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UNITED NATIONS INDUSTRIAL DEVELOPMENT ORGANIZATION

Development and Transfer of Technology Series

No. 20

BAUXITE TESTING LABORATORIES

UNITED NATIONS

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BAUXITE TESTING LABORATORIES

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Preface

The world's prospective bauxite resources are located mainly in developing countries while the processing is concentrated in the developed ones. To successfully process bauxite, and to establish a national processing industry, a thorough knowledge of local bauxite deposits is an important prerequisite.

At the first meeting of the Expert Consulting Group on the Aluminium Industry, organized by UNIDO and held at Vienna in 1967 [1], the following recommendation was made: "[UNIDO should] 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.)"

In 1973, as part of its response to this recommendation, UNIDO made a study on analytical methods for testing bauxite, alumina and intermediate products [2].

As a next step, UNIDO organized a group training course on the production of alumina, at Budapest in 1979 (in co-operation with the Hungarian Aluminium Corporation). The topics outlined by the lectures and a manual for the laboratory investigations were collected in eight volumes [3].

The present study is based on the publications mentioned above. It takes into account the latest innovations in and equipment for the chemical, mineralogical and technological investigation of bauxite and is especially intended for developing countries that have bauxite resources.

The study was prepared for UNIDO by P. Gadó, L. Tomcsányi, D. Bulkai and K. Solymár of the Research, Engineering and Development Centre of the Hungarian Aluminium Corporation (AT-FKI), Budapest.

EXPLANATORY NOTES

References to dollars ($) are to United States dollars, unless otherwise stated.

The following forms have been used in tables:

Three dots (. . .) indicate that data are not available or are not separately reported
A dash (—) indicates that the amount is nil or negligible
A blank indicates that the item is not applicable
Totals may not add precisely because of rounding.

In addition to common abbreviations, symbols and terms, the following have been used in this report:

AAS	atomic absorption spectrometry
ABA	automatic bauxite analyser
a.c.	alternating current
AT-FKI	Research, Engineering and Prime Contracting Centre of the Hungarian Aluminium Corporation
CPU	central processing unit
CRT	cathode ray tube
d.c.	direct current
DTG	differential thermogravimetry
EDAX	energy-dispersive X-ray analysis
EDL	electrodeless discharge lamp
EDTA	ethylenediaminotetraacetic acid
EPMA	electron-probe microanalysis
ETA	electrothermic atomizer
FS	flame spectrometry
Hungalu	Hungarian Aluminium Corporation
ICF	intensity conversion factor
IR	infrared spectrometry
LOI	loss on ignition
PTFE	polytetrafluorethylene
RICF	relative intensity conversion factor
SEM	scanning electron microscopy
TAA	total available alumina
TEM	transmission electron microscopy
TG	thermogravimetry
XRD	X-ray diffraction
XRF	X-ray fluorescence

Summary

World reserves and utilization of bauxite

The world's bauxite resources are mainly located in the developing countries. However, bauxite processing is concentrated in the developed countries.

Despite the expected diminution of the growth rate of consumption during the next decade, the aluminium industry will remain an attractive area for developing countries to invest in. In locating a new processing plant, the following considerations must be taken into account:

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

(b) The bauxite producing countries would benefit from a shift towards more valuable products;

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

Consequently it may be expected that more alumina refineries will be located close to mining areas.

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

Functions of bauxite testing laboratories

There are three levels of bauxite testing laboratories:

Level A: The simplest service laboratory for geological exploration and mining. Performs bauxite analysis, and enables the country to study its ore resources and promote economical marketing.

Level B: For technological evaluation of ores; a laboratory of level A supplemented by technological equipment. Its operations include modelling the recovery of alumina from bauxite. This type of laboratory may be installed independently or connected to an alumina plant or an existing research laboratory.

Level C: A complete research laboratory for bauxite testing and alumina technology. Functions: those of level B plus material investigation and modelling of processing stages of the Bayer process by means of automated and computerized equipment, optionally complemented with a large laboratory and pilot plant; preparation of feasibility studies for the future alumina plants; elaboration and optimization of different procedures and analytical methods.

Establishing a bauxite testing laboratory

The principal considerations when establishing a bauxite testing laboratory are the selection of methods and equipment, the application of the "building block" system, the estimation of analytical work required (including the technological investigations) and the question of whether to conduct large-laboratory or pilot-plant investigations.

The main aim of a pilot plant is to test the critical stages of the whole process and to test new equipment. Two types of pilot plants can be established: one characterized by a high flexibility of parameters in each stage of the process, suitable for studying very different kinds of bauxite; and one in which this flexibility is applied only to selected processing steps. Large laboratories and pilot plants are operated in a closed circuit for the study of the consequences of contaminant enrichment. Thus, pilot plants should be planned individually.

Units and activities of bauxite testing laboratories

The activities of bauxite laboratories can be classified into four main groups: chemical analysis, X-ray investigations, technological testing and simulation, and physical and physico-chemical investigations.

In this study, the estimated costs and personnel needs are given for the various laboratory units. The details of the various investigative methods are in separate annexes.

Requirements for equipment, building and human resources

Detailed equipment lists, building layouts and human resource requirements are given for all levels of laboratory.

Training requirements

Even the graduate-level specialists will require special training in organizational and operational techniques. Two types of training that should be considered are a general review of laboratory practices and special courses in particular techniques. Each programme should consist of two steps: *(a)* a few selected experts are trained as a group in the research centre of the technology-transferring institution; *(b)* a course, supervised by the experts, is organized in the new laboratory. Details of these training programmes are given in an annex.

Estimated cost of bauxite testing laboratories

The approximate costs of different levels of laboratories are as follows:

Laboratory	*Cost (thousands of dollars)*
A level	500-1 000
B level	1 000-3 000
C level	3 000-5 000
Large laboratory (closed-circuit)	500-1 000
Pilot plant (closed-circuit)	5 000-10 000

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I. World reserves and utilization of bauxite

Distribution of bauxite deposits

World bauxite production in 1980 amounted to 81.4 million tonnes [4]. World bauxite reserves were estimated by de Weisse at 8 billion tonnes in 1972 [5], and 38 billion tonnes by Lotze in 1978.

Costs of bauxite are dependent on the geographical location of the ore, the availability of mining infrastructure and transportation costs. Estimated bauxite resources, by country, are given in table 1.

Resources are divided into developed resources and undeveloped resources [6]. Developed resources are, in turn, divided into "mineable reserves" and "potential ores". Resources are deposits of bauxite in or on the Earth's crust such that economic exploration is (currently or potentially) feasible. Developed resources are bauxite deposits or areas currently under exploitation. Undeveloped resources are known bauxite deposits from which future economical exploitation may be expected. Mineable reserves are bauxite deposits currently being exploited economically under existing conditions. Reserves of undeveloped resources are those for which future exploitation is expected. Potential ores of developed resources are that portion of resources in the continuity of known deposits which are insufficiently explored at this time and for which quantitative size estimates are based largely on broad knowledge. Potential ores of undeveloped resources are that portion of currently subeconomic resources which may become reserves as a result of changes in economic conditions or after further exploration.

TABLE 1. ESTIMATED BAUXITE RESOURCES CLASSIFIED BY ECONOMIC EVALUATION
(Millions of tonnes)

	Resources				
	Developed		*Undeveloped*		
Location of deposits	*Mineable reserves*	*Potential ores*	*Reserves*	*Potential ores*	*Total*
Africa					
Cameroon	...	...	680	1 200	1 880
Guinea	1 430	4 000	2 950	1 000	9 380
Other	60	...	650	830	1 540
Total	1 490	4 000	4 280	3 030	12 800
Americas					
Brazil	70	...	1 350	1 900	3 320
Jamaica	2 000	...	...	1 000	3 000
Suriname	200	...	200	1 570	1 970
Colombia	...	...	115	905	1 020
Other	200	250	50	1 535	2 035
Total	2 470	250	1 715	6 910	11 345
Asia					
India	50	...	1 010	1 495	2 555
Indonesia	40	40	...	1 000	1 080
Other	35	5	100	845	985
Total	125	45	1 110	3 340	4 620
Europe	840	350	...	280	1 470
Oceania					
Australia	1 215	1 800	830	2 180	6 025
Total of deposits included above	6 140	6 445	7 935	15 470	36 260
Unclassified deposits not included above					1 965
World total					38 225

Source: Lotze [6].

Lotze [6] indicated that for alumina production, including mining and transportation costs, 12% of the total bauxite reserves belong to the lowest cost category. Ninety-five per cent of these resources are in Jamaica, Guinea, Australia, Suriname and Greece.

Africa has major bauxite reserves with Guinea the largest African producer. Ghana and Cameroon have significant reserves.

In the Americas, Brazil, Colombia, Jamaica and Suriname also have significant reserves.

The information in table 1 indicates that developing countries have abundant bauxite resources and the aim to build up processing facilities seems to be reasonable.

Trends in bauxite processing

Bauxite production and marketing is dominated by large-scale mines located near to large-capacity tidewater ports.

The distribution of bauxite production for selected countries for the years 1950, 1965 and 1978 is shown in table 2. The most significant change from 1950 to 1965 was the rise of Jamaica as a major world supplier of bauxite. A similar rise occurred between 1965 and 1978 in Guinea.

In 1978 Australia took over as the largest bauxite producer in the group (33.3%) and Suriname dropped from first to fourth between 1950 and 1978.

These shifts resulted from changes in the relative production costs. It is expected that the share of production of such relatively new producers as 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 and 1984, are shown in table 3, adopting the data of B. Balkay [7]. It can be seen that the processing of bauxite is especially limited in Africa and in Latin America.

TABLE 2. DISTRIBUTION OF BAUXITE PRODUCTION IN A SELECTED GROUP OF COUNTRIES, 1950, 1965 AND 1978

(Percentage)

Country	*1950*	*1965*	*1978*
Australia	—	3.8	33.3
Brazil	0.3	0.6	1.7
France	11.4	8.6	2.7
Guinea	0.2	6.1	16.5
Guyana	22.9	9.5	4.7
Jamaica	—	28.3	16.1
Suriname	29.4	14.2	7.2
United States of America	19.3	5.5	2.3
Remainder of group	16.5	23.4	15.5
Total	100.0	100.0	100.0

Source: Langton [4].

The predicted primary aluminium consumption, alumina production and bauxite production in the world is shown in table 4 [4].

Despite the expected diminution of the growth rate of consumption during the next decade, the aluminium industry will remain a potentially attractive area for investments. In locating new alumina plants the following considerations should be taken into account:

(*a*) Shipping charges have become increasingly important;

(*b*) The bauxite producing countries will benefit from a shift towards more valuable products;

(*c*) The developing countries should aim at better control over mining and processing.

Consequently, it can be expected that more alumina refineries will be located close to mining sites.

TABLE 3. BAUXITE MINING AND PROCESSING CAPACITIES, 1978, EXPECTED INCREMENTS UP TO 1984 AND RELATIVE CAPACITIES OF PROCESSING, 1978-1984

	Bauxite mining			*Capacity ratios*[a]		*Alumina refining*		
Country or area	*Capacity 1978 (thousands of tonnes)*	*Increase to 1984 (thousands of tonnes)*	*Percentage of world increase*	*1978*	*1984*	*Capacity 1978 (thousands of tonnes)*	*Increase up to 1985 (thousands of tonnes)*	*Percentage of world increase*
Africa	14 262	6 945	29.5	0.14	0.10	700	...	...
Asia without Japan	3 756	105	0.4	0.86	1.27	1 120	540	7.5
Europe	8 680	2 040	8.7	1.89	2.39	5 675	2 963	41.0
Latin America	30 449	11 200	47.6	0.47	0.49	4 990	1 882	26.1
Northern America	2 150	...	...	11.20	11.66	8 330	135	1.9
Oceania	28 000	3 245	13.8	0.70	0.81	6 800	1 700	23.5
Japan	...	...	...	...	...	2 580	...	...
Total	87 297	23 535	100.0	1.00	1.00	30 195	7 220	100.0

Source: Balkay [7].

[a] The capacity ratio for a given area is the ratio of the amount of bauxite processed into alumina to the amount of bauxite mined within the region.

TABLE 4. WORLD ALUMINIUM CONSUMPTION AND ALUMINA AND BAUXITE PRODUCTION, 1980-1990

Item	*1980*	*1985*	*1990*	*Annual growth, 1980 to 1990 (%)*
	(millions of tonnes)			
Primary aluminium consumption	12.2	15.4	19.6	4.9
Alumina production	27.9	34.7	44.1	4.7
Bauxite production	81.4	103.4	134.3	5.1

Source: Langton [4].

Bauxite processing technology

More than 90% of the world's alumina is produced by the Bayer process, which yields a high-purity final product.

Alumina production is a beneficiation process to extract Al_2O_3 (alumina) from bauxite. This alumina may then be electrolyzed to aluminium in a cryolite melt.

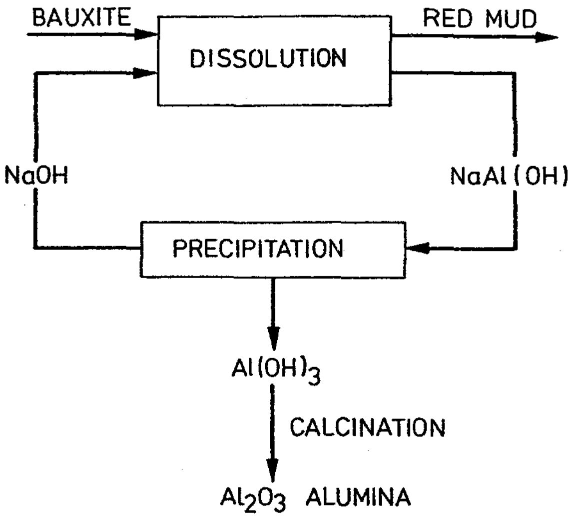
The industrial Bayer process is based on the following reaction:

$$\underset{\text{solid gibbsite}}{Al(OH)_3 + NaOH} \; \underset{\substack{< 100^\circ\ C \\ \text{precipitation}}}{\overset{\substack{\text{dissolution} \\ > 100^\circ\ C}}{\rightleftharpoons}} \; \underset{\text{in solution}}{NaAl(OH)_4}$$

The Bayer process consists essentially of digesting the crushed and ground 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 crystals of trihydrate whereupon precipitation takes place. The precipitated trihydrate is filtered and subjected to calcination at about 1,200° C. The simplified flow sheet is shown in figure 1.

A scheme of material-streams through the main operation units is illustrated in figure 2.

Figure 1. Simplified flow-sheet of the Bayer process

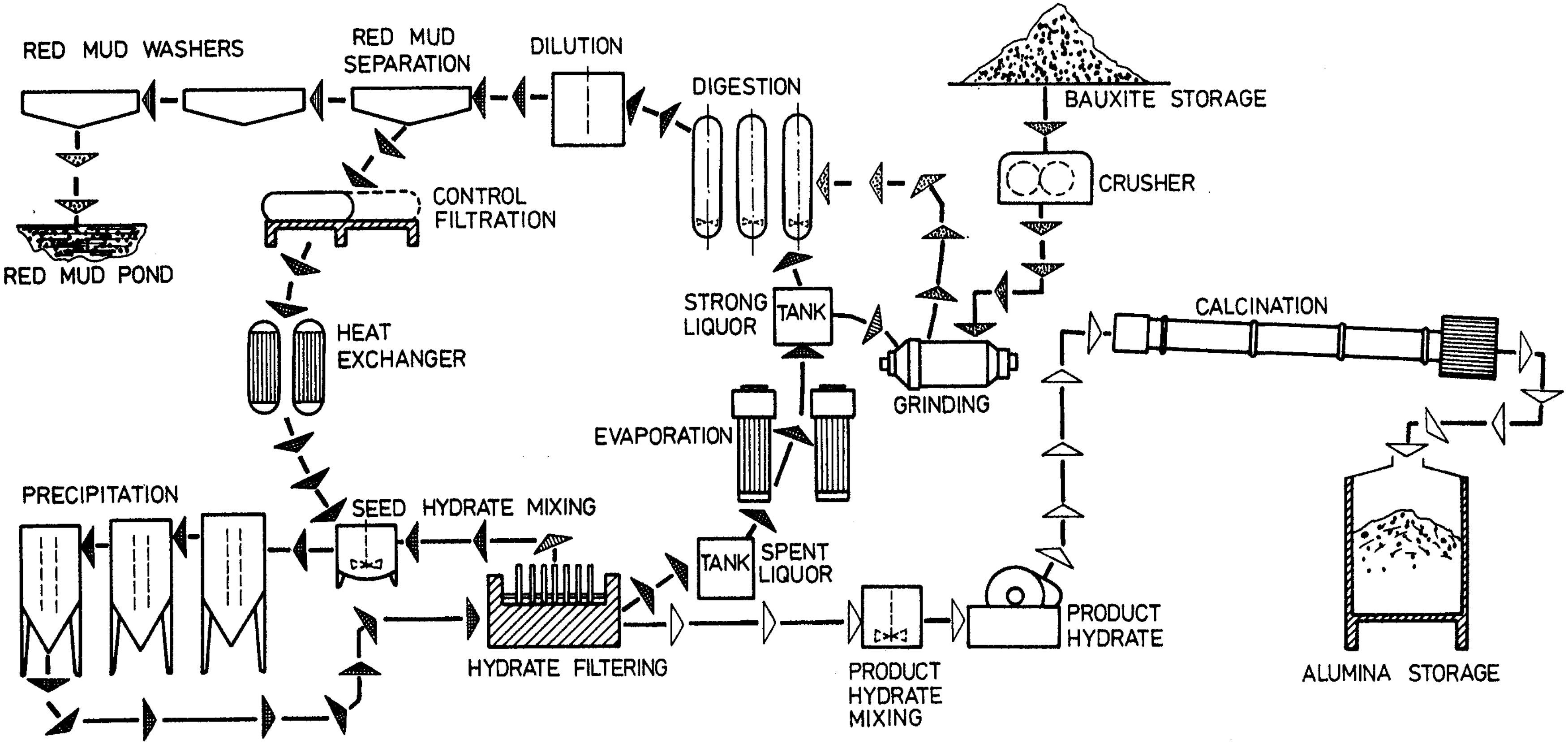


Role of the bauxite testing laboratory in resource utilization

To evaluate bauxite resources, a well-organized bauxite testing laboratory for determination of the chemical and mineralogical composition of a large number of bore-hole samples is needed. Technological studies may often be required for marketing purposes.

There are various levels of laboratories. If processing at the site is planned the laboratory must conduct some tests on pilot scale. The most advanced type of bauxite testing laboratory can serve as a research centre and possibly as a training centre for the staff of alumina plants. Such a laboratory should be installed near bauxite deposits or connected with alumina plants. The research centre may either be independent of or operate as part of existing research institutes, university departments or other institutions. Operation in co-operation with other institutions has the advantage that expensive instruments can be effectively used for many tasks and the laboratory can provide education as well.

Figure 2. Scheme of the Bayer process



Source: Lepori [8].

II. Functions of bauxite testing laboratories

The following is a brief survey of the functions of bauxite testing laboratories beginning with the simplest. The three main types of laboratories share certain functions, and overlapping is inevitable.

Laboratory for geological exploration and mining (level A)

The level A laboratory is the simplest and performs analyses 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 producing countries to determine their ore resources qualitatively and quantitatively. In accordance with earlier international practice, analyses are usually done to determine loss on ignition, and alumina, silica, iron-oxide and titanium-oxide content.

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

Classical chemical analytical methods can be replaced by automatic, fast and highly reproducible methods such as X-ray fluorescence spectrometry.

The task of geological laboratories has broadened recently by mineralogical analysis. Earlier, simple methods, such as thermogravimetry (TG), differential thermogravimetry (DTG), differential thermal analysis (DTA), were applied; however, currently the quantitative phase analysis of bauxite is based on X-ray diffractometry. Fundamental tests of digestibility are also required (determination of Al_2O_3 content soluble in caustic soda).

Laboratory for ore evaluation (level B)

Some technological investigations are necessary for marketing, especially if the bauxite varies considerably in chemical or mineralogical composition. The level B technological laboratory is primarily engaged with digestion tests and settling tests of the obtained red mud in order to determine the industrial value of ores. The pertinent data relate to Al_2O_3 recovery, digestion parameters, expected soda losses and separability or washability of the mud. These measurements help to predict the effects of composition on processing. The laboratory also provides chemical and mineralogical analysis of red mud and the analysis of aluminate liquors. The laboratory should collect information on the technological behaviour of the bauxite, and secure qualified experts to co-operate in establishing the national ore processing facilities in the country.

Such a laboratory can help the ore-producing countries to control the processing of bauxite in existing plants operated by international companies. The facilities may be installed independently or attached to an alumina plant or research laboratory.

Complete research laboratory (level C)

In addition to the functions of the preceding laboratories, the complete laboratory models processing phases of the Bayer cycle on a laboratory and on a pilot-plant scale for the established alumina industry; the laboratory prepares feasibility studies for future plants; and elaborates and optimizes different procedures and analytical methods.

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

Scale	*Quantities handled (kg)*
Laboratory	10^{-3}-1
Large-laboratory	1-10^2
Pilot-plant	10^3-10^4
Industrial	10^6

Laboratory-scale experiments are generally arranged in batches, while large-laboratory experiments and pilot plants are operated in closed circuits; therefore, laboratory-scale equipment can be located independently of production. In the case of a large laboratory or a pilot plant it seems advisable to place the operation near an alumina plant.

The building of a pilot plant can help to implant an industrial facility (especially in the case of inhomogeneous ore composition) by determination of optimal technology and by personnel training.

It is necessary to determine on an individual basis whether it is preferable to establish a more flexible and by far less expensive large laboratory or a pilot plant. Large laboratories are adequate for the general study of all problems arising in the Bayer process.

Pilot plants may provide for the testing of new or newly modified processing equipment and are strongly recommended in advance of industrial application.

III. Establishing a bauxite testing laboratory

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

(a) Select appropriate methods and equipment corresponding to the analyses and to the amount of expected technological investigations as well as to the qualifications of the personnel, taking into account the required maintenance, the related cost, the productivity and the accuracy;

(b) Follow the "building block" system and set up a high-level research laboratory starting with a simple service laboratory for geological exploration;

(c) Examine the necessity of establishing a pilot plant and, if such a need is proved, carefully determine its scale and the problems to be examined.

Analytical tests

In the organization of an industrial laboratory one of the most important considerations is the economic provision of laboratory services. Three different kinds of the analytical laboratories can be organized, to fulfil differing requirements of chemical analysis as required within a given country.

The first laboratory is a simple service laboratory for the determination of the main chemical components (and some specially interesting impurities) of bauxite and other sedimentary rocks by simple, fast, reproducible and accurate methods. Productivity and efficiency of qualified workers are very important.

The laboratory requires:

(a) X-ray fluorescence spectrometry;

(b) Automated atomic absorption spectrometry (AAS);

(c) The combination of different nuclear, thermometric and potentiometric methods for analyses;

(d) Classical wet chemical methods.

The second type of laboratory is a control and analysis laboratory supporting bauxite mining and technological operations. The laboratory must perform all the functions of the laboratory described above plus the chemical analysis of the aluminate liquor and that of the red mud.

The laboratory requires:

(a) 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) A combination of microprocessor-controlled AAS and X-ray diffractometric methods, as well as thermometric, potentiometric, spectrophotometric and some classical wet chemical methods.

The third type of laboratory is a research laboratory for mining and alumina production which will fulfil the functions of the other two and also provide analysis of all relevant components of the raw materials, products and by-products of alumina technology. In addition some research and development should also be carried out in the field of analytical chemistry in order to develop methods for monitoring and control.

The laboratory will make use of a 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 three different laboratories can be developed step by step or established at once according to the alumina production requirements. In the case of gradual development the reorganization of the staff of the laboratory is important.

Technological tests for bauxite processing

The methods and equipment needed are determined by the data needed. The main groups of the technological tests are for:

Crushing and grinding
Pre-desilication
Digestion
Red mud settling
Determination of the effect of contaminants

All of these relate only to high-grade bauxite as low-grade bauxite can not be economically used in the Bayer process. Unfortunately, the commercial availability of technological apparatus that is needed is quite limited. (For example only a few laboratory autoclaves are available.) Therefore, equipment was developed by aluminium companies.

The building block principle

It is reasonable to establish the bauxite laboratories step by step, using the building block principle. An example of a very flexible layout allowing stepwise enlargement of the laboratory from the simplest type to higher levels is shown in figure 3. The basic sketch of the goelogical exploration laboratory, based on the classical wet analytical method, using flame photometry and spectral photometry only, has a central entrance hall along the length of the building. Thus, enlargement is easy by adding further rooms at each end. The simple laboratory may be upgraded to a bauxite testing laboratory, suitable for technological investigations, by attaching two rooms at the 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.

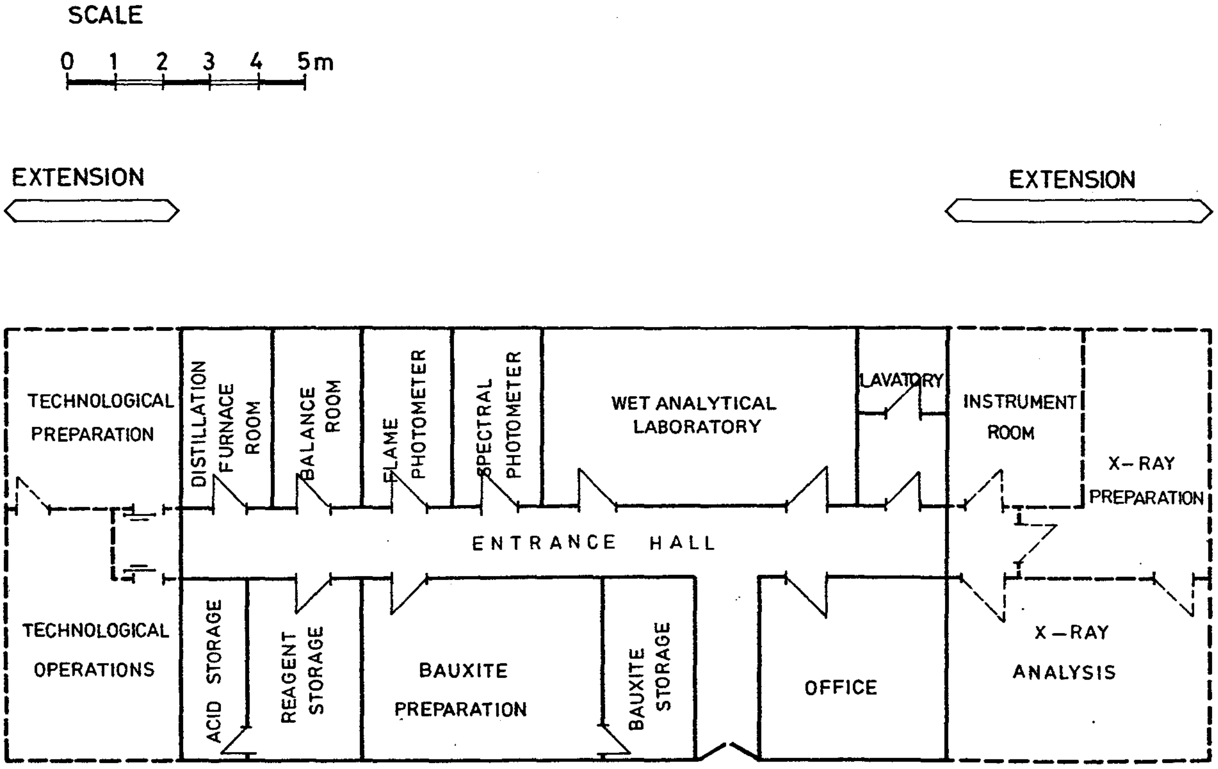
Setting up a pilot plant

Although certain units needed for the Bayer process can be modelled fairly well, the significance of pilot-plant investigations is increasing. In new plants which are now designed with greater and greater capacities, components of the process line must be correctly sized. Also, when using new deposits, unknown ores have to be processed.

These difficulties can be reduced by running pilot plants. Pilot plants are also suitable for designing the start-up of a new plant, and provide economical training. For example, an aluminium company designed an alumina plant of 1 million tonnes per year capacity, located on the bauxite deposit at Gove (Australia), using the experience gained 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 1:1,000.

However, as constructing and operating a pilot plant is fairly expensive, in critical cases in order to save time, the parameters of the plant may be determined in the laboratory and excess capacities of machinery and equipment designed into the process for the critical places.

Figure 3. Typical layout of an A level laboratory, showing possible extensions



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 one thousand times larger plant machinery and equipment. This includes investigations of processes such as fluidization, crushing, grinding and digestion. However, in many cases problems are met when switching from laboratory to pilot plant. These problems become even more pronounced when transferring the results to plant scale. In these cases laboratory results can be evaluated only as reflecting trends. 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 are suitable for process control in a plant or for determining the optimum parameters of a new project. Figure 4 shows two methods for optimization of plant design. In part A of the figure the test material is submitted to laboratory and pilot plant examinations for 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 differences among the results of laboratory, pilot-plant and large-scale experiments can be eliminated by means of modelling.

Although a pilot plant is operated in a continuous closed circuit it is not a "mini" Bayer plant because of the size of the equipment used. The main purpose of the pilot plant is to test the critical stages of the whole process and to examine newly introduced equipment. Thus there are two types of pilot plants, a pilot plant characterized by the ability to vary a wide range of parameters (such as temperature, holding time, and capacity) and a pilot plant where such flexibility is more restricted. In developing countries the latter type is more reasonable due to cost.

A pilot plant connected to a working industrial plant should preferably form a "by-pass" to permit the examination of critical stages in the industrial plant.

A closed circuit is ideal for the study of the effects of the enrichment of contaminants. Pilot plants should be designed individually for local requirements.

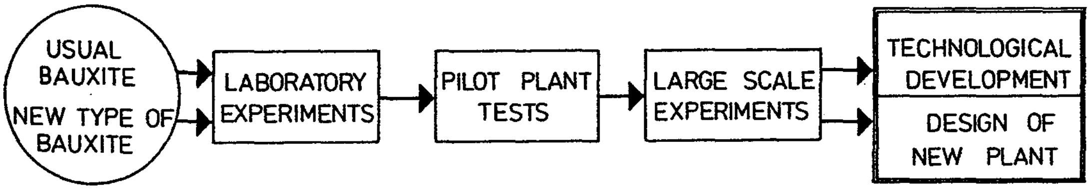
Process control analyses

The analytical part of the bauxite laboratory must be available for large-laboratory and pilot-plant investigations if these are located at the same site. As bauxite testing laboratories are usually extended step by step it is advisable to provide for future requirements.

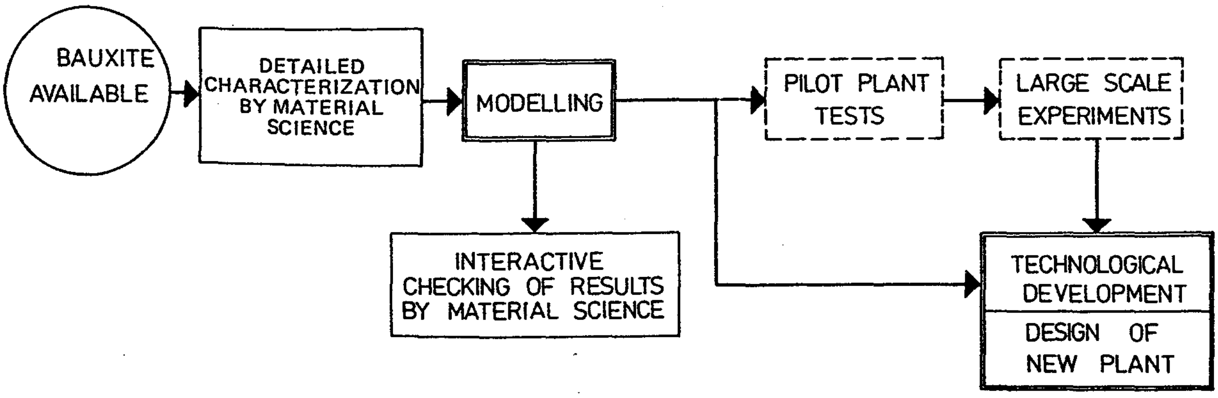
If the pilot plant is planned to be set up near an existing alumina plant then process control analyses should be made at that time in the industrial process control laboratory. Furthermore, process control instruments should be pretested in pilot plants.

Figure 4. Schematic diagram of plant design

A. Former method



B. Current method



IV. Units of a bauxite testing laboratory

This chapter is a review of the functions, principles, equipment and staff requirements in the main areas of the investigations carried out in a bauxite testing laboratory. Details of room requirements will not be given as these vary according to the tasks and conditions. Some examples will be given later, using tables and floor-plans.

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

Chemical analysis
X-ray investigations
Technological testing and simulation
Physical and physico-chemical investigations (including mineralogical and texture analysis)

In order to accomplish these tasks, it is usually necessary to have a storage room for samples, a storage room for reagents, a sample preparation room, a balance room, a distillation and furnace room, a wet chemical analysis laboratory, rooms for spectrophotometry and atomic absorption and flame spectrometry, a laboratory for other analytical instruments, an X-ray laboratory (sample preparation, XRF, XRD), a preparatory room for technological experiments, a laboratory for technological operations (rooms for digestion and settling, for precipitation, calcination, and purification of aluminate solutions), a laboratory for physical investigations, a room for thermogravimetry, an infrared spectrometry room, rooms for the electron beam laboratory (transmission electron microscope, scanning electron microscope, electron probe microanalyser), a data processing and documentation room, a library, an office, and communal rooms.

Dividing a laboratory into several rooms is necessary for equipment protection, hazard isolation (acidic atmosphere), or regulatory protection (X-rays and electron beam). On the other hand, pieces of similar equipment may be concentrated together independently from the material being examined, making their operation more efficient.

Annexes I to XII give details of scientific and technical procedures used in bauxite analysis. Annex XIII provides selected methods of chemical analysis. Annex XIV provides details of a laboratory training programme. Annex XV is about the technological evaluation of bauxite leading to the determination of plant design parameters.

Chemical analytical laboratory

Chemical analysis may be divided here into sample preparation and storage, the analytical procedure itself and data evaluation.

Sample preparation, analysing and data handling units must be separately organized for each level. Their organization and design will be different according to the needs and objectives. In this chapter these units will be discussed in detail.

Sample preparation and storage

Tests made on a sample of a certain amount of material are intended to yield information about the entire material. Consequently, the composition of the sample, within prescribed limits of error, must represent that of the whole. The accepted method of sampling is generally described in standard specifications, such as [9]. Certain deviations may also occur from either well-established experience or a trade contract.

The samples may be solid, fluid or slurries (red mud slurry, alumina hydrate slurry). These three types of samples must be prepared for analysis in different ways to preserve the representative character of the sample. The preparation starts when the material is crushed, reduced in mass, dried, if necessary, and packed. For chemical analysis the sample used is that obtained after quartering and averaging. A separate sample has to be taken for the determination of moisture. This operation is difficult because the sample must not lose any of its original moisture content during the preparation.

The sample may change physically or chemically during storage. By adequate preparation those changes can be avoided or reduced to a negligible level. In the alumina industry the change of moisture content is the one most likely to be reckoned with. For the purpose of chemical analysis and to ensure weight constancy the dried and properly powdered sample should be brought to equilibrium with the humidity of the environment as minimum change in the state occurs if the sample is in equilibrium with its environment. This is ensured by spreading the sample in a thin layer for a few hours.

Similarly, chemical changes may occur under the effect of the environment. For example, during the drying of the causticized red mud sample containing unreacted $Ca(OH)_2$, the composition will definitely change. In the presence of CO_2 from the air, $Ca(OH)_2$ forms $CaCO_3$. Thus, the original $Ca(OH)_2$ and CO_3^{-2} content of the sample cannot be determined. When the relationship of SiO_2, Al_2O_3 and Na_2O has to be determined in red mud, the sample may be dried straightforwardly. If the amount of unreacted $Ca(OH)_2$ retained in the red mud has to be determined the sample should be isolated from the CO_2 of the air.

A problem of preparation is what size the particles in the sample should be reduced to. No definite answer can be given to this question. The sample dissolves at an instantaneous rate which varies with the instantaneous dispersity of the sample. Therefore, samples slow to dissolve should be ground finer than faster dissolving samples. Generally, the harder a sample and the higher its mechanical strength the slower it dissolves. Fine grains may contain foreign material worn off from the grinding equipment. Thus, with respect to the question of how fine a sample is needed, a compromise must be reached. The sample should be pulverized 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 (μm):

Fairly reactive gibbsitic bauxites	150
Mixed-typed gibbsitic-boehmitic bauxites and red mud	100
Hard diasporic bauxites	50

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 so that the entire sample passes through a prescribed mesh aperture.

The prepared sample should be placed into a proper air-tight container. The label should state the material, place and date of origin, the amount of the sample, code number of the sample, other data and the signature of the sampler. For actual working analysis another coded container is used.

The International Organization for Standardization (ISO) specifies how to take a representative sample of alumina. 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.

Liquid samples may be of purely liquid phase or may contain a considerable amount of solids.

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

During the analysis of liquid samples (provided that they were extracted from a system of metastable state), troubles frequently arise from the instability of the sample. The decomposition must be prevented by dilution, the maintaining of a proper temperature or by simply carrying out the analysis before the sample decomposes. In any case, the procedure preventing the decomposition should be efficient so that the representative character of the sample should not be affected.

For 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.

Generally, the liquid samples do not require further treatment; however, dilution or the addition of some special reagent may be needed.

Solid samples can be analysed immediately by nuclear and X-ray spectrometric methods. For X-ray methods fusion of the material is desirable to eliminate the matrix effects.

Solid samples are generally dissolved before being analysed. Sometimes, the dissolution process is preceded by an alkaline fusion. When dissolving the samples preference should be given to methods which introduce the least amount of foreign substance into the solution.

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 for 3 h. If the sample is heated at 1,100° C for 2 h, the loss in weight corresponds to the water and CO_2 content; however, some side-effects originate 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. A very small (maximum 100 mg) sample can be heated under automatic control and the weight-loss is displayed as a percentage.

The ordinary laboratory facilities required are:

Turbomill for sample pulverization
Air jet sieve
Laboratory disc mill
Metal trays
Electric drying oven
Electric furnace
Melting apparatus
Micro thermobalance

Electronic analytical balance
Sampler containers
Storing racks with a one-year capacity
Computer terminal with keyboard and interfaces to the balances

Special equipment is described in table 5.

TABLE 5. SPECIAL INSTRUMENTS FOR THE CHEMICAL ANALYTICAL LABORATORY

Kind of instrument and description of features	*Approximate price (1980 dollars)*
Spectrophotometer	
Single-beam optical system, automatic sample changer, data-handling system	5 000-10 000
Thermometric titrator	
Automatic thermometric end-point detection	6 000
pH meter	
Digital display	1 000-2 000
Sequential fusion apparatus	
Time-selected fusion of 6 samples in Pt crucible at 1 200° C	2 000
Bauxite analyser	
Automatic sample and data handling	20 000

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

Laboratories for chemical analysis

The principles of the different methods are introduced here; details are in the annexes. Computer- and microprocessor-operated instruments are now dominant in instrumental analytical chemistry. These instruments can produce savings in labour and decrease of errors.

AAS and FS laboratories

The atomic absorption spectrometry analytical technique developed in the late 1950s was adopted quite rapidly by the alumina industry because of the relatively low expense and easy sample preparation (in some cases solutions can be used directly without any other procedure). The technique can be applied if the analytical problems need otherwise complicated procedures (i.e. determination of Mg, Zn, Na etc.).

The AAS analytical technique may be used for the determination of the main components of bauxite and red mud with high reproducibility and accuracy and for the determination of a specially interesting trace element in bauxite, red mud or alumina samples.

Principles of atomic absorption analysis are discussed in annex I.

Equipment

The following equipment is required:

Sequential fusion apparatus
PTFE lined steel bombs for the dissolution of alumina
Equipment for sample treatment and dilution
AAS equipment and accessories
ETA system
Compressed air, acetylene, nitrous oxide and argon supplies
Fume hood
Data processing system

Staff requirements

Usually, a university-level chemist and two to three technicians are able to analyse 2,000 to 2,500 bauxite samples in one shift. With a modern AAS system, using external assistance, the staff may consist of only trained technicians.

Table 6 shows the approximate prices of two types of absorption spectrophotometer.

TABLE 6. ATOMIC ABSORPTION SPECTROPHOTOMETERS FOR THE BAUXITE TESTING LABORATORY

Description	*Approximate price[a] (1980 dollars)*
Single-beam optical system with microprocessor data handling, electronic gas control, automatic sampler changer	28 000
Double-beam optical system, microprocessor, automatic gas control, automatic sample changer	25 000-35 000

[a] Accessories (e.g. lamps, gas regulators etc.) are not included in the price.

Wet chemical analysis laboratory

The function of the wet chemical analysis laboratory is to determine components of bauxite, red mud and alumina, to determine the main components of aluminate liquor, to calibrate (preparation of standard solutions, determination of the concentration of the reference solution etc.), and to perform special investigations (e.g. reactive SiO_2, available Al_2O_3 etc.).

The principles of wet chemical analysis applied to the alumina industry are discussed in annex II.

The structure and performance of the wet chemical laboratory depends on the tasks and the levels of the other instrumental laboratories. If all the other analytical routine investigations are carried out by other well-organized groups only the standardization and very special analytical tasks are performed in this laboratory. In that case a limited number of highly trained chemists is required. However, all the analytical chemistry services can be performed by the classical wet chemical methods if justified by the level of technical development or by financial considerations.

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.

The final detection of the components to be analysed may also be performed by spectrophotometric or potentiometric methods. These methods, however, are not purely classical wet chemical methods in the sense discussed here.

Equipment needed includes common glass beakers, volumetric flasks, pipettes, burettes etc., depending on the capacity of the laboratory. Sequential fusion equipment is very useful because of its high productivity and will be described later.

Generally, the leader of the laboratory must be a chemist with experience in the analytical chemistry of alumina industry. Technicians and laboratory assistants and unskilled workers comprise the rest of the staff.

Instrument laboratory

The instrument laboratory provides routine identifications required by the wet chemical laboratory, special investigations in the field of analytical chemistry of the alumina production, and research and development for the application of monitors in the testing or technological process.

The scientific principles of the instruments used in this laboratory are discussed in annex III.

Several instruments are needed in order to successfully carry out analysis. These instruments are generally installed in the same area, the instrumental laboratory. The equipment can be divided into spectrophotometers, electroanalytical instruments and other special devices. The equipment needed includes: a spectrophotometer, a precision pH meter, an automatic potentiometric titrator, a conductivity meter, polarograph (with d.c., a.c. and pulse mode) and a thermometric titrator. Some automatic titrators are described in table 7.

TABLE 7. AUTOMATIC TITRATORS

Type	*Used for determination of*	*Approximate price (1980 dollars)*
Potentiometric[a]	S, Cl, Al	6 000-10 000
Thermometric	Caustic Na_2O, total Na_2O, Na_2CO_3, Al_2O_3, $FeCO_3$, FeS_2	6 000-10 000
Mhometer	Caustic Na_2O, Al_2O_3, caustic molar ratio, total N_2O	1 500

[a] Some models incorporate other types of titration, e.g. voltametric (for Zn), colorimetric (Si, P, V, Cr, Mo) and thermometric.

For routine analytical tasks, an analyst specialized in instrumental analytical chemistry should supervise the work of a staff of three or four technicians. For research and development, experts in instrumental methods are needed.

Neutron activation laboratory

The neutron activation laboratory is used for the determination of some main components of bauxite (and red mud). Annex IV discusses the scientific basis of neutron activation analysis.

An automatic bauxite analyser (ABA) is needed; costs range from approximately $20,000 to $30,000.

The ABA instrument can be operated by unskilled workers who have undergone brief training.

Data processing unit

The functions of the data processing unit include the handling, formatting, reducing and storing of analytical data originated from different equipment; and direct control over laboratory operation.

The productivity and the objectivity of a laboratory can be increased by a data handling system. Human resources may be more efficiently utilized by use of a computerized data handling unit.

Modern laboratory instruments have (generally digital) outputs that can be connected to a minicomputer terminal. Some instruments have self-contained microprocessors.

The appropriate data handling unit is chosen after reviewing technical and financial considerations. Some automatic instruments equipped with a microprocessor do not require a separate on-line data centre even for producing laboratory reports and storing data. In such cases it is better to use a punched tape, tape cartridge, or 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 used.

The organization of the analytical laboratory into an integrated data processing system is becoming more and more common. This integration provides vast possibilities for increasing the productivity of the analytical work.

The equipment required varies with the needs of the laboratory. Therefore, first of all, the laboratory planner should employ an expert in laboratory data processing. The expert will be able to suggest equipment options with due regard to laboratory goals and purposes.

No extra personnel are needed for data processing but special training will be required for current staff.

X-ray laboratory

In bauxite laboratories, X-rays are generally used for X-ray fluorescence spectrometry (XRF) and X-ray diffraction analysis (XRD).

XRF is an analytical technique used to determine the elemental constitution of a substance, both qualitatively and quantitatively. In this capacity it is an up-to-date instrumental substitute for wet chemical analysis, and it can be highly automated. However, XRF is not an absolute determination procedure and thus requires calibration by standards which may be purchased or prepared. Alternative instrumental methods are atomic absorption spectrometry (AAS), thermometric analysis, emission spectrometry, neutron activation analysis or mass spectrometry. Each has its particular advantages and disadvantages. Although XRF could have been discussed along with the various other methods of modern chemical analysis, the proposal for a separate X-ray unit has mainly practical reasons.

X-ray fluorescence spectrometry can be used to give an overall check of the chemical elements where they are present in an unknown sample at a concentration of greater than 0.01%. In bauxite exploration this is used to define major impurities to be reckoned with in new resources. In alumina technology XRF is usually used for trouble shooting in the investigation of abnormal deposits, intermediate products or 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, it seems to be economical in 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 easily and quickly, and with high confidence. In alumina plants, regular analysis of red mud samples is the most advantageous application for XRF, although there are many other advantages.

XRD is used to qualitatively and quantitatively investigate the crystalline components of a substance. It is a complementary technique to elemental analysis methods.

As the mineral forms of the aluminium, iron and silicon bearing compounds are decisive in determining the parameters of processing, XRD is indispensible in assessing technical criteria such as new plant design. During the operation of plants the efficiency of steps such as desilication, digestion and separation, can be controlled by XRD.

XRD in itself can yield highly sensitive reliable results in routine qualitative analysis. However, quantitative mineralogical phase analysis requires a knowledge of the chemical constitution. Alternative and complementary tools for obtaining mineralogical information are infrared spectrometry, thermal analysis and microscopic methods. Thus, XRD should be considered as part of the group of mineralogical analysis. A combined XRF and XRD analysis allows 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 use several complementary methods, although the problems faced are similar when highly variable, poorly crystallized components have to be dealt with.

XRF spectrometers and XRD systems can be installed as independent equipment in separated laboratories; however, both need X-ray sources which make a hazardous environment. Combination of XRF and XRD measurements under common computer control can lead to enhanced information obtained from a single sample.

Sample preparation

XRF

The aim of sample preparation for fluorescence measurements is to assure adequate homogeneity in a geometrical form for the introduction of the sample into the instrument.

An XRF preparation room needs a set of mortars and pestles, a set of sieves, a pulverizer or grinding mill, a balance, a hydraulic press with mould, and means for preparing "beads". The "beads" may be prepared in a furnace with heat regulation into which the crucibles are manually placed. After a predetermined heat programme the melted samples are cooled and solidified to the required shape in rings or moulds. On the other extreme are automated bead machines. Aids for bead preparation of intermediate automation and price are also available.

XRD

For X-ray powder diffractometry, samples of 1 to 1.5 g mass with $< 10\,\mu m$ particle size are necessary. Aluminium or plastic sheet sample holders are used with about 10 × 15 mm windows which should be filled by the powder to give 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 mesurements it may be necessary to mix the sample with a known amount of a reference material. Frequently used references are alpha-Al_2O_3 (corundum) and KCl.

In addition to a balance, a mill and a set of sieves and a manual press are the only equipment needed.

Measurements and data collection

Having prepared the samples the phenomena following the interaction with X-rays must be recorded and then evaluated. Annex V considers the apparatus used for the generation of X-ray analysis, and for the fluorescence and diffraction studies.

Equipment and staff for the X-ray laboratory

The X-ray laboratory is used to assist in geological survey work, mining activities and alumina plant control. Owing to the high investment costs which range from $80,000 to $540,000 (without building and related investments) as listed in table 8, 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 have one laboratory for several enterprises. The appraisal of geological samples can be extended to other ores and minerals. Many other industrial sectors of a country may also benefit from such a facility. Investigation of soils, cements, labour safety control in connection with silica and asbestos, and many branches of chemical, ceramic and dye-stuff industries need similar measurements. Thus a well-arranged central service can provide analyses for all. Further gradual expansion from a modest start is also a cost-effective possibility.

TABLE 8. AVERAGE INVESTMENT COSTS OF X-RAY EQUIPMENT

Equipment	*Approximate price (1980 dollars)*
Generator and diffractometer assembly	80 000
Generator and sequential spectrometer	110 000
Sample preparation laboratory for XRF	32 000
Closed-loop water cooling	12 000
Updating of diffractometer assembly by micro-processor control	30 000
Generator and computer-controlled diffractometer	160 000
Simultaneous spectrometer for 20 channels under computer control	350 000

The size of the staff of the laboratory depends on the tasks required. The reasonable minimum number of personnel is two, provided maintenance and repair service is available externally. The team of two should consist of a (research) chemist and a technician trained for the operation of complicated instrumentation. For external maintenance and repair, the laboratory should have its own engineer or technician for the decoding of circuit diagrams and for locating or repairing faulty components in modern semiconductor electronics.

Full exploitation of the capacity of an up-to-date diffractometer or spectrometer justifies the employment of four to five persons (two scientists and two to three technicians). If both X-ray methods are fully used, 10 persons may be needed.

Layout of an X-ray laboratory

A typical layout is shown in figure 5. The sample preparation room is approximately 12 m^2 in area if only XRD or XRF are used and 20 m^2, or preferably two separate rooms of 12 m^2 each, if both are used. An instrument room approximately 15 m^2 in area for the first unit and about 10 m^2 for each additional unit is required. The water cooling apparatus must be placed in a separate room of at least 8 m^2, 3 to 5 m from the measuring units and working places. Working rooms for the staff should be, depending on national standards, about 10 m^2 in area for each scientist and about 6 m^2 for each technician. A stockroom for spare parts and other storage of about 4 m^2 will be required.

Technological testing and simulation laboratory

In the technological testing laboratory, the operations of the Bayer process are simulated under laboratory conditions. The process-modelling units serve to simulate different stages of the Bayer process: pre-desilication, digestion, red mud separation and washing; precipitation of $Al(OH)_3$ from an aluminate solution rich in contaminants; and calcination (including physico-chemical control of alumina). The laboratory is also used to evaluate the effects of bauxite contaminants.

A simple technological laboratory is shown in figure 6. It consists of three basic units: sample preparation, technological examination and sample storage. This laboratory can only be operated in close co-operation with other laboratories (chemical, physico-chemical, mineralogical).

Figure 5. A typical layout for an X-ray laboratory

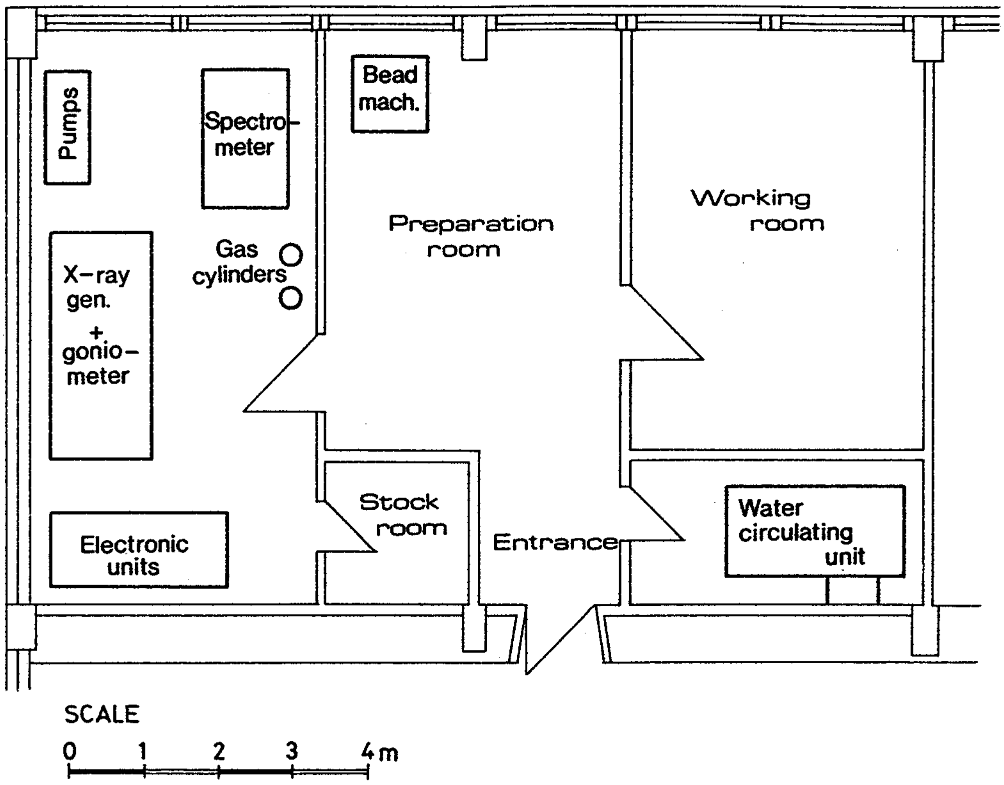
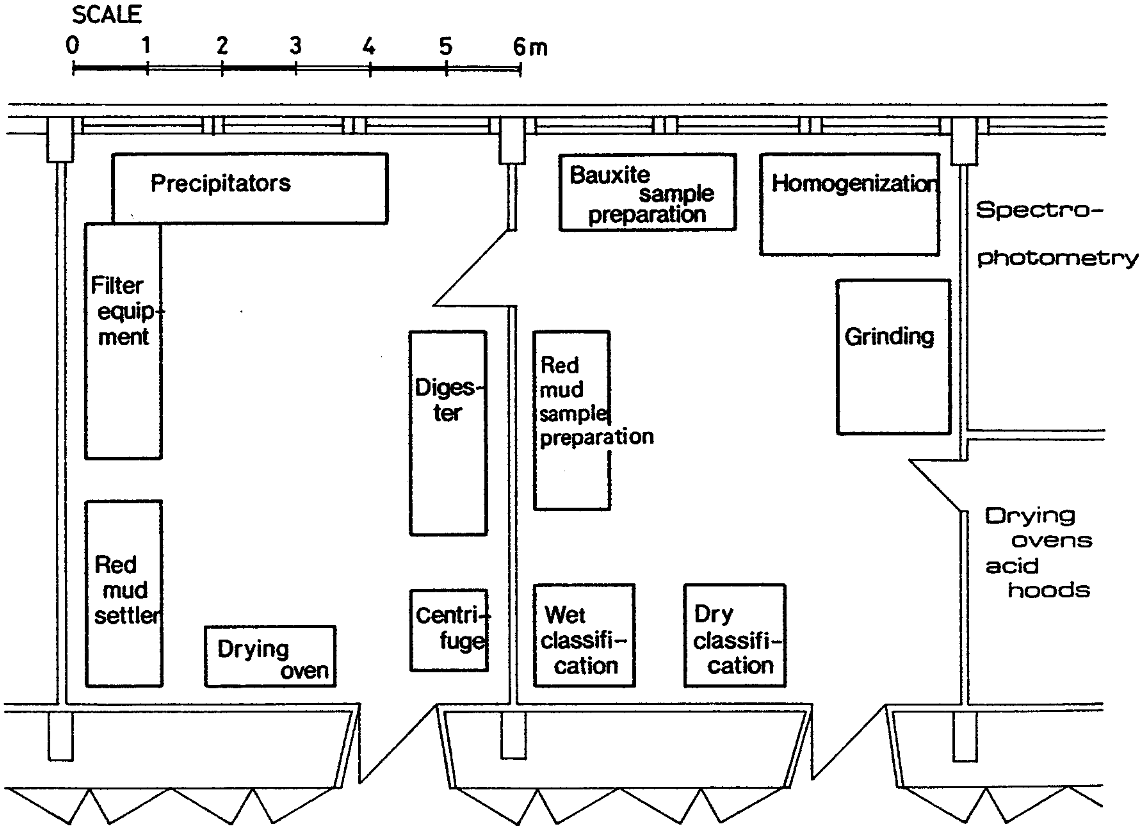


Figure 6. A typical layout for the technological testing and simulation laboratory



Sample preparation

The function of the sample preparation laboratory is to do preparatory work for technological tests, including preparation of representative samples, coding and storage of samples, drying, grinding, weighing of bauxite samples, preparation of aluminate solutions, and fast analysis of aluminate solutions. The laboratory also tests grindability and ore beneficiation. More details about the operation of this laboratory are given in annex VI.

The equipment needed includes:

- Jaw crushers
- Drum crushers
- Wet- and dry-grinding mills
- Separators
- Dry and wet sieves
- Sample reducers and splitters
- Vibration mill
- Cyclones
- Drying ovens
- Analytical balances
- Scales
- Sample containers
- Tanks for solutions with stirrers
- Thermometric titrator
- Potentiometric titrator

Tables 9-16 show typical equipment specifications and approximate prices.

A staff of two technicians and three to four skilled workers is needed.

Laboratory for digestion and settling tests ("red lab")

The "red lab" is used for the determination of pre-desilication parameters, the selection of digestion parameters and the determination of settling and washing characteristics of red mud.

The principles of operation of the red lab are discussed in annex VII.

The equipment required consists of:

- Thermostats, and a series of tanks equipped with mechanical stirrers (not commercially available)
- Autoclaves (mechanically stirred, with temperature control up to at least 260° C; cost, \$5,000-\$8,000)
- Digester assembly with 6-10 bomb autoclaves (150-250 ml each) rotated in a temperature control (up to 260° C) oil or melted-salt bath
- Centrifuges for red mud separation (size corresponding to autoclave bombs) (see table 16)
- Filter presses (see table 17)
- Vacuum filters (see table 17)
- Thermostatic settling tubes
- Soft γ-ray absorption apparatus for settling experiments (cost, about \$50,000)

TABLE 9. JAW AND DRUM CRUSHERS

Type	*Specifications*	*Approximate price (1980 dollars)*
Jaw Crusher	Feed opening: 100 × 100 mm Capacity: 150-700 kg/h Product size: 1-15 mm Push-plate: tungsten-carbide + cobalt Cr-Ni steel	7 000 7 000 (additional) 700 (additional)
Jaw Crusher	Feed opening: 125 × 150 mm Capacity: 300 kg/h Product size: 10-30 mm Push-plate: Manganese steel	7 000
Drum Crusher	Drum diameter and width: 250 × 150 mm Capacity: 200-1 200 kg/h Maximum feed-piece size: 12 mm Product size: 0.2-3 mm Drum casting: hard steel (550 Brinell)	10 000
Drum Crusher	Drum diameter and width: 200 × 160 mm Capacity: 200-1 200 kg/h Maximum feed-piece size: 12 mm Product size: 0.2-3 mm Drum casting: hard steel, stainless steel or hard porcelain	20 000
Roll Crusher	Drum diameter and width: 250 × 150 mm Capacity: 2 000 kg/h Maximum feed-piece size: 10 mm Product size: 0.25 mm	10 000

TABLE 10. LABORATORY WET AND DRY GRINDING MILLS

Specifications	*Approximate price (1980 dollars)*
Mill	7 000
Feed charge: 1-20 kg	
Product size: 45 μm (at 5 Mohs hardness)	
Mill	7 000
Capacity: 100 kg/h	
Product size: 5-100 μm (at 5 Mohs hardness)	
Grinds and separates	
Mill	10 000
Capacity: 500 kg/h	
Product grain size: 1 μm to 2.5 mm	
Teeth, perforated steel grinding discs or simple corundum discs	
Ball mill	12 000
Procelain or stainlesss-steel grinding containers of 1-25 l capacity	
Product size: 30-100 μm	
Autostop at 100 rpm for Bond index determination	
Jar mill with rolls	8 000
Ceramic and steel grinding containers of 6-10 l capacity	
Product size: 30-100 μm	
Separator mill	25 000
Capacity: 1-2 kg/h	
Cut off: 1 to 100 μm	
Separator mill	25 000
Capacity: 50 kg/h	
Cut off: 0.1 to 10 mm	
Universal mill	1 000
For regrinding of red mud or other dry minerals	

TABLE 11. MAGNETIC SEPARATORS

Type and specifications	*Approximate price (1980 dollars)*
Wet magnetic separator	10 000
Feed charge: 200 g	
Feed grain size 3 μm to 3 mm	
Dry disc magnetic separator	10 000
Capacity: 20-50 kg/h (continuous or batch operation)	
Magnetic filter separator	10 000
Fully automatized	
Feed: maximum 100 μm, minimum below 3 m	
Capacity up to 5 000 kg/h	
Wiefley-type concentrating table	1 500
Table size: 1 000 × 450 mm	
Capacity: 25-75 kg/h	

TABLE 12. DRY AND WET SIEVES FOR ANALYTICAL PURPOSES

Type and specifications	*Approximate price (1980 dollars)*
Air jet sieve	11 000
Includes cyclone, small filter, fine filter, vacuum cleaner, normal and micro sieve series	
Relative error: less than 1%	
Measuring time: 2-3 min per sieve	
Cyclone: 15 W high-pressure type	
Small filter: 1 m	
Fine filter: 300 mm diameter	
Vaccum cleaner: 800 W	
Normal series: 32 to 250 μm, 200 mm diameter	
Micro series: 10 to 25 μm, 75 mm diameter	
Wet screen with three jetrings	10 500
Relative error: < 1.5%	
Measuring time: 6-60 min for 4 sieves	
Normal and micro series sieves	
Wet sieve	11 500
Measuring time: 3-30 min for 6 sieves	
Relative error: < 1.5%	
Sieve diameter: 203 mm	
Screen: 20-100 μm	
Sieve cleaners	2 200
Ultrasonic bath	2 100

TABLE 13. SAMPLE REDUCERS AND SPLITTERS

Type and specifications	*Approximate price (1980 dollars)*
Laboratory size reducer	1 500
Volume: 30 l	
Sampling ratio: 1/10 to 1/200	
Speed: 1-60 min per charge	
Pilot-plant size reducer	3 500
Equipped with ball mill	
Sampling ratio: 1/10 to 1/80	
Capacity: 75-750 kg/h	
Feed size: 60-120 mm	
Electromagnetic laboratory splitter	1 500
Sample receiver: 10 × 500 cm^3	
Minimum sample size: 20 cm^3	
Maximum particle size: 6 mm	
Universal sample splitters	
Pilot-plant size, up to 250 kg capacity	200-850
Laboratory size, 50 g-25 kg	750
Hand size, maximum 1.8 kg	250
Automatic samplers	1 100-5 700
Feed size: 10-100 mm	
Volume: 50-80 l	
Splitters	1 400-4 000
Feed size: 10-800 mm	
Volume: 50-250 l	
Sampling ratio: 1/8 to 1/64	

TABLE 14. VIBRATION MILLS AND CYCLONES

Type and specifications	*Approximate price (1980 dollars)*
Vibration cup mill	3 400
Dry or wet	
Cycle: 1-3 min	
Feed size: 7-12 mm	
Cup volume: 50, 100, 250 ml	
Cup material: hardened steel	1 600 additional
tungsten carbide	15 000 additional
agate	3 500 additional
Pilot-plant scale wet-grinding mill	4 000
Capacity: 10 l	
Product size: 1 μm	
Balls: Mn-steel or ceramic	
Cyclone	5 000
Separation: 10 100 μm (optimum 30 μm)	
Capacity: 2-15 m^3/h	
Pressure gauge: 2 MPa	
Laboratory testing cyclone	8 000
Separation: 1-250 μm	

TABLE 15. FURNACE, DRYING OVENS, WATER STILL, BALANCES AND SCALES

Type and specifications	*Approximate price (1980 dollars)*
Furnace	
Temperature: 1 600° C max.	25 000
Dimensions: 170 × 220 × 535 mm	
Input power: 8.5 kW	
Drying ovens	
Temperature: 300° C max.	4 000
Dimensions: 600 × 600 × 580 mm	
Input power: 2.7 kW	
Temperature: 250° C max.	6 000
Dimensions: 1 024 × 1 350 × 580 mm	
Input power: 5.0 kW	
Temperature: 200° C max.	1 800
Dimensions: 1 000 × 600 × 520 mm	
Input power: 4 kW	
Water still	10 000
Double distillation	
Capacity: 8 l/h	
Input power: 11.5 kW	
Analytical balances	
Electronic; precision 0.1-0.01 mg	1 000-4 000
Mechanical; precision 0.1-0.01 mg	2 000-8 000
Scales	
Electronic; capacity 15-60 kg, precision 0.01-1 g	3 000-4 000
Mechanical; capacity 3 kg, precision 1 g	1 000

TABLE 16. STIRRED TANKS, THICKENERS, CENTRIFUGES

Type and specifications	*Approximate price (1980 dollars)*
Stirred tank	
Diameter, 1 800 mm; height, 1 800 mm	2 500
Diameter, 1 500 mm; height, 1 600 mm	9 000
Diameter, 2 100 mm; height, 2 100 mm	4 500
Agita tank	10 000
Diameter, 900 mm; height, 900 mm	
Attrition machine (2 cells)	5 000
Size: 250 × 250 mm	
Thickener	4 500
Diameter, 900 mm; height, 750 mm; pumps (for underflow)	
Four stage agitator unit	6 500
With 4 × 22 l cells propeller agitators	
Centrifuges	
Speed: 4 000 rpm	20 000
Volume: 6 × 1 000 ml or 4 × 1 500 ml	
Input power: 4.5 kW	
Speed: 1 700 rpm	7 000
Volume: 6 × 500 ml	
Input power: 1.2 kW	
Spiral decanter type for pilot plant capacity 0.5-1.5 m^3/h	50 000

TABLE 17. PRESSURE AND VACUUM FILTERS

Type and specifications	*Approximate price (1980 dollars)*
Centrifuge	7 000
Heatable to 100° C	
Speed: variable to 16 000 rpm	
Capacity: 10 l/h	
Rotor: porcelain	
Filter paper: special	
Filter press	
Laboratory size; counterbalanced	2 000
Capacity: 22 l	
Diameter: 300 mm	
Laboratory size; complete with layers, precoat, compressor, pump etc.	20 000
Heatable to 134° C	
Pilot-plant size	50 000
Filtering area: 5 m^2	
Discharge: automatic	
Filling pressure: 10 MPa	
Pressure gauge: 36 MPa	
Disc filter	
Laboratory size; double-disc	25 000
Area: 0.4 m^2	
Pilot-plant size	20 000
Area: 1 m^2	
Drum filter	
Laboratory size; with agitator, scraper, washing nozzles and belt	20 000
Material: Cr-Ni-Mo steel	
Filter cycle: 0.2-1.8 rpm	
Area: 0.25 m^2	
Pilot-plant size	20 000
Area: 1 m^2	
Pan filter	1 000
Laboratory size, 12.5 × 12.5 cm	

Laboratory for precipitation and calcination tests ("white lab")

The "white lab" determines the parameters of control filtration and the parameters of precipitation (yield and granulometry of $Al(OH)_3$ as a function of holding time, seed quality and quantity, temperature and caustic soda concentration) and conducts filtrability and calcination tests for $Al(OH)_3$.

The equipment needed includes:

A pressure filter (for control filtration) (see table 17)
Air-agitated precipitator tanks of about 20 litres (see table 16)
Mechanically agitated precipitators, of 1 to 3 litres (see table 19)
A vacuum filter for hydrate filtration (see table 17)
A furnace with heat control up to 1,400° C (see table 15)

The staff requirements are one engineer, one technician and one assistant.

Bauxite contaminant laboratory

The laboratory for bauxite contaminants provides dissolving and accumulation tests, analyses of the effect of contaminants in certain parts of the process, and elaboration of methods for recovery and removal.

The principles of bauxite contaminant studies are given in annex VIII.

No special equipment is necessary. In addition to standard laboratory equipment, a large-scale closed-circuit laboratory or pilot plant is required for studying equilibria and the enrichment process, as well as the recovery of V_2O_5 and Ga_2O_3.

Physico-chemical laboratory

The activities of the laboratory for physical and physico-chemical measurements are divided into the following four groups:

Simple physical and physico-chemical investigations
Thermogravimetry
Infrared (IR) spectrometry
Texture investigations (optical and electron-optical methods)

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

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.

Simple investigations

The function of the laboratory for simple physical and physico-chemical investigations is to:

(a) Supply physical and physico-chemical data during technological investigations;

(b) Provide 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.

The principles of these investigations are discussed in annex X.

The equipment required is:

An analytical balance (see table 15)
Two scales (see table 15)
A sieve set for wet sieving (see table 12)
A light microscope
An air-jet sieve (see table 12)
Pycnometers (see table 18)
A scanning photosedigraph (see table 18)
An instrument for measuring specific surface area (see table 19)
An instrument for measuring pore-size distribution (porosimeter; cost ranges from $10,000 to $50,000)

TABLE 18. INSTRUMENTS FOR GRAIN-SIZE DISTRIBUTION ANALYSIS AND SPECIAL INSTRUMENTS

Type and specifications	*Approximate price (1980 dollars)*
Scanning photosedigraph with built-in calculator	14 000
Grain-size range is 2-100 μm (in water) or to 500-600 μm in a higher viscosity medium; cuvette volume is 200 ml	
Plots the extinction curve and the calculated grain-size distribution curve	
Sedigraph particle-size analyser	29 000
Based on X-ray absorption; uses direct-mass weighing. Automatically plots grain-size distribution; cuvette volume is 5 ml; grain-size range is 0.1-100 μm	
Sedimentometer	3 000
Based on light absorption; cuvette volume is 10 ml; grain-size range 3-50 μm (in water) or 5-100 μm (in glycerine-water mixture); digital display	

Type and specifications	*Approximate price (1980 dollars)*
Automatic grain-size analyser Based on light absorption; particle size range 1 to 9 000 μm. Any fluid may be used as a carrier. Automatic background correction, data processing; result can be printed or plotted	40 000
Sedimentation balance Grain-size range: 1-150 μm Sample weight: 0.3-1.5 g Cuvette volume: 550 ml Maximum weight of sample: 200 mg	5 500
Pipette instrument Used if more than 50% of grains are less than 5 μm. In water, grain-size range is 0.05-5 μm and sample weight is minimum 2 g per 160 ml water	3 000
Calculator For data analysis	5 000
Auto pycnometer Maximum sample volume: 50 cm^3 Accuracy: ± 0.01 cm^3	10 500
Pycnometer Maximum sample volume: 90 cm^3 Accuracy: ± 0.1 cm^3	3 600
Zeta-potential analyser Range: 10 Ω-10 mΩ Sample volume: 110-145 ml Maximum solid content: 50%	7 500
Viscosimeter	
Built-in thermostat, 40° C to 100° C; automatic evaluation with microcomputer	25 000
External temperature control; manual data registration and evaluation	10 000

TABLE 19. COMPARISON OF RANGES AND PRICES OF GAS ABSORPTION INSTRUMENTS FOR MEASURING SPECIFIC SURFACE AREA

Measurement conditions	*Range of measurement (m^2/g)*	*Approximate price (1980 dollars)*
Static; N_2	0.1-1 000	4 000
Static; N_2; automatic	1-2 000	33 000
Static; Ar	0.05-150	9 000
Static	>0.001	29 000
Dynamic; N_2/He	>0.1	8 000

Thermogravimetry laboratory

The thermogravimetry laboratory provides a contribution to complex phase analysis technique (as a unit or as a complement to X-ray and IR), a study of bauxite roasting (for removal of contaminants) and a study of alumina calcination process (especially the effect of mineralizers).

The principles of thermogravimetry are discussed in annex X.

The specifications of a few available thermoanalytical instruments are given in table 20. Further information is provided in [10], [11] and [12].

The staff required is one graduate chemist or physicist.

TABLE 20. THERMOANALYTICAL INSTRUMENTS

Specifications	*Approximate price (1980 dollars)*
Range: 20-1 500° C Heating rate: 100° C/min Automatic base-line correction Differential scanning calorimeter (DSC) Microcomputer	50 000
Range: 20-1 000° C Heating rate: 0.1-320° C/min Sample weight: 5 mg-5 g Sensitivity: 10 μg	20 000
Range: 50-725° C Heating rate: 0.3-320° C/min Sensitivity: 0.1 mcal	25 000
Range: 20-1 000° C Heating rate: up to 100° C/min Weighing range: up to 150 mg Reproducibility: ± 1 mg	10 000
Range: −20 to 600° C Heating range: up to 100° C/min Reproducibility: ± 0.2° C	15 000
Range: −130° to 725° C Calorimetric sensitivity: 0.003 mcal/s Calorimetric precision: ±1% Calorimetric accuracy: ±0.2%	60 000
TG sensitivity: 500 μm/250 mm Sample weight: 1-100 mg DTG sensitivity: 500 μg min^{-1} 250 mm^{-1} 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 to 600° C	50 000
TG sensitivity: 20 mg/200 mm Sample weight: 10 mg to 10 g Accuracy: ± 0.2% Temperature range: 20-1 100° C Purge gas: air/inert	25 000

Infrared (IR) spectrometry laboratory

The infrared spectrometry lab provides information on mineralogical composition, crystallinity and stability. IR spectrometry combined with thermogravimetry can substitute for XRD in an A level laboratory or supplement and refine the results of other methods in an advanced C level laboratory.

The principles of IR spectrometry are discussed in annex XI.

The equipment needed varies with the sophistication of the laboratory. Specifications and prices of some IR spectrometers are shown in table 21. For field work, gibbsite, boehmite and kaolinite content can be determined by portable instruments. For rapid and reliable mineralogical analysis in A and B level laboratories, an automated analyser should be used. For C level laboratories a fully computerized system with data processing should be considered.

TABLE 21. SPECIFICATIONS AND PRICES OF TYPICAL INFRARED SPECTROMETERS

Specifications		*Approximate price (1980 dollars)*
Range (cm^{-1})	*Resolution*	
4 000-600	10 cm^{-1}/3 000 cm^{-1}	4 900
4 000-600	5 cm^{-1}/1 000 cm^{-1}	5 300
4 000-600	5 cm^{-1}/3 000 cm^{-1}	6 400
4 000-600	3 cm^{-1}/1 000 cm^{-1}	7 300
4 000-375	5 cm^{-1}/3 000 cm^{-1}	8 400
4 000-250	3 cm^{-1}/1 000 cm^{-1}	9 100
4 000-200[a]		19 400
4 000-400[a]		17 000
4 000-600[a]		14 500
4 000-600[b]		30 000
4 000-240[b]		32 000
4 000-250[b]		39 700
4 000-600[b]		37 200
4 000-250[b]		46 900
4 000-600[b]		44 900

[a] Microprocessor controlled, automatic double beam, flowchart recording system, five-digit display.

[b] With computer, memory and visual display. Price differences in this group reflect differences in the various computing and memory options available.

Texture investigations

Texture investigations are used for the determination of element distribution (using the optical microscope, EPMA); the determination of particle size and shape for enrichments analysis (SEM, TEM); and the qualitative analysis of alumina hydrates (SEM).

Texture studies are described in more detail in annex XII.

Equipment is shown in table 22. The staff required for texture studies are two graduate physicists or chemists and two technicians (for SEM and EDAX). For further information see [3] to [16].

TABLE 22. EQUIPMENT FOR TEXTURE INVESTIGATIONS

Type and specifications	*Approximate price (1980 dollars)*
Transmission electron microscope (TEM)	
Accelerating voltage: to 100 kV Lattice resolution: 0.22 nm	150 000
Accelerating voltage: to 200 kV Lattice resolution: 0.14 nm	400 000
Accelerating voltage: to 120 kV Lattice resolution: 0.14 nm	400 000
Scanning electron microscope (SEM)	
Accelerating voltage: 25-30 kV Lattice resolution: 6 nm	160 000
Accelerating voltage: 25-30 kV Lattice resolution: 6 nm	70 000
Accelerating voltage: 100 kV Lattice resolution: 3 nm	140 000
Accelerating voltage: 1-30 kV Lattice resolution: 6 nm	160 000
Wavelength and energy-dispersive microanalyser	
Three to five crystal spectrometers	350 000
Crystal detector, computer controlled	300 000
Energy dispersive detector (EDAX) (For use with SEM or EPMA)	30 000
Energy resolution: 150-160 eV Detection limit: 0.1% Element range: ^{11}Na to ^{92}U; optional from ^{6}C	

V. Equipment and human resource requirements

In the previous chapter the functions and activities to be carried out by bauxite laboratories were grouped according to techniques. In this chapter the functions and activities are grouped according to levels of laboratories and also basic equipment. Estimated costs and human resources are given. These characteristics are summarized in table 23.

It should be noted that estimated costs refer to the main equipment which account for 70% to 80% of the total equipment costs. Sometimes alternatives of different level and completion are given. There is overlap between the levels.

In determining staff requirements, overestimates have been made due to equipment-oriented considerations. Consequently, in some cases highly qualified specialists may be partially replaced by technicians and the technicians by skilled workers. The proposed scheme, however, presupposes a highly qualified specialist having ample industrial experience.

TABLE 23. ACTIVITIES OF BAUXITE TESTING LABORATORIES

Level	*Type of laboratory*	*Function*	*Main equipment*	*Estimated costs (1980 dollars)*	*Personnel required*
A	Sample preparation and storage	Sampling, homogenization, reduction of size, coding, determination of moisture and loss on ignition	Turbo mill	10 000	Two (for 2 000 samples per 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	
A	Analytical chemical laboratories (AAS, FS)	Determination of main components and some trace elements of solid phases, chemical analysis of aluminate solutions	Sequential fusion equipment	10 000	One chemist Three technicians (for 2 000 bauxite and red mud samples per 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	One chemist Three technicians One unskilled worker
			Glassware	10 000	
				20 000	
A	Other instrumental methods (spectrophotometry, electroanalytical techniques and thermometric concentration 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)	Spectrophotometer	5 000	One chemist Three to four technicians
			Precision pH-meter	1 000	
			Automatic potentiometric titrator	5 000	
			Conductivity meter	1 000	
			Thermometric titrator with calculator and plotter	6 000	
				18 000	
A	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	Two skilled workers
(A), B	Data processing for analytical chemical units	Handling, reducing and storing of analytical data	Mini computer (CPU capacity 32-48 kbyte, peripheral devices)	10 000	After special training qualified technical staff of the laboratory

TABLE 23 *(continued)*

Level	*Type of laboratory*	*Function*	*Main equipment*	*Estimated costs (1980 dollars)*	*Personnel required*
(A), B	X-ray fluorescence analysis	Determination of the main components and contaminants of solid phases	Sample preparation laboratory for XRF	32 000	Two scientists Two or three technicians (20 000 samples per year)
			Generator plus sequential spectrometer	110 000	
			Generator and simultaneous spectrometer with 20 channels under computer control	350 000	
			Close loop water cooling	12 000	
				540 000	
A	Thermal gravimetry	Determination of gibbsite, boemite, 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	One chemist or technician One (or two) samples per day
(A), B, C	Infrared spectrometry	Semiquantitative determination of minerals (e.g. gibbsite, boemite, kaolinite), characterization of certain minerals (preferably in co-operation with thermogravimetry)	Portable, simple (for A)	5 000	One graduate
			Standard (B level)	30 000	One technician
			Computerized (Fourier transform) connected with gas chromatograph (C level)	300 000	
(A), B	X-ray diffraction	Complete phase analysis of bauxite and red mud	Generator + diffractometer assembly	80 000	Two scientists Two to three technicians (1 000 to 5 000 samples per year)
			Generator + computer controlled diffractometer	160 000	
(A), B	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)	2 000	Two technicians
			Sedigraph (light absorption measuring with time scanning for grain-size distribution analysis)	18 000	
(A), (B), C	Laboratory for texture analysis	Studies of bauxite texture Determination of elementary distribution Particle shape and size analysis (for alumina and hydrate)	Light microscope	2 000	One technician
			Electron probe microanalyser (EPMA)	250 000-350 000	One graduate scientist and two technicians for each electron-microscope
			Transmission electron-microscope (TEM)	150 000-400 000	
			Scanning electron-microscope (SEM)	60 000-160 000	
B	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	20 000	Two technicians
			Mills for wet and dry grinding	40 000	Three skilled workers
			Dry/wet sieving series	6 000	
			Sample divider	3 000	
			Vibration mill	15 000	
			Drying oven	4 000	
			Autoclaves	20 000	One to two engineers
			Digester equipment (with 6-10 bombs)	20 000	Two technicians Two skilled workers
			Centrifuge	6 000	
			Pressure filter	5 000	
			Vacuum filter	5 000	
			Settling tubes	1 000	
			Soft γ-ray model settler	60 000	
			Air-agitated precipitator tanks	5 000	
			Mechanically agitated precipitators	10 000	
				220 000	

Level	*Type of laboratory*	*Function*	*Main equipment*	*Estimated costs (1980 dollars)*	*Personnel required*
(B), C	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 closed-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	Large-scale laboratory or pilot-plant facility	Technological and apparative modelling of Bayer process	Depends on existing plant and aims and on 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 $50 000 to $100 000. A pilot plant costs about 10 times as much	

VI. Laboratory layouts

The principles of setting up a bauxite testing laboratory were discussed in chapter III, the main units of these laboratories were set forth in chapter IV, and the activities, equipment and human resources of the laboratories at different levels were given in chapter V. This chapter gives examples for the layout of bauxite testing laboratories at different completion levels.

A characteristic A level laboratory is shown in figure 3. An example of a B level laboratory, where studies of process control in an alumina plant and research and development are carried out, is shown in figure 7. All laboratories require water, electricity and gas, and most require a compressed-air, vacuum and hot-water network. A C level laboratory depends to a much greater extent on the technical and economical infrastructure within a country. C level laboratories generally work in co-operation with other research laboratories and with educational institutions.

A level

Activities of an A level bauxite testing laboratory include physical investigations, such as volumetric weight for wet bauxite and dry bauxite and bauxite moisture. Other activities are:

(a) Fast determination of Al_2O_3 and SiO_2 by neutron activation;

(b) Chemical analysis of bauxite (main components: Al_2O_3, SiO_2, Fe_2O_3, TiO_2, LOI; contaminants: CaO, MgO, CO_2, P_2O_5 etc.; rarely occurring compounds: V_2O_5, Ga_2O_3;

(c) 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^{2-}, SO_3^{2-}, P_2O_5, V_2O_5, Cl^-, F^-);

(d) Chemical analysis of trace elements from bauxite, carbonate and silicate clays;

(e) Mineralogical analysis (thermogravimetry, X-ray diffractometry, infrared spectrometry);

(f) Geological investigations, including mineralogical analysis of clays (micromorphological analysis, determination of residual after extraction, microscopy investigations, calcimetric investigations, grain-size distribution analysis); and paleontological investigations (nannoplankton analysis, foraminifera analysis, macrofaunal investigations).

An example organization chart for a bauxite testing laboratory group is given in figure 8.

B level

A main function of the laboratory is to monitor the various parameters. For some plant liquids, the analysis is required every two or four hours, or during every shift. To meet this requirement, a section is provided in the laboratory where only these analyses are performed as an around-the-clock operation. The other sections of the laboratory operate only during the date shift.

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

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

Layout

The proposed laboratory layout (figure 7) is a well-proven system, based on the number and frequency of various analyses to be performed.

Utilities

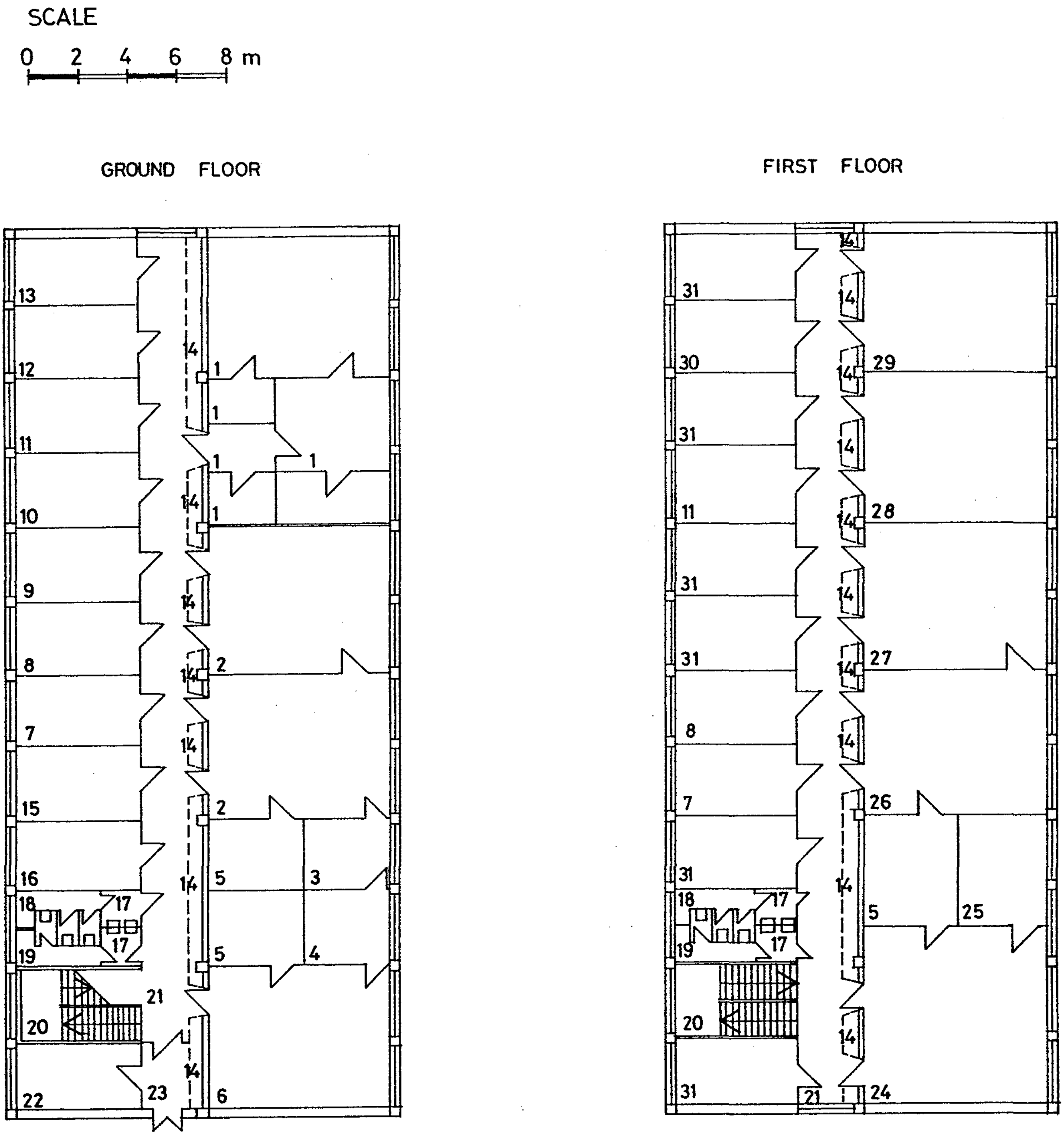
Electricity, water and compressed air are supplied from the plant network. Vacuum pumps are installed as necessary. Gas supply is from cylinders. Hoods with air-suction installed on each hood for extracting gases and vapours are located in separate rooms. The various waste solutions will be collected and after neutralization transferred to the red mud disposal facility.

C level

In setting up a large-scale or pilot-plant laboratory, it is first advisable to prepare a study of the proposed production. Table 24 indicates specifications and prices of equipment for a large-scale plant.

A flow-sheet for a large-scale laboratory is given in figure 9. Some of the equipment for the laboratory or pilot plant must be individually designed and constructed.

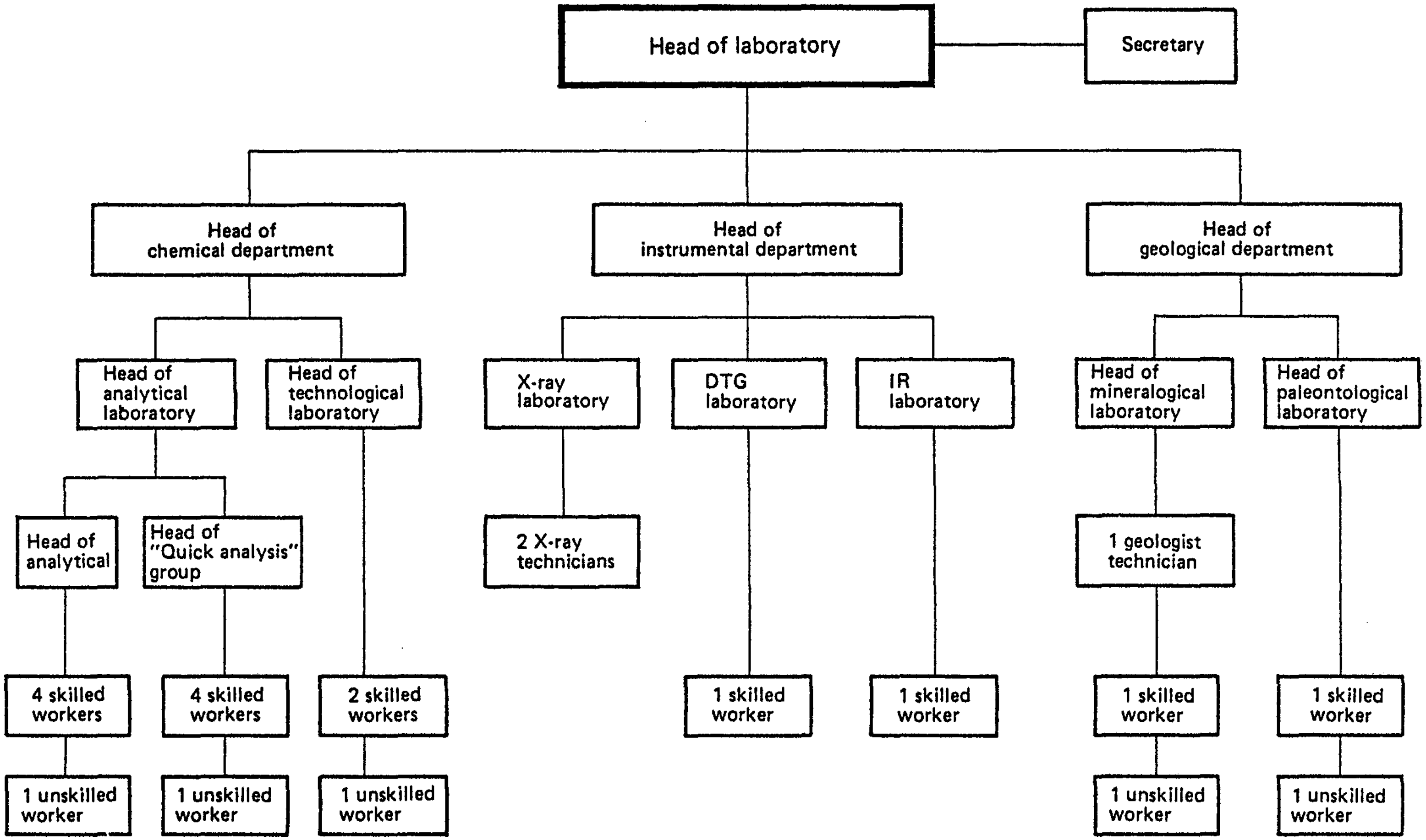
Figure 7. Layout of a B level laboratory



Key:

1. X-ray laboratory (see figure 5)
2. Wet chemical and shift laboratory (see figure 14)
3. Flame-photometer (see figure 11)
4. Atomic absorption spectrometer (see figure 11)
5. Acid hoods, drying ovens (see figure 11)
6. Bauxite and red mud sample preparation for analysis
7. Balance
8. Chemicals storage
9. Acid storage
10. Distillation
11. Control samples storage
12. Glassware storage
13. Vacuum pumps
14. General storage
15. Changing room
16. Changing room
17. Lavatory
18. Toilet
19. Toilet
20. Staircase
21. Passage
22. Switch room
23. Entrance
24. Hydrate and alumina analysis
25. Instruments
26. Technological investigations
27. Technological investigations (see figure 6)
28. Lubricants testing
29. Research laboratory
30. Library
31. Office

Figure 8. Organization chart for a bauxite testing laboratory (36 people)



Chemical department
3 chemists or chemical engineers
2 chemical technicians
10 skilled chemical workers
3 unskilled workers
18

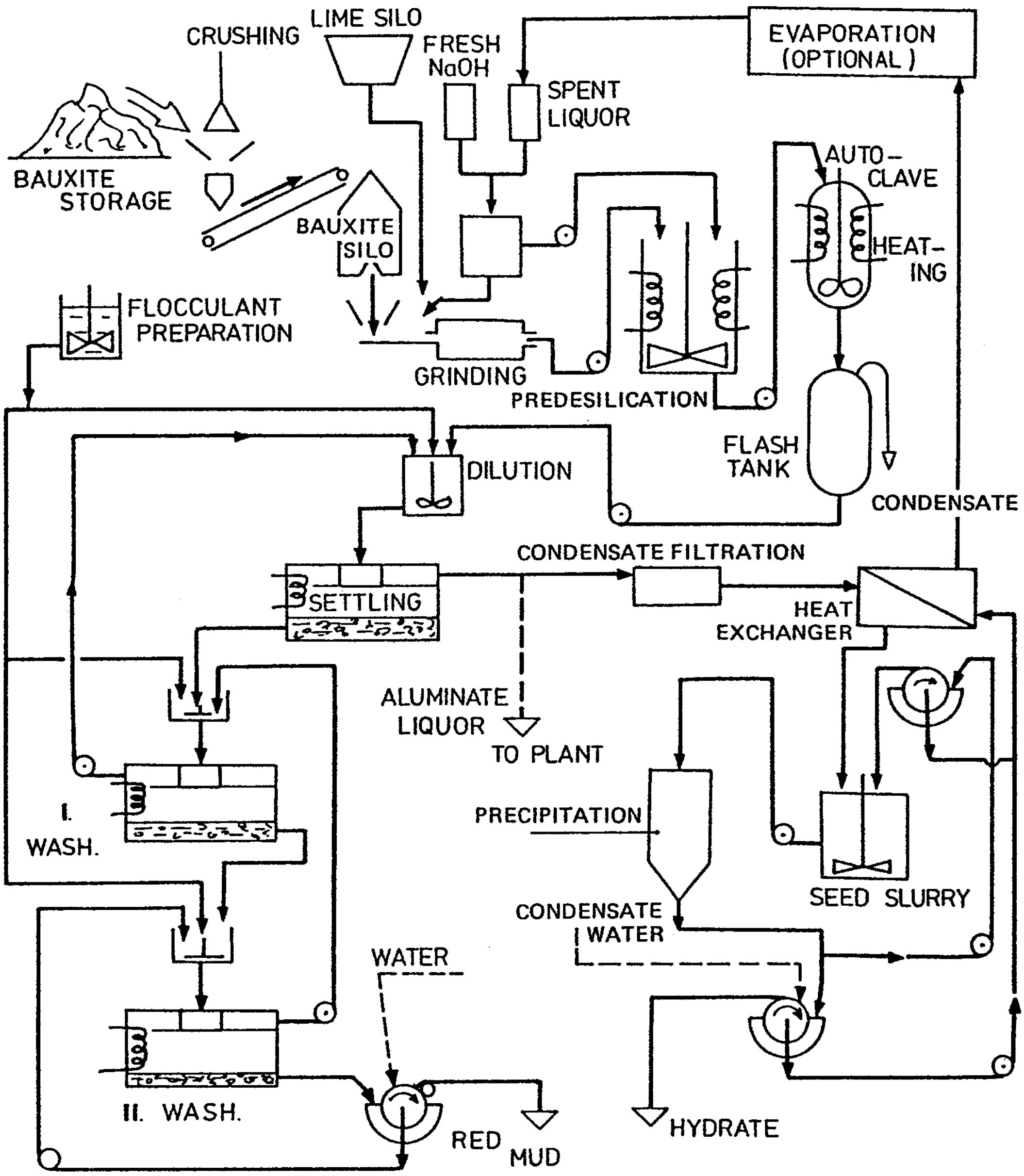
Instrumental department
4 chemists or physicists (or engineers)
2 X-ray technicians
2 specially skilled workers
8

Geological department
3 geologists
1 geologist technician
2 specially skilled workers
2 unskilled workers
8

TABLE 24. COMMERCIALLY AVAILABLE EQUIPMENT FOR LARGE-SCALE LABORATORIES

Specifications	*Approximate price (1980 dollars)*
Jaw crusher Feed opening: 200 × 125 mm Capacity: 300-1 500 kg/h Product size: 8-20 mm Push-plate: Mx steel	8 000
Jaw crusher Feed opening: 250 × 150 mm Capacity: 2 000-4 000 kg/h Product size: 5-30 mm Push-plate: Mn steel	4 500
Laboratory crushing roll Diameter: 250 mm; width: 150 mm Maximum feed size: 10 mm; product size: 0.25 mm Capacity: 2 000 kg/h Operating speed: 25-320 rev/min	10 000
Drum crusher Diameter: 300 mm; width: 250 mm Maximum feed size: 20 mm; product size: 0.1-10 mm Capacity: 4 000 to 6 000 kg/h	7 000
Ball mill Diameter: 600; length: 800 mm Screen classifier diameter: 200 mm Capacity: 30-100 kg/h Feed size: 15 mm Product size: 40-300 µm Operating speed: 43 rev/min Continuously operable	60 000
Laboratory ball/rod mill and spiral laboratory classifier and pump Mill diameter: 400 mm Length: 400 or 800 mm Spiral diameter: 225 mm Capacity: 70-150 kg/h Feed size: 6 mm	50 000
Dry sieve Single-deck screen; frame size: 250 × 500 mm Grain-size range: 0.2-10 mm Capacity: 0.2-1.2 m^3/h	4 000
Dry sieve Continuous-flow screen Screen frame size: 400 × 600 mm Single or double deck Grain-size range: 38 µm to 10 mm Capacity: 0.5-2.5 m^3/h	4 500
Dry sieve Continuous; double deck Flow-screen; dust-sucking accessories Grain-size range: 60 µm to 10 mm Capacity: 1-6 m^3/h Sieve surface: 2 × 0.3 m^2 (round)	15 000
Vibration sieve Diameter: 305 mm Four decks; for wet operation Capacity: 1-4 m^3/h Grain-size range: 60 µm to 10 mm	4 000
Grading screen: diameter 460 mm Diameter: 460 mm Four decks; for wet operation Capacity: 1-4 m^3/h Grain-size range: 60 µm to 10 mm	4 000
Air-jet sieve Screen size: 320 × 1 000 mm Grain-size up from 40 µm Capacity: 40-320 kg/h Equipped with automatic dust extractor	30 000
Autoclave Volume: 95 l Temperature limit: 350° C Strain capability: 13 MPa Material: steel	
Thickener Surface: 5.7 m^2 Volume: 10.4 m^3 Continuously operable	13 000
Mobile laboratory drum filter Area: 0.5 m^2 Zoned; precoat with scraper roll, ring or bell discharge, swivel agitator	25 000
Drum vacuum filter Area: 1 m^2 Zoned; with standard scraper belt discharge, spiral agitator	30 000
Disc filter Area: 1 m^2 consisting of 8 segments Zoned; with swivel agitator and water-ring vacuum pump	25 000

Figure 9. Flow-sheet of a large-scale laboratory



VII. Training requirements

Training programmes depend on the level of training of existing personnel. For the purpose of this discussion it is assumed that staff, including the management of the bauxite testing laboratory, have scientific training but have not had experience in organizing and operating a bauxite laboratory. Thus specialized training is required.

Two kinds of training may be used:

(a) A general review course on laboratory practice in the chemical, mineralogical, physical, physico-chemical, textural and technological evaluation of different kinds of bauxite;

(b) Special courses on particular investigation techniques (including AAS, FS, XRF, XRD, TG, IR, SEM, TEM, EPMA). This training may be given in combination with *(a)* above.

Both training programmes consist of two stages:

(a) An initial stage in which experts are trained in a group in the research centre of the technology transferring institution;

(b) A second stage consisting of an on-site course at the new laboratory, under the supervision of the experts invited for the installation and in co-operation with the scientists trained in the first stage.

A group training programme on the production of alumina was organized by UNIDO from September to December 1979. An outline for a similar training programme is included in annex XIV.

VIII. Laboratory costs by level

The estimated cost for the three levels of laboratories are shown below.

A level laboratory (for layout, see figure 3):

	Cost range (thousands of 1980 dollars)
Building	50-100
Infrastructure	50-200
Equipment	220-900
Total	320-1 200

B level laboratory (to be added to A level; see figure 7):

Building	100-300
Infrastructure	50-100
Equipment	250-1 100
Total	400-1 500

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

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

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

The costs (especially for infrastructure) vary considerably due to the local conditions, the building system used and the equipment selected.

The following main considerations determine constructing costs:

Technical-economical situation in the country
Availability of the chosen building materials
Building system used
Climatic situation of the site
Subsoil conditions
Existing level of infrastructure
Distance from sources of the building materials
Domestic regulations on construction, safety and fire protection
Labour costs

Depending on these factors the investment costs can vary from $180 to $420 per square metre.

Annex I

ATOMIC ABSORPTION AND FLAME SPECTROSCOPIC ANALYSIS

A component present in solution is excited to the atomic state by thermal energy. The light absorbed by the atomic vapour at a specific wavelength depends on the concentration of the component.

To calibrate the measuring device, the 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, more than one source must be used for determination of different chemicals. In practice the exchange of light sources is very quick. Consequently, atomic absorption spectroscopy (AAS) may be more efficient for multi-elemental determinations than other methods. Due to the small number of resonant lines spectral interference is rare.

The AAS equipment consists of an atomizing unit, the resonant radiation source, a light dispersion unit and a light detector. Recent equipment also has a data-processing unit.

The task of the atomizing unit is to produce a volume of 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. 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 is related to the design of the atomizer, the rate of atomization, the temperature determined by the chemical composition of the flame and the character of the flame (construction and shape of the burner head). The accuracy of the measurement is thus a function of the design of the atomizing gas burner.

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

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

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

The measurement of light intensity is done by a photomultiplier. Recent devices generally provide digital display for the absorbance and enable automatic zero-setting and measurement during the various integration times. There may also be direct electronic display of concentration and curve correction as required from time to time. The AAS instrument can also be used in emission (as a flame spectrophotometer).

The accessories, mainly the sample changer and the data-processing system, are very important from the point of view of productivity. The basic AAS equipment is designed for operation over long periods of time and allows the analysis of more than 100 samples per hour. Classical methods of data collection and evaluation need much more time and are sources of subjective errors. Therefore, modern AAS systems have a data-processing system controlled by a microprocessor. This system is able to supply the automatic calibration for concentration measurements using marked "set in" probes.

The relative error of the AAS method is generally 1% to 5% depending on the matrixes and the nature and concentration of the element to be determined. Several conditions determining the sensitivity and the accuracy of the method can be controlled by the operators. To obtain the best performance from the AAS system, the operator has to choose operating conditions appropriate to the element being analysed and decide whether to optimize sensitivity, detection limit or precision. The most important operator-adjustable conditions are the wavelength of the absorption line, monochromator bandpass, lamp current, fuel flow-rate, burner height, burner rotation and position of the impact bead in the case of the flame AAS method. If the ETA method is used other conditions can also be controlled.

The detailed theory of the AAS method and the main principles of various AAS instruments are described in the handbooks (see, for example, [17] or [18]).

The materials of the alumina industry have for a long time been thought of as particularly difficult to analyse by AAS because of problems associated with dissolution and also because the materials contain a number of elements that are known to cause particularly serious interference effects. This interference originates from the chemical composition of the samples. The so-called refractory elements, aluminium, titanium, silicon etc., form relatively stable compounds. However, if a suitable method is chosen and the correct precautions are taken, these determinations should not present any serious problems [19].

The determination of the main components of bauxite and red mud may be done by a combination of AAS and flame spectroscopy (FS) [20]. The components of such an AAS analytical system and the relevant flow-chart can be seen in figure 10, a laboratory layout in figure 11 and the equipment itself in figure 12.

The bauxite or red mud samples are fused with strontium carbonate/boric acid flux in platinum crucibles with a suitable sequential fusion apparatus operating at the required temperature and providing for proper fusion time. After the solidified material is dissolved, without heating, in diluted hydrochloric acid, the solutions are fed to the sample changer together with the standard solutions consisting of the required quantities of flux, hydrochloric acid and different volumes of the Al, Fe, Ti, Si, Ca, Mg and Na standard solutions. One of the standards is used as the "set in" probe and occurs several times in the sample tray, which has 50 sample positions. The intensity data are produced from the samples by the AAS equipment working with properly selected operation conditions. In the case of Al, Si, Ti and Ca an acetylene/nitrous oxide flame is used; for Fe, Mg and Na (in FS mode), acetylene/air. The weights 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 figure 13. The results are printed as a laboratory report. The analysis time is less than six hours for all the main components of six samples.

In the case of alumina, Ca, Mg, Na, Zn and Cu are generally measured by AAS and FS after proper dissolution; best is 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 is used as a flux.

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

Figure 10. Flow-chart of an AAS analytical system

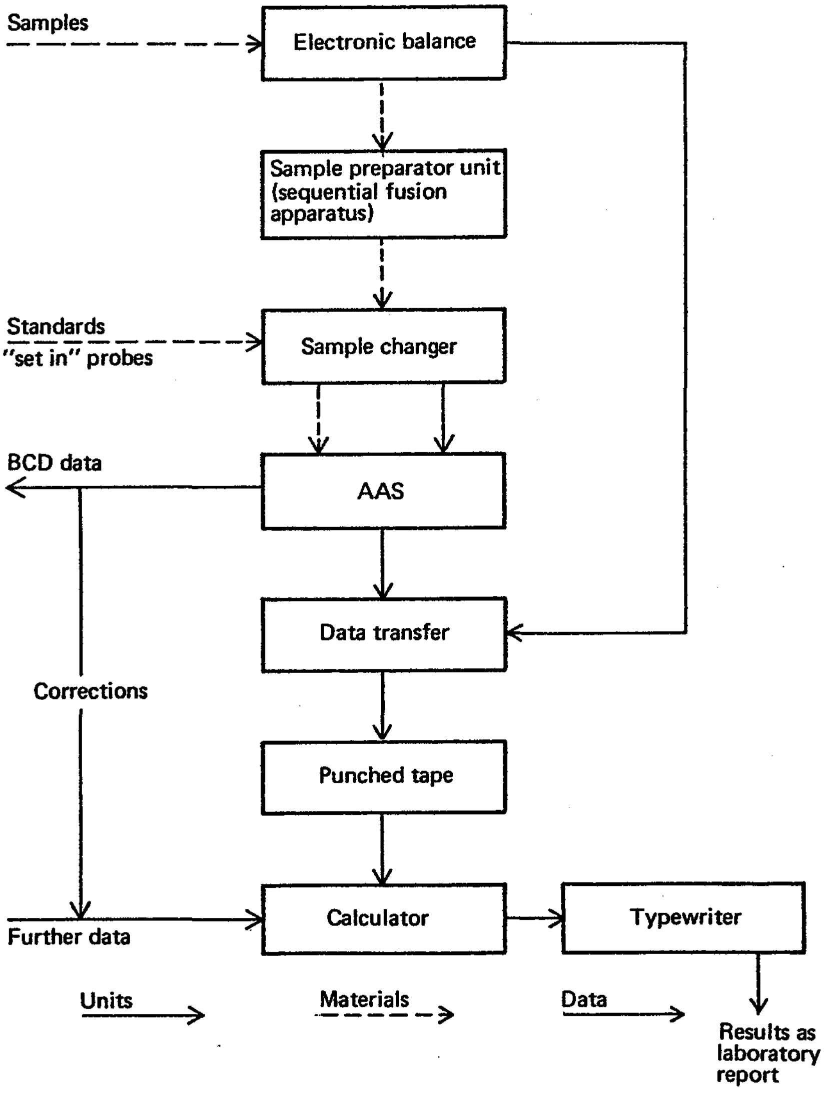


Figure 11. Layout of a laboratory for atomic absorption and flame photometric spectroscopy

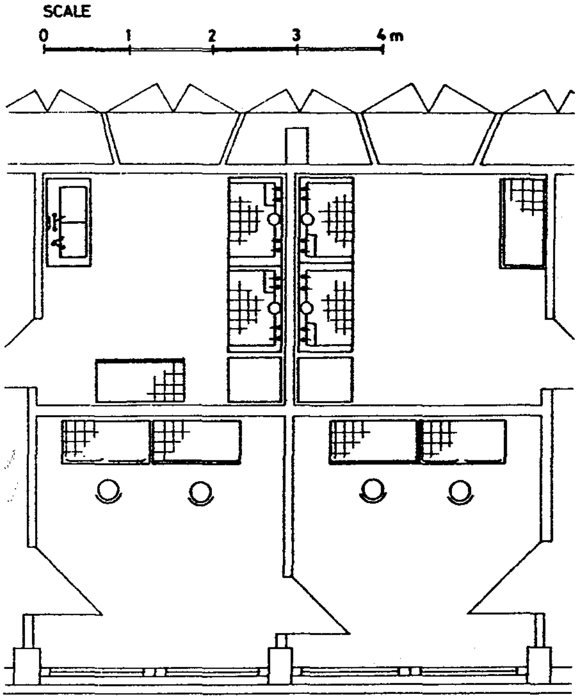


Figure 12. An atomic absorption spectrometer

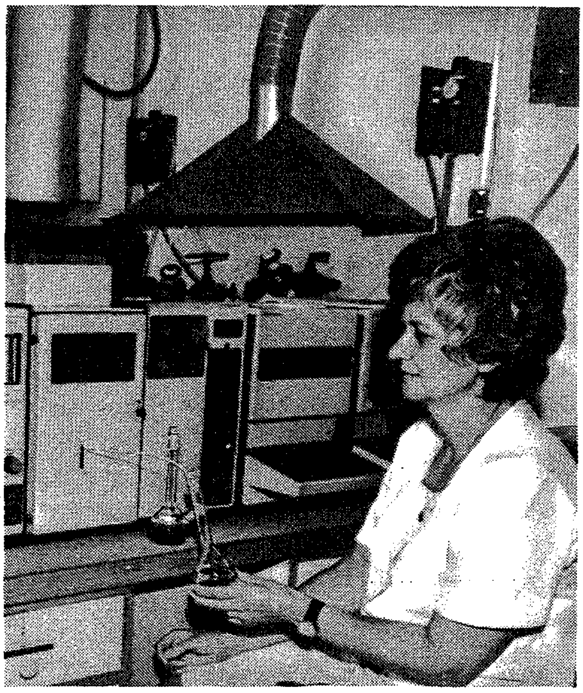
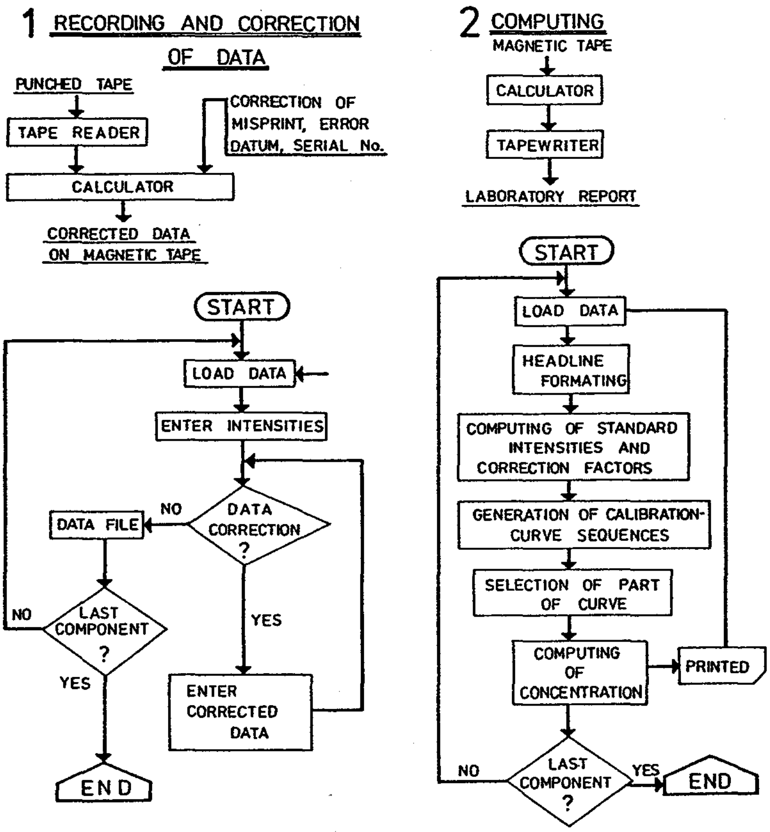


Figure 13. Data processing for an AAS/FS analysis



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

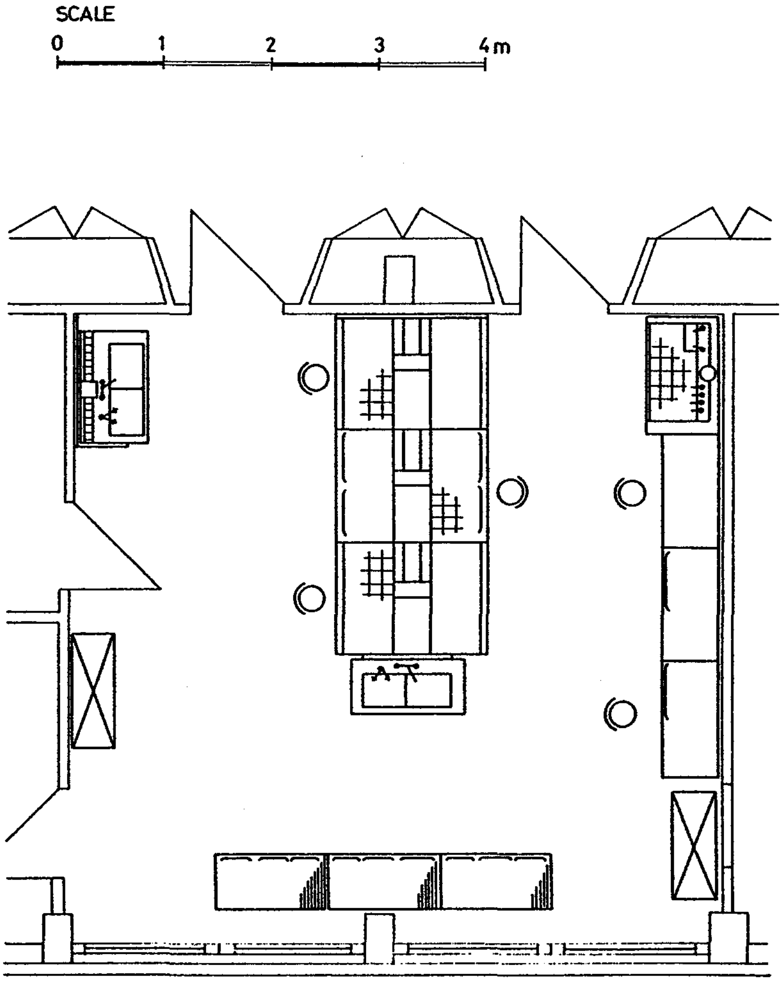
Annex II

WET CHEMICAL ANALYSIS

Since the principles of classical wet chemical analysis can be found elsewhere quite readily, only special methods of interest to the alumina industry are discussed here.

Figure 14 shows a typical layout of a laboratory for wet chemical analysis.

Figure 14. Layout of an analytical laboratory



Bauxite and red mud

The determination of the SiO_2, Al_2O_3 and TiO_2 content of bauxite and red mud requires a special dissolution procedure. Usually, the sample is dissolved in a mixture of H_2SO_4 and H_2O_2; however, dissolution in a three-acid mixture is 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 and KNO_3. The precipitate is filtered off, washed and ignited at 1,000° C, then evaporated in the presence of HF and H_2SO_4. 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, which remain as insoluble hydroxides. The aluminium can be determined by titration, taking into account the loss due to the adsorption of aluminate on to the hydroxide precipitate. If the sample has a large CaO content, a preliminary separation with urotropine is needed. The TiO_2 forms peroxo complexes of yellow colour with hydrogen peroxide and the adsorbance 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 Fe_2O_3 content of the sample is determined after fusion with KOH flux and oxidation with permanganate in H_2SO_4 solution. The ferric ions are reduced with $SnCl_2$ and titrated with potassium dichromate in the presence of diphenylamine sulphonic 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, and the flux is then extracted with hot distilled water. The chromium and vanadium can be measured together (in the form of chromate and vanadate) by titration performed with a ferrous titrating agent and the end-point can be detected potentiometrically. The vanadium can be selectively oxidized after titration with permanganate. The vanadium content can be measured again with ferrous titrant.

Alumina

There are internationally standardized procedures for the determination of the most important contaminants of alumina, which is quite fast with a spectrophotometer. The sample is dissolved after an alkaline fusion. The Fe_2O_3 content is measured as the Fe(II)-1,10-phenanthroline complex at 410 nm. The SiO_2 content is measured as reduced molybdo-silicate 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-phenylhydroxylamine. The P_2O_5 content is determined at 730 nm as the phospho-molybdic complex extracted by 2-methylpropane-1 after reduction.

Aluminate liquors

The caustic soda content is determined after the elimination of the disturbing anions (CO_3^{2-}, PO_4^{3-}, VO_4^{3-}, F^-) by precipitation with large excess of $BaCl_2$. An aliquot of the clear solution can be used for the titration with HCl titrant in the presence of phenolphthalein. The alkalinity originated from sodium hydroxide and sodium aluminate is measured this way. The disturbing effect of Al can be eliminated with addition of potassium sodium tartrate. The aluminium content of aluminate liquor forms an EDTA (ethylene diaminotetraacetic acid) complex. The excess of a known quantity of EDTA can be back-titrated with zinc acetate in the presence of a xylenol-orange indicator. The carbonate content of aluminate liquor can be determined utilizing the $BaCO_3$ content of the precipitate from the caustic-soda determinated. The precipitate can be decomposed with HCl and the excess of the acid can be back-titrated. The Na_2CO_3 content can be calculated from the consumed HCl. The silica content of an acidified aluminate liquor can be determined as the reduced silica-molybdenic blue complex, the absorbance of which 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 methods described above can be applied to analyse the standard and "set in" probes used in 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 interlaboratory testing activities.

Annex III

SPECTROPHOTOMETRIC, ELECTROANALYTIC AND THERMOMETRIC TECHNIQUES

Spectrophotometric techniques

The absorbance of a solution containing the component to be determined is proportional to the distance the light travels in the solution (size of the cuvette) and the concentration of the component. The wavelength of the investigation is characteristic for the component concerned [13].

The spectrophotometer has a light source of uniform intensity over the entire wavelength range, a light-dispersion unit, a cuvette compartment and a detector unit. Some instruments have a double-beam optical system and are able to measure absorbance values in the range 0.01-4. The latest spectrophotometers have automatic sample changers and data-processing systems.

Electroanalytical techniques

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

During equilibrium measurement the electromotive force is measured at zero current (potentiometry) [21]. In dynamic measurements the electron is used as a universal reagent and the current through the cell is maintained (voltametry, polarography) [21]. In ionic conductivity measurements, ion transport is measured (conductometry, oscillimetry) [21].

Potentiometry is done with a voltmeter that has a high input impedance and a cell that 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 used for direct potentiometry or potentiometric titration and for monitoring different components of an aluminate liquor. Thus 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 directly monitored.

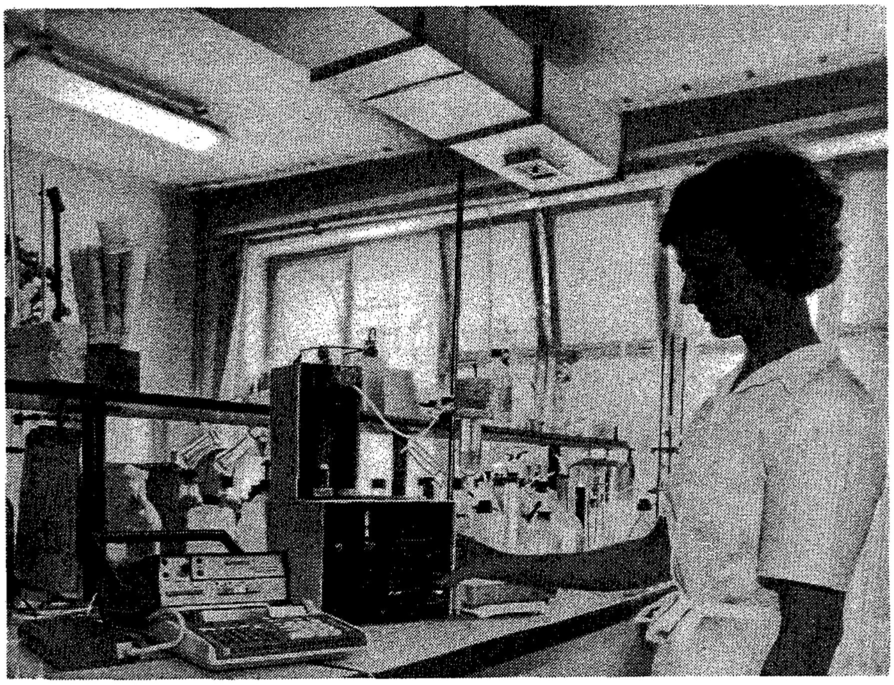
Modern polarographs consist of a potentiostat as a voltage source, a signal generator for the control of the production of the polarization voltage signal with the potentiostat and a compensograph or oscilloscope for the measurement of the current. For coulometric measurements a current integrator is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

Conductivity measurements are commonly carried out with a 1,000 Hz bridge. For oscillometry an electrodeless inductive or capacitive measuring cell is used. Oscillometry has an important role to play in the rapid, direct control of technological processes. For instance, from oscillometric measurements, caustic soda, total sodium and aluminate content can be determined.

Thermometric techniques

Thermometric concentration measurements are particularly useful for determining components at high concentrations. The heat effects arising from chemical and physical changes are detected and measured to yield information on concentration. Either direct thermometry (entalpymetry) or thermometric titration can be used. The latter is very useful in the analysis of aluminate liquor. An automatic thermometric titration system has been developed by the R and D Centre of the Hungarian Aluminium Company for the determination of caustic soda, total sodium and aluminate content, and the molar ratio of the aluminate liquor. The relative error of this thermometric determination is less than 1% and the procedure is very fast and inexpensive. Figure 15 is a photograph of the instrument.

Figure 15. Thermometric titrator



Annex IV

NEUTRON ACTIVATION ANALYSIS

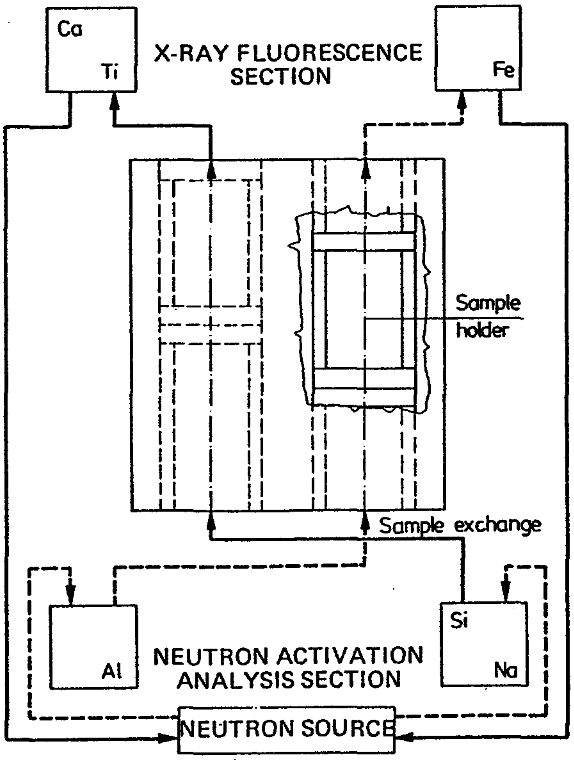
Nuclear techniques, primarily neutron activation analysis, have been widely used for rapid quantitative analysis of the main components of bauxite and red mud. The method is based on the phenomenon that some atomic nuclei in a sample exposed to neutron radiation are made radioactive, that is, "activated". The resulting radioactivity can be detected, and as the activation is related to the chemical composition and concentration of the elements in the sample, a quantitative analysis can be made. Short-time irradiation of bauxite and red mud by slow neutrons, in general, mainly activates Al. Irradiation by fast neutrons activates primarily Si and Na.

The circumstances are such that the activation procedure produces very short-lived radioisotopes the activity of which virtually ceases within a few minutes. Thus, there is no radiation hazard. The advantage of the method is that, in contrast to wet chemical analysis, little sample-preparation work is required. Because of the high penetration power of the neutrons, the irradiation may be regarded as uniform over the entire sample. The only requirement is to assure uniform packing of the sample into its holder (with the material ground to 1 mm particle size).

The resulting radioisotopes emit beta and gamma rays that are measured automatically without contacting the material. Thus, after having the sample filled into the tubes no further manual work is required. The Hungarian automatic bauxite analyser is extensively used for routine measurements. Its sample holder can accommodate 10 pairs of sample tubes. 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 lowered if the sample comprises other easily activated elements; for example, P and V. Such interference can be eliminated only by a calibration procedure.

In another version of this instrument, X-ray fluorescence analysis is applied for the determination of Ca, Ti and Fe. The fluorescence is excited by suitably selected radioactive sources emitting soft gamma rays, and a radiation detector of low selectivity is used for measurement. The measuring accuracy is less than that of the energy-selective systems: however, the unit is simple and provides a cheap, fast analytical method. A schematic diagram of the instrument is shown in figure 16.

Figure 16. Schematic diagram of an automatic bauxite analyser



Annex V

X-RAY, FLUORESCENCE AND DIFFRACTION STUDIES

Radiation sources

Spectrometric and diffraction X-ray tubes for use in R and D laboratories have different shapes, but both are water-cooled thermionic tubes working from a smoothed and stabilized, 30-100 kV power supply. Spectrometric tubes are usually rated at 3 kW and diffraction tubes vary from 1 to 2 kW.

The generators supplying high voltage may be integrated into the device, as in many sophisticated spectrometers, or separated, permitting the operation of several X-ray sources, including a combination of spectrometric and diffractometric tubes.

Generators and X-ray tubes require intense water cooling (5-10 l/min per tube at 20° C). As cold, clean tap-water may be expensive and its pressure may not be steady, closed-loop water cooling systems have been developed. These are filled with a few litres of distilled water to which anticorrosion reagents have been added and are capable of cooling at the rate of 3-4 kW.

X-rays are harmful to man, and certain safety precautions must be taken. All goniometers, spectrometers etc. must be equipped with safety devices. 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 should be worn, area monitors should be provided, and a regular schedule of medical examinations should be established.

Spectrometers

There are two types of wave-dispersive X-ray fluorescence (XRF) spectrometers: sequential and simultaneous. The sequential type is cheaper and slower, but it is more flexible. 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 even continuously fed spectrometers coupled on-line to a sampling mechanism. If coded holders are used, the measuring and evaluating program can be selected by the controlling microprocessor in accordance with the nature of the sample. The element requiring the longest period of data collection determines the cycle, which is typically 60-70 s per sample. The investment in a simultaneous spectrometer is only justified when a very large number of samples must be measured by a few fixed programs. A cheaper, less accurate variant of simultaneous spectrometers is the recently introduced energy-dispersive X-ray spectrometer.

Diffractometers

An X-ray diffractometer (XRD) consists of a goniometer (with or without a monochromator or sample changer), a detector with electronics (microprocessor, computer), a recorder, a printer and a tape punch.

A goniometer with a reflected-beam monochromator is indispensable in bauxite and red mud analyses. A radiation recording and measuring instrument and a sample changer add convenience. The performance of commercially available vertical or horizontal wide-angle goniometer devices is quite acceptable for bauxite and red mud analysis. Sample changers can handle 20 to 40 samples and the most recent also permit spinning. The X-rays are detected by a proportional or scintillation counter. Simple analogue units are being replaced by digital units in modern apparatus. The data corresponding to the X-ray intensity diffracted from the sample can be strip-chart recorded on a strip-chart, printed or stored on punched tape. If an on-line computer is present, evaluation and storage is not separated from the measurement.

Data interpretation

XRF

The integrated microprocessor or computer accomplishing data reduction and corrections at different levels, gives a list of the concentrations on a display or hard copy. Qualitative analysis is carried out using comparative tables.

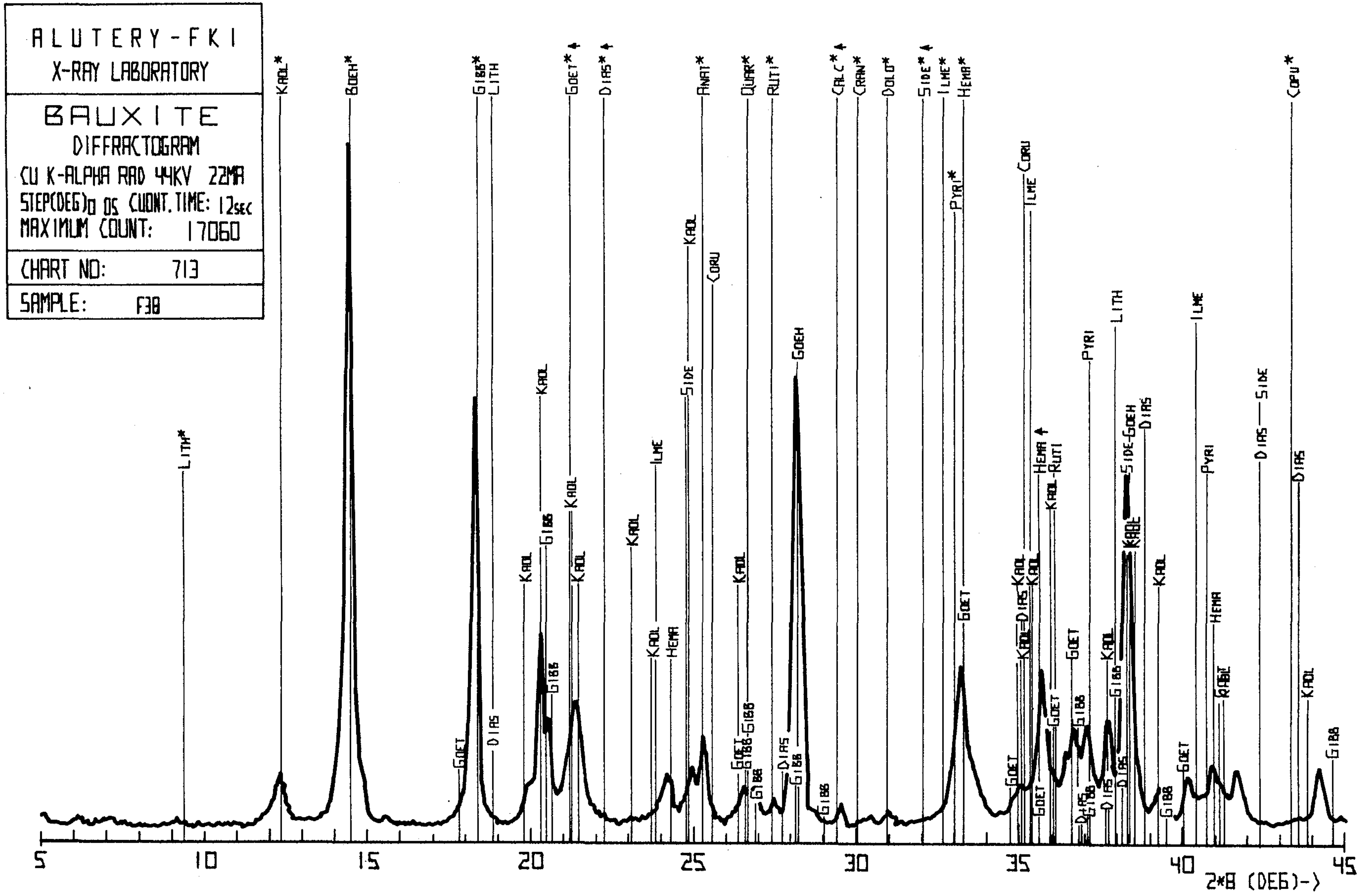
XRD

XRD analysis requires several steps. 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 or the integrated intensities of the maxima I and their width B.

Analogue 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 and I values must be compared to data files, which may be available in card, book, microfiche or magnetic-tape form. General requirements are most frequently satisfied by the Powder Diffraction File (PDF) of the Joint Committee for Powder Diffraction Standards (JCPDS) [23]. In the more restricted field of bauxite mining and alumina technology the "Minerals" subfile, covering

Figure 17. Computer-drawn X-ray diffractogram of bauxite sample F38



about 5,000 minerals, is sufficient. When the same kinds of minerals are studied, a small local file including some dozen or hundred data sets should be compiled or ordered from JCPDS.

The whole work of data reduction and interpretation can be aided by a microcomputer; nevertheless, human control seems to be important, and 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 Gy. Bárdossy [22] and P. D. Johnson [23].

The mineralogical composition of a Hungarian bauxite sample gained by this method is given in figure 17 and table 25. Further information on X-ray analysis can be found in books and journals [24-33].

TABLE 25. MINERALOGICAL COMPOSITION OF BAUXITE SAMPLE F38

ALUTERV-FKI X-RAY LABORATORY
BUDAPEST, 79.06.20.

SAMPLE: F38
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. H_2O										0.43		0.4	BOEHMITE REF		
Calculated	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			

Annex VI

SAMPLE PREPARATION FOR TECHNOLOGICAL TESTS

The laboratory tests of representative samples require about 50 kg of material. Pilot-plant tests require about 100 t of material. If ore-dressing investigations are necessary all individual samples will be required in lumpy form.

For representative sample preparation, about 100 individual samples of 0.5 to 1.0 kg are needed. The mining programme should be taken into consideration, and it may be advisable to prepare two or three composite samples of different grades. The samples have to be mixed, usually in a rotating drum, for one or two 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 and lifetime on one hand and operating costs (bauxite, caustic soda and energy consumption) on the other. A sample corresponding to this optimum grade is called a representative sample.

For modelling individual stages of the Bayer process aluminate solutions can be obtained by:

(a) Using appropriate liquor from an existing plant in the required concentration;

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

Industrial liquor (contaminated) can be reproduced only on a large-laboratory or pilot-plant scale after a longer period of continuous operation.

To evaluate the tests a large number of fast and accurate liquid analyses are required, and it is necessary to carry them out on the site. Thermometric titrimetry seems to be an adequate method for this analysis.

The aim of bauxite grinding is to render the ore more permeable and digestible by the digesting liquor. However, excessive grinding adversely affects red mud settling and uses more energy. Consequently, grinding is for an optimal grain-size corresponding to minimum energy consumption.

A good way to characterize the grindability is to determine the Bond index [34].

In alumina manufacturing, wet grinding is carried out in the presence of caustic liquids (digesting liquor) that have both chemical and physical effects on the ore. It is advisable to also simulate this process in the laboratory. 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.

Ore dressing (beneficiation) is accomplished on the industrial scale for some lateritic bauxites (of high kaolinite content) by washing off the highly reactive silica with water. The process can, and should, be modelled in the laboratory by means of a grinding and wet-sieving test before large-laboratory and pilot-plant scale experiments are carried out.

Besides silica, a few other contaminants can also be successfully eliminated by ore dressing methods provided the contaminants are favourably distributed (with respect to texture); for example, siderite by magnetic separation and calcite by heavy suspension or magnetic separation.

Annex VII

SIMULATION OF DIGESTION, SETTLING, PRECIPITATION AND CALCINATION

The transformation of reactive SiO_2 into sodium aluminium hydrosilicates is called "predesilication", the principal aim of which is to reduce the scale-formation rate and thereby increase the operation cycle time and decrease the specific energy consumption for heating during digestion. Another aim is to ensure a low SiO_2 content in the alumina, especially when processing gibbsitic bauxites.

Predesilication tests are usually performed in heat-regulated stirred tanks (95° to 100° C) or in the bomb autoclaves used in the digestibility tests. The digestion technology is determined principally by the chemical composition and mineral components of the bauxite. Whether floury or sandy alumina is going to be produced is also important.

Digestion

During digestion the equilibrium molar ratio (saturation of the solution with Al_2O_3) of the aluminate solution is the most important determinant for the technological parameters for digestion. The equilibrium molar ratio of aluminate liquor to be used for the digestion of different types of bauxite is shown in figure 18.

The simplest method of determination of extractable alumina is to treat 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 a given time at the temperature selected on the basis of phase analysis. The aim is to reach, after digestion, the equilibrium molar ratio in some samples and a higher final molar ratio in others. Digestion is carried out in steel autoclaves rotated in electrically heated, temperature-controlled oil, salt or air baths. Digestion characteristics are plotted from the results of 6 to 10 simultaneous tests. The working volume should correspond to the experimental requirements. Digestion tests are complemented by preliminary and subsequent desilication. After digestion, the aluminate liquor is separated from red mud by centrifuging. In figure 19 the Al_2O_3 recovery is plotted as the function of the A/C ratio, which is defined as the ratio of the Al_2O_3 concentration to the concentration of caustic soda expressed as Na_2CO_3 [35].

The gibbsitic and boehmitic types of bauxite are usually processed without additives. Diasporic and goethitic types require the addition of lime and other catalysts. If catalysts are added, some other parameters are of importance. These include:

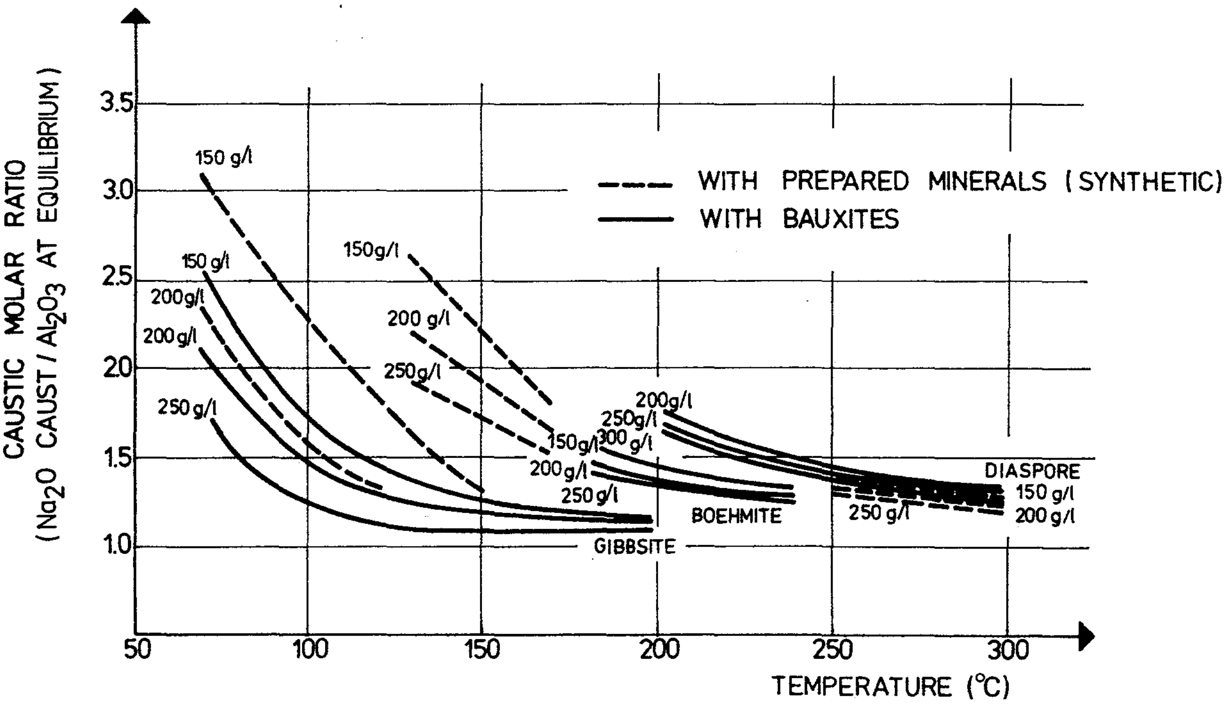
Determination of the optimal quantity of lime for Al_2O_3 recovery and NaOH consumption

Kinetics of dissolution of Al_2O_3 minerals

Kinetics of goethite-hematite transformation in the presence of additives

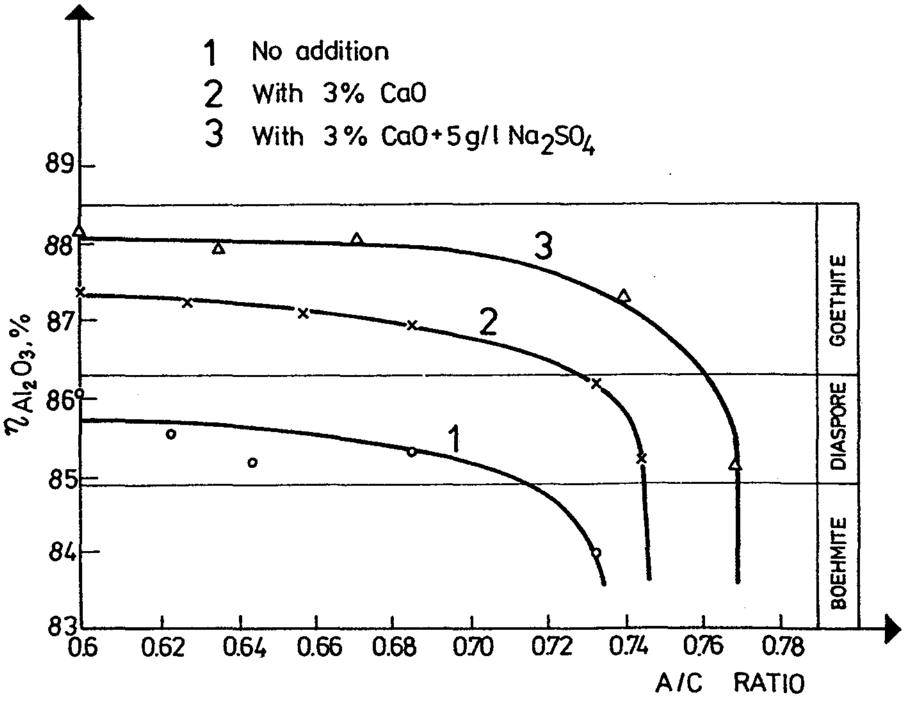
The temperature, holding time, caustic concentration equilibrium molar ratio etc. usually have to be determined, also.

Figure 18. Equilibrium molar ratio during digestion of bauxites of different mineralogical types



Source: Tikhonov and Lapkin [36].

Figure 19. Al_2O_3 recovery plotted as a function of the A/C ratio of digested liquor



The following data are necessary for a routine calculation:

Digestion liquor caustic concentration
Digestion temperature
Holding time
Caustic molar ratio after digestion
CaO required
Expected alumina recovery
Expected Na_2O/SiO_2 ratio in red mud
Degree of goethite-hematite transformation
Data on the preparation of the catalyst for the goethite-hematite conversion
Digestibility of various contaminants of bauxite

There are two types of laboratory digesters: standard autoclaves, with a mechanical stirrer (figure 20); and equipment with bomb-autoclaves developed for the determination of digestion characteristics (figure 21). A bomb autoclave is shown in figure 22.

Red mud settling

During settling, the solid red mud residue separates from the aluminate liquor. The Al_2O_3 and NaOH content of the red mud slurry is washed out. Red mud separability and washability depend on the bauxite characteristics and the digestion technique. The process used is influenced by the previous stages.

The investigation of red mud settling provides solutions to two main problems: minimizing the red mud settler and washer volume; and maximizing the efficiency of flocculation. Mud settling behaviour is expressed in terms of the settling area required per weight of dry mud per day for various mud densities. The required clarity of overflow is another consideration.

A quick comparative evaluation can be made in simple settling tubes. This method compares red mud samples obtained from different types of bauxite or from different digestion techniques or determines the effect of different flocculants, by observing the settling rate of the mud/liquor interface in the slurry using temperature-controlled cylinders with appropriate graduation.

A recently developed technique allows measurement of the actual settling rate versus the solid concentration for non-ideal suspensions in a sedimentation tube, over any interval. A schematic diagram of the technique is shown in figure 23.

Red mud sedimentation is carried out in a temperature-controlled glass tube and the solid concentration is measured by means of a system which may be moved along the tube and detects the absorption of gamma radiation. A photograph of the apparatus is shown in figure 24. The radiation source and the detector are located on opposite sides of the sedimentation tube. The measurement of the intensity of radiation passing through the suspension is displayed on a recorder. The experiment is continued for up to 48 hours to determine final concentration at the bottom of the settling tube. From the results proper dimensions for settlers and washers can be calculated and the optimum operating conditions can be established.

Figure 20. Laboratory autoclave supplied with mechanical stirrer

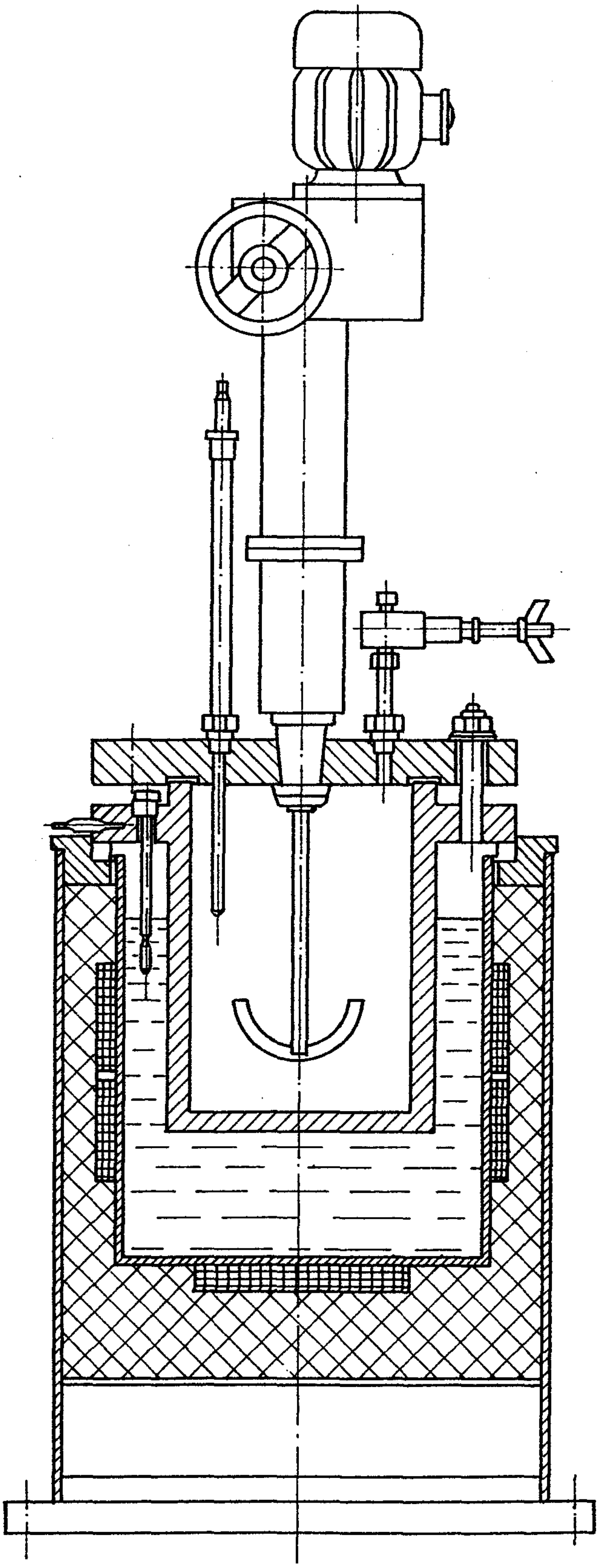
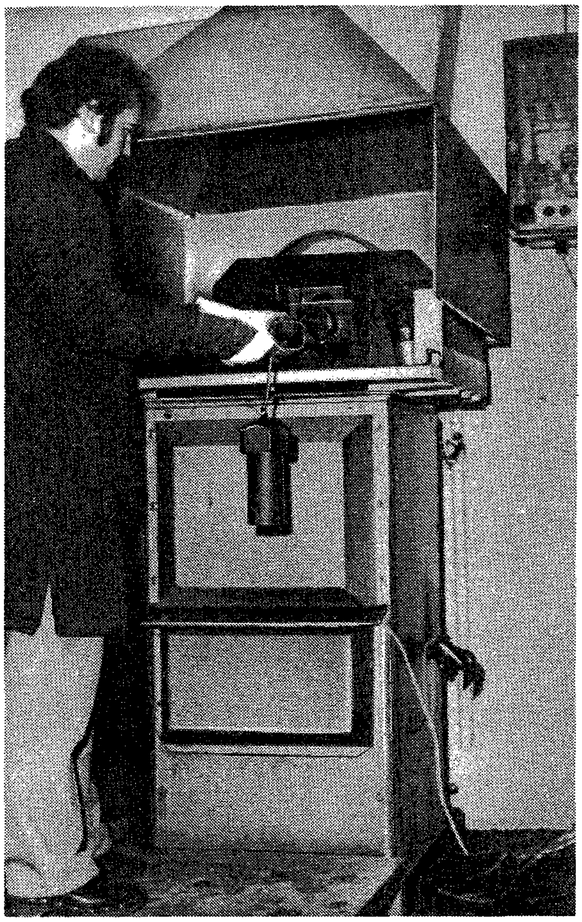


Figure 21. Oil bath with programmed temperature control for bomb autoclave



For modelling red mud washability in thickeners, the slurry is diluted step by step. Centrifugation follows. The procedure is iterated repeatedly with a more dilute aluminate solution. The filtering behaviour of red mud slurries is measured by laboratory-scale pressure filters and vacuum filters to obtain data adequate for preliminary evaluation.

Precipitation

Before precipitation a controlled filtration is carried out to remove suspended Fe_2O_3 from the aluminate liquor. Removal of other contaminants (such as Zn, Cr, and organic compounds) requires special techniques. Laboratory investigations are carried out using small, specially coated laboratory filters.

The precipitation is carried out continuously in commercial practice, but the principal parameters can be derived using laboratory-scale batch-precipitators. For studying the changes of grain-size distribution continuously operated large-laboratory or pilot-plant scale equipment is recommended. The effect of liquor contaminants, and the crystallization kinetics can be studied fairly well under laboratory conditions.

Figure 22. Bomb autoclave

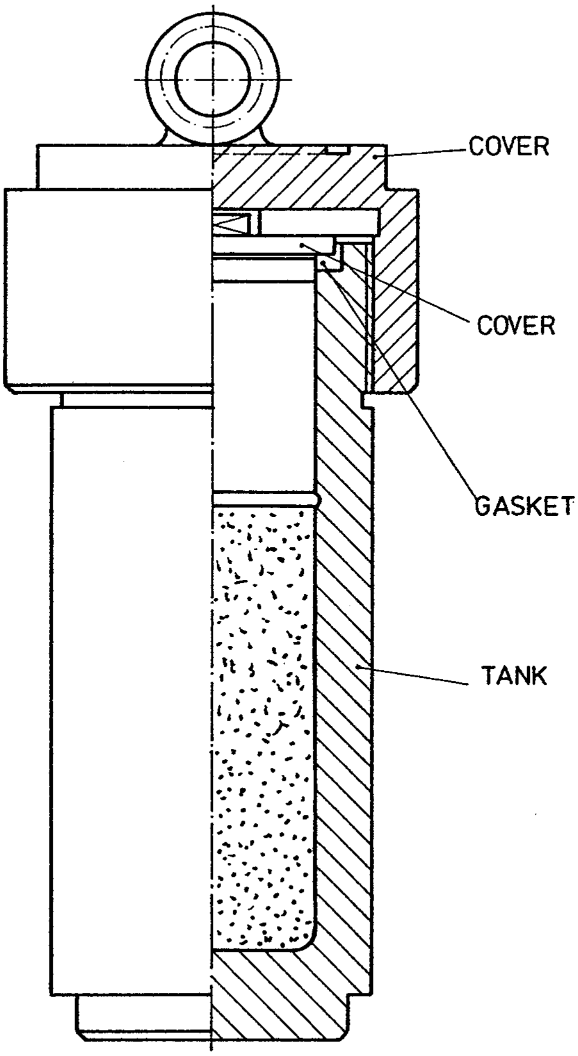
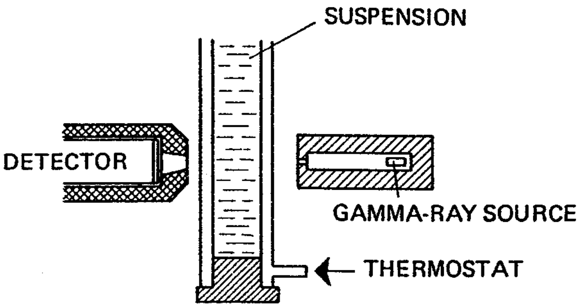


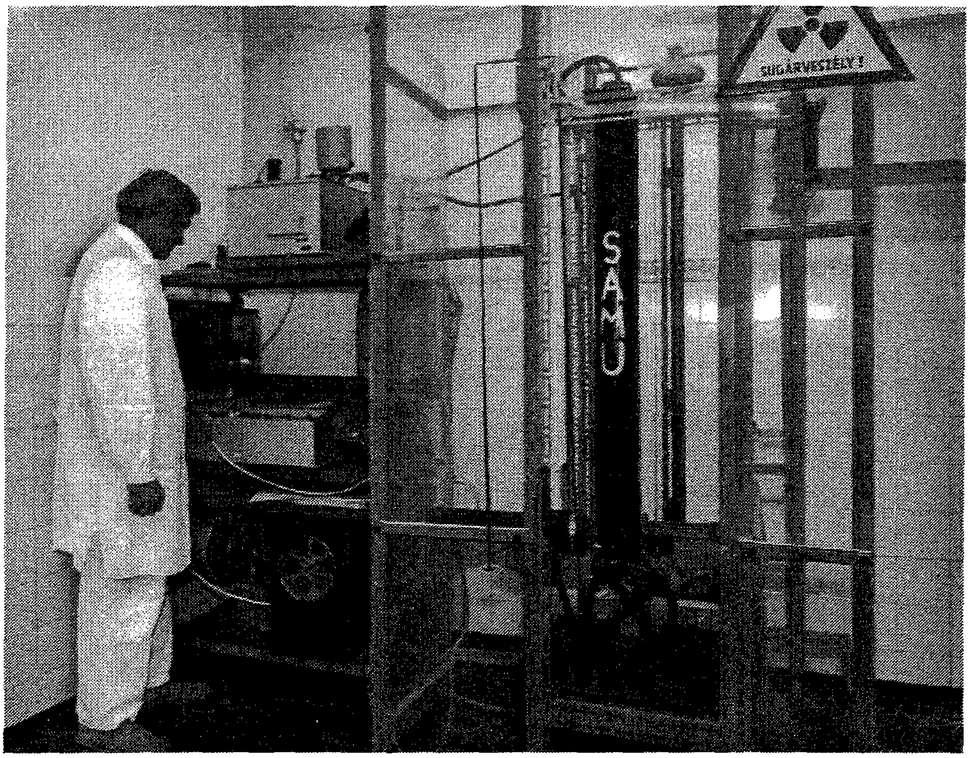
Figure 23. Schematic diagram of a soft gamma-ray absorption model settler



The precipitation experiments moved into focus recently partly because of the efforts invested in the conversion of European plants from floury to sandy alumina and partly because of the increase made in the liquor concentration at precipitation in the traditional sandy-type plants in an attempt to save energy.

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

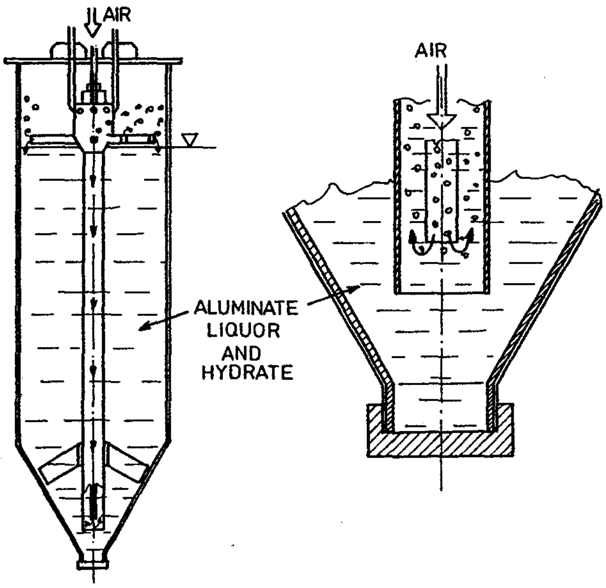
Figure 24. Soft gamma-ray absorption model settler equipment



used. One possibility is to use digester equipment with bomb-autoclaves. A laboratory air-agitated precipitator tank is illustrated in figure 25. It is important to use CO_2-free air for agitation, and the seed must be filtered before use but not dried or aged.

Hydrate filtration is performed in small-scale vacuum filters, and data on filtration period or capacity are characteristic for different hydrate types. The effect of various de-watering aids can also be studied. The calcination process and the effect of mineralizers can be studied in thermogravimetric instruments. A batch-type laboratory furnace cannot be used to model industrial calciner kilns, but they can be used to investigate the alumina obtained in precipitation tests.

Figure 25. Laboratory air-agitated precipitator tank



Annex VIII

BAUXITE CONTAMINANTS

Contaminants of bauxite may be classified into three groups:

(a) Valuable by-products (to be recovered from the Bayer cycle, such as Ga, V);

(b) Contaminants transferred to the alumina which affect its quality (P, Zn);

(c) Contaminants having a detrimental effect on the process (calcite, dolomite, siderite, pyrite and organic compounds).

If a contaminant must be removed from the Bayer cycle, the rate of dissolution of the contaminant should be measured. On the basis of dissolution tests the designer can take into consideration such measures as soda salt removal and regeneration, lime addition for P_2O_5 control etc.

In certain cases, the conditions of the digestion process must be modified to reduce the detrimental effect of a contaminant. For example, at a lower liquor concentration the rate of dissolution is lower and at a higher temperature phase transformations may occur that eliminate the effects of the contaminant.

It is a very difficult technical task to investigate the accumulation of contaminants as these influence the solubility of other contaminants. The accumulation must be studied in a pilot plant that models the Bayer cycle. Thus, research may be needed to investigate the mechanism of contamination, so as to determine a maximum acceptable level and a means for removal.

The recovery of useful components accumulated in the cycle liquor and the removal of contaminants form separate investigations. For certain contaminants standard techniques have been developed, as for recovery of vanadium, soda salt and oxalate removal.

Although the removal of organic compounds has been widely explored, no well-established process exists. A significant objective is the recovery of V and Ga (in the form of V_2O_5 and Ga_2O_3). Further, in order to reduce the caustic soda losses it is very important to realize the causticization of soda within the cycle. 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 the laboratory, using 500 to 1,000 ml volumes and different amounts of CaO.

Annex IX

PHYSICAL AND PHYSICO-CHEMICAL CHARACTERISTICS OF BAUXITE, RED MUD AND ALUMINA

The parameters to be determined for the characterization of the physical and physico-chemical characteristics of bauxite, red mud, alumina-hydrates and alumina are listed in table 26.

Density affects: mining, storage, transport, alumina plant design (for bauxite); designing of the equipment of the alumina plant, control of the alumina plant (for red mud); design, control of the production and the metallurgical utilization, grading alumina (for alumina and its hydrates).

Grain-size distribution affects: grinding tests, determination of degree of disintegration, control of crushing and grinding operations (for bauxite); control of precipitation and alumina-hydrate classification (for alumina hydrate); characterization of alumina, control of dust-exhausting equipment (for alumina); settling and washing of red mud. Grain-size distribution equipment includes sieves comprising 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 sieve (200 wire intersections per square inch). The error of sieve analysis is generally 1-1.5%.

Microscopy (electron and optical) is another direct method for the determination of grain-size distribution. Evaluation by direct observation, microphotographs or electronic counting.

Pipette methods (including the Köhn and Andreasen method) are based on sedimentation principles, and 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-absorption ability of suspensions. The light-absorption ability depends on the number and size of grains floating in the suspension, that is, it is related to their surface area. Material characteristics also have impor-

TABLE 26. 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	Density Grain-size Specific surface area Immersion heat
Aluminium	Grain-size Density, apparent density Specific surface area Angle of repose Fluorine-bonding capacity Dusting Rate of dissolution Heat transmission

tance, and the equipment has to be calibrated for all types of new materials by sedimentation measurements that can be reduced to bulk weighing.

There are several modern instruments that analyse grain-size distribution automatically 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 A and B level laboratories to install such an instrument.

The specific surface area is a very important parameter for characterizing bauxite, red mud, alumina hydrate and alumina. For bauxite, the specific surface area depends on its geological history and composition (such as the amount and quality of iron-bearing constituents). The specific surface area of the red mud produced will be similar to that of bauxite; therefore, by measuring the specific surface area of the bauxite, information can be obtained for the settling behaviour and washability of the relevant red mud. For red mud, the specific surface area depends (as well as on that of the bauxite) on the processing technology. The surface area of red mud determines its settling, washing and filtration behaviour. For alumina hydrate, the specific surface area affects the control of the precipitation process (seed charge etc.). For alumina, measurement of the surface area is important for controlling the calcination process.

The most accurate method for determination of specific surface area is the BET gas-absorption method, in which the specific surface area is determined by the amount of gas absorbed in a monomolecular layer.

The angle of repose of alumina determines its flow properties. For the measurement of the angle of repose a specially developed measuring instrument is used (figure 26). The angle of repose expressed in terms of degrees is determined by:

$$R = \mathrm{arc\,tan}\,\frac{2\,H}{D-d}$$

where:

H is the height of the alumina cone;

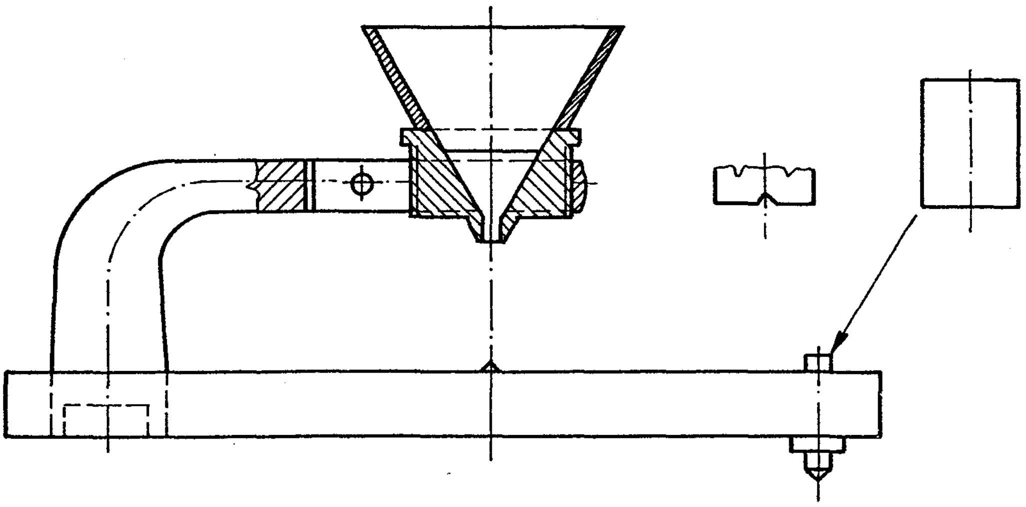
D is the diameter of the alumina cone;

d is the internal diameter of the orifice of the funnel.

The angle of repose of "highly flowing alumina" is less than 35°, that of "flowing alumina" is from 35° to 40° and that of the "sticky" alumina is greater than 40°.

Measurement of porosity and pore-size distribution gives additional information for the characterization of the morphology of bauxite and red mud.

Figure 26. Equipment for determining the angle of repose



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

The porosity can be calculated with highest accuracy using volumes measured in helium and mercury. In this vase 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 only for research laboratories). The pore-size distribution of bauxite indicates the pore-size distribution of the corresponding mud. Knowledge of porosity and pore-size distribution of red mud can be used to determine the morphology of red mud and its washability. Further, it is important to know the porosity of special kinds of alumina (adsorbents etc.).

Immersion heat is the heat involved when a material (dust) is immersed in liquid. It gives information about the solid/liquid surface interaction. Immersion heat is measured in sensitive calorimeters. The method is generally used in research laboratories.

The fluorine-bonding 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.

Annex X

THERMOGRAVIMETRIC ANALYSIS

The chemical and mineralogical composition of certain minerals changes as they are heated. In thermal investigations the sample is placed in a controlled atmosphere, and various parameters characterizing the physico-chemical state of the sample are measured or recorded as a function of the temperature, which is varied according to a predetermined programme. The most frequently used methods of thermal analysis are differential thermal analysis (DTA), thermogravimetry (TG) and differential thermogravimetry (DTG).

DTA is a technique in which the temperature difference between a substance and a reference material is measured as a function of temperature while the substance and reference material are subjected to controlled temperature changes. TG 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 the TG curve so that the changes in the sample can be followed more sensitively.

The most frequently occurring minerals in bauxite and red mud that 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 illustrated by figure 27 and table 28.

Figure 27. Thermogravimetric curves of a Halimba bauxite

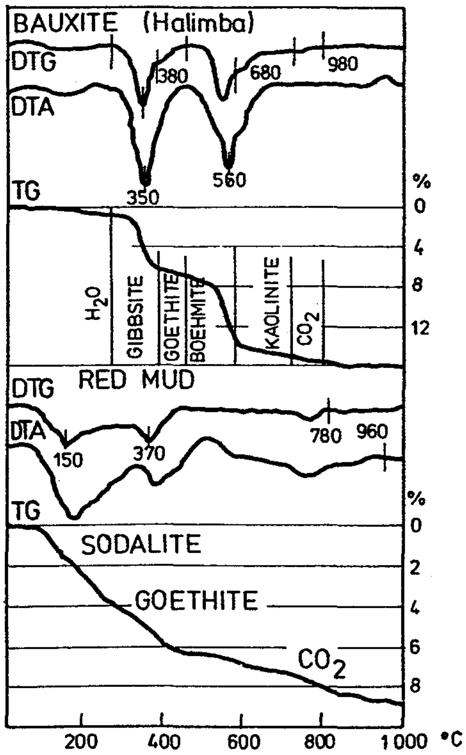


TABLE 27. THERMOGRAVIMETRIC CHANGES OF THE MINERALS OCCURRING MOST FREQUENTLY IN BAUXITES AND RED MUD

Mineral	*Weight loss in change corresponds to*	*Peak temperature of change (° C)*	*Temperature range of change (° C)*	*Calculation formula*[a]
Gibbsite $Al_2O_3 \cdot 3H_2O$	2.75 H_2O 0.25 H_2O	320-340 540-560	260-380 500-600	Al_2O_3 % = 101.94G/49.555 Gibbsite % = 156.0G/49.555
Boehmite $Al_2O_3 \cdot H_2O$ (Diaspore)[b]	H_2O	540-560	440-600	Al_2O_3 % = 101.94G/18.02 Boehmite % = 119.96G/18.02
Kaolinite $Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O$	$2H_2O$	590-610	560-700	Al_2O_3 % = 101.94G/36.03 SiO_2 % = 120.12G/36.04 Kaolinite % = 258.10G/36.04
Goethite $Fe_2O_3 \cdot H_2O$	H_2O	360-380	340-420	Fe_2O_3 % = 159.70G/18.02 Goethite % = 177.72G/18.02
Calcite $CaCO_3$	CO_2	760-780	680-820	CaO % = 56.08G/44.01 Calcite % = 100.09G/44.01
Dolomite $CaMg(CO_3)_2$	$2CO_2$	800-820 960-980	520-920	CaOMgO % = 96.40G/88.02 Dolomite % = 184.42G/88.02
Sodalite $1.3Na_2O \cdot Al_2O_3 \cdot 1.7SiO_2$	$1.7H_2O$	—	100-400	Al_2O_3 % = 101.94G/30.63 Na_2O % = 80.58G/30.63 SiO_2 % = 102.10G/30.63 Sodalite % = 315.25G/30.63
Ca-Al-silicate-hydrate $2CaO \cdot Al_2O_3 \cdot 2SiO_2 \cdot 4H_2O$	$4H_2O$	360-380	280-440	CaO % = 168.24G/72.08 Al_2O_3 % = 101.94G/72.08 SiO_2 % = 120.12G/72.08 Ca-Al-silicate-hydrate % = 462.38G/82.08
Ca-Al-silicate-hydrate $3CaO \cdot Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O$	$2H_2O$	360-380	280-440	CaO % = 168.24G/36.04 Al_2O_3 % = 101.94G/36.04 SiO_2 % = 120.12G/36.04 Ca-Al-silicate-hydrate % = 426.34G/36.04
Calcium hydroxide $Ca(OH)_2$	H_2O	520	470-550	CaO % = 50.08G/18.02 $Ca(OH)_2$ % = 74.10G/18.02
Calcium aluminate $3CaO \cdot Al_2O_3 \cdot 6H_2O$	H_2O	350	280-420	CaO % = 168.24G/72.08 Al_2O_3 % = 101.94G/72.08 Calcium aluminate hydrate % = 378.30G/72.08

[a] G is the measured weight change in the given temperature range.

[b] For calculating boehmite the residual 0.25 mole water of gibbsite should be subtracted from the weight change.

TABLE 28. EVALUATION OF A THERMOGRAVIMETRIC ANALYSIS OF BAUXITE AND RED MUD FROM HALIMBA

Sample	*Temperature range (° C)*	*Weight loss (%)*	*Composition of sample*		*Analysis*					
			Mineral	*Percentage*	*Al_2O_3 (%)*	*Na_2O (%)*	*SiO_2 (%)*	*Fe_2O_3 (%)*	*CaO (%)*	*LOI (%)*
Bauxite (Halimba)	270-380	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	37.0	31.4	—	—	—	—	
	570-720	2.1	Kaolinite	15.1	6.0	—	7.0	—	—	
	720-800	0.8	Calcite	1.8	—	—	—	—	1.0	
			Total by DTA		48.1	—	7.0	8.9	1.0	8.3
			Total by wet analysis		49.2	—	6.8	23.7	1.1	8.4
Red mud (Halimba)	100-320	4.5	Sodalite	46.3	—	11.8	15.0	—	—	
	320-460	1.9	Goethite	18.8	—	—	—	16.9	—	
	460-690	0.9	Boehmite (0.4) + pyrite	2.4	2.0	—	—	—	—	
	690-820	0.9	Calcite	2.0	—	—	—	—	1.1	
			Total by DTA		17.0	11.8	15.0	16.9	1.1	8.8
			Total by wet analysis		16.3	10.8	14.3	44.2	1.5	8.0

Notes:

1. See figure 26.
2. Boehmite was calculated by subtracting the 0.25 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%.

Annex XI

INFRARED SPECTROSCOPY

Infrared (IR) spectroscopy is based on the fact that the absorption of radiation takes place in solids at infrared frequencies characteristic of chemical bonds in the material. In the 1 to 10,000 μm range of wavelength (the infrared range), absorption is due to the change in vibrational or rotational states of molecules. Until recently, commercially available IR spectrometers operated in the more restricted wavelength range of 2-200 μm; however, the latest instruments, exploiting the principle of Fourier transformation spectrometry, cover almost the entire spectrum.

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, and the study of bond strength (e.g. that of hydrogen bridges) by means of precise measurement of the shifts of certain IR bands. Usually, for the purposes of such studies laboratory-made series of samples are compared to natural minerals.

A fast IR method of determining the reactive silica content of lateritic bauxites has been developed by White [37]. It depends on the facts that gibbsite has a characteristic Al-OH band at 965 cm^{-1} and a common band with kaolinite at 910 cm^{-1} (see figure 28) and that there is a strict relationship between the ratio of 910 cm^{-1}/965 cm^{-1} band intensities and the reactive (kaolinite) SiO_2 content (figure 29). The analysis can be carried out at a geological laboratory and gives very important information about the technological value of the ore from a very small sample.

Figure 28. Infrared spectrum in the 1,600 to 600 cm^{-1} region of low grade Jamaican bauxite

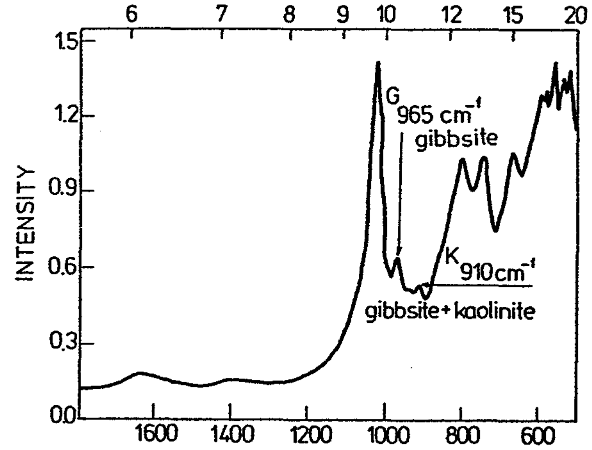
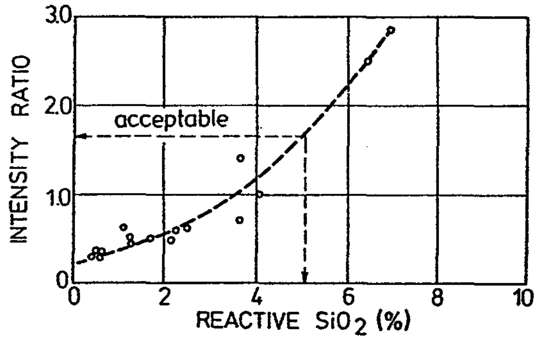


Figure 29. Relationship between reactive SiO_2 concentration and intensity ratio K910 cm^{-1}/G965 cm^{-1}



Annex XII

TEXTURE STUDIES

Texture studies yield data on the distribution of chemical elements and minerals within the ore as well as information on the size, form and distribution of pores and flaws etc.

For light microscope studies, microsections have to be prepared. These are polished, almost plane-parallel slices of some 0.1 mm thickness. These are then glued onto a glass backing and polished to the required final thickness. Microphotographs should be taken with magnifications between 2× and 1,000×.

In mineralogy complex shapes often have to be studied to resolutions of the order of 1 μm with a large depth of focus. In this range instruments based on electron optics play a highly significant role. The following deals with three major methods, X-ray microanalysis, scanning electron microscopy and transmission electron microscopy.

In the majority of bauxite ores the individual mineral particles are so small that they must be studied by transmission electron microscopes (TEM). In general, kaolinite usually forms plate-like 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 findings form the basis of TEM identification of mineral components the determination of their particles sizes and their aggregation. A more definite but also laborious identification requires preparation and evaluation of electron diffraction photographs. Energy-dispersive X-ray analysis (EDAX) can be applied also in TEM to obtain chemical data on particles as small as 10 nm in diameter. The shape and morphology of the particles—as revealed by electron microscopy—vary to a great extent depending on the mineralogical composition and origin of the sample.

The evaluation of texture data from TEM microphotographs can be greatly improved if up-to-date image-analysis techniques are applied. In this case the particle-size distribution is calculated and statistically interpreted by a computer collecting data via an optical system that senses the co-ordinates of each particle.

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

Image produced by	*Information gained*
Secondary electron	Geometrical relief (morphology)
Back-scattered electron	Average atomic number Geometrical relief
Absorbed electron	Average atomic number
Transmitted electron	Internal structure
Cathodoluminescence	Distribution and concentration of fluorescent materials
X-ray	Distribution and concentration of chemical elements from Na to U

Obviously, more than one image type must be used to get complete information. Secondary electron images plus back-scattered electron images plus energy dispersive X-ray analysis seems to be basic for SEM studies. For bauxite the above combination is normally sufficient. The other images may be used as necessary.

Scanning electron microscopes magnify from 30 to 150,000 times. The resolving power is approximately 6-7 nm. Thus they partially bridge the gap between light microscopes and transmission electron microscopes.

Figure 30 shows an alumina hydrate agglomeration (magnification 1,000 times) consisting of large gibbsite crystals with small secondary crystals on their surfaces.

Figure 31 illustrates alumina (magnification 4,750 times) calcined in the presence of a mineralizer.

Figure 30. SEM image of an alumina hydrate sample

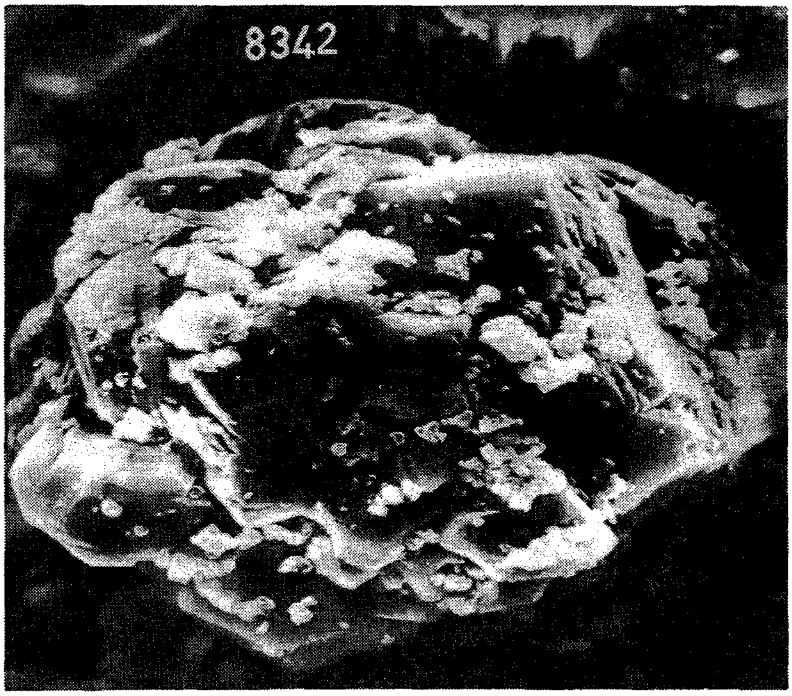
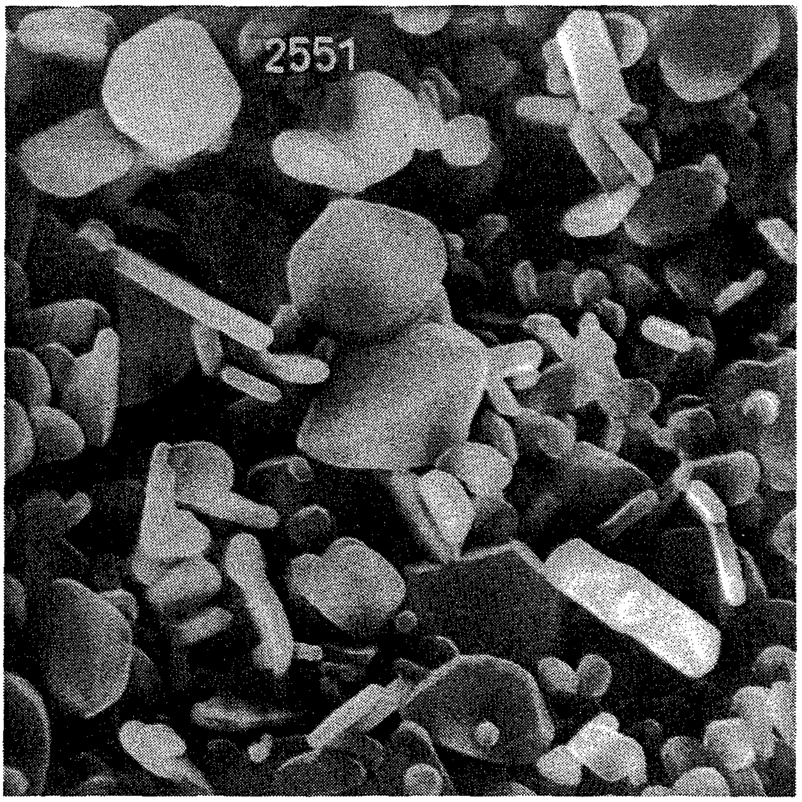


Figure 31. SEM image of an alumina sample



Electron-probe microanalysis (EPMA) reveals the textural characteristics of bauxite samples so that the microdistribution of the constituent chemical elements can be detected. The samples for EPMA investigation must have a polished surface of optical quality.

The main parts of a microanalyser are the electron optics column with vacuum system, the spectrometers and detectors, the electronic operation and control system including power supplies, and the computer-controlled measuring and display units.

The electron probe at the analysing position can be observed by means of its cathodoluminescent image, seen through a light microscope that has an illuminator for visual observation and microphotography. A magnification of up to 400 times is possible. The resolving power should be better than 1 μm. If accelerating voltage and beam current are fully stabilized, prolonged measurements can be made. The probe diameter can be reduced such that the volume analysed by X-ray spectrometry is about 2 to 5 μm^3.

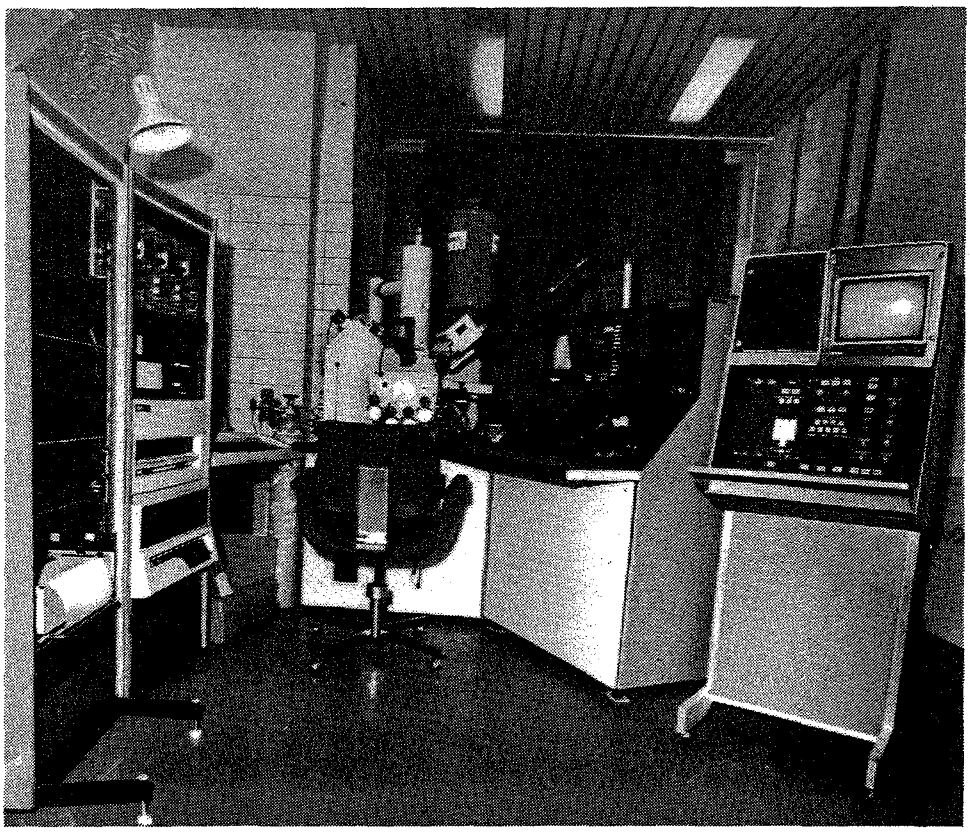
Recent microanalysers incorporate several vacuum spectrometers so that three or four elements can be analysed simultaneously. Each spectrometer is equipped with several analyser crystals selected according to the chemical elements to be measured and with detectors, such as proportional or scintillation 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.

Spectrometers exist for the detection of all elements between ^{5}B and ^{92}U. The counts representing X-ray intensity can be read from scales, digitally printed or recorded on punched tape; strip-chart recorders permit analogue representation; cathode-ray tubes are available to display the signals coming from each spectrometer; the operation of microanalysers may be controlled by microcomputers or microprocessors, which also collect, process and store data.

EPMA has been applied extensively in textural investigations of bauxite.

An EPMA laboratory is shown in figure 32. For further information about electron microscopy see references [13], [14], [15] and [16].

Figure 32. Electron probe microanalyser (EPMA)



Annex XIII

SELECTED METHODS OF CHEMICAL ANALYSIS

The chemical analysis of bauxite (and red mud), the determination of the main components of red mud by AAS and PS methods, and the chemical analysis of aluminate liquor may be found in one of the references [3, vol. 8]. For the analysis of alumina several ISO methods are available, as follows:

Determination	*ISO method No.*
Loss of mass at 3,000° C	803-1976
Loss of mass at 1,200° C	806-1976
Na_2O	1617-1976
Fe_2O_3	805-1976
SiO_2	1232-1976
TiO_2	900-1977
V_2O_5	1618-1976
P_2O_5	2829-1973
ZnO	2072-1971

Annex XIV

TRAINING PROGRAMME

General review

The following programme for a general review course is based on the experience gained during the UNIDO group training course for geologists, chemical engineers and technologists in the production of alumina, held at Budapest September-December 1979 and organized by ALUTERV-FKI, Hungalu.

Part I, Lectures

First week	Bauxite terminology, karst-bauxite deposits of the world, setting types 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 bauxite 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, porosity, optical microscopy Special electron microscope methods: (EPMA, TM, SEM + EDAX)
Fourth week	Modelling of technological process stages of the Bayer technology; technical evaluation of bauxite; tasks of large-scale laboratory and pilot plants, their organization; main features of preparing feasibility studies

Part II, Laboratory practice

First week	Laboratory safety; 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 gamma-ray absorption measurements
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

Note: The demonstration of computer techniques is performed as required.

Part III, Getting acquainted with field and plant work

One week of visits to a bauxite prospecting company and alumina plant (two or three weeks if installation of large-scale laboratory or pilot plant is expected).

The full programme may be reduced to eight weeks if necessary. In this case the methodological and practical training must be combined. Investigations on alumina hydrate and alumina may be omitted and research conducted with complex instruments may be reduced to a demonstration of the methodology and applications (if A level laboratory staff are trained). In that case, the visits to the bauxite prospecting company and the alumina plant take only one week.

Special courses in selected exploration techniques

Special courses may be complementary to a review course or held independently for the study of special fields. An example follows:

First week	Theoretical background, principles; laboratory safety regulations; short review of all methods applied in a typical bauxite testing laboratory
Second and third weeks	Laboratory practice
Fourth week	Laboratory investigations; data evaluation and processing; principles of organizing a special laboratory and its operation

Note: If this course is held in conjunction with the general review, the first week can also be used for laboratory practices.

Annex XV

TECHNOLOGICAL EVALUATION OF BAUXITE

The industrial evaluation of bauxite aims to determine its technical properties within an existing alumina plant, and to provide information for technical development. In the first case, plant conditions have to be simulated, while in the second, a detailed study of technical parameters variants and their optimization is necessary.

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 [38] for the period 1955-1970, the bauxite price A (in dollars) was related to B by the formula $A = (B-29) \times 0.40$ per short ton (900 kg). 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 distinction is made between reactive SiO_2 (kaolinite) and non-reactive SiO_2 (quartz).

The real value of a bauxite sample can be derived from the cost of its processing into alumina. Therefore, all essential technological properties of the bauxite affecting its processing must be considered. The principal ones are the TAA and the amount of caustic soda consumed in producing a ton of alumina.

Basic principles of the technological evaluation of bauxite

The technological behaviour of bauxite is controlled by its mineralogical composition. However, the texture (morphology) is also of great importance, and the role of these two characteristics often can not be separated. 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 large specific surface area of the bauxite will, for instance, promote rapid dissolution of the aluminium minerals but will cause difficulties in the separation and washing of the red mud thereby increasing dissolved caustic soda losses. These generalizations hold true, however, only in so far as the individual minerals have not been subjected to fundamental changes in the course of digestion and no phase transformation or recrystallization has taken place. In most cases such processes do occur, at least partially. This demonstrates the importance of the utilization of textural and physico-chemical investigations in the technological evaluating system.

The system for bauxite grading at Hungalu is divided into studies of texture and mineralogical compositions, providing a scientific basis, and technological studies for the purpose of economic and technical development. Scientific studies include determination of texture (thin section microscopy, electron microscopy, electron microprobe); quantitative phase analysis of bauxite (X-ray diffraction, IR spectrometry, thermal analysis); quantitative phase analysis of red mud; physico-chemical investigations (specific surface area, pore-size distribution, heat of immersion) and chemical analysis. Technical studies include grindability, digestibility and digestion parameters, determination of caustic soda losses, separability and washability of red mud, technological effects of bauxite contaminants. The application of these studies is to be found in the laboratory evaluation of different types of bauxites (gibbsitic, boehmitic, diasporic) and special ores (e.g. goethitic, low-grade, contaminated).

As an 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 react with the digesting liquor. Different types of chamosite exist. If the chamosite does not react during digestion, not only its Al_2O_3 content, but also its SiO_2 content will remain undissolved and hence there will be no caustic soda losses. Goethite and hematite will increase undigested Al_2O_3 losses proportional to the isomorphous substitution of Fe by Al in their lattices. Hematite usually improves, and goethite usually impairs, the settling and washing characteristics of red mud. The carbonate content of calcite and dolomite will raise the Na_2CO_3 content of the liquor but will reduce caustic soda losses bound to red mud. On the other hand, these minerals increase Al_2O_3 losses as well.

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 should be analysed. For a given case, the selling price of alumina and the profit of alumina manufacture can be regarded as constants. Then the value V of the bauxite can be expressed by the following formula [39]: $V = (C' - C_{Na})/G$, where C' is a constant, C_{Na} is the cost of making up caustic soda losses, and G is the amount of dry bauxite consumed. This value depends on the "soluble" Al_2O_3 content of the bauxite and on undigested and operational 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.

Determination and technological significance of the texture of bauxites

The methods for studying the texture of bauxites are thin-section optical microscopy, electron-beam microanalysis (electron microprobe) and 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 permits determination of the distribution of individual elements (figure 33).

From the texture, conclusions may be drawn regarding the crushability of the bauxite and the constituent minerals, the reactivity towards the digesting liquor and the potential of physical enrichment. By using a colour photograph technique, the amount of information recorded in a single picture can be substantially increased. The study of pulverized samples produces results that reflect averages to a greater extent than thin sections. Linear element-distribution diagrams can also be made with samples ground to different particle sizes. The true average composition, particle-size distribution and element distribution, may then be determined by statistical analysis.

This method allows identification of undigested aluminium minerals, the need for addition of CaO, scale formation, and reactions of calcite, dolomite and titanium minerals. Well-defined particles usually suggest bauxite origin, while homodisperse systems imply secondary precipitation in red mud.

Phase analysis as the basis of technological grading

The quantitative phase analysis of bauxite gives direct information on the expected yield of Al_2O_3. The efficiency of the digestion and the distribution of Al_2O_3 losses among the individual mineralogical components [40] can be determined from the phase analysis of the red mud.

The phase analysis of bauxite and red mud is generally based on X-ray diffractometry combined with thermogravimetry and sometimes combined with infrared 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 modified theoretical percentage yield η^* is given by:

$$\eta^* = \frac{Al_2O_3\,(\text{reactive}) - k_1 \cdot SiO_2\,(\text{reactive})}{Al_2O_3\,(\text{total})} \times 100$$

where:

Al_2O_3 (reactive) = Al_2O_3 (total) − Al_2O_3 (undigested). (Both SiO_2 (reactive) and Al_2O_3 (undigested) are values for the given digestion conditions.)

$k_1 \geq 1.00$ is the number of Al_2O_3 molecules for each molecule of SiO_2 in the sodium aluminium hydrosilicate.

In practice, k_1 has values in the range 1.00-1.10.

The "efficiency of digestion" η' expresses the undigested and hydrolysis losses. It is obtained by dividing the actual yield η calculated from the composition of the red mud by the modified theoretical yield η^*: $\eta' = \eta/\eta^*$.

Technological investigations

Technological investigations 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.

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 planned. The usual particle size ranges from less than 0.1 mm to 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 sedimentation methods (Sartorius balance or sedimentometer).

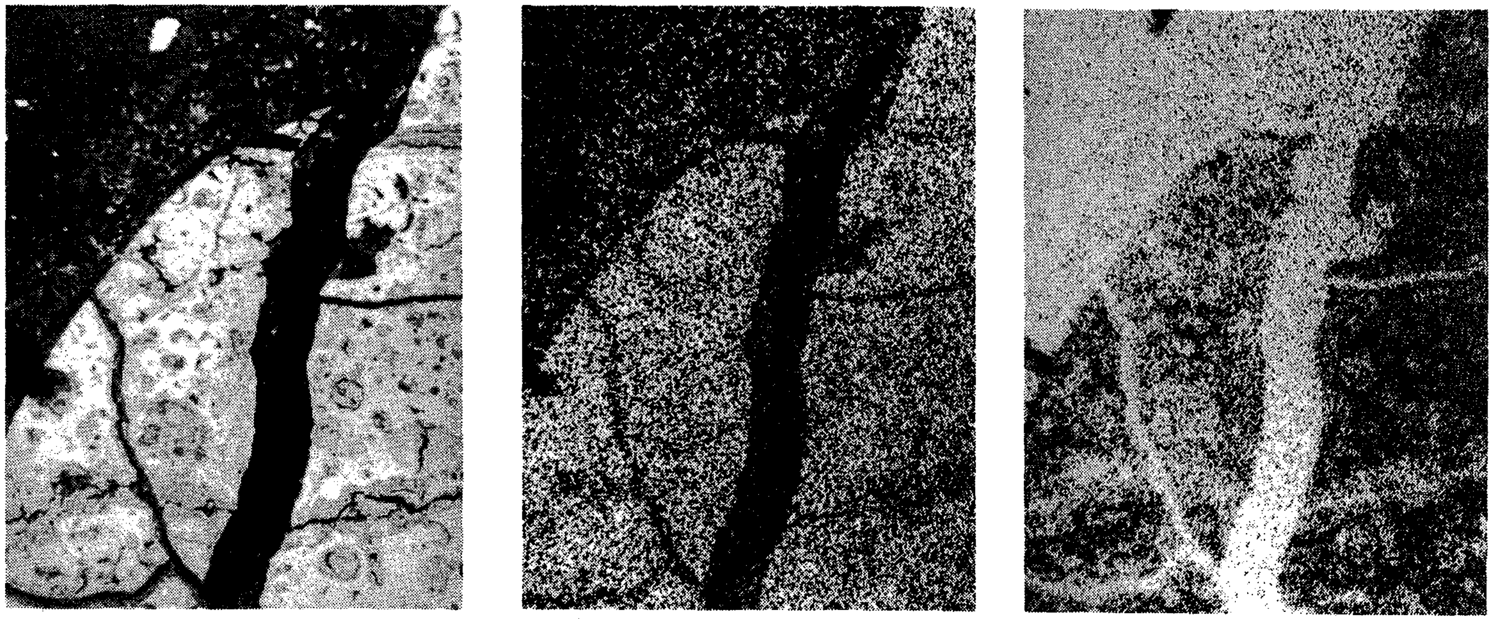
Grinding, complemented by IR studies, can also be used for the morphological characterization of the individual aluminium minerals.

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, such as appropriate temperature and optimum molar ratio. For this reason, digestion is simulated in the laboratory by determining the characteristic digestion curves [40].

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.

Figure 33. Electron microprobe photos of a Hungarian bauxite
(Magnification 200×)



A. Reflected electron image

B. Al distribution

C. Fe distribution

Caustic soda losses

Caustic soda losses in the Bayer cycle decisively affect the economics of bauxite processing [39]. The losses to be expected can only partially be determined from red mud analysis (using flame photometric atom absorption techniques), since its results allow the calculation only of bound caustic and so-called "absorbed" caustic soda losses. Caustic soda consumption of bauxite contaminants (such as carbonate, sulfate, organic matter) which leads to a higher salt level is not included.

The expected caustic losses are equal to the sum of bound Na_2O, dissolved Na_2O and Na_2O transformed into salt. They must be determined experimentally and involve many different technological investigations.

Separability and washability of red mud

The knowledge of the sedimentation and compaction characteristics of red muds is essential in the design of settler equipment. To study them a γ-ray absorption apparatus has been developed that allows measurement of the density of the red mud slurry versus time [41].

If a large number of measurements are required, or if the bauxite sample is not large enough, one may derive conclusions about the settling behaviour from the specific surface area of the red mud, as the sedimentation and filtrability of the red mud is inversely proportional to its specific surface area.

The data obtained for red muds of Iszkaszentgyörgy (Hungary) and Obrovac (Yugoslavia) bauxites are presented in figure 34. 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.

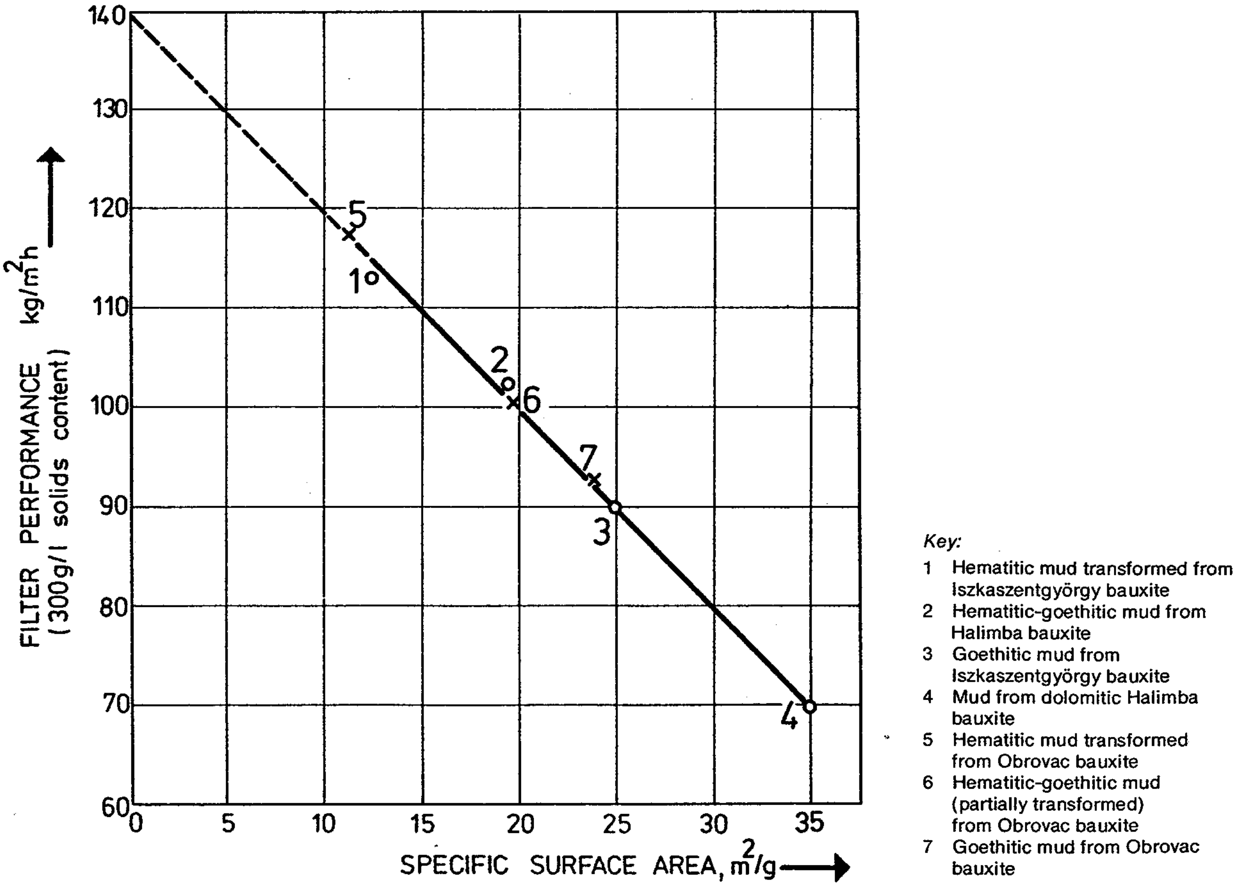
The washability of red mud is of major importance for dissolved Na_2O and Al_2O_3 losses. The experimental data becomes more understandable with regular measurements of the specific surface area and pore-size distribution of the red mud and its individual constituents [42].

Technological investigation of bauxite contaminants

Among the bauxite contaminants, sulfates and carbonates are detrimental, as they react with caustic soda. Fluorides and phosphates have a similar effect.

Zinc is also detrimental, as it impairs the purity of the alumina. In some bauxites it is dissolved in such high percentages that it must be removed from the liquor at some expense.

Figure 34. Dependence of filter performance on specific surface area of red mud



Some trace contaminants (for example, vanadium and gallium) are not so detrimental and may even increase the value of the bauxite if they are recovered from the Bayer cycle on a commercial scale. This is the case in many countries, including Hungary. According to experience, V_2O_5 recovery is possible even if CaO has been added to the digestion [43].

The behaviour of the salt contaminants, 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 a large-scale laboratory. That is very important, since those conditions frequently have a significant effect on the cost of alumina production.

The organic content of bauxites has various detrimental effects. The quantitative evaluation of these effects has proved difficult; however, a methodology for qualitative and quantitative analysis of organic contaminants in the Bayer cycle has been developed [44].

Some practical applications of bauxite evaluation

The grading system used will be illustrated for a goethitic type bauxite and for two characteristic low-grade bauxites. These examples are proofs of the dynamic character of the system.

Evaluation of goethitic bauxite

In figure 35, the digestion characteristics of a 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 29.

Aluminium substitution in the goethite lattice was 20 mol.%. For this bauxite, the theoretical alumina yield is 88.1%. If the Al_2O_3 combined in alumogoethite is considered undigestible, the (modified) yield is only 85.5%. This is confirmed by figure 35 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 the calculations it was assumed that the total silica content was reactive, although, as indicated by the digestion curves, a substantial part of the quartz did not react at 180° and 210° C. The broken line in figure 35 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.

The same bauxite was digested by a recently developed technology from Hungary [45] in the presence of hydrogarnet catalyzer added to convert goethite into

Figure 35. Digestion characteristics of a goethitic bauxite from Iszkaszentgyörgy

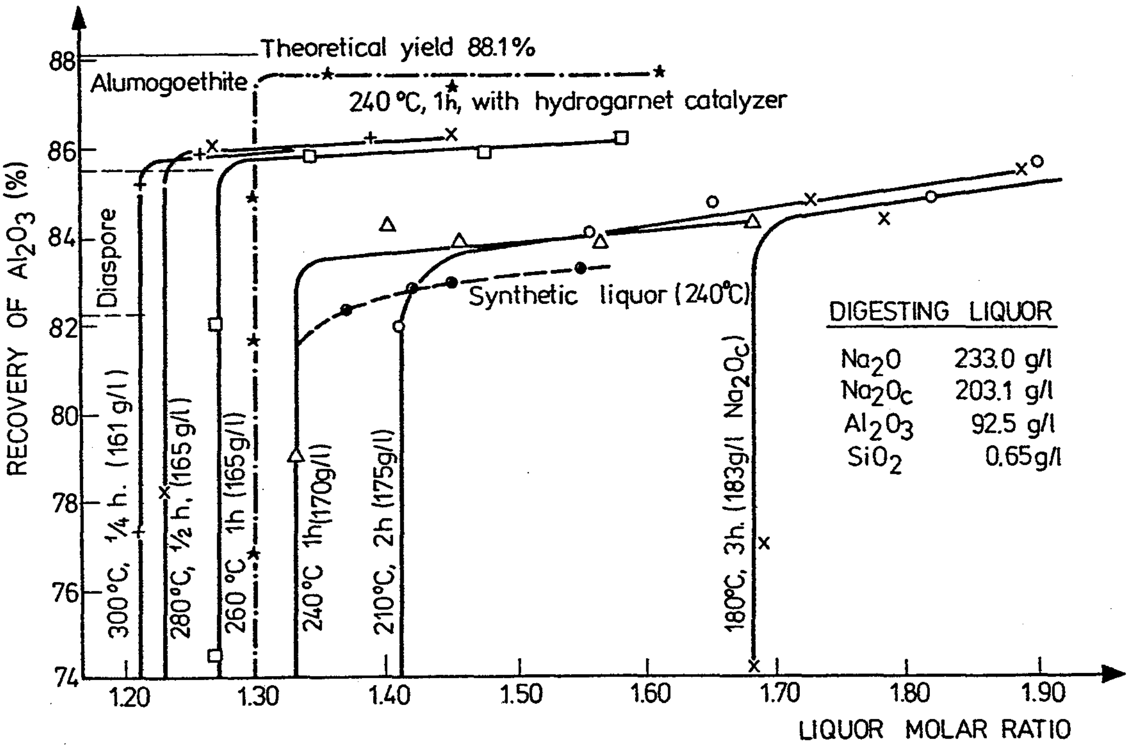


TABLE 29. MINERALOGICAL COMPOSITION OF HUNGARIAN GOETHITIC BAUXITE

(Percentage)

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			
Anatase				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

hematite. According to the digesting curve plotted in figure 35, which was taken at 240° C for 1 h, the Al_2O_3 yield almost reached the theoretical one. At the same time, the filtrability improved, as is indicated in figure 34 (about 25%). The bound soda losses were reduced because of the additive. The goethitic bauxite samples are now evaluated and processed by this new method; consequently the industrial value of goethitic bauxites has increased considerably.

Evaluation of low-grade bauxites

The starting point for the determination of the value of bauxite is the cost of alumina manufactured from it. This depends on the grade of the ore, and 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, and pertinent technological, economical and political considerations.

The following example of an Australian quartz-rich bauxite and an Iranian chamositic bauxite shows 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 30.

Of the SiO_2 content in the Australian bauxite that did not react with the digesting liquor at 150° C, quartz accounted for 75%. This explains the low Na_2O content of the red mud. As may also be seen in figure 36, 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 were 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 soda losses to be expected as a result of the low reactive SiO_2 content.

TABLE 30. COMPOSITION OF AUSTRALIAN AND IRANIAN BAUXITES AND OF RED MUDS OBTAINED FROM THEM

(Percentage)

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
LOI	6.8	7.0

The chemical and phase analysis of the red mud formed from the Iranian bauxite (figure 37) also revealed that 50% of the SiO_2 content did not react with the digesting liquor and chamosite passed unchanged into the red mud, despite the addition of CaO. The reactive SiO_2 content of this Iranian bauxite was therefore 8.2-4.1 = 4.1% and its reactive Al_2O_3 content was 46.2-7.0 = 39.2%. The predicted alumina yield was 77.3%; the experimentally found value, 79.7%. The Na_2O content of the red mud was low (4.9%), so that the processing of this bauxite may be profitable, too.

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

Figure 36. Digestion curves of an Australian bauxite

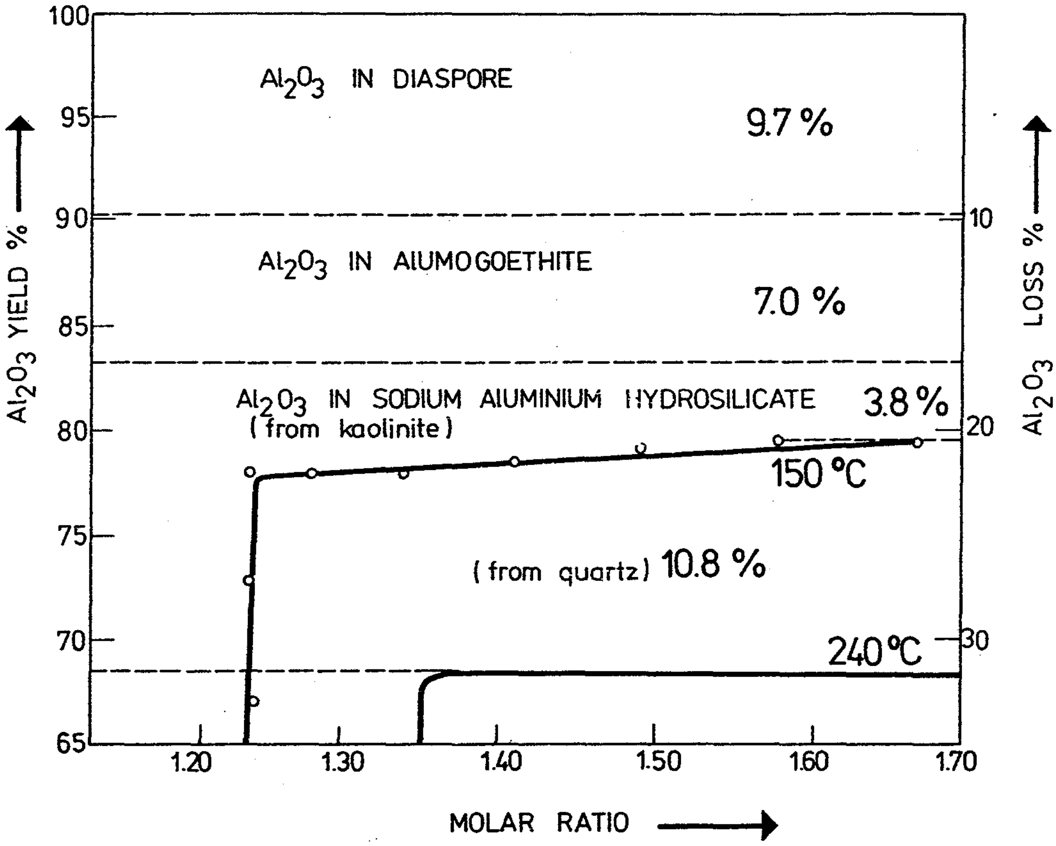
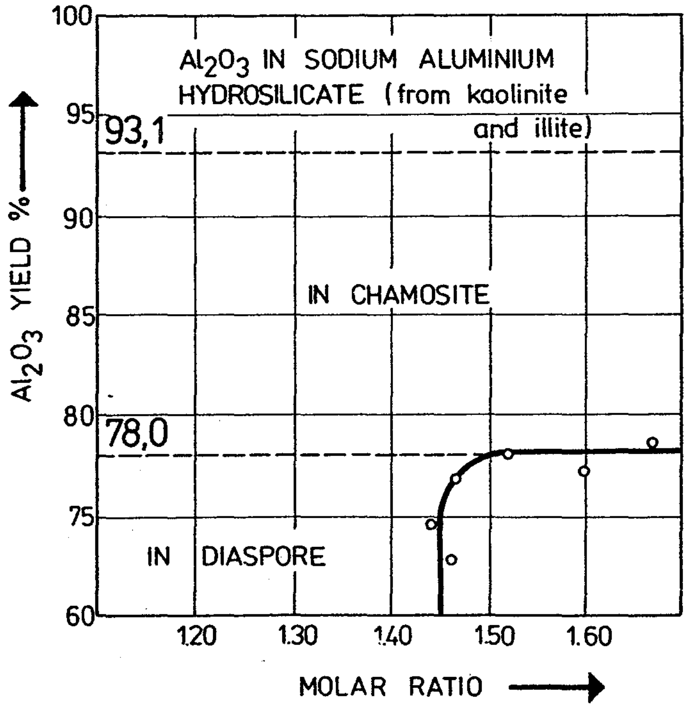


Figure 37. Digestion curve of an Iranian bauxite



In connection with poor-grade bauxite, the question of bauxite enrichment 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 analyser.

The economics of enrichment depend on what increase in value can be obtained compared to the cost of enrichment.

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 that adversely affect the process.

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

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.

As an example, the design parameters of a hypothetical alumina plant processing gibbsitic bauxite and producing sandy alumina are presented below. All parameters depending on the bauxite concerned should be determined experimentally on a large-laboratory scale or pilot-plant scale.

Design parameters for a plant producing sandy alumina

Production

Alumina production	500,000 t/a
Hourly production	62.5 t
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%
LOI	23%
Free water in wet bauxite (year's average)	9%
Lump size of ore	maximum 30 mm

Grinding, predesilication

Solids content	600 g/l
Lime added	0.1% of dry bauxite
Predesilication holding time	8 h
Predesilication temperature	100° C
Test-tank liquor, caustic Na_2O	140 g/l
Test-tank liquor, caustic molar ratio	2.8
Temperature of grinding liquor	106° C

Digestion

Temperature	140° C
Holding time at 140° C	0.5 h
Digestion efficiency for gibbsite	98%
Soda losses at digestion: for reactive silica	0.68 kg Na_2O/kg SiO_2
carbonation	0.5 kg Na_2O/t dry bauxite
Bound in other salts	0.9 kg Na_2O/t dry bauxite
Digestion residue mud to dry bauxite ratio	50%
Sand to mud ratio	5%
Mud specific gravity	3.5 g/cm^3
Final caustic molar ratio	1.4

Red mud settling, washing

Caustic Na_2O content of the settler overflow	105 g/l
Molar ratio of the settler overflow	1.55 g/l
Solids content of the settler overflow	500 g/l
Solids content of the washer underflow	500 g/l
Flocculant addition to settler	50 g/t dry mud
Flocculant addition to washers	60 g/t dry mud
Settling temperature	102° C
Temperature of interstage causticizing	85° C
Retention time at causticizing	2 h
Lime addition (per t alumina)	3 kg
Temperature of red mud wash water	80° C
Wash water for sand washing (per t)	3 t
Moisture content of sand	30%

Control filtration

Flow rate	0.5 m/h
Lime precoat (per m^3 aluminate liquor)	0.2 kg

Heat exchange

Aluminate liquor in	99° C
Aliminate liquor out	75° C
Spent liquor in	65° C
Spent liquor out	88° C

Precipitation

Aluminate liquor:	
Caustic Na_2O	105%
Caustic molar ratio	1.55%
Causticity (caustic Na_2O/total Na_2O)	0.88%
Average seed ratio	1.0
Holding time	40 h
Molar ratio of spent liquor	2.8
Mid-stage cooling of precipitators	5° C
Solids content as $Al(OH)_3$ in tray thickener overflow	1.0 g/l
Various dilutions (per t alumina)	0.5 t water

Product washing and calcination

Moisture of product hydrate	12%
Wash water to filters (per t alumina)	0.6 m³
Na_2O in alumina	0.4%
Kiln and loadout losses	0.5% of product
Product purity	99.4% (calculated on an ignited basis)

Evaporation

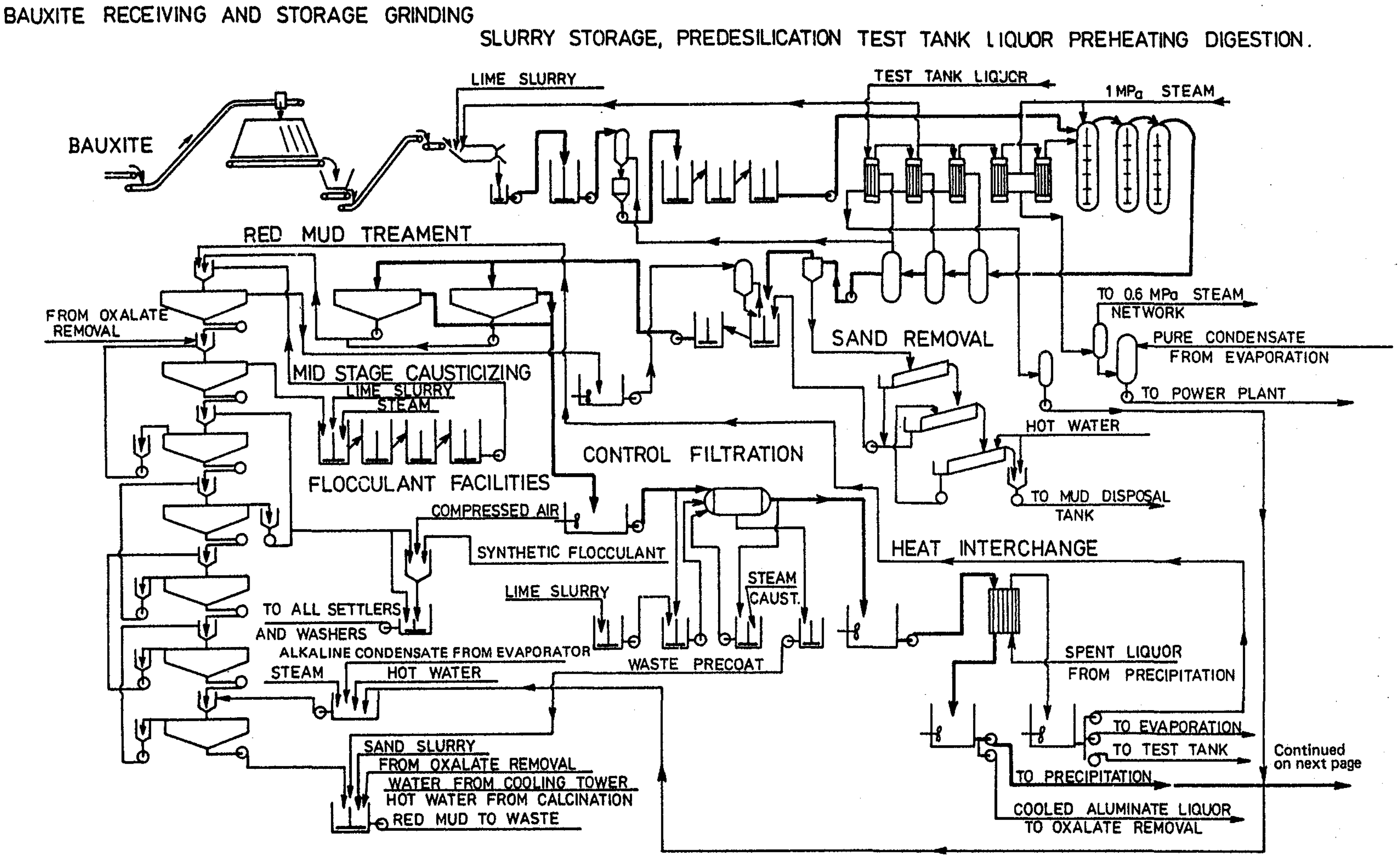
Evaporated water (per t alumina)	4.2 t

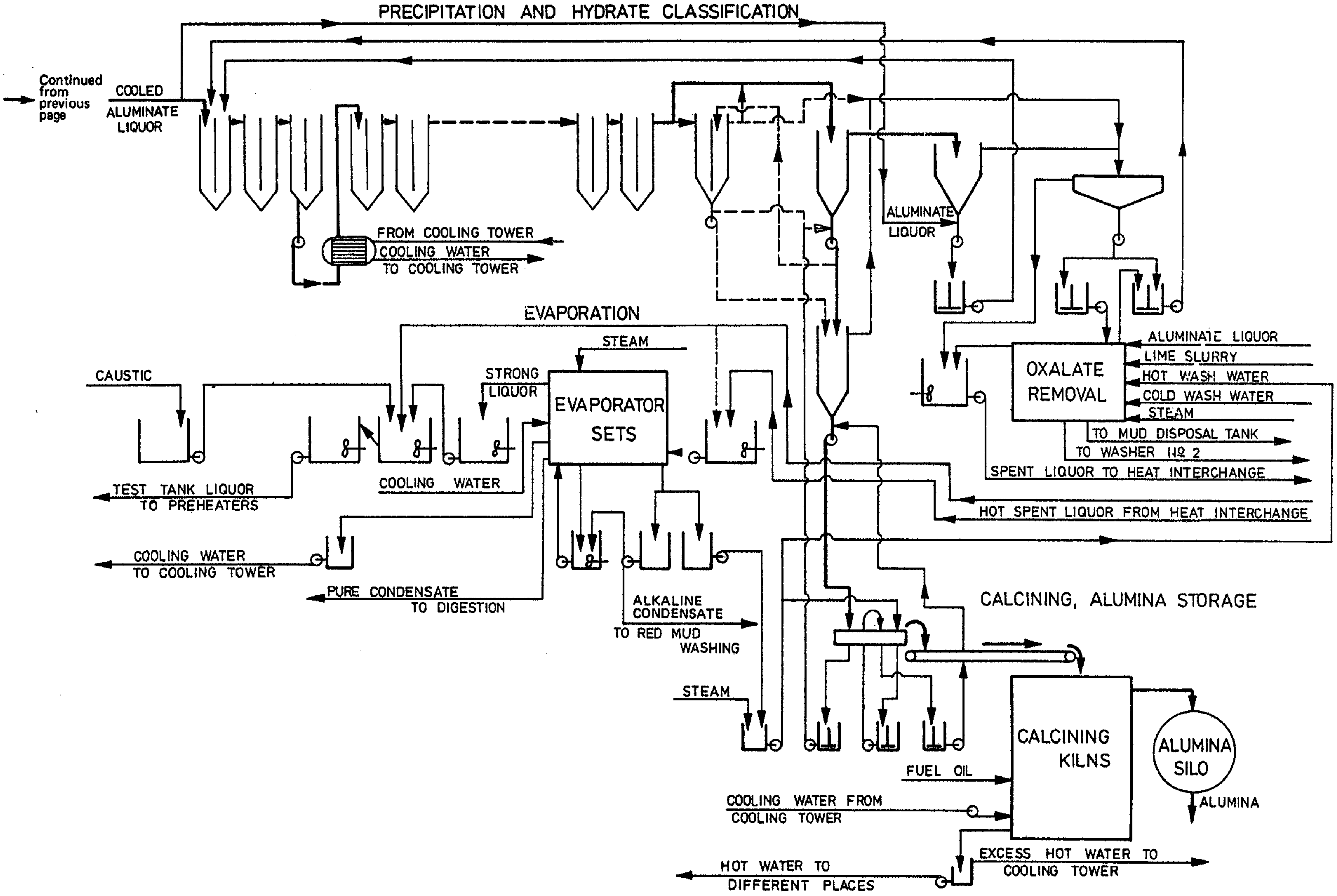
Oxalate removal

Wash water (per t alumina)	0.4 m³

A schematic flow-sheet of an alumina plant characterized by the design parameters above is shown in figure 38.

Figure 38. Schematic flow-sheet of an alumina plant




PRECIPITATION AND HYDRATE CLASSIFICATION
Continued from previous page
COOLED ALUMINATE LIQUOR
FROM COOLING TOWER
COOLING WATER
TO COOLING TOWER
ALUMINATE LIQUOR
EVAPORATION
CAUSTIC
STEAM
STRONG LIQUOR
EVAPORATOR SETS
TEST TANK LIQUOR TO PREHEATERS
COOLING WATER
COOLING WATER TO COOLING TOWER
PURE CONDENSATE TO DIGESTION
ALKALINE CONDENSATE TO RED MUD WASHING
OXALATE REMOVAL
ALUMINATE LIQUOR
LIME SLURRY
HOT WASH WATER
COLD WASH WATER
STEAM
TO MUD DISPOSAL TANK
TO WASHER № 2
SPENT LIQUOR TO HEAT INTERCHANGE
HOT SPENT LIQUOR FROM HEAT INTERCHANGE
CALCINING, ALUMINA STORAGE
STEAM
CALCINING KILNS
ALUMINA SILO
ALUMINA
FUEL OIL
COOLING WATER FROM COOLING TOWER
EXCESS HOT WATER TO COOLING TOWER
HOT WATER TO DIFFERENT PLACES

REFERENCES

1. Report of the Expert Consulting Group Meeting on the Aluminium Industry, Vienna, Austria, 10-16 November 1967 (ID/WG.11/10).

2. "Studies in alumina and aluminium production; analytical methods for testing bauxite, alumina and intermediate products" (UNIDO/ITD.171, 1973).

3. Group Training in Production of Alumina (Budapest, UNIDO/ALUTERV-FKI, 1979), 8 volumes.

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4. G. Thomas Langton, "Economic aspects of the bauxite/aluminium industry", *Journal of Metals,* August 1980, pp. 12-16.

5. G. de Weisse, "Die Entwicklung der Versorgung Europas mit Bauxit", *Erzmetall,* No. 11, 1972, pp. 535-539.

6. J. Lotze, "Economic evaluation of world's bauxite resources", *Proceedings of 4th International Congress of ICSOBA* (Athens, National Technical University of Athens, 1978), vol. 2, p. 494.

7. B. Balkay, "Options and opportunities of metallurgical progress in Africa" (UNIDO/IO.391, November 1980).

8. W. Lepori, "Das Bauxit- und Tonerdeprojekt der Alusuisse in Australien", *Schweizerische Bauzeitung,* vol. 90, No. 45 (1972), pp. 1143-1151; vol. 90, No. 51 (1972), pp. 1327-1333; vol. 91, No. 2 (1973), pp. 21-28; vol. 91, No. 24 (1973), pp. 582-588; vol. 91, No. 27 (1973), pp. 668-679.

9. ISO/TC 129, 1977.

10. T. Kotsis, "Derivatographische Untersuchungen von Bauxitmineralien", *Geologie,* No. 13, 1964, pp. 159-167.

11. *Proceedings of First International Conference on Thermal Analysis, Aberdeen, United Kingdom, 1965* (London, Macmillan); *Proceedings of Second International Conference on Thermal Analysis, Worcester, Massachusetts, 1968* (New York, Academic Press); *Proceedings of Third International Conference on Thermal Analysis, Davos, Switzerland, 1971* (Basel, Birkhäuser Verlag); *Proceedings of Fourth International Conference on Thermal Analysis, Budapest, 1974* (Budapest, Akadémia Kiadó); *Proceedings of Fifth International Conference on Thermal Analysis, Kyoto, Japan, 1977* (London, Heyden); *Proceedings of Sixth International Conference on Thermal Analysis, Bayreuth, Federal Republic of Germany, 1980* (Stuttgart, Birkhäuser Verlag); *Proceedings of Seventh International Conference on Thermal Analysis, Kingston, Ontario, Canada, 1982* (New York, Wiley).

12. G. Liptay, *Atlas of Thermoanalytical Curves* (Budapest, Akadémia Kiadó, 1975), 4 vols.

13. L. Reiner, *Elektronmikroskopische Untersuchungsmethoden* (Berlin, Springer Verlag, 1977).

14. H. R. Wenk, *Elektronmicroscopy in Mineralogy* (Berlin, Springer Verlag, 1976).

15. O. C. Wells, *Scanning Electron Microscopy* (New York, McGraw-Hill, 1974).

16. E. Köster, *Granulometrische und morphometrische Messmethoden an Mineralkörnern, Steinen und sonstigen Stoffen* (Stuttgart, Ferdinand Ehke, 1964).

17. J. Ramirez-Monoz, *Atomic Absorption Spectroscopy* (Amsterdam, Elsevier, 1968).

18. G. F. Kirkbright and M. Sargent, *Atomic Absorption and Fluorescence Spectroscopy* (London, Academic Press, 1974).

19. F. J. Langmyhr and P. E. Paos, "The analysis of inorganic siliceous materials by atomic absorption spectrophotometry and the hydrofluoric acid decomposition technique", *Analitica Chimica Acta,* vol. 43, 1968, p. 397.

20. L. Tomcsanyi and M. Borsodi-Kovacs, *Determination of Major Components of Bauxite and Red Mud by AAS Analytical System Euroanalysis III* (Dublin, Institute of Industrial Research and Standards, 1978), p. 56.

21. G. W. Ewing, *Instrumental Methods of Chemical Analysis* (New York, McGraw-Hill, 1969).

22. Gy. Bardossy and others, "Automated quantitative phase analysis of bauxite", *American Mineralogist,* No. 65, 1980, pp. 135-141.
23. P. D. Johnson, "Quantitative phase analysis of hydrothermal sedimentary rocks", *Advances in X-ray Analysis,* No. 21, 1978.
24. E. F. Kaelble, *Handbook of X-rays* (London, McGraw-Hill, 1967).
25. E. P. Bertin, *Principles and Practice of X-ray Spectrometric Analysis* (New York, Plenum, 1978).
26. R. Jenkins and J. L. de Vries, *Practical X-ray Spectrometry* (Eindhoven, Philips Technical Library, 1967).
27. H. P. Klug and L. F. Alexander, *X-ray Diffraction Procedures* (New York, Wiley, 1954).
28. *Mineralogical and Technological Evaluation of Bauxites* (Budapest, VAMI-FKI, 1975), vol. 2.
29. Powder Data File, JCPDS, 1601 Park Lane, Sworthmore, Pennsylvania, 1980/30 series.
30. *Advances in X-ray Analysis.*
31. *American Mineralogist.*
32. *Journal of Applied Crystallography.*
33. *X-ray Spectrometry.*
34. H. G. Zeisel, "Eine neue Methode der Mahlbarkeitsprüfung", *Zement-Kalk-Gips,* No. 10, 1957, pp. 435-438.
35. K. Solymar, J. Matyasi and B. Toth, "Digestion of Hungarian Goethitic bauxites with additives", *Travaux de l'ICSOBA* (Zagreb), No. 13, 1976, pp. 299-312.
36. N. N. Tikhonov and A. A. Lapkin, *Mineralogical and Technological Evaluation of Bauxites* (Budapest, VAMI-FKI, 1975), vol. 2, p. 202.
37. J. L. White, "Evaluation of bauxites by infrared techniques", *Travaux de l'ICSOBA,* No. 12, 1974, pp. 25-31.
38. E. Alliquander, B. Balkay and K. Solymar, "Improving low-grade bauxites by physical means of beneficiation", *Travaux de l'ICSOBA,* No. 12, 1974, pp. 34-54.
39. J. Zambo and Z. Osvald, "Aspects of industrial evaluation of bauxites in Bayer processing: Costs of processing different grades of bauxites", *Mineralogical and Technological Evaluation of Bauxites* (Budapest, VAMI-FKI, 1975), vol. 2, pp. 295-305.
40. K. Solymar, "Methods applied in Hungary for the evaluation of bauxites from the view of alumina production", *Mineralogical and Technological Evaluation of Bauxites* (Budapest, VAMI-FKI, 1975), vol. 3, pp. 213-227.
41. L. Toth, "Study of red mud settling with radiation and processing of bauxites", *Characterization and Processing of Bauxites* (Budapest, VAMI/ALUTERV-FKI, 1979), vol. 3, pp. 201-207.
42. A. Imre, "Utilization of heat of immersion, specific surface area and pore distribution measurements in the alumina industry", *Characterization and Processing of Bauxites* (Budapest, VAMI/ALUTERV-FKI, 1979), vol. 3, pp. 159-165.
43. J. Zöldi and G. Major, "Separation of vanadium salt from the Bayer cycle with CaO additive", *Characterization and Processing of Bauxites* (Budapest, VAMI/ALUTERV-FKI, 1979), vol. 3, pp. 275-285.
44. Z. G. Szabo, M. Orban and I. Perl, "Some fundamental research for alumina industry in Hungary", *Proceedings of the 4th International Congress of ICSOBA* (Athens, National Technical University of Athens, 1978), vol. 3, pp. 442-436.
45. Hungarian patents Nos. 164863 (1972), 166061 (1973) and 169641 (1974).
46. Gy. Dobos, "Processing of low-grade bauxites" (UNIDO/ITD.170).

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