Table 1

CLASSIFICATION SYSTEM FOR BAUXITE RESOURCES
(J. Lotze, Ref. 6.)

Resources			
Developed		Undeveloped	
Mineable Reserves	Potential Ores	Reserves	Potential Ores

<u>Definitions</u>:

<u>Resources</u>: Concentration of bauxite in or on the Earth's crust in such form that economic exploration is currently or potentially feasible. Resources = Reserves + Potential Ores.

<u>Developed Resources</u>: Bauxite deposits/areas currently under exploitation.

<u>Undeveloped Resources</u>: Known bauxite deposits/areas of bauxite, from which an economical exploitation can be expected in future.

<u>Reserves</u>: That portion of resources from which bauxite is currently economically exploited under existing conditions, including cost, quality, geologic evidence and technology (category: Mineable Reserves of Developed Resources) or economical exploitation will be expected in future (category: Reserves of Undeveloped Recources).

<u>Potential Ores</u>: That portion of resources in the continuity of known deposits which are insufficiently explored at this time and for which quantitative estimates are based largely on broad knowledge (category: Potential Ores of Developed Resources) or that portion of subeconomic resources which may become reserves as a result of changes in economic conditions or after further exploration (category: Potential Ores of Undeveloped Resources).

Table 2

ESTIMATED BAUXITE RESOURCES CLASSIFIED FOR ECONOMIC EVALUATION

(millions of metric tons) (J. Lotze, Ref. 6.)

Deposits	Resources				
	Developed		Undeveloped		Total
	Mineable Reserves	Potential Ores	Reserves	Potential Ores	
Total Australia	1,215	1,800	830	2,180	6,025
Guinea	1,430	4,000	2,950	1,000	9,380
Cameroon	-	-	680	1,200	1,880
Other Africa	60	-	650	830	1,540
Total Africa	1,490	4,000	4,280	3,030	12,800
Brazil	70	-	1,350	1,900	3,320
Jamaica	2,000	-	-	1,000	3,000
Surinam	200	-	200	1,570	1,970
Colombia	-	-	115	905	1,020
Other America	200	250	50	1,535	2,035
Total America	2,470	250	1,715	6,910	11,345
India	50	-	1,010	1,495	2,555
Indonesia	40	40	-	1,000	1,080
Other Asia	35	5	100	845	985
Total Asia	125	45	1,110	3,340	4,620
Total Europe	840	350	-	280	1,470
Total Western world	6,140	6,445	7,935	15,470	36,260
Total Eastern countries					1,965
World Total					38,225

1.2. TRENDS IN BAUXITE PROCESSING

The world market of bauxite is dominated by very large scale mines in the more or less immediate proximity of large capacity tidewater ports.

The distribution of bauxite production in the West for the years 1950-1965-1978 is represented in Table 3.

Table 3

DISTRIBUTION OF WESTERN WORLD'S BAUXITE PRODUCTION,
1950, 1965 AND 1978, IN PERCENTAGE
(Thomas G. Langton, Ref.4.)

	1950	1965	1978
Guinea	0.2	6.1	16.5
Australia	neg	3.8	33.3
Jamaica	-	28.3	16.1
Surinam	29.4	14.2	7.2
Guyana	22.9	9.5	4.7
United States	19.3	5.5	2.3
France	11.4	8.6	2.7
Brazil	0.3	0.6	1.7
Other	16.5	23.4	15.5
Total	100.0	100.0	100.0

Table 3 indicates that the most significant trend of the fifties had been the rapid ascension of Jamaica as a major world supplier of bauxite. Similar increas happened later in Guinea.

In 1978 Australia took over as the largest bauxite producing nation (33,3 %) and Surinam has dropped from first to fourth between 1950 and 1978.

These shifts have resulted from changes in the relative production costs. It is expected that the share of such relatively new producers like Australia and Brazil will increase during the following decade.

The bauxite mining and processing capacities in 1978 and their expected increments up to 1984 as well as the relative capacities of processing to alumina in 1978/1984 is compiled in Table 4 adopting the data of Mr B. Balkay. [7]

It can be seen that the processing of bauxite is especially limited in Africa, and in Latin America.

The predicted primary aluminium consumption, alumina and bauxite production of the world is shown in Table 5 (data of Chase Econometrics). [4]

Despite of the expected diminution of growth rate during the next decade, the aluminium industry will remain a potentially attractive area for investments. For locating new alumina plants the following factors are to be considered:

(1) Shipping charges have turned to be an increasingly critical cost factor.

(2) The bauxite producing countries would benefit from enhanced processing to more valuable products.

(3) The developing countries should aim at a better control over mining and processing.

Consequently if can be expected that alumina refineries will be located more frequently than not closely to mining sites.

Table 4

BAUXITE MINING AND PROCESSING CAPACITIES, 1978, EXPECTED INCREMENTS UP TO 1984, RELATIVE CAPACITIES OF PROCESSING, 1978/1984

(Balkay, Ref. 7.)

Region	Bauxite mining			Alumina vs. bauxite capacity ratios		Alumina refining		
	Capacity 1978 '000 t	Increment up to 1984 '000 t	Percentage of w. incrmt.	1978	1984	Capacity 1978 '000 t	Incrmt. up to 1984 '000 t	Percentage of world incrmt.
World	87,297	23,523	100.0	1.00	1.00	30,195	7,220	100.0
Africa	14,262	6,945	29.5	0.14	0.10	700	-	0.0
Asia without Japan	3,756	105	0 4	0.86	1.27	1,120	540	7.5
Japan	-	-	-	-	-	2,580	-	0.0
Latin America	30,449	11,200	47.6	0.47	0.49	4,990	1,882	26.1
Oceania	28,000	3,245	13.8	0.70	0.81	6,800	1,700	23.5
Europe	8,680	2,040	8.7	1.89	2.39	5,675	2,963	41.0
N-America	2,150	-	-	11.20	11.66	8,330	135	1.9

Table 5

ALUMINIUM CONSUMPTION, 1980-1990 (millions of tonnes and %) (estimated by T.G. Langton, Ref. 4)

	1980	1985	1990	% Growth/year 1980-90.
World Primary Aluminium Consumption	12.15	15.41	19.59	4.9
World Alumina Production	27.9	34.7	44.1	4.7
World Bauxite Production	81.4	103.4	134.3	5.1

1.3. CHARACTERISTICS OF BAUXITE PROCESSING TECHNOLOGY

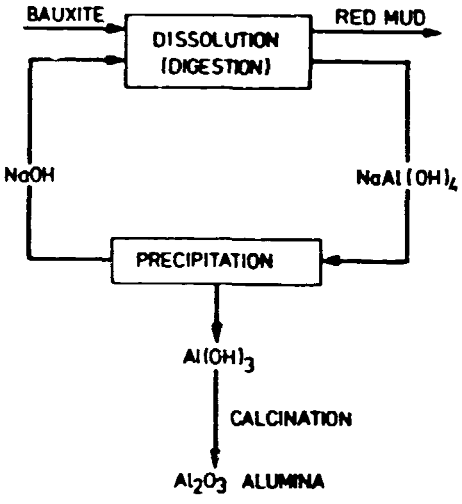
At present, more than 90 % of the world's alumina is produced by Bayer process, providing high purity final product.

Alumina production is a beneficiation process releasing Al_2O_3 content of bauxite from other accompanying oxides. This alumina is suitable for electrolysis in a cryolite melt.

The industrial practice of the Bayer technology is based on the following reaction:

$$\underset{\text{solid gibbsite}}{Al(OH)_3} + NaOH \underset{\substack{< 100\ ^{o}C \\ \text{precipitation}}}{\overset{\substack{\text{digestion} \\ > 100\ ^{o}C}}{\rightleftarrows}} \underset{\text{dissolved in liquor}}{NaAl(OH)_4}$$

The Bayer process consists essentially in digesting the crushed and grined bauxite with strong caustic soda solution at temperatures above 100 °C (for gibbsitic bauxites 100-140 °C, for boehmitic and diasporic bauxites 200-250 °C). After separation from the residue (red mud) and cooling the dissolved alumina is seeded with crystallites of trihydrate whereupon precipitation takes place. The precipitated trihydrate is filtered and calcined at about 1,200 °C. The simplified flow sheet is shown in Fig. 1.



SIMPLIFIED FLOW-SHEET OF THE BAYER PROCESS

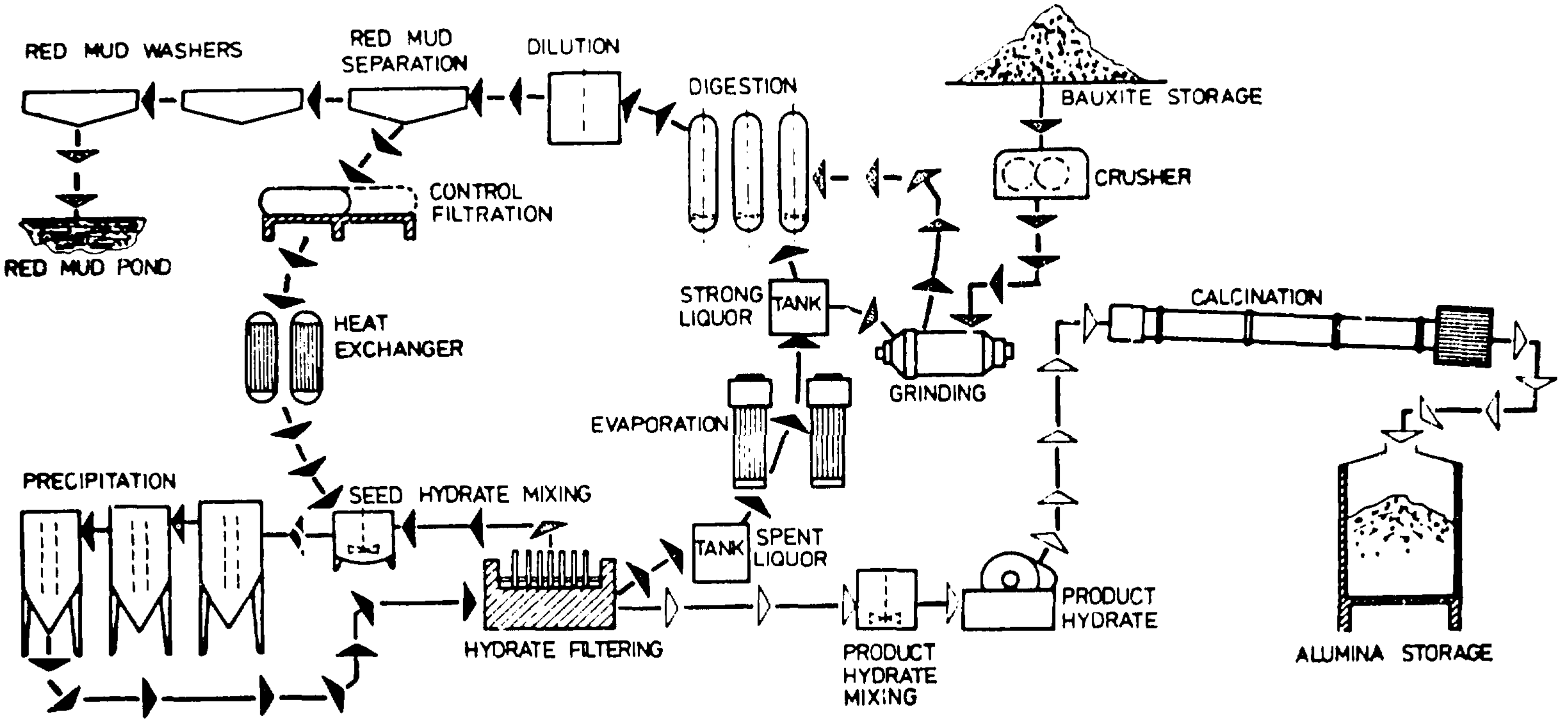
FIG. 1.

A scheme of material-streams through the main operation units is illustrated in Fig. 2. [8].

1.4. THE ROLE OF TESTING LABORATORIES IN THE UTILIZATION OF BAUXITE RESOURCES

The first step to utilize a bauxite reserve is the prospecting and exact evaluation of given resources. This work must be supported by a well organized bauxite testing laboratory in which the chemical and mineralogical composition of a large number of bore-hole samples can be determined. Some technological investigations might also be often required for an economical marketing.

If processing at the site is planned the activity of the laboratory must be extended to technological tests, not only on laboratory scale but - concerning the critical technological units - on pilot scale as well.



SCHEME OF THE BAYER-PROCESS
(by Lepori, Ref. 8)

FIG. 2

The most advanced variety of bauxite testing laboratories serves as a research center for the alumina industry and could be used even to train the staff of alumina plants, and should be installed near bauxite deposits or connected with alumina plants.

Such a research center could be erected either independently or as part of existing research institutes, university departments or other institutions. The latter form has the advantage that the instruments of high value can find effective application in various fields and the laboratory can perform educational tasks, too.

2. THE FUNCTIONS OF BAUXITE TESTING LABORATORIES

Before entering into details a brief survey is given on the functions of bauxite testing laboratories proceeding from the simplest to the more advanced type. Due to specific circumstances there is an overlapping in functions of the three main types of laboratories.

2.1. SERVICE LABORATORY FOR GEOLOGICAL EXPLORATION AND MINING (LEVEL A)

This simplest type of laboratory performs analyses necessary for bauxite prospecting and mining. It may be located on site or form a part of an existing laboratory or educational institute. This laboratory enables the producing countries to determine qualitatively and quantitatively their ore resources. Analyses are usually confined to the main components of the bauxite, i.e. - according to the earlier international practice - loss on ignition, alumina, silica, iron-oxide and titanium-oxide.

More recently the range of bauxite components considered has been extended to the contaminants substantially influencing the processing, i.e. CaO, MgO, P_2O_5, V_2O_5, ZnO, Ga_2O_5, carbonates, sulfates, pyrite, organics, etc. These contaminants are taken into account when determining the industrial value and price of a given ore.

The increase in number of the required analyses and the growing complexity of the same led to a step by step replacement of the classical chemical analytical methods by automatic, fast and highly reproducible methods (e.g. X-ray fluorescence spectrometry).

The task of geological laboratories has broadened recently by mineralogical analysis. Earlier, simple methods, like thermogravimetric measurements (TG, DTG, DTA) were applied, however, currently the quantitative phase analysis of bauxite is based on X-ray diffractometry. In this type of laboratories fundamental tests of digestibility are required as well (i.e. determination of Al_2O_3 content soluble in caustic soda under specific technological conditions, expressed in % of the total Al_2O_3 content).

2.2. TECHNOLOGICAL EVALUATION OF ORES (LEVEL B)

Some technological investigations are necessary also from the point of view of marketing especially if the quality of bauxite varies considerable in chemical and/or mineralogical composition.

The technological laboratory is engaged with technological tests, primarily digestion tests and settling tests of the obtained red mud in order to determine the industrial value of ores reflected by data like Al_2O_3 recovery, digestion parameters, expected soda losses and separability/washability of the mud. These measurements permit to follow the effects of quality variations on processing.

The chemical and mineralogical analysis of red mud and the analysis of aluminate liquors are included in the scope of this type of bauxite laboratories as well.

Beside of monitoring the industrial quality of the ore being delivered, the laboratory should collect information about the technological behavior of the bauxite, and secure qualified experts to co-operate in establishing the national ore processing facilities in the country.

These laboratories help the ore producing countries also to control the processing of bauxite in existing plants operated by international companies.

The facilities may be installed independently or joined to an alumina plant or running research laboratory.

2.3. COMPLETE RESEARCH LABORATORY FOR BAUXITE TESTING AND ALUMINA TECHNOLOGY (LEVEL C)

Beyond the previous two functions this type of laboratory has the following further functions: modelling of processing phases of the Bayer-cycle on laboratory and pilot plant scale for the established alumina industry; preparation of feasibility studies for future alumina plants; elaboration and optimization of different procedures and analytical methods.

The studies related to bauxite processing (based on Bayer-cycle) can be carried out on four different scales:

(1) Laboratory scale (few grams to few kgs)
(2) Large laboratory scale (few kgs to few hundred kgs)
(3) Pilot plant scale (magnitude of several tons)
(4) Industrial scale

The laboratory scale experiments are generally arranged in batches, while large laboratory and pilot plants are operated in close circuits, therefore, the laboratory scale equipment can be located independently of production but in the case of a large laboratory or a pilot plant it seems advisable to seek the proximity of an alumina plant.

On the other hand the building of a pilot plant can introduce the installation of an industrial facility (especially in case of irregular ore behaviour) through the determination of optimal technology and by training the presonnel of the future alumina plant.

The scale of the technological tests has to be considered in respect of the problem to be solved. It is necessary to determine on an individual basis whether it is preferable to establish a more flexible and by far less expensive large scale laboratory or a pilot plant. In addition it should be emphasized that large scale laboratories are adequate for studying in general terms all problems arising in the Bayer process.

Pilot plants might provide for the testing of new or newly modified processing equipment what is strongly recommended in advance of industrial application.

3. ALTERNATIVES FOR SETTING UP BAUXITE TESTING LABORATORIES

The following guidelines should be observed when setting up a bauxite testing laboratory:

- select appropriate methods and equipment corresponding to the number of analyses and to the amount of technological investigations as well as to the qualification of the personnel, taking into account the required maintenance, the related cost, the productivity and the accuracy;
- follow the "building block" system and set up a high level research laboratory starting with a simple service laboratory for geological exploration;
- examine the necessity of establishing a pilot plant and if such a need is proved determine carefully its scale and the problems to be examined;

3.1. SELECTION OF THE METHODS AND EQUIPMENT APPLIED FOR ANALYTICAL TESTS

In the organization of an industrial laboratory one of the most important factors is the economy of the services. According to the required performance and the general technical level of the analytical chemistry in the particular country three different kinds of the analytical laboratories can be organized.

(1) Simple service laboratory for geological exploration

- Task: to determine the main chemical components (and some specially interesting impurities) of bauxite and other sedimentary rocks by simple, fast, reproducible and accurate methods. The productivity and the saving of qualified working power are very important factors.
- Solution:
  - a) X-ray fluorescence spectrometry (quantometer)
  - b) automated AA (atomic absorption) spectrometry
  - c) combination of different nuclear, thermometric and potentiometric methods (mechanized instruments)
  - d) classical wet chemical methods

(2) Control and analysis laboratory supporting bauxite mining and technological operations

- Task: as mentioned in (1) plus the chemical analysis of the aluminate liquor as well as that of the red mud. Standardization and supervision of the monitors built in the technological process. In the case of technological troubles the analysis of many samples can be required for specially influencing trace elements (e.g. F^-, Zn^{++}, organic materials etc.).
- Solution:
  - a) combination of automated X-ray fluorescence spectrometric and X-ray diffractometric methods as well as thermometric, potentiometric, spectrophotometric and some wet chemical methods
  - b) combination of microprocessor controlled AA spectrometrix X-ray diffractometric methods as well as thermometric, potentiometric, spectrophotometric and some classical wet chemical methods

(3) Research laboratory for mining and alumina production

- Task: as mentioned in (2) plus analysis of all relevant components of the raw materials, products and by-products of alumina technology. Besides, some research and development should also be carried out in the field of analytical chemistry in order to develop methods for monitoring and fast control. Up-to-date awareness of the internationally used standard methods and of the requirements regarding quality is very important, too.
- Solution: combination of computer controlled X-ray fluorescence spectrometric and diffractometric methods, AAS and spectrophotometry, electroanalytical methods (especially potentiometry and oscillometry), thermometric methods and some classical wet chemical methods.

The above-mentioned laboratories of different levels can be developed step by step or put up at once according to the requirements of the alumina production. In the case of gradual development the reorganization of the staff of the laboratory is one of the most important factors for the effective operation.

3.2. SELECTION OF METHODS AND EQUIPMENT FOR TECHNOLOGICAL TESTS

The criteria for selecting the methods and equipment are determined by the data needed to evaluate representative bauxite samples from the point of view of feasibility studies. The program involves the selection of alumina processing technology on the basis of the technological investigation. The main groups of the technological tests are the following:

- crushing and grinding tests
- predesilication tests
- digestion tests
- red mud settling tests
- determination of the effect of contaminants.

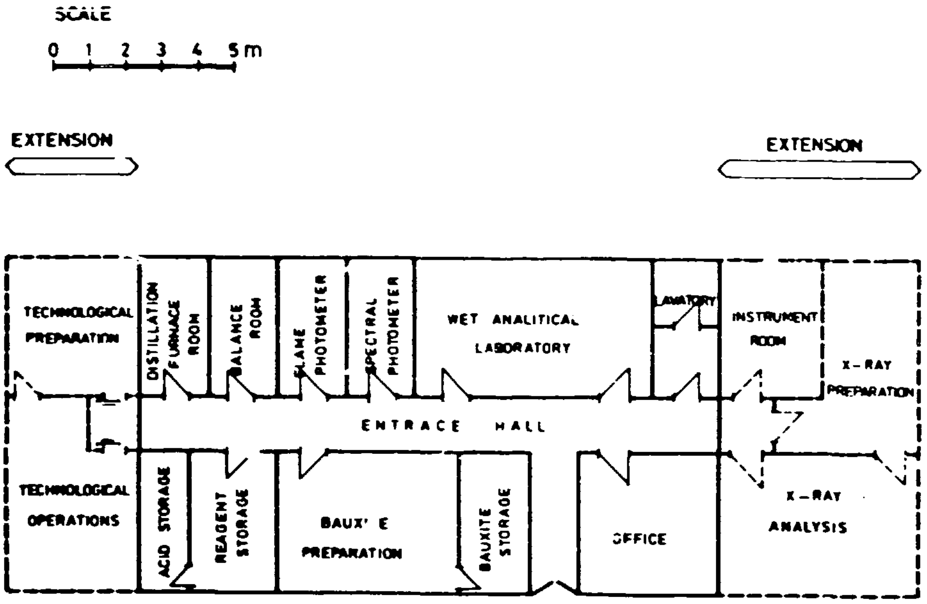
All of these are discussed only for high grade bauxite because low grade bauxite can not be used economically for the Bayer process.

Unfortunately the commercial offer for these technological apparatus is rather poor (e.g. a few types of laboratory autoclaves are available), therefore, dedicated equipment had to be developed by the research centers of aluminium companies. Purchasing of these products is recommended from the very institutions producing the original models.

3.3. APPLICATION OF THE "BUILDING BLOCK" PRINCIPLE

Due to the requirements, the finite capacity of the financial sources and the usually gradual development of bauxite-alumina industry it is reasonable to estabilish the bauxite laboratories step by step, using the "building block" principle.

An example for a very flexible outlay allowing stepwise enlargement of the laboratory from the simplest type to higher levels is shown in Figure 3. The basic sketch of the geological exploration laboratory, based on the classical wet analytical method, using flame photometry and spectral photometry only, is characterized by a central entrance hall along the length of the building. Thus, enlargement is easy by adding further rooms at each end. The bauxite testing laboratory, suitable for technological investigations, too, can be obtained from the simple type by attaching two rooms at the



TYPICAL LAYOUT OF AN „A" LEVEL LABORATORY

FIG.3.

left end (rooms for technological operations). At the right end of the building a sample preparation room, an X-ray analysis room and an instrument room can be added.

3.4. CONSIDERATIONS FOR SETTING UP A PILOT PLANT

Although 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, components of the process line must not be under- or oversized. On the other hand, using new deposits, unknown types of ores have to be processed without relevant plant experiences in advance.

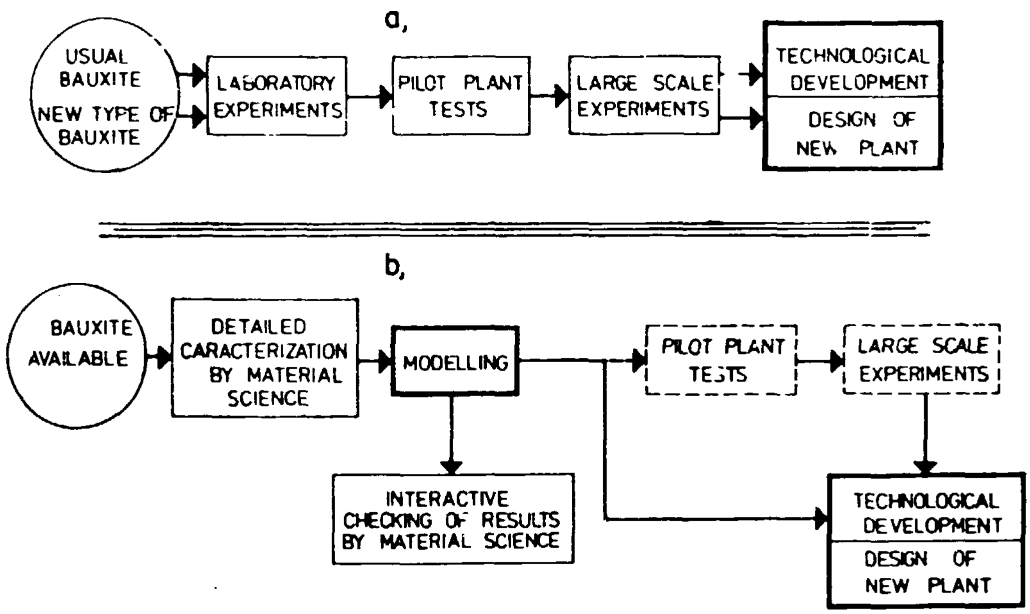
These difficulties can be reduced by running pilot plants. Experiences gained in pilot plants are also suitable for designing the start-up of a new plant, for training the staff, saving thereby considerable expenses. E.g. Swiss Aluminium Ltd. designed an alumina plant of 1 million ton per year capacity, located on the bauxite deposit at Gove (Australia), exploiting the experiences collected in a pilot plant set up partly for that purpose. The ratio between the scale of the pilot plant and that of the industrial plant was about 1:1000.

However, since constructing and operating a pilot plant is fairly expensive, in critical cases in order to save time, very frequently the parameters of the plant are determined by the way of laboratory investigations and then excess capacities of machinery and equipment are designed at the critical places.

Model tests are carried out on laboratory scale. Results of some investigations can be directly used in the design work and during the operation of even 1,000 times greater plant machinery and equipment. Investigations of processes like fluidization, crushing, grinding, digestion, etc. can be listed in this group. However, in many cases problems are met when switching-over from laboratory to pilot plant scale and these become even more pronounced when transferring the results to plant scale. In these cases laboratory results can be evaluated only as reflecting trends and they can not be considered as absolute figures. Only after accumulating appropriate practice can the data resulting from laboratory and pilot plant tests 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 Figure 4. the schemes of elaboration of optimum technology by means of the former philisophy and by up-to-date methods are shown. In part a) of the figure the test material is submitted to laboratory and pilot plant examinations for



THE SCHEME OF ELABORATION OF OPTIMUM TECHNOLOGY BY MEANS OF FORMER (a) AND UP-TO-DATE (b) METHODS, RESPECTIVELY FIG.4.

improving 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 in many cases by applying (mathematical) simulation of the processes. The deviations among the results of laboratory, pilot plant and large scale experiments can be eliminated by means of modelling.

Although a pilot plant is operated continuously and in a close circuit it is not a "mini" Bayer plant in respect of the scale of the applied equipment. The main target is to test the critical stages of the whole process and on the other hand to examine equipment based on new principles. As a consequence two types of pilot plants can be constructed, namely a pilot plant characterized by a wide range of variability of parameters (temperature, holding time, capacity etc.) in each stage of the process suitable for studying very different kinds of bauxite and another one where this flexibility is confined to some preselected processing steps only. In developing countries the latter type is more reasonable for its lower costs without serious limitations.

If a pilot plant can be connected to a plant is should not operate in a close circuit but preferably form a by-pass to permit the examination of critical stages in the industrial plant.

A close circuit is specially favourable to study the effects of the enrichment of contaminants. Because of varying local conditions and technological requirements (i.e. alumina quality specifications) pilot plants should be designed individually.

3.5. CHEMICAL CONTROL OF TECHNOLOGICAL INVESTIGATIONS

The analytical part of the bauxite laboratory must be fitted to the demands of the technological investigations, including large laboratory and pilot plant scale if they are located at the same site. The automation of chemical analysis should be adjusted to the number and complexity of the analyses used to control technological tests. Taking into consideration that bauxite testing laboratories are usually extended step by step it is advisable to reckon already in the first stage with the future requirements to be set from the side of technological investigations going to be introduced.

If the pilot plant is planned to be set up near an existing alumina plant the corresponding process control analyses should be made expediently in the industrial process control laboratory.

It is recommended to test newly developed process control instruments first in pilot plants.

4. THE MAIN UNITS OF A BAUXITE TESTING LABORATORY

In previous chapters the functions of bauxite laboratories and alternatives for their setting up were thoroughly discussed emphasizing that a step by step development is recommended from simple laboratories to high level facilities.

The activities of all bauxite testing laboratories can be classified into four main groups:

- chemical analysis
- X-ray investigations (XRF, XRD)
- technological operations
- physical and physico-chemical investigations (mineralogical and texture analysis)

These main activities may be performed in laboratories organized in different ways, but usually the following laboratories (rooms) are provided: storage room for samples, storage room for reagents, sample preparation room(s), balance room, distillation and furnace room, wet chemical laboratory, spectrophotometer room, atomic absorption and flame spectrometry room, laboratory for other analytical instruments, X-ray laboratory (sample preparation, XRF, XRD), preparatory room for technological experiments, laboratories of technological operations) rooms for digestion and settling; precipitation, calcination, purification of aluminate solutions, laboratory for physical investigations, room for thermogravimetry, infra-red spectrometry room, electron beam laboratory (rooms for transmission electron microscope, scanning electron microscope, electron probe microanalyser), data processing and documentation room, library, office, communal rooms.

It is appropriate to note here that dividing a laboratory into several rooms is necessary because of the protection of valuable equipment and the different kinds of hazardous works (e.g. acidic atmosphere), obligatory protective prescriptions (X-rays and electron beam). On the other hand it is also reasonable if pieces of similar equipment are concentrated independently from the material being examined, consequently it becomes easier to arrange for their operation and the efficiency of qualified personnel is rendered higher.

In the following a review will be given about the functions, principles, equipment, and staff requirements in the main areas of investigations. We do not enter into details concerning room requirements, partly explained by the wide variability of the tasks and conditions and partly by the fact that these will be demonstrated by examples in chapters 5 and 6 using tables and layouts.

In Appendix 3 (Technological evaluation of bauxites) the complex utilization of chemical, mineralogical, textural, physico-chemical and technological investigations will be discussed.

4.1. UNITS OF ANALYTICAL CHEMISTRY

Chemical analytical procedures generally consist of three distinct functional parts a) sample preparation, and storage; b) analysis; c) data processing, formating and documentation.

The nature of the corresponding operations depends greatly on the actual conditions and analytical aims.

Sample preparation, analysing and data handling units must be organized on each level of completness.

Their organization and design will be different according to the needs and objectives. In this chapter these units will be discussed in details.

4.1.1. Sample Preparation and Storage Unit for Chemical Analysis

Functions:
a) sampling, homogenization and reduction of the amount of the sample
b) sample preparation
c) coding, storage

Principles

The sample represents the material to be tested to yield information on one or more properties of the entire amount of the material. Consequently the composition of the sample - at least within certain limits of error - must be identical with the entire material it was taken from. The mode of sampling is generally prescribed in standard specifications [9], however, certain deviations may also occur the basis of which could be either a well established experience or a trade contract.

The samples may be solids or fluids as regards the state of matter or the mixture of the above ones, i.e. slurries (red mud slurry, alumina hydrate slurry). The three kinds of samples have to be prepared for the chemical analysis in different ways. Primary condition is that the sample should preserve its representative character.

The preparation starts already at the sampling when the material is crushed, its mass is reduced, dried if necessary and packed. For the chemical analysis there is used the sample resulting after quartering and averaging.

The materials used in the course of alumina production comprise a more or less large amount of moisture. The quantity of moisture should also be known, therefore, a separate sample has to be taken for the determination of moisture. This operation is not easy to carry out because the sample must not loose any of its original moisture contendt during the preparation.

The sample may alter upon storage. The change may have a physical or chemical character; by adequate preparation those changes can be avoided or reduced to a negligible level. In the alumina industry primarily the change of moisture content has to be reckoned with. Minimum change in the state occurs if the sample is in equilibrium with its environment. For the purpose of chemical analysis and to ensure weight constancy the dried and properly powdered sample should, therefore, be brought to equilibrium with the humidity of the environment. This is ensured by spreading the sample in a thin layer for a considerable period: practically for a few hours.

Similarly, chemical changes might occur under the effect of the environment. As an example it could be mentioned that during the drying of the causticized red mud sample containing unreacted $Ca(OH)_2$, the composition will definitely change. $Ca(OH)_2$ forms $CaCO_3$ under the effect of CO_2 content of the air, thus, the original $Ca(OH)_2$ and CO_3 content of the sample cannot be determined. When the relation of SiO_2, Al_2O_3 and Na_2O contents has to be determined in red mud, the sample may be straightforward dried since the chemical changes do not influence the above relations. If the amount of unreacted $Ca(OH)_2$ retained in the red mud has to be determined the sample should be protected from the CO_2 content of the air.

It belongs to the problems of preparation as well what size the particles in the sample should be reduced to. No definite answer can be given to this question. The velocity of dissolution is directly proportional to the dispersity of the sample. Therefore, samples slow to dissolve should be ground to finer grains than those dissolving faster. It is general experience that the harder a sample and the higher its mechanical strength the slower it dissolves thus the finer grains contain more foreign material which is worn off from the grinding equipment. Considering the above, only a compromise can be searched. The sample should be pulverized to a suitable grain-size so as to achieve an acceptably short time of dissolution in the chemical analysis and at the same time a minimum contamination due to the grinding apparatus. Practical figures for the average grain-size are as follows

- for fairly reactive gibbsitic bauxites 150 µm
- for mixed type gibbsitic-boehmitic bauxites and red muds 100 µm
- for hard diasporic bauxites 50 µm.

In this case the samples meet fairly well the analytical requirements. It is also important to remark that non-uniform breakage of the mineral grains of different hardness occurs during the pulverization. After pulverization the larger grains should be separated by sieving and further pulverized as long as the entire amount of the sample passes the prescribed mesh aperture.

The prepared sample should be placed into a proper container or bottle, sealed air-tight. A label should be provided stating the name of the material, place and date of origin, the amount of the sample, code number of the sample, possible special data and the signature of the sampler, respectively. For the immediate analysis another container is used with adequate coding.

The ISO specifies how to take a representative <u>sample of alumina</u>. The particle size ranges between a few µm to 300 µm. Therefore, the alumina should be ground and screened until all the material passes through the screen of mesh aperture 0.2 mm. Alumina is a very hard material, it should be carefully ground because it can become contaminated by the material of the grinding mill or the mortar.

<u>Liquor samples</u> may be of purely liquid phase or sometimes they may contain solids in considerable amount; the latter ones are the slurries.

Alumina production is a continuous process. The liquid phase (and the slurry) exists in dynamic equilibrium (e.g. in a properly running settler or washer line) or in a metastable state (e.g. batch precipitation).

The aim of sampling is to maintain the material's actual state. If the sampling point is established and sufficient number of samples are taken within a given period the set of samples will be representative regarding the process flow. During the technological tests most frequently the static state prevails. The sampling procedure may be simplified by the fact that sometimes the entire amount of material constitutes the sample. Inhomogenity of the material represents a significant source of errors when sampling liquors and slurries. Segregation according to specific weight and settling of solids from the slurries can be avoided only by thorough stirring. This problem frequently occurs in static systems.

When analyzing liquor samples - provided that they were extracted from a system of metastable state - frequent troubles arise from the instability of the sample. The decomposition must be prevented by an appropriate handling which may be either dilution or the maintaining of a proper temperature or simply carrying out the analysis quickly before the sample starts decomposing. Anyway, the procedure preventing the decomposition should be efficient so that the representative character of the sample should not be affected.

For the analysis the samples must be treated further by different methods depending on the nature of the sample and on the applied analytical methods. These treatments can be discussed as a part of the sampling and preparation or, in the analysis section. Today, when the use of the mechanized and automated instruments and microprocessor controlled or computerized data collection and evaluation systems become widespread, the previous way of discussion is preferable.

Generally, the liquor samples do not require further treatment, however, dilution or the addition of some special reagent are needed sometimes.

The solid samples can be immediately analyzed by nuclear and X-ray spectrometric methods, however, fusion of the material is favourable in the latter case in order to eliminate the matrix effects.

The solid samples are generally analyzed after dissolution. Depending on the mineral composition the bauxite, red mud and alumina samples are soluble in various solvents. Sometimes, the dissolution process is preceded by an alkaline fusion. When dissolving the samples preference should be given to the methods indtroducing the smallest amount of foreign substance into the solution.

Since the dissolution procedure depends on the applied analytical method and the components to be determined it will be described in the analysis part.

From a practical point of view the determination of the moisture content and the loss on ignition can be performed best in the sample preparation room.

A weighed sample of an air-dry bauxite or red mud must be heated at 130 °C during three hours. The loss in weight is equal to the hygroscopic water.

If the sample is heated at 1,100 °C during two hours, the loss in weight corresponds to the water and CO_2 content, however, some side - effects have to be considered, too, originating from the thermal reaction of minor components of bauxite or red mud.

The moisture and the loss on ignition can be determined automatically with a micro thermobalance. The very small (max. 100 mg) sample can be heated under automatic control and the weight-loss is displayed in percentage.

Equipment

- Turbomill for sample pulverization
- Air jet sieve
- Laboratory disc mill
- Metal trays
- Drying oven (electric)
- Electric furnace
- Apparatures for fusing materials
- Micro thermobalance
- Electronic analytical balance
- Containers, bottles for solid samples
- Storing racks with capacity enough to store at least 1 year's samples
- Computer terminal with keyboard and interfaces to the balances

The types, producer firms, specifications and prices of a few special equipment is given in Table 6.

Table 6

DIFFERENT CHEMICAL ANALYTICAL INSTRUMENTS APPLIED IN THE BAUXITE TESTING LABORATORY

Type	Principles of operation	Producer	Price, $
Spectromom 195 Spectro-photometer	single-beam optical system, aut. sample changer data handling system	Hungarian Optical Works Budapest, Hungary	5,000
Pye-Unicam SP-6 Spectro-photometer	single-beam grating optical system, digital display, aut. sample changer	Pye-Unicam Ltd. Cambridge, U.K.	10,000
Thermatic titrator	automatic thermometric end point detection	ALUTERV-FKI Budapest, Hungary	6,000
pH meter OP-208	digital display, BCD output	Radelkis Co. Budapest, Hungary	1,000
Orion 901	microprocessor controlled pH meter	Orion Research Inc. Cambridge, Mass. USA	2,000
Sequence fusion Apparate	time selected fusion of 6 samples in Pt crucible at 1,200 °C	ALUTERV-FKI Budapest, Hungary	2,000
BEA-MTA 1527	nuclear bauxite analyzer with automatic sample and data handling	MTA-ELGI Budapest, Hungary	20,000

Note: Prices in this volume are approximative and based on 1980' price-lists.

Staff Requirements

Sample preparation does not require qualified workers. The number of persons depends on the number of samples, the number of shifts, etc. E.g. for a bauxite analytical laboratory with the capacity of 2,000 samples per year two sample preparators are needed.

4.1.2. Chemical Analytical Laboratories

Nowadays, the application of computer or microprocessor operated instruments becomes dominant in the instrumental analytical chemistry. The use of these instruments can be advantageous in a bauxite testing laboratory, too. Especially, the savings in working power and the decrease of the subjective errors are determining factors. This seems to justify the order of discussion accepted for the analytical units.

4.1.2.1. X-Ray Fluorescence Spectrometric Laboratory

The X-ray spectrometric and diffractometric methods are discussed together in chapter 1.2.

4.1.2.2. AAS and FS (Flame Spectrophotometric) Laboratory

The AAS analytical technique developed in the last years of the fifties has been introduced in the alumina industry quite rapidly for its relatively low expenses and easy sample preparation (in some cases solutions can be used directly without any other procedure). This technique can be applied mainly if the analytical problems need otherwise complicated procedures (i.e. determination of Mg, Zn, Na, etc.).

Functions: a) determination of the main components of bauxite and red mud with the required high reproducibility and accuracy

b) determination of a specially interesting trace element in a series of bauxite, red mud and alumina samples with high productivity.

Principles

A component present at a given concentration in the solution is exited to the atomic state by thermal energy. The atomic vapour will absorb the resonant light frequency of the special light source depending on the concentration of the component. The wavelength of the light absorbed is characteristic for the chemical component, however, the absorbance of the latter is proportional to its amount (concentration). The AAS method is not an absolute one, therefore, the light absorption of suitably prepared standard solutions is used as a reference. Since the light source emits the resonant lines of a single element, in general terms this is not a multi-element method, however, in practice the exchange of light sources is very quick. Consequently, in fact the AAS can be applied more efficiently for multielemental determinations than some other methods declared in theory as true multi-element procedures. Due to the small number of resonant lines spectral interference has to be considered only rarely.

The AAS equipment consists of an atomizing unit, the resonant radiation source, light dispersion part and a light detector; up-to-date equipment has a data processing unit, too.

The task of the nebulizing and atomizing unit is to produce atomic vapour proportional to the concentration of the component to be measured. For this purpose a special gas burner and nebulizer system or an electrically heated metal ribbon or graphite tube are used. The AAS equipment is generally supplied with a gas burner atomizing system. Apart from the viscosity of the solution to be tested and the nature of the component and matrix to be measured the efficiency of atomization depends on the design of the atomizer, the rate of atomization, on the temperature determined by the chemical composition of the flame and on the character of the flow of the flame (construction and shape of the burner head). The design of the atomizing gas burner is concerning the accuracy of the measurement.

Hollow-cathode lamps are generally used as light sources. For certain elements higher sensitivity can be achieved when applying electrodeless discharge lamps (EDL).

The electrothermic atomizer (ETA) equipment with heated graphite tube has been introduced recently. The ETA-system is generally used for trace analyses because of its high sensitivity. Further advantage is that small samples can be tested.

In a high quality instrument a double beam optical system serves for light detecting. For the ultraviclet and visible ranges of wavelengths, the thermal stress quartz or quartz-lined optical systems with high resolving power are used (dispersion of 3 to 4 nm/mm). Correction of the background radiation is normally achieved by means of a deuterium lamp.

The measurement of light intensity is performed using a photomultiplier. Modern devices, in general, provide digital display for the absorbance and enable auto-zero setting and measurement during various integration times. Direct electronic display of concentration and possibility for curve correction required from time to time is also ensured. The AAS instrument can also be used in emission (as flame spectrophotometer).

The different accessories, mainly the sample changer and the proper data processing system, are very important from the point of view of productivity. The basic AAS equipment is designed for long time operation and permits to analyze more than 100 samples per hour. The open end type sample changers can feed the equipment with the adequate number of samples. The evaluation is based on a calibration curve plotted according to the absorbance and concentration data of reference solutions prepared similarly to the sample or by the method of standard addition. These classical ways of data collection and evaluation need a lot of time and are sources of subjective errors. Therefore, modern AAS systems have a data processor system controlled by microprocessor. This system is able to supply all the automatic calibration for concentration (chemical or weight/volume units) using the marked "set in" probes applied in a suitable sequence of positions of the sample changer.

The relative error of the AAS method is generally 1 to 5 % depending on the matrixes and on the behaviour and concentration of the element to be determined. Besides, several factors determining the sensitivity and the accuracy of the method can be controlled by the operators. In order to obtain the best performance from the AAS system the operator has to choose operating conditions appropriate to the element being analyzed and decide whether it will be desired to optimise for sensitivity, detection limit or precision. The most important operator adjustable conditions are as follows: the wavelength of absorption line, monochromator bandpass, the lamp current, fuel flow rate, burner height, burner rotation and the position of the impact bead in the case of the flame AAS method. If the ETA method is used some other factors can also be controlled.

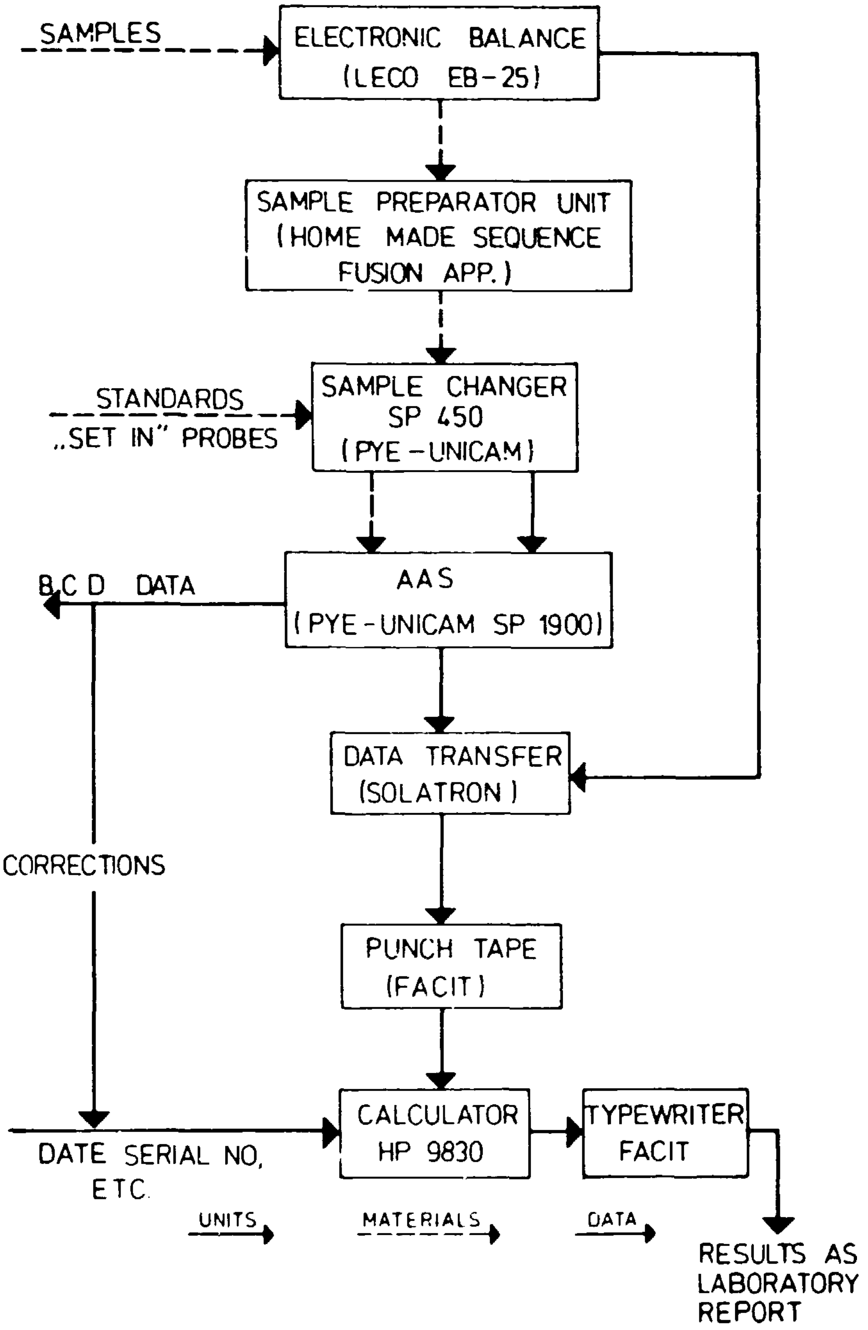
The detailed theory of the AAS method and the main principles of various AAS instruments are described in the different analytical AAS handbooks [10, 11]. However, some examples interesting especially from the point of view of a bauxite testing laboratory are discussed here.

The materials of the alumina industry have for a long time been thought of as particularly difficult to analyse by the AAS methods. This is because of the problems associated with dissolution and also because they contain a number of elements which are known to cause particularly serious interference effects. This interference originates from the chemical composition of the samples. Chemical compounds formed are relative stable in the case of so-called refractory elements as aluminium, titanium, silicon, etc. However, if a suitable method is chosen (e.g. application of a releasing agent, etc.) and the correct precautions are taken these determinations should not present any serious problem [12].

Determination of the main components of bauxite and red mud by AAS and FS method [13].

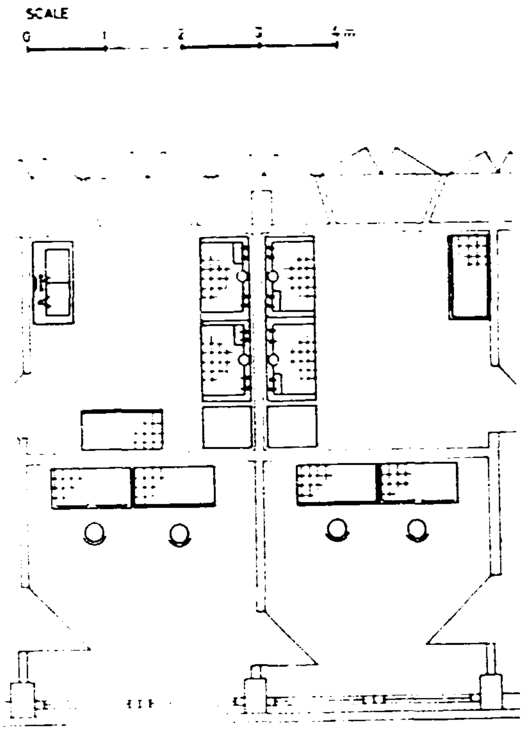
In the case of fast analytical exploration and technological control the application of the AAS and FS methods to determine the main components of red mud and bauxite is favourable due to their suitable reproducibility, little time and sample consumption as well as economic operation. The components of such an AAS analytical system and the relevant flow-chart can be seen in Fig. 5, a laboratory layout in Fig. 6 and an AAS equipment in Fig. 7.

The bauxite or red mud samples are fused with strontium carbonate boric acid flux in platinum crucibles with a suitable sequencial fusion apparatus operating at the required temperature and providing for proper fusion time. After dissolving the solidified material, without heating, in diluted hydrochloric acid the solutions are fed to the sample changer (e.g. Pye-Unicam SP 450 type) together with the five standard solutions consisting of the required quantities of flux, hydro-



FLOW CHART OF AAS
ANALYTICAL SYSTEM

FIG. 5



ROOMS FOR ACID-HOODS, DRYING OVENS, ATOMIC ABSORPTION AND FLAME PHOTOMETRIC SPECTROSCOPY

FIG. 6

Fig. 7
Atomic Absorption Spectrometer

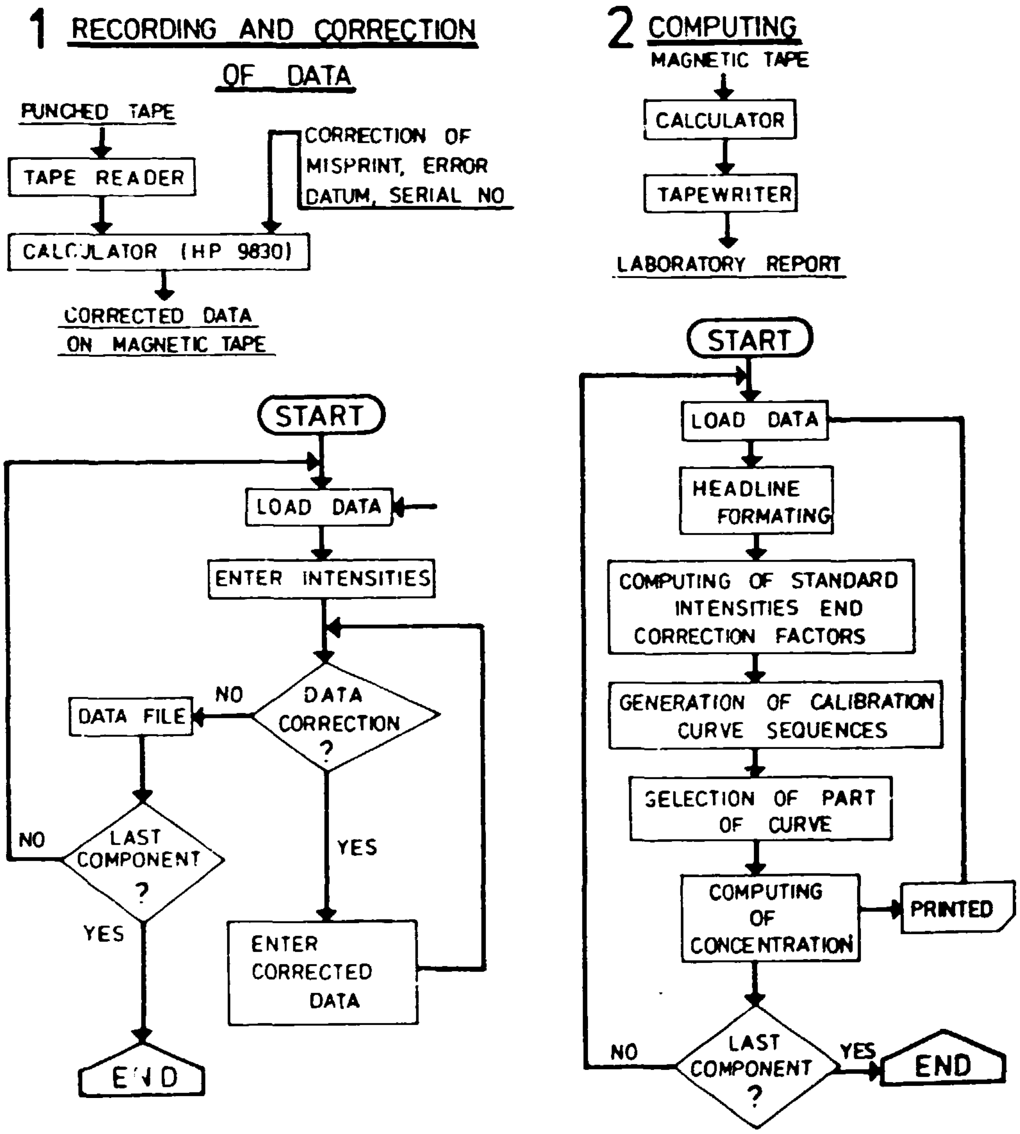
chloric acid and different volumes of the Al, Fe, Ti, Si, Ca, Mg and Na standard solutions, respectively. One of the standards is used as "set in probe" and applied several times on the sample tray having 50 sample positions. The intensity data are produced from the samples by an AAS equipment (e.g. Pye-Unicam SP 1900 type) working with properly selected operation conditions which are described elsewhere. In the case of Al, Si, Ti and Ca acetylene-nitrous oxide flame and for Fe and Mg as well as Na (in FS mode) acetylene-air flame are used. The weight of the samples and the intensity data of the AAS are recorded on a punched tape and fed to a desk calculator. The main steps of the evaluating program can be seen in Fig.8. The results are printed as a laboratory report. The variation coefficients for the different elements are as follows: for Al, Fe and Na 1 and for other elements 3. The analysis time is less than 6 hours/6 samples for all the main components.

In the case of alumina the Ca, Mg, Na, Zn and Cu content are generally measured by AAS and FS methods after proper dissolution process. Best is the treatment with hydrofluoric acid at high pressure and temperature in a PTFE lined steel vessel. After cooling boric acid is added to the dissolved sample to form fluoroboric compounds. In this case the matrix effect originating from the dissolution process is negligible. Sometimes, the fusion method is applied and lithium metaborate or sodium carbonate are used as a flux.

Other minor components of alumina can be determined by the application of the ETA method to increase the sensitivity.

Concerning the analysis of aluminate liquors minor sample preparation (only dilution and acidic treatment) is needed for the AAS method. The Ca, Mg, Mn, Fe, Zn content can be analyzed by AAS routinly. As an example the Zn content of the aluminate liquor can be analyzed by AAS after the addition of HCl until the precipitate is dissolved and after dilution by distilled water to 1:20 volume ratio.

700 DATA 6 samples / DAY
WITH APPLICATION OF A COMPUTERIZED
DATA PROCESSING SYSTEM



DATA PROCESSING AT
AAS/FS ANALYSIS.

FIG. 8.

Equipment

- Sequencial fusion apparatus
- PTFE lined steel bombs for dissolution of alumina
- Bakers, volumetric flasks, pipets, etc. for sample treatment and dilution
- A good quality AAS equipment with the usual accessories
- An ETA system for the work in the field of trace analysis
- Gas supply assembly with compressed air, acetylene, nitrous oxide and argon
- Fume hood particularly for the work with nitrous oxide acetylene flame
- Data processing system with the proper interfaces

The Pye-Unicam SP 1900 AAS equipment completed with the SP 450 Automatic Sample Changer, a Solatron Data Transfer Unit and a Facit Tape Punch Unit, can be regarded as an example of a high quality instrument. If ETA is also required the Pye-Unicam SP 9 system is suitable with the SP 9 Computer, SP 4-01 Automatic Sample Changer, the Video Furnace and the Furnace Autosampler. Pye-Unicam delivers all the other required accessories, e.g. hollow-cathode lamps, accessories for the gas supply, etc. (see also Table 7).

Staff Requirements

According to the number of the samples to be analyzed and the level of the analytical activity workers skilled in different disciplines are needed for the AAS and FS operation. In a usual combination a well qualified analyst (with university degree) and 2-3 technicians are able to analyze completely 2,000-2,500 bauxite samples in one shift. Applying an up-to-date AAS system (such as the Pye-Unicam SP 9) and in the case when effective service is provided externally the staff may consist of trained technicians only.

Table 7

ATOMIC ABSORPTION SPECTROPHOTOMETERS APPLIED IN THE BAUXITE TESTING LABORATORY

Type	Principle of operation	Producer	Price, $
Pye-Unicam SP 9	single-beam optical system with microprocessor data handling, electronic gas control, automatic sample changer	Pye-Unicam Ltd. Cambridge, U.K.	26,000
Varian AA-775	double-beam optical system, system, microprocessor, aut. gas control, aut. sample changer	Varian Techtron Pty. Ltd. Mulgrave Victoria Australia	30,000
Perkin-Elmer M 5000	double-beam optical system, microprocessor, aut. gas control, aut. sample changer	Perkin-Elmer Co. Norwalk, Connecticut, USA	35,000
IL 551 Video I	double-beam optical system, microprocessor, aut. gas control, aut. sample changer, video display	Instrumentation Laboratory Inc. Wilmington, MA USA	25,000

Note: Accessories (e.g. lamps, gas regulators, etc.) are not included in the price.

4.1.2.3. Wet Chemical Laboratory

The structure and performance of the wet chemical laboratory depends on the tasks and the levels of the other instrumental laboratories. If all the analytical routine investigations are carried out by well organized instrumental groups only the standardization and very special analytical tasks are solved in this laboratory. In that case a limited number of workers is required but they should be good experts. However, all the analytical chemistry services can be performed by the classical wet chemical methods if justified by the level of technical development or financial considerations. A number of workers and a lot of time (generally three shifts) are needed for the proper operation of the laboratory in this latter case.

Since the actual situation is usually somewhere in between, the description of the function and principles of the wet chemical laboratory takes into account both possibilities but with constraints.

The final detection of the components to be analyzed is performed by spectrophotometric or potentiometric methods, too. This methods, however, are not purely classical wet chemical methods in the sense discussed here.

Functions

a) Determination of components in bauxite and red mud as well as in alumina.
b) Determination of the main components of aluminate liquor.
c) Calibration of the instrumental methods (preparation of standard solutions, determination of the concentration of the reference solutions, etc.).
d) Performance of special investigation of technological significance (e.g. reactive SiO_2, available Al_2O_3, etc.).

Principles

The general principles of the classical wet chemical methods can be found elsewhere, only the special methods interesting from the point of view of the alumina industry are discussed here.

Bauxite and red mud. For the determination of the SiO_2, Al_2O_3 and TiO_2 content of these materials a special dissolution procedure is required. Generally, the sample is solved in a sulfuric acid, hydrogen peroxide mixture, however, the triacide mixture is applied as an alternative method. If the sample contains Al_2O_3 in the form of diaspore a preliminary transformation is needed by a heat treatment with Na_2CO_3-KNO_3. The filtrated and washed precipitate is ignited at 1,000 °C and evaporated with hydrofluoric acid in the presence of sulfuric acid. The residue is ignited again. The loss in weight is equal to the SiO_2 content. The Al_2O_3 content of an aliquot of the dissolved sample is separated as soluble sodium aluminate from the other components remaining as insoluble hydroxides. The aluminium can be determined by complexometric titration, taking into account the loss due to the adsorption of aluminate on the hydroxide precipitate. If the sample has a large CaO content a preliminary separation is needed with urotropine. The TiO_2 forms peroxo complexes of yellow colour with hydrogen peroxide. The absorbance of the complex is directly proportional to the concentration of titanium. The disturbing effect originating from the yellow colour of the ferric complexes can be eliminated by means of phosphoric acid. The light absorption is measured with a spectrophotometer. The Fe_2O_3 content of the sample is determined after fusion with KOH flux and oxidation with permanganate in sulfuric acidic solution. The ferric ions are reduced to ferrous state with $SnCl_2$ and titrated with potassium dichromate titrant in the presence of diphenylamine sulfonic acid after addition of $HgCl_2$ and phosphoric acid. The Cr_2O_3 and V_2O_5 contents of the sample are determined after an alkaline oxidative fusion. The flux is extracted with hot distilled water. The chromium and vanadium can be measured together (in the form of chromate and vanadate) by titration performed with Fe^{II} titrant and the end point can be detected potentiometrically applying a platinum and a reference electrode pair. The vanadium can be selectively oxidized after titration with permanganate. The vanadium content can be measured again with Fe^{II} titrant.

Alumina. There are internationally standardized (ISO) procedures for the determination of the most important contaminants of alumina. The fast detection of the components is generally performed by a spectrophotometer. The sample is dissolved after an alkaline fusion. The Fe_2O_3 content is measured as iron (II)-1-10-phenantroline complex at 510 nm wavelength. The SiO_2 content is measured as reduced molybdosilicate at 815 nm. The TiO_2 content is determined at 420 nm after the formation of the titanium-diantipyryl-methane complex. The V_2O_5 content is measured at 524 nm after extracting the violet coloured complex of N-benzol-N-phenylhydroxilamine. The P_2O_5 content is determined at a wavelength of 730 nm as the phosphormolybdic complex extracted by 2-methylpropane-1 after reduction.

Aluminate liquors. The caustic soda (Na_2O_c) content is determined after the elimination of the disturbing anions (CO_3^{2-}, PO_4^{3-}, VO_4^{3-}, F^-) by precipitation adding large excess of barium chloride. An aliquot of the clear solution can be used for the titration with hydrochloric acid titrant in the presence of phenolphtalein indicator. The alkalinity originated from sodium hydroxide and sodium aluminate is measured this way. The disturbing effect of aluminium content can be eliminated with addition of potassium sodium tartarate. The aluminium content of aluminate liquor forms an EDTA (ethylene diamine tetra acetic acid) complex.

The excess of a known quantity of EDTA can be titrated back with zinc acetate titrant in the presence of xylenolorange indicator. The carbonate content of aluminate liquor can be determined utilizing the $BaCO_3$ content of the precipitate originated from the caustic soda determination. The precipitate can be decomposed with hydrochloric acid and the excess of the acid can be titrated back. The Na_2CO_3 content can be calculated from the consumed hydrochloric acid. The silica content of an acidified aluminate liquor can be determined as the reduced silica-molybdenic blue. The absorbance of this complex can be measured spectrophotometrically.

Standardization. The most important task of the wet chemical laboratory is to support the operation of the instrumental laboratories having large productivity. The above described methods can be applied to analyze the standard and "set in" probes applied within the instrumental procedures. The accuracy is an outstanding factor in this work because it affects the quality of the output of the whole testing laboratory. This is also valid for the interlaboratory test activity.

Equipment

- The common glass bakers, volumetric flasks, pipets, burets, etc. depending on the capacity of the laboratory.
- A sequencial fusion equipment is very useful for its high productivity (type will be described in paragraph 4.2.2.).

Staff Requirements

Generally, the leader of the laboratory must be a well educated analyst (with university degree) experienced in the analytical chemistry of alumina industry.

The members of the staff comprise some technicians and laborants as well as a number of unskilled workers who can be employed for some routine jobs.

4.1.2.4. Laboratory for Other Instrumental Methods

At the present level of the analytical chemistry several instruments are needed in order to carry out successfully a definite analytical task. For the proper productivity and quality of the supplied results these instruments are generally installed in the same place and operated by skilled workers. That is the instrumental laboratory. The equipment applied in the field of analytical chemistry of alumina production can be divided into spectrophotometers, electroanalytical instruments and other special devices.

Functions

a) Routine identifications as required by the wet chemical laboratory.
b) Special investigations in the field of analytical chemistry of the alumina production.
c) Research and development for the application of monitors in the testing or technological process.

Principles

Spectrophotometry is an analytical method to determine the light absorption of materials. The absorbance of a solution containing the components to be determined is proportional to the distance the light have travelled in the solution (size of the cuvette) and the concentration. The wavelength of the investigation is characteristic for the component concerned [13].

Generally, the instruments of ultraviolet-visible wavelength range are used as final detection units in the laboratory, however, the infrared spectrometers are also applied in the field of the alumina industry.

The spectrophotometer consists of a light source of uniform intensity in the entire wavelength range, a light dispersion part, a cuvette compartment and a detector unit. The advanced instruments have a double beam optical system and are able to measure 0.01-4 absorbance values. The up-to-date spectrophotometers have productivity oriented automatic sample changers and data processing systems, too.

The spectrophotometric analytical procedures are described in paragraph 4.2.3.

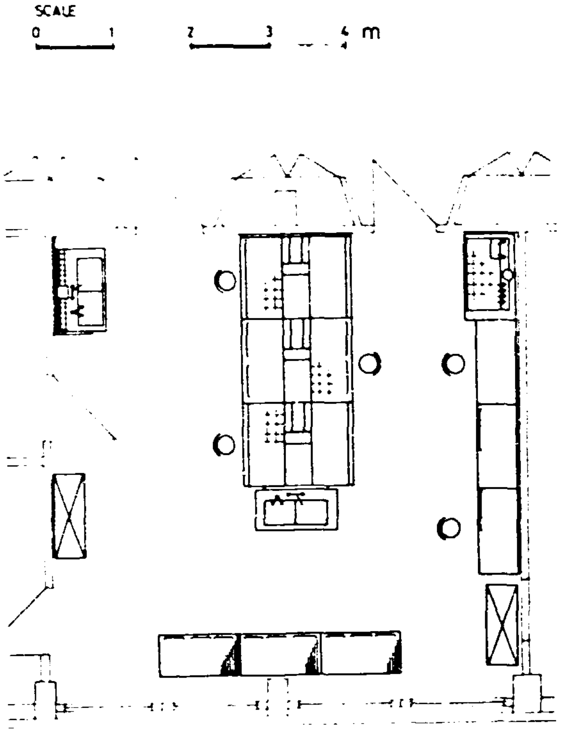
The electroanalytical techniques used in the field of analytical chemistry of the alumina production can be divided into three main groups: equilibrium and dynamic measuring methods, as well as those making use of the conductivity of ions.

During equilibrium measurement the electromotive force is measured in currentless state (potentiometry) [14]. In the dynamic measurements the "electron" as a universal reagent is used so that the current through the cell is maintained constant (voltametry, polarography) [14]. When measuring electric conductivity in fact the transport of ions is measured (conductometry, oscillimetry) [14].

Corresponding to the above the electroanalytical instruments can be divided into three groups. The instrument of potentiometry is a voltmeter with high input impedance and a cell which consists of reference and indicator electrodes. Recently ion-selective electrodes have been widely used for the detection of different ion-activities. The technique can be utilized in the laboratory as direct potentiometry or potentiometric titration, as well as for monitoring different components of an aluminate liquor (e.g. the aluminate content can be determined with an automatic titrator on the basis of potentiometric end point detection, or the sodium content of the aluminate liquor can be monitored on the process directly).

The modern polarographs consist of a potentiostat as a voltage source, a signal generator for the control of the production of the polarization voltage signal with the potentiostat and of a compensograph or oscilloscope for the measurement of the current. For coulometric measurements a current integrator is also needed. The methods are very accurate and can be used for calibration in the field of analytical chemistry related to the alumina industry.

The conductivity measurements are commonly carried out by a 1,000 Hz frequency bridge. For oscillometry an electrodeless inductive or capacitive measuring cell is used. Oscillometry has an important role in rapid direct control of the technological process. For instance, from oscillometric measurements the caustic soda, the total sodium and aluminate content, respectively, can be determined. The thermometric concentration measurement proved to be an instrumental control method particularly adequate for the measurement of high concentrations. Simultaneously with the chemical (and physical) changes also heat effects can be detected which are proportional to the concentration. In the field of chemical analysis the direct thermometry (entalpymetry) and the thermometric titration can be used. The last mentioned method is very useful in the analysis of aluminate liquor. An automatic thermometric titration system had been elaborated by the R/D Centre of the Hungarian Aluminium Co. dedicated to the determination of the caustic soda, total soda and aluminate contents (and for the molar ratio) of the aluminate liquor. The error of this thermometric determination is less than 1 rel. % and the procedure is very fast and economic. A picture taken of the instrument is shown in Fig. 10, a layout of an analytical laboratory is given in Fig. 9.



WET CHEMICAL LABORATORY

FIG 9

Fig. 10

Thermometric Titrator

Equipment

- A good quality spectrophotometer with the usual accessories (see Table 6)
- Precision pH meter (see Table 6)
- Automatic potentiometric titrator (see Table 6)
- Conductivity meter (see Table 8)
- Polarograph (with d.c., a.c. and pulse mode) (see Table 6)
- Thermometric titrator (see Table 8)

Staff Requirements

If only some routine analytical tasks are to be solved in this laboratory, an analyst specialized in instrumental analytical chemistry should supervise the work of the staff consisting of three or four technicians. In the case of research and development activity the staff must comprise experts in the various special fields of the instrumental methods.

4.1.2.5. Laboratory for Neutron Activation Methods

Function

Determination of some main components of bauxite (and red mud).

Table 8

AUTOMATIC TITRIMETERS

Type	Producer Firm	Specifications	Price, US$
Memo Titrator DL 40	Mettler Instrumente AG CH-8606 Greifensee SWITZERLAND	for potentiometric titration of S, Cl, Al for voltametric titration of Zn for colorimetric titration of Si, P, V, Cr, Mo	6,000
TA-2	Technicon International 12-14 Chemin Rien 1208 Geneva SWITZERLAND	Potentiometric titration: S, Cl, Al thermometric end point indication caust Na_2O	~ 10,000
Thermatic	ALUTERV-FKI 1389 Budapest Pf 128 HUNGARY	thermometric titrations (caust Na_2O, total Na_2O, Na_2CO_3, Al_2O_3, $FeCO_3$, FeS_2) Reaction heat range 0.2...25 kJ/mol Concentration 0.1...0.001 mol/dm^3 Titrating time: 3-5 Minutes	6,000
Thermometric Titrator	Perkin Elmer Corp Norwalk/Conn. USA	for thermometric titrations	~ 10,000
DVR	SANDA INC 4343 East Riner Drive Philadelphia, PA 19129 USA	Detection limit: 0.2...25 kJ/mol or 0.1...0.001 mol/dm^3	~ 10,000
Oscimhometer	ALUTERV-FKI 1389 Budapest Pf 128 HUNGARY	caust Na_2O, Al_2O_3, caustic molar ratio, total Na_2O determination with conductometry measurement	~ 1,500

Principles

The nuclear technique, primarily neutron activation analysis is recently widely used for the rapid quantitative analysis of the main components of bauxite and red mud. The method is based on the phenomenon that a part of the atomic nuclei in the sample exposed to neutron radiation is converted to radioactive atomic nuclei. After activation the resulting radioactivity can be readily detected by radiation measurement. The rate of activation of the sample depends on the intensity and duration of the radiation, moreover, on the natures and concentrations of the elements in the sample. With usual bauxite and red mud on applying a short time irradiation by slow neutrons mainly the aluminium gets activated, and on the other hand by fast neutrons the silicium and sodium.

Under these circumstances the activation procedure produces short half-life radioisotopes the activity of which ceases within a few minutes. Thus, the samples are practically inactive after the measurement and no protection is required against radiation hazards. The advantage of the method lies in the fact that in contrast to the wet chemical analysis little sample preparation work is required. Due to the high penetration power of the neutron radiation, as the irradiation can be regarded uniform for the total volume of the sample; the only condition is to fill up uniformly the speciment holder tube with the material ground to 1 mm particle size. The resulting radioisotopes also emit highly penetrant beta and gamma radiation. The measurement of these is carried out automatically without contacting the material. Thus after having the sample filled into the tubes no further manual work is required. The Hungarian "Automatic Bauxite Analyzer" is extensively used for routine measurements. Its sample magazin accommodates 10 pairs of sample tubes simultaneously. A pneumatic tube dispatch system serves for passing the tubes to the activation and measuring positions. Any number of runs or repeated measurement of the samples can be carried out. The measuring accuracy is disturbed if the sample comprises other elements easy to get activated. For instance, if the samples contain phosphorous and vanadium even in hardly changing quantities their interference can be eliminated only in the calibration procedure.

In an updated version X-ray fluorescent analysis is applied for the determination of Ca, Ti and Fe contents of the bauxite. X-ray fluorescence is exited by means of suitably selected radioactive sources emitting soft gamma radiation and a low-selectivity radiation meter is used for detection. The measuring accuracy is less than that of the energy-selective systems, however, the unit is simple and provides a cheap, fast analytical method. Operating principle is shown in Fig.11.

Equipment

- Automatic Bauxite Analyzer (ABA) (see Table 9)

Staff Requirements

The ABA instrument can be operated by unskilled workers having attended a short special training.

4.1.3. Data Processing for Analytical Chemical Units

Functions

a) The handling, formating, reducing and storing of analytical data originated from different equipment.
b) Direct control over the proper operation of the laboratory.

Table 9

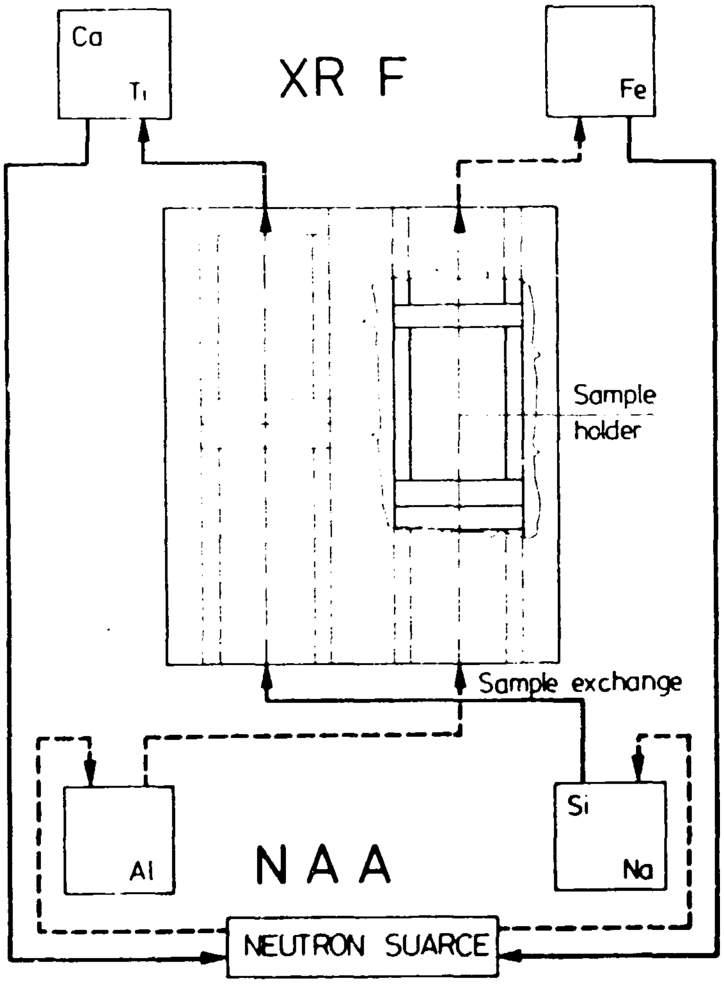
AUTOMATIC BAUXITE ANALYZERS

Type	Producing firm	Specifications	Price in US$
MTA-1527-1000	Magyar Állami Eötvös Loránd Geofizikai Intézet, Budapest, XIV. Kolombusz u. 17-23. Hungary	Al_2O_3 (accuracy: 0.5 %) SiO_2 (accuracy: 0.3 %)	~ 20,000
MTA-1527-2000	Magyar Állami Eötvös Loránd Geofizikai Intézet, Budapest, XIV. Kolombusz u. 17-23. Hungary	Al_2O_3 (0.5 %) SiO_2 (0.3 %) CaO (0.2 %) Fe_2O_3 (0.2 %) TiO_2 (0.2 %)	~ 25,000
MTA-1527-3000	Magyar Állami Eötvös Loránd Geofizikai Intézet, Budapest, XIV. Kolombusz u. 17-23. Hungary	Al_2O_3 (0.5 %) SiO_2 (0.3 %) CaO (0.2 %) Fe_2O_3 (0.2 %) TiO_2 (0.2 %) Na_2O (0.2 %)	~ 30,000
Neutron beam source	E.g. Saclé Institute Isotope, Paris, France	AmBe neutron source: 550 GBq	~ 5,000
X-ray source	Amersham Buckinghamshire England HP 79LL	Fe^{55}: 3.7 GBq Pu^{238}: 0.37 GBq	

<u>Note</u>: To MTA-1527-1000 neutron source, to MTA-1527-2000 and MTA-1527-3000 X-ray source as well, which are not included in price. They are supplied directly by producers.

XRF Ca and Ti generated by 55 Fe radiation
Fe generated by 238 Pu radiation

NAA Na and Si activation by fast neutrons
Al activation by slow neutrons



MEASURING PRINCIPLE OF AUTOMATIC BAUXITE ANALYZER

FIG. 11.

Principles

The productivity and the objectivity of the work of a bauxite laboratory or any other one can be increased by the application of a data handling system. Saving of human resources is also an important factor in organizing such a computerized data handling unit.

The modern laboratory instruments have adequate (generally digital) outputs to link a minicomputer terminal. Some instruments have their own microprocessor operated data acquisition systems to evaluate and produce the results in a preselected form.

The actual realization of a data handling unit is mainly determined by the technical and financial background. Some automatic instruments equipped with microprocessor operated data handling system do not require a real-time data center even if the formation of laboratory reports and storing of data is considered. In that case it is better to use a punched tape or tape cartridge, or some times a disc storage facility and apply the computer in off-line mode. In a more complex laboratory and if the immediate control of its production is required on-line mode can be applied.

The organization of the analytical laboratory into an integrated system becomes more and more frequent and this yields enormous possibilities in the productivity of the analytical work.

Equipment

Depending on the applied methods and instruments various possibilities can be considered.

In the organization of a computerized laboratory system it is recommended first of all to employ an expert of this interdisciplinary field. Abundant offer of hardware and software is available for automation of the data handling in an analytical chemistry laboratory. Firms, like Hewlett-Packard, Perkin-Elmer, Data General, DEC or the Central Research Institute for Physics (KFKI) of the Hungarian Academy of Sciences may be considered, among others, as suppliers.

Staff Requirements

No extra personnel is needed for the data processing of an analytical laboratory but special training should be provided in the operation of the relevant equipment.

4.2. X-RAY LABORATORY

X-ray laboratories oriented towards R & D in the field of bauxite exploration and alumina production may extend their activities mainly to:

X-ray fluorescence spectrometry (XRF) and
X-ray diffraction analysis (XRD).

XRF is an analytical technique used to determine qualitatively and quantitatively the elemental constitution of a substance. In this capacity it is an up-to-date instrumental substitute for wet chemical analysis which can be highly automated. However, XRF is not an absolute procedure and requires calibration by standards which might be either purchased or prepared by a background chemical laboratory. Alternative instrumental methods are atomic absorption spectrometry (AAS), thermometric analysis, emission spectrometry, neutron activation analysis or mass spectrometry each having its particular advantages and drawbacks when well defined analytical problems arise. In fact XRF could have been handled among the various methods of modern chemical analysis (4.1.2.1). The proposal for a separate X-ray unit has mainly practical reasons.

X-ray fluorescence spectrometry can be applied for an overall check of the chemical elements present in an unknown sample in proportions exceeding some 0.01 %. The limit of detection depends partly on the matrix and decreases for the elements with higher atomic number. In bauxite exploration this qualitative aspect is used to define major impurities to be reckoned with in new resources. In alumina technology identification is usually the means of trouble shooting by the investigation of abnormal deposits, intermediate products or even of the incoming raw ore.

XRF is used more frequently for routine quantitative analysis of a large number of rather similar samples. Despite the relatively substantial investments needed to establish a well equipped X-ray fluorescence laboratory,this application seems to be economical on the long run. In geological field exploration the quantities and topological distribution of the main constituents (Al, Si, Fe, Ti, Ca) and that of the major impurities (S, Mn, K, Mg, etc.) can be determined with little labour, within short periods, with high confidence enabling finally a good estimate of the resources. During the mining stage control of the produced ore and guiding of the operations can be achieved. In the alumina plants regular analysis of red mud samples is the most advantageous application for XRF leaving room for many others not listed yet.

XRD is exploited to investigate qualitatively and quantitatively the crystalline components of a substance. As such it represents a complementary technique and not an alternative to the elemental analysis methods listed above. Mineralogical and industrial X-ray diffraction is mainly concerned with comparing measured data to reference diffraction patterns in order to identify or characterize as well as to determine quantitatively the phases present in a multicomponent mixture of polycrystalline materials.

Among the large variety of diffraction techniques such studies can benefit first of all from the application of powder diffractiometers which lend themselves readily to automation.

XRD is indispensable for assigning technological value to a given bauxite deposit. The mineral forms of the aluminium, iron and silicon bearing compounds are decisive in determining the parameters of processing, i.e. in designing a new plant. During the operation of plants the efficiency of steps like desilication, digestion, separation, etc. can be controlled by XRD checks. Alternative and complementary tools for obtaining mineralogical information are infrared spectrometry, thermal analysis and microscopic methods. Thus, XRD should belong systematically to the group of mineralogical analysis. (Section 4.4.)
The proposal for separation has merely practical advantages.

XRD in itself can yield highly sensitive reliable results in routine qualitative analysis. However, quantitative mineralogical phase analysis requires for the problems met in alumina industry the knowledge of the chemical constitution. Therefore, a combined XRF and XRD analysis allows to accomplish a self-consistent quantitative phase analysis, which is more straightforward for natural ores like bauxite, than for synthetic products with ill-defined stoichiometry. Consequently it seems desirable to apply complex analysis involving several complementary methods, although the problems faced are similar for all when highly variable, poorly crystallized components have to be dealt with.

XRF spectrometers and XRD goniometer systems can be installed as independent equipment in separated laboratories, however, both being supported by X-rays providing a similarly hazardous environment for the operators and since they use a great deal of analogous instrumentation it is even feasible to join them is a single "workshop". Close combination of fluorescence and diffraction measurements under common computer control can lead to enhanced information density acquired from a single sample. Separation or coupling of the two services can be governed also through the capacity required from each.

In the following the main functions and the relevant apparatus of an X-ray laboratory will be described.

4.2.1. Sample Preparation

XRF

The aim of sample preparation for fluorescence measurements is to assure homogenety in a geometrical form adequate to introduce the sample into the instrument. Physical properties which render impossible to determine the true chemical composition should be eliminated. The spectrometers usually accommodate sample discs of 30-50 mm diameter and a few mm height. These can be prepared from finely dispersed powders by pressing into thin aluminium cups, metal rings, etc. with or preferably without binder. Pressed pellets can be used only if the samples to be compared with each other and the references have identical crystalline character, what is rarely a priori warranted. Therefore, a solid solution should be made from the sample. After mixing in appropriate proportions with a flux material (e.g. lithiumborate) the minerals occurring in bauxite-alumina technology melt at about 1,100 °C and "beads" can be casted manually or in a more or less automatized fashion.

Items needed in an XRF preparation room are:

a set of mortars and pestles,
a set of sieves,
a pulverizer or grinding mill,
a balance,

a hydraulic press with mould,
means for preparing "beads".

The last item may consist of a furnace with heat regulation in which the crucibles are placed manually and after a predetermined heat program the melted samples are cooled and solidified to the required shape in rings or moulds. On the other extreme bead machines prepare by high frequency program heating automatically the required uniform, smooth sample discs from the powder fed in. Aids for bead preparation of intermediate automation and price are also available. These need more human inspection and are, therefore, less productive. E.g. the heating time and temperature are electronically controlled, but the crucibles must be placed into a holder one by one and the melted sample poured into the cooled mould manually.

XRD

For X-ray powder diffractometry samples of 1-1.5 g mass with < 10 µm particle size are necessary. Aluminium or plastic sheet sample holders are used with about 10x15 mm windows which should be filled by the powder realizing a random orientation of the particles while forming a stable, flat surface. There are several ways to achieve this goal. Back pressing against a piece of fine abrasive paper gives sufficiently good results in most cases. For some measurements it may be necessary to mix the sample with weighed in amount of a reference material. Frequently used references are alpha-Al_2O_3 (corundum) and KCl.

Thus besides a balance, a mill and a set of sieves a manual press is the only equipment needed. Standard materials belong also to the stock of the preparation room.

4.2.2. Measurement, Data Collection

Having prepared the samples the phenomena following the interaction with X-rays must be recorded and then evaluated. Let us consider in turn apparatus used (i) for the generation of X-rays, (ii) for producing the physical responses of fluorescence and diffraction, respectively, and (iii) for registration of these effects.

4.2.2.1. Radiation Sources

X-rays are produced in industrial R & D laboratories by means of sealed off tubes. Spectrometric and diffraction tubes have different geometries, but both are water cooled thermionic tubes working under stabilized, smoothed 30-100 kV high tension. The ratings of the former ones are somewhat higher (usually 3 kW) and that of the diffraction tubes varies between 1 and 2 kW depending on the focus size.

The generators supplying high voltage may be integrated into the device as in many sophisticated spectrometers, but can also be separated, feeding the tubes via a HT (high tension) switch and cables, permitting alternative (or simultaneous) operation of several X-ray tubes, including a combination of spectrometric and diffractometric measuring places. The smallest generators are rated for 1 kW but there seems a tendency to build larger ones and 3-4 kW generators became quite common in the early 80 s.

Generators and X-ray rubes require intense water cooling (5-10 l/min/tube, 20 ^{o}C). Since cold, clean tap water becomes expensive and stable pressure is not always available, closed-loop water cooling aggregates have been developed. This are filled up with a few liters of distilled water and added anticorrosion reagents and consume 3-4 kW electrical power in exchange of supplying cooling water enough for 1 or 2 X-ray sources.

Provided that frequent failures occur in the electrical net it might be reasonable to install (at least a stand-by) motor generator for safe power supply.

X-rays are harmful for the human body. Therefore, every undue exposure of the personnel should be avoided. Warning signals should make people alert that the X-rays are on. All goniometers, spectrometers must have safety features that enable operation exclusively if the direct beam is stopped and shields are in place. Automation of sample changing and remote control of most measuring activities is desirable to minimize the time spent by the operators in the region where stray radiation is present. Personal badge detectors, area monitors should be provided and a regular practice of medical inspections established.

Thus the production of X-rays requires the following items:

a generator
X-ray tubes (with Cr and Au anodes for spectrometry and provided a reflected beam monochromator is available (cf. below) only with Cu anode for bauxite-alumina diffractometry)
means of water cooling
safety devices for radiation protection.

4.2.2.2. Spectrometers

XRF spectrometers fall into two major classes: sequential and simultaneous ones.

Sequential spectrometers are usually cheaper, work slower, but permit more flexible operation. Full possibilities of X-ray spectrometric analysis in general can be practiced. The signals characterizing individual elements are measured by the radiation detector in sequence. The average measurement time necessary for observing the fluorescent radiation of one element must be multiplied by the number of elements looked for to get the total time spent on one sample. About 2 minutes is a typical value for a bauxite analysis covering 6 elements. Nevertheless, all elements from B to U can be considered and full automation is also a realizable option. Thus such instrument should be selected for research and development work, where the topics change, the nature of samples varies from series to series.

Simultaneous spectrometers have independent detecting channels for each element, however, the number of channels is generally limited to 25-30. Microprocessor or computer control is mandatory, automatic changers for up to 100 samples are common. There are even continuously fed spectrometers coupled on-line to a sampling mechanism, i.e. a closed loop leads from the material flow through sampling, specimen preparation, measurement to the final information. If appropriately coded holders are used the measuring and evaluating program can be selected by the controlling microprocessor in accordance with the nature of the very sample. The element requiring the longest period of data collection determines the cycle, which is typically 60-70 sec/sample. The investment in a simultaneous spectrometer is not justified but when very large number of samples have to be measured according to a few (5-10) fixed programs extending to a limited number of elements.

Another, cheaper, however, less accurate variant of simultaneous spectrometers is the energy dispersive X-ray spectrometer introduced recently. This detects the entire fluorescent spectrum in a single step by a semiconductor device and analyses it by a minicomputer using a hard wired or a software multichannel analyser. Sensitivity decreases towards the light elements Na being the limiting case. The measurement is as fast as with the wavelength dispersive simultaneous spectrometers but some physically determined interferences set the accuracy one order of magnitude lower.

Thus basically three types of spectrometers can be considered for an XRF laboratory: sequential
simultaneous or
energy dispersive ones.

4.2.2.3. Diffractometers

A diffractometer consist of a goniometer with or without sample changer, a reflected beam monochromator which is found to be indispensable in bauxite red mud analyses, and of radiation recording and measuring instrumentation. The performance of goniometers well established for the commercialized vertical or horizontal wide angle devices is completely acceptable in this field. Sample changers can handle 20-40 samples and the latest ones permit spinning, too. The parameters of the measurement for the different samples can vary during unattended operation only if the diffractometer is brought under computer control. The X-rays are detected by a proportional or scintillation probe and the signal thereof is processed by sophisticated electronics. Simple analog units are more and more replaced by digital ones in up-to-date apparatus. The data corresponding to the X-ray intensity diffracted from the sample can be strip chart recorded or printed digitally or stored on punched tape. If an on-line computer is present the phase of evaluation and storage is not separated from the measurement.

Thus XRD measuring equipment consists of
a goniometer (monochromator, sample changer)
detector + electronics (microprocessor, computer)
recorder, printer, tape punch.

4.2.2.4. Interpretation

XRF

Spectrometers are usually of the "quantometer" type, i.e. the integrated microprocessor or computer accomplishing data reduction and corrections at different levels, yields as output a table of the concentrations on a display or in a hard copy.

Qualitative analysis is carried out using comparative tables.

XRD

In the first step of evaluation the measured data of the diffraction patterns are reduced to the very parameters usually interpreted. These are the interplanar spacings (d) of the crystallographic planes producing the reflected maxima, the peak and/or the integrated intensities of the maxima (I) and the breadth of the same (B).

Analog records can be interpreted using rules and scales. Digital data are corrected and reduced by appropriate computer algorithms.

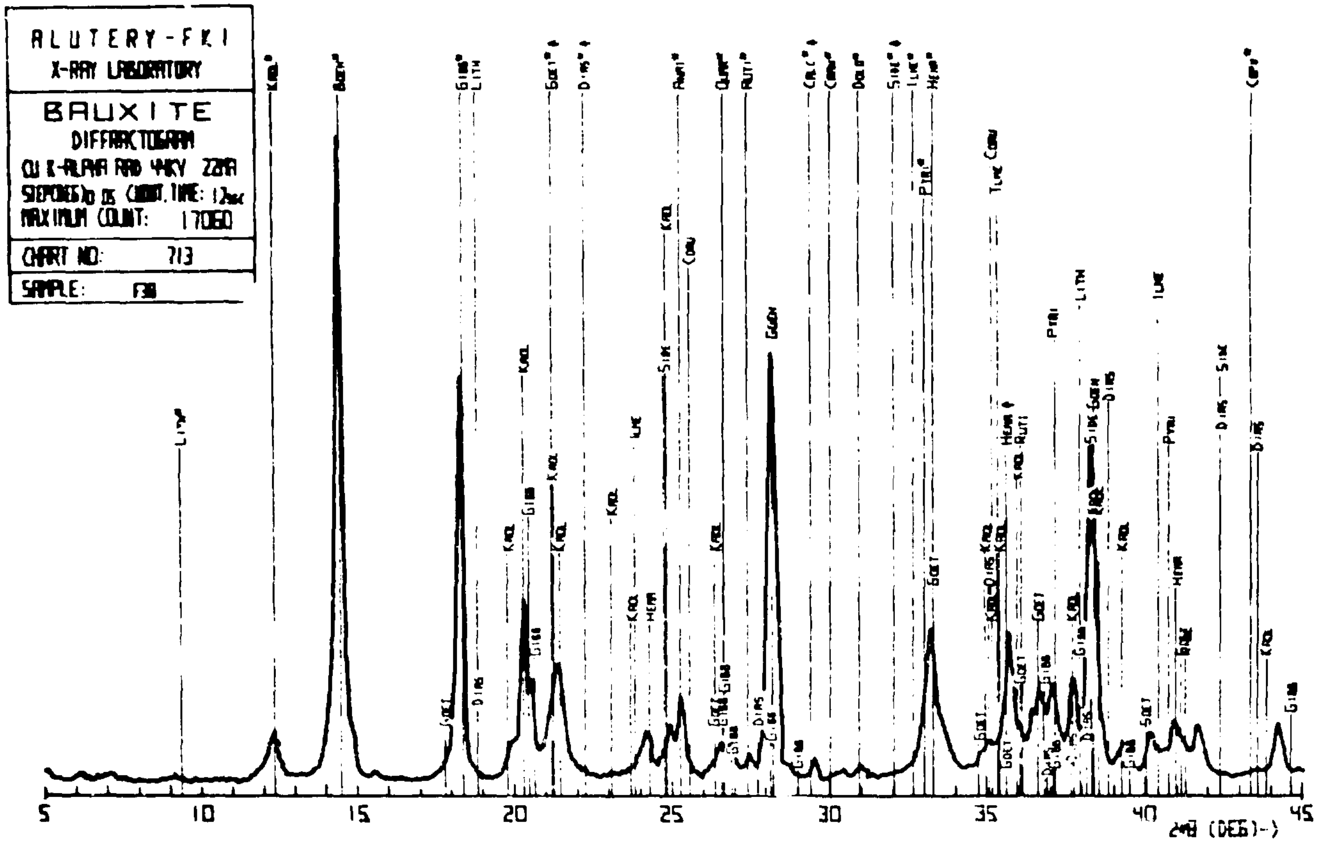
For identification the set of observed d, I values must be compared to some data files which might be available in card, book, microfiche or magnetic tape form. General requirements are most frequently satisfied by the PDF (Powder Diffraction File) of the JCPDS (Joint Committee for Powder Diffraction Standards). [23] In the more restricted field of bauxite mining and alumina technology the "Minerals" subfile covering about 5,000 minerals is sufficient. When repeatedly the same kinds of minerals are studied a small local file including some dozen or hundred data sets should be compiled, or taylored upon order by JCPDS.

Conversion of the measured intensities to the relevant concentrations of the individual phases can be carried out by relatively simple manual or machine calculations [15]. The whole work of data reduction and interpretation can be aided by a microcomputer, nevertheless, human control seems to be important, thus interactive execution is deemed to be optimal. Descriptions of appropriate routines for the computerized quantitative phase analysis of multicomponent mineral mixtures can be found in the works of Bårdossy, Gy. [16] and P.D. Johnson [17].

The mineralogical composition of a Hungarian bauxite sample gained by this method is demonstrated in Fig. 12 and Table 10. For further information on X-ray analysis it is proposed to study books and periodicals given under Ref. Nos. 18-27.

4.2.3. Size and Organization of an X-Ray Laboratory Staff

As already mentioned an X-ray laboratory can do service in the bauxite-alumina industry to the geological survey work, to the mining activities and for the alumina plant control. Because of the high investment costs ranging from 80,000 US$ to 540,000 US$ (without expenditure on building and related investments) as listed in the Table 11, it is questionable whether the analyses needed for the exploration of a single bauxite field can justify the establishment of a laboratory. It seems to be desirable, therefore, to run a laboratory to serve several enterprises and it should be borne in mind that many other industrial sectors of a country might simultaneously benefit from the existence of such a facility, provided that it is well organized. The appraisal of geological samples can be extended to other ores and minerals. Investigation of soils, cements, labour safety control in connection with silica and asbestos, many branches of chemical, ceramic



COMPUTER DRAWN XR DIFFRACTOGRAM OF BAUXITE F 38.

FIG. 12.

Table 10

ALUTERV-FKI X-RAY LABORATORY
BUDAPEST, 79.06.20.

SAMPLE: F 38
RECORD No.: 713
FILE No.: 50/19

GOETHITE: 22.7 m% AlOOH d(110) = 4.152 d(110)st = 4.200 A

	Al_2O_3%	Fe_2O_3%	SiO_2%	TiO_2%	CaO%	MgO%	FeO%	SO_3%	P_2O_5%	LOI%	Integr.i	PHASE %	ICF	REL.DEV.	RICF
HEMATITE		8.89									36634	8.9	0.78	-1.8	1.00
BOEHMITE	28.13									4.96	106737	33.1	1.00	0.0	1.00
GOETHITE	1.85	9.86								1.44	27080	13.1	1.57	-1.3	1.50
GIBBSITE	15.81									8.36	59994	24.2	1.30	2.0	1.00
KAOLINITE	5.38		6.33							1.91	11931	13.6	3.68	-1.1	5.40
ANATASE				1.55							13552	1.5	0.37	0.7	0.80
CALCITE					0.32					0.25	1663	0.6	1.11	3.5	1.00
DOLOMITE					0.56	0.40				0.88	1751	1.8	3.40	15.0	1.00
RUTILE				0.65							4549	0.7	0.46	0.6	1.00
CRANDALL	0.23				0.09				0.29	0.10	432	0.7	5.27	0.1	2.00
PYRITE		0.25					0.22	0.50		0.12	853	0.4	1.41	4.1	1.30
ADS H2O										0.43		0.4	BOEHMITE REF		
CAL.COMP	51.40	19.00	6.33	2.20	0.97	0.40	0.22	0.50	0.29	18.45		99.0			
ANALYSIS	51.40	19.00	6.33	2.20	0.97	0.40	0.19	0.50	0.29	18.34		99.4			

and dye-stuff industries need similar measurements as the alumina production, thus a well arranged central service can provide analyses for all. The level of completeness of the equipment can be adjusted to the actual needs and gradual expansion from a modest start is subsequently possible.

Table 11

AVERAGE INVESTMENT COSTS OF X-RAY APPARATUS
(1981)

Equipment	Characteristic price (US$)
Generator + diffractometer assembly	80,000
Generator + sequential spectrometer	110,000
Sample preparation laboratory for XRF	32,000
Closed loop water cooling	12,000
Updating of diffractometer assembly by microprocessor control	30,000
Generator + computer controlled diffractometer	160,000
Simultaneous spectrometer for 20 channels under computer control	350,000

Table 12

LIST OF SOME MANUFACTURERS OF X-RAY APPARATUS

Name	Address	Type
XRF		
Philips	S & I Export, Eindhoven, The Netherlands	PW 1410 Sequential PW 1600 Simultaneous PV 9100 EDAX HAG Bead Machine ZEM 3 Water Cooling
Siemens	pf.211080, D-7500 Karlsruhe 21, BRD	SRS 200 Sequential MRS 400 Simultaneous HSM 100 Mill HTP 40 Press USG Melting apparatus
ARL	9545 Wentworth St., Sunland CA 91040, USA	72000 S Simultaneous
Toshiba	1-1 Uchisaiwaicho Chiyoda-ku, Tokyo, Japan	AFV - 201
JEOL	1418 Nagakami Akishima Tokyo 196, Japan	JSX 60S4
Rigaku Denki	2-9-8 Sotokanda Chiyoda-ku, Tokyo, Japan	Geigerflex 3063 Sequ. Simultix
VEB Präzisions Mechanik	Hainicher Str. 2a. 92 Freiberg (Sachsen) GDR	VRA 20 Sequential
SKB RA "Burevestnik" (Leningrad)	V/K Techsnabexport Smolenskaja-Sennaja, 32/34 Moscow G-200 USSR	FRK 1 b FRS 4 KRF 11
Telsec	Sandy Lane West, Littlemore, OXFORD, U.K.	TXRF Simultaneous
CGR	48 Bd. Galliéni, 92 Issy-Les-Moulineaux (Seine) France	Spectrometre à Rayons X
SECASI	Avenue Alexander Eiffel Parc Industriel de Pessac Bordeaux - 33	Spectrometre à Rayons X

Table 12 (cont.)

Name	Address	Type
XRD		
Philips	see above	PW 1050 APD 15
Siemens	see above	D 500/501
VEB Freiberger Präzisionsmechanik	see above	HZG - 4, HZG - 5
SKB RA "Burevestnik"	see above	DRON 0.5
Rich.Seifert& Co.	Bogenstr. 41 2070 Ahrensburg bei Hamburg, BRD	ISO Debyeflex MZ - III
General Electric	4855 Electric Ave Milwaukee, WIS 53201 USA	SPG Spectrogoniometer
JEOL	see above	JDX 7S
Rigaku Denki	see above	Geigerflex 2034

The staff of a laboratory should be sized in accordance with the required number of analysis. The reasonable minimum number of personnel may be set at two, provided maintenance and repairment service is available externally. The two manned team should consist of a (research) chemist and a technician trained for the operation of complicated instrumentation. If maintenance and repair must be hired from remote location, the laboratory should rather include its own engineer or technician capable to decode circuit diagrams and locate/repair faulty components in modern semiconductor electronics with digital technique.

The full exploitation of the capacity of an up-to-date diffractometer or spectrometer justifies the enrolment of 4-5 persons (2 scientists and 2-3 technicians/laborants) and if both X-ray techniques are practiced extensively it seems reasonable to double this number.

4.2.4. Layout of an X-Ray Laboratory

Rooms necesary in an X-ray laboratory are as follows:

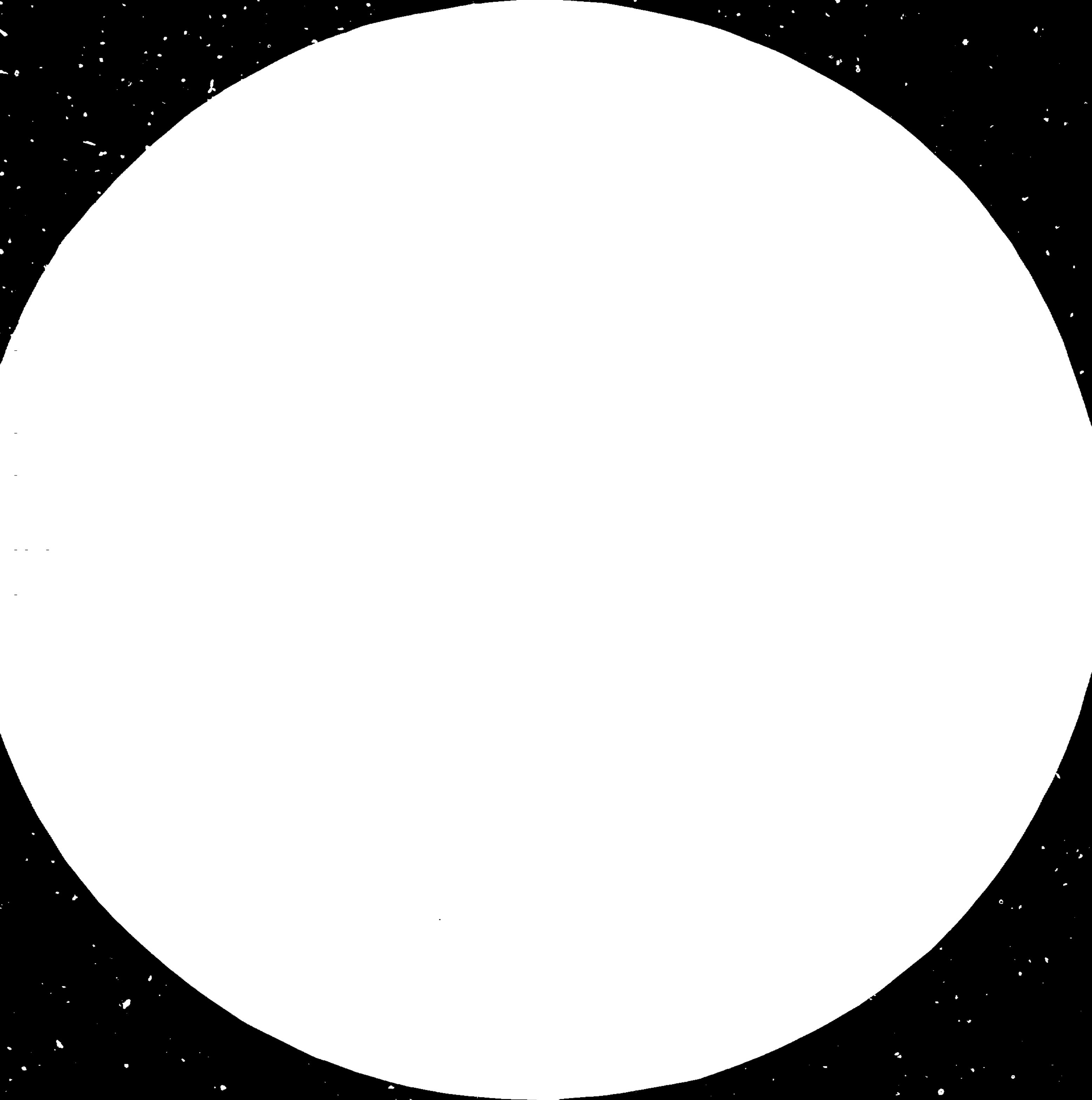
sample preparation room (approximately 12 m^2 if only XRD or XRF are used and 20 m^2 or preferably two separate rooms of 12 m^2 each, if both are applied)

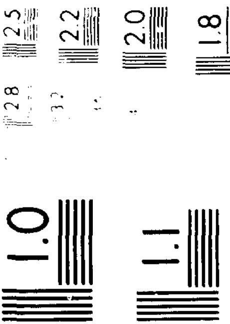
instrument room (15 m^2 for the first unit and about 10 m^2 for each additional; the water cooling apparatus must be placed in a separate room of least 8 m^2 area, 3-5 m apart from the measuring units and working places)

working rooms of the staff (depending on national standards it can be recommended to provide about 10 m^2 separate for each scientist and about 6 m^2 for each technician (laborant)

stock room for spare parts, consumables (about 4 m^2)

A typical lay-out is shown in the sketch below (Fig. 13).

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1.1
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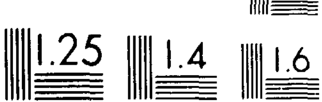
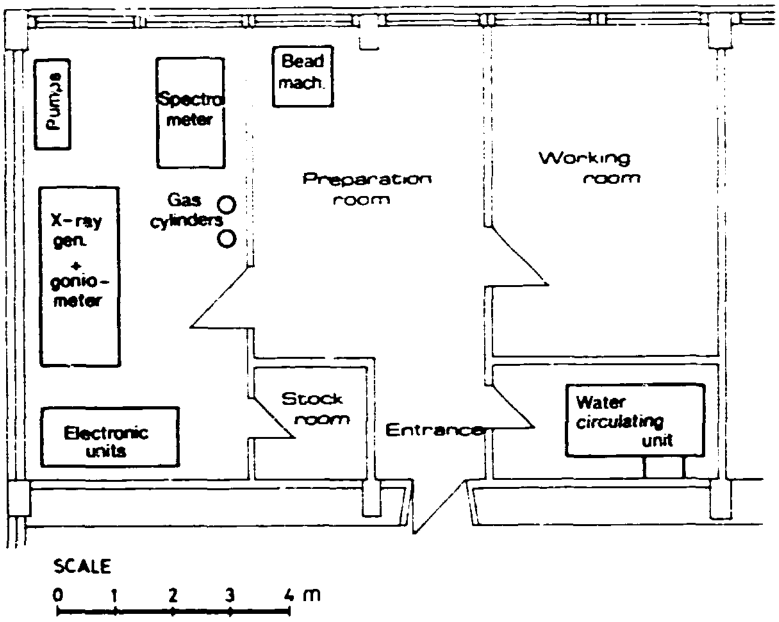
2.5
2.8


1.25
1.4
1.6

[illegible]



A TYPICAL LAYOUT FOR X-RAY LABORATORY FIG. 13.

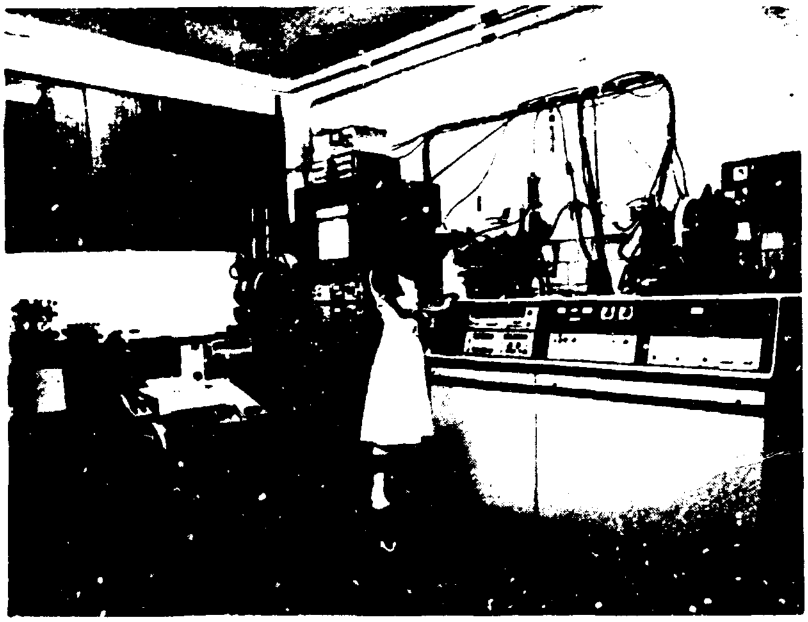
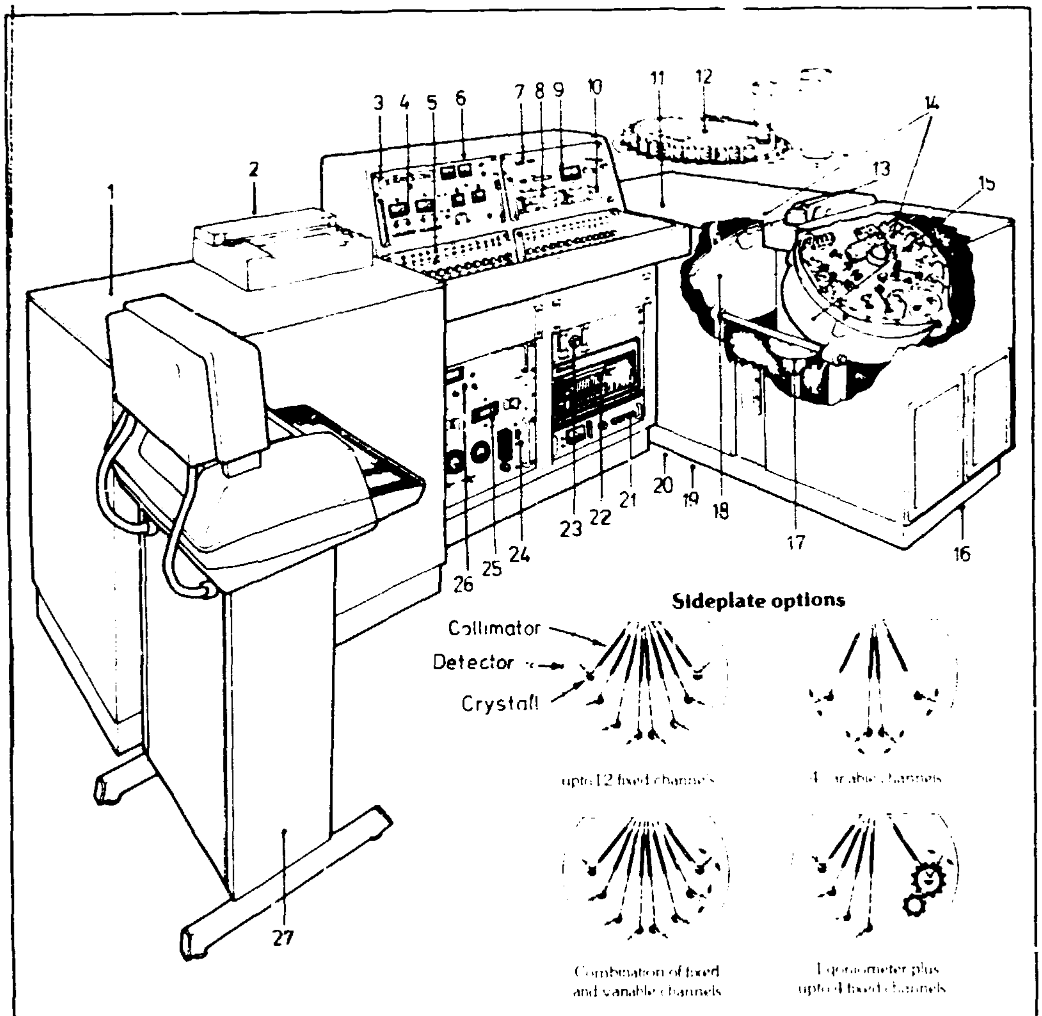


Fig. 14
Combined XRD and XRF Equipment



1 High Voltage generator for X ray tube
2 Optional chart recorder
3 X ray warning light
4 Generator control unit
5 Nucleonics units (One module per channel)
6 Vacuum monitor
7 Sample presenter control
8 Programme control
9 Rate meter
10 Goniometer control
11 Temperature stabilizer
12 Optional 50 position automatic sample presenter
13 Sample holder
14 Side plates
15 Spectrometer drum I
16 Deionised water system
17 X ray tube
18 Spectrometer drum II
19 Hydraulics
20 Vacuum system
21 HT supply for counter
22 Computer
23 Counter gas control
24 Low voltage power supply
25 Safety interlocks
26 Mains stabilizer
27 Printer

Fig. 15

Sketch of an XRF Instrument
(Telsec, U.K.)

4.3. LABORATORY FOR TECHNOLOGICAL TESTS

In this laboratory the operations of the Bayer-process are simulated under laboratory conditions. The so called process modelling units serve for the simulation of different stages of the Bayer process, namely to study predesilication, digestion, red mud separation and washing; precipitation of $Al(OH)_3$ from aluminate solution enriched in contaminants; and calcination (including physico-chemical control of alumina) and to evaluate the effects of bauxite contaminants on the whole circuit.

This laboratory includes, of course, sample preparation units satisfying the special requirements of technological investigations (preparation of representative samples and grindability tests).

Some information for technological evaluation can be gained through the geological reports, based on chemical and mineralogical analyses of bore-hole samples.

The total amount of the available (economically recoverable) bauxite reserves sets the limit of the maximal capacity of the projected alumina plant. 40-50 years lifetime is usually considered for a project.

The mineralogical composition should be determined in the first approach to fix the main technological parameters, in such a way that for the sake of a more simple (more economical) processing max. 7 to 10 % of the total alumina can remain undigested.

The expected specific bauxite consumption can be calculated by the following formulae:

$$\text{dry bauxite, t/t} = \frac{105}{\text{digestible } Al_2O_3\text{, \% in bauxite}}$$

$$\text{wet bauxite, t/t} = \frac{\text{dry bauxite, t/t}}{100 - \text{adsorbed moisture, \%}} \cdot 100$$

The number 105 allows for the fact that estimated Al_2O_3 losses in various processes amount to 50 kg/t of Al_2O_3 product. The different types of bauxite are processed by different technologies, so "digestible Al_2O_3 content" means Al_2O_3 in gibbsite, in gibbsitic bauxite; in gibbsite + boehmite, in boehmitic bauxite; and Al_2O_3 in gibbsite + boehmite + diaspore, in diasporic bauxite, respectively.

During the chemical reaction between the bauxite and digesting liquor 1 kg reactive silica causes roughly 1 kg of NaOH loss and further 20 kg NaOH(t Al_2O_3 product) soda loss is non-silica related. Consequently the estimated total caustic soda losses are:

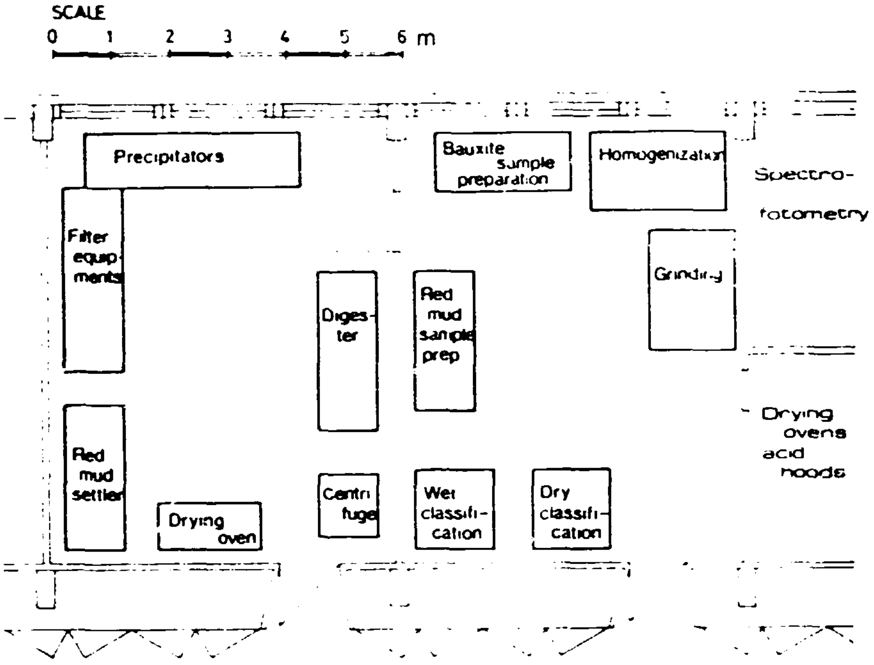
Total NaOH loss, kg/t = 10 . dry bauxite t/t . reactive SiO_2, % + 20

These estimations are only preliminary, for an accurate calculation thorough technological tests are required.

Information about the realization of technological investigations are supplied in Appendix 3 and in the recommended literature.

In this volume technological evaluation and examinations are discussed only for the Bayer process, since that is only prospectivic and economical.

A simple technological laboratory is shown in the sketch of Figure 16. This laboratory consists of 3 basic units: sample preparation, technological examinations and sample storage. Naturally this laboratory can be operated only in close co-operation with other laboratories (chemical, physico-chemical, mineralogical) engaged with the study of the raw materials, the intermadiate - and final - products.



A TYPICAL LAYOUT FOR TECHNOLOGICAL LABORATORY FIG 16

4.3.1. Laboratory for Sample Preparation, Grindability Tests

Functions

a) all preparatory work for technological tests:
  - preparation of representative samples
  - coding and storage of samples
  - drying, grinding, weighing of bauxite samples
  - preparation of aluminate solutions
  - fast analysis of aluminate solutions

b) grindability and ore benefitiation tests

Principles

The tests planned to be carried out determine the form and amount of the representative samples. All laboratory tests considered herein require a quantity of about 50 kg representative sample.

The amount of representative sample required for the test of a pilot plant is of the order of magnitude of 100 tonnes.

If ore dressing investigations are necessary all individual samples are required in a lumpy form.

The principles of representative sample preparation are as follows:

- the number of individual samples may be about 100 (they are chosen on the basis of the analytical results)
- each individual sample weights 0.5 to 1.0 kg
- in the determination of the cut-off limit (depending on the quality) the mining program should be taken into consideration
- it can be advisable to prepare two or three composite samples of different grades by applying different cut-offs
- the samples have to be mixed, usually in a rotating drum for 1 or 2 hours
- after applying the standard quartering and coning practice a part of each composite is bottled and sealed.

Laboratory tests and subsequent economic calculations will show the "optimum grade" determined by a trade-off between plant capacity as well as life-time on one hand and operating costs (bauxite, caustic soda and energy consumption) on the other. The sample corresponding to this optimum grade is called "representative sample".

The principles of sample preparation, coding and storage techniques were discussed in chapter 4.1.1. and these can be applied here as well.

For modelling individual stages of the Bayer process aluminate solutions can be obtained in the following way:

a) using appropriate liquor of an existing plant in the required concentration

b) synthetizing clear sodium aluminate solutions ($NaOH+Al/OH/_3$ or NaOH + 99.99 % Al) and adding contaminants as required.

It should be noted here that industrial liquor quality (contaminated) can be reproduced only on a large laboratory or pilot plant scale after a longer period of continuous operation.

To evaluate the technological tests a large number of fast and accurate liquor analyses is required and it is necessary to carry out them on the site. The thermometric titrimetry, elaborated by ALUTERV-FKI-VKI, Budapest, seems to be an adequate method for liquor analysis.

The aim of bauxite grinding is to render the ore more permeable and digestable for the digesting liquor. However, excessive comminution affects adversely the red mud settling and it is detrimental to the energy consumption, too. Consequently, comminution has to warrant optimal grain-size (corresponding to minimal energy consumption).

A suitable practice, for characterizing the grindability is to determine the Bond index (described in Ref. [28]).

Nevertheless, in the practice of manufacturing alumina, wet grinding is carried out in the presence of caustic (digesting liquor) where the caustic has also chemical and disintegrative effects on the ore. It is advisable to simulate this process also in the laboratory. If it is wanted to determine the optimal conditions of grinding (liquor concentration, solid concentration, ball charge in mill, comminution time) tests with various, systematically altered parameters should be carried out.

The ore dressing (beneficiation) is accomplished on industrial scale for some lateritic bauxites (of high kaolinite content) by washing off the highly reactive silica with water. This can be modelled in laboratory by means of a grinding and consecutive wet sieving test, but before the industrial realization large laboratory and pilot plant scale experiments are needed.

Besides the diminution of silica content by means of washing, a few other contaminants can also be successfully eliminated by ore dressing methods provided the contaminants are favourably distributed (texture), e.g. siderite by magnetic separation, calcite by heavy suspension or magnetic separation.

Equipment

- jaw crushers (see Table 13)
- drum crushers (see Table 13)
- wet and dry grinding mills (see Table 14)
- separators (see Table 15)
- dry/wet sieving series (see Table 16)
- sample reducers and splitters (see Table 17)
- vibration mill (see Table 18)
- cyclons (see Table 18)
- drying ovens (see Table 19)
- analytical and physical balances (see Table 19)
- sample containers
- tanks for solutions with stirrers (see Table 20)
- thermometric titrator (see Table 8)
- potentiometric titrator (see Table 6)

Table 13

JAW/DRUM CRUSHERS

Type	Producer Firm	Specifications	Price, $
Pulverisette 1 Typ 2 Jaw Crusher	Fritsch GmbH D-6580 IDAR-OBERSTEIN-1 FRG	Feed opening: 100x100 mm Capacity: 150-700 kgs/h Product size: 1-15 mms	3,200 (1980)
		Push-plate: Tungstencarbide + Cabalt	7,000
		CrNi-steel	700
5"x6" Laboratory Jaw Crusher	Denver Equipment Co. 1400 Seventeenth St DENVER 17, Colo USA	Feed opening: 125x150 mm Capacity: 300 kgs/h Product size: 10-30 mms Push plate: Mn steel	7,000
MN 934/1	KHD Ind. AG Humboldt Wedag D-463 Bochum BOBox 2730 FRG	Drum diameter: 250 mms, width: 150 mms Capacity: 200-1,200 kgs/h Max. feed piece size: 12 mms Product size: 0.2-3 mm Drum casting: hard steel (550 Brinell)	10,000
Flex Roller Mill (Drum Crusher)	Karl Marz Maschienenfabrik D-7890 Waldshut-Tiengen 2 BOBox 1220 FRG	Drum diameter: 200 mms, width: 160 mms Capacity: 200-1,200 kgs/h Max. feed piece size: 12 mms Product size: 0.2-3 mm Drum casting: hard steel or stainless stul or hard porcellain	20,000
10"x6" Laboratory Crushing Rolls	Denver Equipment Co. 1400 Seventeenth St DENVER 17, Colo USA	Drum diameter: 250 mms, width: 150 mms Capacity: 2,000 kgs/h Max. feed piece size: 10 mms Product size: +0.25 mm	10,000

Table 14

LABORATORY DRY/WET GRINDING MILLS

Type	Producer Firm	Specifications	Price, $
Contraplex Labormühle 63-C	Alpine AG D-89 Augsburg-1 POBox 101109 FRG	Feed charge: 1-20 kgs product size: +5 µm (at 5 Mohs hardness)	7,000
Universal Labormühle C 100 LU	Alpine AG D-89 Augsburg-1 POBox 101109 FRG	Capacity: ~ 100 kgs/h product size: 50-100 µm (at 5 Mohs hardness) grinds and classifies	7,000
Combimühle-95	Fryma Maschienen AG POBox 325 Theodorshofweg CH-4310 Rheinfelden SWITZERLAND	Capacity: 500 kg/h product grain size: 1 µm - 2,5 mm teeth or perforated steel grinding disks or simple corundum disks	10,000
Laboratory ball mill 1-25-*LK*	Alpine AG D-89 Augsburg-1 POBox 101109 RFG	Porcellain or stainless steel grinding pots: 1...25 l product size: 30-100 µm Autostop at 100 rpm for Bond index determination	12,000
Laboratory jar mill + rolls	Denver Equipment Co. 1400 Seventeenth St DENVER 17, Colo USA	Ceramic and steel grinding pots: 6...10 l product size: 30-100 µm	8,000
Separator mill A 100 MZR	Alpine AG D-89 Augsburg-1 POBox 101109 RFG	Capacity: 1-2 kgs/h cut off: 1...100 µm	25,000
Separator mill 40 MZM	Alpine AG D-89 Augsburg-1 POBox 101109 RFG	Capacity: 50 kg/h cut off: 0.1...10 mm	25,000
M-20 Universal Mühle	IKA-Werk D-7813 FRG	for regrinding of red mud or other dry minerals	1,000

Table 15

MAGNETIC SEPARATORS

Type	Producer Firm	Specifications	Price, $
LHW Laboratory Wet Magnetic Separator	BoxMag Rapid Chester Street, Aston Birmingham B64AJ ENGLAND, U.K	Feed charge: 200 g Feed grain size 3 µm-3 mm	10,000
LOG 14 Dry Disc Magnetic Separator	BoxMag Rapid Chester Street, Aston Birmingham B64AJ ENGLAND, U.K	Capacity: 20-50 kgs/h also for continuous operation	10,000
Magnetic Filter Separator	N.M.E. Mineral Dressing Department Miskolc-Egyetemváros HUNGARY	Fully automatized Feed: max. 100 µm also works below 3 µm Capacity up to 5,000 kgs/h	10,000
Wiefley Type Concentrating Table	Denver Equipment Co. 1400 Seventeenth St DENVER 17, Colo USA	Table size: 1,000x450 mms Capacity: 25-75 kgs/h	1,500

Table 16

DRY/WET SIEVES FOR ANALYTICAL PURPOSES

Type	Producer Firm	Specifications	Price, US$
A-200 LS Air jet sieve completed with: - cyclone - small filter - fine filter - vacuum cleaner - normal - micro sieve series	Alpine AG D-89 Augsburg-1 POBox 101109 FRG	relative error < 1 % measuring time: 2...30 min/sieve 15 W high pressure 1 m^2 filterpaper, 300 mm diameter 800 W 32...250 um dia 200 mm 10...25 um dia 75 mm	11,300 (1979)
AJ-200 (Alpine licence)	Gilson Co.Inc. POBox 99 Malinta 43535 OH USA	see at Alpine	10,500 (1980)
Analysette 3 wet screen + 3 jetrings	Fritsch GmbH D-6580 IDAR-OBERSTEIN-1 FRG	relative error < 1.5 % measuring time 6...60 min/4 sieves normal: 25 mm...20 um micro: 5...100 um sieve diameter: 200 mm (normal) 110 or 75 mm (micro)	10,500 (1980)
Wet-Vac 2	Gilson Co.Inc. POBox 99 Malinta 43535 OH USA	measuring time: 3...30 min/6 sieves relative error: < 1.5 % sieve diameter: 203 mm screen: 20...100 um energy consumption: 2.2 kW	11,500 (1980)
Sieve Cleaners Laborette 17 Grösse 2	Fritsch GmbH D-6580 IDAR-OBERSTEIN-1 FRG		2,200 (1980)
Ultrasonic bath Model UB-5	Gilson Co.Inc. POBox 99 Malinta 43535 OH USA		2,100 (1980)

Table 17

SAMPLE REDUCERS AND SPLITTERS

Type	Producer Firm	Specification	Price, US$
Laboratory size reducer: PKZ + DR40	Retsch GmbH, F.Kurt Fabrik Chemischer Apparate Reinische Str.36 5657 Haan 1, FRG	volume: 30 l sampling ratio: 1/10...1/200 speedness: 1...60 min/charge	1,500
Pilot plant size reducer PT-50	Retsch GmbH, F.Kurt Fabrik Chemischer Apparate Reinische Str.36 5657 Haan 1 FRG	continuous ratio: 1/10...1/80 equipped with ball mill capacity: 75...750 kgs/h sample holder change in every hour feed: 60...120 mm (depending on crusher type)	3,500
Electromagnetic laboratory splitter, Laborette 10	Fritsch GmbH D-6580 IDAR-OBERSTEIN-1 FRG	sample reciener: 10x500 ccm minimum sample: 20 ccm max. particle size: 6 mm	1,500
Universal sample splitter SP 1,2,3 pilot plant	Gilson Co.Inc. P.O.Box 99 Malinta 43535 OH USA	feed: 60 µm... 50 mm capacity: up to 250 kg	200...850
SP-10 Hand model		capacity: max. 1.8 kg	250
SP-25 Laboratory model		capacity: 50 g...25 kg	750
automatic sampler SM-1...6-L		feed: 10...100 mm capacity: 50...80 l	1,100...5,700
split-O-matic splitter SM-30...66-A		feed: 10...800 mm capacity: 50...250 l ratio: 1/8 or 1/64	1,400...4,000

Table 18

VIBRATION MILLS AND CYCLONES

Type	Producer Firm	Specifications	Price, US$
Pulverisette 9 (vibration cup mill)	Fritsch GmbH D-6580 IDAR-OBERSTEIN-1 FRG	sound filter, timer dry or wet 1 cycle: 1...3 min feed: up to 7...12 mm cup volume: 50, 100, 250 ml	3,400
		cup material: hardened steel	1,600
		tungsten carbide	15,000
		agate	3,500
Pilot plant scale: Laboratory vibrating wet grinding mill M-18	Denver Equipment Co. 1400 Seventeenth st DENVER 17, Colo USA	capacity: 10 l product size ~ 1 µm Mn steel or ceramic ball	4,000
Diameter 304.8 mm vibration mill	William Boulton Ltd Burslem Stoke-on-Trent ST63 BQ U.K.	similar to Denver	~ 4,000
		CYCLONES	
MN-918/1	KHD Ind. AG Humboldt Wedag D-4630 Bochum P.O.Box 27[illegible] FRG	material: Plexi-glass	4,500
CT 8/5 +8xGA80	Bányászati Kutató Intézet Pf. 115 1300 Budapest HUNGARY	separation at 10...100 µm optimum at ~ 30 µm capacity: 2...15 m^3/h pressure gauge: 2 MPa	5,000
Laboratory Testing Cyclone also for pilot plants	Krebs Engineers 1205 Chrysler Dr Menlo Park, CA 94025 USA	set of cyclones (D-4...D-50, D-6-CA, PC-1) separation at: 1...250 µm	8,000
Laboratory Cyclones	Amberger Kaolinwerke GmbH Equipment + Construction D-8452 Hirschau P.O.Box 1140 FRG	set of cyclones (5 µm...2 mm)	3,000
		antiblocking filter	2,000

Table 19

FURNACES, DRYING OVENS, THERMOTANKS, WATER DISTILLERS, ANALYTICAL/PHYSICAL BALANCES

Type	Producer Firm	Specifications	Price, US$
KSL 170 furnace	W.C.Herans GmbH P.f. 1553 6450 Hanau 1 BRD	temp: up to 1,600 °C useful room: 170x220x535 mm energy consumption: 8.5 kW	25,000
D-5060 E drying oven	W.C.Herans GmbH P.f. 1553 6450 Hanau 1 BRD	temp: up to 300 °C useful room: 600x600x580 mm energy consumption: 2.7 kW	4,000
T-5100 E drying oven	W.C.Herans GmbH P.f. 1553 6450 Hanau 1 BRD	temp: up to 250 °C useful room: 1,024x1,350x580 mm energy consumption: 5.0 kW	6,000
LP-309 drying oven	Labor MIM 1450 Budapest P.f. 33 HUNGARY	temp: 200 °C useful room: 520x600x1,000 mm energy consumption: 4 kW	1,800
LP-312	Labor MIM 1450 Budapest P.f. 33 HUNGARY	temp: 300 °C useful room: ~ 300 l	4,000
Water Still Double Distiller for Water Typ 2108	Gesellschaft für Labortechnik mbH Schulze-Delitzsch Str.4 D 3006 Burgwedel 1	capacity: 8 l/h energy consumption: 11.5 kW	10,000
Waterbath Typ 1042	Gesellschaft für Labortechnik mbH Schulze-Delitzsch Str.4 D 3006 Burgwedel 1	6x13[illegible] mm	500
Analytical balances	Sartorius GmbH P.f. 19 D-3400 Göttingen FRG	electronical precision 0.1...0.01 mg mechanical precision 0.1...0.01 mg	1,000-4,000 8,000-2,000
Physical balances	Sartorius GmbH P.f. 19 D-3400 Göttingen FRG	electronical max. weight: 30-60 kg precision 0.1-1 g	3,500-4,000
Toledo Model 48[illegible]	Metripond Mérlegyár Hódmezővásárhely HUNGARY	electronical max. weight: 15 kg precision ~ 1 g digital weigh/price display	3,000
Florence	Metripond Mérlegyár Hódmezővásárhely HUNGARY	optical weigh/price display max. weigh: 3 kg precision ~ 1 g	1,000

Table 20

STIRRED TANKS, THICKENERS, CENTRIFUGES

Type	Producer Firm	Specifications	Price, US $
180/180 150/160 210/210	Apritógépgyár 5101 Jászberény Pf. 65 HUNGARY	diameter: 1,800 mm, height: 1,800 mm diameter: 1,500 mm. height: 1,600 mm diameter: 2,100 mm, height: 2,100 mm	2,500 9,000 4,500
Agitator tanks 3'x3'	Denver Equipment Co. 1400 Seventeenth St DENVER 17, Colo, USA	900 mm x 900 mm	~ 10,000
Attrition machines (2 cells) 11"x11"	Denver Equipment Co. 1400 Seventeenth St DENVER 17, Colo, USA	250 mm x 250 mm.	~ 5,000
Thickener [illegible]'x2.5'	Denver Equipment Co. 1400 Seventeenth St DENVER 17, Colo, USA	diameter: 900 mm height: 750 mm SP diagram pumps (for underflow)	~ 4,500
Four stage agitator unit 4x12"x15"	SEPOR INC. P.O.B. 1252 Wilmington, CA 90748, USA	4 x 22 l cells propeller agitators	6,500
Ultra Turrax T-652 + R-652 adapter tiristor control	IKA-Werk D-7813 Staufen BRD	for attrition, dispergion emulgeation in laboratory	1,000
Roto Magna/K Centrifuge	Andreas Hettich Pf. 391 D-7200 Tuttlingen	speed: 4,000 rpm volume: 6x1,000 ml or 4x1,500 ml Energy cons. 4.5 kW cooling facility down to - 10 °C	~ 20,000
LV-418 TV + LV-418 HV rotor	Budapesti Vegyipari Gépgyár Budapest, X. P.O.B. 69, HUNGARY	heating facility up to 68 °C volume: 6x500 ml max. speed: 1,700 rpm Energy consump. 1.2 kW	7,000
Total K-120-MS	Budapesti Vegyipari Gépgyár Budapest, X. P.O.B. 69 HUNGARY	spiral decanter centrifuge capacity: 0.5 ... 1.5 m^3/h for pilot plant	50,000

Staff

- 2 technicians
- 3-4 skilled workers

4.3.2. Model Laboratory for Digestion and Settling Tests ("Red Lab")

Functions

- determination of predesilication parameters
- selection of digestion parameters
- determination of settling and washing characteristics of red mud

Principles

Predesilication of bauxites means the transformation of reactive SiO_2 contents into sodium-aluminium-hydrosilicates. The principal aim of predesilication is to reduce the scale formation rate at the slurry side of the indirectly heated equipment of alumina production (slurry preheaters, autoclaves), bringing about an increase in the operation cycle time and a decrease in the specific energy consumption for heating. Similarly, a significant objective is to ensure low SiO_2 content of alumina - first of all in the case of processing gibbsitic bauxites.

The predesilication tests are usually performed in a heated stirred tanks the temperature of which can be regulated in the range of 95-100 °C or in bomb autoclaves used in the digestability tests.

The digestion technology is determined principally by the bauxite's chemical composition and mineral components, however, it has also a significant influence whether floury or sandy alumina is going to be produced.

During digestion the equilibrium molar ratio of the aluminate solution is the most important factor determining in fact the optimum technological parameters of the digestion. The equilibrium molar ratio of aluminate liquor to be used for the digestion of different types of bauxite was determined by Soviet authors. Their results are shown in Fig. 17 [29].

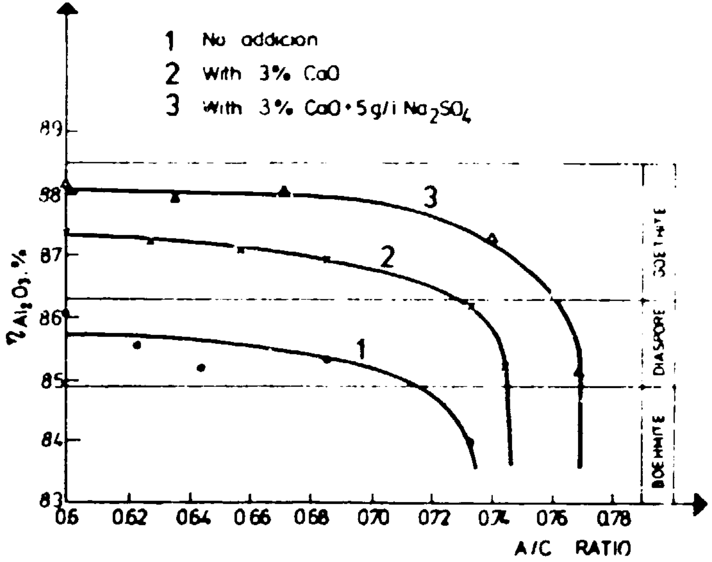
The simplest way for the determination of extractable alumina is the treatment of the bauxite sample by aluminate liquor under standard conditions. However, this method provides no information on the parameters of digestion (optimum temperature, attainable molar ratio etc.). Therefore, the digestion process is modelled in order to determine digestion characteristics under laboratory conditions.

Samples with different bauxite to digesting liquor ratios are digested for given time at the temperature selected on the basis of phase analysis. The aim is set to reach after digestion in some samples the equilibrium molar ratio (saturation of the solution with Al_2O_3), while in others a higher final molar ratio, i.e. the maximum alumina extraction attainable in practice. Digestion is carried out in steel autoclaves rotated in electrically heated, temperature controlled oil, salt or air bath. Digestion characteristics are plotted from the results of 6 to 10 simultaneous tests. The working volume should correspond to the further experimental requirements. Digestion tests are complemented by preliminary and subsequent desilication. After digestion, the aluminate liquor is separated from red mud by centrifuging. In Fig. 18 the Al_2O_3 recovery is plotted as the function of the A/C (alumina per caustic) ratio. The term A/C ratio is defined as the Al_2O_3 gpl per caustic soda expressed as Na_2CO_3 gpl [30].

EQUILIBRIUM CAUSTIC MOLAR RATIO DURING DIGESTION OF BAUXITES OF DIFFERENT MINERALOGICAL TYPES

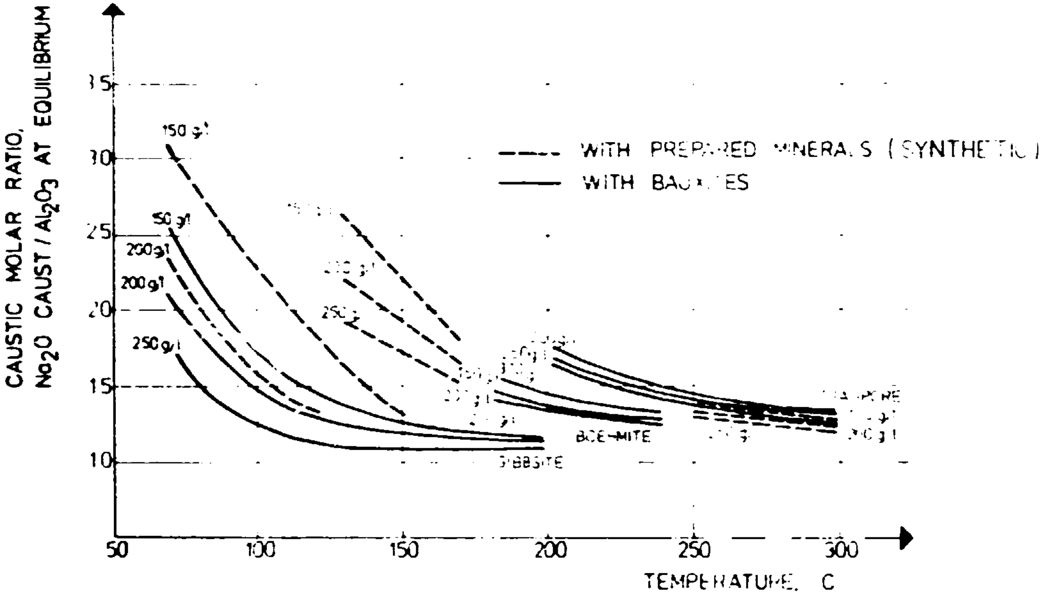
FIG 17

(BY TIKHONOV & LAPKIN, REF. 26)



Al_2O_3 RECOVERY PLOTTED vs. A/C RATIO OF DIGESTED LIQUOR

FIG 18


CAUSTIC MOLAR RATIO,
Na_2O CAUST / Al_2O_3 AT EQUILIBRIUM
35
30
25
20
15
10
50
100
150
200
250
300
TEMPERATURE, C
WITH PREPARED MINERALS (SYNTHETIC)
WITH BAUXITES
150 g/l
50 g/l
200 g/l
200 g/l
250 g/l
200 g/l
250 g/l
GIBBSITE
BOEHMITE
DIASPORE

The gibbsitic and boehmitic types of bauxite are processed usually without additives, while the processing of diasporic and goethitic types 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 Al_2O_3 recovery and NaOH consumption;
- kinetics of dissolution of Al_2O_3 minerals;
- kinetics of goethite-hematite transformation in the presence of different quantities of selected additives.

Invariably, the common parameters like temperature, holding time, caustic concentration and equilibrium molar ratio of the liquor has to be determined as well.

Summarizing the results of the digestion tests and taking into account the varying grade of bauxite to be processed the recommendable parameters will be fixed. Sometimes alternatives of the 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:

Digesting liquor: caust. Na_2O gpl
Digestion temperature, ^{o}C
Holding time, hours
Caustic molar ratio after digestion
CaO addition (referred to dry bauxite)
Expected alumina recovery
Expected Na_2O/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 effects

The digester autoclaves belong to the most important equipment of the technological laboratory. Principally two types are distinguished:

a) standard autoclaves, supplied with mechanic stirrer, available commercially demonstrated in Fig. 19.

b) equipment developed specially for the determination of digestion characteristics. An equipment developed in ALUTERV-FKI is shown in Fig. 20. and one of the six autoclave bombs in Fig. 21.

During settling the solid red mud residue of the digestion is separated from the aluminate liquor and by washing the Al_2O_3 and NaOH content of the red mud slurry is eliminated. Red mud separability and washability depends on bauxite characteristics and the technology (digestion) applied. The optimization of this part of the process is mainly influenced by the previous stages (first of all parameters of the digestion).

By the investigation of red mud settling two main problems should be solved:

a) minimizing the red mud settler and washer capacity,

b) selection of the most efficient flocculant and economical optimization of their dosage.

Mud settling behaviour is expressed in terms of the settling area required per ton of dry mud per day for various mud compactions. In the same time the required clarity of overflow has to be considered, too.

A quick comparative evaluation can be made in simple settling tubes. This method is suitable to compare red mud samples obtained from different types of bauxite or by different digestion technologies or to determine the effect of different flocculants, by observing the settling rate of the mud/liquor

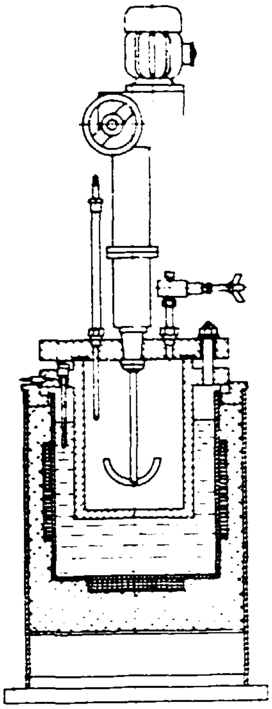


FIG 19 LABORATORY AUTOCLAVE SUPPLIED WITH MECHANIC STIRRER

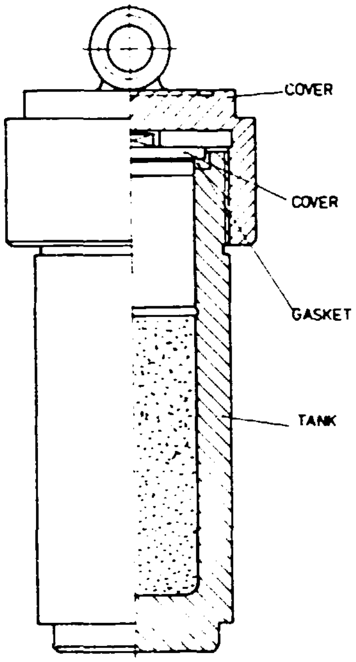


FIG 21 AUTOCLAVE BOMB

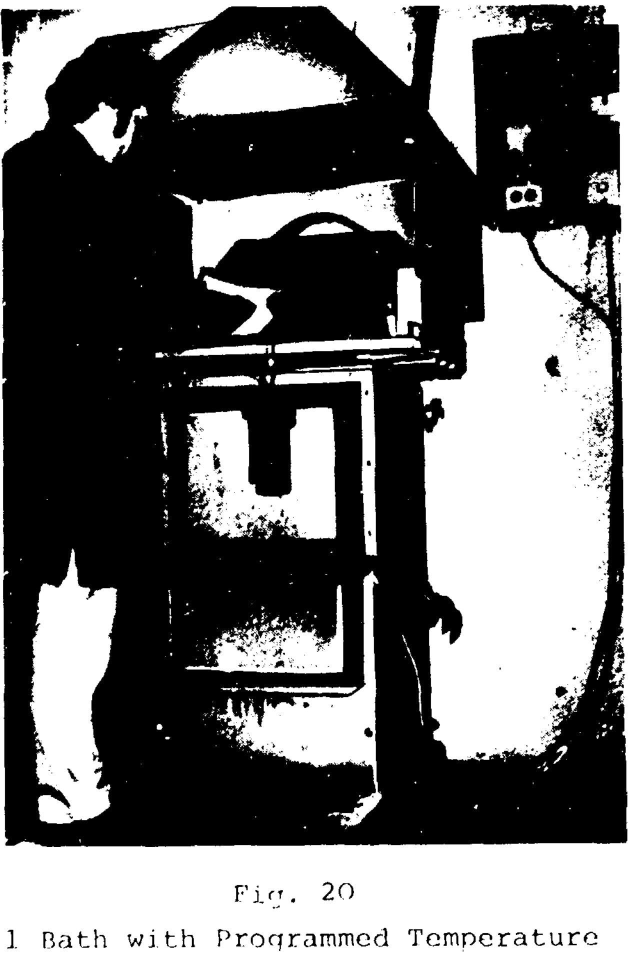
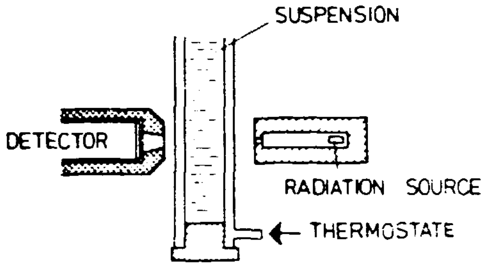


Fig. 20

Oil Bath with Programmed Temperature
Control for Bomb Digester Autoclaves

interface in the slurry using temperature controlled cylinders with appropriate graduation.

According to a recently developed technique the actual settling rate versus the solid concentration can be measured for non-ideal suspensions, too, in a sedimentation tube, at any arbitrary intervals. The principle of the measurement elaborated in ALUTERV-FKI is shown in Fig. 22.



PRINCIPLE OF SOFT GAMMA RAY
ABSORPTION MODEL SETTLER

FIG 22

Red mud sedimentation is carried out in a thermostated glass tube and the solid concentration is measured by means of a system movable along the tube and detecting the absorption of γ-radiation. The photograph of the apparatus is shown in Fig. 23. The radiation source and the detector are located on opposite sides of the sedimentation tube. The result of the measurement, i.e. the intensity of radiation passing through the suspension is displayed by a recorder. The experiment is continued up to 48 hours to determine final concentration formed at the bottom of the settling tube. Exploiting the results thus gained proper dimensions of settlers and washers can be calculated by a computer program and the optimum operating conditions can be established.

For modelling red mud washability in thickeners the slurry will be diluted step-by-step in correspondance with the concentration prevailing at the washing stages, after that cen-

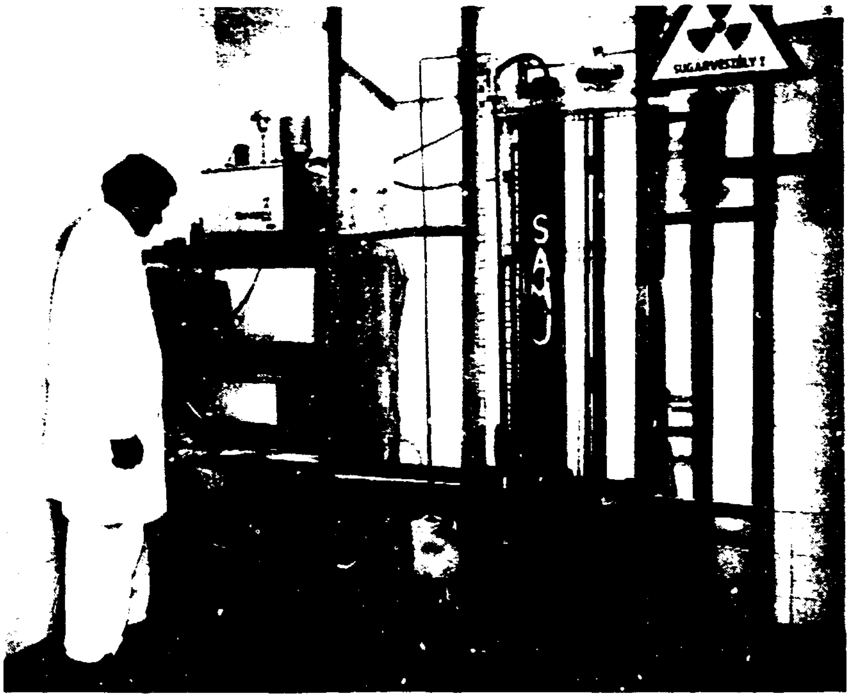


Fig. 23
Soft γ-Ray Absorption Model Settler Equipment

trifugation takes place and then more diluted aluminate solution is pulped again in the next stages and after the required holding time the procedure will be repeated beginning from the centrifugation.

The filtering behaviour of red mud slurries is measured by laboratory scale pressure filters and vacuum filters. The data obtained in this manner are adequate for preliminary evaluations.

Equipment

- thermostates, series of tanks equipped with mechanical stirrers (non available commercially) (see Table 20)
- autoclaves (mechanically stirred, temp. control, up to min. 260 °C) (see Table 21)
- digester assembly with 6-10 bomb autoclaves (150-250 ml each) rotated in a temperature controlled (up to 260 °C) oil or melted salt batch

Table 21

AUTOCLAVES

Type	Producer Firm	Specifications	Price, $
Hochdruck Laborautoklav PM 15 or. PM 25	Friedrich Uhde GmbH Werk Hagen i W Dortmund P.O. Box 425 FRG	Volume: 1; 2; 5 l Operation temperature up to 350 °C Operation pressure up to 25 MPa Magnetic coupling	5,000
High pressure autoclave	Andreas Hofer GmbH Millheim (Ruhr) FRG	Volume: 5 l Operation temperature up to 350 °C Operation pressure up to 20 MPa Coupling: magnetic	5,000
Model No. 4552	Dorr Instrument Co. 211 Fifty-Third Street Moline, Illinois 6165 USA	Volume: 7.5 l Max. temperature: 350 °C Sampling valves: 2	8,000 (1979)

- centrifuges for red mud separation (size corresponding to autoclave bombs) (see Table 20)
- pressure filters (see Table 22)
- vacuum filters (see Table 22)
- settling tubes in thermostate
- soft γ-ray absorption apparatus for settling experiments (ALUTERV-FKI model) ~ 50,000 $

Staff Requirements

- 1-2 engineer(s)
- 2 technicians
- 2 skilled workers

4.3.3. Model Laboratory for Precipitation and Calcination ("White Lab")

Functions

- determination of the parameters of control filtration
- determination of the parameters of precipitation (yield and granulometry of $Al(OH)_3$ as a function of holding time, seed quality and quantity, temperature and concentration of caustic)
- $Al(OH)_3$ filtrability tests
- $Al(OH)_3$ calcination tests

Principles

Before precipitation the clarity of the liquor required for the production of a marketable alumina quality must be ensured.

The aim of control filtration is to remove the suspended Fe_2O_3 from the aluminate liquor. It must be emphasized here that the removal of other contaminants (like Zn, Cr, or-

Table 22

PRESS/VACUUM FILTERS

Type	Producer Firm	Specifications	Price, US $
Laborfilter FC 10	IKA-Werk D-7813 FRG	heatable up to 100 °C centrifuge filter rpm: 0...16,000 capacity: 10 l/h rotor: porcellan special filter paper	~ 7,000
Laboratory press filter	Sartorius GmbH Pf. 19		~ 10,000
SM-16260	D-3400 Göttingen	Thin layer (d: 100 mm)	1,[illegible]00
SM-16540	BRD	Thick precoke (d: 142 mm) heatable up to 134 °C,	1,000
SM-16277		Thin layer (d: 273 mm) d = 100 mm, volume 1 l	1,600
SM-16263 accessories		tank, compressor, high pressure slurry pump, etc.	1,000 ~ 5,500
Laboratory Cabinet drum filter 0.25 m²	KHD Ind. AG Humboldt Wedag D-463 Bochum P.O.Box 2730, FRG	with scaper, washing nozzles, washing belt material: Cr-Ni-Mo-steel filter cycle: 0.2 ... 1.8 rpm Swivel agitator (43 double stroke/min)	20,000
Laboratory drum filter 18"x6" station	Denver Equipment Co. 1400 Seventeenth St DENVER 17, Colo, USA	same as Wedag	20,000
Laboratory disc filter 4.5 sq Ft station	Denver Equipment Co. 1400 Seventeenth St DENVER 17, Colo, USA	filtering area: 0.4 m² double disc	25,000
Duplex laboratory pan filter 5"x5"	Denver Equipment Co. 1400 Seventeenth St DENVER 17, Colo, USA	area: 0.62	1,000
Laboratory counter balanced press filter	Denver Equipment Co. 1400 Seventeenth St DENVER 17, Colo, USA	Capacity: 22 l diameter: 300 mm (12")	2,000
Drum filter	*Láng Gépgyár* Budapest, Pf. 62 HUNGARY	filtering area: 1 sq.m (for pilot plant)	20,000
Drum filter PTA 1/1-A	Tatabányai Szénbányák Tatabánya, Pf. 320 HUNGARY	1 sq.m filter area disc filter	20,000
AJMO press filter	*Láng Gépgyár* Budapest, Pf. 62 HUNGARY	filtering area: 5 sq.m (for pilot plant) automatic discharge filling pressure 10 MPa pressing gauge 36 MPa	50 000

ganics oxalate etc.), requires the elaboration of special technologies and the accumulation of experiences relevant to the particular conditions.

Laboratory investigations are carried out by small laboratory filters with different precoats.

The precipitation occurres continuously in the commercial practice, but the principal parameters can be derived using laboratory scale batch precipitators as well. For studying the changes of grain-size distribution a continuously operated large laboratory or pilot plant scale equipment is recommended.

The effect of liquor contaminants, and the cristallization kinetics can be studied fairly well even in laboratory equipment.

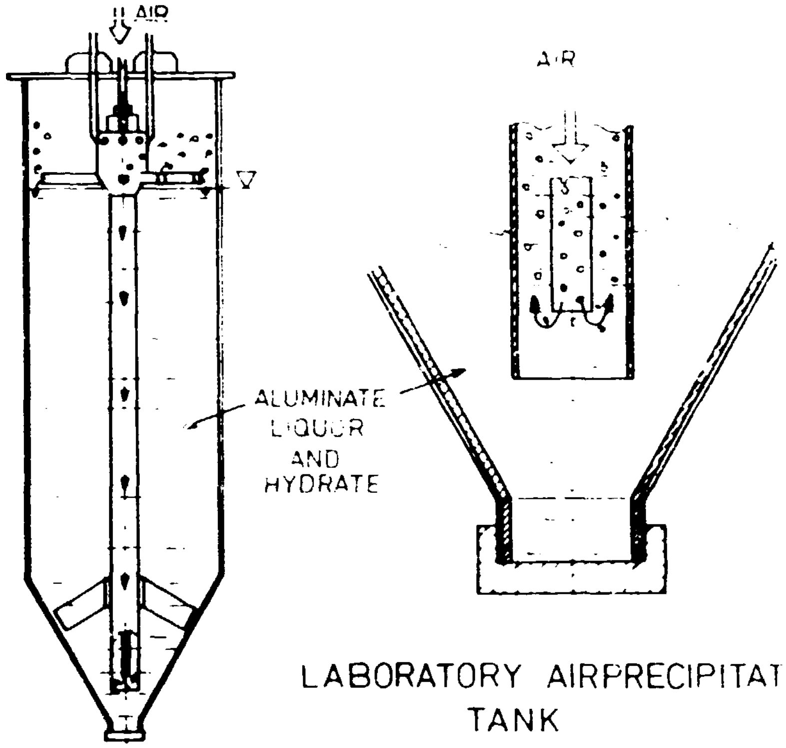
The precipitation experiments moved into focus recently partly due to the efforts invested into the conversion of European plants from floury to sandy alumina and partly due to the aim of increasing the liquor concentration at precipitation in the traditional sandy type plants in order to save up energy.

For carrying out precipitation tests various types of equipment with mechanical and air agitation are used.

One possibility is to use digester equipment with autoclave bombs (see Figs 20 and 21). A laboratory air precipitator tank is illustrated in Fig. 24. It is important to use CO_2-free air for agitation in this equipment and the seed must not be dried or aged by any means but only filtered before use.

The hydrate filtration is performed in small scale vacuum filters and data obtained on filtration period or capacity compare fairly characteristically for different hydrate types. The effect of various dewatering aids can be studied also here.

The physical and physico-chemical studies of hydrate and alumina are described in chapter 4.4.1.



LABORATORY AIRPRECIPITATOR TANK FIG.24

The calcination process and the effect of mineralizers can be studied in thermogravimetric instruments (see chapter 4.4.2.). A batch type laboratory furnace can not model the industrial calciner kilns, it may serve only to investigate the alumina obtained by precipitation tests.

Equipment

- pressure filter (for control filtration) (see Table 22)
- air agitated precipitator tanks (∿ 20 l) (see Table 20)
- mechanically agitated precipitators (1.0-3.0 l) (see Table 20)
- vacuum filter (for hydrate filtration) (see Table 22)
- furnace (heat control up to 1,400 °C) (see Table 19)

Staff Requirements

- 1 engineer
- 1 technician
- 1 assistant

4.3.4. Laboratory for Studying Effects of Bauxite Contaminants

Functions

- dissolving and accumulation tests
- analysis of the effect of contaminants in certain parts of the process
- elaboration of methods for recovery and removal

Principles

The technological evaluation of bauxite must be extended to the study of the contaminating components of bauxite.

These can be classified into three groups regarding their technological effects:

a) Valuable by-products to be recovered from the circuit, e.g. vanadate, gallium.

b) Contaminants trasferred to the alumina and directly affecting its quality, e.g. zinc, phosphorous.

c) Contaminants having detrimental effect on different phases of the process, e.g. calcite, dolomite, siderite, pyrite, organic compounds.

Dissolution and Accumulation Tests

If a certain contaminant is present in the bauxite in such a large amount, that technological measures have to be taken to remove it or to diminish its effects, the rate of dissolution of the contaminant should be measured.

Dissolution tests are performed in connection with the digestion tests at the suggested optimal digestion parameters. On the basis of dissolution tests the technological designer can take into consideration the required practical measures, e.g. designing of soda salt removal and regeneration, lime addition for P_2O_5 control, etc.

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

It seems to be a very difficult technical task to investigate the accumulation of contaminants because they influence each-other's solubility. The accumulation can not be studied in a usual bauxite evaluating laboratory equipped for batch system investigations only, but in pilot plants modelling the entire Bayer circuit. Useful experiences can be gained on contaminant accumulation during the starting up of new alumina plants.

Investigation of the effect of contaminants in certain stages of the process:

A particular bauxite might contain such kinds of contaminants that the operation of the process becomes difficult or, in certain cases, even impossible (e.g. high pyrite content spoils settling etc.). In such cases separate research should be devoted to the mechanism of the effect of the contaminant, to the determination of its acceptable maximum level and to the method of eliminating its detrimental influence, e.g. removal in advance or neutralization within the process.

Study of the possibilities for recovering or removing the contaminants:

The recovery of useful components accumulated in the circuit liquor and the removal of contaminants form separate parts of research. For certain contaminants commercial technologies had been worked out, like recovery of vanadate, soda salt removal, oxalate removal etc. These might be introduced to definite plant conditions after adaptation.

Although the removal of detrimental organic content appears to be one of the most widely explored problems there exists not a single well established process which could be applied generally in plants. Promising investigations are in progress.

A significant objective is the recovery of V_2O_5 and Ga_2O_3. In order to reduce the caustic soda losses it is very important to realize the causticization of soda within the circuit. The causticization of red mud plays a considerable role in the processing of bauxite with relatively high silica content. Both of the causticization procedures can be modelled fairly well in laboratory, using 500-1,000 ml volumes and different dosages of CaO.

Equipment

- no special equipment is necessary on laboratory scale
- over the above-mentioned standard laboratory equipment, however, a large scale close circuit laboratory or pilot plant is required for studying equilibria and the enrichment process, as well as for the recovery of V_2O_5 and Ga_2O_3.

Staff Requirements

- 1 experienced research worker
- 1-2 assistant(s)
- 1-2 technician(s)

4.4. UNIT FOR PHYSICAL AND PHYSICO-CHEMICAL MEASUREMENTS

The activities of this laboratory are divided into the following four groups: *

- simple physical and physico-chemical investigations
- thermal gravimetry
- infra red spectrometry
- texture investigations (optical and electron optical methods)

Some physical and physico-chemical investigations must be carried out in bauxite laboratories of all levels. Thermal gravimetry is usually used in parallel with X-ray diffraction analysis to determine the mineralogical composition. (Sometimes also IR spectrometry.)

Electron beam methods are not used in "A" or "B" level laboratories due to their high investment costs and the complicated operation technique requiring highly qualified and experienced personnel.

*(XRD belouging to the same tapic was handled already in Chapter 4.2.)

4.4.1. Laboratory for Simple Physical and Physico-Chemical Investigations

Functions

(1) Supplying physical and physico-chemical data during technological investigations

(2) Quality control of the raw materials, intermediate and final products (bauxite, $Al(OH)_3$, Al_2O_3, red mud) as a service for existing alumina plants

Principles

The parameters to be determined are listed in Table 23. The number and types of the applied procedures depend on the fact whether the laboratory is run for process control or research and on its level.

The knowledge of the density respectively volumetric weight of the solid phases occurring in bauxite processing is important in the following fields:

- bauxite: mining, storage, transport, alumina plant design
- red mud: designing of the equipment of the alumina plant, control of the alumina plant
- alumina hydrate and alumina: design, control of the production and the metallurgical utilization, grading alumina

The density of materials is the mass in the unit volume. Dimension: g/cm^3; kg/m^3.

Several methods may be used for the determination of density, however, the well-known pycnometric method is the most widespread.

Determination of the volumetric weight is very simple, too. The tested material is filled under fixed conditions into a container having unit volume and the material is weighed.

Information on grain-size distribution is applied mainly in the following fields:

- bauxite: grinding tests, determination of desintegration degree, control of crushing and grinding operations
- alumina hydrate: control of precipitation and alumina-hydrate classification

Table 23

PHYSICAL AND PHYSICO-CHEMICAL PARAMETERS USED FOR THE CHARACTERIZATION OF BAUXITE, RED MUD, ALUMINA HYDRATE AND ALUMINA

Material tested	Parameters used for the characterization of the materials
Bauxite	Density Volumetric weight Grain-size Specific surface area Porosity and distribution of pore volume Immersion heat
Red Mud	Density Grain-size Specific surface area Porosity Immersion heat
Alumina Hydrate	Grain-size Density Specific surface area Immersion heat
Alumina	Grain-size Density, appearent density Specific surface area Angle of repose Flourine-bounding capacity Dusting Rate of dissolution Heat transmission

- alumina: characterization of alumina, control of dust-exhausting equipment
- red mud: settling and washing of red mud

There exist direct and indirect methods for the measurement of grain-size distribution.

The equipment of sieve analysis are the screens of different apertures or sieve sets, where the apertures form a geometrical progression. The most common is the sieve set of quotient 2, based on geometrical considerations. The basis of the set is the so-called 200 mesh (wire intersections sieve/sg.in.) having 0.075 mm apertures, according to the USA Tyler, ASTM E 11-61 or English 410 standards.

The error of sieve analysis is generally 1 to 1.5 per cent.

Microscopy is the other direct method for the determination of grain-size distribution including tests performed by optical or electron microscopes. Evaluation can be carried out on the basis of direct observation, microphotographs or electronic counting.

Pipette methods (Köhn and Andreasen method) based on sedimentation principles, utilize the fact that only a small fraction of the sample is taken from a depth corresponding to the given length of settling path.

Turbidimetry is based on measuring the light-absorbtion ability of suspensions. The light-absorbtion ability depends on the number and size of grains floating in the suspension, that is, it is proportional to their projected surface area. Material characteristics have also importance, consequently, the equipment has to be calibrated for all types of new materials by sedimentation measurements which can be reduced to mass weighing.

In the course of the analysis absorbance (logarythm of intensity ratio, $A = \ln I_o/I$) is measured instead of the absorption itself. I_o is the intesity of light passing through the pure measuring medium, whereas I represents the same passing through the suspension.

Nowadays there are several modern instruments which analyse grain-size distribution automatically (using a small amount of sample and being fast due to time scanning method) and evaluate the collected data by means of built in computers. Data can be obtained either graphically or in digital form for direct transfer into a processing system. It is expedient for "B", "A" level laboratories to install such an instrument (e.g. Fritsch sedigraph).

The specific surface area is a very characteristic parameter for bauxite, red mud, alumina hydrate and alumina:

- Bauxite: the specific area of bauxite depends on its geological history and composition (such as the amount and quality of iron-bearing constituents). The specific surface area of the produced red mud will be similar to that of bauxite, therefore, measuring the specific surface area the bauxite information can be obtained for the settling behaviour and washability of the relevant red mud.
- Red mud: the specific surface area of red mud depends besides the quality of bauxite on the processing technology. The magnitude of the surface area of red mud determines its settling, washing and filtration behaviour.

- Alumina hydrate: the specific surface area of alumina hydrate is used to control the precipitation process (seed charge etc.)

- Alumina: measurement of the surface area provides control of the calcination process, grading of the produced alumina capability for dry cleaning of flourine containing gases).

The most accurate method for determination of specific surface area is the BET gas-absorption method.

The specific surface area is determined via the amount of gas absorbed in monomolecular layer.

Measuring the angle of repose of the alumina is important in relation to the flowing of alumina in the course of transportation while in the electrolyzing cells from the point of view of heat isolation.

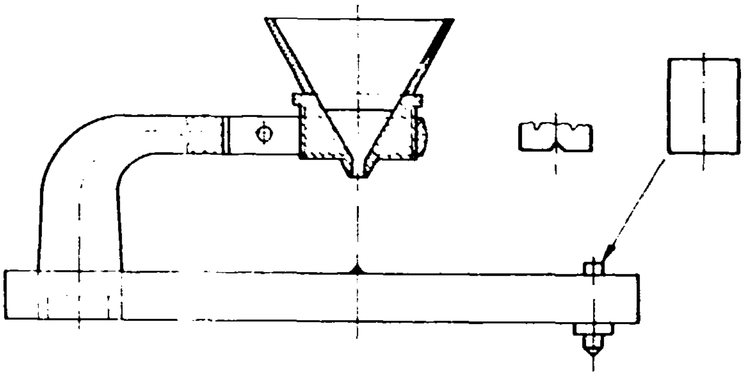
For the measurement of the angle of repose a specially developed measuring instrument is used (Fig. 25.).

The angle of repose expressed in terms of degrees is determined by the following formula:

$$R = \text{arc tg} \frac{2\,H}{D-d}$$

where: H - the height of the alumina cone, mm
D - distance in mm, diameter of the alumina cone
d - internal diameter of the orifice of the funnel, mm

The angle of repose of highly flowing alumina is less than 35^{o}, that of the flowing alumina is in the range of 35-40^{o}, while that of the sticky alumina larger than 40^{o}.



EQUIPMENT FOR DETERMINING THE ANGLE OF REPOSE FIG.25.

Measuring of the porosity and pore size distribution gives additional information for the characterization of the morphology of bauxite and red mud, respectively:

Bauxite: relying on the bauxite's pore size distribution, conclusion can be drawn on the pore size distribution of the corresponding mud.
- Red mud: the knowledge of porosity and pore size distribution of red mud can be utilized to determine the morphology of red mud and in its washability
- Alumina: it is important to know the porosity of special kinds of alumina (adsorbents, etc.)

Porosity; the ratio between pore volume (V_{pore}) and the total volume (V_{total}) of the porous material is called porosity

$$\varepsilon = \frac{V_{pore}}{V_{total}}$$

The porosity can be calculated with highest accuracy using volumes measured in helium and mercury. In this case V_{total} represents the volume measured in mercury, and V_{pore} is the difference of the volumes measured in mercury and helium.

The pore size distribution is determined by gas-absorption and by mercury porosimeters. This method is proposed only for research laboratories.

The immersion heat evolves when immersing the tested material (dust) into some liquid and it gives information about the solid/liquid surface interaction.

Immersion heat is measured in semi-micro- or microcalorimeters. The method is used meanly in research laboratories.

Fluorine-bounding capacity, dusting, rate of dissolution in cryolite and thermal conductivity of alumina are very important characteristics from the point of view of electrolysis.

These parameters are measured by special instruments mainly in laboratories dealing with problems of aluminium electrolysis.

Equipment

- analytical balance (see Table 19)
- 2 technical balances (see Table 19)
- pycnometers (see Table 24)
- Andreasen pipets (see Table 24)
- sieve set for wet sieving (see Table 16)
- light microscope
- air jet sieve (see Table 16)
- scanning photosedigraph (see Table 24)
- instrument for measuring the angle of repose (see Table 25)
- instrument for measuring specific surface area (see Table 26)

Table 24

INSTRUMENTS FOR GRAIN-SIZE DISTRIBUTION ANALYSIS AND SPECIAL INSTRUMENTS

Type	Producer Firm	Specifications	Price, $
Analysette 20 Scanning Photosedigraph with inbuilt calculator	Fritsch GmbH D-6580 Idar-Oberstein 1 FRG	Grain-size range: 2...100 µm (in water) or up to 500-600 µm (in higher viscosity measuring medium) cuvette volume: 200 ml Plots the extinction curve and the calculated grain-size distribution curve The results are convertable to sieve or any other analysis	14,000
Sedigraph particle size analyzer Model 5000 D	Micromeritics Instrument Corp. 5680 Goshen Springs Road Norcross, Georgia 30093 USA	Based on X-ray absorption principle, direct mass weighing. A microprocessor automatically plots the grain-size distribution cuvette volume: 5 ml (reason of unaccuracy!) grain-size range: 0.1...100 µm	29,000
Sedimentometer	Evans Electroselenium Ltd. Halstead, Essex, U.K.	Works on light absorption principle Cuvette volume: 10 ml (disadvantageable) grain-size range: 3...50 µm (in water) or 5...100 µm (in glycerine and water mixture) digital display	~ 3,000
HIAC PA-720	Pacific Scientific HIAC Instruments Division P.O.B. 3007 4719 West Brooks Street Montclair, CA 91763, USA	Measuring principle: light absorption Particle size range: 1-9,000 µm Carrier fluid: any fluid Automatic background correction, data processing, result is printed and/or plotted	40,000
Sedimentation balance 4610	Sartorius GmbH A-1020 Wien Alliirtenstraße 2	Grain-size range: 1...150 µm Sample weight: 0.3...1.5 g Cuvette volume: 550 ml, weight: 200 mm	5,500
Analysette 21 Pipetten centrifuge	Fritsch GmbH D-6580 Idar-Oberstein 1 FRG	Application if grains below 5 µm are more than 50 % Grain-size range: 0.05-5 µm (in water) Sample weight: min. 2 g/160 ml water	3,000
Commodore calculator	Fritsch GmbH D-6580 Idar-Oberstein 1, FRG	for automatic evaluation	5,000
Auto pycnometer Model 1320	Micromeritics Instrument Corp. 5680 Goshen Springs Road Norcross, Georgia, 30093, USA	max. sample volume: 50 cm^3 accuracy: ± 0.01 cm^3	10,500
Helium Pycnometer II. Model 1303	Micromeritics Instrument Corp. 5680 Goshen Springs Road Norcross, Georgia, 30093, USA	max. sample volume: 90 cm^3 accuracy: ± 0.1 cm^3	3,600
Zeta Potential Analyzer Model 1202	Micromeritics Instrument Corp. 5680 Goshen Springs Road Norcross, Georgia, 30093, USA	Range: 10 Ω - 10 MΩ Sample volume: 110-145 ml Solid content: max. 50 %	7,500
Rheomat 115	Seibold Messgerätefabrik Wipplingerstraße 24 A-1010 Wien, Austria	inbuilt thermostate: - 40 °C... + 100 °C automatic evaluation with microcomputer	25,000
Rheomat 30	Seibold Messgerätefabrik Wipplingerstraße 24 A-1010 Wien, Austria	external temperature control manual data registration and evaluation	.

Table 25

SPECIFIC SURFACE AREA MEASURING INSTRUMENTS

Type	Producer firm	Measuring principle, range	Price in US $
AREA-Meter	Ströhlein GmbH D 4000 Düsseldorf 1 Aderstr. 91-94. Postfach 7829	gasadsorption volumetric, static (N_2) 0.1-1,000 m^2/g	4,000 (Nov. 1980)
Sorptomatic 1800	Carlo-Erba P.O.Box 4342 20 100 Milano	gasadsorption volumetric, static (N_2) automatic 1-2,000 m^2/g	33,000 (Apr. 1980)
Surf.Area Analyzer 2205	Micromeritics Instrument Corp. 5680 Goshen Springs Road Norcross, Georgia 30 093 USA European distributor: SY-LAB GmbH Prager Gasse 11 A-3002 Purkersdorf, Austria	gasadsorption volumetric, static (Ar) (0.05-150 m^2/g	9,000 (Apr. 1981)
Accusorb 2100 E		gasadsorption volumetric, static over 0.001 m^2/g	29,000 (Apr. 1981)
Digisorb 2500		gasadsorption volumetric, static over 0.001 m^2/g	110,000 (Apr. 1981)
Monosorb	Quantachrome Corp. 101 Park Street, Montclair New Jersey, USA 07 042	gasadsorption volumetric, dynamic (N_2/He) over 0.1 m^2/g	8,000 (Nov. 1980)
Quantosorb	European distributor: Donau Electronic AG, Zürich Birmendorferstr. 483. P.f. 149 CH-8055 Zürich	gasadsorption volumetric, dynamic (N_2/He)	7,000 (Nov. 1980)

Table 26

PORE DISTRIBUTION MEASURING INSTRUMENTS

Type	Producer firm	Measuring range	Price in US $
Porosimetro Mod. 225	Carlo-Erba P.O.Box 4342 20100 Milano	7,500-3.7 nm	20,700
Macropore Unit Mod.120		600,000-1,875 nm	10,000 (Nov.1980)
Pore Sizer 9300	Micromeritics Instrument Corp. 5680 Goshen Springs Road Norcross, Georgia 30 093 USA	600,000-3.7 nm	20,100
Autopore	European distributor: SY-LAB GmbH Prager Gasse 11 A-3002 Purkersdorf, Austria	600,000-3.1 nm	51,000 (Jan.1981)
60 000 PSI Porosimeter	Aminco Corp. Silver Spring Maryland 20 910	300,000-9 nm	
15 000 PSI		600,000-3 nm	
Scanning porosimeter	Quantachrome Corp. 101 Park Street Montclair, New Jersey USA 07042 European distributor: Donau Electronic AG. Zürich Birmerndorfstr. 483. Postfach 149 CH-8055 Züch	600,000-3 nm	

4.4.2. Laboratory for Thermogravimetry

Functions

- contribution to complex phase analysis technique (complementary to X-ray and IR or can be used even alone)
- studying bauxite roasting (for removal of contaminants)
- studying alumina calcination process (especially the effect of mineralizers)

Principles

In the mineralogical investigation of bauxite and red mud thermal methods are used in order to gain complementary information to those obtained by other methods. Chemical and mineralogical composition of certain minerals change during heating. In thermal investigations the sample is placed in a controlled atmosphere and various parameters characterizing the physical-chemical state of the sample are measured or recorded as a function of the temperature varied according to a predetermined program. The most frequently used methods of thermal analysis are differential thermal analysis (DTA), thermogravimetry (TG) and differential thermogravimetry (DTG).

Difference thermal analysis (DTA) is a technique in which the temperature difference between a substance and a reference material is measured as a function of temperature whilst the substance and reference material are subjected to a controlled temperature program.

Thermogravimetry is a technique in which the mass of a substance is measured as a function of temperature. The DTG curve is obtained by electronic derivation of TG so that the changes of minerals can be followed more sensitively.

Table 27

DERIVATOGRAPHIC DETERMINATION OF THE MINERAL PHASES OCCURRING MOST FREQUENTLY IN BAUXITES AND RED MUDS

Mineral, Formula	Component lost	Peak temperature °C	Temperature range °C	Calculation formula
Gibbsite $Al_2O_3.3H_2O$	$3H_2O$ $2.75H_2O$ $0.25H_2O$	 320-340 540-560	 260-380 500-600	Al_2O_3 % = 101.94.G/49.555 Gibbsite % = 156.0.G/49.555
Boehmite $Al_2O_3.H_2O$ (Diaspore)	H_2O	540-560	440-600	Al_2O_3 % = 101.94.G/18.02 Boehmite % = 119.96.G/18.02
Kaolinite $Al_2O_3.2SiO_2.2H_2O$	$2H_2O$	590-610	560-700	Al_2O_3 % = 101.94.G/36.03 SiO_2 % = 120.12.G/36.04 Kaolinite % = 258.16.G/36.04
Goethite $Fe_2O_3.H_2O$	H_2O	360-380	340-420	Fe_2O_3 % = 159.70.G/18.02 Goethite % = 177.72.G/18.02
Calcite $CaCO_3$	CO_2	760-780	680-820	CaO % = 56.08.G/44.01 Calcite % = 100.09.G/44.01
Dolomite $CaMg(CO_3)_2$	$2CO_2$	800-820 960-980	520-920	CaOMgO % = 96.40.G/88.02 Dolomite % = 184.42.G/88.02
Sodalite $1.3Na_2O.Al_2O_3.1.7SiO_2$	$1.7H_2O$ $1.7H_2O$	-	100-400	Al_2O_3 % = 101.94.G/30.63 Na_2O % = 80.58.G/30.63 SiO_2 % = 102.10.G/30.63 Sodalite % = 315.25.G/30.63
Ca-Al-silicate-hydrate $2CaO.Al_2O_3.2SiO_2.4H_2O$	$4H_2O$	360-380	280-440	CaO % = 168.24.G/72.06 Al_2O_3 % = 101.94.G/72.08 SiO_2 % = 120.12.G/72.08 Ca-Al-sil.-hydr. % = 462.38.G/82.08
Ca-Al-silicate-hydrate $3CaO.Al_2O_3.2SiO_2.2H_2O$	$2H_2O$	360-380	280-440	CaO % = 168.24.G/36.04 Al_2O_3 % = 101.94.G/36.04 SiO_2 % = 120.12.G/36.04 Ca-Al-silicate-hydrate % = 426.34.G/36.04
Calcium hydroxide $Ca(OH)_2$	H_2O	520	470-550	CaO % = 50.08.G/18.02 $Ca(OH)_2$ % = 74.10.G/18.02
Calcium aluminate $3CaO.Al_2O_3.6H_2O$	$4H_2O$	350	280-420	CaO % = 168.24.G/72.08 Al_2O_3 % = 101.94.G/72.08 Calcium aluminate hydrate % = 378.30.G/72.08

G = measured weight change in the given temperature range.

For calculating boehmite the residual 0.25 mole water of gibbsite shall be subtracted from the weight change.

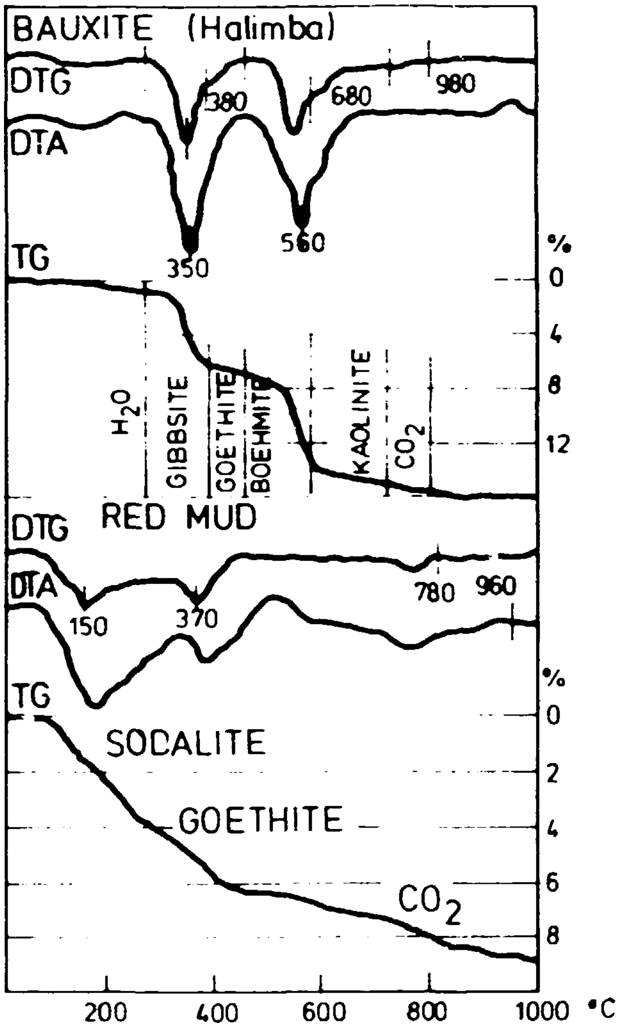
Table 28

EVALUATION OF DERIVATOGRAMS OF BAUXITE AND RED MUD FROM HALIMBA

(see Fig. 26)

Sample	Temperature range °C	Weight loss %	Mineral comp.	[illegible] comp.	Al_2O_3	Na_2O	SiO_2	Fe_2O_3	CaO	Ign. loss
Bauxite (Halimba)	270-360	5.2	Gibbsite	16.4	10.7	-	-	-	-	
	380-450	1.0	Goethite	9.9	-	-	-	8.9	-	
	450-570	5.54 (6.0-0.46)	Boehmite	37.	31.4	-	-	-	-	
	570-720	2.1	Kaolinite	15.1	6.0	-	7.0	-	-	
	720-800	0.8	Calcite	1.8	-	-	-	-	1.0	
			Derivatograph.		48.1	-	7.0	8.9	1.0	8.3
			Wet analysis		49.2	-	[illegible]	23.7	1.1	8.1
Red mud (Halimba)	100-320	4.5	Sodalite	46.3	15.0	11.8	15.0	-	-	
	320-460	1.9	Goethite	18.5	-	-	-	16.9	-	
	460-690	0.9	Boehmite (0.4)+Pyrite	2.4	2.0	-	-	-	-	
	690-820	0.9	Calcite	2.0	-	-	-	-	1.1	
			Derivatograph.		17.0	11.8	15.0	16.9	1.1	8.8
			Wet analysis		16.3	10.8	14.3	44.2	1.5	8.0

Boehmite was calculated by subtracting the 2.25 mole water of gibbsite from the weight loss. The difference between the amounts of Fe_2O_3 determined by wet analysis and Fe_2O_3 determined in form of goethite gives the hematite content of samples. Thus hematite content of bauxite is 23.7 - 8.9 = 14.8 %; hematite content of red mud is 44.2 - 16.9 = 27.3 %.



DERIVATOGRAM OF A HALIMBA BAUXITE

FIG. 26.

Table 23

THERMOANALYTICAL INSTRUMENTS

Type	Producer Firm	Specifications	Price, US $
DTA-1700	Perkin-Elmer Corp. Main Avenue - M.S. 12 Norwalk, CTO 6856 USA	Temperature range: 20-1,500 °C Heating rate up to 100 °C/per min. Automatic base line correction + DSC (Differential Scanning Calorimeter) MODE + microcomputer	~ 50,000
TGS-2	Perkin-Elmer Corp.	Sensitivity: 10 μg Temperature range: 20-1,000 °C Heating rate: 0.1-320 °C/min. Sample weight: 5 mg-5 g	~ 20,000
DSC-2	Perkin-Elmer Corp.	Range: 50-725 °C Heating rate: 0.3-320 °C/min. Sensitivity: 0.1 mcal	~ 25,000
TA Processor	Mettler Instrumente AG CH-8606 Greifensee, Switzerland	Accuracy 0.1 °C Microprocessor controlled	~ 40,000
Thermowage TG-50	Mettler Instrumente AG	Temperature range: 20-1,000 °C Heating rate: 0...100 °C/min. Weighing range: 0...150 mg Reproducibility: ± 1 mg	~ 10,000
DSC 20	Mettler Instrumente AG	Temperature range: - 20...+ 600 °C Heating range: 0...100 °C/min. Reproducibility: ± 0.2 °C	15,000
910 DSC	DuPont (U.K.) Ltd. 64 Wilbury Way, Hitchin, Herts England	Temperature range: - 130 °C to 725 °C Calorimetric sensitivity: 0.003 mcal/sec Calorimetric precision: ± 1 % Calorimetric accuracy: ± 0.2 %	~ 60,000
DTA	Perkin-Elmer Corp. Main Avenue - M.S 12 Norwalk, CTO 6856, USA	Temperature range: - 180 °C to 1,600 °C	
DuPont 990 DSC/TGA	DuPont Instruments Scientific and Process Division Wilmington, DE 19898 USA	TG sensitivity: 500 μg/250 mm Sample weight: 1...100 mg DTG sensitivity: 500 μg/min/250 mm Temperature range: 25...1,200 °C Purge gas: air/inert DSC sensitivity: 6 μg/cm Calorimetric precision: ± 1 % Heating rate: 0.5...100 °C/min. DSC temperature range: - 180 °C...+ 600 °C	50,000
LP-839	Chinoin, Budapest Exporteur: METRIMPEX Budapest HUNGARY	Temperature programmer Accessory to any furnace	5,000
Quasy 1500	MOM, Hungarian Optical Works 1525 Budapest, Pf. 52 HUNGARY	TG sensitivity: 20 mg/200 mm Sample weight: 10 mg...10 g Accuracy: ± 0.7 % Temperature range: 20...1,100 °C Purge gas: air/inert	25,000
Gas titrimeter	MOM, Hungarian Optical Works 1525 Budapest, Pf. 52 HUNGARY	Operating range: 0-14 pH -600...+1,800 mV Suitable for Cl^-, Br^-, SO_2, SO_3 CO_2, SeO_2, NH_3, CO, H_2O determination	10,000

The most frequently occurring bauxite and red mud phases which have a characteristic transformation caused by heat are given in Table 27.

The mineralogical composition of a bauxite or red mud sample can be calculated from the chemical composition and the thermogravimetric curve as it is illustrated by Fig. 26 and Table 28.

Staff Requirement

- 1 person (graduated chemist or physicist)

Equipment

A list of a few available thermoanalytical instrument is given in Table 29.

For further informations about application of thermoanalysis look up items [31...33] of references.

4.4.3. Laboratory for Infra Red Spectrometry

Functions

- information on mineralogical composition
- information on crystallinity and stability (while X-ray diffraction spectroscopy shows sensitivity towards the chemical bonds as well as towards the order of molecules)
- IR spectrometry in union with thermogravimetry can substitute XRD in an "A" level laboratory or supplement and refine the results of other methods in an advanced ("C" level) organization

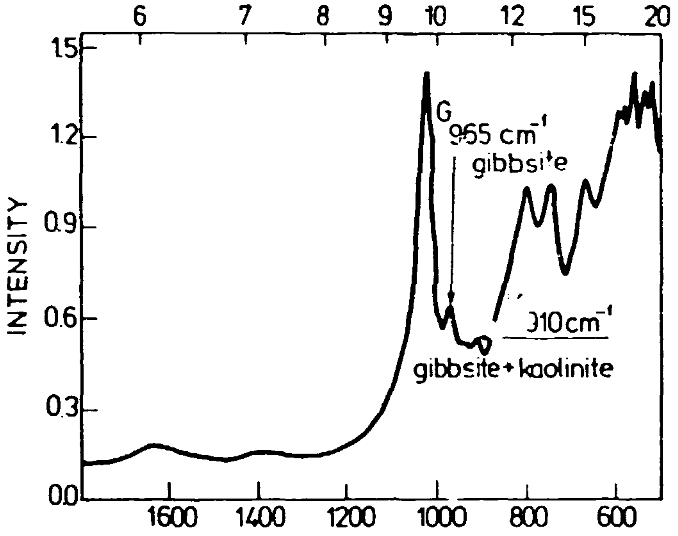
Principles

This method of instrumental physical-chemistry is based on the fact that the absorption of radiation takes place in solids at particular frequencies characteristic for chemical bonds in the material. In the 1 to 10,000 μm range of wavelength - infrared region - absorption is due to the change in vibrational or rotational states of molecules. Commercially available IR spectrometers operate in the more restricted wavelength range of 2-200 μm, however, the latest apparates exploiting the principle of Fourier transformation spectrometry, cover almost the entire spectrum of interest.

Important applications of IR spectrometry are the identification of minerals, their quantitative phase analysis in mixtures using the characteristic absorption bands, the determination of water content in materials as well as the study of bond strength (e.g. that of hydrogen bridges) by means of measuring precisely the shifts of certain IR bands. For the purposes of such studies usually laboratory made sets of samples and natural minerals are compared.

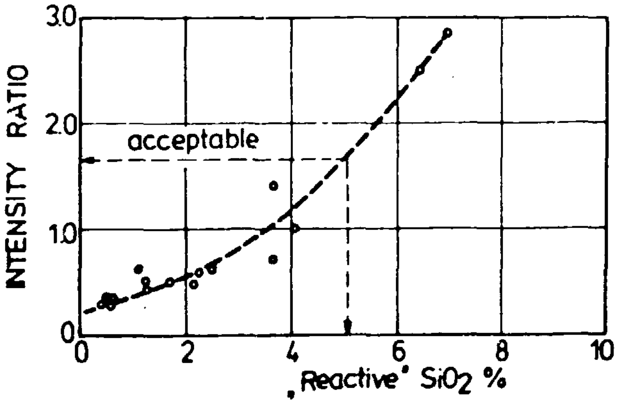
There is a fast method to determine the "reactive silica" content of lateritic bauxites developed by White [34].

Gibbsite has a characteristic Al-O-H band at 965 cm^{-1} and a common band with kaolinite at 910 cm^{-1} (see Fig. 27). There is a strict relationship between the ratio of 910 cm^{-1}/965 cm^{-1} band intensities and the reactive (kaolinite) SiO_2 content (Fig. 28). This fast and simpl analysis can be carried out at a geological laboratory as well and gives very important information about the technological value of the ore, by investigating even a very small amount of sample.



INFRARED SPECTRUM OF 1600 TO 600 cm^{-1} REGION OF LOW GRADE JAMAICAN BAUXITE.

FIG. 27



RELATIONSHIP BETWEEN „REACTIVE' SiO$_2$ % AND INTENSITY RATIO $K_{910 cm^{-1}}/G_{965^{-1}}$

FIG. 28

Table 30

INFRA RED SPECTROMETERS

Type	Producer firm	Specifications		Price in US$
		Range cm^{-1}	Resolution	
Acculab 1	Beckman Instruments	4,000-600	10 $cm^{-1}/3,000\ cm^{-1}$	4,860
Acculab 2	GmbH, Austria	4,000-600	5 $cm^{-1}/1,000\ cm^{-1}$	5,300
Acculab 3	A-1191 Wien	4,000-600	5 $cm^{-1}/3,000\ cm^{-1}$	6,400
Acculab 4	Stefan Esders Platz 4	4,000-600	3 $cm^{-1}/1,000\ cm^{-1}$	7,300
Acculab 5		4,000-375	5 $cm^{-1}/3,000\ cm^{-1}$	8,400
Acculab 6		4,000-250	3 $cm^{-1}/1,000\ cm^{-1}$	9,100
Model 598	Perkin-Elmer GmbH	4,000-200		
Linear wavenumber	Austria	microprocessor controlled automatic		19,400
grating spectro-	A-1101 Wien	double beam, flowchart recording		
photometer	Postfach 78	system, five digit display		(Feb.1981)
		4,000-400		17,000
Model 398	Perkin-Elmer	4,000-600		14,500
Model 298	- " -	computer, keyboard, floppydiscs		20,400
IR Data Station	- " -	visual display, DOS, compatable		
+ PECDS II.		to Models 298, 398, 598		(Feb.1981)
Microlab 600MX	Beckman	4,000-600	one floppy disc no compuset	30,000
Microlab 250MX	- " -	4,000-240	two floppy discs programming	32,000
Microlab 252MX	- " -	4,000-250	two floppy discs memory stat.	39,700
Microlab 620MX	- " -	4,000-600	- " -	37,200
Microlab 252 CMC	- " -	4,000-250	- " -	46,900
Microlab 620 CMC	- " -	4,000-600	- " -	44,900
Optimal video display	- " -			10,300 (Jan.1981)

Equipment

- for field work; gibbsite, boehmite, kaolinite content can be determined after calibration using portable instruments

- in "A" and "B" level laboratories for rapid and reliable mineralogical analysis an automatic instrument can be recommended

- for "C" level laboratories fully computerized system with data processing could be considered.

4.4.4. Texture Investigations

Functions

- determination of element distribution (optical microscope, EPMA)
- determination of particle size and shape for enrichments analysis (SEM, TEM)
- qualification of alumina hydrates (SEM)

Principles

The size, shape and mutual arrangement of the mineral grains and various particles forming any rock is referred to as its texture. We mean by textural investigations all those studies which yield information and accurate data about the distribution of chemical elements and mineral phases as well as about the size, form and distribution of pores, flawes, etc. within the ore.

For the purposes of textural studies by means of a light microscope microsections have to be prepared. These are polished almost plan parallel slices of some 0.1 mm thickness. Fairly hard bulk material is necessary that one should be able to cut rather thin slices, glue them on a glass substrate and than polish to the required final thickness.

Depending on the particles to be discerned microphotographs are taken with magnifications between 2x and 1,000x.

Part of the challenge in many technological problems arises from the way in which some small, microscopic particles control the macroscopic development of the whole process. In mineralogy often complex shapes have to be studied to resolutions of the order of 1 µm with a large depth of focus. In this range electron optical instruments are playing a highly significant role. Here we will deal with the applications of three major methods: X-ray microanalysis, scanning electron microscopy and transmission electron microscopy.

In the majority of bauxitic ores the individual mineral particles are too small to be studied by any instruments but transmission electron microscopes (TEM). As a result of a large number of observations it can be summarized that kaolinite usually forms platy crystals, goethite appears needle like, gibbsite and hematite tend to be more isometric and some minerals crystallize in, or are crushed to, irregular forms. These experiences form the basis of TEM identification of mineral components and the determination of their particles sizes and the form of their aggregation. More definite but also laboursome way of identification is to prepare and evaluate electron diffraction photographs. Energy dispersive X-ray analysis (EDAX) can be applied also in TEM to obtain chemical data on particles as small as some times 10 nm in diameter. It is clear that TEM techniques yield the most detailed information about texture with the highest resolution. The shape and morphology of the particles - as revealed by electron microscopy - vary to a great extent depending mineralogical composition and origin of the sample.

The evaluation of texture data from TEM microphotographs can be accelerated a great deal if up-to-date image analysis methods and apparates are applied. In this case the particle size distribution is calculated and statistically interpreted by a computer which collects data via an optical digitizer system sensing the co-ordinates of the perimeters of each particle.

Scanning electron microscopes (SEM) are used in mineralogic studies usually with six different detectors producing the following images:

Image	Information
1. Secondary electron	Geometrical relief (morphology)
2. Backscattered electron	Average atomic number Geometrical relief
3. Absorbed electron	Average atomic number
4. Transmitted electron	Internal structure
5. Cathodoluminescence	Distribution and concentration of fluorescent materials
6. X-ray	Distribution and concentration of chemical elements in the Na-U range

From the "Information" column of this table it can be seen that not all images give unique data, therefore, the use of secondary electron images + backscattered electron images + energy dispersive X-ray analysis seems to be a basic requirement in SEM studies and the other signals can be used additionally as the particular problem renders it indispensable. For bauxite the above combination is normally sufficient.

Scanning electron microscopes yield magnifications between about 30x and 150,000x and the resolving power is approximately 6-7 nm. By this virtue they partly bridge the gap between the optical area of light microscopes and that of transmission electronmicroscopes, and partly provide characteristic advantages as compared to these two rivals.

Fig. 29 (SEM image No. 8342) shows alumina hydrate agglomeration in magnification of 1,000x consisting of large gibbsite crystals bearing on their surface small secondary ones.

Fig. 30 (SEM image No. 2551) illustrates alumina at a magnification of 4750x, calcined in presence of mineralizer.

Electron Probe Microanalysis (EPMA) reveals the textural characteristics of bauxite samples in a sense that the special distribution of the constituting chemical elements can be detected on a micro-scale.

The samples for EPMA investigation must have a polished surface of optical quality.

The main parts of a microanalyser are:

- the electron optical column with vacuum system
- the spectrometers and divers detectors
- electronic operation and control system including power supplies
- computer controlled measuring and display units.

The electron probe at the analysing position can be observed by means of its catodoluminescent image through an optical microscope provided also with illuminator for visual ob-

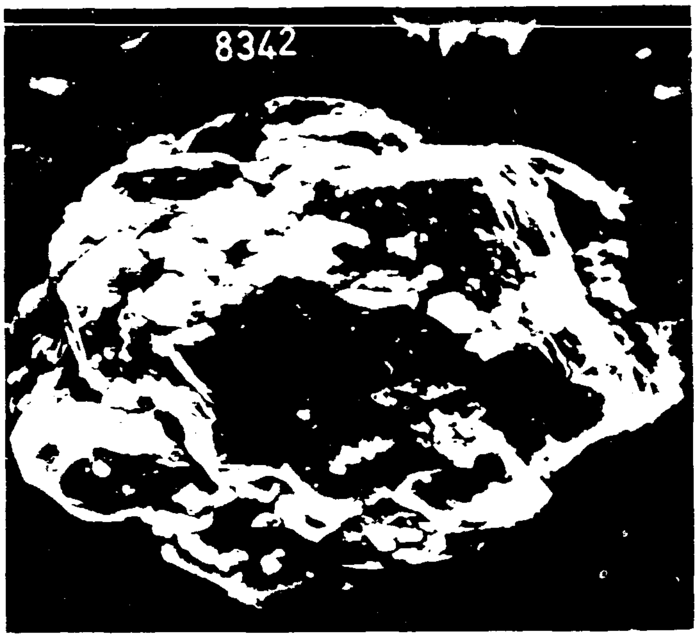


Fig. 29
SEM image of an Alumina Hydrate Sample

Fig. 30
SEM image of an Alumina Sample

servation and microphotography. The magnification of the optical device goes typically up to 400x, the resolving power should be better than 1 µm. Accelerating voltage and beam current being fully stabilized prolonged measurements can be carried out effectively. The probe diameter can be reduced so far that the volume analysed by X-ray spectrometry becomes restricted to about 2-5 μm^3.

Up-to-date microanalysers incorporate several vacuum spectrometers so that three or four elements can be analysed simultaneously. Each spectrometer is equipped with several analyser crystals selected according to the chemical elements to be measured and with detectors, like proportional or scintillation counters. The spectrometer may scan a definite range of X-ray wavelength or be set to a particular wavelength. In the first case one can identify the elements present in a microvolume, in the second the particular distribution of an element can be surveyed.

The spectrometers detect currently any elements between ^{5}B and ^{92}U.

(1) the counts representing X-ray intensity can be read from scalers, digitally printed or recorded on punched tape;
(2) strip chart recorders permit analogue representation;
(3) cathode ray tubes (CRT) are available to display the signals coming from each spectrometer;
(4) recently the operation of microanalysers is controlled by microcomputers or microprocessors which also collect the measured data and if required process or store them. Data storage occurs usually on magnetic floppy discs.

EPMA has been applied extensively in the textural investigations of bauxite.

An EPMA laboratory is shown in Fig. 31. Instruments are listed in Table 31.

Staff Requirements

- 2 graduated physicists or chemists and 2 technicians for SEM & EDAX
- 2 graduated members and 1 technicians for EPMA and TEM

For further information about electron microscopy look up [3/4], [3/5], [35]...[38].

Fig. 31
Electron Probe Microanalyzer (EPMA)

Table 31

ELECTRON BEAM DEVICES

Type	Producer Firm	Specifications	Price in US $ *
TRANSMISSION ELECTRON MICROSCOPES			
JEOL 100 CX	JEOL Ltd. Tokyo New Tokyo Building 3-3-1 Marnnouchi, Chiyoda - Ku TOKYO 100 JAPAN	Accelerating voltage: up to 100 kV Lattice resolution: 0.22 nm	150,000
JEOL 200 CX	JEOL Ltd. Tokyo New Tokyo Building 3-3-1 Marnnouchi, Chiyoda - Ku TOKYO 100 JAPAN	Accelerating voltage: up to 200 kV Lattice resolution: 0.14 nm + scanning equipment	from 350,000 to 400,000
Philips EM 400	N.V. Philips' Gloeilampenfabricken Eindhoven Netherlands	Accelerating voltage: up to 120 kV Lattice resolution: 0.14 nm + scanning equipment	from 300,000 to 400,000
SCANNING ELECTRON MICROSCOPES /SEM/			
JEOL 35 C	JEOL Ltd. Tokyo New Tokyo Building 3-3-1 Marnnouchi, Chiyoda - Ku TOKYO 100 JAPAN	Accelerating voltage: 25–30 kV Lattice resolution: 6 nm	~160,000
ISI 40	International Scientific Instruments Inc. 3 255-6C Scott Blvd Santa Clara, CA 95050, USA	Accelerating voltage: 25–30 kV Lattice resolution: 6 nm	~ 70,000
ISA-DS-130	International Scientific Instruments Inc. 3 255-6C Scott Blvd Santa Clara, CA 95050, USA	Accelerating voltage: 100 kV Lattice resolution: 3 nm	~ 140,000
SEM-505	N.V. Philips' Gloeilampenfabricken Eindhoven/Netherlands	Accelerating voltage: 1 to 30 kV in four ranges Lattice resolution: 6 nm + multi video control + detector control + EDAX + Split (Data) Rotation	~ 160,000
WAVELENGTH AND ENERGY-DISPERSIVE MICROANALYZERS /EPMA/			
Scanning Electron Microprobe Quantometer	Applied Research Laboratories 9545 Wentworth Street, Sunland California 91040, U.S.A.	3-5 crystal spectrometers	~ 350,000
CAMEBAX MICRO	CAMECA 103, Boulevard Saint Denis 92403 Courbevoie Cedex France	crystal detector, computer controlled	~ 300,000
CAMSCAN	Cambridge Instruments Wiking Way, Bor Hill Cambridge, CB 38 EL England		~ 250,000
Superprobe 733	JEOL Ltd., Tokyo New Tokyo Building 3-1-1 Marnnouchi, Chiyoda-Ku TOKYO, 100, JAPAN		~ 300,000
Accessories to SEM or EPMA: ENERGY DISPERSIVE DETECTORS /EDAX/			
EDAX-711	EDAX P.O.B. 135. Praise View Ill. 60069 U.S.A.	Energy resolution: 150 to 160 eV Detection limit: 0.1 % Elemental range: ^{11}Na to ^{92}U with ECON: from ^{6}C	30,000
ETEC	ETEC Corp. 3392 Investment Boulevard Hayward, California 94545	N. B.: cooled by liquid N_2!	30,000
	LINK SYSTEMS 32-34 Abbey Barn Road High Wycombe, Bucks, England		30,000

* Prices are highly dependent on the selection of accessories

5. ACTIVITIES OF BAUXITE LABORATORIES

In the previous chapter the functions and activities to be carried out by bauxite laboratories were grouped according to different types of work and special methodologies. In this chapter these activities and functions are summarized in terms of the typical levels of laboratories and simultaneously the main equipment required with estimated costs and human resources are given. These information are compiled in Table 32.

It should be noted that estimated costs refer only to the main equipment but these make up the substantial part (70-80 %) of the total equipment costs. Sometimes alternatives of different level and completion are given to leave for the users discretion the choice of the appropriate one. According to our opinion outlined above there are mutual overlappings among the three levels and different activities characterizing a highly developed unit appear at "A" level also, however, the applied procedures are here usually simplified and carried out by more simple (common) equipment.

When determining staff requirements there are over-estimates due to equipment oriented considerations. Consequently, if regarding the laboratory as an integrated entity highly qualified specialist partially can be replaced by technicians and the latters by skilled workers. Our system supposes highly qualified specialist having ample industrial practice.

Table 32

ACTIVITIES OF BAUXITE TESTING LABORATORIES

Level	Type of laboratory	Activities	Description of the main [illegible]	Estimated costs,US$	Personal requirement
A	1. Analytical Chemical Laboratories				
	1.1 Sample preparation and storage	sampling, homogenization, reduction of size, coding determination of moisture and LOI	turbo mill	10,000	2 persons (2,000 samples/year/
			air jet sieve	15,000	
			laboratory disc mill	3,000	
			electric oven	1,000	
			moistomat	10,000	
			analytical balance	1,000	
			interface	2,000	
				42,000	
	1.2 Analytical Chemical Laboratories	determination of main components and some trace elements of solid phases, chemical analysis of aluminate solutions			
A	AAS-FS		sequential fusion equipment	10,000	1 chemist 3 technicians 2,000 bauxite and/or red mud samples/year
			PTFE lined steel bombs for dissolution of alumina	3,000	
			AAS equipment with accessories	50,000	
				63,000	
A	Wet chemical laboratory		fusion equipment	10,000	1 chemist 3 technicians 1 unskilled worker
			glassware	10,000	
				20,000	
	1.3 Other instrumental methods	supplying the routine detection work required for the wet chemical laboratory and performing special investigations for some simple technological operations (determination of the total available alumina - TAA - analysis of aluminate solutions)			
A	Spectrophotometry		spectrophotometer	5,000	1 chemist 3-4 technicians
A	Electroanalytical techniques		precision pH-meter	1,000	
			automatic potentiometric titrator	5,000	
			oscimhometer	1,000	
A	Thermometric concentration measurements		thermometric titrator with calculator and plotter	6,000	
				18,000	

Table 32 (cont.)

Level	Type of laboratory	Activities	Description of the main equipment	Estimated costs,US$	Personal requirement
A	1.4 Laboratory for neutron activation methods	rapid analysis of Al_2O_3, SiO_2, Fe_2O_3 needed for bauxite explorations and mining	automatic bauxite analyser Type BEA	26,000	2 skilled workers
(A) B	1.5 Data processing for analytical chemical units	handling, reducing and storing of analytical data	mini computer (CPU capacity 32-48 Kbyte, peripherical devices)	10,000	after special training qualified technical staff of the laboratory
(A) B	1.6 X-ray fluorescence analysis	determination of the main components and contaminants of solid phases	sample preparation laboratory for XRF	32,000	2 scientists 2-3 technicians 20,000 samples/year
			generator + sequential spectrometer	110,000	
			generator + simultaneous spectrometer with 20 channels under computer control	350,000	
			close loop water cooling	12,000	
				540,000	
	2. Laboratories for qualitative and quantitative phase analysis				
A	2.1 Thermal gravimetry	determination of gibbsite, boehmite, diaspore as well as kaolinite, carbonates, sulphates in bauxite	thermobalances with simultaneous recording of TG and DTG curves preferably with titration of released gases	40,000	1 chemist or technician 1 (or 2) sample(s)/day
(A) B	2.2 Infra red spectrometry	semiquantitative determination of minerals (e.g. gibbsite, boehmite, kaolinite) characterization of certain minerals (preferably in co-operation with thermogravimetry)	portable simple (for A)	5,000	1 graduated 1 technicians
			standard (B level)	30,000	
C			computerized (Fournier transf.) connected with gaschromatograph (C level)	300,000	
(A) B	2.3 X-ray diffraction	complete phase analysis of bauxite and red mud	generator + diffractometer assembly	80,000	2 scientists 2-3 technicians 1,000-5,000 samples/year
			generator + computer controlled diffractometer	160,000	

Table 32 (cont.)

Level	Type of laboratory	Activities	Description of the main equipment	Estimated costs, US$	Personal requirement
(A) B	3. Physical and physicochemical laboratories				
	3.1 Laboratory for determination of basic physical parameters	determination of density, volumetric density, grain-size distribution, specific surface area, porosity and pore size distribution in bauxite, red mud ("A" level) and $Al(OH)_3$, Al_2O_3 ("B" level)	sieve series (dry and wet), porosimeter BET equipment (for determination of specific surface area), sedigraph (light absorption measuring with time scanning for grain-size distribution analysis)	2,000 18,000	2 technicians
(A), (B), C	3.2 Laboratory for texture analysis	studies of bauxite texture by means of polarized light microscope ("A" level)	light microscope	2,000	1 technician
		determination of elementary distribution by electron probe microanalyzer (EPMA) in bauxite red mud, alumina hydrate ("C" level)	electron probe microanalyzer (EPMA)	250,000 - 350,000	1 graduated scientist and 2 technicians associated to each
		particle shape and size analysis (for alumina and hydrate), TEM, SEM with EDAX	transmission electron-microscope (TEM) scanning electron-microscope (SEM)	150,000 - 400,000 60,000 - 160,000	electron-microscope

Table 32 (cont.)

Level	Type of laboratory	Activities	Description of the main equipment	Estimated costs, US$	Personal requirement
B	4. Laboratory for technological operations	simulation of the operations of the Bayer process: Ore dressing, grinding, preliminary desilication, digestion, red mud separation, washing and causticization decomposition, calcining	crushers mills for wet and dry grinding dry/wet sieving series sample devider vibration mill drying oven autoclaves digester equipment with 6-10 bombs (Hungalu) centrifuges pressure filter vacuum filter settling tubes soft γ-ray model settler (Hungalu) air agitated precipitator tanks mechanically agitated precipitators	20,000 40,000 6,000 3,000 15,000 4,000 20,000 20,000 6,000 5,000 5,000 1,000 60,000 5,000 10,000 220,000	2 technicians 3-3 skilled workers 1-2 engineers 2 technicians 2 skilled workers
(B) C	5. Laboratory for studying bauxite contaminants	dissolution and accumulation tests, analysis of the effect of contaminants in certain parts of the process elaboration of methods for recovery and removal	no special equipment is required on laboratory scale, however, a close circuit large scale laboratory or pilot plant is required for studying equilibria and enrichment processes and for recovery of V_2O_5 and Ga_2O_3		
C	6. Large scale laboratory or pilot plant facility	technological and apparative modelling of Bayer process	depends on existing plant and aims whether the entire circuit or certain stages are to be modelled	cost estimation is possible if given tasks and conditions are known. A large scale laboratory costs 50-100,000 $, pilot plant about 10 times of that.	

6. REQUIREMENTS FOR EQUIPMENT, BUILDING AND HUMAN RESOURCES

The principles of setting up a bauxite testing laboratory were discussed in chapter 3., the main units of these laboratories were analysed in chapter 4., the activities, equipment and human resources were projected to the laboratories of different levels in chapter 5. This chapter tries to give examples for the layout of bauxite testing laboratories at different completion levels.

In chapter 3.3. a characteristic "A" level laboratory is shown in Fig. 3.

An example for a "B" level laboratory, where tasks of process control in an alumina plant and also research and development tasks were predicted to be carried out, is shown in Fig. 33. A legend (i.e. the list of laboratory rooms allotted to different functions) to the figure is in Table 33.

It should be mentioned here that a laboratory of any level requires basic infrastructure: water supply, electricity, canalization, gas supply, likely compressed air, vacuum and hot water network. If the first three are missing no laboratory can be operated. The letter four can be substituted somehow by special facilities.

A "C" level laboratory can't be described so generally because it depends to much greater extent on the technical and economical background of the country. As a rule "C" level laboratories work in a co-operation with other research laboratories and educational institutions.

6.1. AN EXAMPLE FOR AN "A" LEVEL LABORATORY (Fig. 3)

Activities of an "A" level bauxite testing laboratory:

- Physical investigations:
    - Volumetric weight
        - wet bauxite
        - dry bauxite
    - Moisture of bauxite (%)

- Fast determination of main chemical components of bauxite by neutron activation method (Al_2O_3, SiO_2)

- Chemical analysis of bauxite (main components, Al_2O_3, SiO_2, Fe_2O_3, TiO_2, LOI, and contaminants, CaO, MgO, CO_2, P_2O_5..., and some rare elements, V_2O_5, Ga_2O_3)

- Chemical analysis of carbonate and silicate clays (14 components: CaO, MgO, Fe_2O_3, FeO, TiO_2, Al_2O_3, SiO_2, CO_2, SO_4^{--}, SO_3^{--}, P_2O_5, V_2O_5, Cl, F^-)

- Chemical analysis of trace elements from bauxite, carbonate and silicate clays

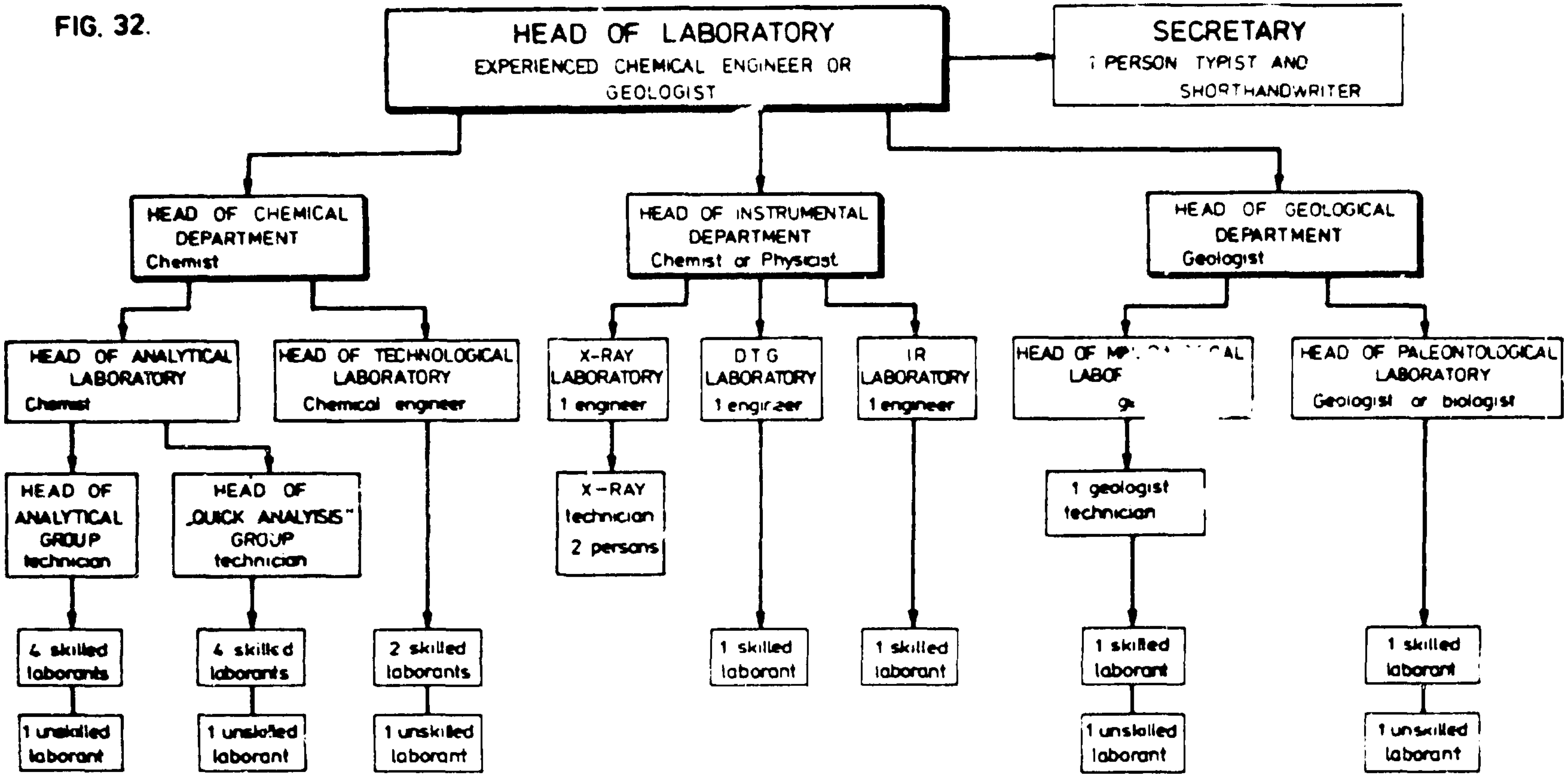
- Mineralogical analysis (thermogravimetry, X-ray diffractometry, infra red spectrometry)

- Geological investigations:
    - Mineralogical analysis of clays (micromorphological analysis, determination of residual after extraction, microscopy investigations, calcimetric investigations, grain-size distribution analysis)
    - Paleontalogical investigations (nannoplancton analysis foraminifera analysis, macrofaunal investigations)

An example for organizing a bauxite testing laboratory group is given in Fig. 32.

ORGANIZATION SCHEME FOR A BAUXITE TESTING LABORATORY (WITH 36 PERSONS)

FIG. 32.



CHEMICAL DEPARTMENT	
3	Graduated chemists or chemical engineers
2	Chemical technicians
10	Skilled chemical laborants
3	Unskilled laborants
18	Persons

INSTRUMENTAL DEPARTMENT	
4	Graduated chemists or physicists (or engineers)
2	X-ray technicians
2	Specially skilled laborants
8	Persons

GEOLOGICAL DEPARTMENT	
3	Graduated geologists
1	Geologist technician
2	Specially skilled laborants
2	Unskilled laborants
8	Persons

6.2. AN EXAMPLE FOR A "B" LEVEL LABORATORY (Fig. 33)

6.2.1. Scope of Capabilities and Activities of the Laboratory

In order to have a comprehensive knowledge on the state of the technological circuit at any time, continuous and regular checking of various parameters is necessary.

The most important parameters are continuously measured, recorded and sometimes automatically controlled. However, a considerable part of the technological parameters and information, essential for adequate operation of the process, can be checked only by taking samples at regular intervals and analysing them. This is one of the main functions of the laboratory.

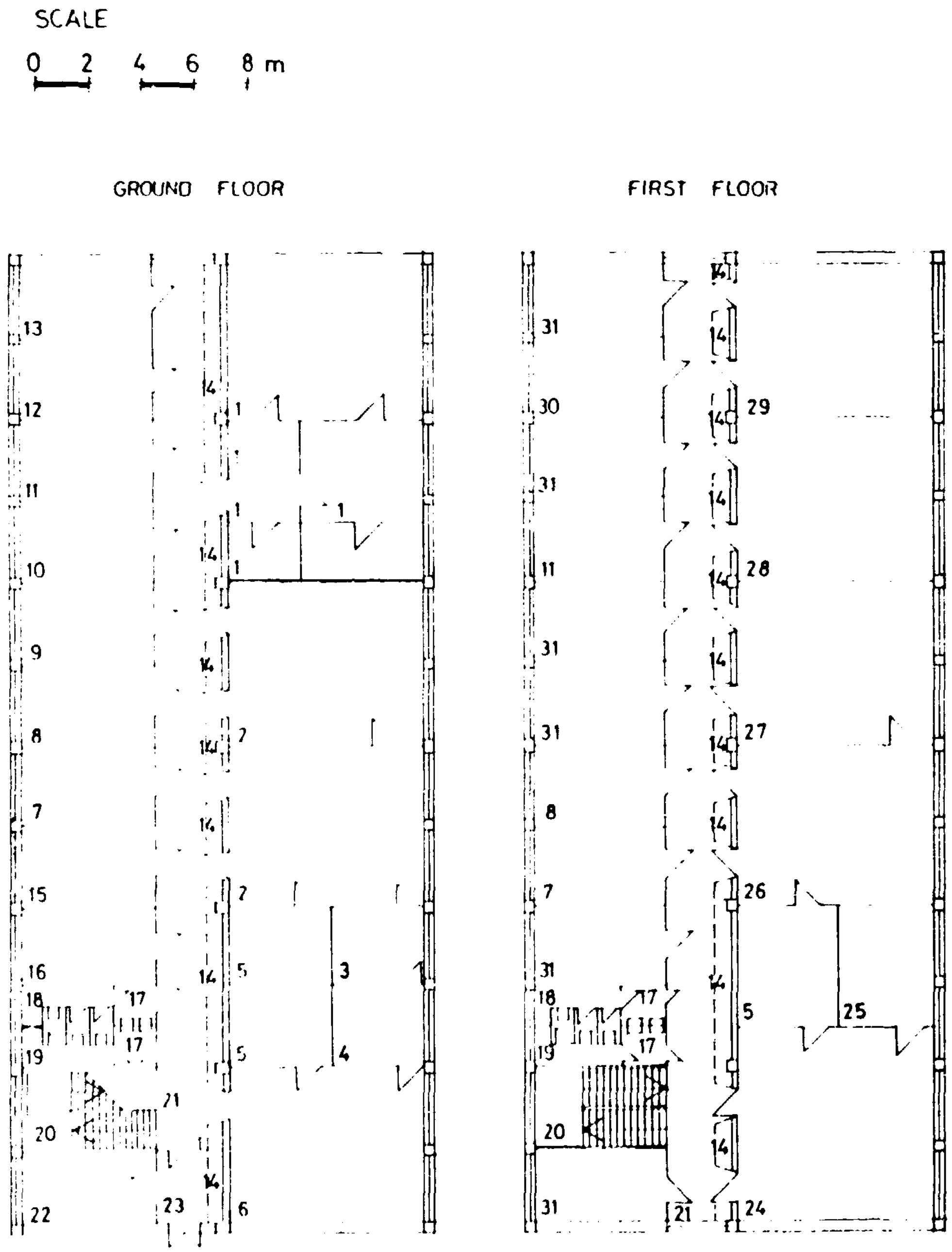
There are several plant liquors, the analysis of which is required at short intervals, in every two or four hours, or in every shift. To meet this requirement, a section is provided in the laboratory where only these analyses are performed as around the clock operation. The other sections of the laboratory operate only during the day shift.

Another important function of the laboratory is the regular quality control of raw and auxiliary materials as well as the product quality control.

A separate section is the research laboratory. Here the possible operating porblems are dealt with. Moreover process research and development activities are practised.

6.2.2. Layout of the Laboratory

The proposed laboratory layout is a well-proven system, based on the number and frequency of various analyses to be performed.



LAYOUT OF „B" LEVEL LABORATORY

FIG 33.

Table 33

LEGEND TO FIGURE 33.

1. X-ray laboratory (a 1:50 layout is given in Fig.13)
2. Wet chemical laboratory (a 1:50 layout is given in Fig. 9) (and shift laboratory)
3. Flame-photometer room (Fig. 6)
4. Atomic absorption spectrometer room (Fig. 6)
5. Room for acid hoods, drying ovens (Fig. 6)
6. Room for bauxite and red mud sample preparation for analysis
7. Balance room
8. Storage of chemicals
9. Storage of acids
10. Room for water distillation
11. Storage of control samples
12. Storage of glass ware
13. Room for vacuum pumps
14. Utilities and cupboards for storage of various materials
15. Changing room for ladies
16. Changing room for gents
17. Lavatory
18. WC for ladies
19. WC for gents
20. Staircase
21. Passage
22. Switch room
23. Entrance way
24. Hydrate and alumina analysis
25. Room for instruments
26. Preparatory room for technological investigations
27. Room for technological investigations (see Fig. 16 1:50 layout)
28. Room for tests of oil and lubricants
29. Research laboratory
30. Library
31. Office

The total area requirement is provided in two storeys. In addition to the laboratory working rooms, provision is made for storage of acids, chemicals and glass-ware, as well as for offices, changing rooms and other facilities. The design of the floor in each room is appropriate to its function.

The layout of the laboratory is shown in Fig. 33.

6.2.3. Utilities

Provision is made for utilities which are necessary for the operation of the laboratory. Power, water and compressed air will be supplied form the plant network. Vacuum pumps will be installed to meet the requirements. Gas will be supplied from cylinders.

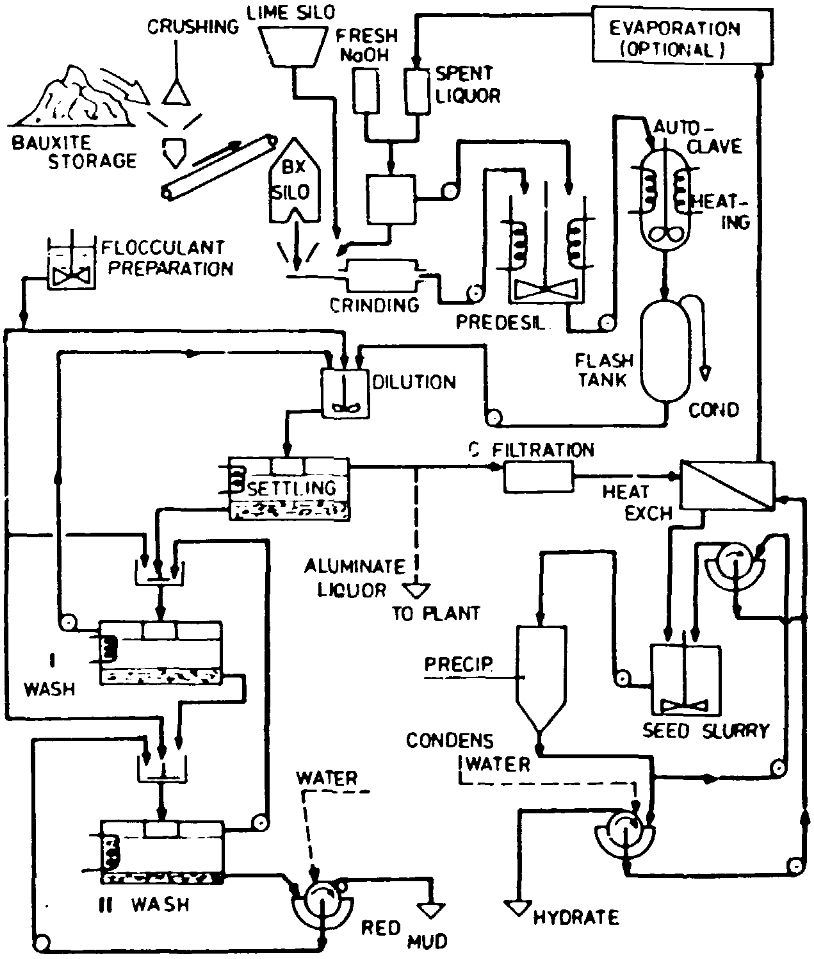
Acid hoods are located in separate rooms and an effective air-suction installed on each hood for extracting gases and vapours.

The various waste solutions will be collected and after neutralization transferred to the red mud disposal.

6.3. EXAMPLE FOR SETTING UP A LARGE SCALE LABORATORY, PILOT PLANT

Concerning to chapter 2.3. setting up a large scale or pilot plant laboratory is advisable, first of all to study the steps connected to the quality of bauxite. Although a large scale laboratory or a pilot plant is to be planned individually, it is aimed to purchase those equipment which are available commercially (see Table 34).

A conceptual flow sheet of such large scale laboratories is given in Table 34. Unfortunately some equipment the whole laboratory or pilot plant is to be designed and constructed individually by experienced specialists.



FLOW SHEET OF A LARGE SCALE LABORATORY.
FIG. 34.

Table 34

COMMERCIALLY AVAILABLE EQUIPMENT FOR LARGE SCALE LABORATORIES

Type	Producer Firm	Specifications	Price, $
MN 931/1 Jaw crusher	KHD Ind. AG Humboldt Wedag D-463 Bochum P.O.B. 2730 FRG	Feed opening: 200x125 mm Capacity: 3.00 ... 1,500 kg/h Product size: 8...20 mm Push-plate: Mn-steel	8,000
FE-02 Jaw crusher	Apritógépgyár, Jászberény Exporteur: NIKEX Budapest, I. Mészáros u. HUNGARY	Feed opening: 250x150 mm Capacity: 2,000 ... 4,000 kg/h Product size: 5...30 mm Push plate: Mn-steel	4,500
VA-60	Dorogi Szénbányák, NIKEX	Vibration feeding cell	500
10"x6" Laboratory Crushing roll	Denver Equipment Co. 1400 Seventeenth St. Denver 17, Colo, USA	Diameter: 250 mm, width: 150 mm Max. feed size: 10 mm, product size: 0.25 mm Capacity: 2,000 kg/h Operating speed: 250...300 rpm	10,000
HS 30/25 Drum cursher	Apritógépgyár, Jászberény NIKEX, HUNGARY	Diameter: 300 mm, width: 250 mm Max. feed size: 20 mm, product size: 0.1 ... 10 mm Capacity: 5,000 ... 6,000 kg/h	7,000
ACM-25	Apritógépgyár, Jászberény	Rotary cell feeding device	500
MN 940/1 Ball mill	KHD Ind. AG Humboldt Wedag	Diameter: 600 mm, length: 800 mm Screen classifyer, diameter: 200 mm Capacity: 30...100 kg/h Feed size: 15 mm Product size: 40...300 µm Operating speed: 43 rpm Continuously operatable	60,000
Laboratory ball/rod mill 10"x16" or 16"x32" and Spiral labor. Classifyer and pump	Denver Equipment Co. 1400 Seventeenth St. Denver 17, Colo, USA	Mill diameter: 400 mm Length: 400 or 800 mm Spiral diameter: 225 mm Capacity: 70 or 150 kg/h Feed size: 6 mm	50,000
MN 945/1 dry sieve	KHD Ind. AG Humboldt Wedag	Single deck screen, frame size: 250x500 mm Grain-size range: 0.2 ... 10 mm Capacity: 0.2 ... 1.2 m^3/h	4,000
Model CF-1 dry sieve	Gilson Co.	Continuous flow screen Screen frame size: 400x600 mm Single or double deck Grain-size range: 38 µm... 10 mm Capacity: 0.5 ... 2.5 m^3/h	4,500
APTS-600 dry sieve	Algaier Werke GmbH D-7336 Uhingen/Würt. FRG	Continuous double deck Flow screen, dust sucking accessories Grain-size range: 60 µm...10 mm Capacity: 1...6 m^3/h Sieve surface: 2x0.3 m^2 (round)	15,000
Vibration sieve Dia: 304.8 mm Wet sieve	William Boulton Ltd. Burslem, Stoke-on-Trent ST6 3 BQ U.K.	4 decks, for wet operation Capacity: 1...4 m^3/h Grain-size range: 60 m ... 10 mm	4,000 (1980)
Grading screen Dia: 18"	Denver Equipment Co. 1400 Seventeenth St. Denver 17, Colo, USA	4 decks, for wet operation Capacity: 1...4 m^3/h Grain-size range: 60 µm ... 10 mm	4,000
Air jet sieve 32/100, DBP	Alpine AG D-89 Augsburg 1 P.O.B. 101109	Screen size: 320x1,000 mm Grain-size up from 40 µm Capacity: 40 ... 320 kg/h Equipped with automatic dust reperator	30,000
Stehende Magnet Hub Rührautoklav 100 l	Andreas Höfer Hochdruck Apparatebau GmbH Mülheim Ruhr, FRG	Volume 100 l Strain capability: 10 MPa Temp. limit: 350 °C Material: steel	
25 gallon autoclav	Parr Instrument Co. 211 Fifty-Third Street Moline, Illinois, 61265 USA	Volume: 95 l Temp. limit: 350 °C Strain capability: 13 MPa Material: steel	
Thickener 8' diam. 6' deep	Porr Laboratory Suppiy P.O.B. 1252 Wilmington, California 90744 USA	Thick. surf.: 5.7 m^2 Volume: 10.4 m^3 Continuously operatable	13,000
PTA 3 m diameter 1 m deep thick.	Tatabányai Szénbányák Vértanuk tere 1, Pf. 323 2803 Tatabánya I. HUNGARY	Thick. surf.: 10 m^2 Volume: 10 m^3 Continuously operatable	10,000
Mobile labor. Drum filter 0.5 m^2	KHD Ind. AG Humboldt Wedag	Sucking, drying and blasing zones, precoat scraper roll, ring or ball discharge swivel agitator	25,000
Láng 1 m^2 drum vacuum filter station	Láng Gépgyár P.O.B. 320, Budapest 62. XIII. Váci ut 152-156. HUNGARY	Lower and higher vacuum zones, drying and blasing zones, standard scraper belt discharge spiral agitator	30,000
PTA 1/1-A or 2/1-A disc filter station	Tatabányai Szénbányák Vértanuk tere 1, Pf. 323 2803 Tatabánya I. HUNGARY	1 or 2 x 1 m^2 discfilter, consisting of 8 segments sucking, drying and blasing zones, swivel agitator water ring vacuum pump	25,000

7. TRAINING REQUIREMENTS

Supposing that the specialists (including management) selected for the bauxite testing laboratory have graduated in science, however, have not experiences in organizing and operating such kind of a laboratory it can be stated that special training is required.

Two kinds of training might be proposed:

- a general review course and laboratory practices on chemical, mineralogical, physical and physico-chemical, textural and technological examinations for the evaluation of different kinds of bauxite
- special courses in particular investigation technique according to the main activities and the equipment accessible (e.g. AAS, FS, XRF, XRD, TG, IR, SEM, TEM, EPMA - separately or in combination corresponding to the major fields of chemical, physical or tehcnological studies).

Both kind of training program consists of two stages:

- at first a few selected experts are trained (in form of group training) in the research center of the technology transferring institution
- in the second stage a course is organized in the installed new laboratory, on site, under supervision of the experts invited for the installation and in co-operation with scientists trained in the first stage.

A group training programme was organized by UNIDO in Sept.-Dec. 1979 on the subject "Production of Alumina". An outline for a similar training programme which may be organized in the future is included in the Appendix 2.

3. COST ESTIMATES FOR SETTING UP ALTERNATIVE TYPES OF BAUXITE TESTING LABORATORIES

In chapter 4., 5. and 6. the layout and main equipment with cost estimates are demonstrated. Using the listed data the costs of a definite system can be estimated. Informative costs for different level of laboratories can be calculated as follows:

"A" level laboratory (layout Fig. 3)

Building:	50,000 - 100,000 $
Infrastructure:	50,000 - 200,000 $
Equipment:	220,000 - 900,000 $
Total:	820,000 - 1,100,000 $

"B" level laboratory (to be added to "A" level, Fig.33)

Building:	100,000 - 300,000 $
Infrastructure:	50,000 - 100,000 $
Equipment:	250,000 - 1,100,000 $
Total:	400,000 - 1,500,000 $

"C" level laboratory (to be added to "B" level)

Building:	100,000 - 300,000 $
Infrastructure:	50,000 - 100,000 $
Equipment:	400,000 - 1,000,000 $
Total:	550,000 - 1,400,000 $

Large scale laboratory	500,000 - 1,000,000 $
Pilot plant	5,000,000 - 10,000,000 $

The costs vary considerably due to local conditions (especially infrastructure), the applied building system and equipment selected according to the tasks of the given bauxite testing laboratory.

The following main factors determine the constructing costs:

- the technical-economical situation of the given country
- the availability of the chosen building materials
- the applied building system
- the climatic situation of the site: (tropic, subtropic, continental, mediterranean)
- subsoil conditions
- existing level of infrastructure: road-, rail- and waterway network, drink water supply, canalization, supply with electricity and gas
- distance from the building material sources
- domestic regulations on construction, labour safety and fire protection
- labour costs.

Depending on these factors the investment costs can vary from 180 to 420 US$/$m^2$ of area built in.

9. RECOMMENDATIONS

9.1. INSTALLATION OF A BAUXITE LABORATORY

This profile serves as a basis to select the tasks and the level of a bauxite testing laboratory in the given developing country.

If a decision is passed, detailed project and cost calculation must prepared by a proper organization.

9.2. PROJECTING AND INSTALLATION

It seems desirable to charge one organization with all tasks connected with the installation of model laboratories, and preparation of detailed projects and cost calculations, preparation of work instructions and training of the personnel. This is chiefly motivated by the fact that very little open literature is available on specific experiences and detailed techniques for bauxite analysis and technological evaluation. For this reason eminent laboratories and experts of big aluminium companies or industrial corporations, respectively, (operating in either free market or centrally planned economics) can only is factory manner.

APPENDIX 1

SELECTED METHODS OF CHEMICAL ANALYSIS

1. CHEMICAL ANALYSIS OF BAUXITE (AND RED MUD)

UNIDO-ALUTERV-FKI, Group Training in Production of Alumina [3], Vol. 8, pp. 41-52.

2. DETERMINATION OF MAIN COMPONENTS OF RED MUD BY AAS AND PS METHODS

Vol. 8, pp. 89-95. [3].

3. CHEMICAL ANALYSIS OF ALUMINATE LIQUOR

Vol. 8, pp. 128-133. [3].

4. CHEMICAL ANALYSIS OF ALUMINA

For the analysis of alumina **several** ISO methods are available.

Components to be determined	ISO Method No.
Loss of mass on 3,000 °C	803-1976
Loss of mass on 1,200 °C	806-1976
Na_2O	1617-1976
Fe_2O_3	805-1976
SiO_2	1232-1976
TiO_2	900-1977

Components to be determined	ISO Method No.
V_2O_5	1618-1976
P_2O_5	2829-1973
ZnO	2072-1971

APPENDIX 2

PROPOSAL FOR TRAINING PROGRAM

1. PROPOSAL FOR A GENERAL REVIEW TRAINING PROGRAM

As an example the following group training program can be proposed on the basis of the experiences gained during the UNIDO Group Training for geologists, chemical engineers and technologists in production of alumina held in Budapest (Sept.-Dec. 1979) and organized by ALUTERV-FKI, Hungalu:

PART I.: Lectures

First week:
- Bauxite terminology, karst-bauxite deposites of the world, setting types and genetics
- Bauxite types and reserves, their evaluation for alumina production
- Laterite and its types, the distribution and characteristics of lateritic bauxite and karstic bauxite deposits of the world
- Short history of the Bayer process, circuit of the Bayer technology, present state and future trends in development of alumina processing

Second week:
- The main process stages, their characteristics (technology and equipment of the Bayer process)
- The chemical, analytical requirements for bauxite testing from the point of view of bauxite prospecting, control of technological investigations and plant processing

- Special chemical analytical methods for the determination of the main components and contaminants (AAS, FS, XRF, thermometric methods)

Third week:
- Quantitative determination of mineralogical composition of bauxite and red mud by means of different methods, applied alone or in combination: TG, IR, XRD methods and their fields of application
- Physical and physico-chemical investigation methods and equipment: focusing on the measurements of granulometry, specific surface area, and porosity, optical microscopy
- Special electron optical methods: EPMA, TEM, SEM + EDAX

Fourth week:
- Modelling of technological process stages of the Bayer technology
- Technological evaluation of different kinds of bauxite
- Tasks of large scale laboratory and pilot plants, their organization
- Main features of preparing feasibility studies

PART II.: Laboratory Practices

First week:
- Laboratory safety, regulations
- Chemical analysis of bauxite
- Bauxite grinding test and digesting liquor preparation

Second week:
- X-ray spectrometry and automatic bauxite analysis by neutron activation
- XRD
- SEM + EDAX

Third week:
- Thermogravimetry, IR spectrometry
- Conductivity measurement of aluminate liquor
- Precipitation test

Fourth week:
- Predesilication test
- Digestion test and red mud settling
- Control of red mud settling by γ-ray absorption measurement

Fifth week:
- Chemical analysis of red mud
- Determination of pore volume distribution in red mud
- Red mud causticization test
- Thermogravimetric measurements on $Al(OH)_3$

Sixth week:
- Determination of specific surface area of red mud
- Thermometric analysis
- Determination of grain-size distribution
- Chemical analysis of alumina
- Determination of physical properties of alumina

Remark: The demonstration of computer technique is performed in connection of each testing method if it is required to the evaluation.

PART III.: Getting Acquainted with Field and Plant Work

1 week (2 or 3 weeks required if installation of large scale laboratory or pilot plant is expected) visit to a bauxite prospecting company and alumina plant.

The full program can be reduced to 8 weeks if required. In this case the methodological and practical training must be performed in combination, investigations on alumina hydrate and alumina can be omitted and also the performance of research conducted by means of instruments of high value can be reduced to a demonstration of the methodology and applications (if "A" level laboratory staff is trained) and naturally the visits to bauxite prospecting company and alumina plant takes only 1 week.

2. PROPOSAL FOR SPECIAL COURSES ON SELECTED EXPLORATION TECHNIQUES

Such trainings can be organized as complementary to a review course or independently to study special fields of research (methodology and operation).

Topics: for an independently organized course:

First week:
- Theoretical background, principles of the selected method and equipment
- Laboratory safety regulations
- Short review on all methods applied in a typical bauxite testing laboratory

Second, third week:
- Laboratory practice in the selected field

Fourth week:
- Laboratory investigations
- Examples for data evaluation and data processing

- Principles of organizing a special laboratory and its operation

Remark: If this course is connected to the review training (proposal) the first week can also be used for laboratory practices.

APPENDIX 3

TECHNOLOGICAL EVALUATION OF BAUXITE

The aim of the industrial evaluation of bauxite is, on the one hand, the determination of its technological behaviour in an existing alumina plant, and, on the other hand, elaboration of optimum technology for a new plant in correspondence with the expected bauxite reserve. In the first case, given plant conditions have to be modelled, while in the second case, the task consists in a detailed study of technological variants and finding the optimum one.

The commercial bauxite is usually graded by the chemical analysis of the main components. Supply contracts are made on the so-called basic number, $B = Al_2O_3, \% - 2SiO_2, \%$. According to data [39] for the period 1955-1970, the bauxite price A was related to B by the following formula·
A = (B-29)x0.40 US$/ton. In the United States grading is often based on total available alumina (TAA) which is Al_2O_3 content digestible under standard conditions (instead of total alumina (TA) content), and a difference is also made between reactive SiO_2 (kaolinite) and non-reactive SiO_2 (quartz).

The real value of a bauxite can be derived from the costs of its processing into alumina. Therefore, all essential technological properties of the bauxite affecting its processing are to be considered.

1. BASIC PRINCIPLES OF THE TECHNOLOGICAL EVALUATION OF BAUXITES

The value of bauxite is finally deduced from the digestible Al_2O_3 content, TAA (bauxite consumption per ton alumina) and the caustic soda consumed in producing one ton alumina. These technological parameters determine the economics of the process.

The technological behaviour of bauxite is controlled by the mineralogical composition. However, the texture (morphology) is also of great importance and the role of these factors often can not be separated at all. As a first approach, the mineralogical composition will control equilibrium or pseudo-equilibrium states, while the texture will affect the kinetics of the processes. A finely disperse structure, a large specific surface area of the bauxite will, for instance promote rapid dissolution of the aluminium minerals, but - on the other hand - will cause difficulties in the separation and washing of the red mud thereby increasing so-called dissolved caustic soda losses. This is true, however, only until the individual minerals are not subjected to essential changes in the course of digestion, no phase transformation or recrystallization takes place. In most cases such processes do occur, at least partially, demonstrating the importance of the utilization of textural and physico-chemical investigations in the technological evaluating system.

Table 35

SYSTEM FOR BAUXITE GRADING AT HUNGALU

	Studies of texture and mineralogical compositions	Technological studies
Objective	Scientific basis, dynamic nature	Economical optimum, technological development
Test Methods	1. Determination of texture (thinsection microscopy, electron microscopy, electron microprobe) 2. Quantitative phase analysis of bauxite (X-ray diffraction, IR spectrometry, thermal analysis) 3. Quantitative phase analysis of red mud 4. Physico-chemical investigations (specific surface area, pore size distribution, heat of immersion) 5. Chemical analysis	1. Grindability 2. Digestibility and digestion parameters 3. Caustic loss 4. Separability and washability of red mud 5. Technological effects of bauxite contaminants
Application	Evaluation of different types of bauxites (gibbsitic, boehmitic, diasporic), furthermore special ores (e.g. goethitic, low-grade, contaminated)	

By way of example: when evaluating the technological effect of the most important minerals in bauxite, it is important to know that quartz and rutile are harmful only if they reacting with the digesting liquor. Different types of chamosite exist. If the chamosite does not react at digestion, not only its Al_2O_3 content, but its SiO_2 content will remain undissolved and hence will cause no caustic losses. Goethite and hematite will increase undigested Al_2O_3 losses proportionally to the isomorphous substitution of Fe atoms by Al atoms in their lattice. Hematite usually improves, goethite usually impairs settling and washing characteristics of red mud. The carbonate content of calcite and dolomite will largely raise the Na_2CO_3 content of the liquor, but will reduce caustic losses bound to red mud. On the other hand, these minerals increase Al_2O_3 losses as well.

In addition, the behaviour of individual components can change due to the interaction between various minerals. In the technological evaluation of bauxite these different effects and their origin are to be analyzed.

The most important factors determining the industrial value of a bauxite are: digestible Al_2O_3 content and sodium hydroxide consumption. The value of a bauxite for the alumina industry should expediently be determined so as to ensure reasonable profit [40].

For a given case, the selling price of alumina and the profit of alumina manufacture can be regarded as constants. Then the value of the bauxite can be expressed by the following formula [40]:

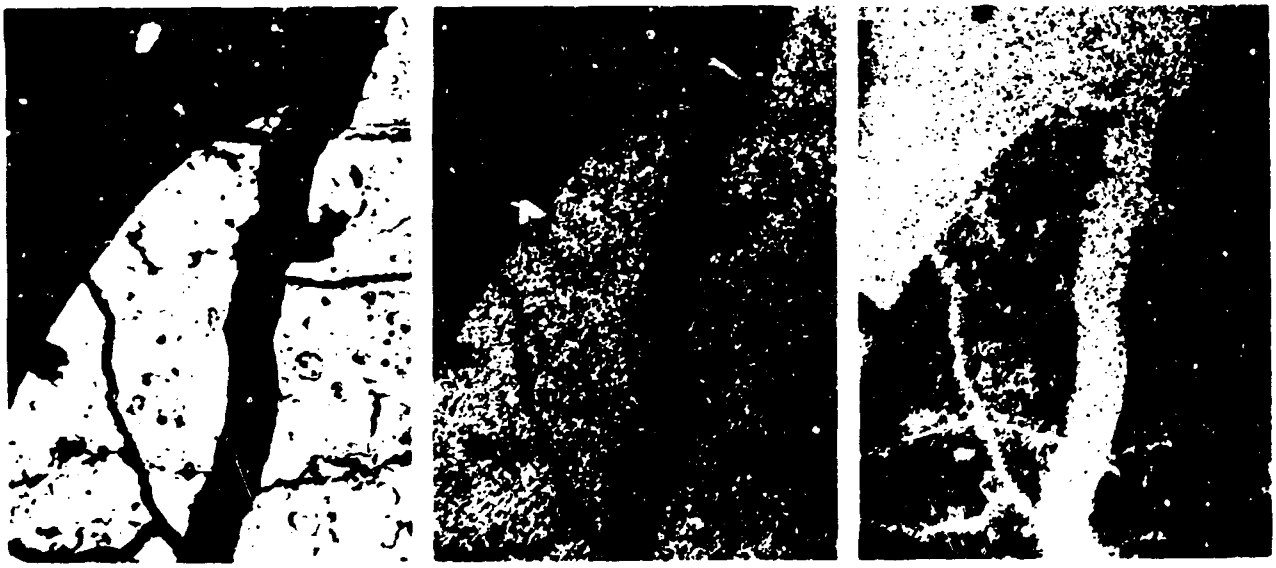
$$\text{value}_{\text{bauxite}} = \frac{C' - C_{Na_2O}}{G_{\text{bauxite}}}$$

Where C' is a constant, C_{Na_2O} the cost of making up caustic losses, and $G_{bauxite}$ is the amount of dry bauxite consumed. This value depends on the "soluble" Al_2O_3 content of the bauxite and on undigested and operation losses.

The effect of the contaminants in the bauxite and that of the separability of the red mud can be accounted for in the caustic consumption.

2. DETERMINATION AND TECHNOLOGICAL SIGNIFICANCE OF THE TEXTURE OF BAUXITES

The methods for studying the texture of bauxites are thin-section optical microscopy, electron beam microanalyzis (electron microprobe), transmission and scanning electron microscopy. These yield information concerning the size of the bauxite particles, the formation conditions of the bauxite, and the degree of crystallization. Also, the electron microprobe allows to determine the distribution of individual elements. Fig. 35.



a) b) c)

Fig. 35

Electron Microprobe Photos of a Hungarian Bauxite (Magnification 200x) a) Reflected Electron Image; b) Al Distribution; c) Fe Distribution

From the texture, conclusions may be drawn regarding the crushability of the bauxite and of the individual minerals presented in it, the reactivity towards the digesting liquor and the potential of physical enrichment.

By using colour photograph technique, the amount of information recorded in a single picture could substantially be increased. By studying powdered samples instead of thin sections, the results will reflect the average to a much higher extent, allowing to find out the technological behaviour of individual minerals and mineral aggregates and the potentials of enriching, and also to check the effectiveness of individual enriching operations.

Linear element distribution diagrams can also be made with samples ground to different particle sizes. Statistical-mathematical data processing will then yield results that characterize the true average composition, particle size distribution and element distribution.

This method, allows to answer important technological question as e.g.: identification of undigested aluminium minerals; correctness of CaO addition; formation of scales; reactions of calcite, dolomite and titanium minerals. Well-defined particles usually suggest bauxitic origin, while homodisperse systems imply secondary precipitation in red mud.

3. PHASE ANALYSIS AS THE BASIS OF TECHNOLOGICAL GRADING

The quantitative phase analysis of bauxite gives direct information on the Al_2O_3 yield to be expected.

From the phase analysis of the red mud, the efficiency of the digestion can be determined, as well as the distribution of Al_2O_3 losses among the individual mineralogical components [41].

The phase analysis of bauxite and red mud is generally based on X-ray diffractometry combined with thermogravimetry and sometimes combined with infra red spectroscopy as well. The mineralogical composition of certain bauxite samples can be determined purely by IR or TG technique.

For determination of Al_2O_3 yield (soluble Al_2O_3) on the basis of phase analysis the following formula is of general validity:

$$\eta^*, \% = \frac{Al_2O_{3reactive} - K_1 . SiO_{2reactive}}{Al_2O_{3total}} . 100$$

where $Al_2O_{3reactive} = Al_2O_{3total} - Al_2O_{3undigested}$. (Both $SiO_{2reactive}$ and $Al_2O_{3undigested}$ are values valid under the given digestion conditions.) $K_1 \geq 1.00$ is the number of Al_2O_3 mols relative to SiO_2 in the sodium aluminium hydrosilicate.

In practice, $K_1 = 1.00...1.10$.

To characterize the effectiveness of operation, we introduced the term of the efficiency of digestion. This value expresses the undigested and hydrolysis losses. It is obtained by dividing the actual yield calculated from the composition of the red mud by the modified theoretical yield η^*:

$$\eta' = \frac{\eta_{actual}}{\eta^*}$$

4. TECHNOLOGICAL INVESTIGATIONS

Our technological investigations serve the purpose to determine the expected behaviour of the bauxite in processing, and the optimum technological parameters for the given bauxite sample in the operations of ore preparation, digestion and red mud settling and washing. The expected harmful effects of the bauxite contaminants and the methods to reduce them must also be determined.

4.1 Grindability of the Bauxite

In the course of this test the optimal grain-size required to achieve satisfactory efficiency of digestion is determined and the grinding technology is elaborated. The usual particle size is $< 0.1 \ldots 0.3$ mm, but in the case of hard. diasporic bauxites finer particles are necessary. The tests are carried out in laboratory ball or rod mills, and the result of grinding is checked by sieve analysis, and for the finer fractions by using sedimentation methods (Sartorius balance or Sedimentometer).

Grinding, completed by IR studies, can also be used for the morphological characterization of the individual aluminium minerals.

4.2 Amount of Digestible Al_2O_3 and Digestion Parameters

The simplest method to determine the amount of digestible Al_2O_3 is to treat the bauxite sample with aluminate liquor under standard conditions. This test does not, however, yield any information on the parameters of digestion, like appropriate temperature, optimum molar ratio etc. For this reason, digestion is simulated in the laboratory by determining the characteristic digestion curves [41]. See examples in chapter 5.

The distribution of the Al_2O_3 content of the red mud among the mineralogical phases is checked by X-ray diffractometry. Characteristic digestion curves serve to determine not only the expected Al_2O_3 yield, but also the appropriate conditions of digestion.

4.3 Caustic Soda Losses

Caustic soda losses in the Bayer cycle decisively affect the economics of bauxite processing [40]. The losses to be expected can only partially be determined from red mud analysis (using flame photometric atom absorption techniques), since its results only allow to calculate bound caustic and so-called "absorbed" caustic soda losses, while the caustic soda consumption of bauxite contaminants (carbonate, sulfate, organic matter stc.) leading to a higher salt level is not included.

Caustic losses to be expected (L_{Na_2O}) are expressed by the following formula:

$$L_{Na_2O} = Na_2O_{bound} + Na_2O_{dissolved} + Na_2O_{transformed\ into\ salt}.$$

Na_2O losses to be expected must be determined experimentally, involving many different technological investigations.

4.4 Separability and Washability of Red Mud

The knowledge of the sedimentation characteristics of the red muds is essential for dimensioning the settler equipment and establish its operating conditions. To study the sedimentation and compaction of red muds, ALUTERV-FKI (HUNGALU) developed a γ-ray absorption apparatus, allowing the measurement of the density of the red mud slurry versus time [42].

If a great number of measurements are required, or if the bauxite sample is not large enough, one can conclude upon settling behaviour from the specific surface area of the red mud, using the BET technique of measurement, since we found that the sedimentation and filtrability of the red mud is reversely proportional to its specific surface area.

The data obtained for red muds of Hungarian and Obrovac (Yugoslavia) bauxites are presented in Fig. 36. Filtrability was measured, in large-laboratory scale. As shown by the figure, the hematitic red mud obtained from the goethitic-type Iszkaszentgyörgy and Obrovac bauxite by a slightly modified Bayer process separates more readily than the hematitic-type red mud formed from Halimba bauxite, and filtrability is also improved by about 25 % as compared to the initial goethitic red mud.

The explanation for indicated points is the following:

1. Hematitic mud transformed from Iszkaszentgyörgy bauxite
2. Hematitic-goethitic mud from Halimba bauxite
3. Goethitic mud from Iszkaszentgyörgy bauxite
4. Mud from dolomitic Halimba bauxite
5. Hematitic mud transformed from Obrovac (Yugoslavia) bauxite
6. Hematitic-goethitic mud (partially transformed) from Obrovac bauxite
7. Goethitic mud from Obrovac bauxite

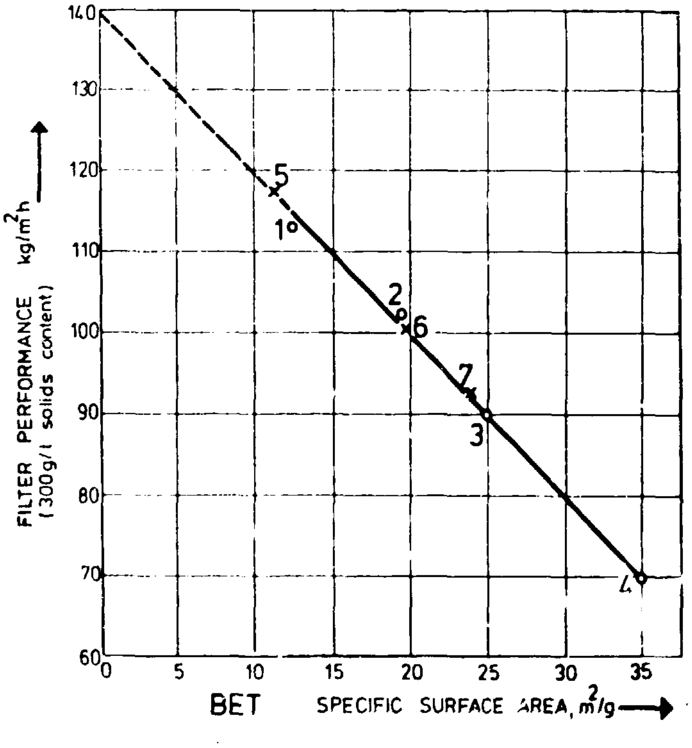
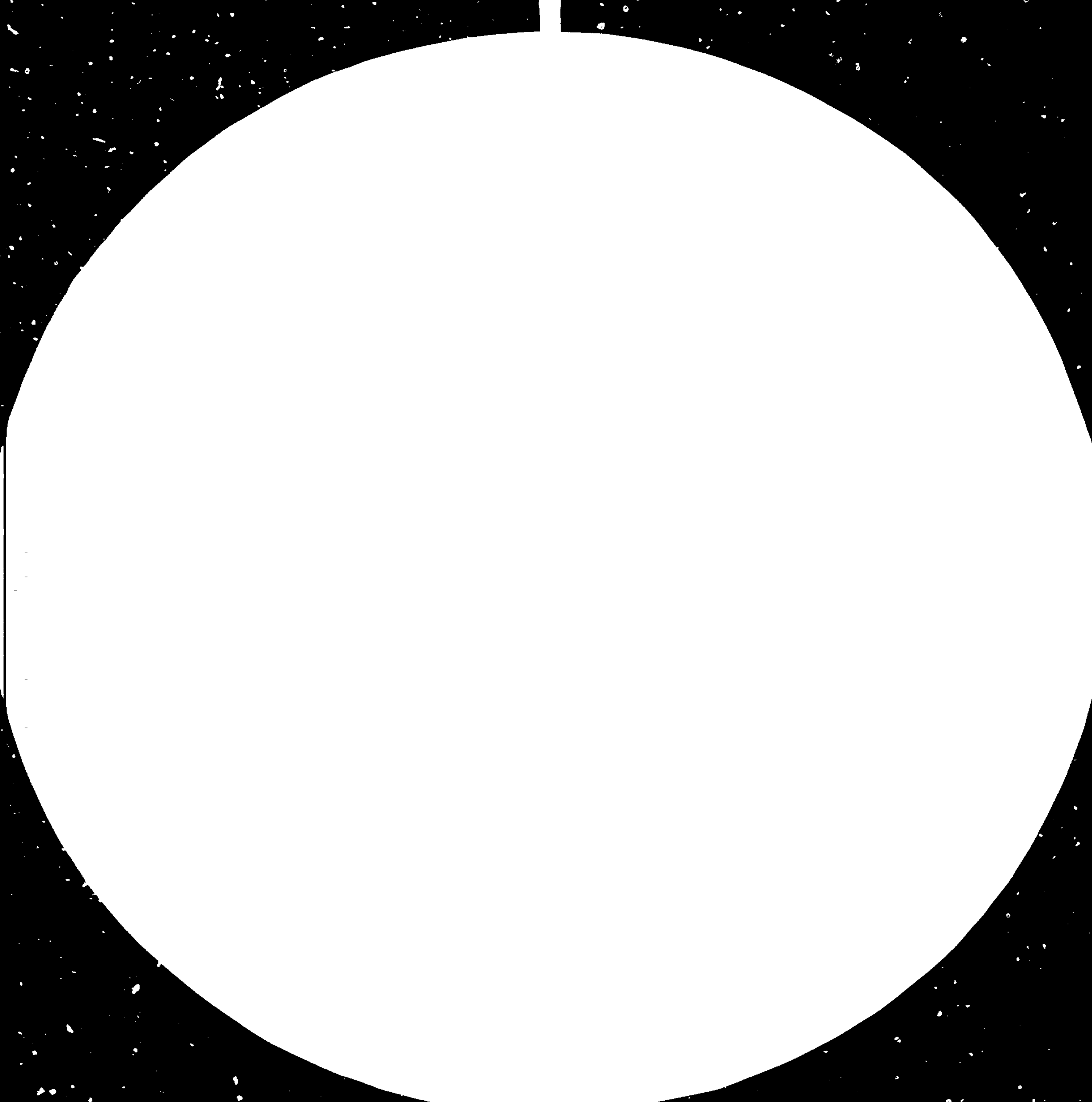
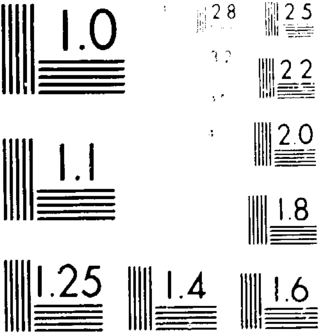


Fig. 36

Dependence of Filter Performance on Specific Surface Area of Red Mud




1.0
2.8
2.5
3.2
2.2
3.6
2.0
4.0
1.1
1.8
1.25
1.4
1.6

[illegible]

Washability of the red mud is of high importance regarding dissolved Na_2O and Al_2O_3 losses. The frequently contradictional experimental data became explainable on the basis of regular measurements of the specific surface area and pore size distribution of the red mud and its individual constituents [43].

4.5 Technological Investigation of Bauxite Contaminants

Among the bauxite contaminants, sulfur and carbonate content is detrimental primarily as consumer of caustic. The effect of fluorine and phosphorus is similar.

Zinc is also harmful. From some bauxites it is dissolved in such high percentages that it must continuously be removed from the liquor even at the expense of raising the costs of operation, since it would impair the required purity of the product alumina.

Some trace contaminants, e.g. vanadium and gallium, are not so detrimental, and can even increase the value of the bauxite if they are being recovered from the Bayer cycle at a commercial scale. This is the case in many countries, among them in Hungary, too. According to the plant experiences it is proved that V_2O_5 recovery is possible even at CaO addition to the digestion [44].

The behaviour of the salt contaminants concentrating in the circuit, their expected equilibrium level, the technology of their separation and their effect on the Bayer cycle, as well as the control filtration of the aluminate liquor (which also depends on the bauxite contaminants) can only be studied in a continuously operated, closed-circuit pilot plant or large scale laboratory. This is very important, since these factors frequently have a significant effect on the cost of alumina production.

The organic content of bauxites has various detrimental effects. We have not yet succeeded to find a suitable method for the quantitative evalution of these effects, however, the methodology of qualitative and quantitative analysis of organic contaminants in Bayer cycle has been developed [47].

5. SOME PRACTICAL APPLICATIONS OF BAUXITE EVALUATION

The application in practice of our grading system shall be illustrated on the example of a goethitic type bauxite and on that of two characteristic low-grade bauxites. These examples are, at the same time, proofs for the dynamic character of our system.

5.1 Evaluation of Goethitic Bauxite

In Fig. 37, the digestion characteristics of a Hungarian boehmitic-gibbsitic-goethitic bauxite from Iszkaszentgyörgy, determined at temperatures in the range of 180 to 300 °C are presented. The quantity and distribution of the main constituents in individual mineralogical phases are given in Table 36.

Table 36

MINERALOGICAL COMPOSITION OF HUNGARIAN GOETHITIC BAUXITE

Mineral	Al_2O_3%	SiO_2%	Fe_2O_3%	TiO_2%	CaO%	MgO%
gibbsite	20.5					
boehmite	22.2					
diaspore	1.6					
kaolinite	4.4	5.4				
quartz		1.6				
alumogoethite	1.3		12.6			
hematite			6.1			
anataze				2.3		
rutile				0.6		
calcite					0.3	
dolomite					0.3	0.3
Total	50.0	7.0	18.7	2.9	0.6	0.3

Aluminium substitution in goethite was 20 mol %.

For this bauxite, theoretical alumina yield is 88.1 %. If Al_2O_3 combined in alumogoethite is considered undigestible, the modified yield is only 85.5 %. This is confirmed by Fig.37 at temperatures of 240 °C and below, determined without additives, where diaspore (at least its major part) remains undigested, and the modified alumina yield to be expected is reduced to 82.3 %. In our calculations we assumed that total silica content is reactive, though - as indicated by the digestion curves - a substantial part of quartz did not react at 180 and 210 °C. The dotted line in Fig. 37 represents the characteristic obtained with synthetic, salt-free digesting liquor. In this case, owing to the higher alumina content of sodium aluminium hydrosilicate, alumina yield was lower than in the digestion test with industrial digesting liquor.

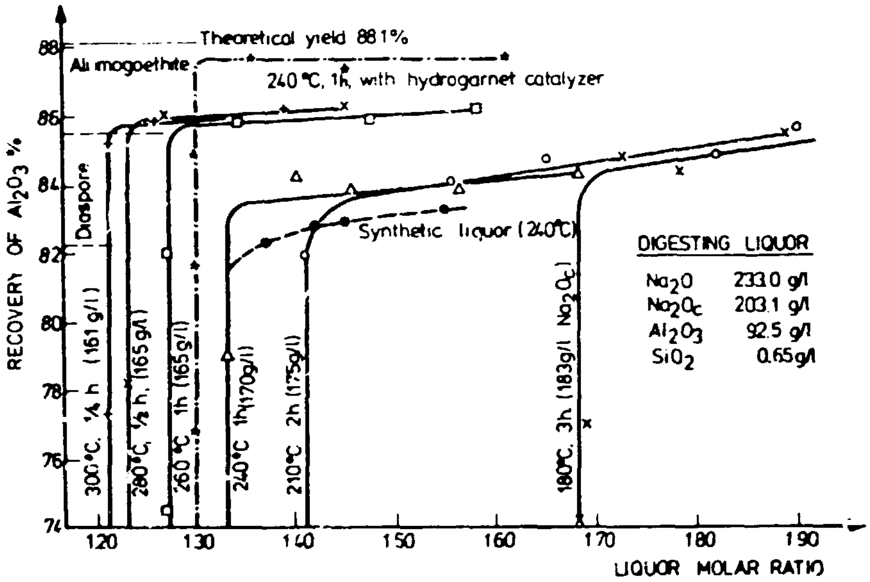


Fig. 37

Digestion Characteristics of a Hungarian Goethitic Bauxite from Iszkaszentgyörgy

The same bauxite has been digested by a recently developed Hungarian technology [45] in presence of hydrogarnet catalyzer, too, to convert goethite into hematite. According to the digesting curve, plotted in Fig. 37 the Al_2O_3 yield almost achieved the theoretical one, at 240 °C, 1 hour. At the same time the filrability improved as it is indicated on Fig. 36 (about 25 %). The bound soda losses have been reduced due to the additive. Nowadays the goethitic bauxite samples are evaluated and processed by this new method, consequently the industrial value of goethitic bauxites increased considerably.

5.2 Evaluation of Low-Grade Bauxites

The starting point for the determination of the bauxite's value is again the cost of alumina manufactured from it.

Evidently, this depends, besides the grade of the ore, to a large extent on the technology chosen and on the technical, level of processing. As a first approach, low-grade bauxites can be characterized by a reactive silica content exceeding 7 % and a reactive (digestible) Al_2O_3 content below 40 %. In a more general formulation, those bauxites are regarded as low-grade from which alumina cannot be manufactured economically (with a profit of 5 %) although the technology is fitted to the ore, the equipment is modern and the size is suitable [46].

For processing such low-grade bauxites, a decision can only be made taking into account all local conditions, all technological, economical and political factors acting at the given place and period.

We will illustrate by the example of an Australian quartz-rich bauxite and an Iranian chamositic bauxite that the processing of apparently very poor-grade bauxites can be economical by means of a properly chosen technology. The composition of the two bauxites and of the red muds formed from them is listed in Table 37.

Table 37

COMPOSITION OF AUSTRALIAN AND IRANIAN BAUXITES AND OF RED MUDS OBTAINED FROM THEM

Component		Australian bauxite	Iranian bauxite
Al_2O_3, % in	gibbsite	31.9	-
	boehmite	-	0.8
	diaspore	3.9	35.2
	kaolinite	1.5	2.8
	illite	-	0.4
	chamosite	-	7.0
	goethite	2.8	-
	Total	41.1	46.2
SiO_2, % in	kaolinite	1.8	3.3
	quartz	5.1	-
	illite	-	0.4
	chamosite	-	4.1
	Total	6.9	8.2
Fe_2O_3, % in	goethite	14.7	-
	hematite	13.1	16.8
	chamosite	-	8.9
	Total	27.8	25.7
TiO_2, % in	anatase	3.4	5.7
	rutile	traces	0.7
	Total	3.4	6.4
Component		Red mud, 150 °C, 150 g/l Na_2O_c	Red mud, 240 °C, 240 g/l Na_2O_c+4 % CaO
Al_2O_3 %		15.7	15.0
SiO_2 %		14.6	12.2
Fe_2O_3 %		53.5	41.2
TiO_2 %		6.6	10.5
Na_2O %		2.4	4.9
Loss on ign., %		6.8	7.0

75 % of the SiO_2 content in the Australian bauxite was quartz that did not react with the digesting liquor at 150 °C. This explains the low Na_2O content of the red mud. As it may also be seen in Fig. 38, Al_2O_3 in diaspore and in the goethite lattice remains undigested under such conditions. One must always reckon with these two loss sources when processing lateritic bauxites of the gibbsitic type, since the above minerals could be digested only under conditions where quartz would also become reactive. Thus, although the Al_2O_3 content of the bauxite is low (41.1 %) and the yield of alumina recovery did not reach even 80 %, this bauxite can be processed economically, owing to the low caustic losses to be expected as a result of the low reactive SiO_2 content.

The chemical and phase analysis of the red mud formed from the Iranian bauxite also revealed that 50 % of the SiO_2 content does not react with the digesting liquor and chamosite passes

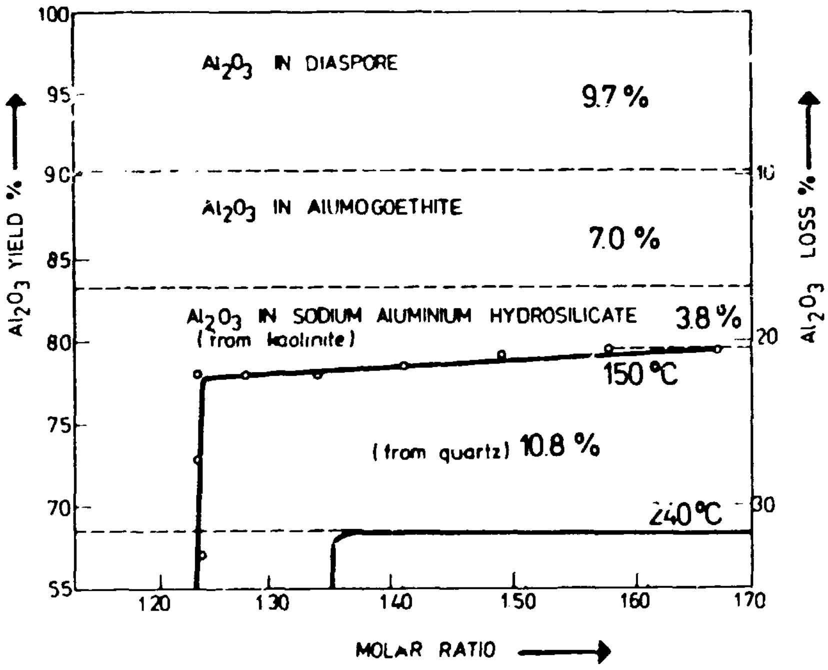


Fig. 38

Digestion Curves of an Australian Bauxite unchanged into the red mud, dispite of CaO addition. The reactive SiO_2 content of this Iranian bauxite is hence 8.2-4.1 = 4.1 % and its reactive Al_2O_3 content 46.2-7.0 = 39.2 %. Calculated alumina yield is 77.3 %, the experimentally found value 79.7 %. The Na_2O content of the red mud is low (4.9 %), so that the processing of this bauxite may be profitable, too.

These examples demonstrate how carefuly one must proceed in the grading of bauxite, and that correct evaluation can only be carried out based on the cost of their processing into alumina. It is reasonable to introduce, besides the term of reactive SiO_2, the term of reactive Al_2O_3.

In connection with poor-grade bauxite the question of bauxite enriching turns up frequently. The determination of the potentials of enrichment was discussed above, in the section on the texture of bauxite and its investigation by means of the electron microprobe analyzer.

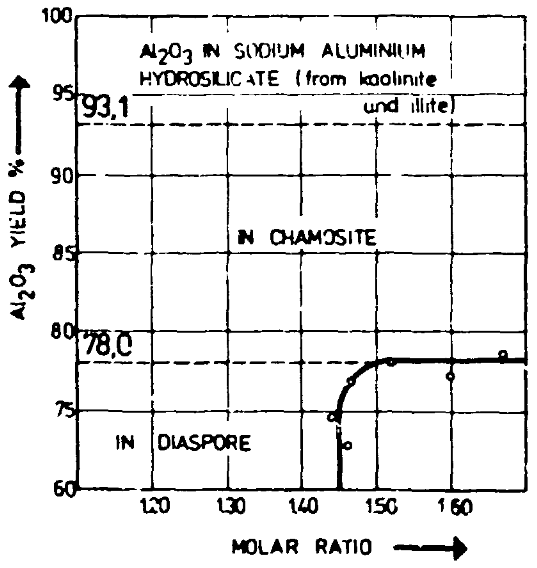


Fig. 39
Digestion Curve of an Iranian Bauxite

The economics of enrichment depends on what value increase can be obtained against the cost of enrichment, that is, to what extent the overall manufacturing cost of the alumina will decrease.

The question of enrichment arises in a different manner in the case of medium and high-grade ores suited for the Bayer process. In these cases it is always expedient to reduce - if economical conditions can be satisfied - the SiO_2, calcite, dolomite and pyrite contents affecting adversely the process.

Lateritic bauxite can often be enriched by washing and classification. Hence, when grading such bauxite, physical enrichment with simple methods must also always be envisaged.

6. DETERMINATION OF DESIGNING PARAMETERS

The design of a new alumina plant or the optimization of an old one is based on technological evaluation of a representative sample.

One part of the parameters must be determined by thorough calculations based on experiences of operating plants as well.

As an example the design parameters of a non specified alumina plant processing gibbsitic bauxite and producing sandy alumina are presented below (Table 38). All parameters depending on the bauxite concerned should be determined experimentally on laboratory scale (large laboratory scale) or pilot plant scale respectively.

The conceptual process flow sheet of an alumina plant, characterized by the design parameters collected in Table 38, is demonstrated in Figs 40, 41.

Table 38

DESIGN PARAMETERS

Production		
Alumina production,	tpy	500,000
Hourly design rate,	t	62.5
Design operating factor,	%	92.0 (8,000 hrs/year)
Bauxite (average sample)		
Al_2O_3, total,	%	48
in gibbsite,	%	40
in boehmite,	%	4
SiO_2, total,	%	3
in kaolinite,	%	2
in quartz,	%	1
Fe_2O_3,	%	25
TiO_2,	%	3
P_2O_5,	%	0.1
V_2O_5,	%	0.1
Organic carbon,	%	0.2
L.O.I.,	%	23
Free water in wet bauxite (year's average),	wt%	9
Lump size of ore,	mm	max. 30

Table 38 (cont.)

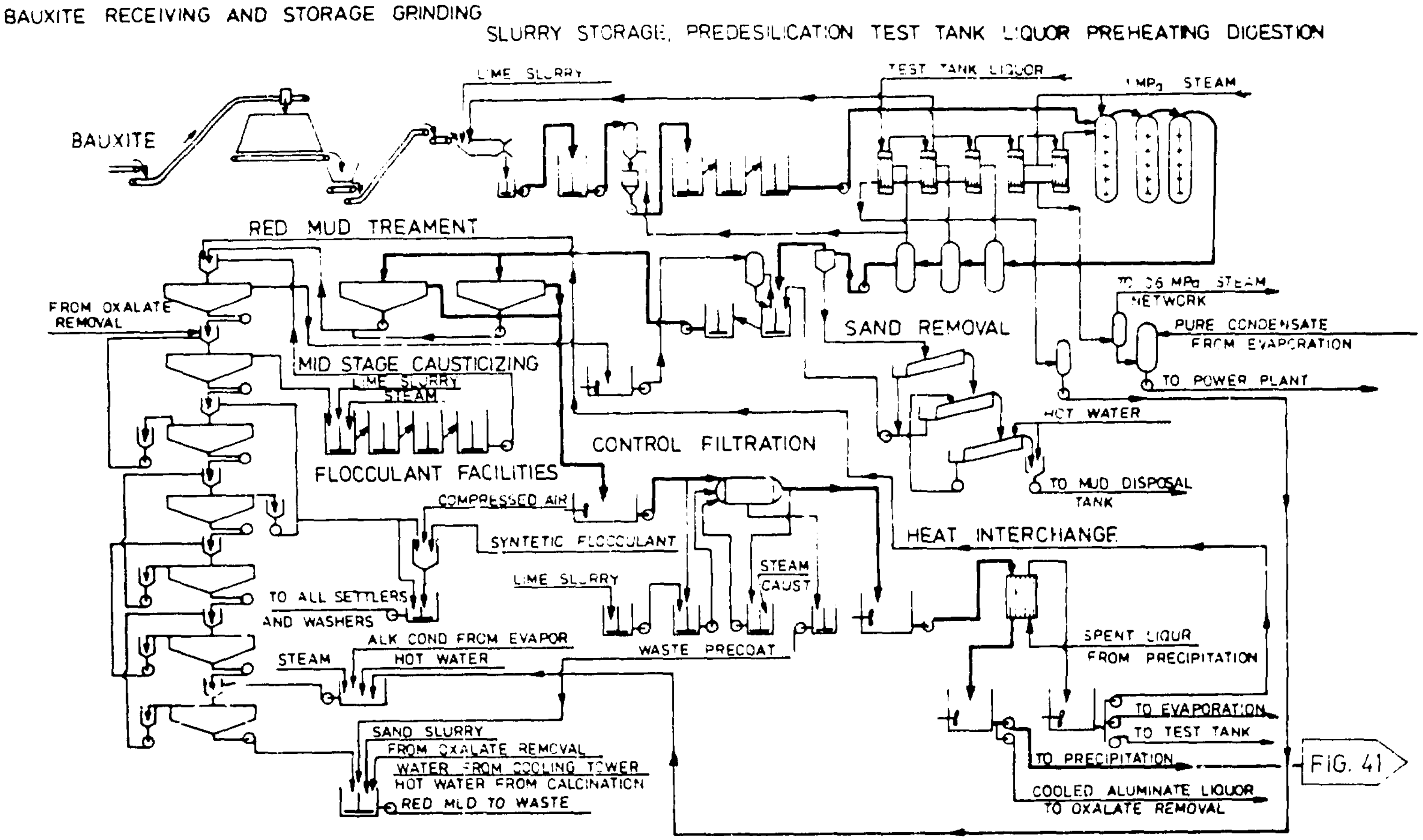
Grinding, predesilication		
Solids content,	g/l	600
Lime added,	% of dry bauxite	0.1
Predesilication holding time,	hrs	8
Predesilication temperature,	°C	100
Test tank liquor caustic Na_2O,	g/l	140
Test tank liquor caustic molar ratio		2.8
Temperature of grinding liquor,	°C	105
Digestion		
Temperature,	°C	140
Holding time at 140 °C,	hrs	0.5
Digestion efficiency for gibbsite,	%	98
Soda losses at digestion:		
for reactive silica,	kg Na_2O/kg SiO_2	0.69
carbonation,	kg Na_2O/t dry bauxite	0.5
Bound in other salts,	kg Na_2O/t dry bauxite	0.9
Digestion residue,	t mud/t dry bauxite	0.5
Sand/mud,	%	5
Mud specific gravity,	g/cm^3	3.5
Final caustic molar ratio,	caustic Na_2O/Al_2O_3	1.4
Red mud settling, washing		
Caustic Na_2O content of the settler overflow,	g/l	105
Molar ratio of the settler overflow,	g/l	1.55
Solids content of the settler overflow,	g/l	500
Solids content of the washer underflow,	g/l	500
Flocculant addition to settler,	g/t dry mud	50
Flocculant addition to washers,	g/t dry mud	60

Table 38 (cont.)

Settling temperature,	°C	102
Temperature of interstage causticizing,	°C	85
Retention time at causticizing,	hrs	2
Lime addition,	kg CaO/t alumina	3
Temperature of red mud wash water,	°C	80
Wash water for sand washing,	t/t	3
Moisture of sand,	%	30
<u>Control filtration</u>		
Flow rate,	m^3/m^2h	0.5
Lime precoat,	kg/m^3 aluminate liq.	0.2
<u>Heat interchange</u>		
Aluminate liquor,	°C in	99
Aluminate liquor,	°C out	75
Spent liquor,	°C in	65
Spent liquor,	°C out	88
<u>Precipitation</u>		
Aluminate liquor:		
Caustic Na_2O,	g/l	105
Caustic molar ratio		1.55
Causticity, caust.Na_2O/total Na_2O, %		0.88
Average seed ratio		1.0
Holding time,	hrs	40
Molar ratio of spent liquor		2.8
Mid-stage cooling of precipitators,	°C	5
Solids content as $Al(OH)_3$ in tray thickener overflow,	g/l	1.0
Various dilutions,	t water/t alumina	0.5

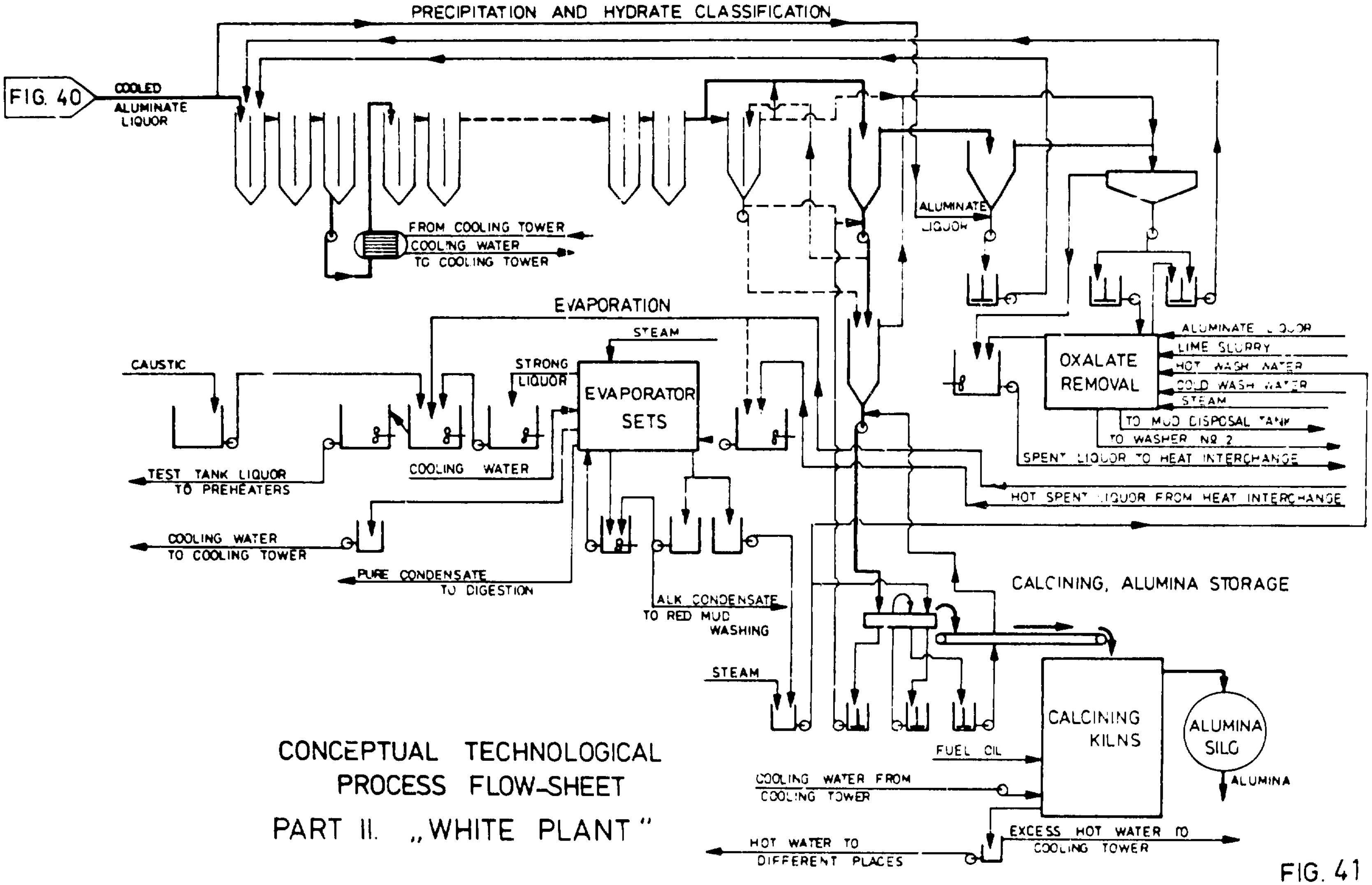
Table 38 (cont.)

Product washing and calcination		
Moisture of product hydrate,	%	12
Wash water to filters,	m^3/t alumina	0.6
Na_2O in alumina,	%	0.4
Kiln and loadout losses,	% of product	0.5
Product purity,	%	99.4
	(calculated on an ignited basis)	
Evaporation		
Evaporated water,	t/t alumina	4.2
Oxalate removal		
Wash water,	m^3/t alumina	0.4



CONCEPTUAL TECHNOLOGICAL PROCESS
FLOW-SHEET
PART I. „RED PLANT"

FIG. 40



CONCEPTUAL TECHNOLOGICAL
PROCESS FLOW-SHEET
PART II. „WHITE PLANT"

FIG. 41

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