

Effect of Various Furnace  
Atmospheres on the Pyrometric  
Cone Equivalent of Certain  
Typical Refractory Materials

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ABSTRACT

The effect of four different atmospheres on the pyrometric cone equivalent values of five typical refractory materials was investigated.

The atmospheres used were: (1) oxidizing, (2) reducing, (3) sulfur dioxide, and (4) water vapor. Two replications were made on each material, using three test cones for each.

It was found that siliceous fireclay brick and flint clay brick had significantly lower P.C.E. values under all atmospheres other than oxidizing, while the other three samples were not affected.

It was also found that the modified Denver Fireclay P.C.E. furnace was capable of producing satisfactory results under different operators.

## I INTRODUCTION

The pyrometric cone equivalent test has long been a standard measure of refractoriness in the ceramic industry. Standard cones have been perfected which give excellent results under ordinary firing conditions; however, they are subject to a certain degree of variation.

It has long been recognized that furnace atmosphere has been a major factor in cone deviation. The purpose of this investigation is to bring into the light some of the variations in P.C.E. value of certain typical refractory materials.

Clay constituents such as iron and titanium oxides are readily reduced under an atmosphere of CO. Atmospheres of SO<sub>2</sub> and H<sub>2</sub>O also have a reducing effect on these oxides.

The high-temperature series of cones, 23 to 42, contain no known sources of variation under various atmospheres. It should therefore be apparent that refractory materials containing fluxes could be tested along with the high-temperature standard cones, and any deviation in P.C.E. value noted.

An investigation of this nature was conducted by Llewellyn Wood at the Virginia Polytechnic Institute during the 1948-49 session in partial fulfillment of the requirements for the master of science degree in ceramic engineering. The present investigation then, will serve to determine the reproducibility of results by different operators, and also furnish additional atmospheres for study of P.C.E. deviation.

The materials which were used were five typical refractory materials\*, both in the raw state and the fired state. The investigation was designed to allow testing and evaluation of results by means of statistics in order to assign variances to their proper causes.

\*See Appendix D.

## II REVIEW OF LITERATURE

In performing P.C.E. (pyrometric cone equivalent) tests, one of the factors which affects the true P.C.E. value is the furnace atmosphere in which the test is conducted.

According to Geijsbeek<sup>2</sup>, those cones which contain iron oxide, O22 to O11, are subject to change by kiln gases, and the formation of iron silicates will produce lower softening points. For the O10 to 6 cones, the volatilization of the boracic acid in these cones will raise their softening points. The action of the boracic acid will also help the crystallization of silicates. The viscosity of the silicates requires higher temperatures, especially when they contain but little alumina.

Iron oxide which goes into the make-up of the low temperature series of cones is capable of oxidation or reduction in different kiln atmospheres, giving results which vary from the theoretical values.<sup>3</sup>

Vickers and Theobald<sup>4</sup> have shown that  $\text{SO}_2$  and  $\text{CO}_2$  act slightly as reducing gases, lowering the fusion points as  $\text{Fe}_2\text{O}_3$  increases. Hydrogen gas acts strongly as a reducing gas, lowering the fusion point  $250^\circ\text{C}$  in Thiver's earth with an  $\text{Fe}_2\text{O}_3$  content of 2.5%.

Furnace atmospheres containing 0.3 to 0.4 per cent  $\text{SO}_2$  were used on the low series of cones by Fairchild and Peters<sup>5</sup>. All cones but O11, which has a small content of bases, fused at higher temperatures in the presence of such atmospheres.

Sulfuric acid may replace boric acid from materials which contain the latter acid.<sup>6</sup>

Coal ashes contain those oxides found in impure clays. Therefore, because accurate results may be obtained for these mixes, they should be obtainable for the clays<sup>7</sup>.



Fieldner and Hall<sup>8</sup>, using coal ash in their work, found that the most important factor in causing large variation in softening temperature was furnace atmosphere. Atmospheres of air, or strongly reducing CO, gave the highest softening points. The lowest softening temperatures occurred with mixed gases in which reduction of  $\text{Fe}_2\text{O}_3$  proceeded mainly to FeO. Maximum variation in softening points due to atmosphere was from  $143^\circ$  to  $495^\circ$  C.

According to Wartenberg<sup>9</sup>, quartz-glass and porcelain are attacked by hydrogen at temperatures of  $1200^\circ$  C. and above with formation of silicon vapors, which under certain conditions condense to brown silicon. In the presence of metals, the silicon is dissolved. A reaction between  $\text{H}_2\text{O}$  and the FeO of the porcelain appears to take place at  $1200^\circ$  C. as oxygen disappears.

Chlorine reacts with the  $\text{Fe}_2\text{SiO}_4$  of the porcelain at  $600^\circ$  C. with the formation of  $\text{FeCl}_3$ .<sup>10</sup>

At temperatures over  $1150^\circ$  C., and in the presence of alumina-siliceous material, water vapor has greater reducing properties for the oxides of iron than has hydrogen itself.<sup>11</sup>

Ruprecht, Pierce, and Harvey<sup>12</sup> studied the effect of natural gas and of hydrogen on various refractories. They concluded that certain fireclay brick may be completely disintegrated into a powder if subjected to natural gas under certain conditions, but diaspare and silica brick were unaffected by the gas. Hydrogen gas passed over fireclay brick, silica brick, diaspare brick, and magnesia brick at temperatures from  $800^\circ$  to  $1800^\circ$  F. failed to produce any disintegration.

Searle<sup>13</sup> has found the following results from tests on 100 clays:

(1) All the clays with more than 29 percent alumina had a softening point above  $1650^\circ$  C. (2) Fifty-three clays contained 20 to 28.9 per cent alumina; of these, 39 softened at temperatures above  $1650^\circ$  C., whilst 14 softened be-

tween 1120° and 1650° C. (3) Thirteen clays contained less than 20 per cent alumina; of these, seven softened above 1650° C., and six below.

It appears that the fluxing effects on clays are in the following order, beginning with the highest, soda, potash, oxide of iron, lime, and magnesia.<sup>14</sup>

### III INVESTIGATION

#### A. Purpose

The purpose of this investigation was to determine the effect of various furnace atmospheres on the pyrometric cone equivalent values of certain typical refractory materials, and to determine the reproducibility of results by different operators. Results were to be evaluated by means of statistics.

#### B. Procedure

The testing of the cones was done as prescribed by the American Society for Testing Materials (A.S.T.M. Designation C 24-46), following the schedule shown in Table 1. The only deviation from the designation was in the furnace atmospheres.

The samples used in conducting the tests were: (1) silica fire clay (silica cement), (2) siliceous fireclay brick, (3) plastic fire clay, (4) fireclay brick, and (5) flint clay brick.

Cones of all five materials were molded in a molding machine by the Edward Orton, Jr. Ceramic Foundation in order to insure uniformity between cones.

The test cones were set in the plaques as shown in Plate I, and placed in an electric Glo-bar kiln, where they were calcined to a temperature of 2370° F. This was done because of the large percentage of bonding material which had to be used in preparation of the cones.

All five samples were first subjected to testing under the influence of an oxidizing atmosphere indicated by a flue gas analysis showing  $\Delta$  eight to ten per cent carbon dioxide. This is the prescribed atmosphere for the standard P.C.E. (pyrometric cone equivalent) test of refractory materials. The above analysis indicates that the excess air passing into the combustion chamber of the furnace <sup>lies</sup> between 35% and 65%.

The second atmosphere used in testing the five materials was a reducing

atmosphere of from four to five per cent carbon monoxide. This was the highest percentage that could be maintained and cone 34 brought to its end point.

The percentages of  $\text{CO}_2$ ,  $\text{CO}$ , and  $\text{O}_2$ , were measured by using a portable gas analysis apparatus of the Orsat type.

An atmosphere of sulfur dioxide was also used in testing the five materials. The  $\text{SO}_2$  gas was introduced into the gas line of the furnace just prior to mixing with air. A needle valve on the cylinder containing the gas was opened until the  $\text{SO}_2$  fumes escaping with the flue gases became too obnoxious for the comfort of the operator. The amount of this gas entering the furnace was roughly approximated to be 0.25 per cent.

Water vapor was used as a fourth atmosphere in testing the materials. It was obtained by catching steam under a canopy over the air intake of the blower which supplies air to the furnace. Concentration was measured by inserting a wet-dry bulb thermometer into the air line near the furnace, and measuring relative humidity. One hundred per cent relative humidity was readily attainable with the system, and since air in the room remained fairly constant, the amount of moisture per unit of incoming air to the furnace was fairly constant: approximately 0.0018 lb. moisture per cu. ft. dry air.

During each test, the cone plaque was placed in the same relative position in the furnace to take care of the effect of any variation which may have taken place within the chamber of the furnace.

Samples of flue gases were taken at three different temperatures during each test run, namely  $2400^\circ\text{F.}$ ,  $2700^\circ\text{F.}$ , and the temperature at which the lowest cone on the plaque began to tip. These samples were taken at the tip of the flame coming from the furnace, and were drawn through a porcelain tube and rubber hose to the Orsat apparatus. Frequent temperature checks were made on the furnace with a Leeds and Northrup Optical Pyrometer.

The data were recorded as the tests were run, and transformed to decimal equivalents, Table II. These decimal equivalents were subjected to statistical evaluation and testing.

### C. Apparatus

The apparatus used in the investigation is diagrammed in Plate II. It consisted of a modified Denver Fireclay P.C.E. Furnace, AR.I Type, Model A122, a flue gas analysis apparatus of the Orsat type, and cylinders of  $\text{SO}_2$  and  $\text{O}_2$  gases. A section of stove pipe with a heavy ground-coat of enamel was used as a flue, and a porcelain tube and rubber hose connection was used in transferring the flue gases to the Orsat apparatus.

The 12 oz. blower was fitted with a canopy made from roofing tin to concentrate steam for the  $\text{H}_2\text{O}$  atmosphere.

The pressure of the incoming gases was measured by means of a 15 inch water column manometer.

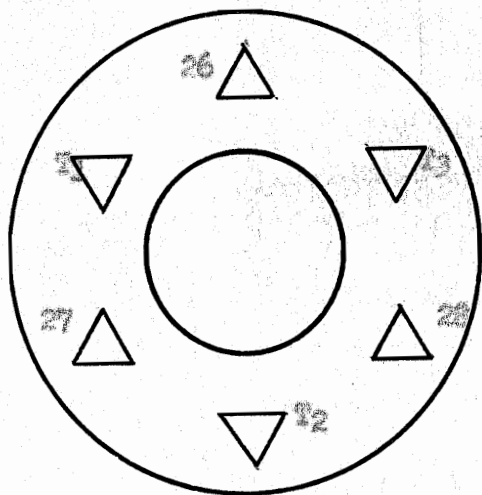
Table I

## A.S.T.M. Heating Schedule for Refractory P.C.E. Test

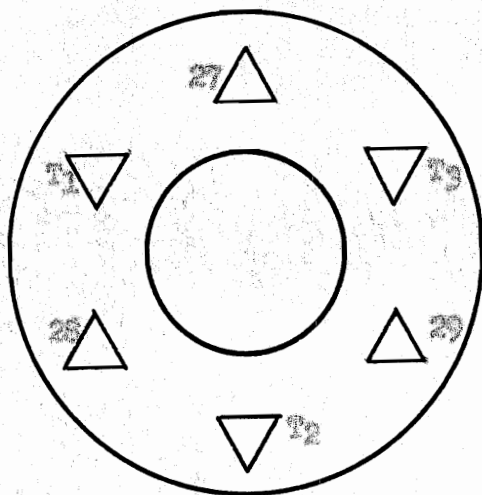
To reach cone 20 .....	45 minutes ....	2786°F
Time interval to		
Cone #23 .....	20 minutes ....	2876°F
Cone #26 .....	6 minutes ....	2903°F
Cone #27 .....	4 minutes ....	2921°F
Cone #28 .....	4 minutes ....	2939°F
Cone #29 .....	10 minutes ....	2984°F
Cone #30 .....	4 minutes ....	3002°F
Cone #31 .....	12 minutes ....	3056°F
Cone #32 .....	8 minutes ....	3092°F
Cone #32½ .....	9 minutes ....	3137°F
Cone #33 .....	9 minutes ....	3173°F
Cone #34 .....	6 minutes ....	3200°F
Cone #35 .....	10 minutes ....	3245°F

## Plate I

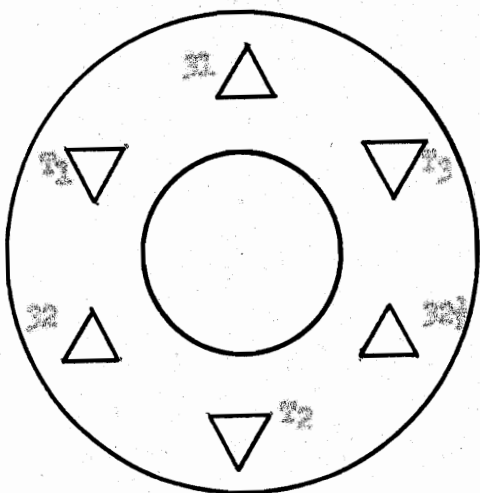
## Positions of Cones in Plaques



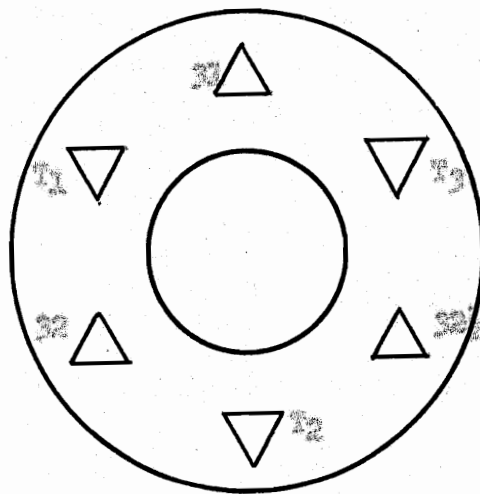
Material #1



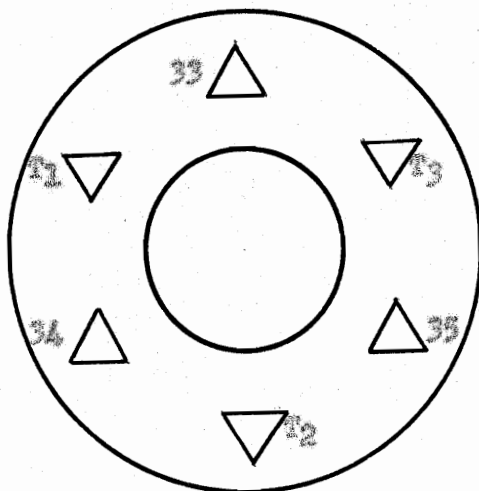
Material #2



Material #3



Material #4



Material #5

Table II

Table for Converting O'clock Positions of Cones to Decimal Equivalents

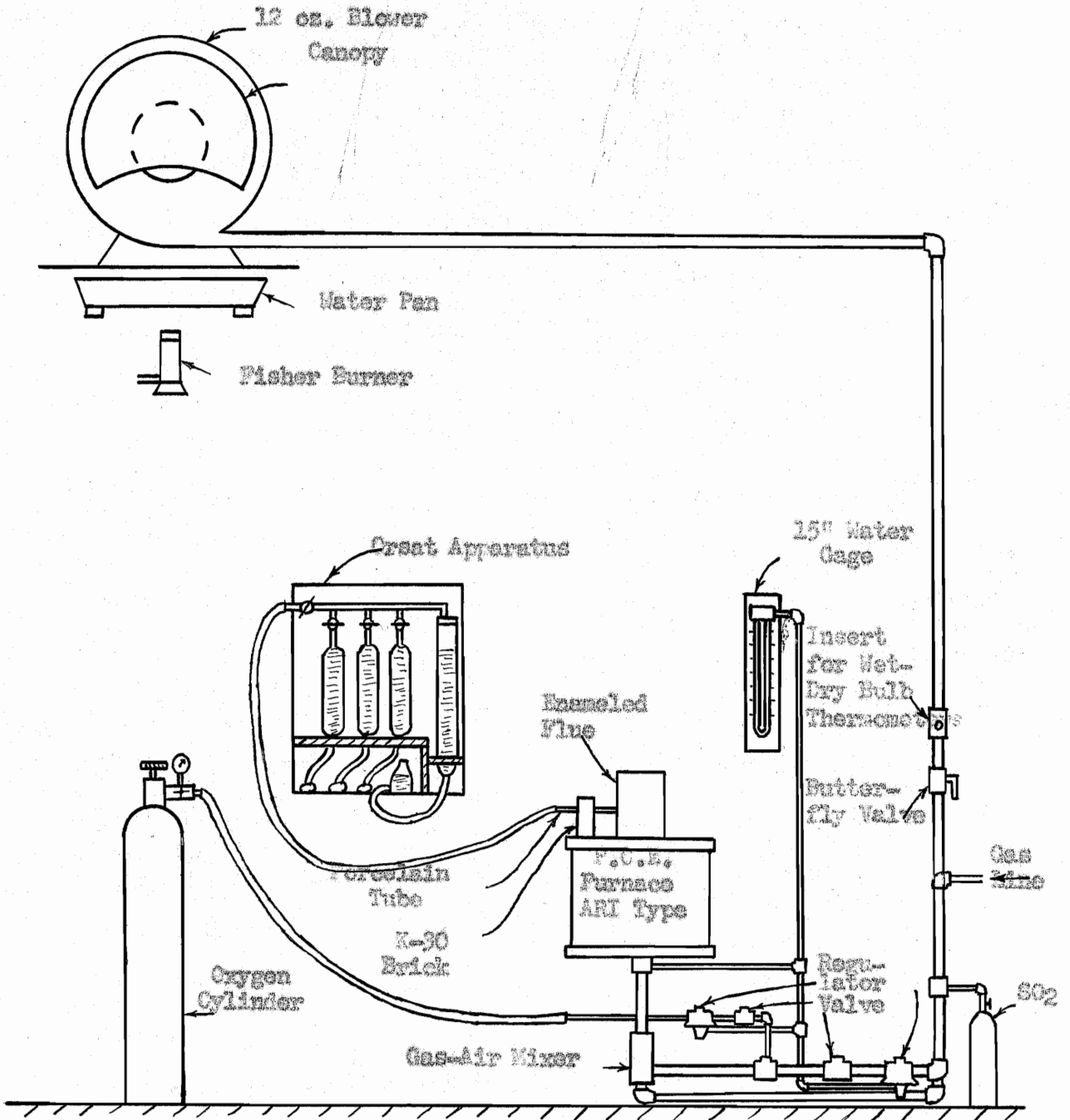
26- 6 o'clock or 27- 3 o'clock .....	26.0
27- 4 o'clock .....	26.3
27- 5 o'clock .....	26.7
27- 6 o'clock or 28- 3 o'clock .....	27.0
28- 4 o'clock .....	27.3
28- 5 o'clock .....	27.7
28- 6 o'clock or 29- 3 o'clock .....	28.0
29- 4 o'clock .....	28.3
29- 5 o'clock .....	28.7
29- 6 o'clock or 30- 3 o'clock .....	29.0
30- 4 o'clock .....	29.3
30- 5 o'clock .....	29.7
30- 6 o'clock or 31-3 o'clock .....	30.0
31- 4 o'clock .....	30.3
31- 5 o'clock .....	30.7
31- 6 o'clock or 32- 3 o'clock .....	31.0
32- 4 o'clock .....	31.3
32- 5 o'clock .....	31.7
32- 6 o'clock or 32½- 3 o'clock .....	32.0
32½- 4 o'clock .....	32.2
32½- 5 o'clock .....	32.3
32½- 6 o'clock or 33- 3 o'clock .....	32.5
33- 4 o'clock .....	32.7
33- 5 o'clock .....	32.8
33- 6 o'clock or 34- 3 o'clock .....	33.0
34- 4 o'clock .....	33.3



34- 5 o'clock .....	33.7
34- 6 o'clock or 35- 3 o'clock .....	34.0
35- 4 o'clock .....	34.3
35- 5 o'clock .....	34.7
35- 6 o'clock .....	35.0

Plate II

Diagram of Apparatus



IV Experimental Data

## A. Code for recording data:

First Number: 1--oxidizing atmosphere  
2--reducing atmosphere  
3--sulfur dioxide atmosphere  
4--water vapor atmosphere

Second Number 1--Sample 1  
2--Sample 2  
3--Sample 3  
4--Sample 4  
5--Sample 5

Third Number: 1--Replication 1  
2--Replication 2

## B. Observations of Cone Tests:

## Replication 1

111

Test Cone 1 ----- 27 at 5 o'clock (26.7)  
 Test Cone 2 ----- 27 at 5 o'clock (26.7)  
 Test Cone 3 ----- 27 at 6 o'clock (27.0)

121

Test Cone 1 ----- 28 at 6 o'clock (28.0)  
 Test Cone 2 ----- 28 at 5 o'clock (27.7)  
 Test Cone 3 ----- 28 at 5 o'clock (27.7)

131

Test Cone 1 ----- 32 at 6 o'clock (32.0)  
 Test Cone 2 ----- 32 at 5 o'clock (31.7)  
 Test Cone 3 ----- 32 at 6 o'clock (32.0)

141

Test Cone 1 ----- 32 at 6 o'clock (32.0)  
 Test Cone 2 ----- 32 at 5 o'clock (31.7)  
 Test Cone 3 ----- 32½ at 4 o'clock (32.2)

151

Test Cone 1 ----- 34 at 6 o'clock (34.0)  
 Test Cone 2 ----- 34 at 5 o'clock (33.7)  
 Test Cone 3 ----- 35 at 4 o'clock (34.3)

211

Test Cone 1 ----- 27 at 6 o'clock (27.0)  
 Test Cone 2 ----- 27 at 5 o'clock (26.7)  
 Test Cone 3 ----- 27 at 6 o'clock (27.0)

## 221

Test Cone 1 ----- 27 at 6 o'clock (27.0)  
 Test Cone 2 ----- 28 at 4 o'clock (27.3)  
 Test Cone 3 ----- 28 at 4 o'clock (27.3)

## 231

Test Cone 2 ----- 32 at 5 o'clock (31.7)  
 Test Cone 2 ----- 32 at 5 o'clock (31.7)  
 Test Cone 3 ----- 32 at 5 o'clock (31.7)

## 241

Test Cone 1 ----- 32 at 5 o'clock (31.7)  
 Test Cone 2 ----- 32 at 5 o'clock (31.7)  
 Test Cone 3 ----- 32 at 5 o'clock (31.7)

## 251

Test Cone 1 ----- 34 at 4 o'clock (33.3)  
 Test Cone 2 ----- 33 at 6 o'clock (33.0)  
 Test Cone 3 ----- 34 at 4 o'clock (33.3)

## 311

Test Cone 1 ----- 27 at 5 o'clock (26.7)  
 Test Cone 2 ----- 27 at 5 o'clock (26.7)  
 Test Cone 3 ----- 27 at 6 o'clock (27.0)

## 321

Test Cone 1 ----- 27 at 6 o'clock (27.0)  
 Test Cone 2 ----- 28 at 4 o'clock (27.3)  
 Test Cone 3 ----- 28 at 4 o'clock (27.3)

## 331

Test Cone 1 ----- 32 at 5 o'clock (31.7)  
Test Cone 2 ----- 32 at 5 o'clock (31.7)  
Test Cone 3 ----- 32 at 4 o'clock (31.3)

## 341

Test Cone 1 ----- 32 at 5 o'clock (31.7)  
Test Cone 2 ----- 32 at 5 o'clock (31.7)  
Test Cone 3 ----- 32 at 6 o'clock (32.0)

## 351

Test Cone 1 ----- 34 at 4 o'clock (33.3)  
Test Cone 2 ----- 34 at 4 o'clock (33.3)  
Test Cone 3 ----- 34 at 4 o'clock (33.3)

## 411

Test Cone 1 ----- 27 at 6 o'clock (27.0)  
Test Cone 2 ----- 27 at 5 o'clock (26.7)  
Test Cone 3 ----- 27 at 6 o'clock (27.0)

## 421

Test Cone 1 ----- 27 at 6 o'clock (27.0)  
Test Cone 2 ----- 27 at 6 o'clock (27.0)  
Test Cone 3 ----- 28 at 4 o'clock (27.3)

## 431

Test Cone 1 ----- 32 at 6 o'clock (32.0)  
Test Cone 2 ----- 32 at 5 o'clock (31.7)  
Test Cone 3 ----- 32 at 5 o'clock (31.7)

21

442

Test Cone 1 ----- 32 at 6 o'clock (32.0)  
Test Cone 2 ----- 32 at 5 o'clock (31.7)  
Test Cone 3 ----- 32 at 6 o'clock (32.0)

451

Test Cone 1 ----- 34 at 6 o'clock (34.0)  
Test Cone 2 ----- 34 at 4 o'clock (33.3)  
Test Cone 3 ----- 34 at 6 o'clock (34.0)

Replication 2

112

Test Cone 1 ----- 27 at 6 o'clock (27.0)  
Test Cone 2 ----- 27 at 6 o'clock (27.0)  
Test Cone 3 ----- 27 at 5 o'clock (26.7)

122

Test Cone 1 ----- 28 at 5 o'clock (27.7)  
Test Cone 2 ----- 28 at 6 o'clock (28.0)  
Test Cone 3 ----- 28 at 6 o'clock (28.0)

132

Test Cone 1 ----- 32 at 6 o'clock (32.0)  
Test Cone 2 ----- 32 at 5 o'clock (31.7)  
Test Cone 3 ----- 32 at 6 o'clock (32.0)

142

Test Cone 1 ----- 32 at 6 o'clock (32.0)  
Test Cone 2 ----- 32 at 5 o'clock (31.7)  
Test Cone 3 ----- 32 at 6 o'clock (32.0)

22

152

Test Cone 1 ----- 34 at 6 o'clock (34.0)  
Test Cone 2 ----- 34 at 6 o'clock (34.0)  
Test Cone 3 ----- 35 at 4 o'clock (34.3)

212

Test Cone 1 ----- 27 at 5 o'clock (26.7)  
Test Cone 2 ----- 27 at 5 o'clock (26.7)  
Test Cone 3 ----- 27 at 5 o'clock (26.7)

222

Test Cone 1 ----- 27 at 6 o'clock (27.0)  
Test Cone 2 ----- 27 at 6 o'clock (27.0)  
Test Cone 3 ----- 28 at 4 o'clock (27.3)

232

Test Cone 1 ----- 32 at 6 o'clock (32.0)  
Test Cone 2 ----- 32 at 5 o'clock (31.7)  
Test Cone 3 ----- 32 at 5 o'clock (31.7)

242

Test Cone 1 ----- 32 at 6 o'clock (32.0)  
Test Cone 2 ----- 32 at 5 o'clock (31.7)  
Test Cone 3 ----- 32 at 6 o'clock (32.0)

252

Test Cone 1 ----- 34 at 4 o'clock (33.3)  
Test Cone 2 ----- 33 at 6 o'clock (33.0)  
Test Cone 3 ----- 34 at 5 o'clock (33.7)



## 312

Test Cone 1 ----- 27 at 6 o'clock (27.0)  
 Test Cone 2 ----- 27 at 5 o'clock (26.7)  
 Test Cone 3 ----- 27 at 6 o'clock (27.0)

## 322

Test Cone 1 ----- 27 at 6 o'clock (27.0)  
 Test Cone 2 ----- 27 at 6 o'clock (27.0)  
 Test Cone 3 ----- 28 at 4 o'clock (27.3)

## 332

Test Cone 1 ----- 32 at 5 o'clock (31.7)  
 Test Cone 2 ----- 32 at 5 o'clock (31.7)  
 Test Cone 3 ----- 32 at 5 o'clock (31.7)

## 342

Test Cone 1 ----- 32 at 6 o'clock (32.0)  
 Test Cone 2 ----- 32 at 5 o'clock (31.7)  
 Test Cone 3 ----- 32 at 5 o'clock (31.7)

## 352

Test Cone 1 ----- 34 at 5 o'clock (33.7)  
 Test Cone 2 ----- 34 at 4 o'clock (33.3)  
 Test Cone 3 ----- 34 at 4 o'clock (33.3)

## 412

Test Cone 1 ----- 27 at 6 o'clock (27.0)  
 Test Cone 2 ----- 27 at 5 o'clock (26.7)  
 Test Cone 3 ----- 27 at 5 o'clock (26.7)

24

422

Test Cone 1 ----- 27 at 6 o'clock (27.0)

Test Cone 2 ----- 28 at 4 o'clock (27.3)

Test Cone 3 ----- 28 at 4 o'clock (27.3)

432

Test Cone 1 ----- 32 at 5 o'clock (31.7)

Test Cone 2 ----- 32 at 5 o'clock (31.7)

Test Cone 3 ----- 32 at 5 o'clock (31.7)

442

Test Cone 1 ----- 32 at 6 o'clock (32.0)

Test Cone 2 ----- 32 at 6 o'clock (32.0)

Test Cone 3 ----- 32 at 5 o'clock (31.7)

452

Test Cone 1 ----- 34 at 6 o'clock (34.0)

Test Cone 2 ----- 34 at 4 o'clock (33.3)

Test Cone 3 ----- 34 at 5 o'clock (33.7)

## V Results

## REPLICATION 1

## Atmosphere 1

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
	26.7	28.0	32.0	32.0	34.0
	26.7	27.7	31.7	31.7	33.7
	27.0	27.7	32.0	32.2	34.3
$\Sigma$	80.4	83.4	95.7	95.9	102.0
$\bar{v}_i$	26.80	27.80	31.90	31.97	34.00
			$\Sigma \Sigma = 457.4$		
			$\bar{v}_i = 30.49$		

## Atmosphere 2

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
	27.0	27.0	31.7	31.7	33.3
	26.7	27.3	31.7	31.7	33.0
	27.0	27.3	31.7	31.7	33.3
$\Sigma$	80.7	81.6	95.1	95.1	99.6
$\bar{v}_i$	26.90	27.20	31.70	31.70	33.20
			$\Sigma \Sigma = 452.1$		
			$\bar{v}_i = 30.14$		

## Atmosphere 3

Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
26.7	27.0	31.7	31.7	33.3
26.7	27.3	31.7	31.7	33.3
27.0	27.3	31.7	32.0	33.3
$\Sigma$ 80.4	81.6	95.1	95.4	99.9
$\bar{y}_i$ : 26.80	27.20	31.70	31.80	33.30
$\Sigma \Sigma = 452.4$				
$\bar{y}_i = 30.16$				

## Atmosphere 4

Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
27.0	27.0	32.0	32.0	34.0
26.7	27.0	31.7	31.7	33.3
27.0	27.3	31.7	32.0	34.0
$\Sigma$ 80.7	81.3	95.4	95.7	101.3
$\bar{y}_i$ : 26.90	27.10	31.80	31.90	33.77
$\Sigma \Sigma = 454.4$				
$\bar{y}_i = 30.29$				

$$\Sigma \Sigma \Sigma = 1816.3$$

$$\bar{y} = 30.27$$

## REPLICATION 2

## Atmosphere 1

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
	27.0	27.7	32.0	32.0	34.0
	27.0	28.0	31.7	31.7	34.0
	26.7	28.0	32.0	32.0	34.3
$\Sigma$	80.7	83.7	95.7	95.7	102.3
$\bar{x}_i$	26.90	27.90	31.90	31.90	34.10
$\Sigma \Sigma = 458.1$					
$\bar{\bar{x}}_i = 30.54$					

## Atmosphere 2

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
	26.7	27.0	32.0	32.0	33.3
	26.7	27.0	31.7	31.7	33.0
	26.7	27.3	31.7	32.0	33.7
$\Sigma$	80.1	81.3	95.4	95.7	100.0
$\bar{x}_i$	26.70	27.10	31.80	31.90	33.83
$\Sigma \Sigma = 452.5$					
$\bar{\bar{x}}_i = 30.17$					

## Atmosphere 3

Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
27.0	27.0	31.7	32.0	33.7
26.7	27.0	31.7	31.7	33.3
27.0	27.3	31.7	31.7	33.3

---


$$\Sigma \quad 80.7 \quad 81.3 \quad 95.1 \quad 95.4 \quad 100.3$$

$$\bar{x}_i \quad 26.90 \quad 27.10 \quad 31.70 \quad 31.80 \quad 33.43$$

$$\Sigma \Sigma = 452.8$$

$$\bar{\bar{x}}_i = 30.19$$

## Atmosphere 4

Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
27.0	27.0	31.7	32.0	34.0
26.7	27.3	31.7	32.0	33.3
26.7	27.3	31.7	31.7	33.7

---


$$\Sigma \quad 80.4 \quad 81.6 \quad 95.1 \quad 95.7 \quad 101.0$$

$$\bar{x}_i \quad 26.80 \quad 27.20 \quad 31.70 \quad 31.90 \quad 33.67$$

$$\Sigma \Sigma = 453.8$$

$$\bar{\bar{x}}_i = 30.25$$

$$\Sigma \Sigma \Sigma = 1817.2$$

$$\bar{\bar{\bar{x}}} = 30.29$$

Table III

## Average F.C.E. Values of Materials

Material No.	Oxidizing Atmosphere	Reducing Atmosphere	Sulfur Dioxide Atmosphere	Water Vapor Atmosphere
1	26.85	26.80	26.85	26.85
2	27.85	27.15	27.15	27.15
3	31.90	31.75	31.70	31.75
4	31.94	31.80	31.80	31.90
5	34.05	33.26	33.36	33.72

## VI DISCUSSION OF RESULTS

In order to properly test and evaluate results, the investigation was subjected to testing by statistical analysis. The F-test of significance was used in conjunction with the analysis of variance, and results were taken as significant at the one per cent level of testing.

Two replications were made on each material, giving one degree of freedom. The F-test value for replications showed no significance and indicated that the investigation was under control from that aspect.

Atmospheres were sub-grouped into: (1) atmosphere one against atmosphere two, (2) atmosphere three against atmosphere four, and (3) atmospheres one and two against atmospheres three and four. Atmosphere three against atmosphere four was found to be not significant, showing that the significant effect of atmospheres is distributed among deviation of the reducing, sulfur dioxide, and water vapor atmospheres from the oxidizing atmosphere.

Materials were sub-grouped in a manner similar to that used for atmospheres, and significant results were found for all comparisons except the comparison of material three with material four.

It was decided not to sub-group the interactions into their component parts since they would give results according to the significant or not significant components going into their make-up. The interactions are shown in the orthogonal comparison table to indicate how they may be broken up into components.

Chemical analyses of the five test materials were made by the General Refractories Company, Baltimore, Maryland. They fail to yield any absolute reasons for the behavior of materials number two and number five against the other test materials. It may be seen from the analyses that refractori-



ness increases with the content of alumina. The constituents which were believed to influence P.C.E. values under different atmospheres were ferric oxide and titanias. Neither of those materials, however, in a position in the test materials to cause discrepancies unless the reason is coupled with an interaction with other constituents. The chemical analysis furnished much less information than it was believed could be derived therefrom.

The furnace behaves differently on different days. From relative humidity data taken during the conducting of the experiment, it is to be concluded that testing is more readily done on days when the relative humidity is comparatively low. When the water vapor atmosphere was used, it was necessary to use oxygen to reach the end point of even the lower cones.

When sulfur dioxide was introduced into the furnace, it was necessary to increase the flow of gas, indicating that some of the  $SO_2$  may possibly have combined with oxygen from the air resulting in the formation of  $SO_3$ .

There is good reason to suspect that there is a strong source of variation due to personal causes in operating the furnace since it is necessary to perfect technique to a fine degree before testing is feasible. Results of P.C.E. values which lie fairly close to those of a previous operator, Llewellyn Wood indicate that the furnace is adaptable for different operators. Results of P.C.E. values which lie within the values given by the A.S.T.M. for testing refractory materials\* indicate that the furnace is satisfactory for making F.C.E. determinations.

\* See Appendix D.

## VII CONCLUSIONS

The following conclusions appear to be justified from application of the F- test at the one per cent level to the analysis of variance and from application of standard error to the average results of P.C.E.

1. The P.C.E. of Sample #1 (a silica cement) is not affected by atmospheres of carbon monoxide, sulfur dioxide, or water vapor.

2. The P.C.E. of Sample #2 ( a siliceous fireclay brick) is lowered by atmospheres of carbon monoxide, sulfur dioxide, or water vapor. These three atmospheres show no differences among themselves.

3. The P.C.E. of Sample #3 (a plastic fire clay) is not lowered by atmospheres of carbon monoxide, sulfur dioxide, or water vapor.

4. The P.C.E. of Sample #4 (a fireclay brick) is not lowered by atmospheres of carbon monoxide, sulfur dioxide, or water vapor.

5. The P.C.E. of Sample #5 (a flint clay brick) is lowered by atmosphere of carbon monoxide, sulfur dioxide, or water vapor. The water vapor atmosphere for this sample gives results higher than the CO or SO<sub>2</sub> atmospheres.

6. There is no difference between the P.C.E. values of Sample #3 (a plastic fire clay) and Sample #4 (a fireclay brick).

7. The modified Denver Fireclay P.C.E. furnace is capable of producing similar results for different operators.

8. The chemical analysis of the test materials indicate no apparent constituent or constituents responsible for the lowering of the P.C.E. of Sample #2 and Sample #5.

VIII SUMMARY

The pyrometric cone equivalents of five typical refractory materials were tested under the influences of four furnace atmospheres.

It was found that a siliceous fireclay brick and a flint clay brick have significantly lower P.C.E. values when tested under atmospheres of Carbon monoxide, sulfur dioxide, or water vapor than when tested under an oxidizing atmosphere. A silica cement, a plastic fire clay, and a fireclay brick showed no significant lowering of P.C.E. under the other atmospheres.

Chemical analyses of the materials failed to divulge any clue as to why two of them should behave differently when subjected to foreign atmospheres. Perhaps the reason lies in the interaction of a combination of constituents.

IX APPENDIX

## Appendix A

Bibliography

1. Wood, Llewellyn, Virginia Polytechnic Institute, Department of Ceramic Engineering, M.S. Thesis, (1949).
2. Geijsbeek, Samuel, "The Melting Points of Pyrometric Cones Under Various Conditions", Trans. Amer. Ceram. Soc., Vol. XIV (1912), pp. 862-63.
3. Gorton, E.H., "Remarks on Seger Cones", Trans. Amer. Ceram. Soc., Vol. II (1900), p. 60.
4. Vickers, A.E., and Theobald, L.S., "The Influence of Oxidizing and Reducing Atmospheres on Refractory Materials", Trans. Ceram. Soc. (Eng.), Vol. 24 (1925), pp. 87-104.
5. Fairchild, C.O., and Peters, M.E., "Characteristics of Pyrometric Cones", Journ. Amer. Ceram. Soc., Vol. 9 (1926), p. 9.
6. Ortman, F.B., "The Composition of Kiln Gases and Their Effect on Terra Cotta Glazes and Colors", Journ. Amer. Ceram. Soc., Vol III (1920), pp. 476-89.
7. Kirkpatrick, F.A., "A fusion Study of the Mineral Systems Feldspar-Calcite and Feldspar-Magnesite", Trans. Amer. Ceram. Soc., Vol. XVIII (1916), p. 585.
8. Fieldner and Hall, "Softening Temperatures of Coal Ash", Journ. of Ind. and Eng. Chem., May and June (1915)
9. Wartenberg, Hans V., "The Effect of Gases on Silicates", Æ Anng. Allgem. Chem. (1925), pp. 335-6. From: Cer. Abs., Vol. 4 (1925) p. 229.
10. Nernst and V. Wartenberg, Æ Physik. Chem. (1905) p. 535.

11. Vickers, A.E.J., "Effects of Furnace Atmospheres on Refractory Materials", Trans. Inst. Gas Eng. (1926), pp. 26-33. From: Cer. Abs. Vol. 10 (1931), p. 41.
12. Ruprecht, B.O., Pierce, R.H.H., and Harvey, F.A., "Study of the Effect of Natural Gas and of Hydrogen Gas Upon Various Refractories", Journ. Amer. Ceram. Soc., Vol. 17 (1934) pp. 185-93.
13. Searle, A.B., Refractory Materials. London: Charles Griffin and Company, Ltd. (1924) p. 116.
14. Ibid 12, p. 117.
15. Davies, O.L., Statistical Methods in Research and Production. London: Oliver and Boyd, (1949), p. 73.
16. Fisher, R.A., The Design of Experiments. Edinburgh: Oliver and Boyd, (1937).

## APPENDIX B

Statistical Analysis

When data are subject to variations due to a number of separate causes, a technique known as analysis of variance will allow separation of the different causes of variation and determine their magnitudes. It may be profitably applied to almost any type of experimental results involving quantitative measurements, particularly when several independent sources of variation are present.

The variance is numerically the square of the standard deviation. Owing to its additive properties, the former is to be desired in preference to the latter. The additive properties, of course, hold strictly true only for an infinite number of tests; for any finite number of tests, the value is only approximate, and increases in accuracy directly with the number of observations. The arithmetical splitting up of a total variance into its component parts is the analysis of variance. Since it is impossible to have an infinite number of tests, there must be a source of error, and in the analysis of variance, the error group is obtained by subtracting from the total, all assignable causes of variation. It is possible to classify the data with respect to each independent source of variation, and a complete classification is a necessary first step to the application of the analysis of variance.<sup>15</sup>

From data on this investigation, there are five materials that were tested, and therefore, four possible independent comparisons that may be made between materials, or as they are termed in statistics, degrees of freedom. One comparison goes to the grand mean and cannot be used in the variance. The analysis of variance table, Table IV, shows the general breakdown of the variance. The degrees of freedom for each one of the

parts represents the number of independent comparisons that can be made on that part. The other parts of the analysis are obtained in a similar manner to that for materials.

The next step in the table is to calculate the individual sum of squares for the parts that were broken down into sources of variation. The sums of squares are assigned to the same parts as are the degrees of freedom. The total sum of squares is the sum of squares of the deviations between the actual values obtained and their means. It measures the total amount of variation due to all causes.

The mean squares of the individual parts are obtained by dividing the sums of squares of the parts by their corresponding degrees of freedom.

The ratio of the mean squares of the components to the mean square of the experimental error term is known as the F- test of significance. Tables<sup>16</sup> have been prepared which give the values of significance for both the one per cent and the five per cent levels at the proper degrees of freedom. If the value of the quotient is found to be less than that for the five per cent level that source of variation is said to be not significant. If it lies between the five per cent and the one per cent values, it is said to be significant; and if it is greater than the value for the one per cent level, it is said to be highly significant.

The standard error for the difference between any two cone plaques subjected to two replications was computed from the experimental error. Because of the highly significant results found under analysis of variance, it was decided that certain valid conclusions could be drawn from this test (T-test). The value from the t-table for 19 degrees of freedom at the one per cent level was multiplied by the computed standard error between any two cone plaques ( $\hat{\sigma}_d$ ), and the product was used for testing

the average P.C.E. results. When the P.C.E. of a material was lowered numerically greater than the value for the product thus computed, that material was said to have a significantly lower P.C.E. under the other atmosphere. For example, the P.C.E. of material number one under an oxidizing atmosphere was found to be 26.85, while under a reducing atmosphere it was found to be 26.80. The lowering of 0.05 was less than the allowable value of 0.22; hence, the P.C.E. of material one was said to be unaffected by a reducing atmosphere.

By the proper use of statistical methods, an experiment may be evaluated, and the sources of error pointed out for possible correction, elimination, or recommendations.



## Meanings of Symbols Used in the Analysis of Variance

### Table and Orthogonal Comparison Table

#### Analysis of Variance Symbols:

- Not Significant;  $F <$  tabular value for 5% level.

\* Significant;  $F >$  tabular value for 5%, but  $<$  tabular value for 1% level.

\*\* Highly significant;  $F >$  tabular value for 1% level.

$M_1$  vs  $M_2$  — Material one compared with material two

$M_3$  vs  $M_4$  — Material three compared with material four

$M_{1,2}$  vs  $M_{3,4}$  — Materials one and two compared with materials three and four

$M_{1,2,3,4}$  vs  $M_5$  — Materials one, two, three, and four compared with material five.

$A_1$  vs  $A_2$  — Atmosphere one compared with atmosphere two

$A_3$  vs  $A_4$  — Atmosphere three compared with atmosphere four.

$A_{1,2}$  vs  $A_{3,4}$  — Atmospheres one and two compared with atmosphere three and four.

#### Orthogonal Comparisons Symbols:

$A_1 M_2$  — Material one in an oxidizing atmosphere

$A_1 M_2$  — " two " " " " "

$A_1 M_3$  — " Three " " " " "

$A_1 M_4$  — " four " " " " "

$A_1 M_5$  — " five " " " " "

$A_2 M_1$  — " one " " reducing " "

$A_2 M_2$  — " two " " " " "

$A_2 M_3$  — " three " " " " "

$A_2 M_4$  — " four " " " " "

$A_2 M_5$  — " Five " " " " "

$A M$  — " one " " SO<sub>2</sub> " "

$A_3 M_2$	—	Material two	in an $SO_2$	atmosphere.	
$A_3 M_3$	—	"	Three	" " "	"
$A_3 M_4$	—	"	four	" " "	"
$A_3 M_5$	—	"	five	" " "	"
$A_4 M_1$	—	"	one	" " $H_2O$	"
$A_4 M_2$	—	"	two	" " "	"
$A_4 M_3$	—	"	three	" " "	"
$A_4 M_4$	—	"	four	" " "	"
$A_4 M_5$	—	"	five	" " "	"

Table IV

Analysis of Variance

Source of Variation	D/F	Sum of Squares	Mean Squares	F
Replications	1	0.01	0.010	1 -
Atmospheres	(3)	(2.51)	(0.837)	46.5**
$A_1$ vs $A_2$	1	1.98	1.980	110.0**
$A_3$ vs $A_4$	1	0.15	0.150	8.3*
$A_{1,2}$ vs $A_{3,4}$	1	0.38	0.380	21.1**
Materials	(4)	(871.95)	(217.988)	12,110.4**
$M_1$ vs $M_2$	1	2.85	2.850	158.3**
$M_3$ vs $M_4$	1	0.09	0.090	5.0-
$M_{1,2}$ vs $M_{3,4}$	1	538.18	538.180	29,898.9**
$M_{1,2,3,4}$ vs $M_5$	1	330.83	330.830	18,379.4**
Interactions	12	2.22	0.185	10.3**
Experimental Error	19	0.34	0.018	—
Sampling Error	80	2.85	0.036	2.00-
Total	119	879.88	—	—

Calculation of Sums of Squares

$$\text{Total sum} = 3633.5$$

$$(\text{Total sum})^2 = 13,202,322.25 = (\sum x)^2$$

$$\frac{(\sum x)^2}{n} = \frac{(13,202,322.25)}{120} = 110,019.35 = C$$

$$\text{Total sum of squares} = \sum (x)^2 - C$$

$$= (26.7)^2 + (26.7)^2 + (27.0)^2 + \dots + (33.7)^2 - C$$

$$= 110,899.23 - C = 879.88$$

$$\text{Replications sum of squares} = \frac{R_1^2 + R_2^2}{60} - C$$

$$= \frac{(1816.3)^2 + (1817.2)^2}{60} - C$$

$$= 110,019.36 - C = 0.01$$

$$\text{Atmospheres sum of squares} = \frac{A_1^2 + A_2^2 + A_3^2 + A_4^2}{30} - C$$

$$= \frac{(915.5)^2 + (904.6)^2 + (905.2)^2 + (908.2)^2}{30} - C$$

$$= 110,021.86 - C = 2.51$$

$$\text{Materials sum of squares} = \frac{M_1^2 + M_2^2 + M_3^2 + M_4^2 + M_5^2}{24} - C$$

$$= \frac{(644.1)^2 + (655.8)^2 + (762.6)^2 + (764.6)^2 + (806.4)^2}{24} - C$$

$$= 110,891.30 - C = 871.95$$

$$\text{Treatment sum of squares} = \frac{T_1^2 + T_2^2 + T_3^2 + \dots + T_{20}^2}{6} - C$$

$$= \frac{(161.1)^2 + (167.1)^2 + (191.4)^2 + \dots + (202.3)^2}{6} - C$$

$$= 110,896.03 - C = 876.68$$

$$\text{Interaction sum of squares} = \text{treatments} - \text{Atmospheres} - \text{materials}$$

$$= 876.68 - 2.51 - 871.95 = 2.22$$

$$\begin{aligned}
 \text{Sub-total sum of squares} &= \frac{G_1^2 + G_2^2 + G_3^2 + \dots + G_{40}^2}{3} - C \\
 &= \frac{(80.4)^2 + (83.4)^2 + (95.7)^2 + \dots + (101.0)^2}{3} - C \\
 &= 110,896.38 - C = \underline{877.03}
 \end{aligned}$$

$$\begin{aligned}
 \text{Sampling error sum of squares} &= \text{total} - \text{sub} - \text{total} \\
 &= 879.88 - 877.03 = \underline{2.85}
 \end{aligned}$$

$$\begin{aligned}
 \text{Experimental error sum of squares} &= \text{total} - \text{all others} \\
 &= 879.88 - 879.54 = \underline{0.34}
 \end{aligned}$$



Estimate of Variance

The calculation of the standard error ( $\hat{\sigma}$ ) for a cone plaque containing three test cones, subjected to two replications, and the standard error for the difference between any two cone plaques ( $\hat{\sigma}_d$ ) containing three test cones, is obtained as follows:

$$\text{Experimental error} = 0.018$$

$$\text{Standard deviation} = \sqrt{0.018} = 0.134$$

$$\text{Standard error } \hat{\sigma} = \frac{0.134}{\sqrt{n}} = \frac{0.134}{\sqrt{6}} = \pm 0.0547$$

$$\begin{aligned} \text{Standard error for the difference } \hat{\sigma}_d &= 0.134 \sqrt{\frac{1}{N_1} + \frac{1}{N_2}} \\ &= 0.134 \sqrt{\frac{1}{6} + \frac{1}{6}} \\ &= \frac{0.134}{1.732} = \pm 0.0773 \end{aligned}$$

$$T - \text{value at } 1\% \text{ level for } 19 \text{ degrees of freedom} = 2.861$$

$$\text{Significance level at } 1\% \text{ level for average values} = 2.861 \times 0.0773 = 0.22$$

## Appendix C

Combustion Data

Table VI

## Combustion Data of Commercial Propane (Pyrofax)

Cu. ft. dry air required for perfect combustion of one cubic foot of gas -----	23.82
Cu. ft. of moisture produced for each cu. ft. of gas burned -----	4.17
Cu. ft. of CO <sub>2</sub> produced for each cu. ft. of gas burned -----	3.00
Cu. ft. of N <sub>2</sub> produced for each cu. ft. of gas burned -----	18.82
Total volume of flue gases -----	25.99
Dry volume of flue gases (H <sub>2</sub> O assumed condensed) -----	21.82
Dry volume of flue gases for various CO <sub>2</sub> analyses = $\frac{3.00 \times 100}{\% \text{ CO}_2}$	
Ultimate per cent CO <sub>2</sub> = $\frac{3.00 \times 100}{21.82}$ =	13.7 %

## Flue Gas Data Based on Burning One

## Cubic Foot of Commercial Propane

Analysis of Flue Gases			Excess Air	
% CO <sub>2</sub>	% O <sub>2</sub>	% N <sub>2</sub>	%	Cu. Ft.
13.7	---	86.3	---	---
12.0	2.6	85.4	13.3	3.18
11.0	4.0	85.0	23.0	5.48
10.0	4.5	84.5	34.3	8.18
8.0	8.6	83.4	65.7	15.68
6.0	11.8	82.2	118.5	28.18
			Total Volume Including 4.17 cu. ft. Moisture	
N <sub>2</sub>	O <sub>2</sub>	CO <sub>2</sub>	Dry Volume	
18.82	---	3.00	21.82	25.99
21.33	0.67	3.00	25.00	29.17
23.15	1.15	3.00	27.30	31.47
25.28	1.72	3.00	30.00	34.17
31.21	3.29	3.00	37.50	41.67
41.09	5.91	3.00	50.00	54.17



Graph 1

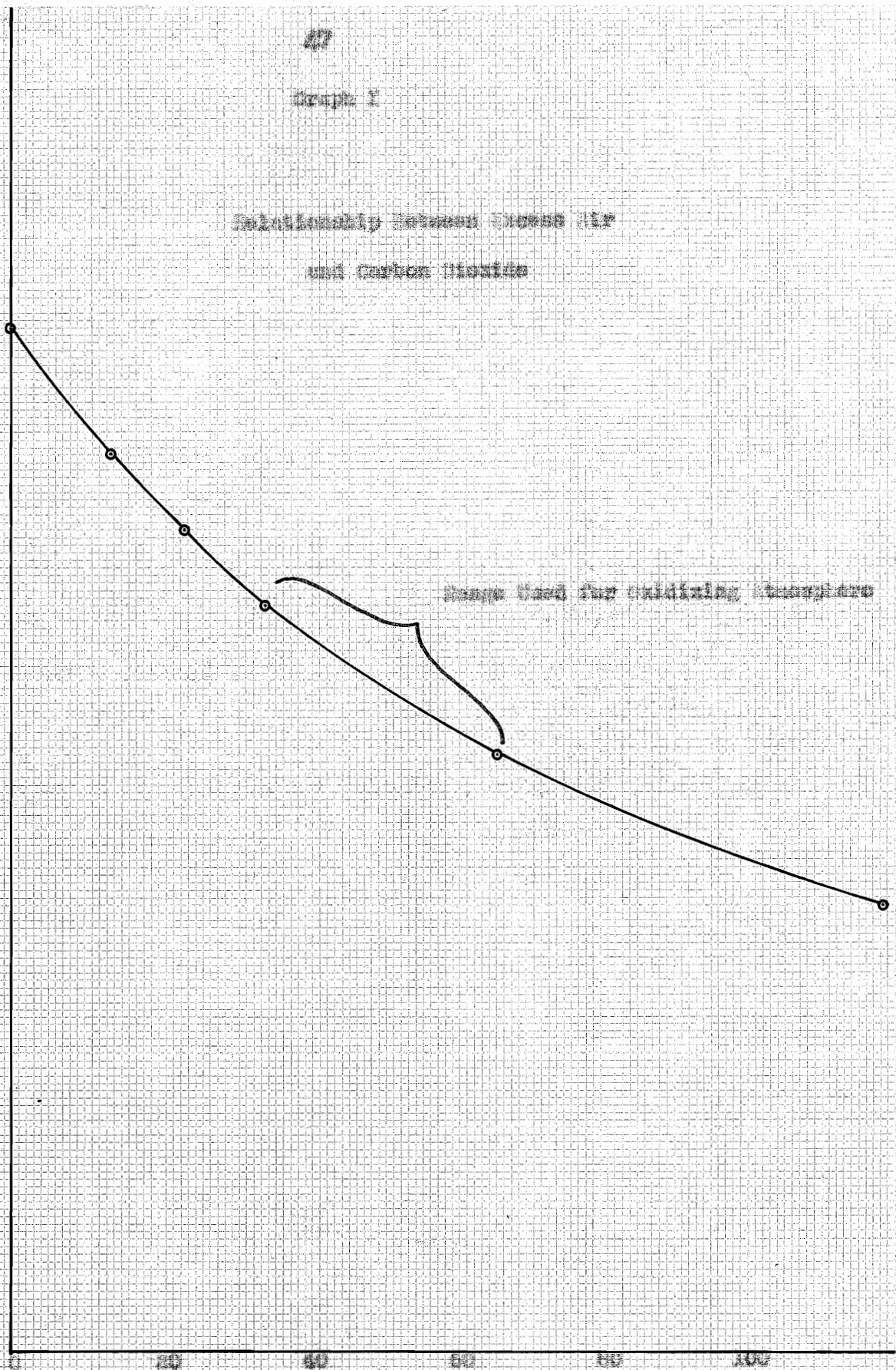
Relationship Between Oxygen Air  
and Carbon Dioxide

PER CENT CARBON DIOXIDE

14  
12  
10  
8  
6  
4  
2  
0

Range Used for oxidizing atmosphere

PER CENT OXYGEN AIR



## Appendix D

Standardized Samples for the  
Pyrometric Cone Equivalent Test

Five materials have been selected and standardized for use with the pyrometric cone equivalent test of refractories. They serve as standards for use by different laboratories for checking P.C.E. determinations. The five samples are comprised of the following materials:

- No. 1 -- Silica fire clay (silica cement)
- No. 2 -- Siliceous fireclay brick.
- No. 3 -- Plastic fire clay.
- No. 4 -- Fireclay brick.
- No. 5 -- Flint clay brick.

In cooperation with this project, the chemical analyses of these materials have been obtained from the General Refractories Company, Baltimore, Maryland. The analyses as received, and as converted to zero loss on ignition are given in the following table:

Table VII  
Chemical Analyses of Standard Samples\*

Sample No	1		2		3		4		5	
	As Rec'd	Altered	As Rec'd	Altered	As Rec'd	Altered	As Rec'd	Altered	As Rec'd	Altered
Ign. Loss	1.79	0.00	0.00	0.00	11.37	0.00	0.06	0.00	0.00	0.00
Silica	89.44	91.30	74.58	74.58	48.12	56.05	55.56	56.75	52.11	52.11
Peric Oxide	0.69	0.71	1.78	1.78	2.24	2.61	2.06	2.10	1.25	1.25
Alumina	5.64	5.79	20.49	20.49	32.20	37.50	37.03	37.83	42.64	42.64
Titania	0.28	0.29	1.86	1.86	2.22	2.58	2.56	2.62	2.24	2.24
Lime	1.09	1.12	0.27	0.27	0.38	0.44	0.23	0.24	0.41	0.41
Magnesia	0.30	0.31	0.28	0.28	0.64	0.74	0.40	0.41	0.45	0.45
Total	99.23	99.52	99.26	99.26	97.17	99.92	97.90	99.95	99.10	99.10

\* As received analyses obtained from the General Refractories Company, Baltimore, Maryland. Altered percentages calculated by author to eliminate loss on ignition.

Sample No. 1 is furnished in the "as received" condition and, according to the A.S.T.M. procedure for P.C.E. determination (C 24), it should be tested "without grinding". All of the remaining samples must be ground to specified particle size. Samples should be selected after careful quartering of the material.

Sample No. 3 was not calcined prior to making the P.C.E. tests used for standardization. Tests have shown that there is little practical difference in values for calcined or non-calcined material.

The P.C.E. values obtained for the samples were obtained in cooperation with five laboratories which were well experienced in making the test. Twelve different operators joined in making the tests, and their work totaled over 30 determinations on each sample. All of the furnaces used were gas fired, and the tests were carried out under an oxidizing atmosphere. The average value for each sample is given as follows:

No. 1 -- Cone 27 - 28

No. 2 -- Cone 28

No. 3 -- Cone 32

No. 4 -- Cone 32 - 32½

No. 5 -- Cone 34

The P.C.E. data obtained from these laboratories show that reproducibility is dependent upon the material being tested. Samples 1, No. 3, and No. 4 checked to within  $\pm \frac{1}{2}$  cone of the average. Sample No. 2 checked to within  $\pm 1$  cone of the average, while sample No. 5 showed a deviation from the average of only  $\pm \frac{1}{4}$  cone.

Standardized samples may be obtained as a set (not sold singly) for

\$6.50 per set. The individual samples weigh approximately 150 grams.

Address inquiries to:

Refractories Fellowship

Mellon Institute

Pittsburgh 13, Pa.

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