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**CHEMICAL AND PHYSICAL STABILITY OF REFRACTORIES  
FOR USE IN COAL GASIFICATION**

**Third Quarterly Progress Report, November 1, 1976—January 31, 1977**

**February 15, 1977**

**Work Performed Under Contract No. EY-76-S-02-2904**

**Missouri University  
Rolla, Missouri**



**ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION**

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15 February 1977

Prepared for the Energy  
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96  
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## I. WORK COMPLETED

During this quarter, the Initial Sample Selection Test at 500 psia and 500°F and the 10-day exposure of the Second Sample Selection at 1000 psia and 1000°F were completed, see Flow Chart of Screening Exposure Tests in Fig. 1. The post-exposure characterization of specimens from these tests has been partially completed. Since this is the first report on the results of the exposure tests, a brief summary of the experimental procedures and analysis methods is described along with the results obtained.

## II. EXPERIMENTAL PROCEDURES

### A. Materials

Eleven calcium aluminate cement-bonded refractory castables designated as High Alumina, Intermediate Alumina, and Insulating; three dense high alumina phosphate-bonded ramming mixes; five calcium aluminate cements; and three Foam-in-Place insulating refractories are being investigated. The general compositions are given in Table I. The dense high alumina castables were selected to be representative of the spectrum of commercial products and for their different cement binder. The UMR specimens are being used to evaluate the relative chemical durability of the

different calcium aluminate cements used in commercial products and for comparison purposes.

#### B. Specimen Preparation

All cement bonded refractory castables, except Group 3, were cast as 12 x 3 x  $\frac{1}{2}$  in. slabs, cured for 24 hrs. in air at 100% humidity, and dried at 100°C for 24 hrs. These slabs were then cut into 3 x  $\frac{1}{2}$  x  $\frac{1}{2}$  in. specimens. Group 3 specimens were cast as 3 x  $\frac{3}{4}$  x  $\frac{3}{4}$  in. bars, cured for 24 hrs. as above, and dried at 100°C for 24 hrs.

Neat cement specimens (water/cement ratio of ~0.3) were cast as  $\frac{1}{2}$  in. cubes, cured in air at 100% humidity for 24 hrs., and dried at 100°C for 24 hrs.

The phosphate-bonded specimens were prepared by hand ramming the mix in 3 x  $\frac{3}{4}$  x  $\frac{3}{4}$  in. molds. Immediately after ramming, the specimens were transferred to an oven at 100°C and dried for 24 hrs. The specimens were then fired at 1000°F in air for 18 hrs.

After drying and/or prefiring, the specimens are labelled, their dimensions and weights recorded, and stored in an oven at 110°C until needed for the exposure tests.

#### C. Test System and Procedure

The exposure test system consists of two high pressure vessels 6" I.D. x 18" deep. One vessel constructed of 316 stainless steel is used as a steam generator, and the other constructed of 347 stainless steel is used for the test vessel. Resistance heating elements on the outer

surface of each vessel are used to heat the vessels and contents to the required temperatures.

At the beginning of a test, each vessel is purged with  $N_2$ , pressurized and checked for leaks. The nitrogen is then exhausted to the atmosphere and the vessels heated at a rate of about  $200^\circ F/hr.$  Each group of refractory specimens in the test vessel, are separated from each other by thin alumina plates. During the heating of the test vessel, the gas exhaust to the atmosphere is open so that the water of hydration from the refractory specimens can readily escape.

When the steam vessel has attained the desired steam pressure, the remaining gases are added from a premixed mixture such that the overall atmosphere has the calculated composition given in Fig. 1. When the test vessel has attained the desired temperature, it is pressurized with the steam/gas mixture from the steam vessel. The steam and gases from the test vessel flow through a condensor where the steam is condensed and the remaining gases exhausted to the atmosphere through a flowmeter. The flow rate was adjusted to  $\sim 4 \text{ l/hr. (STP)}$  which provided for one complete change in the atmosphere of the test vessel every 24 hrs.

At the completion of the exposure test, the gas flow into the test vessel was stopped and the vessel cooled to  $\sim 200^\circ F.$  The pressure is then reduced to atmospheric and the vessel is allowed to cool to room temperature. The specimens are then removed, checked visually for any surface

deterioration, dried in an oven at 110°C for 24 hrs., and their weights and dimensions recorded. Flexural strength is measured in three-point bending (ASTM C268-70) using a span of 2 in. and crosshead speed of 0.5 cm/min. Parts of the fractured specimens are used to measure the bulk density, % apparent porosity, X-ray and DTA/TGA analyses. Seven specimens were used in each group.

### III. RESULTS

#### A. Flexural Strength

The flexural strength (MOR) of the cement- and phosphate-bonded refractory specimens are given in Tables II and III, respectively. Unless indicated otherwise, seven specimens were measured for each refractory.

The MOR of the high alumina castables, Table II, exposed for 10 and 30 days at 500 psia and 500°F is higher than that of the as-fired at 500°F specimens. For reasons not known at this time, the 20 day specimens are lower in strength than the 10 or 30 day specimens. With the exception of Group 7, the MOR of the castables exposed for 10 days at 1000 psia and 1000°F are lower than that of the as-fired at 1000°F specimens.

The intermediate alumina and insulating castables (Table II) exposed to 500 psia at 500°F or 1000 psia at

1000°F do not show any large changes in MOR for either the 500 or 1000 psia exposure tests.

The MOR of the phosphate-bonded specimens (Table III) exposed to 500 psia at 500°F decreased for the first 10 days, but then increased for the 20 and 30-day exposures. The specimens exposed to 1000 psia at 1000°F for 10 days showed a slight decrease in strength. Except for Brikram 90-R, the phosphate-bonded specimens in general show no significant loss in strength.

#### B. Density and Porosity

Table IV summarizes the bulk density and apparent porosity of the cement-bonded castables. The density and porosity of specimens exposed to 500 psia at 500°F either show a slight increase or no change in density but a small increase in porosity. Specimens exposed to 1000 psia at 1000°F have lower density and higher porosity compared to those exposed to 500 psia at 500°F.

Table V gives the density and porosity for the phosphate-bonded specimens. No significant changes in porosity are observed. The density decreases slightly for the 10-day exposures at both 500 psia and 1000 psia.

#### C. Dimensional and Weight Changes

No dimensional changes of significance were observed in any of the materials tested at 500 or 1000 psia. In the high alumina castables, a contraction of less than 0.3%

was observed in some groups exposed for 20 or 30 days at 500 psia and 500°F and 10 days at 1000 psia and 1000°F. The intermediate alumina and insulating castable groups showed even smaller changes in dimensions. The dimensional change in the phosphate-bonded specimens was barely measureable.

Table VI summarizes the data on weight change in the as-fired and exposed cement-bonded castable specimens. The weight change in each group represents an average percent weight change with as-fired weight as the initial weight.

As evident from Table VI, specimen in most of the groups of cement-bonded castables show an average of 3% weight decrease at 500°F and over 4% weight decrease at 1000°F with specimens in Group II showing the smallest decrease at both temperatures. Most of the high alumina castable specimens exposed to 500 psia for 10 or 30 days show weight losses of less than 1%. Those exposed to 20 days, however, have larger losses. The intermediate and insulating castables, exposed to 500 psia show 1-2% decreases in weights with the 20 day specimens having the higher decreases. For the 10 day exposure at 1000 psia, different groups in the high alumina castables show varying amount of weight changes while the intermediate and insulating castable group show more consistent weight losses.

Table VII summarizes the weight change in the hydrated calcium aluminate cement cubes exposed for 20 days at 500 psia and 10 days at 1000 psia. The data represents an

average percent weight change with the as-dried weight as the initial weight. At 500 psia, the CA-25 specimens gained only 0.1% weight, while all other cements showed gains of over 1%. At 1000 psia, except for the Secar 250 specimens which gained 2.4%, the cement specimens had weight losses ranging 0.5-6.3%.

The Foam-in-Place insulating refractories, exposed to 500 psia at 500°F or 1000 psia at 1000°F, all showed weight changes of less than 1%.

The phosphate-bonded specimens, exposed to 500 psia or 1000 psia, barely showed any measureable change in weights.

#### D. Chemical Reaction

The examination of the exposed samples by x-ray diffraction and DTA/TGA is currently in progress. The major chemical changes detected to-date in the cement bonded castables is the appearance of boehmite ( $\text{Al}_2\text{O}_3 \cdot \text{H}_2\text{O}$ ) and calcite ( $\text{CaCO}_3$ ) in all samples exposed at 500 psia. Boehmite is absent in all the specimens tested at 1000 psia.

### IV. TENTATIVE CONCLUSIONS

(1) The MOR of the high alumina castables exposed to 1000 psia at 1000°F for 10 days is only about one half of that at 500 psia and 500°F.

(2) The MOR of the high alumina castables after exposure (ERDA atmosphere) at 500°F is higher than that of as-fired in air at 500°F.

(3) The MOR of the high alumina exposed castables at 1000°F is significantly lower than of the as-fired in air in 1000°F.

(4) The MOR of the intermediate alumina and insulating castables remain generally constant during exposures at either 1000 or 500 psia.

(5) The change in the MOR of phosphate-bonded specimens is much less than in the cement-bonded high alumina specimens. No significant change in the MOR of Green Pak 90-P and 90 Ram HS ramming mixes occurred during exposures at either 500 or 1000 psia.

(6) If all the refractory products used in this investigation are compared with one another, no significant difference between the commercial products of the intermediate alumina and insulating refractories is apparent. On the other hand, a significant difference in strength is observed for the various commercial dense high alumina and to some extent, for the phosphate-bonded high alumina products.

(7) From the results obtained so far, the ERDA atmosphere appears to be less corrosive than the 50% steam-50% CO atmosphere reported in literature.<sup>1,2</sup> Both Gac<sup>1</sup> and Wistrom<sup>2</sup> investigated dense and insulating castables exposed

to the saturated steam-CO atmosphere at  $\sim 200^{\circ}\text{C}$  for 18 days and reported significant weight and dimensional changes. Calcium formate,  $\text{Ca}(\text{HCO}_2)_2$ , was one of the major products of reaction besides boehmite and calcite.

In the present investigation no trace of calcium formate has been detected at 500 or 1000 psia. The intensity with which the boehmite phase is found in the three castables and the effect on the flexural strength seems to be related. The high alumina castable was found to be stronger at 500 psia where boehmite appears as one of the major phases, but weaker at 1000 psia where no boehmite was detected. Change in strength for the intermediate and insulating castables was much less at either 500 or 1000 psia and boehmite was present as a minor phase only in the castable exposed to 500 psia. Based on the reported<sup>3</sup> use of boehmite as a bonding phase in high alumina compositions and the strength data obtained so far in this investigation, it is tentatively concluded that the strength increase of the high alumina castables is due to the boehmite formed at 500 psia and  $500^{\circ}\text{F}$ .

#### V. FUTURE WORK

The x-ray diffraction and DTA/TGA analyses of the specimens will be continued. The 20 and 30 day exposures at 1000 psia and  $1000^{\circ}\text{F}$  will be completed and characterization of

these specimens will be undertaken. Based on the preliminary results of these exposure tests, a decision will soon be made as to continue the third Screening Tests which contains H<sub>2</sub>S. Because of the less corrosive nature of the ERDA atmosphere experienced so far, it may be deemed necessary to investigate atmospheres of different composition, particularly CO-steam ratio, for short time exposures.

## VI. PERSONNEL

During this quarter the following personnel worked on this project:

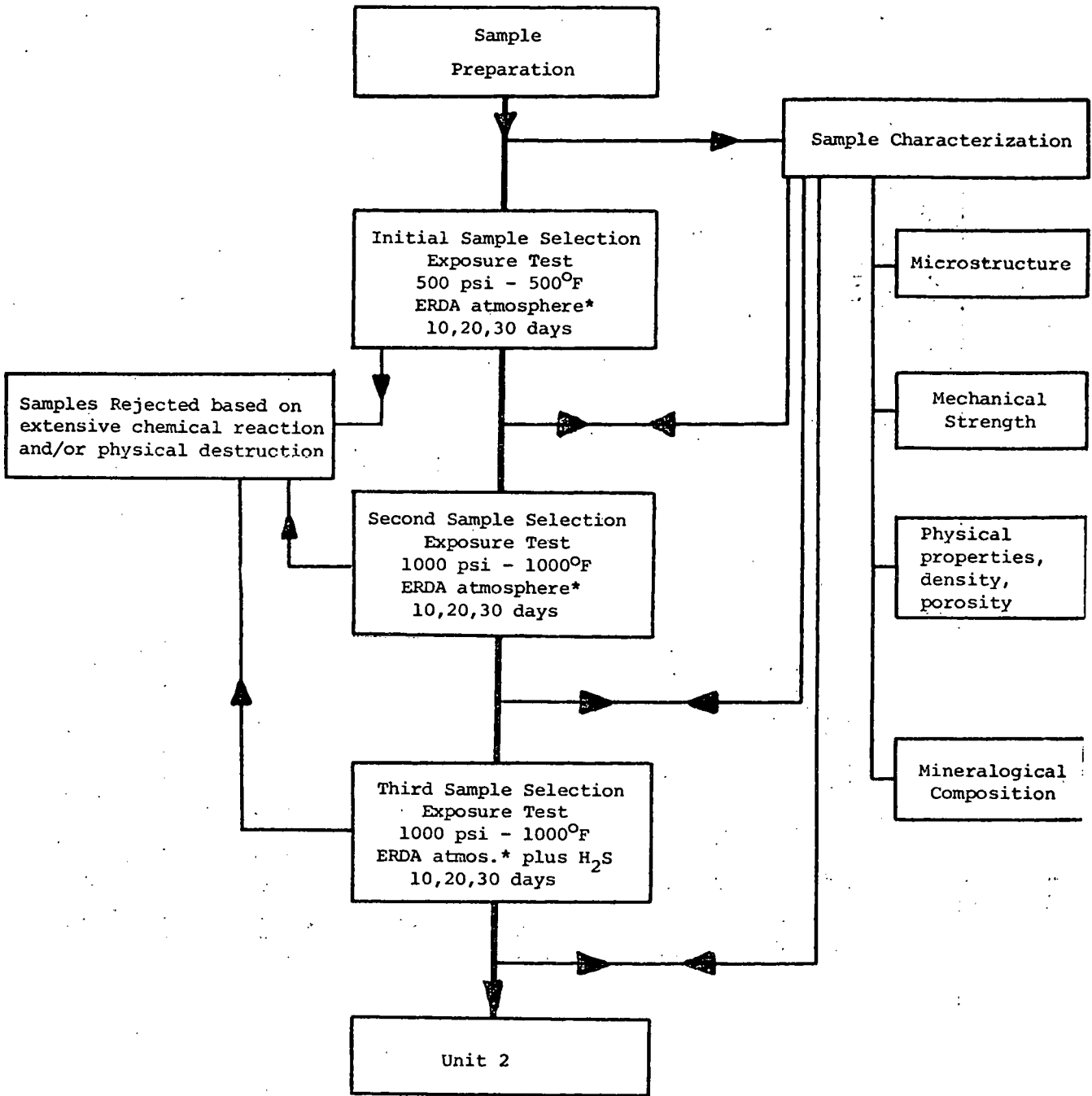
- (a) Delbert E. Day, Principal Investigator;
- (b) Gordon Lewis, Co-investigator;
- (c) Syed Rahman, Postdoctoral Fellow in Ceramic Engineering (full-time); and
- (d) Undergraduate Research Technicians (four).

## VII. REFERENCES

1. Frank Gac, "Stability of Refractory Castables to Steam-N<sub>2</sub> and Steam-CO Atmosphere," M.S. Thesis, University of Missouri-Rolla, 1975.
2. Ken Wistrom, "High Pressure Steam-CO Corrosion of High Alumina Castables," M.S. Thesis, University of Missouri-Rolla, 1977.
3. K. S. E. Forssberg, "Mechanical and Thermal Properties of Boehmite Bonded Alumina Bodies," *Ceramurgia Intern.* 2 (3) 136-40 (1976).

Figure 1

SUMMARY FLOW CHART OF SCREENING EXPOSURE TESTS



\*ERDA atmosphere will be CO-18%, CO<sub>2</sub>-12%, H<sub>2</sub>-24%, H<sub>2</sub>O-41%, CH<sub>4</sub>-5%

Table I  
Refractory Materials Used in Exposure Tests

Group No.	Trade or Given Name	Manufacturer	Remarks
<u>Dense High Alumina Castable</u>			
1	Greencast 94	A.P. Green	94% Al <sub>2</sub> O <sub>3</sub> bonded with CA-25 Cement*
2	Kaotab	Babcock & Wilcox	93% Al <sub>2</sub> O <sub>3</sub> bonded with C-3 Cement*
3	Castolast G	Harbison - Walker	90-95% Al <sub>2</sub> O <sub>3</sub>
4	Purotab	Kaiser Refractories	90-95% Al <sub>2</sub> O <sub>3</sub>
5	UMR-1	University of Missouri	93% Al <sub>2</sub> O <sub>3</sub> (70% of Tabular Alumina** + 30% CA-25 Cement)
6	UMR-2	University of Missouri	91% Al <sub>2</sub> O <sub>3</sub> (70% of Tabular Alumina** + 30% Secar 250 Cement*)
7	UMR-3	University of Missouri	91% Al <sub>2</sub> O <sub>3</sub> (70% of Tabular Alumina** + 30% C-3 Cement)
<u>Intermediate Alumina Castable</u>			
8	LO-Abrade	A.P. Green	54-57% Al <sub>2</sub> O <sub>3</sub> , 34-37% SiO <sub>2</sub>
9	RC-3	General Refractories	57% Al <sub>2</sub> O <sub>3</sub> , 34% SiO <sub>2</sub>
<u>Insulating Castable</u>			
10	Kastolite 30	A.P. Green	59% Al <sub>2</sub> O <sub>3</sub> , 33% SiO <sub>2</sub>
11	Cerlite-75	C.E. Refractories	54% Al <sub>2</sub> O <sub>3</sub> , 34% SiO <sub>2</sub>
<u>Dense High Alumina Phosphate-Bonded Ramming Mix</u>			
	Green Pak 90-P	A.P. Green	96% Al <sub>2</sub> O <sub>3</sub>
	90 Ram HS	C.E. Refractories	96% Al <sub>2</sub> O <sub>3</sub>
	Brikram 90-R	General Refractories	90% Al <sub>2</sub> O <sub>3</sub>
<u>Calcium Aluminate Cement</u>			
	CA-25	Aluminum Company of America	79% Al <sub>2</sub> O <sub>3</sub> , 18% CaO
	Secar 250	Lone Star Lafarge Co	72% Al <sub>2</sub> O <sub>3</sub> , 26% CaO
	C-3	Babcock & Wilcox	71.5-72.5% Al <sub>2</sub> O <sub>3</sub> , 24-25% CaO
	Refcon	Universal Atlas Cement	58% Al <sub>2</sub> O <sub>3</sub> + TiO <sub>2</sub> , 33% CaO
	Lumnite	Universal Atlas Cement	44% Al <sub>2</sub> O <sub>3</sub> + TiO <sub>2</sub> , 36% CaO

Table I continued

Group No.	Trade or Given Name	Manufacturer	Remarks
<u>Foam-in-Place Ceramic Insulation</u>			
	Alumina Foam Type C-2 Zirco Foam	Horizons Incorporated Horizons Incorporated Horizons Incorporated	

\* Information on various cements appears elsewhere in this table.

\*\* Aluminum Co. of America, Bauxite, Ark. Tabular Alumina T-61, used as grog in the three castables prepared in this laboratory, contained the following fractions (Wt% of the total amount of castable):

-8 to 14 mesh=14%  
 -14 to 28 mesh=13%  
 -28 to 48 mesh=11%  
 -48 mesh=32%

Table II  
Modulus of Rupture of Unexposed and Exposed Cement Bonded Refractory Castables

Refractory Specimen	Modulus of Rupture in psi						
	As Dried* 110°C	Fired 500°F, Air, 18 hrs.	Exposed to 500 psia and 500°F in ERDA atmosphere			Fired, 1000°F Air, 18 hrs.	Exposed to 1000 psia and 1000°F in ERDA atm. 10-Days
			10-Days	20-Days	30-Days		
<b>High Alumina</b>							
Group 1	2870+290 <sup>**</sup>	1940+130	3030+190	1950+180	2760+420	1880+53	1270+230
Group 2	2500+140	1185+180	2510+330	1630+210	2610+170	1380+150	735+77
Group 3	3080+240	2510+190	2880+260	2520+190	2690+400	2650+86	1460+150
Group 4	3300+370	2410+260	3120+760	2580+470	2990+380	2220+350	1780+300
Group 5	2850+190	2220+170	2680+870	2480+420	2860+600	2200+140	1820+210
Group 6	2930+340	970+180	2930+430	1800+210	3120+160	1810+290	700+77
Group 7	2370+300	1700+300	2260+560	1870+220	2740+230	970+230	1290+120
<b>Intermediate Al<sub>2</sub>O<sub>3</sub></b>							
Group 8	2830+560	1870+340	2810+400	2270+310	2700+450	1760+180	2160+450
Group 9	2200+240	1570+230	2770+900	2000+310	2130+340	1390+99	2390+170
<b>Insulating</b>							
Group 10	650+100	460+51	570+100	440+77	460+53	480+34	530+17
Group 11	970+120	790+77	890+190	670+64	710+57	940+36	1160+36

\* As dried data are based on 9 specimens in each group.

\*\* The + limit corresponds to one standard deviation.

Table III  
 Modulus of Rupture of Unexposed and Exposed Phosphate Bonded High Alumina Refractories

Refractory Specimen	Modulus of Rupture in psi				
	Fired 1000°F, Air, 18 hrs.*	Exposed at 500 psia and 500°F in ERDA atmosphere			Exposed at 1000 psia and 1000°F in ERDA atm
		10-Days	20-Days	30-Days	10-Days
Green Pak 90-P	3910+320**	3320+270	3960+450	4040+380	3340+420
90 Ram HS	3470+250	2420+520	2970+280	3120+380	3240+410
Brikram 90-R	2650+600	1260+620	2370+560	2520+510	1810+390

\* As fired data are based on 9 specimens in each group.

\*\* The + limit corresponds to one standard deviation.

Table IV  
Density & Porosity of Unexposed and Exposed Cement Bonded Refractory Castables

		High Al <sub>2</sub> O <sub>3</sub> Castable						Intermediate Al <sub>2</sub> O <sub>3</sub> Castable		Insulating Castable		
		Group 1	Group 2	Group 3	Group 4	Group 5	Group 6	Group 7	Group 8	Group 9	Group 10	Group 11
As Dried #	Bulk Density gm/cc	2.77	2.62	2.85	2.88	2.75	2.66	2.47	2.33	2.31	1.53	1.58
	% App. Porosity	14	19	14	14	16	16	23	13	16	43	39
Fired at 500°F in Air	Bulk Density gm/cc	2.75	2.56	2.81	2.84	2.71	2.58	2.45	2.29	2.27	1.52	1.55
	% App. Porosity	20	25	17	17	20	24	25	17	19	46	41
Fired at 1000°F in Air	Bulk Density gm/cc	2.67	2.49	2.74	2.78	2.68	2.52	2.35	2.26	2.21	1.46	1.54
	% App. Porosity	23	28	20	22	23	28	31	20	23	49	42
Exposure Test I* 10-Days	Bulk Density gm/cc	2.81	2.59	2.87	2.87	2.74	2.70	2.50	2.32	2.30	1.50	1.55
	% App. Porosity	19	25	19	19	23	22	27	18	19	48	45
Exposure Test I* 20-Days	Bulk Density gm/cc	2.78	2.59	2.85	2.88	2.76	2.62	2.47	2.30	2.28	1.50	1.51
	% App. Porosity	21	26	20	20	22	23	29	19	21	49	44

Table IV continued

		High Al <sub>2</sub> O <sub>3</sub> Castable						Intermediate Al <sub>2</sub> O <sub>3</sub> Castable		Insulating Castable		
		Group 1	Group 2	Group 3	Group 4	Group 5	Group 6	Group 7	Group 8	Group 9	Group 10	Group 11
Exposure Test I* 30-Days	Bulk Density gm/cc	2.81	2.63	2.88	2.90	2.75	2.68	2.52	2.32	2.30	1.50	1.52
	% App. Porosity	20	24	19	18	21	22	27	18	20	48	44
Exposure Test II** 10-Days	Bulk Density gm/cc	2.77	2.57	2.85	2.87	2.73	2.61	2.46	2.26	2.24	1.48	1.53
	% App. Porosity	24	28	21	21	24	26	31	21	22	52	50

# As dried data are based on 9 specimens in each group.

\* 500 psia and 500°F in ERDA atmosphere.

\*\* 1000 psia and 1000°F in ERDA atmosphere.

Table V  
Density and Porosity of Unexposed and Exposed  
Phosphate-Bonded High Alumina Refractories

Refractory Group	Fired at 1000°F, Air, 18 hrs.*		Exposed to 500 psia, 500°F in ERDA atm.						Exposed to 1000 psia, 1000°F in ERDA atm.	
	Bulk Density gm/cc	% App Porosity	10-Days		20-Days		30-Days		10-Days	
			Bulk Density gm/cc	% App Porosity	Bulk Density gm/cc	% App Porosity	Bulk Density gm/cc	% App Porosity	Bulk Density gm/cc	% App Porosity
Green Pak 90-P	2.95	17	2.91	19	2.97	18	2.97	18	2.90	18.2
90 Ram HS	2.92	20	2.90	20	2.94	20	2.94	20	2.91	18.2
Brikram 90-R	2.96	17	2.90	20	2.95	18	2.94	18	2.94	16.3

\* As fired data are based on 9 specimens in each group.

Table VI  
Weight Change in Unexposed and Exposed Refractory Castables

Specimen Group	Percent Weight Change compared to As Dried Weight					
	Fired 500°F, Air, 18 hr.	Fired 1000°F, Air, 18 hr.	Exposed 500 psia, 500°F in ERDA Atmosphere			Exposed 1000 psia, 1000°F ERDA Atm.  10-Days
			10-Days	20-Days	30-Days	
High Alumina Castable						
1	-3.1	-4.5	-0.3	-1.5	-0.2	-2.6
2	-2.9	-4.7	-0.8	-2.0	-0.7	-4.3
3	-2.3	-3.9	+0.2	-0.7	---	-1.1
4	-3.0	-4.1	+0.4	-0.6	+0.6	*
5	-3.2	-5	-0.4	-1.6	-0.3	-2.5
6	-4.3	-5	---	-1.8	-0.1	-3.7
7	-3.0	-4	+1.0	-1.4	-0.2	-5.0
Intermediate Alumina Castable						
8	-3.0	-4.7	-1.0	-1.8	-0.8	-4.2
9	-3.2	-5.0	-0.8	-2.2	-1.1	-4.0
Insulating Castable						
10	-3.4	-5.0	-1.5	-2.0	-1.7	-4.2
11	-2.1	-3.5	-1.0	-1.5	-1.1	-2.9

Symbols: +, Weight gain.

-, Weight loss.

--, Weight Change less than 0.1%.

\*, Three specimens had average weight gain of 7%, and four specimens had average weight loss of 4.6%.

Table VII

## Weight Change in Hydrated Calcium Aluminate Cements

Percent Weight Change compared to As Dried Weight

Cement	500 psia, 500 <sup>o</sup> F 20 Days	1000 psia, 1000 <sup>o</sup> F 10 Days
CA-25	+0.1	-2.2
Secar 250	+3.4	+2.4
C-3	+4.8	-6.3
Refcon	+1.0	-0.5
Lumnite	-1.4	-2.3

+, Weight gain

-, Weight loss