OCCASION

This publication has been made available to the public on the occasion of the 50th anniversary of the United Nations Industrial Development Organisation.

DISCLAIMER

This document has been produced without formal United Nations editing. The designations employed and the presentation of the material in this document do not imply the expression of any opinion whatsoever on the part of the Secretariat of the United Nations Industrial Development Organization (UNIDO) concerning the legal status of any country, territory, city or area or of its authorities, or concerning the delimitation of its frontiers or boundaries, or its economic system or degree of development. Designations such as "developed", "industrialized" and "developing" are intended for statistical convenience and do not necessarily express a judgment about the stage reached by a particular country or area in the development process. Mention of firm names or commercial products does not constitute an endorsement by UNIDO.

FAIR USE POLICY

Any part of this publication may be quoted and referenced for educational and research purposes without additional permission from UNIDO. However, those who make use of quoting and referencing this publication are requested to follow the Fair Use Policy of giving due credit to UNIDO.

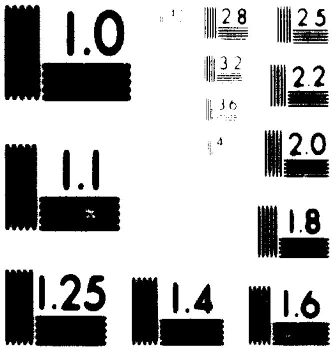
CONTACT

Please contact publications@unido.org for further information concerning UNIDO publications.

For more information about UNIDO, please visit us at www.unido.org

UNITED NATIONS INDUSTRIAL DEVELOPMENT ORGANIZATION
Vienna International Centre, P.O. Box 300, 1400 Vienna, Austria
Tel: (+43-1) 26026-0 · www.unido.org · unido@unido.org

1 OF 2



MICROCOPY RESOLUTION TEST CHART

NATIONAL BUREAU OF STANDARDS-1963-A

24x

E

Distr.
RESTRICTED

UNIDO/ITD.39
8 March 1971

ENGLISH

UNITED NATIONS INDUSTRIAL
DEVELOPMENT ORGANIZATION

MODEL LABORATORY FOR TESTING BAUXITE, ALUMINA, INTERMEDIATE PRODUCTS: PURPOSES, EQUIPMENT, METHODS AND OPERATIONS

by

Rajindra Manocha

72693

The views and opinions expressed in this paper are those of the author and do not necessarily reflect the views of the secretariat of UNIDO. This document has been reproduced without formal editing.

MODEL LABORATORY FOR TESTING BAUXITE, ALUMINA AND INTERMEDIATE PRODUCTS

INDEX

	Item	Page
1.	Motivation	[illegible]
2.	Applications of bauxite	1
3.	Salient features of bauxite consuming processes, as influencing bauxite quality demands	2
4.	Bauxite testing requirements for the Bayer process	6
4.1	Exploration test work	6
4.2	Process conditions working out	7
4.2.1	Digestion conditions	7
4.2.2	Silica sensitivity and desilication	8
4.2.3	Reversion tendency of pregnant liquors	9
4.2.4	Settling and filtration behaviour of mud	9
4.2.5	Mud utilisation	10
4.2.6	Precipitation behaviour of digested slurry	10
4.2.7	Behaviour of minor impurities	11
4.2.8	Crushing and grinding tests	12
5.	Testing requirements for alumina trihydrate and alumina	14
6.	Bauxite chemical analyses methods (excluding spectrophotometric)	16
6.1	Sampling	16
6.2	Moisture	15
6.3	Loss on ignition	19
6.4	Bauxite decomposition and dissolving	19
6.5	Total alumina	22
6.6	Alumina trihydrate	25
6.7	Alumina monohydrate	25
6.8	Total available alumina	26
6.9	Available trihydrate alumina	27
6.10	Available monohydrate alumina	29
6.11	Total silica	29
6.12	Quartz	29
6.13	Iron oxides	31
6.14	Titania	32

6.15 Phosphorus 34
6.16 Calcium oxide 35
6.17 Magnesium oxide 36
6.18 Manganese 37
6.19 Gallium 37
6.20 Organic carbon 38

7. Mud chemical analysis 40

8. Chemical methods for the liquor analysis 43
8.1 Caustic 43
8.2 Total alkaline soda 44
8.3 Alumina 45
8.4 Sodium carbonate 46
8.5 Total soda 47
8.6 Organics 47
8.7 Silicate 48
8.8 Vanadium 49
8.9 Phosphorus 50
8.10 Dissolved iron 50
8.11 Gallium 50
8.12 Chloride 51
8.13 Sulphate 51
8.14 Oxalate 52

9. Chemical methods for trihydrate and alumina 53

10. Colorimetric and spectrophotometric methods 57
10.1 Colorimetric techniques 57
10.2 Spectrophotometric techniques 58
10.3 Individual estimations 59

11. Flame spectrophotometry 73
11.1 Normal flame photometry 73
11.2 Atomic absorption spectrophotometry 75

12. X-ray methods 76

13. Mineralogical tests 79

14. Technological tests 80
14.1 Specific gravity of bauxite 80
14.2 Bulk density of bauxite 80
14.3 Digestion tests 81
14.4 Desilication tests 82
14.5 Reversion tendency of liquors 82
14.6 Mud settling behaviour 82
14.7 Filtration behaviour 85
14.8 Precipitation yield 86
14.9 Grain size testing 87

14.10 Surface area measurement 90
14.11 Microscopic examination of hydrate 91
14.12 Product purity 92
14.13 Wet screening 92
14.14 Viscosity of bauxite - liquor slurries 93

15. Filtration testing 94

16. Laboratory description 97
16.1 Field laboratory, model I 97
16.1.1 Scope 97
16.1.2)
16.1.3) Departments, descriptions 98
16.1.4 Rooms and area requirement 104
16.1.5 Staff 106
16.1.6 Training 107
16.2 Central laboratory, model II 109
16.2.1 Scope 109
16.2.2)
16.2.3) Department description 109
16.2.4 Rooms and area requirement 115
16.2.5 Staff 117
16.2.6 Training 119
16.3 Central research institute, model III 119
16.3.1 Scope 119
16.3.2 Departments description 120
16.3.3 Area requirement 123
16.3.4 Staff 126
16.3.5 Training 130

17. Acknowledgement 131

18. References 132

Tables and figures.

MODEL LABORATORY FOR TESTING BAUXITE, ALUMINA AND INTERMEDIATE PRODUCTS

Rajindra Manocha x)

Summary

This study is prepared as work paper for Committee on [illegible] of Bauxite-Alumina Testing Laboratories, set up by United Nations Industrial Development Organisation, Vienna, with emphasis on supplying information needed for detailed feasibility studies.

Following a short description of bauxite applications and salient features of the main consuming processes as influencing bauxite specification demands, various testing requirements for bauxite have been analysed both for immediate and future needs. With 85 percent of total bauxite consumed for alumina production, this aspect has constituted the centre theme. For alumina testing also, stress is on features concerned with the aluminium metal production which accounts for 90 percent alumina produced. Development of special products increases profitability but requires extensive research. Since primary requirement here, in addition to low permissible impurity levels, is that of physical characteristics, which are attained by adjustment of precipitation and calcination conditions, main interest during feasibility studies is to establish the product purity limits. The technological tests described for general precipitation behaviour study, coupled with research facilities indicated in the description of Research Institute, will cover the detailed investigation [illegible] well.

Outline of various analytical methods, both for bauxite and muds, process liquors and alumina, as needed for exploration and technological evaluation has been given. Considered are:

a) classical chemical tests, which are still most commonly used for the major constituents analyses, and also provide standard values for caliberation of new faster instrumental techniques,

b) spectrophotometric and flame photometric methods, including atomic absorption, widely used for minor impurities estimation

c) x-ray diffraction used for semi-quantitative identification of species, and x-ray spectrography, becoming increasingly popular for general analyses (elemental), specially for bauxite, muds and alumina.

---

x) S.A.V.A., Società Alluminio Veneto per Azioni, Divisione ALLUMINIO, Alumina Plant, Porto Marghera, (Italy)

d) Microscopy and differential thermal analysis for bauxite and precipitation studies.

Examined for each above case need and scope for individual application.

Section on "Technology Tests" includes such laboratory tests, which help to establish broad process parameters for final pilot plant studies. These tests establish quality variation of the deposit, help investment decisions on required flexibility at different stages. A well conducted program, statistically planned and evaluated, can shorten the subsequent expensive pilot plant trials considerably.

For pilot plant trials scope considered are

a) overall state of known art of bauxite processing

b) specialised assistance available
from potential equipment vendors (working on competitive basis!)

c) relatively small influence, under practical circumstances, of the equipment cost in overall plant cost

d) risk factor from basic process complication during the plant start-up

f) cost of establishing the facilities and operation.

Continuous run pilot plant trials are needed to establish the overall viability of the process and forestall unexpected extremely expensive complication during the plant start-up. The facilities will correspond to an almost miniature alumina plant, very well instrumented, manned by a skilled operating and maintenance gang. Thus unless the pilot plant can be located next to an operating plant, it will be better to have the feasibility studies conducted at an already established centre and wait to build one's own after the first plant is built. This pilot plant built at a later stage would profitably serve to investigate other deposits, make changes on the running plant without production loss risk and also develop special products. Criteria for selecting the outside party for test work have been listed, and also some general considerations in making the lay-out.

The report ends with description of facilities and requirements of area , staff and training for 3 models of laboratories to suite different circumstances.

[illegible] or [illegible] a file [illegible] needs of [illegible] ation teams to [illegible] to analyse [illegible] for major components [illegible] of [illegible] staff, 4 [illegible] 2 [illegible] and Assistant [illegible] Chemist.

[illegible] to a central lab[illegible] the technological [illegible] the ana[illegible] [illegible] gh [illegible] e ision [illegible] ied [illegible] Assistant Chief [illegible] Ana[illegible] administrat[illegible]

[illegible] with good research potential [illegible] methods [illegible] industry, and [illegible] will be advantageous to [illegible] central [illegible] to meet [illegible] of [illegible] and physical [illegible] work [illegible] ng- [illegible] al condition [illegible] ties. The description [illegible] re- [illegible] is [illegible] base [illegible] the [illegible]

[illegible] consist [illegible] d [illegible] velop[illegible] ve process [illegible] for [illegible] production [illegible] and equipment [illegible] ess [illegible] plant [illegible] and [illegible] improve process economics and meet more stringent [illegible] on to reduce [illegible]. There [illegible] ra[illegible] laboratory. [illegible] microscope will be [illegible] at a [illegible]. A small service [illegible] to take care of [illegible] n and elementary equipment maintenance and modification [illegible] be [illegible] ped at a later stage to [illegible] as the pilot plant programs grow. Library, stores and purchase.

Staff requirement is detailed in [illegible] (covered) area 15,000 sq ft ex[illegible] ding 10,0[illegible] sq [illegible] for continuous [illegible] circuit, [illegible] regular operating p[illegible] including [illegible] for [illegible] technological service and p[illegible] plant (disc[illegible] steps) wo[illegible] and only [illegible] sq ft for the service shop.

A detailed index is given for ready reference to details.

MODEL LABORATORY FOR TESTING BAUXITE, ALUMINA, INTERMEDIATE PRODUCTS: PURPOSE, EQUIPMENT, METHODS AND OPERATIONS

Rajindra Ma[illegible]

1. Motivation

The next few years will see a phenomenal growth of aluminium production, a safe 8 percent exponential rise, with almost corresponding rise in alumina production, most of it produced from bauxite. The developing countries which own about 70 percent of the world bauxite deposits have obviously great benefits to reap if they are aware of various application possibilities.

Much of the aluminium producing facilities will be located close to the metal consumption centres, mostly developed countries, under the pressure of marketing trends and ever-present regional protective tarrifs. Presently, 90 percent of aluminium production comes from such areas. In such circumstances, the alumina industry in developing countries will do well to be export oriented till their indigeneous metal markets grow large enough. Export ambitions carry with them challenges of maintaining low production costs and high dependability. The cost considerations favour large size operations, in which case choice of right type of technology and processes compatible with indigeneous conditions, and rational selection of safety factor for the desired reliability become critical. A large safety factor chosen on the basic process parameters out of panic in the absence of sufficient data increases the costs at every level.

The single important variable element influencing the basic process parameters of alumina production is the nature of bauxite mineral. Thus the importance of an early establishment of adequate facilities to provide such knowledge cannot be overemphasised for the developing countries, hardly any of which can boast of an effective one so far.

2. Applications of Bauxite

Typically, bauxite is a weathered product of aluminous rocks, consisting mostly of aluminium and iron hydroxides, though some depo-

<u>Note:</u> The opinions expressed herein are strictly personal and need not represent any particular official thinking.

ites contain substantial titanium-oxide minerals. Major common impurities are aqueous aluminium-silicates, mostly kaolinites, quarts and limestone/dolomite. Other minor impurities are organics, oxides of vanadium, gallium, zinc, sometimes nickel, chromium, manganese, phosphorus, fluoride and arsenic. The bauxite is valued mostly for its alumina content.

The principal uses of bauxite may be listed as:

1) Alumina production for making aluminium.

2) Refractories.

3) Chemicals, mostly alums and sulfates, used in water treatment, chemicals and pharmaceutical industry.

4) Direct production of aluminium alloys.

Over [illegible]5 percent of total bauxite produced is used in alumina production. The trend of such large proportion going into alumina production will continue despite interesting developments in production of aluminium-silicon alloys direct from bauxite and aluminium by chlorination route.

The bauxite grades for various applications are determined by the nature of impurities, the means available for their removal and possible alternates, the latter factors explaining for large specification differences from country to country. Indian convention is given below for an example 2)

Grade	Al_2O_3 %	SiO_2 %	Fe_2O_3 %	TiO_2 %
Metal	51	1.8–2	6–11	8–11
Chemical	57	1.7–4	1.5–3	5
Refractory	50	1–5	4	12
Abrasive	50	8	15	2
Steel	59–61	2.5	3.5	10

3. Salient Features of Bauxite Consuming Processes as Influencing Bauxite Quality Demands

3.1 Soda Leaching (Bayer and Combination Processes)

Practically all the alumina is produced by leaching out alumina content of the ground bauxite in the hot with caustic alkali, commonly called Bayer process. Among the impurities, iron and titanium-oxides remain almost unattacked. Silica, other than quartz, mostly dissolves and then gets precipitated out in various phases of operation taking along with it an equivalent amount of alumina and soda. Vanadium- and gallium oxides partly dissolve, to the extent depending on the nature of the mineral and severity of the leaching attack. The organates degenerate into various organic acids and salts, such as oxalates and carbonates, at the expense of soda. Limestone reacts to convert active caustic soda to inactive sodium carbonate.

The alumina containing liquor called "pregnant liquor", is characterised by an exceptional degree of supersaturation and has to be conducted under carefully controlled process condition designed to minimise alumina precipitation during the separation of insoluble impurities, called "mud", by decantation and filtration. The alumina is then recovered by cooling the impurity-free liquor and agitating it with earlier precipitated alumina trihydrate seed. The precipitation process is one of the critical steps in the alumina plant flow sheet development, because the precipitated hydrate has to have definite grain size and structure to enable its economic separation from the spent liquor, provide just sufficient seed of the desired quality, and yield the finished calcined product according to set physical and chemical specifications. This has to be done with minimum compromising of conditions conducive to maximum yields, all this excercise requiring detailed knowledge of bauxite composition.

The precipitated alumina is then separated from the mother liquor, called spent liquor, by decantation and filtration, and calcined in rotary kiln to give the product alumina. The spent liquor is, recirculated to leach further quantity of bauxite after evaporating out, if necessary, the extraneous water introduced from the bauxite (moisture content and water of hydration of the alumina oxides) and that used for soda recovery from the mud and the precipitated alumina hydrate. A causticisation process consisting of treatment with slaked lime, is included to convert the inactive sodium carbonate formed in the process back to caustic soda. Certain plants produce consistently from the spent liquor substantial quantities of vanadium salts and gallium as bye-products.

In the production costs, raw materials constitute the largest single item. Bauxite and caustic soda generally account for 80 percent of the raw material costs.

Caustic soda is often the leader where bauxite is not transported over long distances. About 70-80 percent of the total caustic loss comes from silica content of the bauxite. Besides, silica leads to another important source of loss by tying up an equivalent amount of alumina making it unavailable. Thus silica content has long been accepted as a master criterion of bauxite acceptance.

Alumina industry continuously works to develop processes and modifications which will allow economic working of higher silica containing ores, because the tonnage of bauxite resources increases very fast with every single percent increase in the silica content acceptability. Thus the silica acceptability levels have continuously risen and bauxites containing 4 – 6 percent silica are being freely used. Some American plants use even 10–15 percent silica-containing bauxites. The success has come from two sides (a) better unterstanding of the reactivity of alumina and silica content of the minerals, and the desilication process which prevents silica contamination of the finished product (b) development of the so called combination processed which recover combined soda and alumina of the mud by calcining sintering it with soda and limestone. Direct sintering of high silica containing bauxites with limestone and soda followed by aqueous leaching is also being considered. An important requisite for favourable economics of such processes is the nature of sintered product, which should require minimum grinding and be relatively free of dust in preparation for the subsequent leaching step. Ideally, the sinter should be self disintegrating. Identification of the mineral constituents involved and studying the possibilities of their interphase reactions at various temperatures could make remarkable breakthroughs.

Increasing attention is also being given to other possible beneficiation processes to separate silica impurities.

Mention may also be made of growing interest in diasporic bauxites which were generally considered unsuitable for alumina production because of their very poor solubility in practical range of caustic concentration and temperatures. In some cases, heating of ores to 600 – 700 °C leads to certain phase transformations making them reactive and easily soluble in caustic liquors. Sintering with soda also makes the alumina contents soluble. Of great potential practical interest in the same context is the pilot plant success reported on high temperature bauxite digestion (at 280 °C and above) 3).

3.2 Acid Process

The unsuitability of alkali leaching process for higher silica containing bauxites has led to interest in sulphuric acid leaching processes. Silica remains generally insoluble. Objectionable impurities are oxides of iron, alkali metals, titanium and limestone which consume the acid and need more involved chemical separation procedures for their subsequent separation. The precipitated aluminium salt is basic aluminium sulphate, which [illegible] calcined to give the finished product alumina. The released [illegible] gases are regenerated by absorption in the spent liquor [illegible] suitable for recycle and fresh attack of the bauxite. The area requiring critical study is the basic sulphate precipitation process.

3.3 Refractories

Calcined bauxites are used largely for making high alumina refractories.

For chemical composition, important considerations are low contents of oxides of iron and alkali metals which reduce the refractoriness of the material and also its resistance to carbon monoxide attack. Titanium content will also become an consideration in view of recent evidence of a low melting titanate phase (melting point 1300 - 1350 °C) with consequent big loss in mechanical properties [4], and catalysing action of titanium oxide on carbon manoxide dissociation responsible for the brick disintegration.

The mineralogical properties are important for their influence on shrinkage characteristics and [illegible] transformations. Bauxites, that give consistently dimensionally stable calcined product in furnaces operating at 1200 - 1400 °C are considered extremly desirable. Diasporic ores are preferred.

3.4 Production of Chemicals and Pharmaceutical Products

Most of the methods depend on acid attack of the minerals, though chlorination processes will be assuming importance.

For acidic processes, low contents of iron, titanium and limestone are important. Being relatively small production units, only high grade bauxites, containing iron and titanium oxides less than 3 - 5 percent, are used. Reactivity of the mineral is an important consideration.

3.5 Direct Production of Aluminium Alloys

With fast growing applications of aluminium silicon alloys, very promising outlets are expected for direct smelting of high siliceous bauxites in regions of cheap electrical power. The processes consist essentially of slagging out the impurities under controlled reducing conditions leaving behind aluminium silicon alloys. For chemical composition, important will be alumina-silica ratio, titanium and iron oxide content to determine the fluxing requirements. For physical characteristics, the nature of the minerals of aluminium, iron, titanium and silicon, which influence the reducibility and formation of desirable phases necessary for neat slag/metal separation.

4. Bauxite Testing Requirements for the Bayer Process

Attention is restricted here to only those types of investigations, which are needed to define the technology and equipment required in the framework of feasibility studies on the necessary investments and operating costs. The nature of such investigations has been described in the usual order of precedence required in assessment for the alumina industry.

4.1 Exploratory Test Work

First and foremost is establishing the extent of the deposit in terms of its silica and alumina content.

Bauxite deposits are notorious for their irregularity, and sampling grid patterns are chosen conservatively. Large number of samples have thus to be analysed initially for bulk density, moisture content, alumina, iron, titanium, silica and loss-on-ignition till a well-trained eye and some indirect easier method of grading by statistical techniques is developed. *)
For test work here, emphasis is on speed rather than high degree of accuracy. A limited number of samples will have to be analysed for other impurities, perhaps at a later date.

Existence of inert diasporic bauxite has to be established immediately because in the event of substantial presence of this constituent many flow sheets will be elimineted, narrowing down the scope of further studies substantially.

*) In many cases, loss-on-ignition values have provided fair first approximation index. In some cases, specific gravity determination proved quite indicative.

Next in importance is establishing the presence of boehmite, monohydrate form of alumina, because this again narrows down the possible scope of future process investigations and provides a first rough indication of expected investment. Monohydrate-containing-bauxites require more drastic leaching treatments and equipment for their processing. And because of severe attack requirements, bauxite silica behaviour also becomes more important.

In the event of massive deposits occuring in the vicinity of clayey materials, or the excavated material being contaminated with mud, washing tests will be desired to investigate possibility of its separation by straight-forward washing. This would require extensive wet screening tests.

For high moisture containing ores required for export, it may be necessary to calcine them for drying at the site. Since considerable significant transformations are known to occur on heating 6), mineral and phase transformation studies by the T.D.A. method, X-ray and microscope along with digestion tests will be needed for different temperature treatments.

4.2 Process Conditions Working Out

Next come more detailed investigations required to fix optimum process parameters of caustic concentration and temperature which have strong interactions at various stages and largely determine equipment size specifications.

4.2.1 Digestion Conditions

First is chemical reactivity of the bauxite which varies considerably from one bauxite to another despite comparable chemical compositions. It is determined by finding alumina yield on bauxite digestion in autoclaves at different temperatures, caustic concentrations, grind size and residence time selected by statistical methods of experimental design. Refinements include contaminating the digesting liquors with the expected impurities accumulating on continuous liquor recycle in the plant to evaluate their modifying effect on bauxite extraction.

An attempt is made to determine kinetic equations from the extraction data to improve predictability under wide range of conditions. This involves analyses of the liquors for alumina and caustic because most of the bauxites reaction kinetics respond to some function of chemical potential represented by the degree of departure from equilibrium solubility of the concerned alumina hydrate at corresponding caustic concentrations. Sodium carbonate and silicate content of the liquors are analysed to determine the amount of caustic lost in desilication and non-alkaline soda formation and that converted into sodium carbonate. Alumina yield is determined by analysing alumina in the residue mud and calculating quantity of the mud produced by comparing the iron oxide contents of the mud and the bauxite, iron oxide being generally inert in caustic.

Mineralogical examination under the microscope will be done at different mesh-of-grind sizes for possible detection of free quartz. The digestion conditions have to be viewed for their interaction on the desilication process, behaviour of the mud during settling and filtration, premature decomposition, called reversion, of the pregnat liquor during mud removal, and permissible caustic concentration at the precipitation stage. Also, for solubility of other bauxite impurities like titanium, vanadium, gallium, zinc and phosphorus for likely alumina product contamination.

4.2.2 Silica Reactivity and Desilication

Studies on solubility behaviour of silica and subsequent liquor desilication will require liquor analyses for soluble silica content. Rate of silica dissolution and its subsequent separation will have to be examined under different combinations of caustic and alumina concentrations and temperatures. The desilication behaviour and kinetics are notoriously individualistic. Faster method of analyses will be real asset. Chemical and X-ray analyses will be required for identification of the desilication product produced in the mud, in search for the conditions where soda and alumina tied up in the desilication product is relatively less.

In the circumstances, where cheaper bauxite is available or the bauxite contains excessive silica, or caustic soda is expensive, cheaper plants can often be built by accepting less drastic digestion condition (sacrificing some alumina yield thereby) <u>if</u> under such conditions silica impurities showed marked reduction in their reactivity, and residual unreacted alumina did not promote excessive reversion.

In the case of higher silica bauxites, their behaviour on heating will be examined for likely possibility of some reactive silica mineral becoming relatively inert.

4.2.3 Reversion Tendency of Pregnant Liquors

The reversion sensitivity of the pregnant liquor in the mud removal stage is established by prolonged (gentle) agitation with muds containing different amount of undigested bauxite and analysing the liquor and the mud for alumina. Excessive reversion can make mud filtration impossible, apart from the alumina loss it entails.

Reversion tendency of the liquors is one of the important factors influencing alumina yield of the liquors and is influenced by the nature of minerals. The degree of liquor contamination as resulting from enrichment of the various impurities during continuous liquor recirculation influences the results significantly and must be considered.

4.2.4 Settling and Filtration Behaviour of Mud

Besides the fact that the liquor clarification facilities account for about 15 - 20 percent of the plant investment, the dependability of the final pregnant liquor filtration is critical because any mud passing from this section will positively contaminate the product and the seed, and the filter performance is strongly influenced by the clarity of pregnant liquor received from the settler. The settling and filtration characteristics of the mud are very individualistic, reacting differently to bauxite grind size, digestion treatment, caustic concentration, flocculents and solid content of the slurries. In some cases, there may be sharp critical points in their behaviour and digestion conditions may have to be modified in the overall interest.

Settling characteristics determine the settling area requirement for various degrees of mud consolidation.

Filtration characteristics determine the filtration surface required and the practical deliquoring limits.

The above information establishes the most economical combination of the two types of equipment from estimate of the capital cost and soda recovery for various alternates.

Increasing attempts are being made to improve the mud behaviour predictability from the knowledge of bauxite characteristics and flocculating characteristics of the muds by X-ray and mineral examination techniques. For example, presence of goethite is often associated with poor settling muds. Presence certain mineral is reported to upset the filtration drastically. Muds from the bauxites of colloidal origin behave very much differently from the other types. In case of high silica containing ores, coating of mud particles with thin light films could suppress their flocculating capacity or alter their "density" substantially, which might be detectible with improved microscopy techniques. Liquor phosphate concentration is often a suspect during filtration problems on pregnant liquors.

4.2.5 Mud Utilisation

Huge quantities of mud are produced in alumina industry, about 80 - 100 percent of the actual alumina produced.

In case of high silica bauxites, mud is to be examined for recovery of alumina and soda values, as mentioned in section 3.1 for the Combination Processes. Otherwise too, as mud disposal sometimes accounts for 2 - 5 percent of the alumina production costs, good incentive always exists to find an interested party. Its properties of interest are high titania content for titanium rich ores, high iron content, high soda and alumina content for high silica ores, red colour for pigment industry, absorptive power for gas purification industry.

4.2.6 Precipitation Behaviour of Digested Slurry

After the bauxite digestion tests most critical for the process design is precipitation behaviour of the liquor produced under different digest conditions.

Objectives in the precipitation process are to obtain maximum of yield, obtain a product of such grain size, that it can be readily separated and after yielding the fraction of required specification for the product alumina, enough hydrate is left for the seeding operation. Besides, grain structure of the precipitated hydrate should be such that it can be economically washed to yield a product of the desired chemical purity. Many of these demands are contradictory. For example, process conditions that help ready recovery of the hydrate militate against those promoting high yields. So compromises have be accepted.

Precipitation kinetics and nature of the precipitate produced being very sensitive to the liquor impurities, especially organics, detailed liquor analyses become important. Once the impurities are identified, their effect on equilibrium solubility of alumina at the concerned caustic considerations will be examined to predict their deleterious effect on the rate of alumina precipitation influenced by the function: (liquor alumina concentration at time t - equilibrium liquor alumina concentration) 2.

Different types of organates are produced depending on digestion conditions, and they have different influences on the precipitation behaviour. Where-as some of these slow down precipitation kinetics by raising the equilibrium solubility of alumina in caustic, others adversely effect only grain growth processes. Further, the magnitude of their effect is strongly influenced by the precipitation caustic concentration. Correct identity of the influence of these contaminants is thus needed for establishing the precipitation conditions.

The importance of such studies will be appreciated from the fact that the entire process conditions and thus the size of the equipment and unit operations are determined largely by the two terminal conditions of digestion and precipitation steps and large numbers of combinations are possible for selecting the optimum.

The liquor analyses will include estimation of silica, iron, titanium, carbonate, sulphate, chlorides, zinc, vanadium, gallium and different types of organates. On the precipitated products, besides the tests for impurities, sieve analyses, surface area estimation, decantation tests and microscopic examinations will require to be done, as further noted in section 5.

4.2.7 Behaviour of Minor Impurities

Included generally are organics, vanadium, gallium, phosphorus, limestone, dolomite, zinc, manganese, fluoride, nickel, arsenic, beryilium, meroury, sulphur. These are termed minor because their presence rarely exceeds 1 percent and though their presence could certainly demand importantprocess modificatións or supplementary steps as a result of enrichment in liquors from repeated circulation, yet it would not rule out an establishment of an alumina industry if major considerations outlined earlier are favourable. However, for bauxite exports to the running plants, many of these impurities can be critical.

In design of new facilities, the effect of these impurities is examined for the following.

Increase in equilibrium solubility of alumina which reduces the precipitation rate (section 4.2.6).

Product contamination from supersaturation. Change of desilication characteristics 6) and mud settling characteristics.

Reduction in the hydrate precipitate grain size to the extent that it cannot be recovered by the provided facilities.

Excessive foaming.

Besides some of these impurities could be turned into roal assets in terms of valuable by-products, as for example. vanadium, gallium.

The degree of variation of these impurities in bauxites and the magnitudes involved are seen in table I.10) Table II shows an example of impurities distribution in a plant liquor.10)

Organics consitute perhapes most important single minor impurity for their effect on process at most steps. In bauxite digestion, these degrade into various inorganic and organic compounds. Though soda loss from this reaction is small, the compounds produced when present above certain critical concentrations have been known to upset drastically desilication, mud settling and precipitation steps. So much so, that if sufficient provision for keeping them at a controlled level is not provided, the plant capacity could easily fall by 30 percent. The testing would require the liuqor analyses to identify the trouble causing compounds at various stages to provide economic counteracting measures.

Phosphorus is partly reactive to caustic soda, so causes soda loss in digestion. In the soluble form, it can cause some mud filtration problem and sometimes contaminate the final alumina product to the detriment of current yield during aluminium production.

Vanadium also reacts with caustic to varying degrees. Above contain concentration level, it can lead to objectionable product contamination by precipitating along with alumina. As an operating nuissance, it could precipitate in pipes and tanks even before it affected the product quality significantly. Vanadium impurity can be turned into an asset by recovering this from the plant liquors as by-product.

Gallium causes apparently no process complication from its dissolving in soda. Expensive as the gallium metal is ($ 1 - 2/gram) profitable by-product industry for gallium recovery could perhaps be made possible in certain circumstances.

Limestone, dolomite convert part of active caustic soda to inert carbonate. Suitable provision for causticising the extra carbonate formed has to be provided in the plant facilities. In sintering processes, their fluxing behaviour is very important.

Zinc is objectionable because it dissolves and precipitates with alumina to become objectionable impurity. A zinc separation treatment on the digested liquors has to be included in the flow sheet.

Fluoride is mostly of nuissance values in hard scale formation in tanks and pipes. It precipitates out along with vanadium salts.

Iron: Directly affects the alumina product purity, which is objected for some aluminium end uses.

Mercury is not known to upset the process in any way. In certain high temperature digestion practices, elemental mercury has been obtained in the flashed steam during cooling of the digested slurries.

Arsenic. Most of it ends up in the mud as insoluble constituents.

4.2.8 Crushing and Grinding Tests

Once the optimum grain size is established for the bauxite digestion, grinding and crushibility characteristics of the mineral are determined. These, generally expressed as the grindability index and the work index, provide good indication of the equipment required and the involved costs.

Grinding index is generally determined by preparing 35 MOG of the bauxite (80 percent passing 35 mesh screen) by a batch, closed circuit grinding technique in a small ball (or rod) mill, say 8 x 10". Grindability is judged from the slope of Schumann plot drawn from screen analysis of the product -mesh size of the Tyler series as the abscissa on the Cartesian Co-ordinate of semi-log paper and accumulated weight percent finer than the stated mesh size as the ordinate. This plot is linear if the material has been ground in an unbiased manner.

5. Testing Requirements For Alumina Trihydrate and Alumina

Testing requirement considered here are those needed for eventual feasibility studies on bauxite utilisation. As for any other material, these are determined by eventual application of the alumina produced.

Though about 90 percent of the alumina produced is used for the metal production, profitability of the alumina plants can be improved by producing some special varieties of the alumina and trihydrate. The special products are prized by paper, rubber and pigment industry as fillers and coating pigment, by pharmaceutical industry as bodying material and for medicinal purposes, by chemical industry as starting point for organic and inorganic chemicals specially alum, anhydrous aluminium chloride and cryolite, as catalysts, absorbent for chromatographic and filtration applications and by ceramic and refractory industries for special high temperature and high dielectric resistance insulators, certain high duty service bricks and corundum production.

For alumina for metal production, two criteria are used, chemical and physical. Two typical specifications are given in table III. Chemical criteria define the upper impurity limits, these limits being critical because the very nature of aluminium production process is such that the impurities present in alumina cannot be removed. Excessive soda content and loss-on-ignition (representing undecomposed hydrate) contribute to the raw material costs in aluminium production through use of cryolite and aluminium fluoride.

Physical criteria are based on consideration of solubility behaviour of alumina in electrolytic cell baths of the aluminium smelters, and its dusting and flowing characteristics.

Two distinctions are in vogue;

a) sandy alumina
b) floury alumina

Sandy alumina is characterised by free flowing characteristics, closer size distribution, coarseness and dusting tendency.

Floury alumina is marked by relatively poor flowing characteristics, wide size distribution, fine grain size, less dusting tendency and character of a well calcined product with low loss-on-ignition.

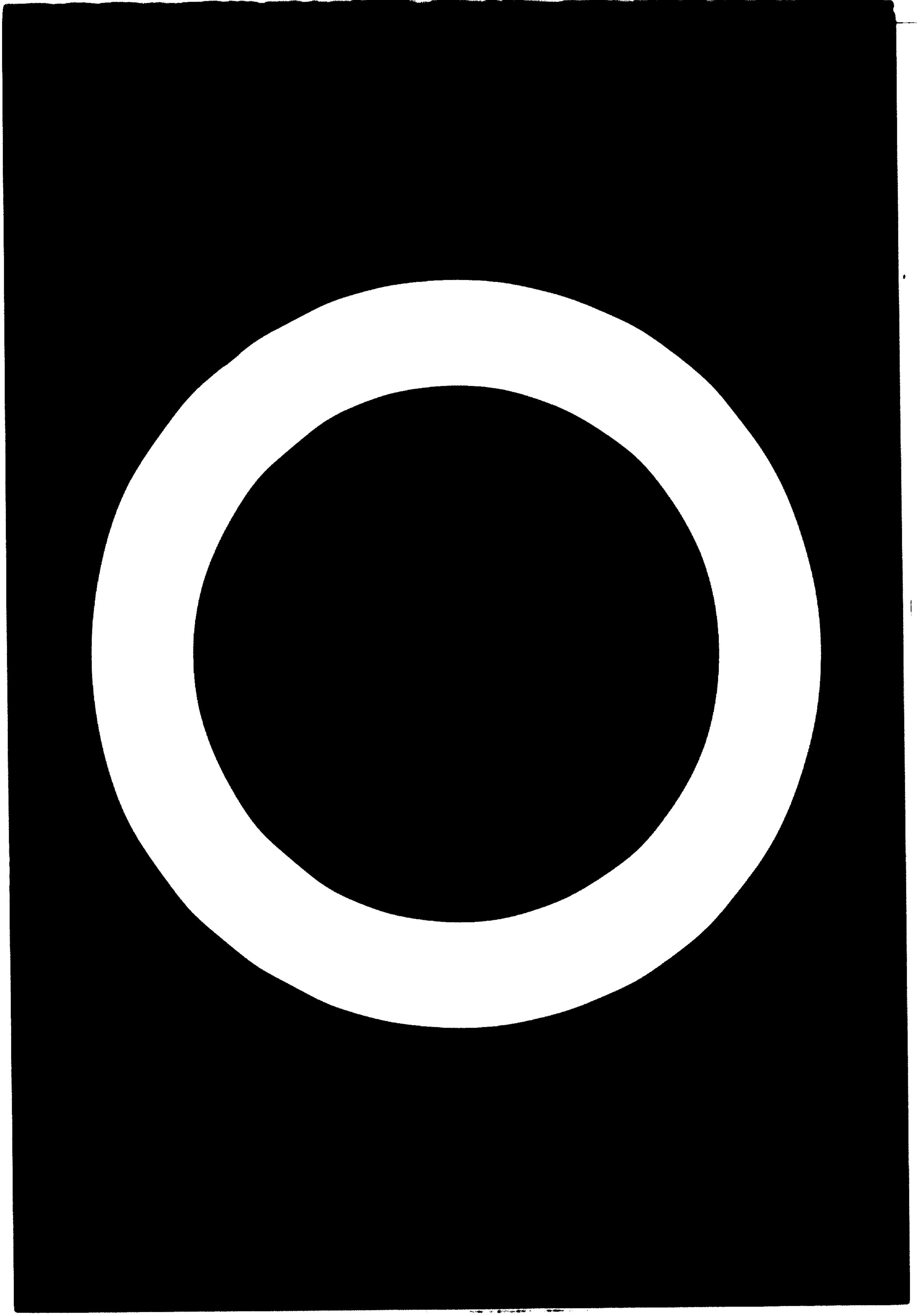
These different physical conditions are realized by adjusting the precipitating conditions and calcination operations.

Though the aluminium producers have always had very strong individual preferences based on their experiences with the electrolytic cells, gradually the emphasis in judgement criteria is shifting to the economics of the alumina production which have been treated in discussions on bauxite. However, it has to be remembered that alumina handling facilities for the two types of aluminas can be considerably different and will have to be given due weightage in the final decisions.

The special alumina products are distinguished more for the physical characteristics, such as density, size distribution, shape and nature of the individual crystals or agglomerates, rather than the chemical composition, except that in most cases, specially low limits are imposed on sodium, iron, titanium and vanadium contents. Here also physical properties are controlled by adjusting the precipitation operating conditions and calcination conditions. Starting point is the hydrate produced.

From the above it will be noted, for the bauxite feasibility studies principal interest in the testing of alumina and the trihydrate is checking for the precipitated hydrate contaminations and washability of the hydrate to remove soluble impurities, especially soda. This will be done as a part of the precipitation studies outlined in section 4.2.6. Needed will be size distribution and surface area determination of the hydrate precipitate and configuration of the crystals and conglomerates under the microscope.

Sophisticated equipment like electron microscope will be needed for special product development.



6.1.1 Field Sampling

In field sampling of bauxite where the size range is wide and mineral very heterogeneous, serious errors result from accidental classification by size or gravity, and different size fractions usually differ considerably in critical constituents like silica and alumina. The risk of accidental classification in the phase of reducing sample volume is particularly pronounced with dry materials. A typical sampling procedure adopted in such cases is described below for a half ton sample collected from a test pit.

The underlying principal is that splitting is done over narrow size ranges.

> The original pile is divided into two lots - one for plus 3" size lumps and the other for smaller ones.
>
> On the coarser, plus 6" pieces are broken by the hammer to minus 6" size without attempting to break them into smaller pieces. The entire pile is then divided into two equal lots on the basis of equal number of lumps. One lot is rejected thus reducing the bulk volume by half.
>
> On the other minus 3" size pile, it is split into two parts by coning and quartering. One part is rejected thus reducing this bulk volume by half.
>
> On both the halved portions of the original coarse and fine piles, larger pieces are individually reduced to minus 2 inch size, followed by mixing together the two piles. The combined pile is then reduced to half the size, about 250 pounds, by the standard method of coning and quartering.
>
> Larger pieces in the above material are then reduced to minus one-half inch size by hand, or by jaw crusher where a small one is available, and reduced to about 7 pound size by usual coning and quartering procedure.

On wet samples the bauxite will have to be dried before effective volume reduction can be done. This is generally done by spreading the material on a clean metal sheet heated by gas burners.

6.1.2 Test Samples

Reducing the bulk volume further to that required for the laboratory is conducted by usual methods of coning and quartering or by rifflers when a crusher (laboratory jaw crusher or rolls) is available to reduce the material to minus 6 mech.

For chemical analyses, it has to be further ground, 100 MOO (passing 100 mesh Tyler screen) generally adequate. However, while looking for minor impurities, or contaminations like quartz which can exist as individual particles, finer grinds may be required. Serious errors can result during above grinding and sample volume reduction, unless following basic considerations are respected.

1. To grind a sample only little at a time to avoid moisture loss from the heat of grinding. The sample is ground, sieved, the operation being repeated on the residues.

2. To avoid loss of representativity during grinding from bias in favour of softer or harder fractions of the mineral by taking all the powder. And also when the fractions passing through the sieve on repeated grinding are mixed, it should be done on a smooth and flexible surface such as glazed paper, rubber or oil-cloth, in such a way that the material in made to roll over and over and does not merely slide along. As many as 100 such operations are recommended for each diagonal action.[11])
   Thus during crushing to minus 6 mesh, the crusher is so adjusted that some particles are retained on the 6 mesh-screen, thereby increasing the amount retained on the finer sieve. The coarser fraction is recrushed until it passes.
   Ultimate fine grinding is done with mechanical grinders, in absence of which agate mortor is used. While using mechanical grinders on extremely hard ores, possibilities of minor contaminations from iron, molybdenum, nickel and chromium should be remembered, though in practice they have been generally found insignificant.

6.2 Moisture Content

Moisture content is defined as that part of water which is physically adsorbed as distinct from that chemically bound such as water of hydration.

According to international convention, all the bauxite analyses are reported and compared on dry basis. In cost transactions, generally based on tonnage, this information is vital.

The detailed procedures differ with the physical characteristics of the ores.

For the dry non-hygroscopic ores, the sample prepared as outlined in section 6.1.2, is dried in an oven to constant weight at 110 - 140 °C. The temperature chosen is such that no organic matter or hydrate of aluminium or iron, limestone or sulfur comound is decomposed and iron oxides are not transformed. Indian standards Istitution prescribes 140 °C 11) which looks reasonable. Use of lower temperature will require longer time for the test.

Sample taken is about 5 - 10 gram. For wet hydroscope ores, the moisture determination should be done in the shortest possible time. The larger pieces in the collected samples are broken only to a pea-size with the hammer and loss in weight determined at 140 °C. The weight of the sample taken for drying is about 50 gram.

6.3 Loss on Ignition (LOI)

To determine all the water of hydration of alumina.

After a few determinations of alumina on a bauxite deposit, this simple quick determination often proves a very useful indication of quality not only with respect to alumina content but also sometimes to the relative occurrence of monohydrate and trihydrate forms of alumina. Possible errors are, inclusion in water of hydration of the losses from decomposition of the organic matter, iron hydrates, limestone, kaolinites etc. but they are generally small.

The sample, as prepares in section 6.1.2, is ignited in platinum crucible at 1100 °C for one-half hour in a furnace or on mecker burner taking care to heat it gently in the beginning and raising the temperature gradually so as to avoid any physical losses from spurting. The ignited sample is cooled in the dessicator and weighed. Though some practice insist on use of expensive magnesium perchlorate as dessicant, 11) others consider activated alumina good enough.

The LOI is generally reported on dry basis i.e. after correcting for the initial moisture determined separately as in section 6.2. In some cases, as per Indian Standards Institution 11), LOI is reported on the original basis, i.e. without correcting for the moisture content.

6.4 Bauxite Decomposition and Dissolving

The first step common to all chemical analytical procedures, consists of decomposing the mineral to get its components of interest into solution - called "main solution". These processes fall into two broad categories depending on the composition and reactivity of the constituent minerals – acid attack and alkali attack, generally fusion. The complete extractability of the concerned constituents is generally checked by X-ray diffraction. For minor impurities, alkali fusion is generally required.

6.4.1 Acid Attack

Acid attack is generally preferred because of the overall ease in handling of liquids and reduced chances for errors in routine analytical work. Also silica dehydration, required for silica estimation, proceeds faster and is more positive. Mostly, mixture of sulphuric-, hydrochloric- and nitric acid is used whereby the metallic elements are converted to the respective sulfate, leaving insoluble silica behind. Sometimes for estimation of calcium and insoluble quartz, hydrochloric acid and phosphoric acid attack are respectively adopted as described later. However many bauxites cannot be readily decomposed by acid digestion, as for example, diasporic ores and high titania containing ores. It is generally not trusted for estimation of minor impurities.

Method

1 – 2 gram finely ground bauxite, as prepared under section 6.1.2 is treated on a hot plate with mixture of sulphuric, hydrochloric and nitric acids different proportions being used by different groups. Nitric acid serves to decompose the organic contaminations, lime-stone, and oxidise any ferrous iron to the ferric state, which is desirable subsequently. For complete decomposition it is important to heat gently before leading to vigorous fuming. The residue is baked for one quarter to one half hour to dehydrate silicic acid and produce readily filtrable silica. The cooled residue is then carefully dissolved in boiling water, using only a small quantity of cold water in the beginning. Too vigorous and prolonged a boiling could lead to precipitation of some difficult-to-redissolve basic salts, especially in the excessive presence of titanium minerals.

The insoluble residue consisting mostly of silica is then filtered out and washed free of acid with hot water, using only small quantities of water every time to avoid any hydrolysis of the entrapped liquor. Pre-heating of the solution on hot plate for 30 – 60 minutes can accelerate filtering considerably. In no case should the solution be kept unfiltered for long because some of the insoluble silica can become soluble, causing interference during subsequent estimations. Medium pore-size filters are recommended. The clear solution is made up to 250 or 500 ml.

If the residue was red, it contained undissolved substances or basic iron sulphate formed from excessive sulphuric acid. While the latter can be easily dissolved by washing with hot dilute sulphuric acid, former will require alkali fusion for decomposition.

6.4.2 Alkali Fusion

Decomposition of bauxite with alkali fusion is more positive, and often recommended for estimation of minor impurities and those containing high silica.
Following alkali mixtures are generally used.

a) Sodium carbonate.
b) Sodium carbonate potassium carbonate mixture.
c) Sodium hydroxide.
d) Sodium hydroxide and sodium peroxide mixture.
e) Sodium carbonate-borax mixture.
f) Lithium carbonate-borax mixture.

Action of sodium carbonate and that of sodium hydroxide is about similar except that sodium hydroxide is considered by some to be more positive and necessary for effective decomposition of some bauxites. Carbonates have the advantage that they are readily available in very pure forms. For caustic fusions, a blank run is a must.
Mixture of sodium and potassium carbonate is preferred in certain cases to use of sodium carbonate alone for avoiding complication of chlorides crystallising out in hydrochloric acid medium. Besides the mixture has lower fusion point.
Sodium hydroxide and sodium peroxide mixture fusion provides decomposition in oxidising conditions and is extensively used for analysis of minor impurities, because aqueous extraction of the fused mass gives quite a straight forward separation of iron, titanium, manganese (in presence of alcohol), magnesium and calcium which remain on the filter from alumina, vanadium, phosphorus and chromium which go into the filtrate. Organics are decomposed.
Sodium carbonate and borax mixture fusion is required for the bauxites extremely difficult to decompose - very seldom needed.
Lithium carbonate borax mixture is used for bauxite decomposition to prepare samples for X-ray spectrometry.

1-2 g of finely ground bauxite are taken and fused with the selected alkali at 900-1000 °C for 30-45 minutes. Platinum crucibles are used for alkali fusion, nickel crucibles with caustic soda. For caustic soda fusion the soda should be first fused to dehydrate it well. Intimate mixing of the reactants before and towards end of the fusion operation is important.

The melt is carefully dissolved in about 200 ml of 10 percent sulphuric acid at slow heat. The solution is evaporated to the evolution of copious white fumes lasting for about 15 minutes, to complete insolubilisation of silica. Correct addition of sulphuric acid for dissolving the fused mass is important to avoid titania precipitation during fuming. If the solution is turbid or showed presence of insoluble pisolites, concentrated hydrochloric acid is also added.

The residue after fuming is diluted with hot water and filtered, washing with hot sulphuric acid, 1 percent. The filtrate serves as stock solution for subsequent analyses. Often for more precise work, another acid fuming operation is recommended on the filtrate to complete silica recovery.

6.5 Total Alumina (TA)

Total alumina includes all the aluminium present as oxide, including diaspore, boehmite, gibbsite and kaolinites. This differs from TAA (total available alumina) often considered in alumina industry, which represents only that component of alumina which is extractable under particular process conditions and excludes most of diaspore and all the alumina eventually combining with silica during the alumina production.

6.5.1 Difference Method

Alumina is determined by substracting the separately estimated weights of iron and titanium oxides expressed as Fe_2O_3 and TiO_2 from the weight of mixed oxides precipitated by ammonium hydroxide after the silica separation.

The method is not accurate in the absolute term, since any other hydroxide precipitated besides iron and titanium will show up as alumina, as for example small amounts of oxides of vanadium, chromium, phosphorus. Besides the precipitate has high adsorption power and it is difficult to eliminate all the occluded impurities. Also any error in the estimation of titanium and iron oxide will show up on alumina value. However, the errore involved are of small magnitude and the method is often accepted as for example by the Indian Standards Institution.11)

Method

An aliquot of the main solution obtained by the bauxite decomposition as per section 6.4 is used. Equivalent bauxite about 200 mg.

Before proceeding with ammonia precipitation, ferrous iron is oxidised in the hot to ferric state with concentrated nitric acid. Some practices add ammonium nitrate before precipitating with ammonia to ensure full oxidation conditions. Aluminium, iron and titanium hydroxides are precipitated in the boiling solution by slowly adding concentrated ammonium hydroxide in the presence of ammonium chloride to the methyl red indicator (0.1 percent) turning yellow. Since the precipitate can be significantly contaminated due to gelatinous and bulky nature of the precipitate, it is dissolved in a suitable acid and reprecipitated as before. All the three main acids (sulphuric, hydrochloric and nitric acid) are being used for dissolving the precipitate. Nitric acid and hydrochloric acid have an advantage that the formation of basic salts is largely avoided. With sulphuric acid, the solution must be boiled for 15-20 minutes to ensure the redissolution of any basic salt formed.

However, some practices do not consider the second precipitation necessary if the first precipitation and washing of the precipitate is carefully done, ending up with good vacuum suction. For complete precipitation, sufficient concentration of ammonium ions in the solution and vigorous agitation during precipitating step are important. The precipitate is washed with hot ammoniacal ammonium nitrate solution (5 percent) until free from the chloride. The precipitate is ignited in a platinum crucible at 1100 °C for an hour. Being very hygroscopic the crucible is kept covered, cooled in a dessicator over activated alumina and weighed soonest possible.

The ignited precipitate represents $Al_2O_3 + Fe_2O_3 + TiO_2$. For more accurate work a blank is run using the same quantities of reagents as employed in practice.

Typical quantities used for 200 mg equivalent bauxite sample are 2 - 3 ml concentrated nitric acid, 4 - 5 g ammonium chloride and 10 ml concentrated hydrochloric acid.

6.5.2 Oxine Method

In contrast to the earlier "difference method" it is a direct and absolute method in the sense that alumina is precipitated by itself, subsequently determined either gravimetrically or volumetrically. Oxine method is a standard check method for alumina estimation. Gallium and zinc are precipitated with aluminium, but their content being very low, no significant errors are introduced.

Method

The method consist in separating out titanium, iron, calcium and manganese from the silica-free filtrate - main aliquote from the solution prepared under section 6.4 - by double precipitation with caustic soda. Alumina is then precipitated with 8-hydroxyquinoline (called oxine) in presence of acetate and ammonium ions and tartaric acid at pH adjusted to avoid vanadium and calcium precipitating with aluminium

For iron and titanium precipitation with caustic soda, the main solution (about 400 mg bauxite equivalent) is added slowly to 36 percent caustic solution (equivalent 18 g caustic), agitating vigorously followed by boiling gently for 10 minutes. This is needed for complete precipitation of iron and titanium in presence of the bulky gelatinous precipitate.

To facilitate filtration of the highly alkaline solution, the solution is diluted and cooled for about one half hour. The washing is done with hot ammonium chloride solution (2 percent), only once to avoid hydrolysis of the entrapped sodium aluminate.

For the second precipitation, the precipitate is dissolved in hot dilute sulphuric acid adding a few drops of hydrogen peroxide, the latter considerably hastening the dissolution step. The precipitation and filtration are done as before, except that only 10 g of caustic soda is used and the precipitate is washed more thoroughly, 4 times to recover all the aluminate. The filtrates from both the precipitations are mixed and evaporated gently (without boiling) to 300 - 400 ml volume after addition of 20 ml sulphuric acid 1 : 1.

For alumina precipitation, an aliquote of the above solution containing no more than 100 mg alumina is taken. With larger quantities, the precipitate is too bulky to allow good washing.

Ammonium chloride (2 - 3 g) and tartaric acid (1 - 2 g) are added and the solution neutralized in the cold by adding slowly liquor ammonia in presence of neutral red. The solution is then heated to 80 °C and alumina precipitated with acetic acid solution of oxyquinoline, adding about 3 - 5 ml excess of the reagent calculated on the basis of 1 ml reagent for every 3 mg alumina. To complete alumina precipitation and eliminate vanadium interference, liquor ammonia is now added to make the solution alkaline to litmus red, and boiled gently for 10 minutes.

Some practices recommend adding hydroxylamine in the cold, heating to be done only after adjustment of pH (by ammonia) 12). This is to avoid possible errors from loss of the oxine reagent before the precipitation step through its own precipitation and evaporation.

For filtration, the solution is allowed to decant on hot plate for 15 minutes, and then passed through sintered glass crucible (G4).

For gravimetric estimation, the precipitate is dried at 110 °C after washing two (2) times with hot water and three (3) times with cold water. It is weighed as aluminium oxinate, $Al(C_9H_6NO)_3$.

For volumetric estimation, the precipitate is dissolved in 6N-hydrochloric acid boiling, diluted, cooled and titrated with standard potassium bromide-bromate mixture (2N), adding 1-2 ml of the latter in excess. The excess reagent is estimated by liberating iodine quantitatively from the potassium iodide solution (10 percent), subsequently added. Liberated iodine is titrated with standard sodium sulphite solution (IN) in presence of starch as indicator.

Oxyquinoline solution is prepared by dissolving 30 g of the reagent in 70 ml hot acetic acid, subsequently made to 1000 ml with water.

6.6 Alumina Trihydrate (THA)

As explained in section 4.1, THA determination becomes important right at the preliminary stage of feasibility studies inasmuch as a purely THA bauxite is easier and cheaper to process.

The methods used are based essentially on very marked differences in reactivity against caustic liquors between three principal naturally occuring alumina hydrates, trihydrate, monohydrates in the form of boehmite and diaspore. The selectivity and accuracy of the respective methods is established by testing the insoluble residues of bauxites by X-ray diffraction. For convenience, this estimation is presented under section 6.8.

6.7 Monohydrate

6.7.1 Boehmite

As explained earlier, monohydrate containing bauxites are more difficult to precess, hence the importance of its estimation at an early stage.

In the proved absence of diaspore, boehmite is generally determined by substracting trihydrate alumina from total alumina.

It can also be analysed by the autoclave digestion tests as described under section 6.10.

X-ray methods are used to confirm the estimated values in the event of doubt.

6.7.2 Diaspore

Diasporic bauxites are particularly prized by the refractory industry, but are generally least desirable for straight Bayer process.

Bauxite digestion tests included in section 6.8.2 give an approximate idea of its degree of occurence. Precise estimation requires X-ray diffraction.

6.8 Total Available Alumina (TAA)

TAA content of bauxite represents only that part of alumina which is available for extraction, excluding the remaining which the reactive silica content of bauxite ties up in any case. Thus from the process viewpoint this figure is of more direct value. In fact many plants hardly ever talk in terms of total alumina content of the bauxites in day-to-day work.

6.8.1 Indirect Method

Percent TAA = percent TA - (percent reactive silica) x k whrere k represents ratio of alumina to silica combined.

k is established from experience on mud analyses and is often taking as unity. The estimated TAA value will be higher than the actual to the extent depending on presence of diaspore.

6.8.2 Direct Methods (autoclave digestion)

The methods consist in digesting bauxite with caustic liquors in rotating autoclaves and analysing the filtrate for alumina content.

The operating conditions are so chosen, that all the alumina present in the bauxite is attacked, except for the diaspore content. This is established by X-ray analyses of the residues. Widely different nature of the test conditions practised is indicated by the following:

a) Pure caustic solution, 100 gpl Na_2O (129 gpl NaOH) 120 °C, 15 minutes digestion time; bauxite charged to give a final alumina-free caustic weight ratio, expressed as Al_2O_3/Na_2O 0.4 (as Al_2O_3/Na_2CO_3) 0.234).7)

b) Caustic 200 gpl Na_2O (260 gpl as NaOH), 0.5 Al_2O_3/Na_2O weight ratio, 180–230 °C, 3 hours digestion time, bauxite charged to 0.8 ratio.7)

c) Caustic 125 gpl (NaOH) 220 °C, 40 minutes digestion time, bauxite charged to give final Al_2O_3/Na_2O 0.650 (Al_2O_3/Na_2CO_3 0.38)

Individual preference of the conditions used is determined by the desire to remain close to the digestion conditions operating in the plant of the particular group.
It is to be noted that the TAA test is not an absolute test but a relative one depending on the details of the test conditions. For example, the degree of desilication achieved during the test operation will influence the alumina present in the filtrate. However, this is usually a very minor quantity, especially under higher temperature digestion conditions and long treatment intervals.

Autoclaves are preferably made of monel metal. They are heated electrically or by gas burners and rotate at a slow speed – 56 rpm. A typical autoclaves is shown in fig. 1.

Handling of the digested slurry. The autoclaves are emptied after releasing the pressure gradually and the insoluble residue filtered out washing with 2 % hot sodium chloride solution. The filtrate is made to 1 litre solution and analysed for alumina. Usually volumetric methods, described later, are used because they are quick and sufficiently accurate. Some plants prefer oxine method for alumina estimation in the filtrate, after acidifying it carefully with hydrochloric acid, making sure that any alumina precipitated is redissolved.

6.9 Available Trihydrate Alumina (Av. THA)

The purpose of this analysis, and principles underlying various procedures have been explained in section 6.6.

6.9.1 Autoclave Digestion Method

Basically identical to the autoclave digestion method discussed in section 6.8.2 on TAA determination.

Modificationsconsist in so adjusting the composition of digestion liquor and the amount of bauxite charged that the available chemical potential*) allows dissolving of only easily soluble trihydrate. Here again the nature of widely varying practices is evident from the following:

a) Caustic solution 100 gpl Na_2O (129 gpl NaOH) 120 °C, digestion time 15 minutes, bauxite charged to give a final Al_2O_3/Na_2O weight ratio 0.8 (or Al_2O_3/Na_2O_3 0.468)

b) Caustic solution, 125 gpl NaOH, 150 °C, digestion time 30 min., bauxite charged to give a final Al_2O_3/Na_2O 1.024 (or Al_2O_3/Na_2CO_3 = 0.600)

---

*) Chemical potential at any temperature and caustic concentration = Al_2O_3 gpl equilibrium concentration minus Al_2O_3 gpl in test slurry on complete extraction.

6.9.2 Atmospheric Pressure Digestion

Method

The bauxite as prepared under 6.1.2 is boiled with dilute caustic soda solution of such concentration that all the trihydrate alumina is extracted without attacking the monohydrate. Generally 2-N solution is adequate. A water reflex condenser is used to avoid increase of caustic soda concentration from evaporation.

The solution is filtered, washing the residue with hot water containing a little caustic - generally 3 washings, washings done without mixing the precipitate. Alumina is determined in suitable aliquote of the filtrate by oxine method as outlined in section 6.5.2.

Typical quantities used are 5 g bauxite, 200 ml 2 N caustic soda solution, 1000 ml stock solution after filtration, 25 ml aliquotes for alumina estimation. Boiling (attack) time about 30 minutes.

Alumina estimated by this method is available trihydrate alumina and does not include that part of trihydrate alumina which has combined with silica during attack.

To determine total THA, the amount of trihydrate alumina not included in the above is estimately separately from amount of the bauxite reactive silica and the mud analyses as explained in section 6.8.1.

The estimated value of available THA is likely to be slightly higher than that met with in the plant practice, because at the low temperature used, desilication process is slow and some of the alumina which should be lost by eventually combining with silica is still in solution.

6.10 Available Monohydrate Alumina (Av. MHA)

Generally determined by substracting THA from TAA. Individually estimated by X-ray methods.

6.11 Total Silica

Total silica includes silica present in all the forms.

Finely ground sample of bauxite from section 6.1.2 is decomposed by alkali fusion except, when sure, that the bauxite is completoly decomposed by mixed acids. Procedure for mixed acid attack and alkali attack are described in section 6.4.

The ultimate insoluble residue obtained in section 6.4 contains mostly silica along with variable amounts of oxides of iron and titanium and perhaps undigested bauxite. The filter still wet is ignited in platinum crucible at 900 - 1200 °C, lower temperature requiring about 2 - 3 times longer time than that needed at about 1100 °C. Silica is determined by volatalising away the silica from the ignited residue as silicon tetrafluoride, on treating it in the platinum cricible itself with sulphuric acid and hydrofluoric acid. Some reccommend repeated silica volatalisation, until the weight of the calcined residues become constant 11) while others consider it unnecessary as long as the weight of the calcined residue does not exceed 1 - 2 mg. For repeated volatalisation, some hydrochloric acid is also added, if the residue indicates presence of some undecomposed bauxite. Important precaution in silica estimation is not to leave the solution containing insoluble silica precipitate for long, otherwise some silica becomes soluble. If the bauxite sample was decomposed by alkali fusion, refinements include evaporation of the filtrate obtained in section 6.4 to the fuming stage to ensure recovery of any soluble silica that did not become insoluble earlier.

6.12 Quartz

Quartz analysis is important, because along with information on total silica content of bauxite it determines a major raw material cost (of caustic soda) in alumina production. A high silica containing bauxite may be quite desirable if large part of it consisted of relatively inert quartz. Absolute method analysis is by X-ray techniques. Chemical methods are principally based on relatively inert nature of quartz.

6.12.1 Elimination by Hydrofluoric Acid [11])

The bauxite is decomposed by acid attack as described in section 6.4.1. To the boiling solution obtained by acid attack, about 5 ml of hydrofluoric acid is added before filtering out the insoluble silica residue. The insoluble residue is then handled exactly as described under section 6.11.

6.12.2 Sulphurous Acid Method

The method determines in the mud reactive silica actually in combination with alumina in the desilication product, on the basis that desilication product dissolves in sulphurous acid. 6)
The value of reactive silica so determined is substracted from total silica estimated on bauxite to give quartz.
The quartz content so estimated will be under-estimated to an extent determined by its reactivity during the bauxite digestion step.

6.12.3 Phosphoric Acid Method

The method is based on complete lack of quartz solubility in pyrophosphoric acid at 250 °C. The determination is done right on bauxite.

0.5 - 1 g of bauxite prepared according to section 6.1.2 is treated with about 25 ml anhydrous phosphoric acid at carefully controlled temperature of 240 - 260 °C for about 10 - 15 minutes. The solution is cooled to about 60°, diluted with about 150 ml hot water and boiled gently till it becomes clear. 10 ml fluoboric acid, 40 percent, is added to solubilise any reactive silica present and the quartz filtered out on fine-pore size filter, washing the residue with 4 percent hydrochloric acid. The initial filtrate may be turbid in which case it should be refiltered till completely clear. Washing is continued to the disappearance of phosphate ions, as checked with molybdenum blue reaction. Quartz is determined on the washed residue by volatalising it away with hydrofluoric acid and sulphuric acid as described under section 6.11 for total silica.

6.13 Iron Oxides

Iron occurs mostly as hematite Fe_2O_3, limonite $FeO.OH.nH_2O$, goethite $FeO.OH$, siderite $FeCO_3$ and nontronite $Fe_2O_3.3SiO_2.5H_2O$. Iron is generally determined as Fe_2O_3 though of recently some interest has arisen for estimating separately iron present in the ferrous state, which mostly is present in small quantities.

Iron estimation is vital in process studies because being almost inert in caustic soda, it serves as key element to calculate expected mud loads and alumina extraction efficiency as explained in section 4.2. Excessive presence of ferrous iron could cause some product purity problem from colloidal iron hydroxide formation.

Methods

Iron is determined in the main solution obtained under section 6.4. Refinements include fusing the insoluble silica residue separated out with potassium bisulphate and adding the acid extracted filtrate to the stock solution. The fusion should preferably be done in porcelain crucible because platinum is partially attacked by potassium bisulphate and interfers slightly with iron. If platinum crucible is used, the solution should be treated with a fast stream of hydrogen sulphide gas for removing the interference. The filtrate is subsequently boiled to remove all the excess hydrogen sulphide gas. The standard accepted method is Zimmermann Reinhardt procedure where all the iron in solution is first reduced to the ferrous state, with stannous chloride, and estimated by oxidising it subsequently to the ferric state, titrating with potassium permanganate in presence of Reinhardt Zimmermann reagent containing manganese sulphate and phosphoric acid. Some laboratories prefer potassium dichromate to permanganate. Potassium dichromate has the advantage that vanadium and chromium do not get oxidised. Potassium permanganate gives a very clear end point and is not disturbed by the presence of chloride ions.

Numerous other procedures are equally accurate and often more convenient. The other procedures commonly in use include reduction of an oxidised solution with zinc or other metal and titration with titanous chloride in an oxidised solution. But these are all checked for acceptability against results obtained by Zimmermann Reinhardt procedure.

Certain practices recommend prior oxidation with potassium permanganate of any ferrous oxide present in the solution to the ferric state before reduction with stannous chloride.[11])
Excess of the permanganate in such cases is destroyed by boiling it

with 50 percent hydrochloric acid.

Reduction with stannous chloride (25 percent solution) has to be done carefully making sure that excess hydrochloric acid is present, the solution is boiling hot and vigorously agitated and that no more than 2 - 3 drop in excess of that required for complete discolcration of the solution are added. Excess of stannous chloride is then destroyed by adding saturated solution of mercuric chloride in the cold.

Titration with potassium dichromate is done using diphenylamine as indicator after adding 10 - 15 ml sulphuric acid - phosphoric acid mixture. At the end point the indicator turns permanent violet. The solution must be sufficiently diluted for ready recognition of the end point.

Titration with potassium permanganate is done on sufficiently diluted solution in presence of Zimmermann Reinhardt reagent (H_2SO_4, H_3PO_4, $MnSO_4$). Phosphoric acid suppresses the iron colour and also promotes the titration oxidation reaction. Standard permanganate solutions, especially dilute ones, are susceptible to decomposition on storage and may require frequent standardisation. Mostly sodium oxalate is used for standardising; some prefering to use ferric chloride solutions made from merck quality Fe_2O_3 dissolved in concentrated hydrochloric acid.

Indicator sodium diphenylamine sulphonate is made by dissolving 0.32 g of barium diphenylamine sulphonate in 100 ml hot water, followed by addition of 0.5 g sodium sulphate. Barium sulphate formed is filtered off.

Zimmermann-Reinhardt reagent: 140 ml concentrated sulphuric acid, 140 ml syrupy phosphoric acid, 720 ml water. Dissolved 200 g hydrated manganese sulphate ($MnSO_4 \cdot 4H_2O$) in the above. Oxidisable impurities removed by adding potassium permanganate drop by drop.

Colorimetric and spectrophotometric method based on developing colour with potassium thiocyanate is given in section 10.3.2.

6.14 Titania

In the Bayer process for alumina production, titania analyses are required toestimate total alumina by the difference method (see section 6.5.1), to investigate possible soda losses from titanium during digestion and to evaluate the economics of mud disposal. Importance of titania analysis for refractory industry is explained in section 3.3

Till recently only classical methods were used, based on reducing all the titanium to the titanous stage followed by oxidation-titration with standard ferric ammonium sulphate. Recently good progress has been made in developing reliable colorimetric spectrophotometric methods.

6.14.1 Volumetric Method

Starting point is aliquote from the main solution prepared in section 6.4. Reducing agents generally used are Jones Reductor consisting of zinc amalgam - see Fig. 2.[11])
Suitability of liquid zinc amalgam method,[13]) especially for titaniferrous bauxites, needs to be examined for the advantages that the liquid amalgam is relatively inert to common impurities present in sinc and its preparation is simpler. The amalgam is more readily cleaned for repeated use, as checked by the results reproducibility.

For reduction by the Jones Reducter, the solution should be hot containing sufficient sulphuric acid. It is poured slowly through the Jones Reductor, protecting the reduced solution from oxidation by collecting it in a flask continuously swept with carbon dioxide. The Jones Reductor is washed free of reduced solution with dilute sulphuric acid (1 - 2 percent).

With liquid amalgam reduction, the solution containing sufficient sulphuric acid or hydrochloric acid is taken over liquid amalgam contained in the [illegible] separatory funnel - see Fig. 3. A pinch of sodium carbonate is added to provide protective atmosphere of carbon dioxide and the top closed with rubber stopper carrying a small glass tube sealed with a glass rod stopper. The rubber tube is given a vertical split with a sharp blade to release any excess pressure resulting from carbon dioxide released from sodium carbonate. The solution is reduced by shaking the separatory funnel assembly with hand. Liquid amalgam is separated into the bottom funnel and the reduced solution titrated with standard ferric ammonium sulphate solution, using ammonium thiocyanate indicator. Blank tests are recommended for special occuracy.

6.14.2 Colorimetric Method

Used for titania content upto about 5 percent. See section 10.3.1.

6.15 Phosphorus Pentoxide

Importance of phosphorus contamination in bauxite is explained in section 4.[illegible].7.

Though phosphorus occurs generally as phosphate of iron, calcium and manganese, it is reported as P_2O_5.

Two methods are considered - one based on precipitation with ammonium molybdate and the other spectrophotometric (in section 10.3.3).

6.15.1 Precipitation Method

Starting point is silica-free solution of the bauxite, obtained by alkali fusion. Phosphorus is precipitated as yellow ammonium phospho-molubdate in a carefully acidified (with nitric acid) solution with ammonium molybdate and determined either gravimetrically or volumetrically by the alkalimetric method.

In the alkalimetric method, the precipitate is washed free of acid and iron, and treated with a measured excess of standard sodium hydroxide solution. The amount of excess sodium hydroxide, as estimated by titration with sulphuric acid, gives the amount of caustic used up by phosphorus.

For gravimetric method, the washed precipitate is dissolved in hot 2.5 percent ammonia and reprecipitated. The ignited product is weighed as $NPO_3 \cdot 12MoO_3$.

For correct adjustment of acidity prior to precipitation step, the starting solution is treated with concentration ammonia in boiling condition till the precipitated hydrate dissolved with difficulty. The precipitated hydrate is redissolved with concentrated nitric acid, thus providing a slight excess of 2 ml/100 ml solution.

For precipitation step, solution should be sufficiently concentrated, say about 100 ml for 1 gram bauxite sample before the acidification step. The ammonium molybdate solution should be filtered before use.

For washing, the precipitate filtered out under gentle sunction is washed free from iron by cold dilute nitric acid (1 percent) till potassium ferrocyanide indicator turning brown. The acid is washed out with dilute potassium nitrate, 1 percent, to the extent that 10 - 15 drops of the final washing do not decolourise the phenolphtaleine indicator containing a drop of 0.1 N sodium hydroxide solution.

6.16 Calcium Oxide

Calcium present in bauxite converts active caustic into inactive sodium carbonate. Calcium content of the bauxite is required to estimate the nature of causticising facilities.

Methods

The methods are based on precipitating calcium as oxalate. Calcium is subsequently estimated gravimetrically or volumetrically.

Three methods of bauxite decomposition are used, depending on circumstances.

a) Routine decomposition methods described in section 6.4. Calcium oxalate precipitation is done on filtrate obtained after the separation of mixed oxides outlined in section 6.5.1.

b) Bauxite fusion with caustic soda in presence of sodium peroxide. The fused mass is dissolved in boiling water containing alcohol. Insoluble residue containing iron, titanium, manganese, calcium and magnesium is dissolved in 50 percent nitric acid and iron, ititanium, manganese and phosphorus eliminated by double precipitation with liquor ammonia in presence of oxidising agent ammonium nitrate. The filtrate is used for calcium estimation.

c) Bauxite decomposed with 50 percent hydrochloric acid in gently boiling condition. Mixed oxides are separated by double precipitation with ammonia and calcium estimation done on the filtrate.

For calcium oxalate precipitation it is important that the solution be fairly concentrated - about 50 ml for 1 g bauxite sample. The precipitation is done in hot boiling solution with ammonium oxalate and a slight excess of concentrated ammonia. Presence of sufficient excess of ammonium oxalate ensures that magnesium is not precipitated. Calcium oxalate precipitation is a slow process and many practices recommend leaving the precipitate for a few hours and even over-night before the filtration. The precipitate is washed first with ammonium oxalate solution (1 gpl) and then with cold water, using only small quantities

For gravimetric estimation, the precipitate is ignited in platinum crucible at 1000 °C, and weighed as calcium oxide. A well conducted test gives white residue.

For volumetric estimation, the precipitate still wet is dissolved in dilute sulphuric acid, heating only gently to the temperature below 80 - 90 °C. The oxalic acid generated is immediately titrated with permanganate to the pink end point persisting for 1 minute. Absence of ammonium oxalate is a must for reliable results.

6.17 Magnesium Oxide

Estimated gravimetrically from the filtrate after calcium precipitation in section 6.16. Two methods are in vogue for magnesium precipitation: oxine and ammonium phosphate.

6.17.1 Oxine Method [11])

Any manganese present is separated as manganese dioxide by boiling with ammonium persulphate in the presence of ammonia. The filtrate is evaporated to syrupy consistency and all the ammonium salts volatalised away by evaporating it to drynes with concentrated nitric acid. The residue dissolved in dilute hydrochloric acid is used for oxine precipitation.

Oxine precipitation is done in boiling ammoniacal solution in the presence of ammonium chloride, making it strongly ammoniacal after oxine addition. The precipitate is separated out on gooch crucible, washing it with hot ammoniacal water. It is dried at 130 - 140 °C and weighed as oxinate of magnesium.
Oxine method is often preferred to the ammonium phosphate precipitation method for its comparative simplicity.

6.17.2 Ammonium Phosphate Method

The filtrate from calcium precipitation is acidified slighty with hydrochloric acid, followed by addition in cold of ammonium phosphate solution, 25 percent. The solution is then heated and made alkaline to phenolphthaleine by careful addition of dilute ammonia (30 percent). Finally concentrated ammonia is added, volumetrically to about one third of total volume. The precipitate corresponds to $MgNH_4 PO_4 \cdot 6H_2O$. The precipitate is left over night, filtered, washed with cold ammoniacal water and ignited in platinum crucible at temperature higher than 1000 °C to $Mg_2P_2O_7$. Any manganese not removed earlier will partly precipitate and show up in magnesium.

The solution taken for magnesium precipitation should be fairly concentrated - about 150 ml for a 3 g bauxite sample. For precision work, the precipitate should be dissolved in hydrochloric acid (25 percent) and re-precipitated as above.

6.18 Manganese Oxide

Always present in small quantities. For estimation, it is converted to permanganic acid and analysed by titration with sodium arsenite solution. In the colorimetric method manganese is converted to permanganate - see section 10.3.4.

Volumetric method

Starting solution is silica-calcium - free solution after bauxite dissolution in section 6.4.1. Alternatively, one may start with the insolute residue obtained after aqueous extraction of bauxite fused with sodium hydroxide - sodium peroxide mixture (section 6.4.2).

Manganese is converted to permanganic acid by oxidation with ammonium persulphate in presence of silver nitrate, 1 percent.

The titration method for permanganic estimation is slightly tricky because reduced manganese reoxidises readily by excess of oxidising agents present (ammonium persulphate) unless the titration is carried out fast. The titration is done in the cold.

6.19 Gallium

Importance explaned in section 4.2.7. Estimation is based on extraction of chlorogallic acid in ether. Flame spectrophotometry based on atomic absorption method is expected to provide more straight forward and simpler method as pointed out in section 11.2.

Extraction as chlorogallic acid

To the boiling silica-free filtrate obtained after acid digestion, outlined in section 6.4.1, a concentrated solution of caustic and potassium hydroxide (1:1) is added until the solution becomes clearly alkaline. Iron and titanium are precipitated thereby.

Alumina and gallium are separated from the filtrate by neutralising it in the boiling with hydrochloric acid. Lyphan paper is used as indicator. The washed precipitate is dissolved in hydrochloric acid for extraction with ether and subsequent precipitation as outlined in section 8.11. The solution prepared for extraction should be 100 ml, corresponding to 5.6 N hydrochloric acid concentration. During evaporation for reducing the solution volume, watch out against crystallising out of the sodium and potassium hydroxide.

6.20 Organic Carbon

Importance of organics contamination in bauxites has been explained in section 4.2.7.

Basic method consists in dry combustion of the powdered bauxite in a current of oxygen and estimating the carbon dioxide evolved. Equally good results are reported by some, using potassium permanganate to oxidise carbon in sulphuric acid medium. A routine method uses potassium permanganate to oxidise the organic matter in solution obtained on alkali digestion of the bauxite. [10])

6.20.1 Dry Combustion Method

The apparatus is shown in figure 4.
Oxygen is first purified and dried using caustic soda pellets and concentrated sulphuric acid, and then passed through soda lime to absorb any acid mist before entering the furnace.

Finely ground bauxite (5 gram) as prepared in section 6.1.2 is introduced into the furnace in a porcelain boat, first heated gradually at 100 - 200 ° to remove associated moisture and then heated at 1000 °C for one hour. Lead chromate and oxidised copper wire gauze in the form of a loose plug are kept in the furnace tube after the boat to oxidise any carbon monoxide formed to carbon dioxide. Myers absorption tube with 15 beads is used to contain the barium hydroxide solution.

Oxygen flow is regulated to avoid any loss of bauxite powder through "draft", generally about 2 bubbles per second being enough. Barium hydroxide solution (N/10) taken in known quantity for carbon dioxide absorption is then titrated with hydrochloric acid (N/10) to determine the amount of barium hydroxide consumed, and from that the amount of carbon dioxide evolved. Indicators used: thymol blue and neutral red. Barium carbonate precipitate separation prior to titration is not necessary.

For calculating organic carbon, the amount of carbon dioxide evolved is corrected for carbon dioxide liberated from inorganic carbonate impurities in the bauxite by a separate run with hydrochloric acid digestion.

A blank run in often recommended.

6.20.2 Potassium Permanganate Oxidation Method

The apparatus used is sketched in Fig. 5. Carbon dioxide evolved is mesured volumetrically.

Before the oxidation step, carbonates are decomposed by adding dilute sulphuric acid to the reaction flask and disconnecting the gas measuring apparatus. Later potassium permanganate, 10 percent, is added drop by drop, heating the reaction flask gently all the time, and evolved carbon dioxide measured as described in section 8.4. A couple of small granules of zinc are kept in the stem below the stop-cock of permanganate feeding funnel, so that they are washed down into the reaction flask by incoming permanganate. On their contacting the dilute sulphuric acid, some hydrogen gas is evolved which serves to shake up the contents.

By this method, the organic content may be underestimated because some organics may get lost during initial decomposition of the carbonates with sulphuric acid. Also some organics may resist oxidation to carbon dioxide or oxidise only to the carbon monoxide stage.

7. Mud Chemical Analyses

As explained in section 3.1, one of the principal preoccupations of alumina industry is to develop economic means of processing higher silica containing ores, which means reducing the soda and alumina losses as a part of desilication product in the mud. Both the Combination Processes and the attempts at modifying the nature of desilication product itself depend on very careful analysis of red mud. Besides the alumina yieldsare calculated from mud analyses. Here again classical chemical analytical procedures provide bulk of information, though need for X-ray and spectrophotometric methods to supplement the above information is being increasingly felt.

The analytical methods are generally the same as for bauxites, except for some adjustments done to allow for different relative proportions of various constituents with respect to alumina content of the muds. Also the muds being somewhat hygroscopic, should be weighed fast. Colorimetric-spectrophotometric methods are described separately in section 10.

7.1 Decomposition and Dissolution

Here again like bauxites, both the acid and alkali attack are used, with preference for the acid attacks wherever possible. Generally the muds from the bauxites that required alkali fusion for complete decomposition also require alkali attack. Muds are generally easier to decompose than the bauxites.

In making the main solution, the insoluble silica residue has always to be redissolved in hydrofluoric and hydrochloric acid followed by baking to fumes with sulphuric acid, since the mass is generally large and substantial iron, titanium and aluminium will be entrapped in it. The filtrate obtained by dissolving the baked residue in water is added to the bulk solution made earlier. For additional precision, specially with respect to minor impurities, the residue remaining after repeated silica volatalisation should be fused with potassium bisulphate followed by dissolving in dilute sulphuric acid and adding to the main solution. If platinum crucible is used for the bisulphate fusion, the filtrate should be treated with hydrogen sulphide gas to precipitate out any resulting platinum contamination, which could interfere in ironanalyses.

7.2 Ignition Loss

As for bauxite, except with the strict requirement that the temperature does not exceed 1100 °C.

7.3 Alumina, Titania, Iron Oxide, Calcium, Oxide, Magnesium Oxide and Manganese Oxide

As for bauxites. For calcium estimation particularly, it is absolutely necessary to do double mixed oxide precipitation (with ammonia), otherwise some calcium is bound to remain adsorbed on the precipitate.

7.4 Total Silica

As for bauxites with the emphasis that the insoluble silica residue obtained after acid or alkali attack will be severely contaminated, and repeated fuming with hydrofluoric, hydrochloric and sulphuric acid may be necessary.

7.5 Reactive Silica

Reactive silica represents that part of total original silica which has combined with alumina in the digestion process, as distinct from the inert quarts, remaining as such.

As outlined in section 6.12.2, based on the fact that desilication product is soluble in sulphurous acid.6)

7.6 Soluble Soda

Soluble soda is defined as that part of the soda, which does not exist in a chemically combined condition, i.e. that present outside the desilication product. It is determined by boiling the dried mud in 10 percent sodium chloride solution and titrating the filtrate against standard hydrochloric acid solution using phenol red as indicator.

Soluble soda is a slightly relative term because the desilication product goes on decomposing on continued washing to release part of the combined soda. By convention, the interval of the water treatment is limited to about 10 minutes. Addition of sodium chloride to wash water reduces solubility of the soda forming part of the desilication product and also prevents mud peptisation.

7.7 Combined Soda

Combined soda represents that part of soda which is present in chemical combination in the desilication product as distinct from the "physically" entrapped soda, usually called soluble soda (see section 7.6).
Combined soda is determined mostly by flame photometer. Gravimetric method based on sodium precipitation with zinc uranyl acetate is used only as check method.

7.7.1 Flamephotometric Method

The dry mud is decomposed by acid digest baking as provided for silica estimation. The residue is washed with 1 percent sulphuric acid to recover sodium and tested on flamephotometric as described under section 11.1.

7.7.2 Gravimetric Method

Sodium is precipitated by zinc uranyl acetate in hydrochloric acid medium from a solution free of silica, titanium, iron, manganese, alumina, magnesium and ammonium ions. Two methods are used for sample preparation:
a) acid attack
b) fusion with calcium carbonate and ammonium chloride.

Acid attack

About 0.5 g mud is decomposed with concentrated hydrochloric acid on water bath and evaporated to dryness. The dried mass is subsequently baked at 130 °C for one-half hour and redissolved in dil hydrochloric acid and filtered. Generally double filtration is needed to obtain a reasonably clear solution. Alumina, iron, titanium, phosphorus and manganese are separated by ammonia precipitation in presence of ammonium chloride and ammonium nitrate. The filtrate is evaporated to dryness to destroy ammonium ions and dissolved in dilute hydrochloric acid to determine soda as described in section 8.5.

Fusion with calcium carbonate and ammonium chloride, where sodium content is converted into carbonate, is carried out as outlined in section 9.10.

8. Chemical Methods for Liquor Analyses

Liquor analyses are needed to complete the process information obtained on the bauxite and mud analyses. Precipitation behaviour is particularly sensitive to the liquor contamination, both from the point of yield and the precipitate quality (hence product) as explained in section 4.2.6.

Major constituents e.g., alumina, soda, caustic, organic soda are mostly determined by classical chemical methods, though great effort is being expended to develop, for the plant process control, continuous on-stream analyses methods making use of physical properties of the liquors. Colorimetric methods and atomic absorption spectrophotometry are increasingly adopted for estimation of minor constituents with continuous efforts to extend their range of applicability. These latter methods are treated separately under individual heads.

Under following description of various titration methods, only indicators are mentioned to determine the end points. The potentiometric methods locate the end point more precisely but they are seldom used for anything more than check or in finding new sharper end-point indicators.

8.1 Caustic Soda

Caustic soda in Bayer process is defined as the sum of free sodium hydroxide and sodium aluminate (bound caustic) expressed as Na_2CO_3 or Na_2O.

Caustic is determined volumetrically by titrating against hydrochloric acid in presence of phenophthaleine taking steps to avoid interference from alumina, phosphates, silicates, vanadates, sodium carbonate.

An important source of error in titration is the acid consumed in various basic salts-precipitation, common in high alumina-to-caustic ratio liquors. These considerations have been discussed in detail by Phillips and McLaughlin. 14)

Carbonate, phosphate, silicate and vanadate interference is minimised through their precipitation with barium chloride. The precipitate is removed by filtration.

Alumina interference is suppressed by complexing it with neutral alkali tartarate (Rochelle salt) in the presence of barium chloride. Tartarate is added after filtering out the barium precipitate.

A well conducted analysis, in sufficient presence of barium chloride and tartarate, will give a clear solution before reaching of the end point. A dull solution indicates the basic salts formation incidence.

Some practices do not consider addition of tartarate necessary for general laboratory analyses. Others, who by convention work on the basis of total soda concentration (as distinct from caustic), determine caustic by substracting from the total soda content the sodium carbonate content, estimated from decomposition with hydrochloric acid.

In excessive presence of sodium carbonate, barium chloride precipitate may become too excessive and absorb significant quantity of caustic.

During separation of carbonate, phosphate, silicate and vanadate, while washing part of the liquor silica ends up in the solution to be titrated, and thus gets included in the caustic value. On fairly well desilicated liquors, this error will be small.

8.2 Total Alkaline Soda

Total alkaline soda is defined as sum of free sodium hydroxide, sodium aluminate and sodium carbonate, expressed as Na_2CO_3 or Na_2O. Total soda is generally determined volumetrically, gravimetric method based on zinc uranyl acetate precipitation being used only for check in the events of doubt.

8.2.1 Volumetric Method

The method consists in adding a known excess of hydrochloric acid to neutralize $NaOH$ and Na_2CO_3 and convert $NaAlO_2$ to $AlCl_3$. Tartarate and barium chloride are added to quantitatively complex aluminium and release stochiometric quantity of hydrochloric acid originally combined with aluminium. Excess hydrochloric acid is now titrated with caustic soda solution in presence of phenolphthaleine to give by difference the acid quantitatively combined with total soda. The end point is approachable from both the sides.

Before adding the complexing agents, barium chloride and tartarate, carbon dioxide is expelled by brief boiling.
Excessive boiling may cause some hydrochloric acid loss.

Acid excess has to be kept within limits to avoid possibility of basic salt formation. Also molar ratio of tartarate to barium chloride should exceed 3, and barium chloride should be added at later stage near the end point. All these considerations are explained in detail by Phillip and McLaughlin. 14)

In this method, vanadate ties up some hydrochloric acid (in the acid medium) so gives slightly high values for soda.

Presence of P_2O_5 above 29 mg gives progressive positive error (because the end product is Na_2HPO_4).

Presence of anions of volatile organic acids gives higher valuo for soda because the former get converted to the corresponding acid state and volatalise during boiling for carbon dioxide removal.

Some practices, however, do not consider necessary above complexing agent requirements and expulsion of carbon dioxide when titration is done in presence of alcoholic phenol red indicator (1 percent) which turns red at the end point.

8.2.2 Gravimetric Method

See under section 8.5.

8.3 Alumina

Alumina is defined and expressed as the content of anhydrous aluminium oxide, Al_2O_3.

Alumina is generally determined by volumetric procedures. Gravimetric method, based on oxime precipitation, is used only for check.

8.3.1 Volumetric Method

A known excess of hydrochloric acid is added to neutralise NaOH, Na_2CO_3 and convert $NaAlO_2$ to $AlCl_3$.

Carbon dioxide from sodium carbonate decomposition is expelled by heating. The solution is divided into 2 parts. On one part, free acid is determined by titrating against sodium hydroxide in presence of mixed indicator (phenol red bromothymol blue) which turns violet at end point. Sodium oxalate is added to suppress alumina precipitation and suppress the release of hydrochloric acid in combination with alumina.

This titration thus gives the acid combined with NaOH, Na_2CO_3 and $NaAlO_2$.

On the second part, free acid along with the acid tied up with alumina (liberated by addition of tartarate and barium chloride as explained in section 8.2.1 for total soda estimation) is determined by titrating with caustic soda in presence of phenolphthaleine. This titration gives the acid combined with NaOH and Na_2CO_3 i.e. total alkaline soda.

Difference of the two above gives the acid combined stochiometrically with alumina.

For sharp end point in the first titration, ratio of oxalate to alumina is very important. (14) Oxalate to alumina molar ratio is recommended at 70 - 95 for 200 - 300 ml solution containing 0.1 - 0.15 g alumina. With these precautions, the error can be reduced to less than one drop of 0.3 - 0.4 N caustic soda used for titration. Because of the above complexing consideration, alumina content of the test solution should be less than 0.15 gram.

The mixed indicator colour changes during titration are as follows: darkens from yellow in the beginning towards a neutral shade at pH 7.0; becomes noticeably lavender at pH 7.2 - 7.4; finally turns sharply violet at pH 7.4 - 7.6. The mixed indicator is made from 1 percent sodium salt solutions of phenol red and bromothymol mixed in equal proportions.

The method suffers from about similar errors mentioned under section 8.2.1 for total soda except that interferences for alumina are relatively less because part of the interferences common to two titrations cancel out during the substraction step.

8.3.2 Gravimetric Method

Based on oxine precipitation, as outlined in section 6.5.2, the method is longer and time consuming. Degree of additional accuracy obtained does not warrant its preference over quicker simpler volumetric method, except for the checks.

8.4 Sodium Carbonate

The estimation is based on classical method of determining the amount of carbon dioxide evolved on reaction with dilute hydrochloric acid (50 percent), see Fig. 5 for the apparatus details. Carbon dioxide is absorbed in 40 percent potassium hydroxide solution.

The test solution quantity should be such that no more than 50 ml of carbon dioxide are produced. About 20 ml of a suitably diluted solution are taken for the analysis.

Hydrochloric acid is first added drop by drop from the feeding funnel. After the carbon dioxide evolution slows down, the flask is heated to dissolve any alumina separated out, conti[illegible] the solution [illegible] starts boiling, showing up as [illegible] vapours on the stop cock 3. More hydrochloric acid is now a[illegible] the feeding [illegible] to fill up the reaction flask and [illegible] the stopcock [illegible] displace all the gas.

The carbon dioxide is now measured by usual method of [illegible]ing the carbon dioxide into potassium hydroxide absorption [illegible] and with-drawing it [illegible] into the burette.

This gas volume [illegible] corrected to normal temperature and pressure conditions taking account of the vapour pressure of the saturated sodium chloride solution used in the levelling bottle.

8.5 Total Soda

It includes all types of soda, alkaline as well as n[illegible]. Soda is determined gravimetrically by precipitating with [illegible]ranyl acetate.

A conveniently diluted solution equivalent to about 3 [illegible] Na_2O is evaporated to dryness on water bath after adding a [illegible] excess of hydrochloric acid. Soda is precipitated in the dried [illegible] residue by adding 15 ml zinc uranyl acetate solution; allowing it to rest for about one-half hour, agitating it frequently. It is filtered through sintered glass crucible, grade G-4, washed with alcohol saturated with trisodium zinc [illegible] acetate and ether or acetone and dried at 105° [illegible] $NaZn(UO_2)_3 \cdot (C_2H_3O_2)_9 \cdot 6H_2O$

Zinc uranyl acetate solution is made by dissolving [illegible] g [illegible] uranyl acetate with 6 g acetic acid (30 percent) in [illegible] ml water on water bath. Alternatively it may be prepared by mixing in equal volumes, (a) uranyl acetate solution prepared by dissolving [illegible] g of uranyl [illegible] and 6 g of acetic acid (30 percent) in a little water and diluting to 65 ml on the water bath, (b) zinc ac[illegible] solution made by [illegible]ing 30 g of zinc [illegible] and 3 g of [illegible] acid in a little water and diluting hot to 65 ml with water.

8.6 Organics

Importance of organics in Bayer process is explained in section

6.2.6 and 6.2.7.

6.6.1 Carbon Dioxide Absorption Method

The method consists in oxidizing the organic impurities to carbon dioxide. Interference from carbon dioxide originating from sodium carbonate is prevented by acidifying it earlier and boiling with [illegible] sulphuric acid. [illegible] dioxide is measured volumetrically [illegible] alternatively, it may be estimated by the [illegible] in [illegible] solution as indicated in section [illegible] (20).

The oxidizing agents used are potassium dichromate, potassium permanganate, and potassium persulphate in presence of silver catalyst. The effect [illegible] is [illegible] by classical combustion. [illegible] Potassium permanganate (KMnO4) is most commonly used.

Refinements include eliminating the carbon dioxide evolved from the sodium carbonate content on acid treatment by bubbling air in the cold instead of by boiling. This [illegible] loss of [illegible] in [illegible] with sulphuric acid. [illegible] the carbon [illegible] is passed through hot [illegible] permanganate to convert any carbon monoxide to carbon dioxide.

6.6.2 Spot Method

A short rapid test of a purely indicative nature (done on spent liquors) consists in titrating the sulphuric acid acidified [illegible] with potassium permanganate of about [illegible]. The end point [illegible] marked by the permanganate pink colour is not sharp and permanent. [illegible] arbitrary standards are set up. For example, considering the end point reached when the pink colour lasts for say 1 minute.

6.7 Silicate

Liquor silicate analyses are needed for the liquor desilication study, one of the [illegible] steps in process design. Spectrophotometric method described in section [illegible] is much more frequently used than the following classical method.

Classical Method

The method is basically the same as that described for silica estimation in bauxite. The solution is acidified with sulphuric and [illegible] nitric acid and evaporated to the evolution of white sulphurous fumes so that the dissolved silica separates out. The process of insolubilising silica is subject to considerable error, because if it is prolonged long some silica may be lost due to the presence of hydrofluoric acid generated from the sodium fluoride impurity in the liquor, and if it is stopped earlier, some silica may escape detection.

8.7.8 Spectrophotometric Method

Based on colorimetry of blue molybdenum complex. Outlined in section 10.3.7.

8.8 Vanadium

Importance indicated in section 4.2.7.

The method is based on titration of vanadium converted to completely vanadic state, with ferrous sulphate in presence of ferrous orthophenantrol, as coloured indicator (called "ferroin"). The organics must be absent because they give strongly coloured compounds with vanadium ions in acid medium.

A 100 ml of solution diluted with equal volume of water is acidified with 200 ml of sulphuric acid (1.6 density) and treated for the organics destruction with a few drops of hydrogen peroxide, (30 percent). The solution turns brick red. It is then boiled for 10 – 15 minutes.

Vanadium content is now oxidised to the vanadic state by repeated oxidation with potassium permanganate as follows.

A slight excess of N/10 potassium permanganate is added to the above solution in the hot, based on the appearance of pink end point lasting for several minutes. The excess potassium permanganate is destroyed with N/10 ferrous sulphate solution adding 20 ml excess of the latter.

The solution is now cooled and reoxidised with potassium permanganate to the point that the pink colour persists for several minutes.

The solution is now ready for titration.

The excess permanganate is destroyed with 3 percent sodium nitride solution, and titrated slowly with N/10 ferrous sulphate, in presence of "ferroin" indicator turning red brown from blue green. It has to be noted that the end point is not sharp and the colour requires some time to develop.

The orthophenantrolin indicator is prepared by dissolving 7.5 g orthophenantration and 5 g mohr's salt in 50 ml water.

8.9 Phosphorus

Importance explained in section 4.2.4 and 4.2.7.

Determined by spectrophotometry of molybdenum blue - see section 10.3.3.

8.10 Dissolved Iron

Defined as the iron content of liquors not separable by filtration. Excessive content contaminates the alumina product.

Determined by spectrophotometry based on ferrous O-phenantroline colour - see section 10.3.2.

8.11 Gallium

Importance explained in section 4.2.7.

Estimation is based on separation of gallium in the form of chlorogallic acid by ether extraction in hydrochloric acid medium, followed by precipitation with 5.7 dibromo - 8 - hydroxyquinolein. Iron should be absent at the precipitation stage. Trace contamination of ferric ion is taken care of by titanium trichloride solution as mentioned later. The gallium present in the test solution should not exceed 0.7 mg to avoid too bulky a precipitate.

In the ether extraction stage, hydrochloric acid concentration should correspond to 5.6 N. The solution is filtered, if not clear. Ethyl ether should be freshly distilled before use. Double extraction is done to ensure complete gallium extraction. Ether: test solution ratio is 2.5 : 1.

The extracted gallium ether solution is purified by adding 5.6 N hydrochloric acid and a few drops of titanous trichloride, the impurities ending up in the aqueous phase. The purification step is repeated with 5.6 N HCl followed by evaporation of ether from the purified ether phase. Gallium is precipitated with 5.7 dibromo - 8 - hydroxyquinolein at 60 °C from the solution containing 30 percent acetone and adjusted to hydrochloric acid concentration 0.06 N. The precipitate is filtered through sintered crucible, grade 4, making sure that filtration is finished before the solution cools to below 50 °C: otherwise some dibromohydroxyquinolein will separate out. The beaker is washed with hot acetone - hydrochloric acid solution.

The precipitate is dried at 105 - 110 °C for 2 hours and weighed. The dessicant used is anhydrous phosphorus pentoxide. The precipitate consists of Ga $(C_9H_4NBr_2O)$.
5.7 dibromo - 8 - hydroxyquinolein solution is made in acetone, corresponding to 3 gpl concentration.
Precipitate wash solution consists of 0.06 N solution of hydrochloric acid, containing 30 percent acetone, obtained by diluting 300 ml of N/5 hydrochloric acid and 300 ml of acetone to 1 litre.

8.12 Chloride

Chloride ion concentration raises the equilibrium solubility level of alumina in caustic liquors and thus needs to be reviewed as a part of liquor contamination study program as mentioned in section 4.2.6.

Method

Estimation is based on precipitation of chloride as silver chloride from a solution free of organics. The organics are destroyed by acidifying the liquors with nitric acid, followed by addition of potassium permanganate crystals and boiling. The 'purified' solution will show persistent pink colour. Chloride is precipitated in the cold with N/10 silver nitrate solution and allowed to stay overnight in the dark.
The precipitate is filtered in sintered glass crucible and dried at 105 °C until constant weight.

8.13 Sulphate

Sulphate ions also raise the equilibrium solubility level and need rewiew as mentioned for chloride.

Method

Based on precipitation with barium chloride in an organic-free solution. The organics are destroyed by potassium permanganate in presence of hydrochloric acid. Precipitation is done in hot, adding

N/10 barium chloride drop by drop. The precipitate is left overnight and solution tested for any further need of barium chloride. The washed precipitate is ignited at 1000 °C.

8.13.1 Oxalate

Excessive presence of oxalates in the liquor generates excessive fines during precipitation, creating problems of hydrate recovery from the spent liquor; thus needs to be checked.

Two methods are generally used. One is titration with potassium permanganate as described in section 8.3.2 on determination of organics by short method. The other method titrates the acidified solution with 2 molar sodium cerate - sodium perchlorate solution.

9. Chemical Analyses for Alumina Trihydrate and Alumina

Type of information required is explained in section 5. Both the trihydrate and alumina are treated here together, because except for soluble soda estimation in trihydrate obtained on precipitation, the methods are generally common to both. Of course, the trihydrate dissolves much more readily in preparation of the samples for analyses. Since International Standards Organisation has standardised methods for practically all the constituents, description here will be purely indicative.

9.1 Moisture

Determined by weight loss on 2 hours heating at 110 °C for the trihydrate and at 300 °C for calcined alumina. For alumina, 100 g sample reduced to pass all through 100 mesh screen is used in a platinum dish as per ISO document 366, ISO/TC 47, October 1964. To estimate the atmosphere-adsorbed moisture, a 5 gm sample is taken in a weighing bottle.

Dessicant used (for the dessicator) anhydrous phosphoric acid, activated alumina or phosphoric anhydride.

9.2 Loss on Ignition (LOI)

Determined by weight loss at 1200 °C, on the material dried as in section 9.1. While handling alumina trihydrate, initial heating should be gradual and careful to avoid spurting losses. Dessicant used as for section 9.1.
ISO document 375, ISO/TC 47.

9.3 Dissolving of Alumina

All the sample should pass 100 mesh tyler screen. Coarser fraction is reduced with corundum mortar and pestle.

5 g alumina is generally fused with 12 g anhydrous sodium carbonate boric acid mixture (3 : 1), except for estimating sodium where fusion mixture of ammonium chloride and calcium carbonate is used, and for gallium where sodium carbonate is replaced by a mixture of sodium and potassium carbonate to avoid interference from sodium chloride crystallisation in hydrochloric acid medium during subsequent step. Of recently, use of hydrochloric and sulphuric acid

to dissolve alumina, at 250 °C and above in sealed tubes, is receiving attention for its merit in eliminating presence of several foreign metallic ions in the main solution.

The soda-boric acid fused mass is dissolved in water and excess 8 N nitric acid to produce a final pH of about 1 when the solution made to 500 ml, or about 0.4 when made to 250 ml.

Fusion temperature used 1000 °C.

ISO document 369, ISO/TC 47, October 1964.

9.4 Silica

On an aliquote of the solution prepared as per section 9.3, by photocolorimetric estimation of yellow molybdenum complex as outlined in section 10.3.7.2.

9.5 Iron

On aliquote from the solution prepared in section 9.3, by spectrophotometry of ferrous orthophenantrolin. See section 10.3.2.3.

9.6 Titanium

On aliquote from the solution prepared in section 12.3, by spectrophotometric estimation with hydrogen peroxide and tiron. See section 10.3.1.2 ISO method uses tiron.

9.7 Phosphorus

On aliquote from the solution prepared in section 9.3, by spectrophotometry of molybdenum blue. See section 10.3.3.3.

9.8 Zinc

On aliquote from the solution prepared in section 9.3, by spectrophotometry of zinc dithizonate in carbon tetrachloride.
An alternate method uses atomic absorption spectrophotometry of the alumina solution obtained on digestion with hydrochloric acid at 250 °C.
See section 10.3.9.

9.9 Soluble Soda for Trihydrate

50 g of alumina is heated for 1 hour on sand bath with 300 ml water and 30 ml N/5 hydrochloric acid in presence of phenol red indicator and excess acid titrated with standard caustic soda. Sand bath heating is used to avoid hydrochloric acid loss from accidental boiling.

9.10 Total Soda

Flame photometric method outlined in section 11.1.3 and is the accepted International Standards Organisation method.

Gravimetric method consists of precipitating with zinc uranyl acetate.

1 g alumina is fused with a mixture of ammonium chloride and calcium carbonate in proportion of 1 : 6. Intimate mixing of the components and gentle heating in the beginning is important because of volatile nature of ammonium chloride. The fused mass is taken up in water and filtered.

The filtrate and washings are evaporated to 200 ml total volume and calcium separated by precipitation with 40 ml saturated ammonium carbonate at 50 – 70 °C in presence of ammonia.
The filtrate is evaporated to dryness and ammonium compounds driven off by heating with an open flame. The residue is taken in a few ml of water and a few drops of hydrochloric acid, neutralised with ammonia followed by addition of one crystal of ammonium carbonate. The solution is filtered and made up to 100 ml. An aliquote equivalent to about 0.25 g alumina is taken for zinc uranyl acetate precipitation as given in section 6.5.

9.11 Calcium Oxide

Calcium is mostly determined by spectrophotometric method given in section 11.1.5. Gravimetrically, calcium oxide is determined by precipitation with ammonium oxalate in solution free of alumina, iron and titanium.

Alumina is separated by treating the aqueous extract of alumina fusion with sodium carbonate - boric acid mixture with caustic soda, followed by precipitation in the hot with saturated sodium oxalate solution. The precipitate is freed of alumina, titanium and iron occlusions by dissolving in 50 percent nitric acid, boiling to decompose the oxalate, and followed by ammonia precipitation as outlined in section 6.5.1. Calcium is then determined in the filtrate by precipitating in the boiling condition with ammonium oxalate. The solution is kept warm for 3 - 4 hours to complete the precipitation.

9.12 Gallium

Gallium is extracted as chlorogallic acid with ether as outlined in section 8.11.

Calcined alumina is fused with a mixture of potassium carbonate, sodium carbonate and sodium borate (in proportion of 1.5 : 1.33 : 1) and extracted with water and hydrochloric acid by boiling. For ether extraction, the solution is evaporated to 100 - 120 ml volume for a 5 gram sample, and hydrochloric acid concentration adjusted to correspond to 5.6 N.

9.13 Chromium

Determined by spectrophotometric method using diphenylcarbazide, as outlined in section 10.3.6.2.

10. Colorimetric and Spectrophotometric Methods

(To include bauxite, mud, liquors and alumina)

Spectrophotometric methods are also called absorptiometric methods.

Both these methods depend on development of characteristic colours. These methods are in a way adjunct to the classical chemical methods described earlier, because the latter are needed for many of the preliminary separations necessary for successful selective colour development.

The methods are particularly suited for estimation of constituents occurring in low concentrations, using fairly simple and relatively inexpensive equipment, easily understood by an average laboratory chemist.

With increasing consciousness of role of minor impurities on the alumina process and the finished product aluminium, for which alumina is produced, these methods have an important role to play. Considerable effort is on foot to improve their range and precision. Complications arise from the effect of foreign elements on the particular colour formation. Classical methods of dissolution are being reviewed to minimize the variety of ions introduced into the solution at various stages. For example, dissolution of alumina in hydrochloric acid and sulphuric acid at as high a temperature as 250 °C - 300 °C is being seriously examined, even if it means use of 80 atmosphere pressure gold lined autoclaves.

10.1 Colorimetric Technique

Colorimetric methods are based on duplication of colours against the standard solutions of known concentration. White light is ordinarily used.

Process of colour duplication may be done subjectively i.e. an individual goes by his own judgement, with consequent varying degrees of personal errors involved. Besides, 7 percent is the minimum difference in colour intensity that an average man can identify.

An objective and more accurate method would use a photoelectric cell- called photoelectric colorimetry, but no instrument simple enough has been yet developed.

For visual colour matching, two methods are generally used - colorimetric titration and balancing method involving use of Duboscq/colorimeter, for example.

In colorimetric titration, a standard solution of the constituent being determined is added from a microburette to the comparison tube containing the colour producing reagent. The only special apparatus required are two flat bottomed tubes 50 - 100 ml capacity, called Nessler tubes, having identical dimensions and optical transparency, and a microburette. An essential requirement is that the colour development must be very rapid and the coloured product stable. Thus it will not really suite iron determination by the thiocyanate method where the developed colour fades rapidly or manganese estimation where permanganate colour development is slow. However, it remains a good field method.

In the balancing method, height of the standard solution in the identical comparison tube is varied till the colours match. Concentration C_x in the test solution will then equal concentration C_s of the standard solution X height of the standard solution / height of the test solution. Thus it is much faster.

Field brightness is important factor in the colour matching precision. So practicability of filters, especially in light coloured solutions, is generally doubtful.

In bauxite feasibility studies, colorimetry is restricted mostly to iron and titanium analyses in the field, and the laboratories have mostly adopted spectrophotometry as a standard equipment.

10.2 Spectrophotometry Techniques

Spectrophotometry uses light consisting of a narrow band of wave lengths (ideally a "single wave length") instead of straight white polychromatic light used in colorimetry. The concentration of the constituent concerned is determined by degree of absorption of the selected radiation.

The constituent concentration is read from the standard absorption curves made from the solutions of known concentrations.

The spectrophotometry has an inherent advantage of sensitivity, precision and versatility over colorimetry, because it allows the choice of different wave length lights, including ultra-violet and infra-red, though the latter are not needed with bauxite and alumina.

Improved versatility and precision result from ability to play with different wave lengths so that the interferences from the foreign ions can be avoided and often single solution can be used for estimation of two different costituents, as for example vanadium and titanium. Thus spectrophotometric methods are generally preferred and form part of every laboratory equipment.

However, colorimetry has its own restricted field [illegible] especially in [illegible] sophisticated [illegible] situations and where [illegible] occasional determinations of iron and titania are made, because [illegible] is simple and does not need the standard curves [illegible] unit, a [illegible] is needed for spectrophotometric [illegible]

10.2.1 Spectro[illegible] Types

For choosing different wave length radiations, [illegible] are possible [illegible] filters and diffraction gratings.

Diffraction gratings give purer wave length selection [illegible] to filters which are at best narrow bands of wave [illegible] latter disadvantage sometimes [illegible] apparent deviations [illegible] Beer's law. [illegible], the wave length choice with the [illegible] necessarily limited.
The filter [illegible] light sources [illegible] with time [illegible] curves should, therefore, be checked from time to time.

For bauxite [illegible] bilities in [illegible], filter [illegible] ters are normally adequate because absorption bands [illegible] generally [illegible] high intensity tungsten bulbs [illegible] for the light source. For photoelectric cells, photo[illegible] are preferred because intensity of light is [illegible] lective filters can be used. The absorption co[illegible] mm.

10.3 Individual Estimations

10.3.1 Titanium

Applicable to bauxites, muds, process liquors and alumina. Hydrogen peroxide is generally used for colour developing because [illegible] lack of interference from common contaminations, [illegible] procedure simple. Beer's law of linearity holds [illegible] concentrations as high as 50 ppm. The method for alumina [illegible] "Tiron" [illegible] um -1, 2-di[illegible] benzene - 3,[illegible]

Both visual colorimetry and spectrophotometers are [illegible] for bauxites and muds. Lowest limit for visual color[illegible] 0.4 [illegible] titania content. In the field testing, [illegible] be extended [illegible] -12 percent titania content bauxite [illegible], only spectrophotometer is used.

10.3.1.1 Bauxite and Muds

Hydrogen peroxide method is used **for bauxites and muds**, starting solution is the silica free liquor from alkali fusion as described in section 6.4.2

For visual colorimetry, both Nessler tubes and Duboscq colorimeter are used. 5 percent sulphuric acid solution is added to the [illegible] solution till the colour matches with the standard.

For spectrophotometry, phosphoric acid is added to suppress the iron colour. Filter [illegible] [illegible] cell 20 mm for bauxite and red mud, and [illegible] mm for alumina.

Standard solutions used for comparison and making standard curves are made from [illegible] [illegible] bisulphate fusion of a calcined titania dissolved in dilute [illegible] acid, or from potassium titanium [illegible] [illegible] [illegible] [illegible] acid a task, depending on the relative purity of the available material. The stock solution contains [illegible] g TiO_2/l. These are subsequently diluted as desired.

The calibration curves [illegible] drawn up adding varying amounts [illegible] standard [illegible] [illegible] percent sulphuric acid, after [illegible] 3 percent [illegible] and waiting [illegible] minutes.

Sulphuric acid concentration influences the colour intensity; for comparative tests it should be about same, normal concentration being 5 percent

Interference. Alkali metal sulphates exert [illegible] [illegible] [illegible] sulphuric acid effect [illegible] counteracted, unless they are present in excessive amount. Phosphates and fluorides interfere considerably to suppress the colour. Vanadium interferes to give [illegible] own colour. Vanadium interference is fairly well cancelled with spectrophotometer, using wave lengths of 41[illegible] and 46[illegible] nm.

10.3.1.2 Alumina

Titanium in alumina is determined by Tiron method as per ISO [illegible] document 42[illegible], [illegible], June 1965.

The method is based on spectrophotometry of yellow titania complex formed with tiron [illegible], bisodium - 1,2 - dihydroxybenzene - [illegible] disulphonate.

Modifications required for alumina with Fe_2O_3 and V_2O_5 contents greater than [illegible].015 and 0.[illegible]06 percent are explained in sections 10.3.1.2.1 and 10.3.1.2.2.

Starting [illegible] buffer [illegible] Fail [illegible] Filter [illegible]

[illegible]

10.3.1.8.1 [illegible]

[illegible] the blank [illegible]

10.3.1.8.2 [illegible]

[illegible] Since [illegible] length [illegible]

10.3.2 [illegible]

[illegible] upto 5 per [illegible]

For [illegible] and [illegible]

The [illegible] is based on formation of [illegible] from [illegible] and [illegible] used as [illegible] during [illegible]

[illegible]

[illegible]

[illegible]

[illegible]

[illegible]

[illegible] [illegible]

[illegible]

[illegible]

10.3.3 [illegible]

[illegible] by [illegible] in presence of [illegible] The meth[illegible] [illegible] and [illegible] Deionised water [illegible]

10.3.3.1 [illegible]

[illegible]

[illegible]

pink colour and the solution [illegible] destroyed [illegible] hydrogen [illegible] and [illegible] in the [illegible] addition with [illegible] turning [illegible] the [illegible] is [illegible] and [illegible] and [illegible] is added to prevent the formation [illegible]

10.3.3.3 [illegible]

[illegible] the [illegible] others, [illegible]

[illegible]

[illegible]

[illegible]

[illegible]

[illegible]

[illegible]

[illegible]

[illegible]

[illegible]

[illegible]

[illegible]

Interfe[illegible] are silica, [illegible] and [illegible] at [illegible] concentrat[illegible] the oxidati[illegible] [illegible]

[illegible]

[illegible] principle as [illegible] used for [illegible]

For [illegible] alumina, [illegible] tubes at [illegible]

[illegible]

[illegible]

[illegible]

[illegible] good

[illegible]

[illegible] of the [illegible]

[illegible]

[illegible]

10.3.5. [illegible]

[illegible] photometry [illegible] extract [illegible] phenylhyd[illegible] complex for [illegible] equivalent [illegible] medium. The [illegible] acid and [illegible] V_2O_5 [illegible]

[illegible] acid [illegible] carbonate [illegible] state [illegible] chloroform [illegible]

[illegible] diluted [illegible]

[illegible]

[illegible]

[illegible]

[illegible]

[illegible]

	[illegible]	[illegible]	[illegible]
	[illegible]	[illegible]	
	[illegible]	[illegible]	[illegible]
	[illegible]	[illegible]	
	[illegible]	[illegible]	[illegible]
	[illegible]	[illegible]	
	[illegible]	[illegible]	[illegible]
	[illegible]	[illegible]	
	[illegible]	[illegible]	[illegible]
	[illegible]	[illegible]	[illegible]
	[illegible]	[illegible]	
	[illegible]	[illegible]	[illegible]
	[illegible]	[illegible]	[illegible]
	[illegible]		[illegible]
	[illegible]	[illegible]	[illegible]
	[illegible]		
[illegible]	[illegible]		
	[illegible]	[illegible]	[illegible]
	[illegible]	[illegible]	[illegible]
	[illegible]	[illegible]	[illegible]
	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]		
	[illegible]	[illegible]	[illegible]
	[illegible]		
[illegible]	[illegible]		
	[illegible]	[illegible]	[illegible]
	[illegible]	[illegible]	[illegible]
	[illegible]	[illegible]	[illegible]
	[illegible]	[illegible]	[illegible]
	[illegible]	[illegible]	[illegible]
	[illegible]	[illegible]	[illegible]

[illegible]

ble) and reduce the [illegible] direct [illegible] to the calcium content. The sol[illegible] red through fine [illegible] rejected. Clear filtrate [illegible] comparison with the blank.

Naphthalhydroxamic acid [illegible] free acid with [illegible] KOH in water [illegible] Naphthalhydroxamic [illegible] anhydride and [illegible] for 5 minutes, [illegible] from the [illegible] with cold water and dried [illegible]

[illegible] by [illegible] For the graph [illegible] mixed with the [illegible] decomposition, [illegible] Filter: 4.5 cm [illegible]

10.1.? [illegible]

[illegible]

a) [illegible]
   [illegible]
   ([illegible]

b) [illegible]
   [illegible]

10.1.? [illegible]

[illegible]

[illegible]

[illegible]

[illegible]

[illegible] [illegible]

[illegible]

[illegible]

[illegible]

[illegible]

[illegible] [illegible]

[illegible]

[illegible] [illegible]

[illegible]

[illegible]

[illegible]

[illegible]

[illegible]

[illegible]

[illegible]

[illegible]

[illegible]

[illegible]

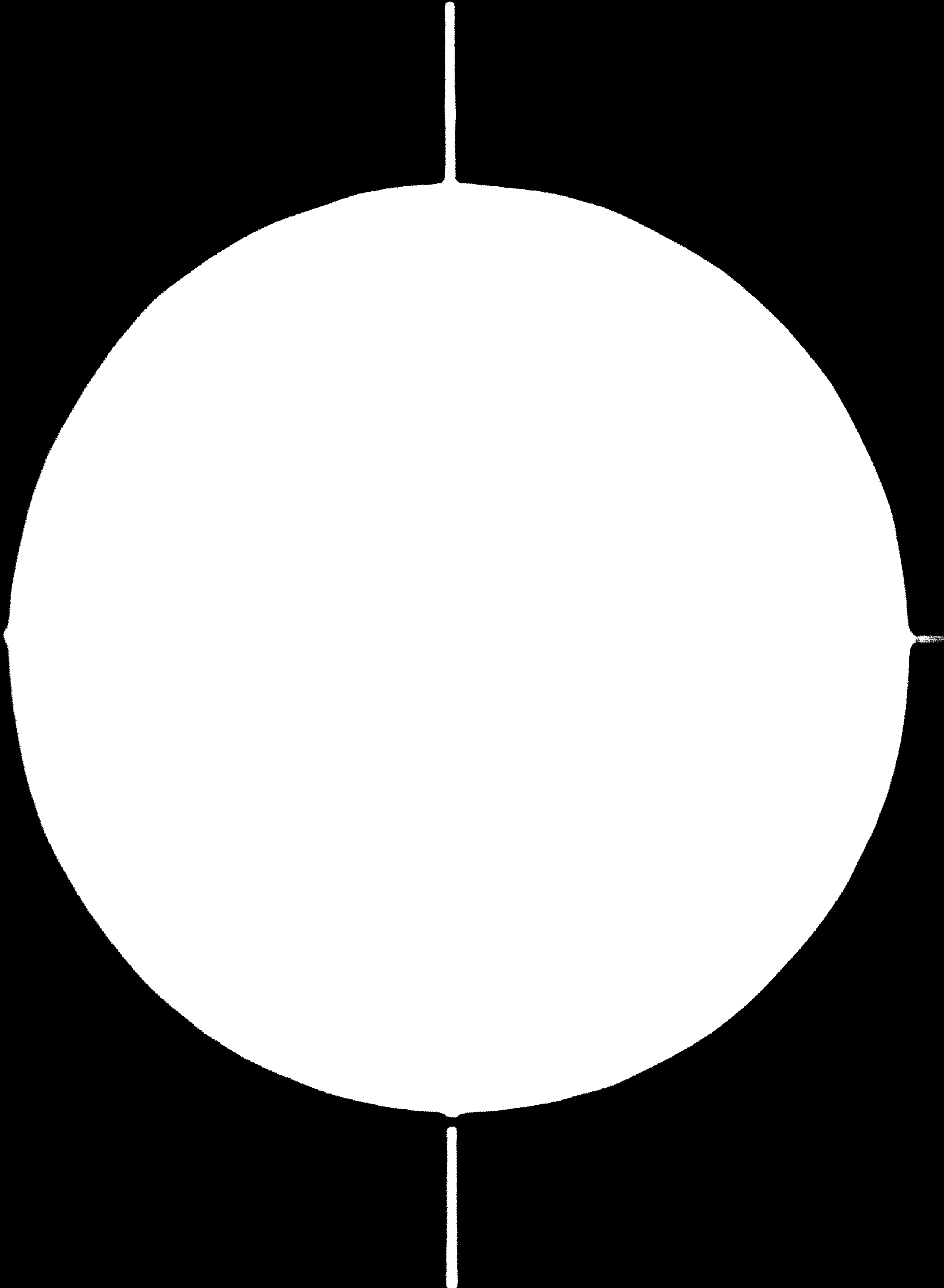
[illegible]

[illegible]

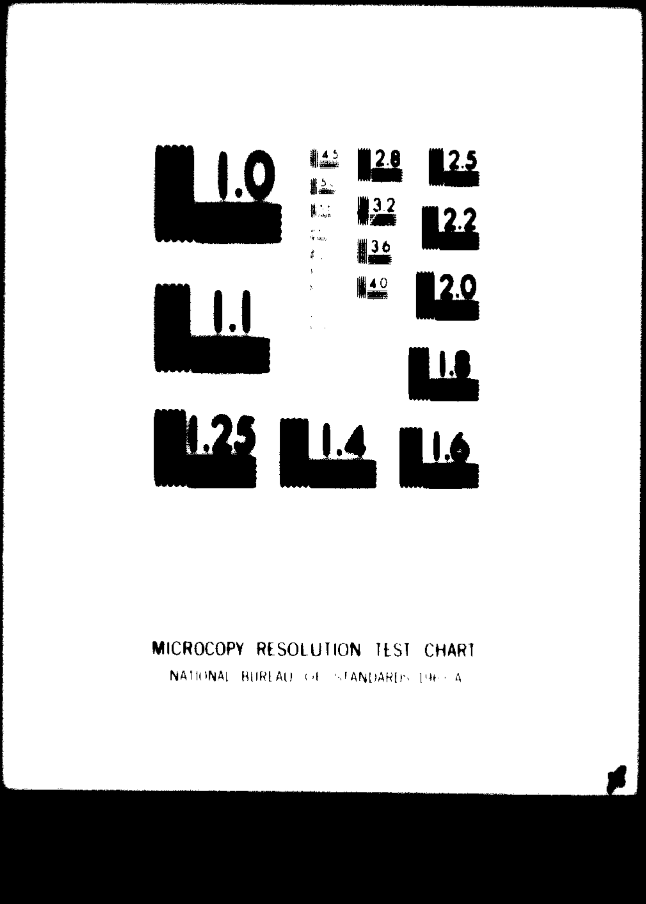
1 - 827

→

82.06.24



2 OF 2



MICROCOPY RESOLUTION TEST CHART
NATIONAL BUREAU OF STANDARDS 1963-A

24×
E

Similarly 35, 10, 5 micron diameter particles will be found after intervals of 2 min. 6 sec., 27 min. 46 sec., and 111 min respectively.

The fluid used for slurrying is 1 gpl sodium tartarate solution.

One of the commonly used pipettes for drawing the sample is shown in Fig. 14. It is fixed to a support which can be moved up or down by a micrometer in front of the scale in mm. The bottom of the pipette is closed. Four holes drilled laterally allow the sampling on a horizontal plane. The volume of the pipette between the two way cock R 2 and the bottom E is approximately 10 ml. This is determined exactly.

The weight of the test sample taken is such that every sample drawn corresponds to about 0.1 g of the bulk sample.

The slurry sample is collected by suction through C. Outlet D serves to discharge the excess slurry collected in the bulb B. Separatory funnel A contains water to flush out the bulb B. The solids drawn in the sample are determined by filtering through sintered glass crucible grade 4, followed by drying at 110 °C.

The results are calculated as follows:

$\leqslant$ 35 micron = a gram
$\leqslant$ 20 " = b "
$\leqslant$ 10 " = c "
$\leqslant$ 5 " = d "

Since every sample is equivalent to 0.1 g sample, $\leqslant$ 5 microns = d

5 - 10 microns	c - d
10 - 20 "	b - c
20 - 35 "	a - b
35 - 44 "	0.1 - a

Recently a method based on the principle of solid particles sedimentation in air has found great favour, because of the readiness with which it can be automated and requirement of comparatively much less operational skill[21]; In the reported procedure using Sharples micromerograph, a 50 mg sample of dried material is placed at the top of a 220 cm long column, both sealed air tight with clamps.

The sample is "blowed" into the column by building up 50 pound nitrogen pressure behind the sample dish followed by sudden pressure release through a solenoid actuated valve. This burst of gas sweeps the sample through a deagglometer and into the very top part of the column as cloud. The particles as they settle down in the order determined by their diameter are continuously weighed so that the resulting chart is a record of accumulated weight versus time. The equipment is, however, too expensive for its justification for bauxite investigations where the number of samples involved is quite limited.

14.10. Surface Area Measurement

Surface area estimation of the hydrate are widely used in precipitation studies for seed balance calculations and provide direct overall size distribution index for both the seed and the product.

Surface area can be estimated from the sedimentation test data as determined in section 14.9. but it is very tedious, and requires much care and attention to give consistent results.

The procedures most commonly used are:

a) Photoelectric
b) Air permeability

Photoelectric method consists in dispersing 0.2 - 0.5 g sample of trihydrate or alumina in ethylene glycol and noting the degree of light absorption with the help of a photoelectric cell, output being registered in milliamperes, (analogy of spectrophotometer).

If I_o represents the light intensity transmitted with the blank, I_t light intensity transmitted through the suspension, the ratio I_t/I_o will be proportional to the ratio of projected area of all the particles to that of the light beam. The quantity of test sample is varied to have milliampere indication in range of 8 - 20. Particles in the size range of 10 ÷ 20 millimicrons are found to influence absorption strongly.

The entire test takes only about 2 - 3 minutes. The appartus is exceedingly simple, similar in essentials to that of a spectrophotometer. Tungsten bulb serves for the light source. The gycol container consists of quartz, generally 5 cm square with capacity of about 200 ml. The apparatus is sometimes called photoelectric sedimentator.

Air permeability method is based on the principle that the air permeability values at a given bulk density are largely dependent on the particle size, and thus the surface area.

A fixed weight of the dry powder is taken in a permeability cell generally made of sintered glass bottom. The permeability is determined either by noting the upstream air pressure for some standard flow of air through the permeability cell or by noting the air flow for a fixed upstream pressure. The quantity of test sample/ varies from 5 - 20 g depending on the apparatus used. Here again the actual test takes only 5 minutes.

Both these methods provide an index of total surface area which may not reflect the actual size distribution of the sample. Besides, the values provided for the total surface are not strictly absolute, for which it will be necessary to go to Andreason method described in the previous section. However, the values obtained so quickly, reflect the process behaviour fairly well and form a generally acceptable base.

Air permeability method is being increasingly preferred over the photoelectric method. A very popular apparatus is Fisher Sub-Sieve Analyser.

14.11. Microscope Examination of the Hydrate

Microscope examination forms an essential part of the precipitation studies. Of special interest is recognising the point where nuclei begin to be formed because these are difficult to recover in decantation and their excessive presence hurts filtration and hydrate washing operations.

For this, the particles have to be recognised in the range of 1 micron size. For quantitative studies, it is necessary to know the sample density mg/cm2 of the surface. One of the recently successful attempts in solving this problem is as follows.

100 ml of well dispersed solution of 0.5 - 50 mg of the sample is filtered through a millipore cellulose acetate filter with a pore size of 0.45 millimicron (Millipore Cat.n° HAWG 047-00). Thus a sample of known weight is distributed over a known area.

The filter is opaque and is itself composed of 1 micron cellulose particles. To allow microscopic identification of 1 micron size hydrate particles, the constituent particles of the filter have to be destroyed into transparent stage. This is done by laying the filter on a microscope slide coated with acetone which fuses the cellulose particles to leave on evaporation a thin transparent film of cellulose acetate on the slide. The hydrate particles can then be clearly examined by transmitted light.

To examine the particle shape, monobromonaphthaline reagent has been used. The reagent has refractive index greater than gibbsite, usual precipitation form of trihydrate, thus bringing out the contours clearly.

To bring out inclusions and inhomogenities present inside the grain, eugenol is added to monobromonaphthaline in ratio of 2 to 1, to depress the overall refractive index and make the inhomogenities appear as dark spots.

Microscopic examination also helps ready recognition of different forms of alumina produced. Alpha-alumina, for example, looks dark in polarised light.

Dispersing agent commonly used is ethylene glycol.

14.18. Product Purity

Product purity considerations are discussed in sections 4.2.7. and 5.

Preliminary evaluation (which is actually rather drastic) of the bauxite impurities effect on final product purity is effected on the desilicated slurries after filtration and dilution.

Carbon dioxide is passed through the clear solution to precipitate all the impurities along with alumina. A material balance performed on the starting liquor and the precipitated product gives the amount of particular impurity dissolved and possible product contamination. This test generally establishes the maximum limit of the product contamination because the alumina precipitated with carbon dioxide has much higher absorptive power than the alumina precipitated in plant practice.

Border line cases of product contamination will require more detailed studies, taking into account interaction of various liquor contaminations on the solubility of concerned impurities. The product contamination will also be studied on the basis of alumina hydrate precipitates produced by procedures outlined for precipitation yield studies in section 14.8.

14.19. Wet Screening of Bauxite and Mud Slurries

Wet screening of bauxite slurries is needed during bauxite washing studies, as mentioned in section 4.1., and during bauxite wet grinding. Wet screening of the mud is needed while establishing optimum grind size of bauxite.

Details of a commonly used apparatus is shown in Fig. 15. The slurry is poured over the top (largest opening screen) and continuously washed with water on all the screens. The vibrating intensity is regulated to avoid excessive spuzing. The finest fraction is recovered by filtering the slurry using buckner funnel. The vibration is about 50 cycles per second.

14.14. Viscosity of Bauxite - Liquor slurries

This information is needed at the time of plant erection to determine criteria for pumps and pipe selection, which will be different for plastic character slurries and sandy character slurries. Plastic slurries (say 50 % bauxite) will show viscosity higher than 10,000 cps while sandy type may show just about 200 - 300 cps.

The determination is made by a model LVT Brookfield viscometer.

15. Pilot Plant Testing

15.1. Scope

The ultimate purpose of pilot plant trials is crystallised very well in the famous phrase "Commit your blunders on a small scale and make your profits on a large scale". Thus need of such trials is determined essentially by the degree of ignorance and to bridge the gap between the laboratory technology tests described earlier and full-scale commercial operation.

For analysing our needs, general motives for pilot plant trials may be broken down as:

a) to reduce capital costs of the plant through more accurate data;

b) to spot basic process complications that could lead to large investments just lying unproductive till the process difficulties got eliminated.

c) to confirm saleability of the product produced

d) to reduce operating costs through improved raw material conversion efficiency

In deciding on pilot plant programs, it is to be remembered that pilot plantsare expensive to build and operate. The studies are time-consuming and any time spent means profit loss for the equivalent time in case of alumina where the plants are built on assurance of ready markets. Principal consideration is that of acceptable quality. So the old concept of pilot plants as "a collection of equipment used to develop the most economically sound method of commercially producing a material" has undergone considerable modification. The pilot plants are used for only very well-defined limited answers, based on hard-headed calculation of pilot plant study costs and expected financial benefits.

A good plant design allowing for considerable flexibility can successfully by-pass a great deal of uncertainty in data. And if the cost of pilot plant trials (including the lost time on delay in building the plant) exceeds the cost of additional flexibility, pilot plant trials are not warranted. With the improvement in knowledge on scale-up techniques, especially in the reactor design from the batch laboratory data, the use of pilot plants to just confirm the scale-up factors may often be an expensive insurance or luxry. Inasmuch as the basic equipment cost constitutes only 20 - 32 percent of the overall plant costs, significant reduction will occur only if there was a hope of eliminating any process step or lessening the severity of processing conditions or reducing material requirements.

For alumina plant feasibility studies, considering the overall state of known art, broad operating parameters can be reasonably well established by competent analyses of the results obtained from technological tests detailed earlier. Potential equipment vendors (working on a competitive basis!) provide very useful supplementary and practical extrapolations on these data to enable equipment sizing. Warranties and demonstrations of practicabilities of their offered equipments are very frequently volunteered. Continuous run pilot plant trials are, however, still considered necessary to establish the overall viability of the process to forestall any unexpected extremely expensive process- and unit-operation complications. Of particular interest are examination of liquor desilication behaviour (thus product purity) mud settling and filtration steps, scaling tendency especially in digesters, heaters and evaporators, and effect of increased liquor contamination on precipitation yield and precipitate grain size.

The operating conditions are those established from the earlier technological tests, taking into account the statistical variability of the bauxite deposit concerned.

15.2. Naure of Facilities and Conditions

The facilities will correspond to an almost miniature alumina plant (including a boiler plant) with the exception of calcination unit, very well instrumentdd and designed for continuous 3-shift operation under well defined and recorded conditions. This requires a well trained operating gang and skilled maintenance workshop help. These conditions demand that the pilot plant be located next to an operating plant, i.e. to build it after the plant! Thus the initial feasibility studies (i.e. before the start of alumina industry) will have to be conducted at an outside source, However, it will still be advantageous to build the pilot plant at a later stage because it could profitably serve as a centre to investigate other deposits, make improvements and changes on the running plant without risking any production loss, and also to develop special products or profitable intermediate products.

Attempt to use the pilot plant for developing special products before obtaining good experience of the existing normal plant will be exceedingly wasteful and frustrating.

While selecting the outside party for pilot plant work, following criteria will be considered:

a) ability to reproduce the range of bauxite grind, digestion temperature and residence time, precipitation holding time predicted from the technological tests.

b) trained staff to ensure continuous operation without loss of time.

c) Sufficient instrumentation and reliable sampling system to ensure authenticity of data.

d) competent laboratory facilities to provide the desired quantity and quality of analytical information.

e) willingness to associate the project process engineers and the laboratory incharge who did earlier work.

15.3. Pilot plant specifications

Necessity to establish a new pilot plant next to an operating plant has been explained in the last section. This naturally makes its initial lay-out oriented to the process already selected. Detailing will be determined by the points of interest established from the operating practice. One outline is indicated in fig. 16. A few general guide lines are given below.

An important consideration is to limit the minimum pipe size used for main flows to 1 1/2" - 2" and still maintain sufficient flow so that the solids do not separate out from the slurries. 8 - 10 t/day bauxite throughput is quite reasonable. For crushing, jaw crusher or hammer mill is used to reduce material to minus 1/2" size.

Bauxite grinding is done in the wet at about 50 - 60 % solids, the mill being such that it can be used as ball mill or rod mill.

Sufficient number of digesters are provided so that the residence time can be varied from 50 percent to 150 % of that roughly selected; flexibility to change the agitator speed on the digesters will be an advantage.

Mud settler should provide area 50 % larger than roughly estimated. For mud washing, one stage is provided followed by a generous size filter. However, space is provided for an additional washer.

For precipitators, the number provided should give a flexibility of 33 percent plus minus on the selected holding time. Invariably there will be desire to study continuous precipitation and relative merits of air and mechanical agitation.
Evaporation capacity should be 15 percent excess of that estimated.

A disc filter is provided to filter the hydrate. Part of this hydrate is used for the seed. Rest is "sold". Space is provided to allow experimentation with other filters and partial classification of hydrate at a later stage.

Need of extensive reliable instrumentation is already explained in section 14.2. Due to operation with slurries and scaling liquors, magnetic flow meters are used for metering.

16. Laboratory Description

Following three models have been considered:

a) Field Laboratory close to mineral exploration areas to satisfy immediate needs expeditiously.

b) Central Laboratory capable of undertaking most of the technological tests and various analyses required in that connection.

c) Central Institute with (i) research potential to develop new analytical methods, processes and technology needed for established alumina industry (ii) pilot plant facilities.

16.1. Field Laboratory - Model I

16.1.1. Scope

The laboratory will be geared to meet the immediate needs of exploration teams required to work in out-of-way places in the spirit mentioned in section 4.1. Important to remember during planning, that living conditions in such areas are generally hard and life rather isolated, which makes it difficult to attract competent help, willing to stay for sufficiently long time. The important demands may be summarized as follows:

a) To receive very large number of bulk samples and ensure their positive identity at all stages. These samples may be very wet and even in slurry form.

b) To make representative samples most scrupulously in the quantities required locally and for outside testing.

c) To estimate large number of samples for moisture, loss on ignition, bulk density.

d) To estimate, quite large number of samples for mixed oxides, iron, titanium, silica and specific gravity. Later, number of titania analyses may decrease substantially. Titania will most probably be determined colorimetrically.

e) To handle large volumes of slurry from some preliminary washing trials on ores occuring near clayey minerals as pointed out in section 4.1.

f) To pack and despatch large number of samples outside and maintain perfect record of details and duplicate samples.

g) Occasional checks to ensure that samples despatched from the mines are representative, consistent with the description in section 6.1.1.

16.1.2. Departments

a) Receipts and sample preparation
b) Analytical department
c) Central office

16.1.3 Department Description - Scope and Facilities

16.1.3.1. Receipts and Sample Preparation

The section will be equipped to receive 100 samples in gunny bags or drums of various sizes. Though the samples from the field will generally be received already reduced to 2" size in the process of sample bulk reduction, the department will have to be prepared for receiving larger sizes. All the samples will be weighed and logged with respect to the source pit location and depth.

Facilites

	Item	N°	~~Required~~ Remarks:
1.	Ten-pound hammers	4	To break bauxite pieces larger than 2"
2.	Manganese steel plate 6'x3'[1)] placed on 6" high concrete slab	1	" " "
3.	Laboratory jaw crusher or roll type crusher, 1 kw	1	To crush the sample from 2" to 6 mesh size
4.	Laboratory grinding mill, hammer or roll type, with changeable built-in sieves, 2 · 2 kw	1	To reduce the 6 mesh size received from the crusher. Finest sieve, 100 mesh.
5.	Laboratory sample grinding machine 0.4 kw Good example, vibratory disc type grinder, Siebtechnic, Mulheim, Germany. Grinding disc made of widium steel (high-tungsten-cobalt-carbon steel)	1	For final grinding of hard bauxite samples to 100 mesh passing from feed size upto 10 mm.
6.	Agat-mortar, diamter about 4"	1	For final grinding, as stand-by for the above
7.	Gas heated hot plate, lined with stainless steel (will be improvised outdoors when required) 6' x 3'	1	For drying the wet samples prior to sampling.
8.	Shovels	4	For coarse bauxite handling
9.	Scoops and spatulas	12	For crushed and ground bauxite
10.	Standard screen set with Ro-tap machine and timer, 8" size screen, spare 6 mesh and 100 mesh screens	1 ~~each~~	
11.	Wooden framed screens, 1'x1' for 28,50 and 100 mesh tyler opening Steel frame sieves, 1 1/2'x 1 1/2' for square opening 1/2" and 1/4" round opening	1 each	For manual handling and rough use
12.	Riffles with six troughs and pans		
	1" size	1	For sample splitting
	1/2" size	1	" " "

1)= All the dimensions indicative. Can be modified to suite prevailing standard sizes, unless otherwise specified.

13. Platform balance, 100 kg capacity	1	
14. Pan balance 1000 g capacity	1	
15. Pans and trays of assorted sizes	24	For handling the samples
16. Sample bottles, plastic		
4 oz. size	1000	maximum use
8 oz. size	250	
16 " "	100	
17. Sample bottles, glass		For sealed samples where required
4 oz. size	48	
8 " "	12	
18. Tarpuline pieces about		To protect the bulk samples temporarily stored outside.
100 sq ft	1	
150 " "	1	
19. 2' x 1' x 1' wooden box with side handles	1	For bulk density determination
20. Steel buckets, 8 l capacity	4	For sample handling
21. Work tables, aluminium lined, 4 1/2' x 2'	3	As indicated in items 1 and 2 of sec. 16.1.3.
22. Wash basin	1	

16.1.[illegible] Analytical Equipment

Types of analyses required are pointed out in section 16.1.1.The facilities should be adequate to handle about 25 samples simultaneously which means about 4 chemists and 2 assistants.

Facilities	Number
1. Drying oven with natural draft adjustable to 300°C	
size 2.5' x 1' - 2 kw	2
size 1.25' x 1' - 1.2 kw	1
2. Electrical furnace with temperature control for 1000 - 1100 °C Chamber size 12" x 6" - 3 kw	2
3. Pan balance, automatic, capacity 200 g, sensitivity 0.01 g	1
4. Analytical balance, quick-weighing type (Mettler e.g.)	3
5. Fume cupboard 6' x 3'	2
6. Meker burners	12
7. Gas supply for the above, 14 cfm capacity generator	1
8. Hot plates electrical, 10"x20", 300°C, 2 kw	4
" " " " ", 100°C, 0.5kw	2
9. Water distillation unit [illegible] [illegible] type, 200 l/hr capacity	1
10. Tripods with triangle supports	12
11. Stainless steel pans for moisture estimation 100 ml capacity, 70 mm dia.	24
12. Platinum (containing 5% gold) crucibles, 30 ml capacity for LoI and precipitate ignition	20
13. Nickel crucible, 70 ml capacity Requirement to be reviewed if type of bauxite is such that caustic soda fusion is needed always.	2
14. Porcelain dish, 200 ml capacity	6
15. Platinum tipped tongs	6
Stainless steel tongs, 1 1/2' long	3

	Number
16. Nickel tongs, laboratory size	6
17. Beakers, glass, 250 ml capacity	24
" " 400 ml "	72
" " 600 ml "	24
" " 2000 ml "	6
" " 5000 ml "	3
18. Beaker, stainless steel, 1 lt capacity	2
19. Beaker, polyethylene plastic, 1 lt. capacity	4
20. Bucket plastic, 2 gallon size	2
21. Desiccators, dia. 150 mm	6
" " 200 mm	4
22. Filter funnels, (long stem) for 12.5 cm filters	24
23. Buchner funnel 135 mm dia.	6
" " 250 mm dia.	2
24. Filter flasks, 500 ml capacity	6
" " 1 lt. "	3
" " 2 lt. "	3
25. Vacuum pump	1
26. Water pump (for filter flasks)	6
27. Titration bench equipment containing self-zeroing 6 burettes, connected to individual stock solution bottles	
28. Conical flasks, 250 ml capacity	48
" " 500 ml "	12
29. Standard volumetric flasks	
100 ml capacity	12
250 ml "	18
500 ml "	24
1 lt "	4
2 lt "	2
30. Graduated cylinders, glass	
50 ml capacity	6
100 ml "	12
1000 ml "	4
31. Graduated cylinders, plastic polyethylene	
250 ml capacity	2
1000ml "	4

	Number
32. Pipettes 5 ml size	6
10 ml "	6
25 ml "	24
50 ml "	6
100 ml "	6
200 ml "	2
33. Graduated pipette 2 ml size	2
" " 5 ml "	6
" " 10 ml "	6
34. Nessler tubes, graduated, 100 ml capacity	6
35. Watch glasses, 2" size	12
3" size	24
4" size	24
5" size	6
36. Wash bottles, 1000 ml capacity	12
37. Weighing bottles, low form, 45 mm dia.	6
38. Spatulas	12
39. Calculating machines	2
40. Specific gravity bottles, 25 ml capacity	6
41. Jones reductor	1 See fig. 2
42. Water bath for 6 beakers - 1 Kw	1
43. Wash basin, common for bulk glass	1
44. Glasscarboys for bulk solution 5-1 capacity	24
45. Reagent bottles 1 lt.	100
" " 250 ml	100
46. Indicator bottles	12
47. Work tables 4'6" x 2'- ovens and furnace room	1
" " 11'6"x 2.5" Analytical lab.hall (sec.16.1.4.4.)	10
" " 15' x 2 ' - Titration bench	1
" " 10' x 1'9''- Balance room	1
Shelf	1
48. Wash basins - for work tables	5
" " - for central glass-ware cleaning centre	1
49. Funnel stands	6

16.1.3.3. Central Office

Since many samples will be received and large number of samples regularly despatched for outside testing, an efficient central office is a must. This will serve as store and central source of all information.

The office should have a large grid map of the entire exploration area, on which every analysis could be recorded for ready reference at any time. The office will ensure that tags on the samples received from the mines carry precise identifying information in the code mutually agreed between the Laboratory and the Exploring group.

The office will have a jeep at its disposal for frequent liason work.

Facilities	Number
Shelf,(1 for 1000 sample bottles of 4 ounce size and 500 bottles of 1 pound size) (1 for glass-ware stock)	2
Cupboard with lock, stationery, records and books	2
" " " Chemicals and glass - ware	1
Filing cabinet	1
Work tables, 4'6" x 2'	2
Typist table, 4'6'' x 2'	1

16.1.4. Rooms and Area Requirement

1. Mines Samples and Miscelleneous

Good stores

To house upto 50 gunny bags of the mine samples as received, one work table, 6' x 3' steel plate mounted on 6" high concrete slab for breaking plus 2" size pieces, plat-form balance and other common utility items such as hammers, shovels, rough screens, bulk density measuring box, tarpuline and buckets. 120 sq.ft.

2. Sample preparation room

To house sample crusher, two grinding mills, sample riffles, pan balance, screens, 2 work tables, wash basin. 150 sq ft

3. Ovens and furnaces room

To house 3 furnaces, 3 drying ovens, 1 work table - 75 sq ft

4. Main analytical laboratory hall

To house 5 work tables for chemists,
2 work tables for making of the reagents,
1 extra work table for any special demand analytical, work or for an invited help,
1 work table for all stock solutions,
1 titration bench,
1 vacuum filtration assembly work bench,
2 fume cup-boards,
1 wash basin for bulk glass-ware cleaning,
1 general service shelf 1000 sq ft total

5. Balance room

To house 3 analytical balances and 1 work table for the dessicators 40 sq ft

6. Records room cum store cum general office

To house 2 shelves for the samples and glass-ware 1 filing cabinet, 2 cupboards for records, stationary and books, 1 cupboard for the chemicals, 1 typist table, 2 work tables and 1 sample counter 300 sq ft

7. Chief Chemist's room

One work table with 4 chairs for the chief chemist, one extra table for any visiting outside help. One cupboard 120 sq ft

8. Distillation unit cell cum switch room 60 sq ft

9. Gas (for the mecker burners) generating room 60 sq ft

10. Toilets 50 sq ft

11. Total area 2000 sq ft

16.1.5. Staff

In view of the earlier mentioned circumstances, that competent non shifting staff will be difficult to get, emphasis will be on training and intensive work load coupled with higher wages.

1. Chief Chemist (1)

Duties: Overall incharge of the laboratory, he will have frequent contacts at all levels with exploration teams, consultants and invited outside help, shipping agencies and local authorities. He will also keep an eye that the samples sent from the mines are representative and tagged with sufficient responsibility. He may personally analyse difficult samples.

Qualifications:

Essential: Graduate degree in chemistry with 5 years proven successful experience in a good chemical mineral testing laboratory. Congenial personality and good health to go with outdoor rough life and people and liason work. Good proficiency in official language , Unquestioned integrity.

Desirable: Knowledge of locally prevalent language, car driving licence.

2. Assistant Chief Chemist (1)

Duties: In absence of the Chief Chemist, he shoulders all the supervision responsibilities independently.

As a part of routine duties, checks all the standard reagents, and watches that standard practices are followed in day to day work. Personally analyses check samples and routine samples during times of heavy work load.

Qualifications:

Essential: 3 years proven successful experience in a good mineral testing laboratory, Higher secondary school certificate. Unquestioned integrity.

Desirable: Good proficiency in the official language and locally prevalent language. Good personality and fair typing skill.

3. **Chemists / Analysts (4)**

Duties: To conduct analyses independently as per set procedures. To supervise sampling of ores.

Qualifications: 2 years experience in a good mineral testing laboratory Alternately 3 months intensive training in analytical techniques to develop discipline of working simultaneously on 6 samples.

Important: evidence of integrity

Desirable: Higher secondary school certificate

4. **Assistants** (4)

Duties: To make bulk reagents, operate water distillation units, undertake sample preparation and screeing independently. To assist the chemists in cleaning and maintaining of every day apparatus and in other routine operations like filtration etc.

5. **Administrative Assistant** (2)

Duties: To maintain complete records of all the samples and correspondence, responsible for all the shipping and stores.

Qualifications:

Essential: Good proficiency in typing and the official language, 2 years experience in record keeping. Neat handwriting and work habits. Unquestioned integrity.

Desirable: Proficiency in local language

6. **Helpers** (2)

For odd jobs specially in sample preparation and shipping.

Essential: Good health and co-operative nature.

16.1.6. Training

Scope of duties for different personnel has been described in the last section. It is very possible that the recruited staff has very limited background . Details of the training program will depend on the background of manpower available, tailored to make up for the deficiencies. The emphasis should be on orientation and training through down-to-earth practical examples rather than longwinding lectures. The more general outline of the orientation program is given below.

Most important to stress is need for objectivity and integrity in work. The staff should be helped to correlate their every day job with feasibility studies for setting up large industries.

With the help of concrete examples, they should be led to understand ultimate financial implications of their analyses being wrong to various extents. Also they should know that the samples analysed there are being analysed in other laboratories for more detailed analyses and are thus being checked.

The staff should be explained and led to discover the following general pitfalls in routine work.

Through specially prepared samples, possibility of errors from accidental classification during sample making. Striking contrasts will be provided by dusty and massive portions of the ores, bothe with respect to silica and alumina. Another interesting sample will be an ore containing distinct high iron pisolitic material.

The difference between well decomposed ores and not too-well decomposed ores during the dissolving step. Especially important are several demonstrations on pitfalls during decomposition of ores with alkali fusion.

Introduction of large errors from improper selection of quantities. For example, during titration contribution of error from one-half or one drop in the context of total titre value. During silica estimation, importance of weighing carefully to the last place of decimal and so on.

Errors introduced from too fastan initial heating during LoI determination and precipitate ignition.

The staff shouid be helped to develop the discipline of simultaneously working on 6 samples through repeated demonstrations of well planned sequences. Importance of using different types of filter papers for different occasions should be known to them.

The staff should be explained the purpose in specifying certain quantities and concentrations of reagents as set in detailed description of the procedures.

The staff should be taught various methods of cleaning different types of apparatus used by them. Needing special attention are pipettes and burettes.
The chemists and assistants should be thoroughly trained in operation and control of the water distillation unit.

The chemists must be repeatedly taught respect for the standard solutions made in bulk. A slight error during one preparation could affect hundreds of analyses. They should be taught to look for any sharp shift in results with a new batch of reagents.

The staff should be aware of general laboratory hazards, such as making dilute sulphuric acid solutions; opening ammonia, nitric acid and hydrochloric acid bottles, storing ammonia and hydrogen peroxide bottles away from heat, very corrosive nature of hydrofluoric acid.

16.2. Central Laboratory - model II

16.2.1. Scope

The laboratory, as mentioned earlier, will conduct most of the technological tests and all the analytical work connected with the feasibility studies. Lateron, with the start of alumina industry in the country its alumina testing section will grow to meet increased demands for minor impurities check analyses. The analytical accuracy expected will be of the highest order, because results of this laboratory will establish the statistical confidence limits of faster field laboratory tests and also used for reference. This Laboratory will also set up standard operating procedures for the field laboratory. Need to complete the analyses quick will remain. On the technological tests, this laboratory will furnish only basic data leaving its interpretation, for details of equipment selection and validity on operation scale-up, to the design engineers. When it is decided to set up a Central Research Institute for bauxite and alumina, this laboratory may become part of the same.

16.2.2. Departments

a) Receipts and sample preparation section
b) Wet analyses laboratory (classical methods.)

c) Spectrochemical section for spectrophotometry, flame photometry (and atomic absorption spectroscopy to be added at a later date)
d) Physical tests to include microscopy, specific gravity and viscosity measurement, surface area measurement.
e) Technological test section
f) Central administrative offices and store.

If dependable arrangement for x-ray diffraction examination of bauxite and muds cannot be made with a nearby competent laboratory, x-ray diffraction section will have to be included.

16.2.3. Departments Description-Scope and Facilities

1. Receipts and sample preparation

Samples for analyses will be received generally in prepared powdered form. Bulk samples in gunny bags and drums will be received for technological tests and many batches as received may have to be preserved for long time unlike that for the field laboratory.

All the facilities mentioned for the field laboratory

2. Wet Analyses

Common for bauxite, liquor and mud analyses. The section will accomodate 10 Chemists - 8 Chemists for routine work and 2 for special analyses.

Facilities

In addition to those mentioned in section 16.1.3.2. for the field laboratory, following will be needed.

Facilities

Item	Number
Fume cupboard, 6' x 3'	2
Pan balance, 200 g capacity, 0.01 g sensitivity	2
Pan balance 1 kg capacity	1
Analytical balance (Mettler) automatic	1
Hot plate, electrical 18" x 18", 100°C, 0.5 Kw	1
" " " 18" x 18", 300°C, 2 Kw	1
Platinum dishes, 100 ml capacity, for alkali fusion of alumina	4
Porcelain dishes, 350 mm capacity	12
Nickel crucibles, 750 ml capacity	12
" " 150 ml "	6
Nickel dish, 10 cm dia. slightly concave	6
Separating funnel, 250 ml, 500 ml capacity	4 each
Typical quick-fit distillation unit for reagent purification etc.	2
TAA determination autoclaves	2
Drive for the above	1
Carbon dioxide measuring apparatus, Fig. 4 Organics in bauxite	1
" " " " Fig. 5 for liquors	1
Watch glasses 30 mm dia.	12
" " 80 mm dia.	12
" " 100 mm dia.	6
" " 125 mm dia.	12
Thermometers, 0 - 110 °C	6
" 0 - 360 °C	6
Wash bottles, 1 litre	12
Kipps apparatus for hydrogen sulphide gas generation	1
Furnaces	
Tube furnace with temperature control 3/4"x18" 800 Watts (For organic determination in bauxite)	1
Furnace with temperature control 12" x 6", 1200°C 4Kw	2

Items	Number
Work tables	6
Titration bench, 7.5' x 2'	1
Wash basins	3
Reagent bottles 1 -litre	50
" " 600 ml	50
" " 250 ml	50
Indicator bottles	24
Conical flasks, 300 ml	24
" " 500 ml	12
Filter flasks 500 ml	12
Water pumps	4
Filter funnels for 12.5 cm dia. filters	12
Buchner funnels, 200 mm dia.	2
Sintered glass cruicibles, grade G-4	12
Dry sieve analysis assembly, 6" dia. sieves, for alumina analyses	1

Spectrochemical Section

Spectrophotometer with filters	1
Flame photometer to be so chosen that it can be converted later for atomic absortpion spectrophotometry at min.cost	1
pH meter (glass electrode type)	1
Gas cylinders, oxygen and acetylene	
Beakers, glass, 100 ml	6
" " 250 ml	6
" " 600 ml	3
" " 1000 ml	1
Standard volumetric flask 25 ml	3
" " " 50 ml	3
" " " 100 ml	6
" " " 250 ml	3
" " " 500 ml	2
" " " 1000 ml	2

Items	Number
Graduated cylinders, glass, 10 ml	2
" " " 25 ml	2
" " " 50ml	1
" " " 100 ml	1
" " " 200 ml	1
" " " 1000 ml	1
Graduated pipette, 5 ml	2
" " 10 ml	2
" " 25 ml	1
" " 50 ml	1
Conical flask, 300 ml	3
Wash bottles, 100 ml	3
" " , 250 ml	3
Reagent bottles, 250 ml capacity	24
" " , 1 l	12
Indicator bottles	12
Filter funnel for 12.5. cm dia.	2
Watch glass 2" size	6
" " 3" size	6

Technological Testing Section

Digestion autoclaves, Fig. 9	3
Furnace for the above, for TAA determination and digestion tests. with temperature control Range 300°C, 5Kw	3
Drive for the autoclaves 0.3 HP	3
Reversion tests and settling tests assembly Fig. 10	1
Filter leaf test assembly Fig. 11	1
Precipitation studies assembly Fig. 12	1
Wet screening assembly Fig. 13 - 0.75 Kw	1
Pipette for Andreasen test, Fig. 14	1
Sub-sieve analyser, Fischer air-permeability type	1
Microscope with attachment for photography	1
Sample preparation equipment for making mineral sections	1
Pycnometer, 25 ml	2
Brookfield viscosity meter	1

Items	Number
Buckets, plastic, 10 l capacity	4
Beakers, stainless steel, 3 l capacity	2
" " " 1 l "	2
Beakers, plastic, 2 l	4
" " 1 l	4
Vacuum pump	1
Buckner funnel, 300 mm dia.	1
Filter flask 1 l	1
" " 2 l	1
Drying oven with natural draft adjustable to 140°C, 1.2 Kw	1
Pan balance, 1 Kg capacity	1
Work tables	6

Stands and clamps.

9. **Various offices, store, record room, conference room etc.**

The facilities will consist of [illegible] tables, [illegible] typist table, [illegible] cupboards, [illegible] filing cabinets, [illegible] multidoor shelves, [illegible] blackboard

For details, see section [illegible].

Overall space requirement will be [illegible]

10.3.6. **[illegible]**

1. Bulk storage of [illegible] bulk samples [illegible] reducing them to [illegible] etc., if necessary, similar to the [illegible] of the [illegible] store [illegible] (... sq ft)

2. **Sample preparation room**

To house [illegible] grinding mills, sample riffles, pan balance, [illegible], [illegible] work tables, each [illegible] [illegible] sq ft

3. **Wet analytical laboratory hall**

(will be conveniently divided to segregate facilities for special analyses)
To house [illegible] work tables, [illegible] titration [illegible] [illegible] small titration bench, [illegible] fume cupboards and [illegible] glass ware cleaning [illegible] [illegible] sq ft

4. **[illegible] and furnace room** (adjacent to the hall)

To house [illegible] furnaces ([illegible]) [illegible] precipitate ignition etc. [illegible] drying ovens and [illegible] work table [illegible] sq ft

5. **Balance room** (adjacent to the hall)
To house [illegible] analytical balances and [illegible] work table [illegible] sq ft

6. **[illegible] room** (adjacent to the hall)
To house [illegible] table and [illegible] chairs [illegible] sq ft

7. **[illegible]** [illegible] sq ft

8. **[illegible]** [illegible] sq ft

9. Instrumental analyses room

To house 1 work table for spectrophotometer and pH meter, 1 table for microscopy work, 1 work table for mineral (section) sample preparation, 1 work table for Fischer sub-sieve analyser, one general work-table and one wash basin. 180 sq ft

10. Technological tests hall

To house 3 digestion autoclaves assemblies, T.A.A. determination assembly, settling test assembly, Wet screen assembly, vacuum filtration bench, drying oven, 4 general service work tables 700 sq ft

11. Annexe (to the technological tests hall)

To house 2 work tables and provide room for any special equipment fabricated but not frequently used 180 sq ft

12. Chief Chemist' room

To house work table with 4 chairs, one side table, one cupboard, one shelf 180 sq ft

13. Assistant Chief Chemist' room

To house 1 table with 4 chairs, one cupboard, one shelf 180 sq ft

14. Records room and general office (will be conveniently split)

To house 3 work tables, 1 typist's table, 2 filing cabinets, 1 library cupboard, 1 sample storage cupboard, 2 miscellaneous cupboard [illegible] sq ft

15. Store

General: To house 4 multi-tier shelves, 1 cupboard [illegible] sq ft

Hazardous bulk chemicals (Hydrochloric, sulphuric, nitric acids, ammonia etc.) [illegible] sq ft

16. Conference room

To accommodate [illegible] chairs, 1 blackboard [illegible] sq ft

17. <u>Toilets</u>	
Two of 60 sq ft each, i.e. 120 sq ft.	120 sq ft
18. <u>Waiting room</u>	
To house 1 table with 4 chairs	120 sq ft
19. <u>Total covered area</u>	<u>4740 sq ft</u>
	say <u>5000 sq ft</u>

[illegible] <u>Staff</u>

Emphasis in this laboratory will be on quality and precision. Accordingly, higher academic preparation will be required.

1. <u>Chief Chemist</u> (1)

<u>Duties</u>: Overall incharge of the laboratory. He will be responsible for setting and maintaining highest standards of precision in all the work. He will have frequent contacts with the project engineers, consultants, continuously trying to improve his analytical and investigation techniques. He will personally analyse difficult samples.

<u>Qualifications</u>:

<u>Essential</u>: Master's degree in Inorganic Chemistry with 5 years proven successful experience in responsible position of a good industrial mineral testing laboratory. Thorough background of analytical chemistry principles and active experience of spectrophotometry. Familiarity with statistical evaluation techniques and good appreciation of importance of these techniques in day-to-day work.

Energetic and congenial personality, willingness to get equainted with new fields.

<u>Desirable</u>: Experience of microscopy techniques, technological test work in the industry. Knowledge of English language, where official language is different.

2. Assistant Chief Chemist (1)

Duties: In absence of Chief Chemist, he shoulders all the supervision responsibilities independently. He should have potential to step into Chief Chemist's shoes.

As a part of routine duties, he keeps check on all the day to day work. Personally analyses check samples and routine samples during times of heavy work load.

Qualifications

Essential: Master's degree in Inorganic Chemistry with 3 years experience of good analytical work. Active congenial personality. Willingness to undergo training for specialised fields.

Desirable: Specialised knowledge in spectrophotometry, atomic absorption spectrophotometry or microscopy, or technological test work. Knowledge of English, where official language is different.

3. Chemists and Analysts (10)

(2 Chemists will be working on special analyses)

2 Chemists should have good graduate degree in Inorganic Chemistry with interest in analytical work.

Essential: 2 - 3 years proven experience in analytical work.

Remaining 8 Chemists / Analysts should have higher secondary school certificate in science with interest in analytical work.

Desirable will be mechanical aptitude useful for technological tests.

4. Assistants (4)

To make bulk reagents, operate water distillation unit, undertake sample preparation, help in technological test work, cleaning and maintenance of apparatus and miscellaneous laboratory operations.

Essential: High school certificate, clean habits, honest, and co-operative nature.

5. **Administrative Assistants** (3)

Stores purchases, despatches, accounts	One
Secretarial work	One
Records	One

6. **Helpers** (2)

For odd jobs, specially in sample preparation, stores.

Essential: good health and co-operative nature.

16.2.6. Training

Scope of duties for different personnel has been described in the last section. High technical standards are expected at all stages, so senior staff with sound background and maturity will be chosen. Training program will be tailored to make up their particular deficiency. One month's orientation for the Chief Chemist and Assistant Chief Chemist in an alumina plant and in a technological testing laboratory will be very useful. Arrangement should also be made for 1 month's orientation coarse in statistical techniques of experiement planning for the Chief Chemist and Assistant Chief Chemist. For the remaining staff, one month on-job orientation will be adequate.

16.3. Central Research Institute - Model III

16.3.1. Scope

The Institute will be research oriented to evolve new analytical methods, deve- processes and technology needed for the established alumina industry, lop special products to increase profitability, and prepare for changing requirements and more rigorous specification standards of the end-product users and economic exploration of complex lower grade minerals. It will have a continuous run pilot plant as indicated in section 15, probably attached to an operating plant, in addition to the various isolated study models it will develop in its own premises for various discontinuous steps. To meet its requirement of sound chemical and physical analyses central ser- vice, it will be advantageous to have the Central Laboratory (model II described in section 16.2.) amalgamated with it so as to make use of specialised talent developed there.

Although fundamental work is carried out where necessary, much of the work will be applied and related to the development or improvement of processes. In more functional terms, the emphasis will be on "How-to-reach-the-goal" attitude rather than "why-it-happened-attitude" concerned with seeking knowledge for its own sake. Profitability would be the motivating factor in choosing programs. Built-in provision should be made to resist cowardly temptation to go for safe, high yielding areas of short term research (small profit quick returns) which could be better done in operating units in the plant and are often responsive to the inevitable and frequent shifting of goals.

In planning of the facilities, local conditions will play a strong role. For example, in a country rich with good quality bauxite, immediate interest in utilisation of low grade ores will be mostly academic. Consequently the establishment will have a very small common mineralogical service department. Emphasis will be instead on improvement of efficiencies, recoveries and development of special products. And this would in turn reflect strongly on size of many auxiliary services.

Accordingly, the following description is of indicative nature for both the organisation and facilities required. Certainly extensive detailing will be required to fit individual circumstances.

The nature of research projects of interest for bauxite processing has been explained in sections 3,4 and 5.

10.3.3. Departments

1. Chemical Engineering

Engaged in development of predictive process models incorporating the dynamic characteristics of various processes. This recently developed discipline has proved a very strong tool in improving plant economics by more accurate process control (by computers and otherwise). It develops scale-up factors enabling realistic plant designs from bench-scale experimental data, yielding great economies. The department setspace for intelligent planning of equipment and process development by the subsequent process development group.

The department designs its own simulation models, using basic instruments like timers, controllers for temperature, flow, viscosity and density. As mentioned earlier, most of the equipment used is of bench-size, Two calculating machines will be must.

2. Process Development

Control of pilot plant operations. Study of new deposits and improvement of the existing processes, equipment development. All the routine technological tests, described for Central Laboratory model II, will be conducted by the anaytical laboratory service group under the programs laid by this department.

If the Institute has to produce its own steam and gas, these facilities will come under this group's jurisdiction.

3. Analytical Laboratory

The laboratory will have 2 distinct wings;

a) General Service

b) New Method Development

For General Service wing, all the facilities indicated for the Central Laboratory, model II, excluding storage space for bulk bauxite samples and its crushing, microscopy and flame spectrophotometry which will be transferred to its "New Methods Development" wing. Naturally all the office work functions will pass to the new Central Office.

If Central Laboratory is not to be amalgamated with it, required facilities will be reduced by 50 %, because in that case this laboratory will not do any outside work.

For New Methods Development wing, will be included atomic absorption spectrography, and research work on various on-stream analysing systems. On-stream analysers promise significant operational saving in the plants, and require very skilled work for standardisation. The research involves use of physical instruments like conductivity, density meters, turbidity-meters, potentiometers and tracer techniques. Of interest here in the Bayer process is stream analysis for soda, and alumina (Bauxite is treated by x-ray methods).

4. X-ray Laboratory

To include both diffraction and emission (spectrography) inclusive of recording units. Auxiliary facilities include sample preparation by fusion with appropriate mixture or by pressing the powders into pellets as mentioned in section 12. Research programs will include standardisation for bauxite, mud and alumina analyses.

5. Mineralogical Laboratory

To include petrological microscope for magnification upto 1000 times with all fittings, including photographic equipment, fittings for direct study of high temperature transformations, mineral-section preparation equipment consisting of cutters, grinders, polishers, and mounting press, thermal differential analyser, two temperature controlled heat treatment furnaces for temperature range upto 1000 °C and upto 1800 °C, dark room.

Provision for electron microscope, only when competent staff is available.

6. Service Shop

Its facilities will be determined by the nature of pilot plant projects. Undertaking of pilot plant projects must be accompanied by creating facilities for welding, lathe, smithy, drilling machine and light fabrication work. Continuous running plant will need a regular maintenance crew. To be reviewed are machine shop and foundry facilities in the neighbourhood. For very routine services, when bauxite pilot plant is located in the plant and most of the equipment fabrication work can be got done outside, needed for mechanical side are 2 lathes, 1 saw, 1 drilling machine and 2 fitters' benches with usual accessories.

If bulk electricity is purchased, substation control will be under this department.

7. Library and Technical Liason

Responsible also for the records.

8. Administrative Offices, including Purchase Stores etc.

16.3.3. Area Requirement

1. Chemical Engineering Department

2 rooms of 200 sq ft each
1 room of 250 sq ft
1 office of 150 " "

Total 800 sq ft

2. Process Development Department

Close circuite continuous running pilot plant as described in section 15.	10,000 sq ft
Laboratories, three, for special research projects	600 sq ft total
One room to store "idle" miscellaneous items specially fabricated for certain studies but not often used	150 sq ft
Three office rooms	400 sq ft
Common mineralogical service and pilot plant building to contain facilities for crushing, grinding, mixing and storage of ores and which can accomodate large items of pilot plant equipment	

If the close circuit bauxite digestion pilot plant is installed near an operating plant, as per section 15, 3,000 sq ft should be provided at the Institute, as ultimately expected in course of time. Otherwise 6,000 sq ft if very extensive mineral beneficiation work is to be undertaken[x], and 3000 sq ft if it is decided to drop the continuous running close-circuit bauxite digestion plant and particularly intensive bauxite beneficiation program is not envisaged in the near future.

Steam producing plant, if required. 500 sq ft

<u>Total</u> without continuous running pilot plant and boiler, 4150 sq ft incl. 3000 sq ft initial area for common mineralogical service and sundry pilot plant investigations.

x). Provided at U.K. Ministry of Technology's Warren Spring Laboratory, Stevenage, Herts.

3. Analytical Laboratory

General Service

Equivalent to area occupied by various technical services listed under items 16.2.4 of Central Laboratory, model II, excluding office space for stores, conference room, waiting room, and reducing area for records room to 180 sq ft	4,000 sq ft

(If no outside work is to be accepted, the area will be reduced by one-half)

New methods Development Wing

To include one room for atomic absorption spectrophotometer and other instruments mentioned under item 16.3.2.3. and 2 work tables	400 sq ft
One room for usual wet analyses techniques like fusions, fuming etc.	200 sq ft
Total	600 sq ft

4. X-ray Laboratory

One main room for the X-ray unit	300 sq ft
One side room for general services and sample preparation	200 sq ft
One office for the section incharge	150 sq ft
Total	650 sq ft

5. Mineralogical Laboratory

One room for microscope, thermal differential analyser, heat treatment furnaces	150 sq ft
One room for miscellaneous sample preparation work,	150 sq ft
One dark room	60 sq ft
Electron microscope section	300 sq ft
Total	660 sq ft

6. Service Shop

For very routine services, when continuous bauxite processing pilot plant is located in the main plant and most of the equipment fabrication work can be economically got done outside, needed is space for 2 lathes, 1 drilling machine, 1 saw, 2 fitter's benches, 2 work tables for the electricians and 2 for the instrument man.

One room for mechanical equipment	300 sq ft
One room for electricians and instrument mechanics	300 sq ft
One room for the shop chief	150 sq ft
Total	750 sq ft

7. Library, Records and Technical Liaison

Library	500 sq ft
Office	150 sq ft
Liaison officer	200 sq ft
Conference room	300 sq ft
Total	1,150 sq ft

8. Administrative Offices, including Purchase, Stores etc.

It is assumed that the Research Institute is not responsible for housing.

Purchase

General office	200 sq ft
Purchase officer	150 sq ft
Total	350 sq ft

Stores

Hazardous chemicals	50 sq ft
General stores (in the absence of continuous running pilot plant)	450 sq ft
Stores officer	150 sq ft
Total	650 sq ft

General office work and typists' pool	300 sq ft
General administrative officer	120 sq ft
Director	
Office	200 sq ft
Secretarys' office	120 sq ft
Waiting room	120 sq ft
Total	440 sq ft
Reception	120 sq ft
Toilets (2 places)	300 sq ft
Total	14.048 sq ft
say	15,000 sq ft

10.6.4. Staff

A research organisation grows slowly. Accordingly considered here is only initial nucleus staff to provide competent base. It would be expanded rationally, to match the demands.

10.6.4.1. Director

Critical to the success of the Institute in meeting the outlined objectives is selection of the Director.

A research organisation is essentially a biological system, unpredictable.[20] Brainwash it, it becomes sullen; spoil it, it will cease to heed the basic industrial needs; cherish it, it will lay golden eggs.

The Director must have a right balance of academic background, industrial experience and conviction to insist on high standards. He must have clear economic awareness of industrial operations to be able to select priorities and define objectives. He should have personality to inculcate his own economic awareness in his assistants and lead them to have personal commitments in the projects. All this may sound big talk. But it was felt necessary because one sees so many instances of expensive research institutes having many individuals of sound academic standing and still failing to make any impact.

[illegible] Chemical Engineering Department

The accent will be on quality rather than quantity.

Head (1)

Master's degree in chemical engineering with [illegible] years' established experience in modern process simulation techniques. He should be able to lead [illegible] - [illegible] research groups.

[illegible] Engineers (3)

One preferably with master's degree in chemical engineering

Two graduates in chemical engineering with excellent theoretical background of kinetics and unit operations.

Assistants (3)

Science graduates with good academic record.

[illegible] (1)

[illegible] [illegible]

Head

Master's degree in chemical engineering with established knowledge of statistical experimental design and data evaluation. [illegible] years' plant experience, preferably process design. Good and energetic personality to control large operating work force of the various pilot plants and also supervision of their installation.

Assistant Engineers (3)

First assistant (1) bachelor's degree in chemical engineering with [illegible] years plant experience; good personality to control the day to day work, especially pilot plant operations.

Two (2) bachelor's degree in chemical engineering with [illegible] years plant experience, preferably in technical control. Aptitude for calculation and project work needed for feasibility studies.

Assistants (4)

Preferably science graduates with mechanical aptitude and interest in investigation work. Good health and congenial personality. Will take up front-line duties of pilot plant operations as the organization develops.

Helpers (3)

For general help; good health and mechanical aptitude. Co-operative.

For Three-Shift Continuous Run Pilot Plant (as per section 10)

Skilled maintenance facilities provided by the plant.

Shift Engineers (3)

Chemical engineering graduate with 3 years plant experience of technical control - preferably of chemical plant. Proven integrity.

General Foreman (1)

5 years chemical plant experience as a foreman or 3 years experience as assistant foreman.

Shift Operators (3)

To attend to the various operation jobs.
Control room experience of a chemical plant. Active habits. Educated.

Labourers (10)

Manual duties including straight forward routine maintenance jobs. Good mechanical aptitude - active habits.

10.4.4. Analytical Laboratory

All the staff indicated for analytical work in Annex II Central Laboratory Organization plus one administrative assistant. (If no outside work has to be done, the number of chemists and assistants will be reduced by half).

[illegible]

[illegible] [illegible]

[illegible] [illegible]

[illegible]

[illegible]

[illegible] [illegible]

[illegible]

[illegible]

[illegible]

[illegible] [illegible]

[illegible] [illegible]

[illegible]

[illegible]

[illegible]

[illegible]

[illegible]

[illegible] [illegible]

[illegible]

[illegible]

[illegible]

[illegible]

[illegible]

[illegible]

[illegible]

[illegible]

[illegible]

[illegible]

[illegible] [illegible]

[illegible]

[illegible]

Engineering Division and Process Development Division have had no experience of alumina plant, 3 months in-plant orientation will be extremely useful.

10. Acknowledgement

The author is thankful to Swiss Aluminium Ltd. for permission to undertake the work, to the Technical Director, Dr. J.P. Bovay, Società Alluminio Veneto per Azioni, Divisione ALLUMINIO (SAVA Porto Marghera) for allowing time and facilities, and Dr. B. Fabbro, Chief Chemist (SAVA) for helpful discussions.

Rajindra Manocha

RAJINDRA MANOCHA

10. References

1. D.D. Mackay, Symposium on Mining and Processing of Bauxite, Mining Geological and Metallurgical Institute of India, 1967, Oxford Book Co., Calcutta-16, India. p.33.

2. H.K. Roy Chowdhury, Symposium proceedings as above. p.24,29

3. Lipperwerke Vereint an Werke 15(2) 7-8 July, 1968. Aluminium Abstracts 16.4.1967, vol 7 item 8.

4. H.Caldwell, A. Mayhurst, R.Webster, Trans. British Ceramic Soc. March 1967, 107-119.

5. Studies in Economics of Industry, Pre-investment Data for the Aluminium Industry, United Nations, New York, 1966.p.7.

6. R.S. Breur, L.R. Barsotti, A.C.Kelly, Extractive Metallurgy of Aluminium, Vol.I, Interscience Publishers, New York 1963, p.133.

7. A.N.Adamson, E.J.Bloore, A.R.Carr, ibid p.26-27.

8. H.R.Rose, ibid. p.3-80.

9. H.P.Bosndrett, ibid, 95-111.

10. T.G.Pearson. The Chemical Background of the Aluminium Industry. Monograph, Royal Institute of Chemistry, London, U.K.1955.

11. Indian Standards Institution, New Delhi, India.1962. IS: 2000-1962.

12. Analyst, Nov. 1967, p. 680-4, Aluminium Abstracts 1 (1968) 116.

13. Hope, Raymond, Moran, Floets, Ind. and Engg. Chem. Anal Ed 8 (1936) 48.

14. R.R.Phillips, A.E.McLaughlin, Regional Chemical Society Meeting, New Orleans, La, U.S.A., Dec. 10, 1953.

15. J.V.Smith et al., "X-ray Powder Data File", Special Technical Publication No. 48-H, ASTM, Philadelphia, Pa., U.S.A. April 1959.

16. G. de Weisse, Mineralium Deposit 2 (1967) 349-56
Bull.Assoc.Suisse Geologues Ing. Petrole, 15 (1949)(49) 19-28.

17. V.A.Altekar, G.S. Mathad, J.Sci. Ind.Res., 20 D(1961) (11) 405.

18. Statistics in Chemical Engineering L.B.Anderson, Chemical Engineering, Oct 29,1962-Sept.2, 1963.

V.Pavelic, U.Saxena,Chem. Engg.Oct 6,1969, 175-180

19. ISO/TC 47/ T8,Zurich meeting, June 17-20, 1969. Committee ISO/TC formed on October 2, 1969.

20. A.Anderson, G.E.Sparkman, Chem.Engg.,Nov.2,1959,p.78.

21. K.B.Kellogg, C.H.Holley, Extractive Metallurgy of Aluminium, Vol.I,Interscience Publishers, New York 1963, p.194-197.

22. K.Norrish, T.R.Sweatman, C.S.I.R.O. Australia,Divisional Report 14/62, July 1962.

23. World Mining 16(4)(1963) p.43.

24. A.N.Van der Veen. Geologie en Mijnbouw, 47(1968)(6) 469-478.

25. Bullettin Philips Pye Unicam on SP 90 Spectrophotometer.

26. Perry's Chemical Engineers' Handbook.IV edition,1963. McGraw-Hill Book Co.Inc. New York, U.S.A.,19-59.

27. Pilot Plants,Models and Scale-up Methods in Chemical Engineering.
R.E.Johnstone, M.W.Thring.1957.
McGraw-Hill Book Co. Inc.New York, U.S.A.p.127,129.

28. D.R. Sperry, Ind.Eng.Chem., 36 (1944)323.

29. Davies, Metals Materials, August 1967,259-65.

30. W.P.Tamlag, E.B.Fitch, Industrial and Engineering Chemistry, 47 (1)(1955) p.39.

Table Nr. 1

IMPURITIES IN BAUXITE

	Gold Coast	Brazil	Ireland	France	Surinam	British Guiana	Jugoslavia	India	Arkansas bauxite and bauxitic clay	Australia
BaO	Tr	N.I.	-	-	-	-	-	-	0.019	-
BeO	-	N.I.	-	-	-	-	-	-	0.0006	-
Bi_2O_3	N.I.	N.I.	N.I.	N.I.	N.I.	N.I.	-	-	-	-
B_2O_3	-	-	-	-	-	-	-	-	0.010	-
CaO	0.35	0.42	0.30	0.20	0.36	0.15	0.3	-	0.17	0.27
CoO	N.I.	N.I.	0.003	0.004	N.I.	N.I.	-	-	0.0001	-
Cr_2O_3	0.023	0.003	0.0008	0.037	0.003	0.03	0.12	0.09	0.016	0.08
CuO	0.001	0.005	0.004	0.004	0.004	0.001	-	-	0.002	-
Ga_2O_3	0.014	-	-	-	-	0.01	-	-	0.012	-
K_2O	<0.012	<0.012	<0.012	<0.012	<0.012	<0.012	-	-	-	-
La_2O_3	-	-	-	-	-	-	-	-	0.012	-
Li_2O	<0.02	<0.02	<0.02	<0.02	<0.02	N.I.	-	-	-	-
MgO	0.22	0.20	0.43	0.15	0.40	0.08	with CaO	-	0.061	0.25
MnO	0.040	0.030	0.17	0.057	0.015	0.0085	0.2	-	0.15	0.11
MoO_3	0.03	0.03	0.072	0.060	0.003	0.003	0.010	0.007	0.003	Tr
Na_2O	<0.013	0.12	<0.013	0.013	0.013	0.04	-	-	-	nil
Nb_2O_5	-	-	-	-	-	-	-	-	0.070	-
NiO	N.I.	N.I.	N.I.	N.I.	N.I.	N.I.	-	-	0.0008	-
PbO	0.001	<0.001	<0.001	0.002	0.002	0.002	N.I.	-	0.0008	-
P_2O_5	0.034	0.032	0.025	0.050	0.025	0.023	0.21	0.05	-	0.40
Sc_2O_3	-	-	-	-	-	-	-	-	0.001	-
SnO_2	-	-	Tr	Tr	Tr	0.013	-	-	-	-
SrO	N.I.	N.I.	N.I.	N.I.	N.I.	N.I.	-	-	0.022	-
V_2O_5	0.030	0.005	0.045	0.011	<0.002	<0.002	0.19	0.13	0.016	0.04
Y_2O_3	-	-	-	-	-	-	-	-	0.019	-
ZnO	-	0.0075	-	-	-	-	-	-	-	-
ZrO_2	-	0.22	-	-	-	0.04	-	-	0.18	-
Total	0.722	0.9625	1.638	0.635	0.814	0.3545	1.030	0.277	0.7653	1.15

Notes N.I. = no indication

Table Nr. 2

COMPOSITION OF ALUMINATE LIQUOR 10)
[illegible]

Na_2O	100	Ga_2O_3	0.22
Al_2O_3	32.80	MgO	0.17
As_2O_3	0.26	MnO_2	0.06
CaO	0.12	P_2O_5	0.90
Cl^-	1.00	SO_3	0.07
CO_2	3.74	SiO_2	0.60
F^-	0.03	TiO_2	Trace
Fe_2O_3	0.05	V_2O_5	0.45

Table Nr. 8

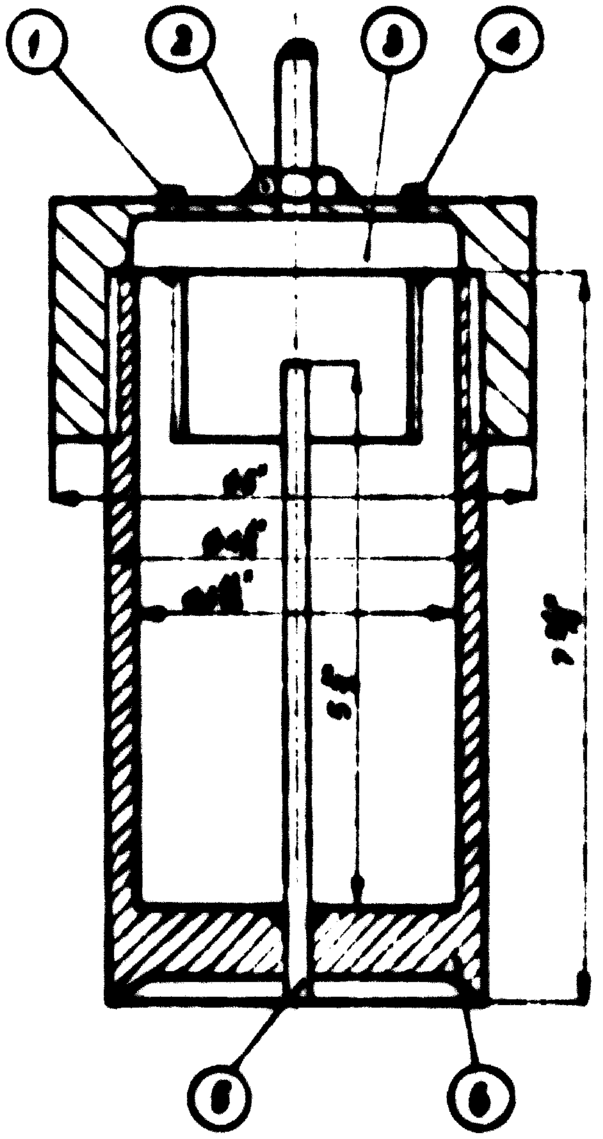
ATOMIC ABSORPTION SPECTROPHOTOMETRY

CONDITIONS FOR DETERMINATION OF VARIOUS ELEMENTS 25)

Elements	Wavelength nm	Flame type	Sensitivity p.p.m.	Elements	Wavelength nm	Flame type	Sensitivity p.p.m.
As	193.7	Air/Acetylene	2.0	Mo	313.3	N_2O/Acetylene	1.0
Be	234.9	N_2O/Acetylene	0.07	Ni	232.0	Air/Acetylene	0.8
Ca	422.7	N_2O/Acetylene	0.08	K	766.5	Air/Propane	0.05
Cr	357.9	Air/Acetylene	0.3	Si	251.6	N_2O/Acetylene	10.0
Co	240.7	Air/Propane	0.2	Na	589.0	Air/Propane	0.03
Ga	287.4	Air/Acetylene	3	Ti	364.3	N_2O/Acetylene	5.0
Fe	248.3	Air/Acetylene	0.5	U	355.1	N_2O/Acetylene	5.3
Mg	285.2	Air/Acetylene	0.012	V	318.4	N_2O/Acetylene	2.5
Hg	253.7	Air/Propane	10.1	Zn	213.9	Air/Propane	0.04
Mn	279.5	Air/Propane	0.05				

FIG. No. 1

AUTOCLAVE FOR T.A.A. ESTIMATION

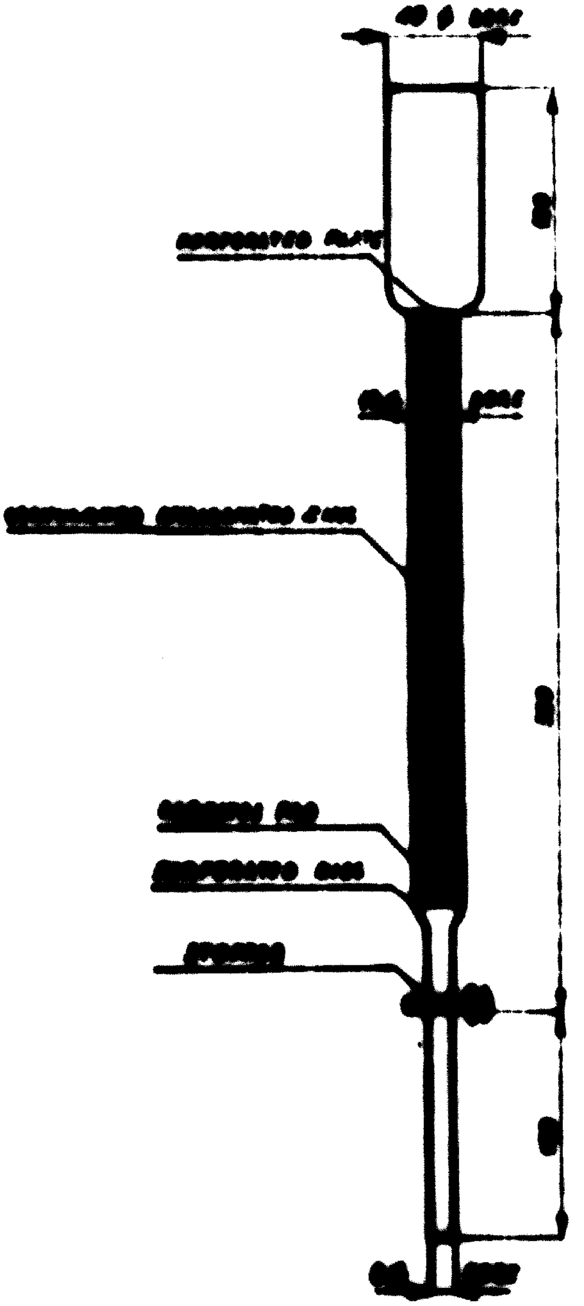


[illegible]

1	[illegible]
2	[illegible]
3	[illegible]
4	[illegible]
5	[illegible]
6	[illegible]

FIG. Nr. 2

JONES REDUCTOR



[illegible]

FIG. Nr. 3

LIQUID AMALGAM METHOD FOR TITANIUM

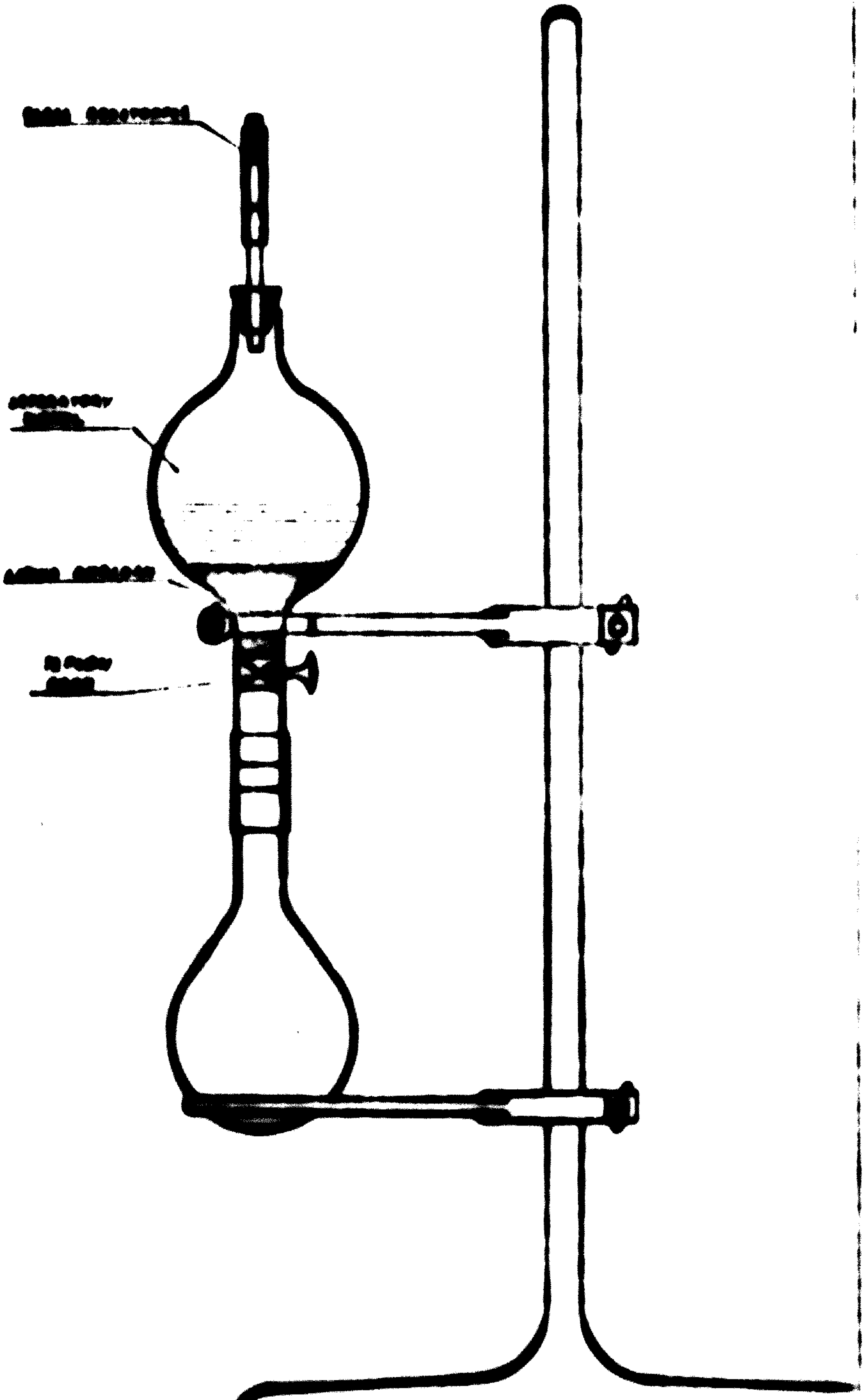
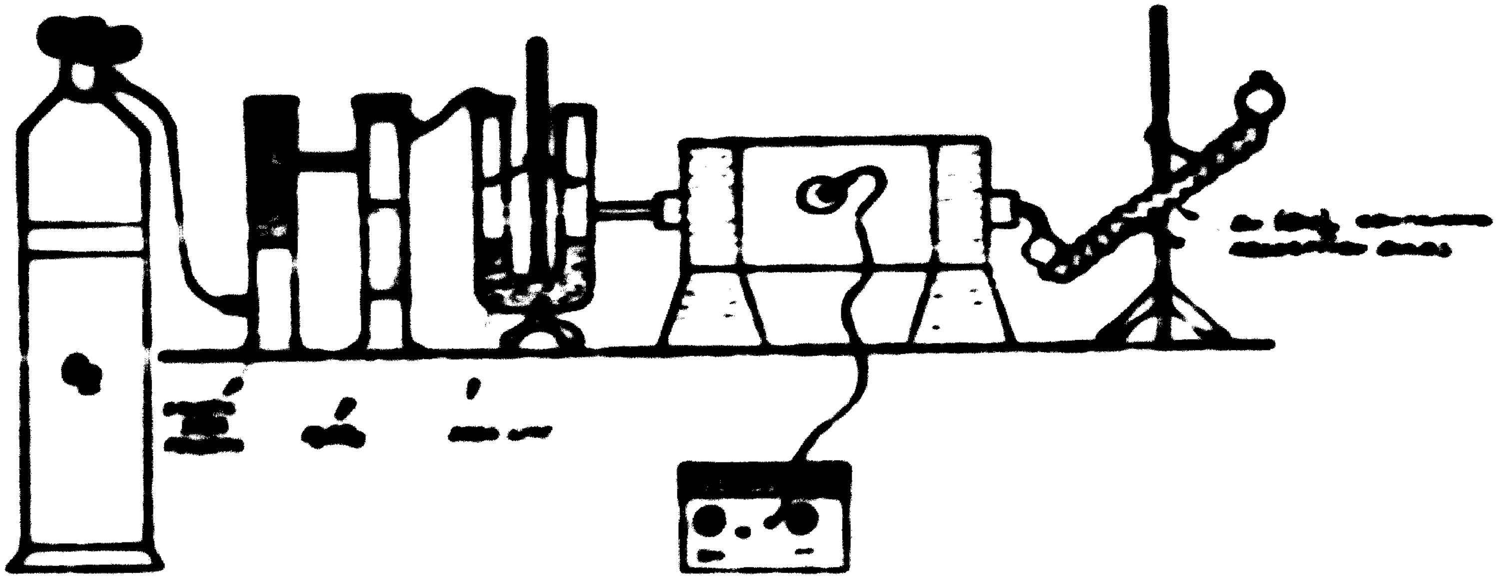


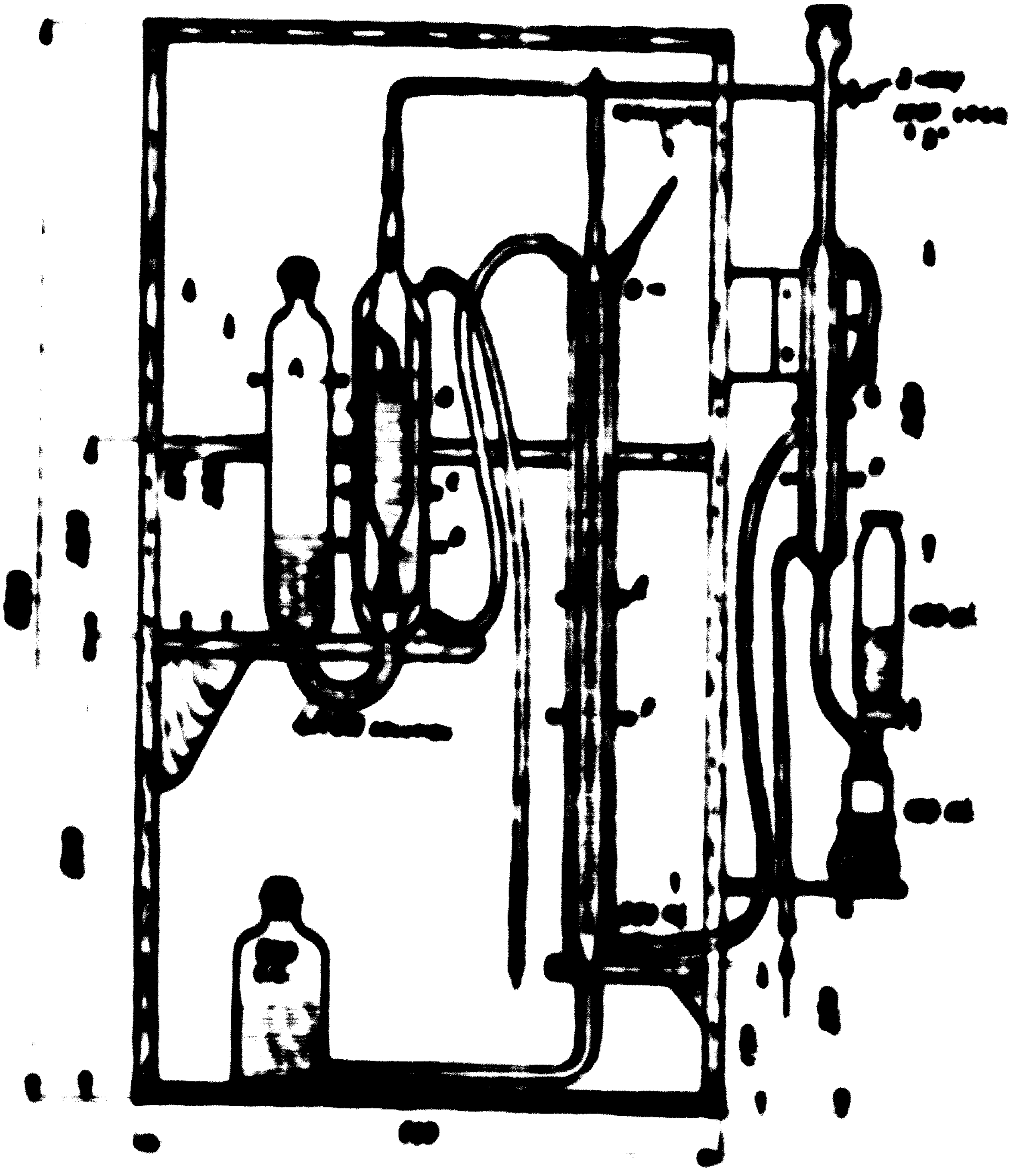
FIG. No. 4

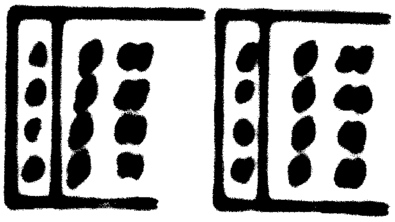
ORGANIC CARBON DETERMINATION BY
COMBUSTION METHOD



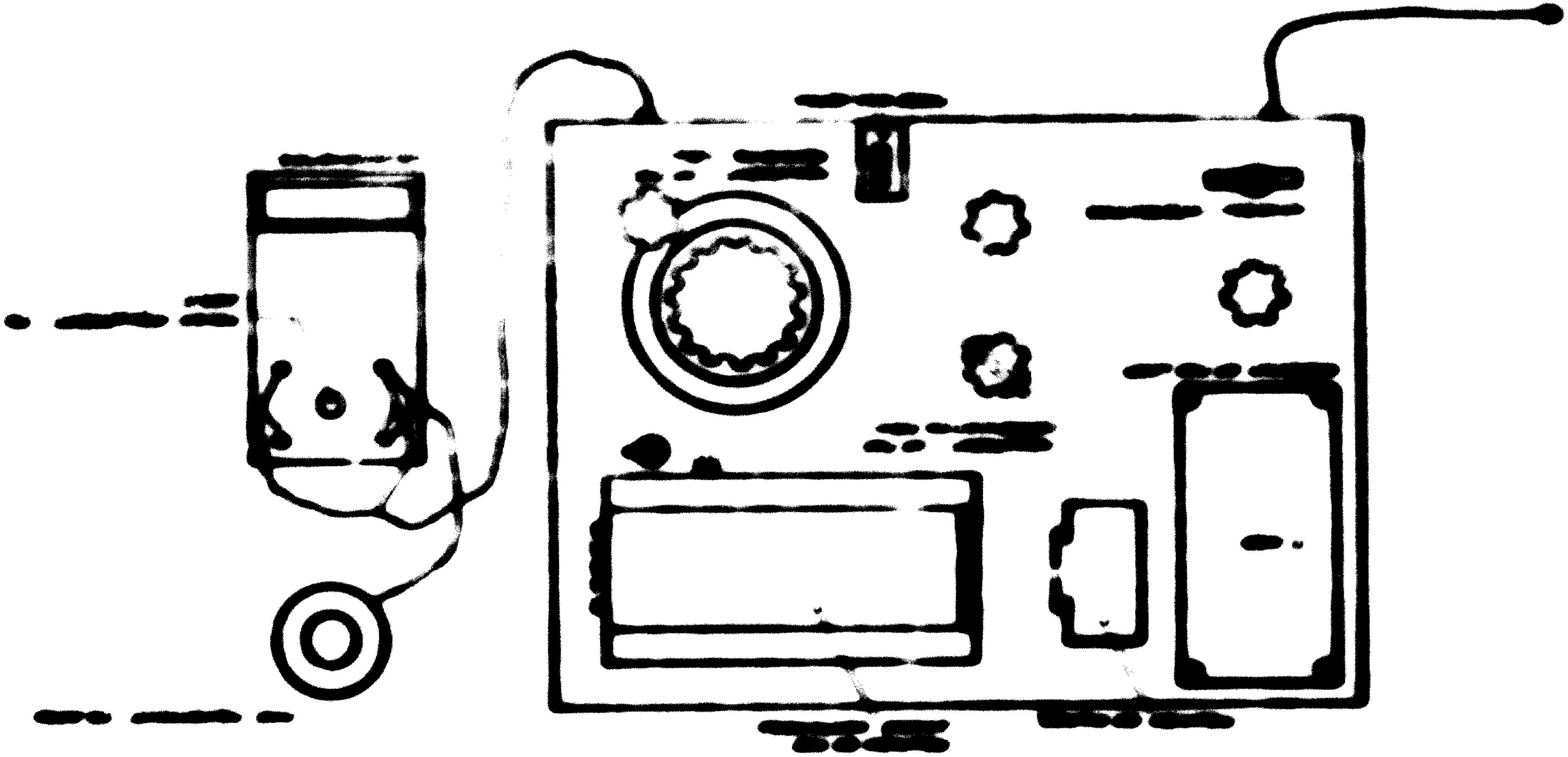
APPARATUS FOR MEASURING [illegible]

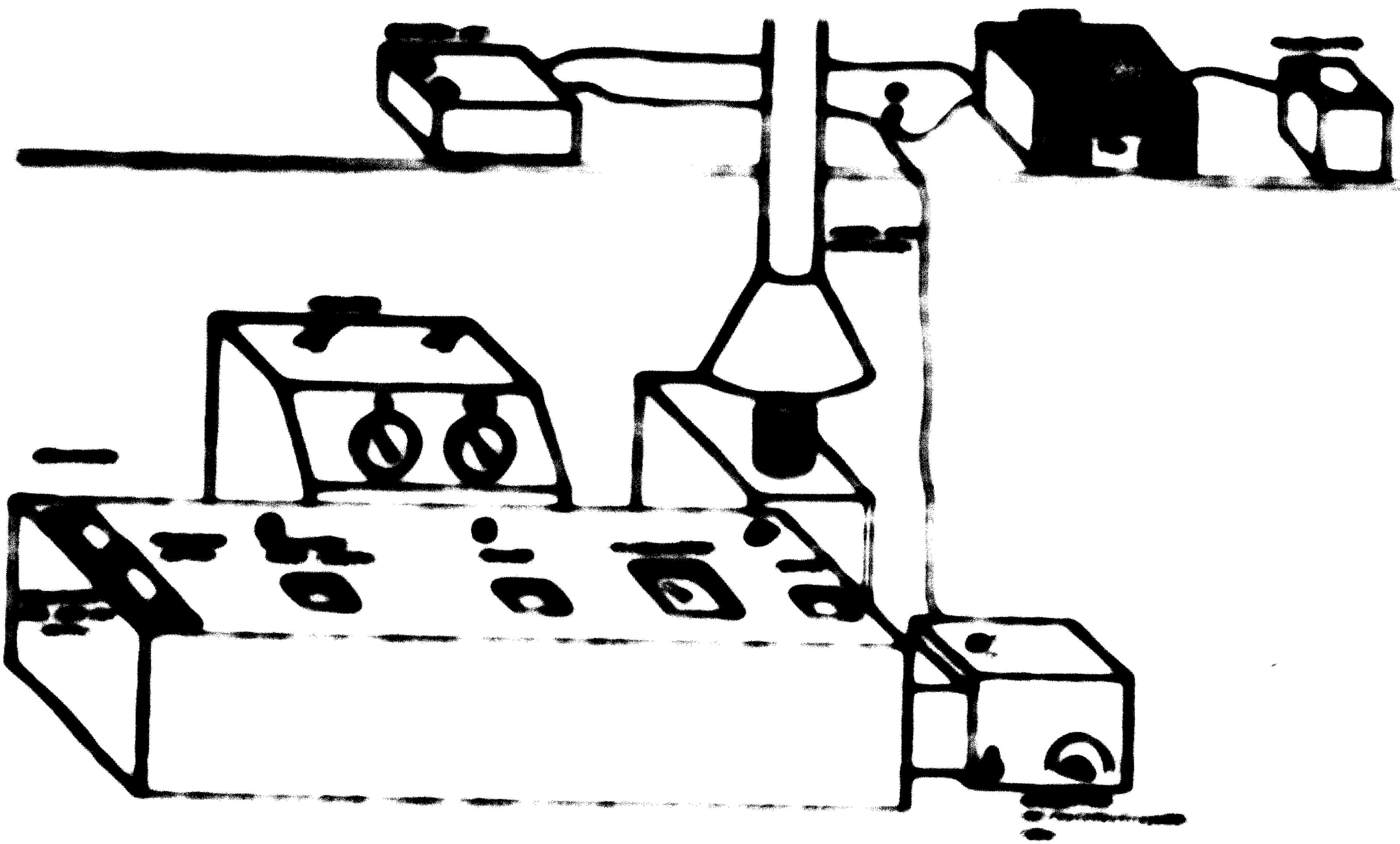
FIG. NO [illegible]





[illegible]





[illegible]

[illegible]

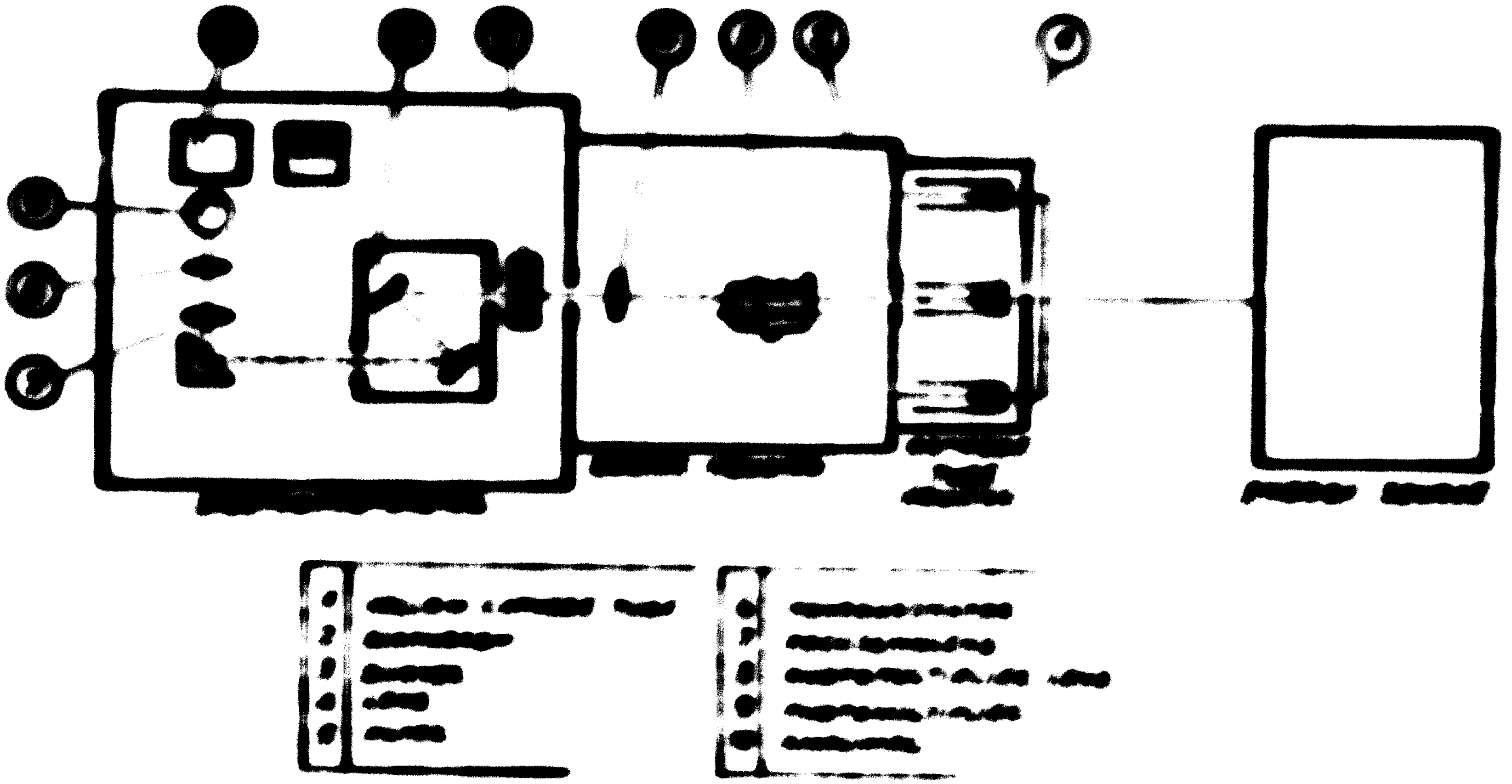
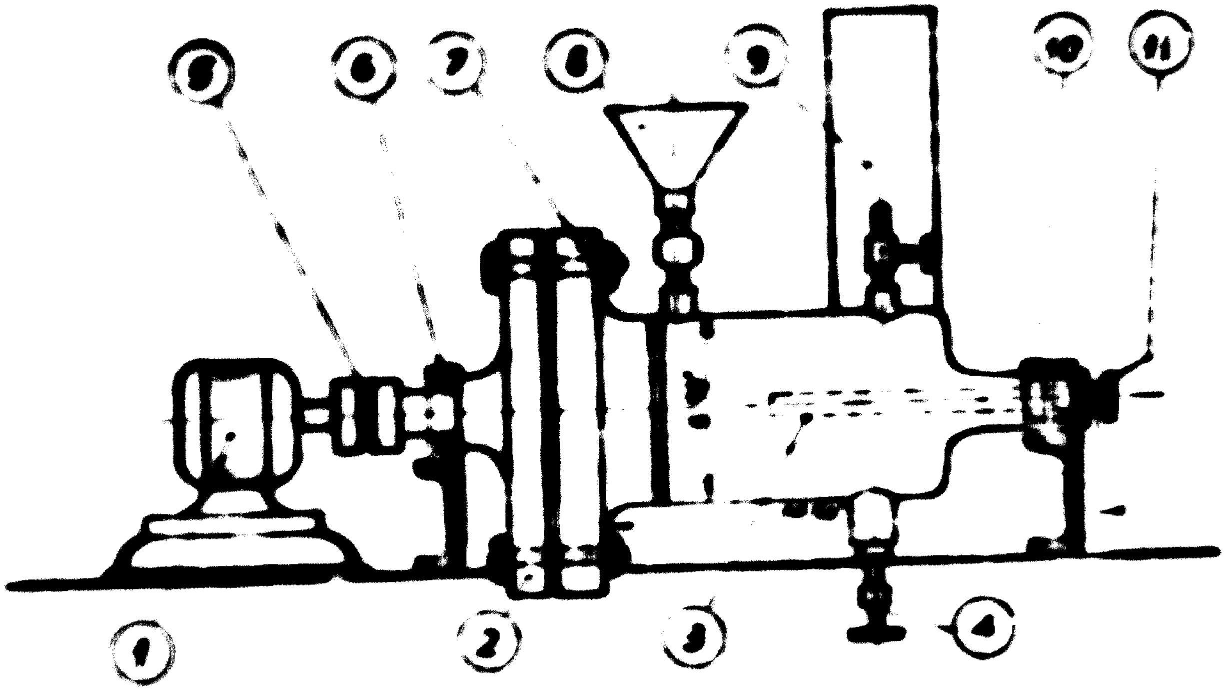


FIG. NR 9

[illegible] TEST AUTOCLAVE

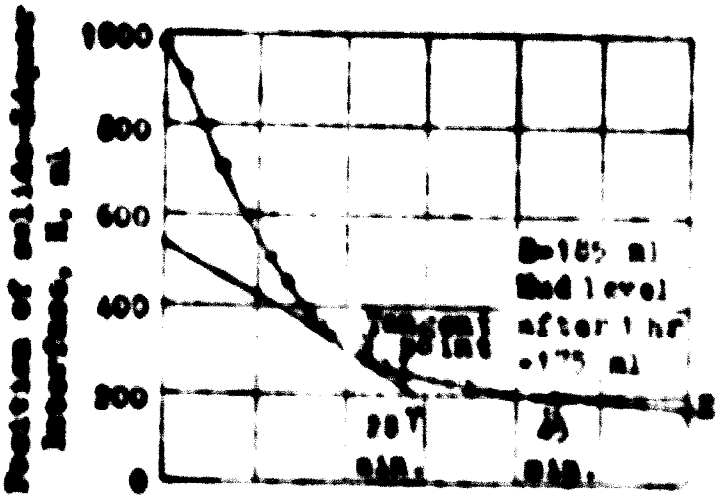


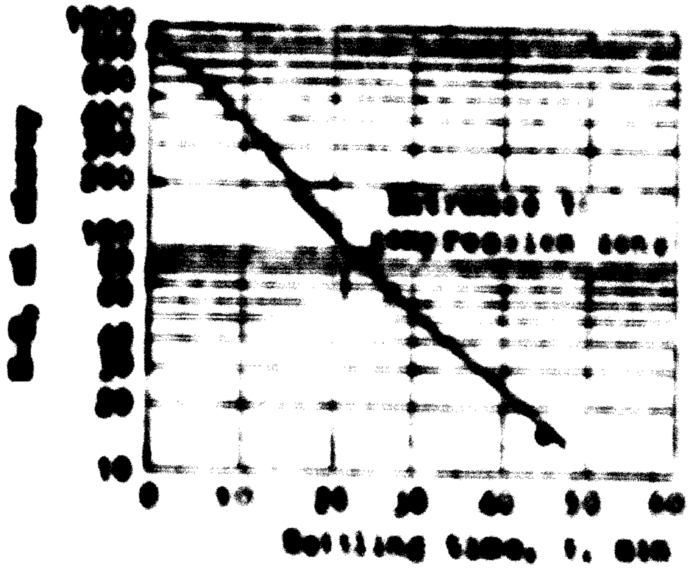
[illegible]

1 [illegible]
2 [illegible]
3 [illegible]
4 [illegible]
5 [illegible]
6 [illegible]

7 [illegible]
8 [illegible]
9 [illegible]
10 [illegible]
11 [illegible]

MUD SETTLING CURVE

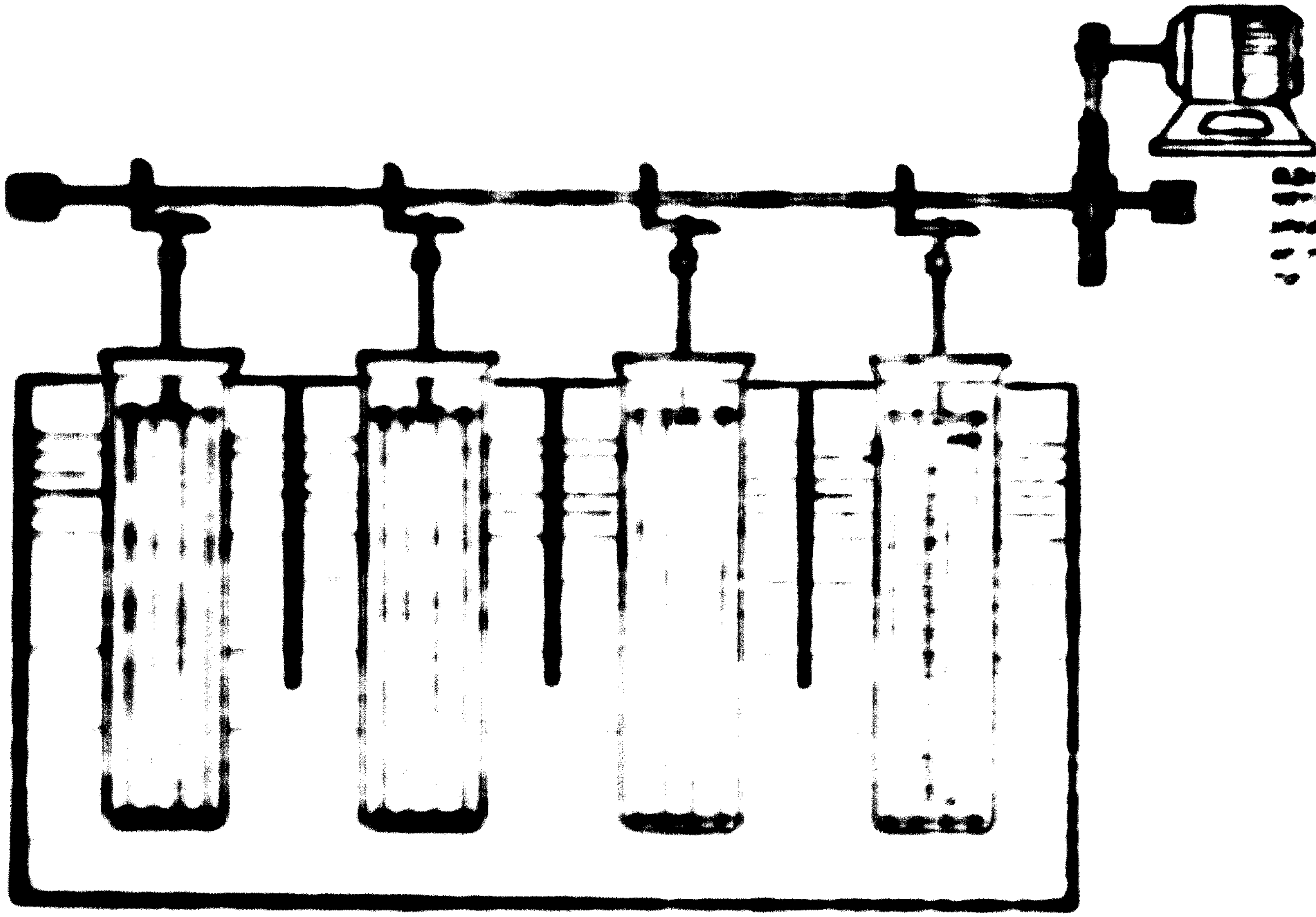


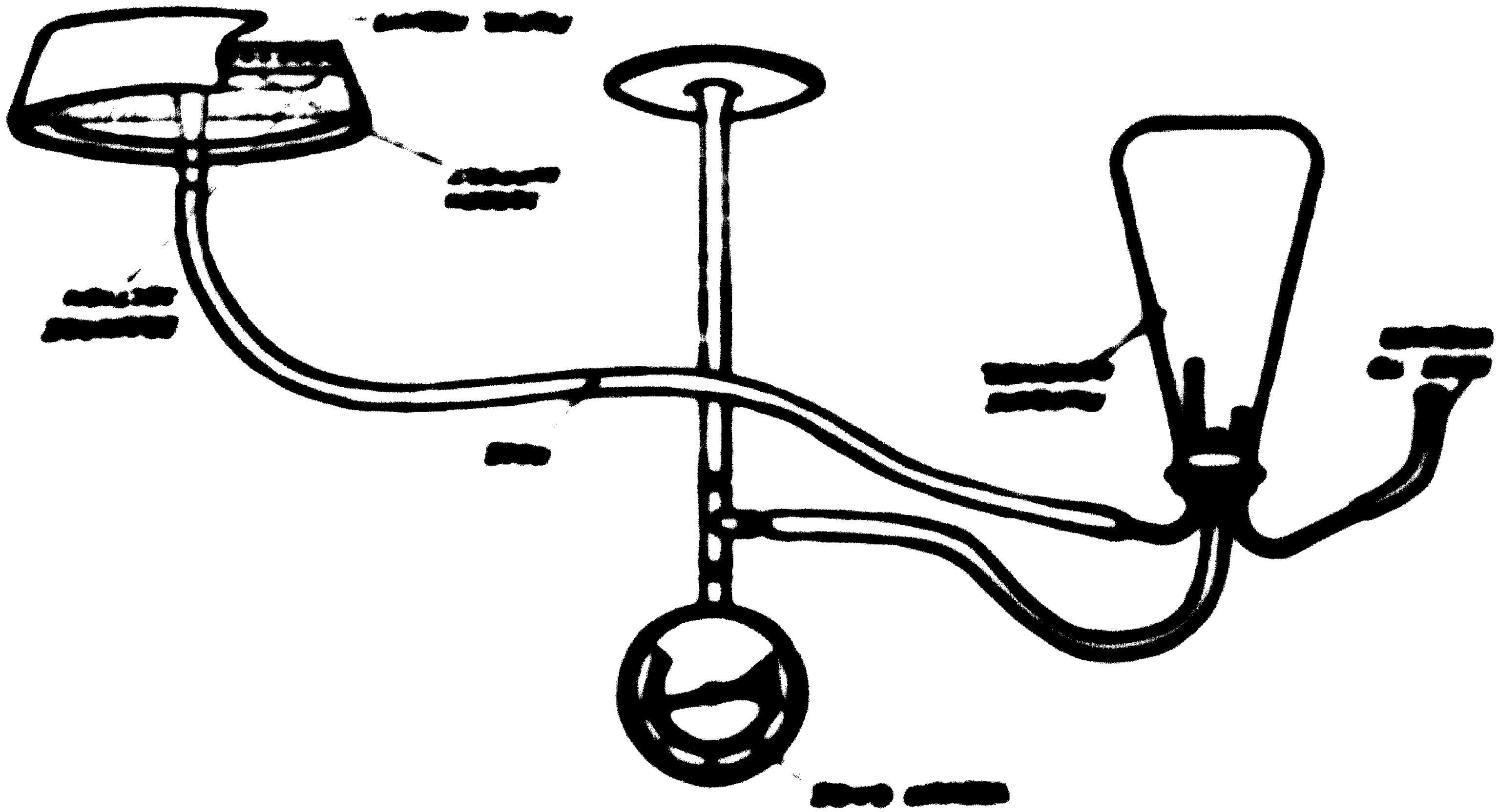


(To locate compression zone start by Roberts method)

[illegible] SETTLING TEST ASSEMBLY

FIG. NR 11





[illegible]

[illegible]

FIG NR. 13

PRECIPITATION YIELD EXPERIMENTS

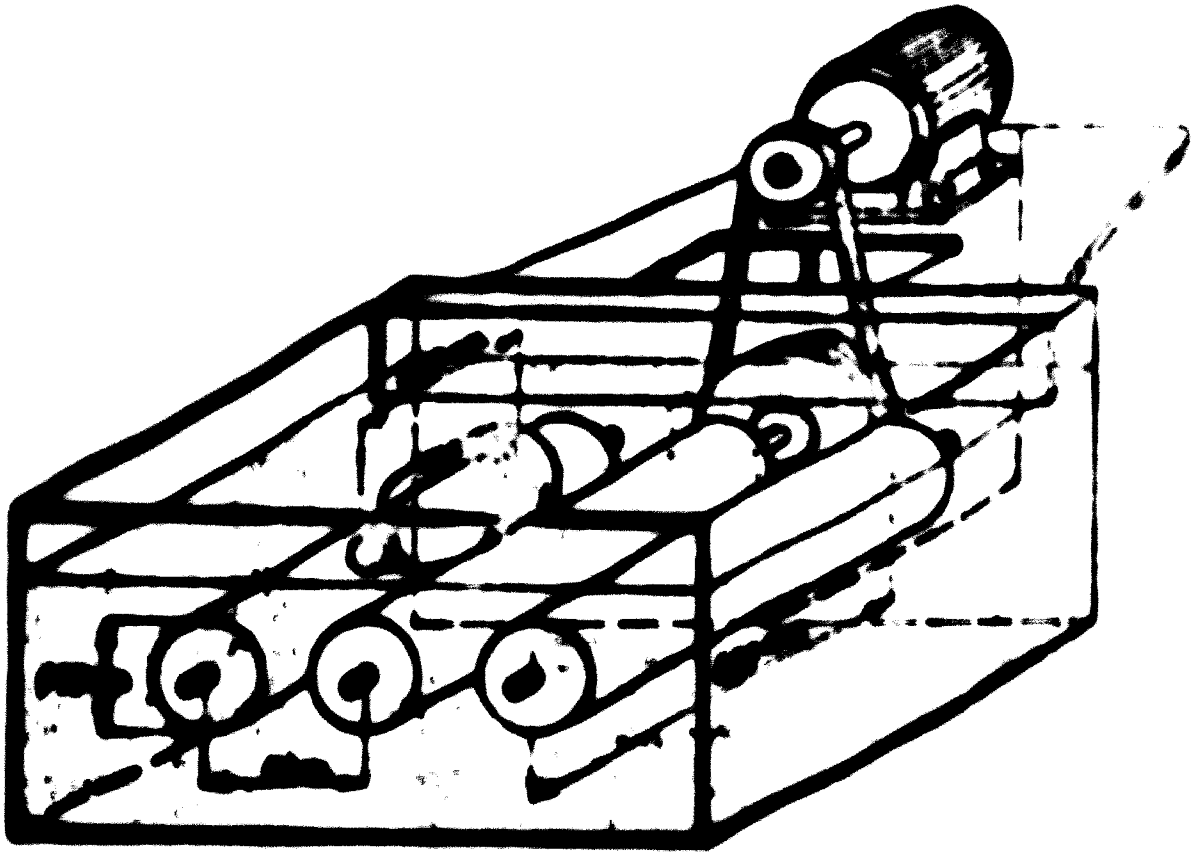
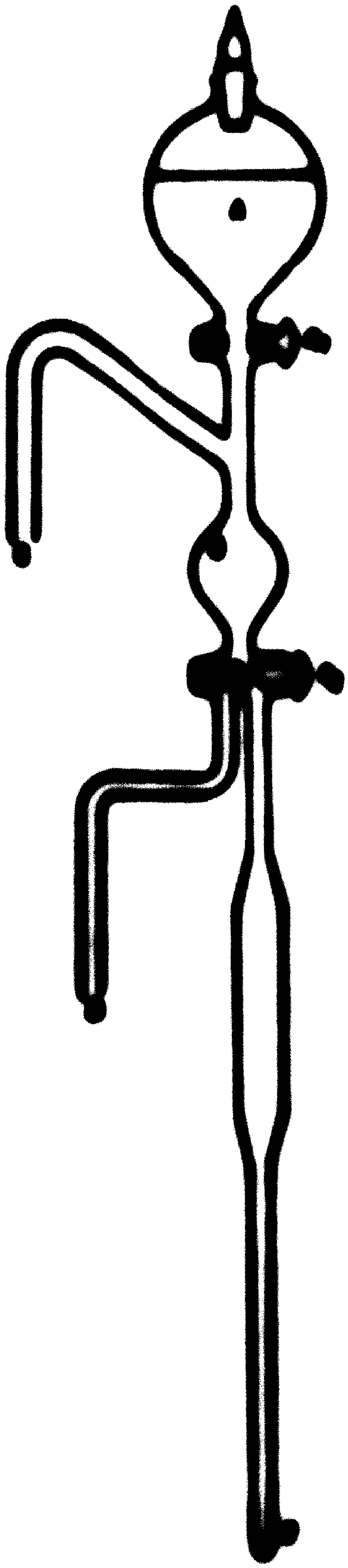
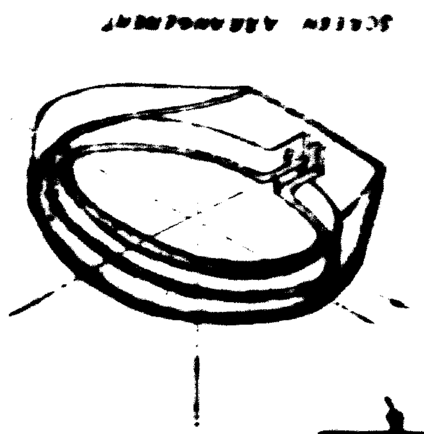


FIG. 14

ANDREASEN SEDIMENTATION TEST [illegible] PIPETTE




SCREEN ARRANGEMENT

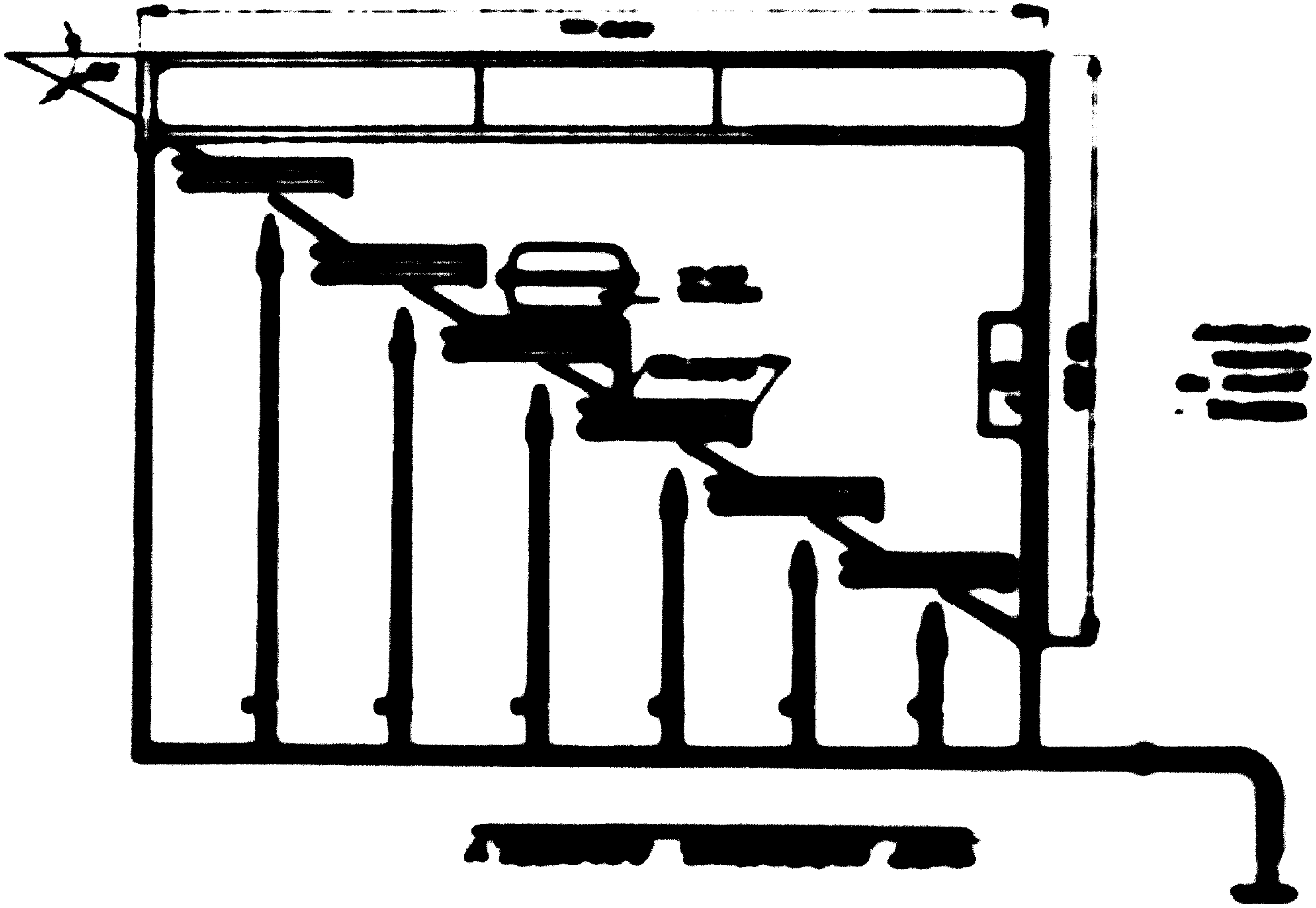
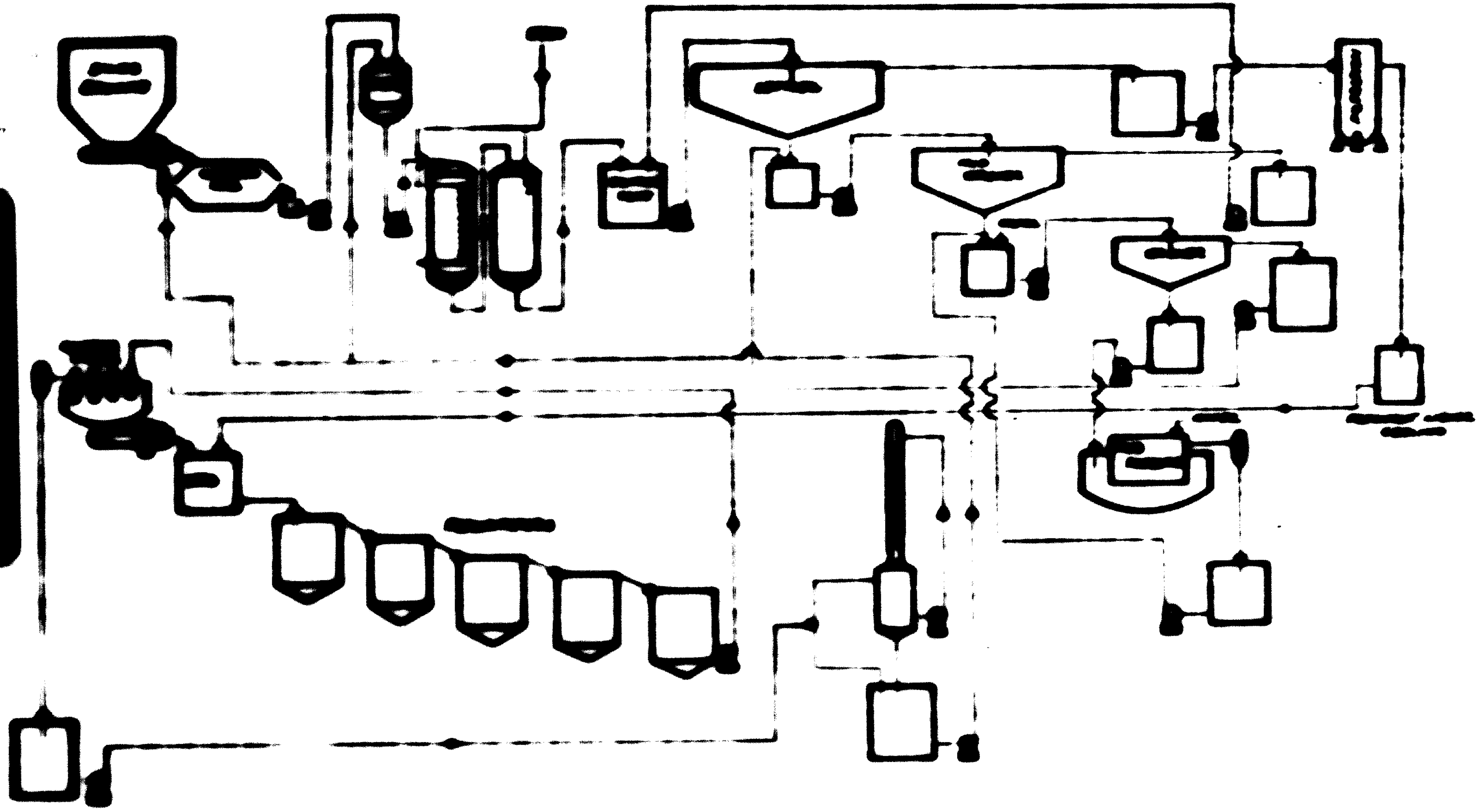


FIG. No. [illegible]

[illegible] PLANT [illegible]-SHEET [illegible]



1 - 827

82.06.24