REPORT OF INVESTIGATIONS

REPORT OF THE NONMETALS DIVISION, FISCAL YEAR 1942

BY

O. C. Ralston and A. George Stern
R.I. 3675,  
November 1942.

REPORT OF INVESTIGATIONS  
UNITED STATES DEPARTMENT OF THE INTERIOR - BUREAU OF MINES

REPORT OF THE NONMETALS DIVISION, FISCAL YEAR 1942

By O. C. Ralston and A. George Stern

CONTENTS

<table>
<thead>
<tr>
<th>Topic</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>Introduction</td>
<td>2</td>
</tr>
<tr>
<td>Acknowledgments</td>
<td>3</td>
</tr>
<tr>
<td>Alumina from clay and low-grade bauxites</td>
<td>3</td>
</tr>
<tr>
<td>Beneficiation of bauxite</td>
<td>4</td>
</tr>
<tr>
<td>Extraction via calcium aluminates</td>
<td>4</td>
</tr>
<tr>
<td>Pedersen extraction of Seailles sinters</td>
<td>7</td>
</tr>
<tr>
<td>Lime-soda process</td>
<td>8</td>
</tr>
<tr>
<td>Electro-fusion method</td>
<td>10</td>
</tr>
<tr>
<td>Electrolysis of alumina</td>
<td>10</td>
</tr>
<tr>
<td>Extraction from Pacific Northwest clays</td>
<td>11</td>
</tr>
<tr>
<td>Potassium carbonate from wyomingite and trona</td>
<td>11</td>
</tr>
<tr>
<td>Optical studies</td>
<td>12</td>
</tr>
<tr>
<td>Mineral dressing and beneficiation</td>
<td>15</td>
</tr>
<tr>
<td>Beneficiation of alunite</td>
<td>16</td>
</tr>
<tr>
<td>New methods of cleaning glass sands</td>
<td>16</td>
</tr>
<tr>
<td>Electrostatic separation</td>
<td>17</td>
</tr>
<tr>
<td>Mineral dressing of Oregon beach sands</td>
<td>17</td>
</tr>
<tr>
<td>Sticky surface concentration of gravel-size min-</td>
<td>19</td>
</tr>
<tr>
<td>erals</td>
<td></td>
</tr>
<tr>
<td>Preparation and beneficiation of graphite</td>
<td>20</td>
</tr>
<tr>
<td>Recovery of coarse flake graphite</td>
<td>20</td>
</tr>
<tr>
<td>Kish graphite</td>
<td>20</td>
</tr>
<tr>
<td>Selection and treatment of domestic talc</td>
<td>21</td>
</tr>
<tr>
<td>Up-grading of Alabama iron ore</td>
<td>22</td>
</tr>
<tr>
<td>Selection and utilization of silica sand of Washington</td>
<td>22</td>
</tr>
<tr>
<td>Clays</td>
<td>22</td>
</tr>
<tr>
<td>Classification by uses</td>
<td>22</td>
</tr>
<tr>
<td>White clays</td>
<td>23</td>
</tr>
<tr>
<td>Electrokinetic treatment of clay</td>
<td>24</td>
</tr>
<tr>
<td>Soluble sulfate content of pottery bodies</td>
<td>25</td>
</tr>
<tr>
<td>Pozzolanic minerals</td>
<td>26</td>
</tr>
<tr>
<td>Refractories</td>
<td>27</td>
</tr>
</tbody>
</table>

1/ The Bureau of Mines will welcome reprinting of this paper, provided the following footnote acknowledgment is used: "Reprinted from Bureau of Mines Report of Investigations 3675."

2/ Principal chemical engineer (formerly chief, Nonmetals Division), Bureau of Mines.

3/ Chemical engineer, Bureau of Mines.

9081
### CONTENTS (Cont’d)

<table>
<thead>
<tr>
<th>Refractories (Cont’d)</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>Electric firing and testing</td>
<td>27</td>
</tr>
<tr>
<td>Electric firing of radio insulators</td>
<td>27</td>
</tr>
<tr>
<td>India kyanite</td>
<td>28</td>
</tr>
<tr>
<td>High-alumina cement</td>
<td>28</td>
</tr>
<tr>
<td>Basic refractory brick from Washington olivine</td>
<td>28</td>
</tr>
<tr>
<td>Thermal studies</td>
<td>30</td>
</tr>
<tr>
<td>Magnesia-silica system</td>
<td>30</td>
</tr>
<tr>
<td>Identification of alumina-bearing minerals</td>
<td>31</td>
</tr>
<tr>
<td>Intercrystalline cracking of boiler steels</td>
<td>31</td>
</tr>
<tr>
<td>Coal Investigations</td>
<td>33</td>
</tr>
<tr>
<td>Utilization of coal refuse</td>
<td>33</td>
</tr>
<tr>
<td>Accelerated attrition tests</td>
<td>33</td>
</tr>
<tr>
<td>Activated carbon</td>
<td>34</td>
</tr>
<tr>
<td>Water softening</td>
<td>35</td>
</tr>
<tr>
<td>Pulverized coal and its preparation</td>
<td>35</td>
</tr>
<tr>
<td>Combustion of coal</td>
<td>36</td>
</tr>
<tr>
<td>Properties and uses of Washington coals</td>
<td>37</td>
</tr>
<tr>
<td>Byproduct coke for war</td>
<td>37</td>
</tr>
<tr>
<td>Summary</td>
<td>38</td>
</tr>
</tbody>
</table>

### INTRODUCTION

During the fiscal year 1942, the Nonmetals Division worked with the strategic minerals needed to win the war. Many of the Nation’s resources of nonmetallics are of utmost importance to vital war activities for purposes other than the extraction of metals, and other projects previously being studied have been suspended for the duration to devote all possible effort to increase the availability of these urgently needed materials.

In addition to the headquarters station at College Park, Md., the three other experiment stations of the Division were at Norris, Tenn., Tuscaloosa, Ala., and Seattle, Wash. The Electrotechnical Laboratory at Norris studied the application of electric power to the nonmetallic minerals while the Southern Experiment Station at Tuscaloosa conducted most of the mineral-dressing treatment tests, and the Northwest Experiment Station at Seattle investigated some problems on the preparation and utilization of coal (other projects relating to the utilization of coal are conducted by the Coal Division of the Bureau of Mines). At the Eastern Experiment Station at College Park attention was directed chiefly to the preparation of alumina from low-grade bauxite and clay and to other problems involving chemical treatment of minerals.
This report is one of a series summarizing accomplishments of the division during each fiscal year. For more detailed technical data, reference to original publications is suggested.

ACKNOWLEDGMENTS

Compilation of this report has been made partly from statements prepared by Hewitt Wilson, Will H. Coghill, and H. F. Yancey, supervising engineers of the Electrotechnical Laboratory, Norris, Tenn.; the Southern Experiment Station, Tuscaloosa, Ala.; and the Northwest Experiment Station, Seattle, Wash., respectively. Material submitted by John E. Conley, chemical engineer, and other section heads at the Eastern Experiment Station, College Park, Md., as well as data from published reports of the division staff also were used.

ALUMINA FROM CLAY AND LOW-GRADE BAUXITES

The unprecedented demand for aluminum for the fabrication of airplane sheet and other parts urgently needed in the war program has made the problem of supplying metallurgical-grade alumina of paramount importance. Also, attention has been focused on the question of raw-material sources for this aluminum. The low-grade, high-iron, and high-silica bauxites of Georgia and neighboring States, and especially the deposits in Arkansas, probably offer the best possibilities.

The engineers of the Bureau of Mines have been checking on the quantities of the various grades of bauxites available. In one estimate of the reserves a total of 8,893,000 long tons of grade B (7 to 15 percent SiO₂) and 8,439,000 tons of grade C (15 to 30 percent SiO₂) are listed. These are the types of bauxites for which the calcium aluminate and lime-soda sintering processes should be suited. Obviously, an appreciable tonnage of alumina would be produced from the processing of these materials.


Beneficiation of Bauxite

A study was undertaken to determine the feasibility of beneficiating low-grade domestic bauxite to obtain a product suitable for refining by the Bayer process to yield alumina for the aluminum industry. Laboratory tests were made on 77 bauxite samples from Arkansas, Mississippi, Georgia, and Alabama. The Arkansas samples were most amenable and with few exceptions yielded concentrates containing 56 percent or more alumina and less than 3 percent silica. Three of the Arkansas samples, representing large tonnages of submarginal material, were tested further by continuous runs on a pilot-plant scale. The results of the laboratory and pilot-plant tests are encouraging and indicate that acceptable products may be prepared from many of the low-grade bauxites should occasion demand.

The problem in beneficiating bauxite is one of recovering gibbsite, the principal aluminum-ore mineral in bauxite, and rejecting clay and other accessory gangue minerals. Various methods of treatment were employed, depending on the physical characteristics of the particular sample. Some of the bauxites concentrated readily, and vigorous blunging by high-speed agitation, followed by desliming, gave a good separation of gibbsite in a granular product relatively free of kaolin. Other bauxites were more difficult and required fine grinding before separation could be effected. Differential grinding and desliming alone or in conjunction with gravity-concentration processes such as tabling and jiggling gave acceptable results. A method of flotation was developed and proved useful as an adjunct process for the recovery of fine gibbsite and rejection of contaminating gangue minerals. On many bauxites flotation alone was satisfactory and gave a high recovery of gibbsite in products of low silica content. A preliminary report on the flotation of several Arkansas bauxites has been published. Flotation has certain limitations, but the process is applicable to many bauxites and should prove particularly useful as an adjunct to other methods when low-silica products are required.

Extraction of Alumina via Calcium Aluminates

An intensive investigation has been made to determine the technical feasibility of the extraction and recovery of metallurgical-grade alumina from low-grade bauxites containing siliceous minerals, chiefly kaolinite, in amounts that make these aluminous materials unsuited for treatment by the Bayer process. This study has been restricted largely to the heating or sintering of these siliceous bauxites with limestone and with limestone and soda ash to make the alumina soluble in aqueous or dilute-alkali solutions and to effect a separation from the major portion of the associated impurities, notably Fe₂O₃, SiO₂, TiO₂, CaO, MgO, and traces of other compounds.

In the process comprising the sintering of bauxite with limestone only, with a subsequent water-leaching treatment, virtually all of the impurities present in the sintered material remain in the insoluble residue, while a solution containing lime and alumina in an approximately equimolar ratio is obtained. Aside from the very low solution concentrations obtainable (1.0 to 1.2 gm. Al₂O₃/liter), certain advantages are associated with this process. The procedure followed was essentially that of Seailles.²/

This process entails treatment of aluminous materials such as clays, coal ash, colliery wastes, shales, and especially siliceous bauxites by sintering with limestone under proper conditions. The limestone is proportioned in the charge to provide enough lime to produce dicalcium silicate (2CaO·SiO₂) and calcium monoaluminate (CaO·Al₂O₃) and also make dicalcium ferrite (2CaO·Fe₂O₃) and monocalcium titanate (CaO·TiO₂) with the iron and titanium oxides. The monocalcium aluminate is the only water-soluble compound in this mixture, so that a quantitative separation can be effected. The dilute solution containing this aluminate can be treated in various ways to recover the alumina.

Most of the work has been done on siliceous bauxites from Arkansas, but some preliminary tests were made on material obtained from Georgia. High-calcium limestones from Arkansas and Pennsylvania have been utilized.

The percentage extraction of the alumina has not been good enough to be of commercial interest, although the process apparently is technically feasible. The tendency of the silica to react and form gehlenite (2CaO·Al₂O₃·SiO₂), which is insoluble in water and dilute alkalies, appears to be a major difficulty. Recoveries are decreased in direct proportion to the amount of this constituent formed during sintering.

Specific results obtained. - Analyses of the bauxite and limestone used in most of the tests are given in the following table:

<table>
<thead>
<tr>
<th>TABLE 1. - Analysis of raw materials</th>
</tr>
</thead>
<tbody>
<tr>
<td>Material</td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td>Arkansas bauxite</td>
</tr>
<tr>
<td></td>
</tr>
</tbody>
</table>

²/ Includes 0.20 percent MgO.
³/ Includes 0.24 percent MgO.

These materials were ground to 200-mesh, thoroughly mixed, and, after being sintered, were reground to again pass the same size sieve. The early extractions were made with water maintained by thermostatic control at the alleged optimum temperature of 29° C. Later tests at prevailing room temperature (25° to 32° C.) indicated this range to be as good as the recommended optimum.

Detailed information on sintering conditions was lacking, as were data on the composition of the bauxite for which the process was adapted. The use of temperatures of 950° to 1,100° C. was reported to be satisfactory. Tests made at these temperatures for periods of heating ranging from 1 hour to as much as 24 hours resulted in the extraction of only 36 percent of the total alumina. Subsequent tests indicated that higher temperatures were necessary. Longer heating with an oxidizing furnace atmosphere increased the extractability of the alumina. However, even heating 24 hours at 1,250° C. and an additional 24 hours at 1,300° C. produced a sinter that yielded only a 53-percent extraction.

Final tests made by sintering the mixture in the strongly reducing atmosphere of a graphite tube heated by a high-frequency induction furnace for periods of 45 to 50 minutes and at temperatures from 1,250° to 1,350° C. produced sinters that would yield approximately 63 percent of their alumina to a water-extraction treatment.

Throughout all the experiments the dissolution tests were made to duplicate the results claimed for the Seailles process. These were a solution concentration of 1.6 gm. of alumina per liter and a 75- to 80-percent extraction. Usually the actual extractions were continued until the optimum concentrations were attained, which in some instances required from 8 to 9 hours. The nearest approach to attainment of the desired results was 1.25 gm. of alumina per liter of water, equivalent to approximately 64-percent extraction of alumina. Attempts made to apply the countercurrent extraction procedure outlined by Seailles indicated a recovery of approximately 50 percent, with a concentration of leach liquor approaching 1.0 gm. of alumina per liter.

All efforts to obtain specific information on the composition of the French bauxites, reputedly satisfactory for treatment by the Seailles process, were unsuccessful. However, from the analysis given for an extracted residue and the assumption that a 75-percent extraction of the alumina had been effected, the estimated composition of the original raw material was calculated. This indicated that before extraction the sinter would contain 41.1 percent CaO, 39.15 percent Al₂O₃, 5.0 percent SiO₂, 13.00 percent Fe₂O₃, and 1.5 percent TiO₂. It will be noted that the weight ratio of alumina to silica is 7.8, as compared with 5.18 for the Arkansas bauxite-limestone mixture tested.
A sample of beneficiated Arkansas bauxite of approximately the composition of the French bauxite, as obtained by the calculations mentioned above, was tested for comparative purposes. This bauxite contained 57.75 percent $\text{Al}_2\text{O}_3$, 5.43 percent $\text{SiO}_2$, 3.04 percent $\text{Fe}_2\text{O}_3$, 2.14 percent $\text{TiO}_2$, and 29.65 percent ignition loss. The alumina:silica ratio obviously is similar, though slightly higher, and the iron oxide somewhat lower than those corresponding to the French material. However, the sinter prepared from this sample was found in duplicate tests to yield 82.1 and 80.7 percent of its alumina content, with concentrations of 1.64 and 1.61 gm. of alumina per liter, respectively, when extracted for 6 hours.

Apparently the tendency to form the insoluble compound gehlenite ($2\text{CaO}.\text{Al}_2\text{O}_3.\text{SiO}_2$) in these sinters is the chief obstacle that prevents high-alumina extraction and recoveries. The presence of this compound has been proved by X-ray diffraction patterns. Obviously, the amount that will be formed increases with the increase in silica content. Likewise, the time-extraction curves show that as the alumina is dissolving, some of it is being removed from solution as a hydrated calcium aluminate, probably tricalcium-aluminate hexahydrate ($3\text{CaO}.\text{Al}_2\text{O}_3.6\text{H}_2\text{O}$). These two factors, in conjunction with the impossibility of attaining complete reaction of the lime with all the alumina and the inability to dissolve all of the alumina that has formed monocalcium aluminate, explain the results obtained. Therefore, it appears justified to conclude that a bauxite containing 5 to 6 percent silica will permit extraction and recovery of 75 to 80 percent of the alumina content of the bauxite at 1.6 gm. per liter, whereas recovery will be reduced to 80 to 65 percent with ore containing 9 to 10 percent silica. This latter figure is definitely lower than is considered satisfactory in an economic process.

In other words, the Seailles results could be duplicated only when using bauxite of a grade that works satisfactorily by the orthodox Bayer process. Bauxites with higher silica gave increasing trouble and lower extraction of alumina by Seailles' process, and it is these siliceous bauxites that constitute the main problem in America.

**Pedersen Extraction of Seailles Sinters**

Numerous extraction tests were made by employing the Pedersen alkali solutions on various sinters from a mixture of limestone and bauxite proportioned to make monocalcium aluminate and dicalcium silicate in accordance with the recommended Seailles process. The Pedersen leaching technique yields much higher concentrations of alumina (20 to 40 gm./$\text{Al}_2\text{O}_3$ liter) by employing leach liquors containing 3 to 5 percent $\text{Na}_2\text{CO}_3$ and 10 percent of the total alkali as NaOH. Some silica is dissolved, and the end liquors, after the alumina has been precipitated, must be recycled. This introduces the additional expense of evaporation to maintain the proper concentrations of alkali.
This extraction process was found to give good yields (80 percent of the Al₂O₃) on synthetic calcium aluminates and on sinters made from a beneficiated bauxite of low silica content (5.43 percent SiO₂). However, the sinters made by the Seailles process, while giving higher extractions than when treated with water, still failed to yield more than 70 to 75 percent of their alumina. Likewise, gehlenite, which has been shown to be present in the Seailles sinters, was but slightly soluble in these sodium carbonate-hydroxide solutions.

**Lime-Soda Process**

The next logical step in the study of the alkaline-extraction process was to investigate the sintering of high-silica bauxites with admixed limestone and soda ash. Information available in the patent and technical literature suggested that good extractions could be expected but that soda losses and purification of the leach liquors to eliminate the silica would be the major difficulties.

The essential steps of this process include (a) sintering of the bauxite or other aluminous material with limestone and soda ash proportioned to provide approximately 2 moles of lime per mole of silica and 1 mole of soda for each mole of total alumina present; (b) extraction of the soluble sodium aluminates from the insoluble calcium silicate and other impurities in the sintered mass, either by a water- or dilute soda leach-liquor; (c) pressure treatment of the sodium aluminate solution with or without added reagents to precipitate the major portion of the soluble silicate; and (d) the controlled carbonation of the desilicated solution to produce a granular crystalline form of hydrated aluminum oxide. Calcination, of course, will be necessary to obtain an anhydrous product adapted for use in the electrolytic aluminum-cells.

The first low-grade bauxite tested by this method was one from Georgia containing 17.0 percent SiO₂. The preliminary series of tests yielded extraction in excess of 90 percent in the small laboratory-size experiments. The composition of this material, as well as that of various other low-grade bauxites used, is given in table 2.
### TABLE 2. - Bauxites used in extraction experiments

<table>
<thead>
<tr>
<th>No.</th>
<th>Type</th>
<th>Al₂O₃</th>
<th>SiO₂</th>
<th>Fe₂O₃</th>
<th>TiO₂</th>
<th>Ignition loss</th>
</tr>
</thead>
<tbody>
<tr>
<td>74</td>
<td>Georgia high SiO₂</td>
<td>54.04</td>
<td>13.63</td>
<td>4.17</td>
<td>1.55</td>
<td>26.75</td>
</tr>
<tr>
<td>414</td>
<td>Arkansas, siliceous</td>
<td>53.90</td>
<td>9.22</td>
<td>6.28</td>
<td>1.78</td>
<td>27.60</td>
</tr>
<tr>
<td>429</td>
<td>Arkansas, concentrates</td>
<td>57.75</td>
<td>5.43</td>
<td>5.04</td>
<td>2.14</td>
<td>29.65</td>
</tr>
<tr>
<td>450</td>
<td>Arkansas, tailings</td>
<td>51.28</td>
<td>21.89</td>
<td>2.56</td>
<td>0.67</td>
<td>23.60</td>
</tr>
<tr>
<td>481</td>
<td>Slime rejects</td>
<td>50.30</td>
<td>1/21.3</td>
<td>2.80</td>
<td>.88</td>
<td>23.0</td>
</tr>
<tr>
<td>Bx40B</td>
<td>Fletcher, high SiO₂ and Fe₂O₃</td>
<td>48.15</td>
<td>14.47</td>
<td>9.00</td>
<td>2.54</td>
<td>25.84</td>
</tr>
<tr>
<td>Bx50</td>
<td>Republic, high SiO₂</td>
<td>58.48</td>
<td>8.30</td>
<td>2.31</td>
<td>1.09</td>
<td>29.82</td>
</tr>
<tr>
<td>Bx24B</td>
<td>Mabelvale, high SiO₂</td>
<td>52.80</td>
<td>18.40</td>
<td>2.41</td>
<td>2.55</td>
<td>23.80</td>
</tr>
<tr>
<td>A83</td>
<td>Arkansas raw bauxite</td>
<td>51.20</td>
<td>21.7</td>
<td>1.9</td>
<td>1.5</td>
<td>22.5</td>
</tr>
<tr>
<td></td>
<td>Limestone</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>416</td>
<td>Arkansas (CaO=54.6)</td>
<td>1.17</td>
<td>1.79</td>
<td>0.38</td>
<td>0.1</td>
<td>42.7</td>
</tr>
<tr>
<td></td>
<td>(MgO=0.24)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

1/ Also contains 0.69 percent CaO.

The various factors affecting the extraction and recovery of high-grade alumina from these siliceous bauxites have been studied in detail, including (a) the effect of composition on the extractability of the sinter, especially the amount of iron oxide present; (b) the effect of time, temperature, and furnace atmosphere; (c) the effect of variation in lime and soda ratios to silica and alumina, respectively; (d) the time, temperature of extraction, particle size, liquid: solid ratio, amount of alkali in the leach liquor, and concentration of the extract liquor; (e) the temperature and pressure as well as nature of reagent added to effect removal of silica from the sodium aluminate solution; and (f) the temperature, rates of carbonation and agitation, and effect of pH on the relative precipitation of alumina and silica.

The various steps have been studied in sufficient detail to determine the optimum conditions for treating the Arkansas siliceous bauxite to obtain a metallurgical-grade alumina containing less than 0.03 percent SiO₂ and less than 0.02 percent Fe₂O₃ with minor amounts of other impurities.

Laboratory-scale experimentation has been conducted involving the treatment of several hundred grams of material per batch and the handling of 2 or 3 liters of solutions that result in the precipitation and recovery of upwards of 100-gm. lots of finished hydrated alumina. However, the calcination step has been extended to the sintering of 200- and 300 pound batches, and, likewise, the extraction treatment has been applied to 5- and 0-gallon lots.
The process has been studied in enough detail to prove its possible merit, and while a few pertinent data remain to be acquired, the process is believed ready for further checking by small pilot-plant operations. A detailed flow sheet has been prepared and a critical study made of possible types of equipment needed to test the scheme as a continuous process. It is believed that the treatment suggested will make alumina of a grade acceptable to the aluminum pot rooms for producing high-grade aluminum. It is further believed that the costs of applying the process will compare favorably with those of the standard Bayer-process treatment method, which employs imported bauxite.

**Electro-fusion Method**

The Electrotechnical Laboratory collaborated with the Eastern Experiment Station in the preparation of electrically fused calcium aluminate samples for production of Al₂O₃. Preliminary work emphasized the difficulties involved in obtaining complete reduction of silica from bauxite-lime charges. Although part of the alumina, silica, and lime was converted to insoluble gehlenite, a large part of the remainder of the Al₂O₃ was shown to be soluble in an alkaline extraction of the pulverized fusions.

It would be desirable to make a ferrosilicon byproduct containing 50 to 75 percent silicon, as this grade is wanted in the Pidgeon process for the reduction of MgC. The furnace tests indicated that this may require more than a single operation.

Furnace manipulations that may eliminate a large part of the silica as ferrosilicon are being investigated. A two-stage treatment suggested by previous work consisted of the preparation of fused alumina by the reduction of silica, iron oxide, etc., followed by reaction with lime in a subsequent treatment in the furnace. A program of work has been laid out to check these assumptions. The tap furnace shown in figure 1 will be used for the new tests.

**Electrodialysis of Alumina**

At the request of the War Metallurgy Committee of the National Research Council, an investigation has been started in cooperation with Kalunite, Inc., Salt Lake City, Utah, for the removal by electrodialysis of the potassium impurity in the alumina derived from alunite. The operators report that potassium causes trouble in the reduction furnaces, and its presence has stymied the production of aluminum from clay and alunite. Beneficiation by electrodialysis involves electrolysis of a suspension of alumina confined by membranes, through whose pores the ions of the soluble salts are forced and eliminated by the impressed electric field.
Figure 1. Two-electrode tapping furnace for melting bauxite, clay, limestone, etc., and draining calcium aluminate into molds.
Extraction from Pacific Northwest Clays

An investigation was undertaken of the extraction of alumina from clays of the Pacific Northwest. Deposits were examined and samples obtained from the Castle Rock, Mica, and Pullman areas in Washington, from the Coeur d'Alene and Moscow areas in Idaho, and from Molalla and Hobart Butte areas in Oregon. Samples were also received from a deposit near Enumclaw and one near Wenatchee, Wash. Three of the clays have been examined in the laboratory.

Thermal analyses were made to determine the mineral composition and chemical analyses for oxide composition. Dehydration, pyrometric cone equivalent, rate of weight loss, carbon content, and petrographic studies also are being carried out.

The percentage of alumina extracted is derived by leaching with a solution of sulfuric acid. Experiments are being conducted to determine the effect of particle size of a clay on rate of solution. Attempts also are being made to associate properties and mineral composition with alumina extraction.

Only one clay showed an appreciable extraction by hot acid without previous calcination. This was due to the presence of the minerals halloysite and gibbsite. Two other clays examined consisted principally of kaolinite, which required calcination in the range between its decomposition temperature and the temperature at which alpha alumina forms. With suitable treatment, the clays tested showed about 90 percent of the alumina to be extractable by the laboratory procedure.

POTASSIUM CARBONATE FROM WYOMINGITE AND TRONA

The discovery of an extensive deposit of trona\(^8\), \(\text{Na}_2\text{CO}_3 \cdot \text{NaHCO}_3 \cdot 2\text{H}_2\text{O}\), underlying the vicinity of Green River, Wyo., has stressed the possibility of adding not only to the soda-ash supply but also of extending the potash resources of the United States through utilization of the immense outcropping of the potassium-bearing rock Wyomingite in the immediate neighborhood.

The wyomingite has among its constituents 50 percent of the mineral leucite, the potassium content of which is amenable to "base exchange" or replacement by sodium\(^9\).

---


It has been proposed to interact trona and wyomingite under such conditions as to effect the production of pearl ash, potassium carbonate. Production and purification require several chemical engineering unit operations, all of which are standard procedures but which may require modification to produce an acceptable grade of potassium carbonate.

A pilot plant has been erected, and tests have been started to determine the best conditions for pearl-ash preparation. Studies will be made of the physical-chemical relations and the chemical-engineering conditions to permit design of a commercial plant should the results warrant it.

**OPTICAL STUDIES**

The optical unit, consisting of the microscopic, x-ray, and spectrographic laboratories, has continued its function of cooperation and assistance in the identification and evaluation of mineral materials, chemical precipitates, and other solid phases originating in the flotation, electrostatic, and chemical laboratories of the station. Also, many samples examined originate from outside sources.

Although this is primarily a service unit, research investigations are being conducted within the unit or in cooperation with other branches.

During the year a publication on the beneficiation of alunite was issued in cooperation with the flotation laboratory.

The domestic reserves of alunite, as estimated by Thoenen, total 47,000,000 tons of rock containing, roughly, 13,800,000 tons of alunite. Of this amount, about 9,400,000 tons of alunite may be high-grade enough to be considered as a source of alumina and byproduct potash.

Domestic alunite can be classified into two categories - the vein type and the disseminated or replacement type. The latter type constitutes about ninety percent of the reserve. It is normally of fine grain size and intimately associated with other minerals, particularly quartz, and with much smaller amounts of clay and iron oxides. This is the type of material on which flotation was tried in an attempt to float the alunite from associated gangue. Results of these tests are discussed on p. 13.

---

10/ Chemical and Metallurgical Possibilities for Wyoming Chemical Industry: Chemical and Metallurgical Engineering, September 1941, pp. 112-113.
A record paper was published on the minor constituents of spodumene.\footnote{13} Spodumene, the lithium-bearing pyroxene, has promising ceramic uses if the content of certain elements, particularly iron, is low enough not to cause tinting in the body in which it is used. Published analyses of spodumene show iron contents usually much higher than that of commercial feldspar. An investigation was therefore made to determine whether the iron and other impurities were present as constituents of the spodumene itself, or whether they occurred as a separate phase.

The cleanest, clearest material from various spodumene deposits was selected under the microscope, and the minor constituents were then determined quantitatively (see table 3) by the spectrographic method developed in this laboratory.\footnote{14}

\begin{table}
\centering
\begin{tabular}{|c|c|c|c|c|c|c|c|c|c|}
\hline
Sample No. & Origin & \multicolumn{9}{c|}{Percent} \\
\cline{3-10}
 & & \text{Fe}_2\text{O}_3 & \text{MnO} & \text{TiO}_2 & \text{SnO}_2 & \text{Ga}_2\text{O}_3 & \text{Na}_2\text{O} & \text{K}_2\text{O} & \text{Rb}_2\text{O} \\
\hline
1 & Lincolnton, N.Car. & 0.44 & 0.54 & 0.012 & 0.15 & 0.07 & 0.33 & 0.34 & 0.048 \\
2 & Dixon, N. Mex. & 0.040 & 0.013 & 0.004 & n.d. & 0.08 & 0.27 & 0.051 & n.d. \\
3 & Kings Mountain, N. Car. & 0.30 & 0.076 & 0.006 & 0.065 & 0.060 & 0.27 & 0.021 & n.d. \\
4 & Kings Mountain, N. Car. & 0.41 & 0.055 & 0.004 & 0.063 & 0.064 & 0.28 & Trace & n.d. \\
5 & Tinton, S. Dak. & 0.17 & 0.014 & 0.004 & 0.062 & 0.032 & 0.48 & 0.37 & n.d. \\
6 & Warren, Maine & 0.89 & 0.074 & 0.005 & 0.084 & 0.058 & 0.29 & 0.32 & n.d. \\
7 & Warren, Maine & 0.73 & 0.082 & 0.011 & 0.12 & 0.054 & 0.28 & 0.12 & n.d. \\
8 & Grover, N. Car. & 0.78 & 0.059 & 0.010 & 0.078 & 0.056 & 0.27 & Trace & n.d. \\
9 & Grover, N. Car. & 0.64 & 0.024 & 0.010 & 0.079 & 0.081 & 0.27 & Trace & n.d. \\
10 & Keystone, S. Dak. & 0.30 & 0.021 & 0.004 & 0.047 & 0.042 & 0.35 & 0.10 & n.d. \\
11 & Pala, Calif. (kun-site) & 0.020 & 0.073 & 0.003 & n.d. & 0.043 & 0.28 & 0.022 & n.d. \\
\hline
\end{tabular}
\caption{Spectrographic analysis of minor constituents in spodumene}
\end{table}

Note. - n.d. = not detected.


The table shows that the impurities lie within the spodumene itself and consequently cannot be removed by mechanical processes. Neither petrographic nor x-ray studies showed any significant differences in the spodumenes. The x-ray diffraction pattern gave no evidence of a general displacement, nor was there a shift in the \( K\alpha \) doublets. The combined data suggest that the impurities exist as isomorphous replacements in the spodumene structure.

The near-gem varieties of spodumene used in this study should not be considered representative of the deposit as a whole, for conceivably the nongem varieties may contain less impurities than the clear grains analyzed. However, potentially commercial deposits of spodumene should be examined closely for objectionable minor constituents, for they may occur in the spodumene itself and therefore may not be separable by mechanical processes; the x-ray powder-diffraction pattern for spodumene is given on the publication.

Quantitative spectrochemical analyses are being applied to a number of products of laboratory investigations, particularly those containing minute amounts of impurities. Thus, \( K_2O \), \( SiO_2 \), and \( Fe_2O_3 \) present in alumina (to be used in the preparation of aluminum metal) are being determined spectrographically. Analysis of glass sands is another example.\(^{15}\)

The reproducibility that may be obtained by spectrographic analysis is shown in Table 4. Eight exposures were made on the National Bureau of Standards Glass Sand Standard No. 81. The iron content is 0.0732 percent \( Fe_2O_3 \), as taken from the average of 10 independent chemical determinations. (See Bureau of Standards Certificate of Analyses of Standard Sample No. 81.)

**TABLE 4. - Comparison of chemical and spectrographic analyses of Bureau of Standards glass sand No. 81**

<table>
<thead>
<tr>
<th>Exposure No.</th>
<th>Spectrographic analysis</th>
<th>Chemical analysis from certificate of analysis B. S. No. 81, ( Fe_2O_3 ), percent</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Weight of sample, mg.</td>
<td>( Fe_2O_3 ), percent</td>
</tr>
<tr>
<td>1</td>
<td>21.5</td>
<td>0.0735</td>
</tr>
<tr>
<td>2</td>
<td>23.4</td>
<td>0.074</td>
</tr>
<tr>
<td>3</td>
<td>27.0</td>
<td>0.069</td>
</tr>
<tr>
<td>4</td>
<td>27.8</td>
<td>0.0795</td>
</tr>
<tr>
<td>5</td>
<td>24.8</td>
<td>0.069</td>
</tr>
<tr>
<td>6</td>
<td>29.2</td>
<td>0.073</td>
</tr>
<tr>
<td>7</td>
<td>24.9</td>
<td>0.077</td>
</tr>
<tr>
<td>8</td>
<td>26.3</td>
<td>0.065</td>
</tr>
</tbody>
</table>

The table shows that the variation in the spectrographic method of analysis is in the same order of magnitude as chemical methods of analyses, at least when they are made by different analysts using different methods.

Table 5 shows the results of duplicate determination on four glass sands determined by the spectrographic method.

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Weight, mg.</th>
<th>Fe₂O₃, percent</th>
</tr>
</thead>
<tbody>
<tr>
<td>1a</td>
<td>23.7</td>
<td>0.038</td>
</tr>
<tr>
<td>1b</td>
<td>26.1</td>
<td>0.036</td>
</tr>
<tr>
<td>2a</td>
<td>25.4</td>
<td>0.088</td>
</tr>
<tr>
<td>2b</td>
<td>24.8</td>
<td>0.071</td>
</tr>
<tr>
<td>3a</td>
<td>28.2</td>
<td>0.018</td>
</tr>
<tr>
<td>3b</td>
<td>28.2</td>
<td>0.018</td>
</tr>
<tr>
<td>4a</td>
<td>22.3</td>
<td>0.23</td>
</tr>
<tr>
<td>4b</td>
<td>24.7</td>
<td>0.26</td>
</tr>
</tbody>
</table>

The results shown in these two tables are typical of a great many obtained on a variety of minerals. The average error of a single observation from the mean of a series is about 6 or 7 percent of the amount present, with occasional errors up to 15 percent. Ordinarily, samples are run in duplicate, so that the larger deviations can be detected easily and the analysis repeated. For duplicate analyses the error for a determination is reduced to about 5 percent. This uncertainty is roughly independent of the percentage present.

An investigation not yet completed of the thermal decomposition of topaz led to the recommendation that a rotary-type kiln be used for calcination and removal of fluorine. Through the cooperation of the Tennessee Valley Authority, the War Production Board, and the United Feldspar and Minerals Corporation, the T.V.A. calcined 15 tons of topaz in an experimental pilot-plant kiln. Tests are now being conducted on the calcined material at various commercial plants to determine if it can be used successfully as a substitute for India kyanite.

MINERAL DRESSING AND BENEFICIATION

Filming and other mineral-dressing processes have been applied to many nonmetallic materials of general interest to the mineral industry and of specific interest for national self-sufficiency. Concentration of various minerals by flotation, agglomeration, adhesion, classification, tabling, screening, conductance, contact potential, and dielectric electrostatic separation, high- and low-intensity magnetic separation, attrition scrubbing, attrition and self-grinding, and by combinations of these methods, was effected. The minerals concentrated included alunite, andalusite, barite, bauxite, calcite, cancrinite,
carborundum, chromite, clay, corundum, feldspar, fluorite, graphite (kish), garnet, gypsum, ilmenite, kyanite, muscovite, nephelite, phosphate rock, pyrite, rutile, sericite, serpentine, silica sand, sillimanite, talc, topaz, and zircon.

**Beneficiation of Alunite**

While making an economic survey of alunite reserves,\(^{16}\) which are to be used for the production of alumina, samples of various medium-grade deposits were obtained for flotation testing. The survey showed that only 10 percent of the reserves were of high-grade material. Flotation\(^{17}\) of the medium-grade (over 30 percent alunite) rock would reduce the proportion of inert gangue, which has to be heated, agitated, and filtered when this material is processed for the production of alumina from alunite.

To make a clean separation of two or more minerals by flotation, the rock containing these minerals must be ground until each grain contains only one mineral. Grains that are not ground fine enough will contain some of two or more minerals "locked" together. These locked grains are neither good concentrate nor good tailing, and they are true "middling" grains.

The alunite deposits contain such small crystals that it is not economical to grind the rock until the grains are fully unlocked. Therefore, tests were made after moderate grinding (through 100-mesh) to float the grains that were mainly alunite from those that were mainly quartz or other gangue. When a high-grade concentrate was produced, many grains containing 25 to 75 percent alunite were rejected into the tailing, and recovery of alunite was poor; but when a low-grade concentrate was made, these lost grains were recovered. The relationship between the grades and recoveries obtained is shown in figure 2. The flotation results obtained depended on the degree of locking. The Close-In sample, which gave the best results, had the fewest middling grains, and the White-Horse sample, which gave the poorest results, had the most.

Flotation reagents used were oleic acid, sodium silicate, and a frother. Figure 3 is the flow sheet used for pilot-plant tests.

**New Methods of Cleaning Glass Sands**

Since presentation of the paper on glass sands\(^{18}\) many new sand samples have been tested. A larger batch scrubber with 10-inch-diameter rotor and 5-horsepower motor has been constructed. If continuous, it would treat 25 to 60 tons a day.

Figure 2. - Relationship between analysis and recovery of alunite in pilot-plant tests.

Figure 3. - Pilot-plant flow sheet.
Figure 4.– Conductance electrostatic separator for Oregon chromite beach sands.
Sand was used to calibrate the device and give working data, although in its application it may be used for the recovery of any of several vital minerals needed for war purposes. By its use as a preliminary treatment previous to flotation, better and cleaner separations can be effected.

At present, where tin cans are being replaced by glass containers for the food industry, new sources of sand to increase production of glass are being sought. Often it is possible to scrub a local sand and make it available for glass-making purposes, avoiding the necessity of shipping other sand from greater distances. Such beneficiation treatment of local raw materials helps to eliminate the transportation bottleneck.

In cooperation with a large glass company, feldspar has been floated from Pacific-coast sand. A product containing 0.05 percent Fe₂O₃ was recovered in pilot-plant tests. Feldspar grains as coarse as 20-mesh were floated. Most of the Pacific-coast sands are geologically young and have not been worn down to a residue of the toughest quartz grains, but contain also feldspars, heavy minerals, and other undesirable material. Before the war, most Pacific coast glass-sand requirements came from Belgium as ship ballast. Details will be published as soon as semicommercial plant results are available.

**Electrostatic Separation**

An analytical study was made of the simple, roll-type electrostatic separator such that its behavior could be expressed in the form of graphs and mathematical equations. These equations not only made it possible to predict the results, but it was also a quantitative means of expressing the results. The usefulness of such a procedure is illustrated in the concentration of chromite-bearing sands (see fig. 4). Here the effect of changing the conditions of separation could be determined quickly by inserting in these equations the values obtained by merely weighing the separated fractions. The alternative is petrographic or chemical analysis, which requires considerably more time. A summary of this investigation is in the process of being published.¹⁹/

**Mineral Dressing of Oregon Beach Sands**

Seven samples from six deposits along 44 miles of the southern Pacific coast of Oregon of sands containing chromite, garnet, zircon, and many other minerals have been tested²⁰/

---


With the following treatment, salable concentrates of chromite and zircon and a fine garnet concentrate that may be salable can be obtained from each sample:

1. Disintegration of the soft sandstone, if necessary by rolling with water in a circular mill.

2. Screening on 4- to 6-mesh to remove pebbles and trash.

3. Attrition scrubbing\(^{21/}\) to remove silicate slime from grain surfaces to improve grade of product and results of electrostatic separation.

4. Classification and hydraulic tabling to reject quartz and green mafic minerals, which are worthless gangue.

5. Drying the table concentrate.

6. Conductance electrostatic separation of sized table concentrates to separate chromite and opaque minerals such as ilmenite and magnetite from garnet, zircon, and traces of green mafic minerals not removed in tabling. Precharging with needle-point corona followed by separation on a simple roll-type separator with a positively charged metal electrode gave the best results. (See fig. 4.)

7. Magnetic separation from chromite of as much of the ilmenite and magnetite-like minerals as necessary to increase the chromite content, reduce the titanium content, or improve the Cr:Fe ratio so that the chromite may be marketed most economically. Magnetic separation of garnet and zircon from the remaining traces of green mafic minerals.

With this treatment, concentrates containing over 43 percent \(\text{Cr}_2\text{O}_3\) were obtained from all six samples tested, with recoveries of 52 to 94 percent of the \(\text{Cr}_2\text{O}_3\). Higher recoveries of 40 percent \(\text{Cr}_2\text{O}_3\) concentrates may be obtained. On one sample a concentrate containing 46.4 percent \(\text{Cr}_2\text{O}_3\) was obtained with a recovery of 83.4 percent of the \(\text{Cr}_2\text{O}_3\).

The zircon concentrates contained as little as 0.03 percent \(\text{Fe}_2\text{O}_3\).

Figure 5. - Diagrammatic sketch of sticky-surface concentrator.
Sticky-Surface Concentration of Gravel-Size Minerals

Particles coarser than 6 to 10-mesh are not treated effectively by flotation and agglomeration processes. For some minerals, a market for coarse products exists. Some coarse-grained rocks liberate their minerals at a size larger than 1/4 inch.

For coarse-grained rocks that need concentration, especially those containing minerals marketed as coarse products, a process has been developed for beneficiation by filming one mineral with flotation reagents and causing it to adhere to a sticky, water-repellent surface.²²/

A photograph of the machine appeared in a previous report,²³/ and a diagrammatic sketch is shown in figure 5.

The sized mixture of coarse mineral grains is treated with the reagents required for flotation separation of the grains, so that one or more minerals are filmed with the collector to give them a water-repellent surface, and the other mineral or minerals are not filmed but are easily water-wettable. The mixture is fed into the revolving cone, whose inside surface is coated with a relatively soft, sticky, water-repellent substance such as petrolatum, soft tar, or pitch.

The filmed grains, which are not readily wetted by water, adhere to the sticky surface and are lifted to the top of the cone, where heat from a gas flame or electric heater softens the sticky surface, so that they drop or are combed out of the sticky surface into the concentrate launder. The other water-wetted grains do not adhere to the sticky surface and, as the cone rotates, roll on out the large end of the cone. The large end may be enclosed to reduce the floor space and the quantity of water required, if it is fitted with lifters for removing grains that do not adhere to the sticky surface. This form is shown in figure 5 as the suggested modification. After the adhering particles are removed, the sticky surface passes under an applicator, which smooths and replenishes the sticky surface with heat.

The warm surface is then quickly hardened and cooled as it enters the water, and the surface is ready for another revolution.

Phosphate rock, barite, feldspar, quartz, fluorite, calcite, and other minerals have been caused to adhere to the sticky surface selectively. Almost perfect separations were obtained on material from 10-mesh to 2-1/2 inches, although material coarser than 1-inch did not lift to the top of the cone and had to be removed by a cutter near the water surface.

Preparation and Beneficiation of Graphite

Recovery of Coarse Flake Graphite

Domestic requirements of crucible graphite have been supplied almost entirely by imports from Madagascar and Ceylon. With the beginning of hostilities imports were cut off, and crucible manufacturers faced the necessity of obtaining domestic graphite to satisfy their needs. A preliminary survey was made of the various deposits in the Alabama district in a search for a possible source of coarse flake. Samples from 35 of the 42 abandoned mines and prospects were obtained and tested by flotation to determine the yield and grade of recoverable graphite suitable for crucible uses. The results of the study were published as a cooperative report by the Geological Survey of Alabama.24/

With the preliminary data as a guide, the investigation was intensified and extended to include graphite from other districts. During the past year 235 samples from Alabama, Georgia, Pennsylvania, New York, Texas, and California have been received and tested at the request of the War Production Board. This study has been supplemented by continuous runs in a small-scale pilot plant on four of the most promising samples. Various flow sheets were used in an attempt to determine the best method for recovering graphite with minimum degradation of the coarse flake. Use will be made of the data in designing the plants proposed by the War Production Board for the Alabama, Pennsylvania, and Texas districts.

Kish Graphite

When a shortage of Madagascar flake graphite was expected, the possibility of using kish was investigated. Kish is a product composed mainly of graphite flakes that crystallize from molten iron on cooling. Although all molten high-carbon iron produces some kish in cooling, the greatest yield of these flakes is from high-silicon iron. Samples were obtained from a

large steel company and were beneficiated by froth flotation, acid leaching, and screening to obtain a 94 percent carbon product of satisfactory size. This product was tested by being used for test crucibles and stoppers. Results were not satisfactory. The artificial flakes are so much thinner than the natural flakes that they require more binder, break down during mixing, and oxidize more rapidly in use. The use of natural domestic flake graphite offers more promise. 25/

Selection and Treatment of Domestic Talcs

One of the most essential and vital pieces of equipment on our airplanes is the radio. High-frequency fields are used in which the ordinary types of insulators suitable for low frequencies become conductors and hence are worthless. Steatite (talc) porcelains, however, stand the work well. Before the heavy war demand these porcelains had been made in relatively small amounts and used as their main ingredient hand-picked talcs obtained largely from a single California source.

With the tremendously increased production now necessary, it is imperative to locate new sources. It would be particularly helpful if deposits closer to the eastern manufacturers could be found. The Bureau has been making a nationwide search with this in mind, and as a result some promising finds have been made in California and Alabama. In the East, there are large deposits of talcs in Alabama, North Carolina, Virginia, and New York, which seem unusually good, especially if processed according to modern flotation milling methods. Most raw talcs are unfit for use in these highly specialized bodies as they usually contain too much iron and lime as impurities. Sometimes, however, they may be beneficiated and brought up to grade, that is having a content of under 1 percent each of Fe₂O₃ and CaO.

Field engineers have inspected the deposits and collected the samples, and the Tuscaloosa laboratory has analyzed and evaluated them. Beneficiating tests were made on all but the most hopeless, and if the results were at all promising large batches were beneficiated and prepared for testing at the Norris laboratory in regular bodies for electrical and other properties. A talc giving excessive firing shrinkage is undesirable, and the final test of suitability is whether a fired ceramic body containing the talc makes an acceptable insulator.

An interim report was prepared after several dozen samples had been studied. The work in all phases is continuing.

Up-grading of Alabama Iron Ores

In the study of the Birmingham iron ores to find their amenability to concentration, 200 surface and underground samples were taken.

Laboratory concentration tests have been made on many of the 200 samples, and the conclusion was reached that with an effective flow sheet and good plant operation any of the ores containing 25 percent or more iron can be made to yield a satisfactory concentrate. This would make the resources almost inexhaustible, whereas without milling for beneficiation some of the companies will soon be short of commercial-grade ore.

Selection and Utilization of Silica Sands of Washington

One-third of the market for silica sands in the Pacific Northwest normally has been supplied by shipments from California, Illinois, and Belgium, with resultant higher cost to users than that of local materials. Because of this situation, an investigation of the properties of deposits of sands, sandstones, and quartzites in the State of Washington was undertaken at the Northwest Experiment Station in cooperation with the College of Mines, University of Washington. The results are given in a publication now in press.

A detailed investigation of all the sands of the State was not made, but only of the best indications known. Figure 6 not only shows the location of the sampled deposits, but also pyrometric cone equivalent (P.C.E.) values or fusion temperatures, which indicate the approximate silica content and, in general, give a clue to the kind of metal that may be cast in a mold from a given sand.

Sand-blast tests of several sands chosen to represent different types of sands and sandstone found in the State showed that they were equal to or better than California sands, although none was as good as sand from Ottawa, Ill.

CLAYS

Classification by Uses

An outline for qualitative testing of clays was prepared. The syllabus is based upon a careful survey and evaluation of various tests in use

Figure 6.—Location of sampled sand deposits, State of Washington, and their P.C.E. values. (Numbers correspond to county sample numbers in the text of University of Washington Bull. 108.)
and development of certain of them. The classification reached is based upon
the indicated uses of the clays, both ceramic and nonceramic, rather than on
the mineral or geological type of the clay. The whole series of tests is so
designed that they may be carried out by a relatively inexperienced operator
using rather simple equipment and yet obtain reasonably accurate results.

A test developed for this outline involved a rather extensive study of the
various types of clay minerals insofar as their dehydation and rehydration
characteristics are concerned. Previous studies on dehydration have shown
some overlapping, but with the results obtained by rehydration, separations
of certain minerals, notably halloysite, are readily possible.28/

A study of the characteristic properties of the Georgia hard and soft
kaolins showed the hard type to be finer-grained and somewhat higher in
silica, iron, and titanium. It was found possible to alter the hard clays con-
siderably by fine grinding29/ or micronizing. For certain uses, this is im-
portant commercially.

To relieve the bauxite shortage, it would be highly desirable to obtain
alums and aluminum from clays at a reasonable cost. The usual method of
attack is by acid extraction. Different clays have been found to react dif-
ferently under the same treatment; hence, an investigation of the reactions
of the various types of clay minerals was carried out. It was found that the
usual commercial type of clay, which contains the mineral kaolinite as its
basic ingredient, must first be heated to 500° to 800° C. before it yields
readily to acid extraction. Certain comparatively rare types, such as
halloysite and allophane, need no precalcination. In general, clays suitable
for this work must be very low in iron and contain little or no potash and
phosphorus.30/

**White Clays**

As part of its cooperation with the T.V.A. on American white clays, the
Electrotechnical Laboratory continued its study of the ball and china clays
from the East and the Pacific Northwest and the white montmorillonites from
Texas and California. As long as the shipping lanes to England are open, a
shortage of English china and ball clay will not develop, but it was well to be
prepared for an emergency. The variety of the many domestic deposits, some
but recently discovered, presents a problem of considerable magnitude,
particularly inasmuch as accurate standards for clay testing have not been
developed.

28/ Berkelhamer, Louis A., A Rehydration Study of Clays. (Approved for pub-
lication.)
(Final report in preparation.)
30/ Mason, C. Morgan, Suitability of Clays for Alum and Aluminum Manufacture.
(Report in preparation.)
A report was made at the April 1942 meeting of the American Ceramic Society on “Testing Whiteware Clays.” Of all the physical tests made to indicate manufacturing behavior after the product classification has been made by fired color, the two most important are bonding strength and shrinkage. Into this problem enter others, including methods of preparation, adjustment of the tempering water, accurate methods of measuring shrinkage, determination of pore space, and correlation of these factors. Classification of the clays in the fired state differs from that in the plastic and dry states because of the loss of organic and volatile plasticizers and the importance of nonplastic fluxing impurities, which act as bonding agents at high temperatures. Correlative tests of standard samples at different laboratories are needed.

Classification charts have been made of 35 different clays for both the dry and fired conditions, using strength versus pore space. Pore space, which varies inversely to shrinkage, has given better correlation values than shrinkage measurements. Figure 7 shows the individuality of clays in the dry state and the great difference in strength between the weak kaolins, the strong ball clays, and the powerful montmorillonites and bentonites.

Electrokinetic Treatment of Clay

The general background and program for investigating electrical methods of beneficiating and dewatering clay were described in the 1941 annual report. The paper on application to a Tennessee ball clay, which was discussed in that report, has been published. The program was continued, and a laboratory study was completed on the electrodeposition of high-alumina clays from Georgia and South Carolina. The results were presented for publication.

Experiments have also been started to apply this process to the rapid casting (electroforming) of ceramic shapes for firing. It has been found that bodies with a wall thickness of 1/3 inch can be formed in less than 1 minute from potters’ slip, but uneven shrinkage and sticking to the metal mold are difficulties that remain to be overcome.

Preliminary work was done on the microscopic measurement of the electrophoretic mobility (standard velocity) of clay particles in dilute aqueous suspension and placed in an electric field. By varying the kind and amount of dispersant it was hoped to detect favorable efforts that could be applied to

---

33/ Moyer, L. S., Part-time work and confidential reports, Univ. of Minnesota, September 1941 to April 1942.
Figure 7.— Correlation between strength and pore space of whiteware clays in the dry condition, including kaolins, ball clays, and montmorillonite.

Figure 8.— Relationship between the soluble sulfate content of two clay mixtures and repeated filter pressing.
improve the control of the electrodeposition of clays and other minerals. The results showed that the Abramson type of all-glass microelectrophoresis cell afforded reproducible measurements on clay particles. The most satisfactory way to prepare the suspensions for study was by the addition of 0.005 percent clay to the ultrafiltrate obtained from more concentrated suspensions, which had been either filtered under pressure on a ceramic filter or else centrifuged.

In connection with this investigation, a survey of the literature and mathematics of the available microelectrophoresis cells was compiled and presented for publication.34/

**Soluble Sulfate Content of Pottery Bodies**

A method of analysis was developed that gave consistent results for soluble sulfate and total soluble salts in ceramic raw materials and bodies. Values for sulfate upon a dry-weight basis ranged from 0.01 to 0.28 percent with ball clays, from 0.002 to 0.015 percent with china clays, and from 0.000 to 0.007 percent with miscellaneous raw materials. Bentonites were relatively high in sulfate, a Wyoming bentonite showing the highest value - 0.194 percent.

The distribution of sulfate in the slip, cake, and press water was determined for 18 ceramic bodies. An increase in the water:clay ratio in slips was found to increase the recoverable soluble sulfate. In experiments on repeated conversion of cake to slip and then filter-pressing, the release of soluble sulfate continued, but the bulk of the sulfate was obtained in the first pressing, as shown in figure 8. Quantitative results were determined on the effectiveness of barium carbonate as a precipitating reagent for sulfate in slips.

Aging from 6 months to 1 year had little effect on the soluble sulfate in stored samples of slips, cakes, and press waters. However, aging for 6 months doubled the soluble sulfate content of a freshly mined ball clay, evidently through oxidation of sulfur compounds. A report on this work was presented at the Cincinnati meeting of the American Ceramic Society, and the manuscript has been completed for publication. This study is of scientific and practical interest to all engaged in casting chemical porcelain, electrical insulators, and general chinaware.

Pozzolanic Minerals

Systematic studies of the chemical and physical properties of pozzolanas (which are siliceous minerals, such as volcanic ash, diatomite, burnt clay or shale, etc., entering as ingredients in concrete and mortar) were continued. The alumina in these minerals may unite with lime to form hydrated calcium aluminates. The experimental procedure consisted mainly in preparing calcium aluminates by precipitation and rotating these in a water bath at 30° C. to equilibrium in contact with lime-water solutions. X-ray diffraction patterns were taken of the residues, and their refractive index was determined. A dicalcium aluminate, 2CaO.Al₂O₃.8H₂O, was found dissolving without decomposing in water to give the following concentration: 8.02 millimoles CaO/1. and 4.00 millimoles Al₂O₃/1. A tricalcium aluminate, 3CaO.Al₂O₃, with about 12H₂O, also was found, giving a characteristically different x-ray pattern. It dissolves without decomposition in water. Both the di- and tri-calcium aluminate formed solid solution with Ca(OH)₂, the former to a ratio of CaO:Al₂O₃ of 2.2 and the latter to about 3.5. The di- and tri-calcium aluminate solid solutions exist in isothermal reversible equilibrium in contact with the following solution: 10.9 ± 0.7 millimoles CaO/1. and 1.20 ± 0.12 millimoles Al₂O₃/1. There was evidence, also, of a β-tricalcium aluminate with characteristic higher ratios of CaO:Al₂O₃ approaching 4. These results were supplemented by observations on complex carbonates and silicates of calcium aluminate.

The tricalcium aluminate solid solutions reverted readily to the stable isometric 3CaO.Al₂O₃.6H₂O. However, dicalcium aluminate was apparently stable, except in contact with lime-water solutions above the invariant concentration.

Several difficulties were experienced in attempting to establish the solubility curve for the isometric compound. A series of solubilities was finally obtained for lime-water concentrations from nil to saturation.

The plasticizing action of pozzolanic minerals is generally recognized. Comparative measurements were made for a series of pozzolanic minerals added to mortar. Plasticity was measured upon the basis of a stress-strain relationship, leading to determination of a coefficient of plasticity, ϕ, and a yield value, ϕ₀, analogous to that for metals. Workability is a more complex property than plasticity and consists of at least four factors. Plasticity is one of these and concerns ease of deformation. The other factors are water retention, stability of the thick suspension as regards rupture,

Figure 9. Globar-heated kiln for firing kyanite, topaz, and forsterite firebrick to 1,500°C. Elements surround the 12-by-12-inch setting space, giving uniformity of firing. The hinged door swings open for loading.
Figure 10.— Typical radio insulators fired in electric, twin-tunnel kiln at Norris, Tenn., in cooperation with the American Lava Corporation.
and adaptability of the suspension to extensive deformation. Particularly noteworthy was the plasticizing action of bentonite, ball clay, and powdered diatomite. Coefficient of plasticity was a good measure of over-all workability and, in line with this, correlated well with the directly measured water retention. The latter appeared to be relatively low for masonry mortars as against those with mineral powder additions.

REFRACTORIES

Electric Firing and Testing

A furnace was needed at the Electrotechnical Laboratory for firing full-sized brick samples made of kyanite, mullite, topaz, and forsterite. Figure 9 shows the furnace constructed by the laboratory staff. It has a capacity of eight bricks and has been fired 29 times to 1,500° and 1,800° C. without renewal of the 16 vertical silicon carbide heating elements arranged around the four walls of the heating chamber. A 24-hour firing, including 4 hours' soak at 1,500° C., requires a maximum input of 13 kw. and an average of only 7 kw. The kiln is easily loaded and unloaded by the hinged wall-door, operates without noise or trouble, and shows a low heat loss at the highest temperatures.

An electrically heated load-test furnace has been developed, using an Arsem-type, spiral, graphite heating element. The novelty of the design adopted lies in the method of protection of the heating element against atmospheric oxidation. No vacuum or special gas atmosphere is used. Instead, easily replaceable graphite plates and cylinders enclose the heating element in such a way that oxygen must contact and react with a cheap and easily replaceable protective graphite part before reaching the element. Three furnaces have been built and have been in regular use for over a year in heat-treating and load-testing samples at temperatures between 1,500° and 1,900° C.

Electric Firing of Radio Insulators

Over 7-1/2 million bisque and 150 thousand glost insulators were fired in an investigation conducted upon a cooperative basis with the American Lava Corporation during the 6-month period while the corporation was enlarging its own kiln capacity. The type of ware fired is shown in figure 10. Two standard cycles were used: 30 hours at cone 04 (1,822° F.) for glazing and 23 hours at cone 14 (2,534° F.) for bisque. The Corporation reported that the quality of the electrically fired ware was equal to its usual oil-fired product and better for certain special bodies and glazes because of cleaner oxidizing conditions. The cost of electricity and heating elements for firing and glazed ware about equaled the cost in the corporation's periodic kilns,

although the full setting capacity of the Norris kiln could not be used because of lack of special kiln furniture and the large variety of sizes and shapes. Many of the insulators fired in the Norris kiln undoubtedly are giving service in war tanks and planes from England to Australia.

**India Kyanite**

The difficulty in obtaining India kyanite from Calcutta started serious testing of American kyanite, topaz, andalusite, diasporé, bauxite, and other high-aluminous minerals that possibly could replace this important foreign refractory material. First, a study was made of the India kyanite, its method of manufacture, and the quality of its products. Comparative tests were made of all known domestic substitutes.

The South Carolina topaz is the immediate logical replacement mineral. Its volume expansion on heating is less than that of kyanite, but its porosity is greater owing to the evolution of water and fluorine with silica. The effect of these differences between topaz and India kyanite is still being investigated for the Miscellaneous Minerals Section of the War Production Board.

Preliminary tests were made on the absorption of waste gaseous fluorine compounds from topaz produced by the calcining of topaz to mullite. The amount of combined fluorine emitted per 24 hours on the proposed commercial scale will be about 10 tons, and this material will be very dangerous in liquid and gaseous form. These tests have not yet been completed, but indications are that the fluorine compounds can be converted into insoluble lime salts for convenient disposal. The possibility of recovering fluorine in the form of byproduct sodium or aluminum compounds for marketing has also been considered.

The 19 million tons of low-grade Southern bauxite represents a potential source of excellent refractory material, which is now being used in comparatively small amounts for electrocast refractories and corundum abrasives. Experiments have been made to reduce the cost of this excellent grog or refractory skeleton material by rapid melting and water quenching instead of slow cooling in large masses. This method not only forms a possible substitute for the India kyanite in the present emergency, but also is an important outlet for a block of Southern power after the war. The iron impurities in the bauxite are reduced to the metallic state and removed as ferrosilicon, and the freed alumina while molten is mixed with silica to form mullite. This artificial mineral is the same high-temperature product obtained by heating India kyanite, except that the electric-furnace mullite is denser, more rigid, and most resistant. Spalling resistance can be produced by inserting the expanding, porous, calcined domestic kyanite fines between the dense, electromelted, mullite grog grains. The variety of domestic materials
possessing a wide range of properties, combined with the use of the superior electric-furnace temperatures, makes it possible to produce mullite refractories for a versatile range of service. The natural massive kyanites are extremely rare, and ultimately, in normal circumstances, we shall be forced to use the electric-furnace product for these superior refractories.

Three progress reports describing this work have been prepared; the first was included in a Bureau of Mines report to the Advisory Committee on Metals and Minerals, National Academy of Sciences, and National Research Council; the second to the American Institute of Mining and Metallurgical Engineers at the New York meeting; and the third to the American Ceramic Society at the April meeting.

**High-Alumina Cement**

The investigation of aluminous cement was initiated as a means of developing a use for low-grade bauxite not suitable for aluminum production. Further impetus was given by the news that Greek bauxite, as a raw material for domestic, quick-setting cement, had been cut off by the conquest of Greece.

Preliminary work has been started to determine the functions of the various mineral constituents of the calcium aluminate cements, including 2CaO.Al₂O₃·SiO₂, 3CaO·5Al₂O₃, CaO·Al₂O₃, 5CaO·3Al₂O₃, and 3CaO·Al₂O₃. Also studied were the effects of their formation methods, such as arc-furnace melting and crucible melting under oxidizing and reducing atmospheres and by sintering below the melting temperatures. Standard and special cement tests for strength and time of set were made.

**Basic Refractory Brick from Washington Olivine**

Refractory brick made from olivine (dunite) from the Twin Sisters Mountains in Washington gave satisfactory service in a test course in the throat of a basic open-hearth steel furnace in place of chromite brick. These brick were made at the Northwest Experiment Station of the Bureau of Mines, where work is conducted in cooperation with the College of Mines, University of Washington. Previously a simulative-service slag test using basic open-hearth steel slag, made to determine the relative resistance of various basic refractories to chemical attack and mechanical erosion, indicated that brick made of 80 percent dunite, 10 percent dead-burned magnesite, and 10 percent chromite were equal to or better than 5 different industrial basic refractories. Although brick sawed from Twin Sisters Mountains (Wash.) dunite tended to become porous upon heating, they had the best slag resistance of any of the brick tested. However, it is doubtful whether sawed brick would be economical. Because dunite is so hard, a diamond saw was found to be the only satisfactory cutting device. Two plastic mixtures were also tried in the slag test - one composed of 80 dunite and 20 chromite and the other of 80 dunite and 20 dead-burned magnesite. These components were mixed with a calcium-ligno-sulfonate binder and applied as plastic patches 1 inch thick.
on the walls of the slag-test furnace. The mixture of 80 dunite and 20 magnesia appeared to withstand the slag attack better than the dunite-chromite mixture.

Petrographic studies of uncalcined olivine from the Twin Sisters Mountains area indicated that possibly olivine and enstatite crystallized together with small chromite crystals. Other samples from the same area were microbrecciated and contained other minerals, suggesting other possible crystallization orders. Calcination caused mineralogical changes and a slight lowering of the index of refraction, but no lowering of fayalite content was indicated. Thin-section studies showed that the pores formed upon heating sawed dunite brick probably were caused by mineralogical changes occurring upon heating enstatite above cone 18 (1,485°C.).

THERMAL STUDIES

Magnesia-Silica System

The properties of forsterite derived from North Carolina olivine by electric melting have been made the subject of investigation33 because of the need of a basic refractory for highest-temperature service in the eastern United States as a replacement for magnesite and chromites. Magnesite is now shipped from the Pacific coast to the points of consumption in the industrial East, involving a long, costly freight haul. Under present conditions of overloaded transportation facilities, the elimination of long shipments is particularly attractive. The Pacific Northwest is likewise in danger of destructive raids.

Tremendous quantities of forsterite olivine occur in dunite formations in the southern Appalachian range in North Carolina, conveniently located between the Birmingham and Pittsburgh steel centers. Combined with low electric-power rates provided by water-power development in the Tennessee Valley, these deposits are well situated for electric furnace production of forsterite.

Although the second part of the investigation has been seriously interrupted by war projects and the erection of a new electric-furnace laboratory, significant data have been obtained and are being prepared as a second manuscript intended for publication in the Transactions of the Electrochemical Society. A melting furnace in the new laboratory, shown in figure 11, will produce two-ton pigs of refractories.

Figure 11.— Water-cooled arc furnace used for preparing artificial mullite and calcium aluminate. Water sprays protect the sides and bottom by maintaining a blanket of unfused charge between the shell and the melt. Capacity, 2 tons.
Figure 12.— Electrocasting molten forsterite at approximately 2,000° C.
Figure 13.— Thermal-analysis curves of typical aluminous minerals.
Formation of shapes by electrocasting molten forsterite into molds has been investigated and shown to be possible, particularly in large shapes, wherein annealing was less troublesome. Casting into a brick mold is shown in figure 12. Small bricks tended to crack, owing to thermal shock. Molds of steel, cast iron, silicon carbide, graphite, or other inert materials were required, as silica sand reacted violently with the forsterite. Electrocast bricks withstood high-temperature load testing up to the melting point, and in several instances a small remaining neck supported the entire load (25 pounds per square inch) originally applied to the full cross section. Specimens containing excess free periclase supported 25 psi. at temperatures above 1,800° C. The electrocast forsterite specimens were equal or superior to commercial magnesite, chrome, and pressed-forsterite specimens in resistance to basic open-hearth slag attack.

Service tests of forsterite bricks in a copper sulfide reverberatory furnace indicated better resistance to slagging than the silica brick currently being used. The bricks used for this panel contained 5 percent calcium aluminate cement (lumnite) and 5 percent calcined magnesite. Larger amounts of the cement seriously reduced the refractoriness of the forsterite.

Identification of Alumina-Bearing Minerals

The principle of the method for identifying alumina-bearing minerals by thermal analysis and a view of the apparatus were given in the 1941 report. Thermal-reaction effects of individual minerals in a sample are registered photographically on a moving chart by means of a differential-thermocouple arrangement and accessory electrical instruments. The kind and percentage of each constituent are determined from the positions of the irregularities and measurements of their magnitude. Some typical heating curves showing characteristic peaks are shown in figure 13.

During the past year attention has been concentrated on applying thermal analysis to ores and possible ores of aluminum (including bauxites and beneficiation products of low-grade bauxites, in cooperation with the program of the Tuscaloosa Station) and to high-alumina clays. Over 150 runs were made on standard minerals, and over 80 runs were made on samples of bauxite, clay, etc. A paper on the results is in preparation.

INTERCRYSTALLINE CRACKING OF BOILER STEELS

Intercrystalline or embrittlement cracking of boiler steel is caused by the action of water containing small amounts of alkalies as it seeps into and through boiler joints and seams. The attack produces a peculiar network of hairline cracks around the crystal grains of the steel, which leads to eventual failure of the joint, possible explosion of the boiler, and attendant
serious damage to adjacent property. Costly repairs or a new boiler may be needed to replace one that has suffered embrittlement cracking, and in any event production of steam will be seriously interrupted.

The embrittlement detector described in two United States Patents\textsuperscript{39} is therefore especially significant to avert such unexpected shut-downs during this period of war. The detector is a test unit that can be permanently attached to a boiler, and it exposes a stressed specimen to the boiler water in exactly the same manner as a riveted seam is exposed under operating conditions. If the specimen cracks, it is a danger sign that the action of the alkalies on the boiler metal is not inhibited and that the boiler may be in danger of cracking. The unit was developed at the Bureau of Mines in cooperation with the Joint Research Committee on Boiler Feedwater Studies.\textsuperscript{40}

A comprehensive bulletin\textsuperscript{41} has been published that traces the development of a basic understanding of the elements that combine to produce cracking. Recognition of the factors involved in the cracking process brought about an evolution of test methods and equipment that permitted thorough investigation of protective compounds under conditions closely related to boiler operation. Tannin substances, particularly those derived from the South American quebracho tree, have successfully inhibited cracking at steam pressures up to 700 pounds per square inch. In addition, they seem to retard corrosion by removing oxygen dissolved in the water and preventing the formation of hard scale by hindering crystal growth. The nitrates are even more certain preventives of embrittlement, and their inorganic nature, definite chemical composition, and stability at boiler temperatures give them advantages over the tannins for some uses. The bulletin gives directions for use of the embrittlement detector and methods of analysis for alkali, nitrate, and tannin. Determination of the latter substances is treated more fully in another publication.\textsuperscript{41}


\textsuperscript{40} Schroeder, W. C., and Berk, A. A., Intercrystalline Cracking of Boiler Steel and Its Prevention: Bureau of Mines Bull. 443, 1941, 85 pp.

The most direct method of treating boiler water to eliminate cracking would be to remove the alkali, which can concentrate to cause the trouble. On the other hand, the pH of the boiler water must not be allowed to fall so low that corrosion and pitting will occur. Sodium phosphate can be used to maintain the pH without introducing caustic soda or permitting it to develop in the boiler water. 42/ When boilers using evaporated make-up are treated with phosphate in the absence of free caustic, total solids in the water are reduced considerably, and carry-over of such solids to interfere with turbine operation may be decreased.

A recent symposium on caustic embrittlement 43/ showed surprising unanimity of opinion concerning methods that are satisfactory for preventing cracking. Data from laboratory research and field study showed that nitrates and quebracho tannin have satisfactorily inhibited cracking in a considerable number of installations. A sharp reduction in the cracking of locomotive boilers following the use of protective agents based upon embrittlement-detector tests affords strong, practical proof of their validity. The more direct procedure of dispensing with inhibitors and substituting phosphate for alkali that may concentrate to attack steel offers a solution for the embrittlement problem at higher pressures. A suitable method to prevent embrittlement can now be designated for almost every boiler water and every operating pressure at which cracking has occurred.

COAL INVESTIGATIONS

Utilization of Coal Refuse

Accelerated Attrition Tests

Carbonaceous zeolites used in water softening are slowly worn down and broken by the motion of the particles in the bed. This motion is caused by the large currents of water passing through the bed and is especially pronounced during the back-washing and reclassification of the column. A zeolite that is resistant to such abrasion would therefore be more desirable than one that is friable, all other factors being equal. Consequently, there has been a demand for a test that will distinguish quickly between zeolites having different degrees of resistance to such attrition. A study was made of the attrition loss under various test conditions, and a satisfactory accelerated test was devised. This test will differentiate well between zeolites and shows the relative degree of abrasion resistance from the softest to the hardest type of zeolite. The test is based upon agitating weighed quantities of the zeolites with a measured quantity of water in a glass container for a definite period.

A reciprocating machine was designed that insures that each sample is agitated under the same conditions. The results of the attrition are measured in two ways: First, the liquid suspension passing 200-mesh is measured for turbidity, and second, the zeolite particles are carefully screened-sized into several fractions. The relative surface areas of the sized fractions are calculated, and a figure called the attrition value is derived. It was found that turbidity values measure the condition of the outer surface of the particles, while the attrition value measured the friability of the deeper layers of the zeolite. The attrition value was found to vary greatly with the types of acid treatment used in making a zeolite from coal. On the other hand, when several types of coal were changed into zeolite by a standard acid treatment it was found that the attrition loss did vary consistently with the physical and chemical properties of the different coals.

Activated Carbon for Sugar Decolorization

The utilization of coal and coal refuse as a raw material for the production of activated carbon was studied. The carbon was activated for the specific purpose of decolorizing raw-sugar solutions. First, the coal was given a low-temperature carbonization at 500°C in a covered iron retort, which allowed the free escape of volatile matter and minimized oxidation of the contents. This preliminary heating drove off large quantities of smoke, tar, and oils that otherwise would have fouled the discharge end of the activating apparatus. This treatment decomposes the complex carbon compounds, drives off the volatile hydrocarbons, and leaves a residue of char consisting largely of carbon. The carbon is considered to have been very active at the time of its formation, but as it was surrounded by volatile decomposition products it instantly adsorbed a surface coating of these materials and became so saturated that little adsorptive power remained. The surface needed therefore to be cleared to restore the original activity. The cokelike residue was then ground to a very fine powder in a ball mill, and the powdered coke was charged into a reaction tube, where it was heated to 900°C in a moving current of steam. This cleared the surfaces of the particles and partly restored their original activity. Activation with steam is a sort of differential oxidation in which the adsorbed constituents burn readily while the carbon burns with difficulty and leaves a clean carbon surface. Further oxidation of the clean carbon is believed to open and enlarge small pores and capillaries in the coal, greatly increasing the total surface.

One interesting point brought out by the investigation was that the difference between a water-purification and a sugar-decolorizing carbon was due to a greater degree of burning in the latter instance. This was attributed to the production of capillaries of larger diameter in the sugar-decolorizing carbon. The color bodies in sugar undoubtedly are much larger than the odor-producing bodies in water and so cannot get into small capillaries as easily.

After steam activation, the decolorizing power of the carbon was increased further by an acid treatment that probably cleaned off soluble salts and removed much of the ash, increasing the relative amount of pure carbon.

The data obtained as a result of these experiments show that there is an optimum degree of burning for each sample tested, any additional burning decreasing the activity; also, the most active carbons were obtained from the raw materials having the lowest ash content.  

Water Softening with Nonmetallic Minerals

Many nonmetallic minerals are used in water softening. Some have been employed for years, and their use and behavior are common knowledge to the industry. They include such products as alum, lime, soda ash, iron salts, and zeolites.

Clays - particularly the bentonitic types, which possess the property of swelling and forming a gelatinous mass - are finding application in water softening. They are good coagulants, removing suspended matter, some dissolved solids, and most of the bacteria.

One of the important advantages of using clays in the softening of water is the saving of more expensive materials that would otherwise be required. By using clays in softening water, less iron and aluminum salts are required for flocculation of suspended matter and less activated carbon for the removal of color.

The use of sodium hexametaphosphate in conditioning domestic and industrial waters has reduced the damage and expense caused by hard water. A small amount of this chemical compound, which has been known for years but only recently was applied to water conditioning, will render a hard water useful for domestic laundry purposes. The addition of sodium hexametaphosphate to boiler feeds will decrease, if not prevent entirely, corrosion in high-pressure boilers.

Pulverized Coal and Its Preparation

Many years have been spent in research on grinding, but the results either have been expressed in terms of gross power consumed, and consequently are influenced by the unknown efficiency of the machinery, or have been expressed as an abstract "grindability index." Such data, for the most part, have fallen short of the ultimate goal - that is, the determination of the net energy consumed in grinding a substance to a specified size.

Frequently in those grinding tests only the most easily ground portion of a certain size of the feed material was ground, and to a size that may have been much different from that desired in a commercial operation. Then, too, the method of grinding in some of the tests did not even remotely simulate commercial practice.

At the Southern Experiment Station a dry pulverizer has been developed that will reduce 3/4-inch material to flotation size or boiler fire-box size. Subsieve sizes may be attained if desired. The method of grinding simulated modern commercial practice in that continuous grinding in closed circuit is accomplished by means of air separation and Cottrell precipitator. This system causes a balanced circulating load to develop, without which accurate power measurement is impossible, particularly on heterogeneous materials. A force recorder that is an integral part of the machine makes possible measurement of the net energy consumed. No friction-loss corrections are involved in the power measurement. A detailed description of the machine has been prepared, and recent work with the pulverizer has even demonstrated that the modifying word "coal" well could have been omitted from the title of the paper, because the operation of the machine has been most gratifying in the grinding of cement clinker, cannel coals, graphite, kaolin, limestone, mica, oil shale, talc, and topaz.

The ultimate degree of fineness to which the machine is capable of grinding has not been determined, but a relatively coarse muscovite mica has been ground in the pulverizer to a mean particle size of 2.5 microns. This was somewhat finer than the product produced from the same feed in a commercial "micronizer."

It is believed that this energy-measuring pulverizer will point the way to new economies in grinding in the range from about 10 mesh down to and including the "superfine" sizes.

The pulverizer is shown in figure 14.

Combustion of Coal

During the past year a study was made of the burning characteristics of eight Washington coals when fired on a domestic ash-remover-type underfeed stoker installed in a standard hot-water boiler comprised of six vertical cast-iron sections. Twenty-one heat-balance trials were made to determine the effect of variations in ash-softening temperatures and in caking properties on the suitability of these coals as fuels for the ash-remover-type stoker.  

Figure 14.— Laboratory grinder with force recorder for continuous measurement of net power.
Figure 15.— Ash-remover-type underfeed stoker, hot-water furnace, and equipment used in coal-burning trials.
The results of the trials showed that certain coals previously found to be unsuitable for burning on the standard underfeed stoker because of their high ash-softening temperatures burned efficiently and without mechanical difficulty on the ash-remover-type stoker. These coals were characterized by their ash-softening temperatures and absence of agglutination or caking. Those giving satisfactory results had ash-softening temperatures above 2,600° F. and no caking properties. Clinker from coals with ash-softening temperatures below 2,600° F. disrupted the fuel bed while being pushed from the retort by the scraper arms of the ash remover and in addition caused excessive grinding noise in the ash-remover mechanism. Coals with agglutinating values even as low as 100 grams breaking strength (Marshall-Bird method) could not be burned efficiently because of a high loss of unburned carbon in the ashes. Figure 15 shows the stoker, hot-water furnace, and other equipment used in the coal-burning trials.

**Properties and Uses of Washington Coals**

Coke of low phosphorus content is required in the electrometallurgical industries recently attracted to the Pacific Northwest by its supply of cheap hydroelectric power. Because little was known about the occurrence of phosphorus in Washington coals, an investigation of its occurrence and its elimination in coal preparation was undertaken jointly by the Northwest Experiment Station and the College of Mines of the University of Washington. The work dealt chiefly with the coals of the Wilkeson-Carbonado and Roslyn-Cle Elum fields, for they are likely to be used in making metallurgical coke.

All of the coals studied contain, in the as-mined condition, more phosphorus than can be tolerated for specific uses. Moreover, in many of the beds the phosphorus is distributed throughout the coal substance itself and therefore cannot be eliminated by ordinary coal-washing methods. In the No. 2 and 3 beds of the Wilkeson-Carbonado field, however, the phosphorus is concentrated largely in the heavy impurities of the bed that can be removed by coal-washing methods. Large-scale washing tests conducted in the laboratory demonstrated that the phosphorus content of these coals can be reduced to as low as 0.04 percent - well within the limit specified by all but the most discriminating of the electrometallurgical industries.

**Byproduct Coke for War**

To explore the possibilities of reviving the coking industry in the Pacific Northwest to supply the growing needs of metallurgical industries engaged on war contracts, the Northwest Experiment Station, in cooperation with the College of Mines, University of Washington, and a group of coal-mine operators, conducted a series of coal-washing and coking trials of coals from Pierce, King, and Kittitas Counties, Wash. The principal matters requiring
study, aside from economics, were the physical and chemical properties of the cokes produced from Washington coals after carbonization under industrial conditions.

After preliminary box tests had been completed, arrangements were made to use the coke plant of the Crow's Nest Pass Coal Co. at Michel, British Columbia, for full-scale oven trials. The ovens were charged with coals straight, blended with each other, and blended with petroleum carbon, the last to give lower ash and phosphorus for special ferroalloy uses.

All the coals and blends yielded commercial coke, but shatter and tumbler tests as well as chemical analysis showed that some cokes were superior to others in certain respects. Based on this work, a new coke plant is being constructed in Tacoma, Wash., and a new mine and washery to supply it is under development at Wilkeson, Wash. Details of the investigation will be discussed in a forthcoming Report of Investigations.

SUMMARY

Almost all of the work of the Nonmetals Division during the year under review has been directed to supplementing and replacing the nonmetallic minerals of the Nation at war, which formerly were imported but which now must be supplied from domestic sources. Of these, the following are especially important:

Alumina. - Study of preparation of alumina for both nonmetallic and metallurgical uses was initiated. One foreign process offered was tested and found unsuitable. A variation of the lime-soda sintering and leaching process for siliceous bauxite and clay was tested mainly on bauxite and found to be quite suitable. Certain improved steps were proved in the laboratory, and pilot-plant proof on larger masses is being organized for early execution. A small amount of work was done on sulfuric acid leaching of alumina from clays, dealing with a study of calcination of clay to make it most soluble and on purification of the aluminum sulfate solutions to remove iron.

Clay. - Clays for complete substitution for imported clays have been under investigation. Britain now wants to divert her clay miners to tin mining, which adds to the problems. However, suitable all-American ceramic bodies have been developed. Complete substitutes for Vallendar enamels' clay and Gross Almerode pot clay have not been worked out to date. Suitable clays for lead-pencil manufacture have been recommended.

Graphite. - The flake-graphite shortage was anticipated one year in advance, and study of the most suitable deposits to produce large flake was
initiated. By the time the need arrived, most of the work on improved grinding processes and flotation had been done. The work has continued in active cooperation with the War Production Board, which has recommended construction of three mills - two in Alabama and one in Pennsylvania - and the rehabilitation of one Alabama and one Texas mill. In all of these instances the Bureau of Mines is to keep in active touch with design, construction, and initial operation and to continue to work out minor improvements in beneficiation.

Talc. - Radio ceramic talc which must consist mostly of talc and contain not over 1 percent each of ferric oxide or lime, was formerly imported mainly from Manchuria, Italy, and France. A single United States mine in Death Valley, California, belonging to Sierra Talc Co., was known to have a reliable supply of suitable grade, but the mine was nearing exhaustion. Examination and sampling of all United States mines thought to have talc of suitable grade showed that a few smaller mines could produce high-grade talc but that most of them required beneficiation. Beneficiation tests by froth flotation were made on all promising samples, and many were found to be amenable. Results have been released to the War Production Board, and plants for mining and flotation have been recommended for Alabama, as well as possibly one in New York and one in California.

Refractories. - Topaz has been shown to be the best domestic substitute for India kyanite, and it is indicated that fused olivine, altered into pure forsterite, should be a superior substitute for some of the former uses of magnesite. Confirmatory service tests are under way.