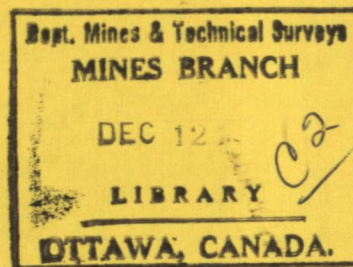




CANADA



THE EFFECT OF THE  
MINERALOGICAL COMPOSITION OF  
WHITEMUD FORMATION CLAYS  
ON THEIR UTILIZATION

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DEPARTMENT OF MINES AND  
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THE EFFECT OF THE MINERALOGICAL COMPOSITION OF  
WHITEMUD FORMATION CLAYS ON THEIR UTILIZATION

by

J. G. Brady\*

## ABSTRACT

Such ceramic properties as plasticity, drying behaviour, and firing behaviour of a wide variety of clays from the Whitemud formation of southern Saskatchewan and southeastern Alberta are investigated. The relationship between the mineralogical composition (determined by X-ray diffraction and differential thermal analysis) and possible commercial utilization is discussed.

Typical samples of good-quality stoneware clays, ball clays, fire clays and kaolinized sands suitable for a wide variety of products contain principally kaolinite and quartz and frequently mica, feldspar and montmorillonite. Fine particle kaolinite and montmorillonite have the greatest effect on increasing the plasticity of these clays while quartz, feldspar and coarse mica reduce their plasticity. The most refractory clays contain principally kaolinite, quartz, and sometimes a small quantity of gibbsite. Mica occurs as the clay mineral "illite" or as coarse mica particles such as muscovite. Mica, feldspar, and montmorillonite lower the refractoriness of the Whitemud clays.

Differential thermal analysis indicates that there should be no problem in firing the Whitemud clays. Contaminating minerals such as carbonaceous material, siderite, jarosite and gypsum should not prove troublesome, because they are present in minor quantities. Usually, the composition of the Whitemud clays results in a medium to long firing range, whereas in many other Canadian clays the firing range is very short.

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Direction des mines

Rapport de recherches R 99

EFFET DE LA COMPOSITION MINÉRALOGIQUE  
DES ARGILES DE LA FORMATION WHITEMUD  
SUR LEUR UTILISATION DANS L'INDUSTRIE

par

J. G. Brady\*

RÉSUMÉ

Le présent rapport traite des recherches faites au sujet de certaines propriétés céramiques telles que la plasticité ainsi que le comportement au séchage et au feu d'une grande variété d'argiles en provenance de la formation Whitemud (Sud de la Saskatchewan et Sud-Est de l'Alberta). L'auteur étudie les relations entre la composition minéralogique (telle que déterminée par la radiocristallographie par diffraction et l'analyse thermique différentielle) et les utilisations possibles dans l'industrie.

Les échantillons représentatifs d'argiles à poterie de grès, d'argiles figulines, d'argiles réfractaires et de sables kaolinisés de bonne qualité qui peuvent convenir à l'élaboration d'une foule de produits contiennent principalement de la kaolinite ainsi que du quartz, et, dans bien des cas, du mica, du feldspath et de la montmorillonite. La montmorillonite et la kaolinite à grain fin ont surtout pour effet d'accroître la plasticité des argiles tandis que le quartz, le feldspath et le mica grossier en réduisent la plasticité. Les argiles les plus réfractaires contiennent surtout de la kaolinite, du quartz et, parfois, une petite quantité de gibbsite. Le mica se présente sous forme d'"illite", minéral argileux, ou sous forme de particules grossières de mica telles que celles de la muscovite. Le mica, le feldspath et la montmorillonite abaissent la qualité réfractaire des argiles de la formation Whitemud.

L'analyse thermique différentielle indique qu'il ne devrait pas y avoir de problème en ce qui concerne la cuisson des argiles Whitemud. Les minéraux accessoires tels que les matières carbonacées, la sidérose, la jarosite et le gypse ne devraient pas avoir d'effets défavorables parce qu'elles ne s'y trouvent qu'en faibles quantités. La composition des argiles Whitemud est telle que la gamme de cuisson est ordinairement de moyenne à longue, tandis que, pour bien d'autres argiles canadiennes, la gamme de cuisson est très courte.

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## INTRODUCTION

### Purpose

To the ceramic industries of Western Canada the Whitemud is one of the most important of the clay formations. It is made up of a wide variety of clays suitable for many applications in industry. Kaolinized sands, ball clays, fire clays, and stoneware clays from this formation have been utilized for many years because of their excellent properties. Extensive reports have been made on the geology and ceramic properties of these clays. Very little information has been reported on their mineralogical composition and, particularly, on the relationship of the mineralogical composition of the clays to their commercial utilization. Consequently, it is the purpose of this investigation to determine the ceramic properties and mineralogical composition of representative Whitemud formation clays and to examine the effect of their composition on commercial utilization. Many of the samples were obtained from existing pits that are being operated by industry. Other samples have been taken from areas containing clays of good quality that would be suitable for commercial use if proper beneficiating and processing techniques are developed, or markets become available.

### Geology

The Whitemud formation occurs in southern Saskatchewan and southeastern Alberta. It was named "Whitemud" by Davis(1) in 1918, principally because of the white or light-grey appearance of the outcrops, which are frequently high up on the sides of valleys.

According to Davis(1), the refractory nature of the clays was first realized about 1909. Prior to that time they were mentioned by Bell(2), Dawson(3), and McConnell(4). Rose(5) discussed the occurrence of Laramie clays, which at that time included the unnamed Whitemud formation, in the Wood Mountain-Willowbunch area of Saskatchewan. Dyer(6), McLearn(7,8,9,10), Fraser et al(11), Hutt(12), Williams and Dyer(13), and Furnival(14) have discussed the geology of the Whitemud clays in Saskatchewan. Dyer(15), Williams and Dyer(13), Russel(16), and Russel and Landes(17) have reported on the geology of the Whitemud formation in Alberta.

The Whitemud formation clays are of non-marine origin and were formed, according to McLearn(10), by sedimentation followed by chemical alteration after deposition. Davis(1) believed the sediments were eroded from the Precambrian in Manitoba, whereas McLearn(10) and Worcester(18) believed they came from the west, although Worcester in a private communication later stated that perhaps some of the eastern sediments came from the east. By 1927 McLearn(7) like some of the geologists preceding him had decided that there were four zones in the Whitemud formation, the type locality being along the valley of the Frenchman river near Whitemud post office in Saskatchewan. McLearn's zones, in ascending order, were as follows: (1) white, sandy clay zone, (2) brown to black shale zone, (3) white clay zone, and (4) upper dark

shale zone. Furnival(14) suggested that the upper dark zone, which he had found to be partly bentonitic and which showed no evidence of chemical alteration to kaolinite, should be named the "Battle" formation. The three lower zones, which are now considered to make up the Whitemud formation, vary in depth from area to area as a result of sedimentation, alteration, and erosion. In some areas one or more of the zones are entirely or partly absent. In the type localities, the lower Whitemud formation zone occurs immediately above the Eastend formation.

#### Previous Ceramic and Mineralogical Investigations

According to Worcester(18), the first test of the Saskatchewan light-coloured clays was conducted in 1907 by Edward Orton, Jr., of Columbus, Ohio, on 27 samples collected from the Dirt Hills by Daniel Diver, a prospector. In 1912 and 1913, Ries and Keele(19,20) described the properties of clays from the Laramie formation in Saskatchewan. Keele carried out further work in this province on Laramie clays in 1915(21) and, for the federal Mines Branch, on the renamed Whitemud formation clays in 1921(22).

In 1934, McLearn and McMahon(23) prepared a comprehensive report on the geology and ceramic properties of southern Saskatchewan Whitemud formation clays. The ceramic tests on the samples were supervised by J. G. Phillips, of the Mines Branch, who contributed a great deal to the knowledge of these clays during this period.

Worcester(18,24,25) made extensive field and laboratory studies on Saskatchewan Whitemud clays, and provided a thorough classification of them according to their ceramic uses. In general, he reported that the Whitemud clays in southwestern Saskatchewan, near Eastend at the eastern edge of the Cypress Hills, are of the stoneware-yellowware type. The Whitemud clays of south-central Saskatchewan, he found, are more varied in their properties, and consist of very refractory plastic fireclays, refractory sandy fireclays, high-strength, light-firing ball clays, kaolinized sand, and stoneware clays. Crockford(26), assisted by the Ceramic Section of the Mines Branch at Ottawa, has described the ceramic properties of numerous samples from the Whitemud formation in Alberta. Worcester(18,25), Humphreys and Brady(27), and Peterson and Tompkins(28) have described the physical properties of many of the better Whitemud ball clays.

Some mineralogical work has been done on the Whitemud formation clays. Little correlation has been made between mineralogical composition, ceramic properties (such as plasticity, workability, casting characteristics, refractoriness, fired colour, fired shrinkage, and fired water absorption), and the problems associated with processing clays and clay products. Fraser (29, 30,31,32,33) studied some 145 Whitemud formation clays by petrographic means. The Whitemud formation of Fraser's time also included the Battle formation (McLearn's upper dark zone), so that some of the upper samples would not be characteristic of the present Whitemud classification. However, he found that typical grey sands and white sandy clays of the present Whitemud formation contained feldspar, quartz, mica, and iron carbonate. In the minus 270 mesh fraction of the sands and sandy clay zones, characteristic heavy minerals, present in very minute amounts in Fraser's samples, were mainly zircon, tourmaline, and rutile. The majority of his Whitemud samples contained kaolinite grains. He found that the sands and sandy clays were non-calcareous



and non-dolomitic.

Brady et al(34) showed that kaolinite and quartz were the principal constituents of some Saskatchewan kaolins, ball clays and fire clays, all samples of which came from the Whitemud formation. They further noted that illite, gibbsite and montmorillonite could be identified in some of the clays. Brady et al(34,35) pointed out that there was some uncertainty in definitely establishing the presence of montmorillonite in some of the "as received" samples, that is, samples that were not fractionated with regard to particle size.

#### Development of the Whitemud Formation Clays

Stoneware clay, for many years, has been shipped from Eastend, Sask., principally to sewer pipe plants, potteries, and brick plants at Medicine Hat, Alta., and a plant at Estevan, Sask. Until recently it was believed that there was too much overburden on the stoneware-type Whitemud clays of Alberta. Recently, however, several pits have been developed in this area where it was found possible to remove the overburden economically and haul the clay to Medicine Hat. Stoneware clay from a pit at Avonlea, Sask., has been trucked to a plant in Regina for three years.

Refractory clays from the Dirt Hills and Cactus Hills, near Claybank, Sask., have been used for many years in the manufacture of refractories and face brick.

Ball clays from the Willows area have been used in whiteware, pottery and refractories manufacturing, principally at Medicine Hat, Estevan, and Vancouver. Some ball clays have been exported to the United States.

A great deal of work has been carried out in an effort to establish a suitable method of extracting a good quality of kaolin from the kaolinized sands of Saskatchewan. Spyker et al(36) investigated this problem for the Saskatchewan government, using air- and water-separation techniques. The federal Department of Mines and Resources at Ottawa(37) reported on the wet separation of kaolin from kaolinized sands at Wood Mountain and Knollys, Saskatchewan.

The Saskatchewan government outlined the extent of the Whitemud clays in many areas by an extensive drilling program(38,39). By this work it has been established that large quantities of good quality clay exist.

#### WHITEMUD FORMATION CLAYS SELECTED FOR INVESTIGATION

The most westerly Whitemud formation samples were obtained in the Alberta Cypress Hills area, approximately 45 miles southeast of Medicine Hat. The most easterly sample was obtained from Halbrite, Sask., southeast of Regina. Representative samples were obtained from the two principal zones: (1) the western or Cypress Hills area, which extends from southwestern Saskatchewan to

southeastern Alberta; and (2) the south-central Saskatchewan area, which lies principally south of Moose Jaw and extends from the Fir Mountain region east to Halbrite. In this report, whenever a series of clays is listed from the same area or project, the smallest number is always identified with the upper bed in the series and the clays are then numbered in order of increasing depth. The areas from which samples were obtained are shown in Figure 1. The numbered lines on the map represent highways.

### Stoneware Clays

#### Alberta Cypress Hills Clays

Two occurrences, "K" and "M", near Eagle Butte, in the Alberta Cypress Hills, were sampled. Six separate seams of a 30-foot section were investigated in area K, and five separate seams of a 22-foot section in area M. A composite mixture of the area K beds, which is herein designated the Alberta Cypress Hills Mixture (K), was also investigated.

#### Samples from Area K

K-1  
K-2  
K-3  
K-4  
K-5  
K-6

Alberta Cypress  
Hills Mixture (K)

#### Samples from Area M

M-1  
M-2  
M-3  
M-4  
M-5

### Eastend Clays

Five beds of stoneware-type clay, representing the upper 20 feet of Project 7(39) at Eastend, near the eastern edge of the Saskatchewan Cypress Hills, were investigated. In addition, a sample of the sewer pipe mixture from the Dempster pit at Eastend was studied.

#### Samples from Eastend

7-S-1  
7-S-2  
7-S-3  
7-S-4  
7-S-5

Eastend (Dempster)

### Avonlea Clays

Five beds of an outcrop at Avonlea, Sask., representing a total thickness of approximately 20 feet, were examined. The properties of a sewer pipe mixture made up from these stoneware-type clays and designated "Avonlea

S A S K A T C H E W A N

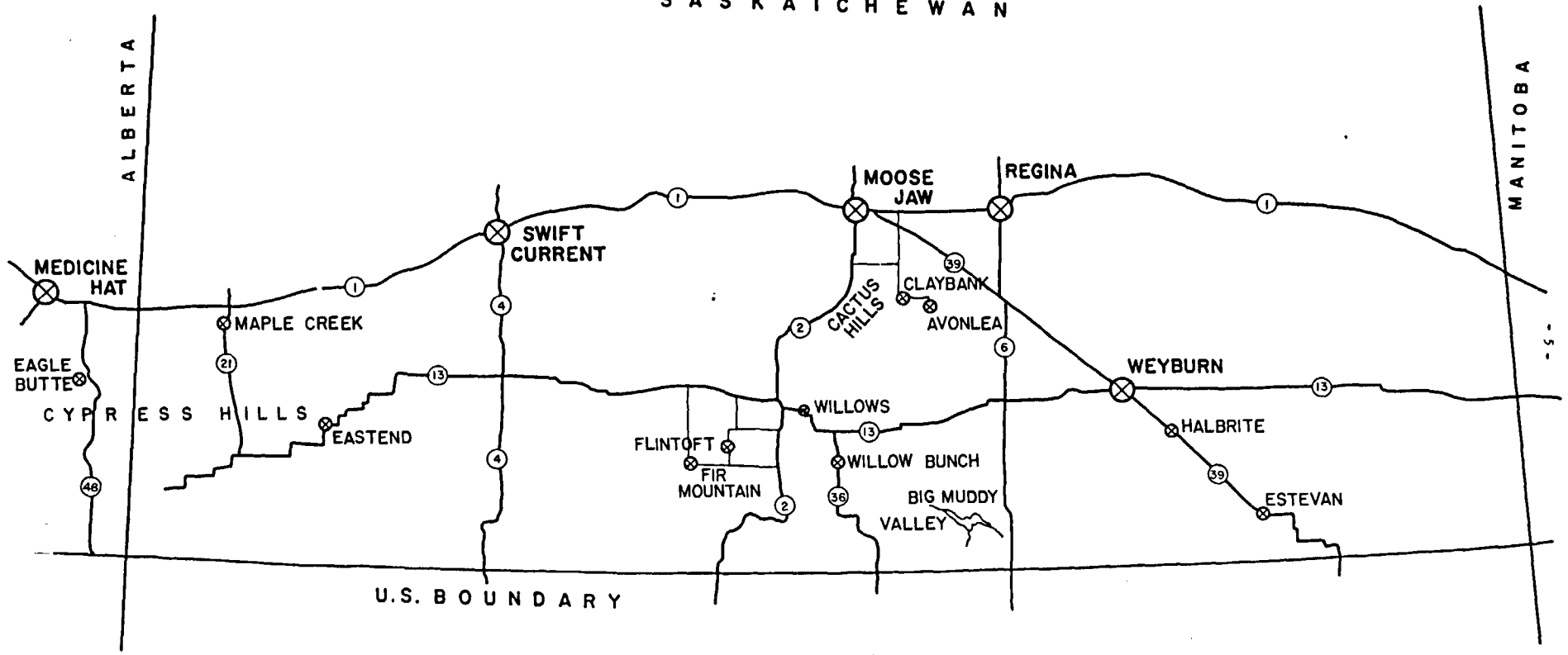


Figure 1. Sample Locations.

Mixture" were determined.

Samples from Avonlea

A-1  
A-2  
A-3  
A-4  
A-5  
Avonlea Mixture

Cactus Hills Fire Clays

Two typical refractory clays, one very plastic and the other sandy, from the Cactus Hills, near Claybank, Sask., are discussed. The plastic one is designated "Cactus Hills Plastic Fire Clay", and the sandy one is designated "Cactus Hills Sandy Fire Clay".

Kaolinized Sands

Various kaolinized sands that were described by Spyker et al.(36) were investigated. The majority of the samples are from Project 12(38,39), near Fir Mountain, Sask. The crude material from this area is designated as Sample 12-S-4&5. This sample was wet-separated by Spyker et al into a coarse clay fraction, a fine clay fraction, and a very fine clay fraction. The very fine fraction was referred to as C5. Samples K07 and K13 are air-separated samples, the former being from Sample 12-S-4&5 and the latter from a stoneware-type deposit at Knollys, near Eastend, Sask.

The core from a drill hole in the isolated Whitemud formation outcrop at Halbrite was investigated.

Designation of Kaolinized Sand Samples

12-S-4&5  
12-S-4&5 Coarse (minus 12 plus 2 microns)  
12-S-4&5 Fine (minus 2 microns)  
12-S-4&5 Very Fine (C5)  
K07  
K13  
Halbrite

Ball Clays

A great deal of work has been carried out by Worcester(18,25), Peterson and Tompkins(28), and Brady et al(34) on the ceramic properties and mineralogical composition of Saskatchewan ball clays. Some of the more important ball clays that were discussed by the above authors are dealt with in this report. Project 10 and Project 11 clays from Willows, Sask., and Project 15 clays from Flintoft, Sask., have been described by Peterson and Tompkins(28), Crawford and Carlson(38), and Brady et al.(34). Ball clays 2622, 92 and 10.

from the Willows area, and Midland ball clay from the Midland mine in the Willowbunch area, have been discussed by Worcester(18). No. 10 ball clay was described by Worcester(18) as having special properties making it suitable for the manufacture of saggars.

Project 10 Samples

10-S-1  
10-S-3  
10-S-5  
10-S-6  
10-S-7

Project 11 Samples

11-S-1  
11-S-3  
11-S-5

Project 15 Samples

15-S-1  
15-S-2  
15-S-3  
15-S-4

Miscellaneous Samples

Midland Ball Clay  
2622 Ball Clay  
10 Ball Clay  
92 Ball Clay

Big Muddy Valley Clays

Four refractory, plastic fire clays (two of which are similar to ball clays), and two stoneware clays occurring below the fire clays from an outcrop below 100 feet of overburden in the Big Muddy Valley, are included in the investigation. These samples were collected by the Saskatchewan Research Council in 1961 from NW 1/4 34-3-24 W2; the upper four are probably similar to Samples 3054, 3055, 3056 and 3057, collected by Worcester(18) in 1930.

Designation of Big Muddy Valley Samples

Big Muddy Fire Clay 6120  
Big Muddy Fire Clay 6121 (can be classed as a ball clay)  
Big Muddy Fire Clay 6122 (can be classed as a ball clay)  
Big Muddy Fire Clay 6123  
Big Muddy Stoneware Clay 6124  
Big Muddy Stoneware Clay 6125

Fractionated Samples

In addition to the fractions of 12-S-4&5 obtained by Spyker et al.(36), the less than 10 micron portion of a few clays was obtained by settling and centrifuging, and investigated mineralogically. The fractionated samples were designated with the original sample number plus a subscript 3A or 3B. The 3A portion was collected from the bottom part of a Sharples Super-Centrifuge bowl, and the 3B (finest) portion, from the upper part of the bowl.

Designation of Fractionated Samples

Cactus Hills Plastic Fire Clay (3A) Fraction  
 10-S-1 (3A) Fraction  
 11-S-1 (3A) Fraction  
 11-S-5 (3A) Fraction  
 15-S-3 (3A) Fraction  
 7-S-1 (3B) Fraction  
 7-S-5 (3A) Fraction  
 Eastend (Dempster) (3A) Fraction  
 A-4 (3A) Fraction  
 K-4 (3A) Fraction  
 Big Muddy 6121 (3A) Fraction

METHODS OF INVESTIGATION

Ceramic Evaluation

Such physical properties of Whitemud formation clays as plasticity, workability, drying shrinkage, fired shrinkage, fired colour, fired hardness, amount of water absorbed by the fired samples, and pyrometric cone equivalent (PCE) or refractoriness, have been reported by McLearn and McMahon(23), and Worcester(18). For the present work, the physical properties of some representative samples of the individual beds from selected Whitemud formation occurrences, and of some typical fire clays, ball clays, stoneware clays and kaolinized sands, are investigated. Procedures used in determining the properties were described previously by Brady(40), where plasticity and workability were empirically judged by a technician. The firing temperatures vary, but are usually cones 06(991°C), 04(1050°C), 02(1101°C), 2(1142°C), 3(1152°C), 5(1177°C), or 10(1285°C).

Special techniques to determine shrinkage and water absorption by the temperature gradient method (41, 42), and plasticity (resistance to shear) by the Brabender Plastograph (43), were used to obtain temperature gradient and plasticity curves of a few of the typical Whitemud formation samples. The procedure and applications of these methods to Canadian clays have been discussed by Brady(44).

Plasticity measurements by the Brabender Plastograph, unless otherwise noted, were made on mixtures of 50% clay sample and 50% potter's flint. Water was added at the rate of 5 ml per minute to a 200-gram dry batch. The flint addition reduced the plasticity of some of the very plastic samples so that the entire peaks could be retained on the chart. A Dundas (referred to occasionally in the literature as 'Lorraine') shale, which has just sufficient plasticity for the production of extruded brick and tile, was used as a standard. The peak heights and the areas under the curves were used as a basis for comparing the plasticities of the clays: the higher the peak and the greater the curve area, the greater is the plasticity. This method of determining relative plasticity is more exact than the empirical method that depends on human



judgment.

### Mineralogical Composition of the Clays

The mineralogical composition of the clays was determined principally by X-ray diffraction (XRD), differential thermal analysis (DTA), and microscopic means. Mineralogical balances were made of a few clays by using the mineralogical composition in combination with chemical analysis. Most of the procedures and apparatuses used were discussed previously by Brady(40,44). Satisfactory DTA thermograms were obtained of the clays by heating the samples in an air atmosphere. A horizontal nichrome-wound furnace was used instead of the Stone atmosphere-controlled furnace previously described by Brady(40). A Philips X-ray diffractometer was used for the XRD investigation. Glycolation and heating techniques were employed in the XRD work, when necessary, to identify certain clay minerals. Packed mounts were used for most of the "as received" samples, and oriented mounts for most of the fractionated samples.

The mineralogical compositions of the plus 200 mesh (Tyler) fraction of a few selected samples were determined by microscopic examination and XRD. Certain clays were fractionated by settling approximately the plus 10 micron fraction and then removing the approximately minus 10 micron fraction from suspension by a Sharples laboratory Super-Centrifuge. In certain cases this procedure concentrated clay minerals, such as montmorillonite, which were believed to be present in small quantities in the fine fraction of some of the samples.

Separation of kaolin from the kaolinized sands was not attempted. However, some of the important samples described by Spyker et al.(36) in the publication entitled "Separation of Kaolin from Kaolinized Sand" were obtained, and are discussed in this report.

### Particle Size

The complete particle size distribution data on the samples were not determined. The per cent plus 200 mesh (Tyler) fractions of several typical samples that were fractionated for mineralogical purposes were calculated. Previous work by Humphreys and Brady(27) had established particle sizes, by the Andreason pipette method, for some Saskatchewan ball clays. Spyker et al.(36) obtained particle sizes, using a Bouyoucos hydrometer, for kaolinized sand 12-S-4&5 and the beneficiated fractions 12-S-4&5 Coarse and 12-S-4&5 Fine. Worcester(18,25) determined the percentages of clay substance and sand for 26 Saskatchewan ball clays from the Whitemud formation.

## RESULTS

### Ceramic Properties

The unfired and fired characteristics of stoneware clays from the Alberta and Saskatchewan Cypress Hills are shown in Tables 1 and 2. The majority of the stoneware samples are plastic and have good workability.

TABLE 1

Physical Properties of Stoneware Clays from the Alberta Cypress Hills

Clay No.	UNFIRED CHARACTERISTICS	P.C.E.*	FIRED CHARACTERISTICS					REMARKS
			Cone No.	Fired Shrinkage %	Absorption %	Colour	Hardness	
K-1	Alberta Cypress Hills Area K Grey clay, very plastic and greasy, water of plasticity 26.7%, cracks in drying, drying shrinkage 7.6%.	23 (1605°C)	06	1.0	14.8	White	Fairly hard	Difficult to dry stoneware clay; vanadium efflorescence (V efflor.**).
			02	3.3	10.0	Cream	Hard	
			2	4.0	8.8	Light buff	Very hard	
K-2	Grey to white clay, fair plasticity, water of plasticity 18.1%, slight cracks in rapid drying, drying shrinkage 4.8%, contains considerable grit.	20 (1564°C)	02	0.7	10.2	Light cream	Fairly soft	Open-firing stoneware clay which contains a high proportion of non-plastic material; V efflor.**
			2	1.0	9.1	Cream	Fairly hard	
			5	1.3	8.2	Cream	Fairly hard	
K-3	Light brown to grey clay, good plasticity, water of plasticity 24.7%, slight tendency to crack with rapid drying, drying shrinkage 7.8%.	28 (1646°C)	06	1.1	14.0	White	Fairly hard	Refractory stoneware or low-duty fire clay; V efflor.**
			02	3.5	10.2	Light cream	Hard	
			2	4.5	8.4	Cream	Very hard	
K-4	White clay, plastic, water of plasticity 24.2%, cracks slightly with rapid drying, drying shrinkage 6.1%.	17 (1512°C)	06	0.2	13.9	White	Fairly hard	Stoneware clay which is inclined to be difficult to dry; V efflor.**
			02	3.3	8.8	Cream	Hard	
			2	3.8	7.4	Light buff	Very hard	
K-5	White clay, short and gritty, water of plasticity 21%, safe drying, drying shrinkage 5%.	19 (1541°C)	06	0	13.5	White	Fairly soft	Open-firing, non-plastic stoneware clay; V efflor.**
			02	0.7	11.5	White	Fairly hard	
			2	1.3	9.9	White	Fairly hard	
			5	1.8	9.0	Light cream	Hard	
K-6	Light buff clay, non-plastic, water of plasticity 10%, safe drying, drying shrinkage 1.5%.	16 (1491°C)	02	0.3	14.5	Brown-buff	Soft	Non-plastic, sandy material.
			2	0.5	15.0	Brown-buff	Soft	
			5	1.3	15.0	Brown	Fairly hard	
Alberta Cypress Hills Mixture (K)	Stoneware-type whitemud clay, fairly plastic, extrudes well under vacuum but body is inclined to be weak, water of plasticity 18.5%, slight tendency to crack with rapid drying, drying shrinkage 7%.	20	04	0.6	12.2	Cream	Fairly hard	Stoneware-type clay with a high proportion of non-plastic material. The plastic portion dries with difficulty. It has a long firing range. V efflor.**
			02	1.5	9.8	Buff	Fairly hard	
			2	2.4	7.6	Buff	Hard	
			6	3.5	5.6	Cream-grey	Hard	
			8	4.1	4.3	Light grey	Very hard	
M-1	Alberta Cypress Hills Area M Brown clay, tough plastic clay, water of plasticity 28.3%, cracks badly in drying, drying shrinkage 10.0%.	20	06	1.1	12.4	Light buff	Fairly hard	Very difficult to dry stoneware clay; V efflor.**
			02	2.5	9.5	Light buff	Hard	
			2	2.5	8.8	buff	Very hard	
			5	3.5	8.2	buff	Very hard	
M-2	White clay, sandy material but inclined to be sticky, water of plasticity 17%, cracks in drying, drying shrinkage 2.0%.	18 (1522°C)	02	0.3	10.4	Light buff	Fairly soft	Difficult to dry, open-firing stoneware clay; V efflor.**
			2	0.3	9.9	Light buff	Fairly soft	
			5	2.0	9.4	Light buff	Fairly hard	
M-3	Brown clay, very plastic, tough, and sticky, water of plasticity 34.7%, cracks in drying, drying shrinkage 11.7%.	16	06	2.5	10.5	Salmon	Hard	Very difficult clay to dry.
			02	5.7	4.4	Salmon	Very hard	
			2	6.3	3.6	Brown-red	Very hard	
M-4	White clay, plastic, water of plasticity 25.8%, slight tendency to crack in drying, drying shrinkage 6.8%.	14 (1398°C)	06	0.8	14.1	Cream	Fairly hard	Stoneware clay with a medium firing range; V efflor.**
			02	5.0	4.1	Light buff	Very hard	
			2	7.1	3.2	buff	Steel hard	
M-5	Brown to grey clay, very plastic and sticky, water of plasticity 37.5%, very difficult to dry, drying shrinkage 12.2%.	14	06	2.0	8.8	Light buff	Hard	Very difficult clay to dry.
			02	5.7	1.5	Brown-buff	Steel hard	
			2	6.3	0.6	Brown-red	Steel hard	

\* P.C.E. - pyrometric cone equivalent (heat softening point).

\*\* V efflor. indicating vanadium efflorescence was observed after the trial pieces were saturated with water and then dried.

TABLE 2

Physical Properties of Eastend, Saskatchewan, Stoneware Clays

Clay No.	UNFIRED CHARACTERISTICS	FIRED CHARACTERISTICS					REMARKS	
		P.C.E.	Cone No.	Fired Shrinkage %	Absorption %	Colour		Hardness
7-S-1	Eastend, Saskatchewan Grey clay, good plasticity, water of plasticity 27.2%, slight tendency to crack with rapid drying, drying shrinkage 7.3%.	26 (1621°C)	06	0.9	15.2	Very pale cream	Fairly hard	Low-duty fire clay which is difficult to dry; V efflor.**
			02	2.9	12.1	Cream	Hard	
			3	4.0	10.6	Cream	Hard	
			5	4.5	9.1	Cream	Very hard	
7-S-2	White clay, good workability and plasticity, water of plasticity 22.5%, satisfactory in rapid drying, drying shrinkage 5.6%.	19 (1541°C)	06	-0.3*	13.4	Nearly white	Fairly hard	Sandy stoneware clay.
			02	1.3	10.7	Light cream	Hard	
			3	2.5	7.9	Creamy buff	Very hard	
			5	2.7	7.5	Creamy buff	Very hard	
7-S-3	White clay, good workability and plasticity, water of plasticity 25%, satisfactory in rapid drying, drying shrinkage 6.6%.	17+ (1517°C)	06	0	14.1	Nearly white	Fairly hard	Stoneware clay; V efflor.**
			02	2.0	10.3	Cream	Hard	
			3	3.0	7.8	Light buff	Very hard	
			5	3.7	5.2	Light buff	Steel hard	
7-S-4	Cream, good workability and plasticity, water of plasticity 35%, satisfactory in rapid drying, drying shrinkage 7.7%.	14+ (1412°C)	06	0.7	14.5	Light salmon buff	Fairly hard	Scums, low grade stoneware clay.
			02	4.5	8.0	Salmon buff	Hard	
			3	6.7	2.4	Partly brownish and salmon buff	Steel hard	
7-S-5	Light grey clay, very plastic (somewhat sticky), water of plasticity 34.7%, slight tendency to crack with rapid drying, drying shrinkage 9.4%.	18+ (1530°C)	06	0.9	13.1	Light salmon buff	Fairly hard	Similar to 7-S-4 except it is difficult to dry.
			02	2.5	8.4	Salmon buff	Hard	
			3	3.5	6.8	Darker salmon buff	Very hard	
Eastend (Dempster) Mixture	Stoneware-type Whitened clay, plastic, extrudes satisfactorily under vacuum, water of plasticity 22%, safe drying, drying shrinkage 6.5%.	19+ (1550°C)	06	0.9	14.9	Cream	Fairly hard	Plastic, easily-dried, stoneware clay with a fairly long firing range; V efflor.**
			04	2.3	12.2	Cream	Hard	
			02	4.5	8.0	Yellow-cream	Very hard	
			2	6.8	3.8	Grey-cream	Steel hard	
			6	8.3	1.0	Grey	Steel hard	

\* A minus sign indicates expansion.

\*\* V efflor. indicates vanadium efflorescence was observed after the trial pieces were saturated with water and then dried.

Drying shrinkages are inclined to be high and, consequently, many of the samples are difficult to dry under rapid-drying conditions. The majority of the clays vitrify readily, and fire to a white, cream, grey, buff, or brown colour. Most Whitemud samples from this area and from other southern Saskatchewan areas contain soluble vanadium salts which appear as a yellow to green efflorescence.

The Avonlea stoneware clays whose properties are shown in Table 3 are, in general, similar to the clays in Tables 1 and 2. Physical properties for typical fire clays, ball clays and kaolinized sands, and for a stoneware clay from the south-central portion of Saskatchewan, are shown in Table 4. The ceramic properties of the plastic fire clays from the Cactus Hills and Big Muddy Valley, and of the ball clays, indicate (Table 4) that these clays are very plastic, are difficult to dry, and vitrify at high temperatures. The ball clays, particularly, are tough, greasy, and inclined to be sticky. The Halbrite, Cactus Hills Sandy Fire Clay, and 12-S-4&5 kaolinized sand (Table 4) are the least plastic, yet have fair workability. They dry safely and are open-firing clays, probably because of the nature of their non-plastic ingredients. All samples in Table 4 fire to a white, cream, grey, or buff colour.

The Brabender Plastograph curves of the 50% clay - 50% potter's flint mixtures are shown in Figures 2,3,4 and 5, where per cent tempering water is plotted against resistance to pugging action (resistance to shear). The peak heights and the areas under the curves are shown in Table 5. The latter areas were used by Marshall(43) as a measure of plasticity. The peak of each curve indicates the water content required for maximum resistance to pugging action or, in effect, maximum plasticity.

Experiments with various clays, a standard shale and some non-plastic Queenston shales have indicated that, in order to extrude satisfactorily, the material should have a peak height of at least 500 units, and a minimum curve area of at least 6 to 7 sq in., under the laboratory conditions employed. The curve for the Dundas shale standard sample, shown in Figure 2, has a broad peak base, a peak amplitude of approximately 500 units, and an area under the curve of 8.50 sq inch. The curves for the Dundas-flint and Halbrite-flint mixtures (Figure 2) indicate that the addition of an equal amount of flint to a clay or shale sample reduces the peak height and the area under the curve to approximately two-fifths to three-fifths of the original value. In general, the per cent tempering water shown for the clay-flint mixtures in Figures 2,3,4 and 5, is several per cent lower than the per cent tempering water (water of plasticity) required for the clays undiluted with flint (Tables 1,2,3,4).

The results shown in Table 5 indicate that if the samples were undiluted with flint their plasticity should be greater, and in some cases much greater, than Dundas 100%. Thus, there is a wide range of plasticities among the Whitemud formation clays, the majority having ample plasticity for extruding. The plastograph curves vary considerably in shape, particularly in the upper portion where maximum resistance to shear, or maximum plasticity, is achieved. The ball clay peak heights and the areas under the ball clay curves are, on the average, considerably higher than those for the fire clays, stoneware clays or kaolinized sands, and indicate that the ball clays are very plastic materials.

TABLE 3

Physical Properties of Avonlea, Saskatchewan, Stoneware Clays from South-Central Saskatchewan

Clay No.	UNFIRED CHARACTERISTICS	P.C.E.	FIRED CHARACTERISTICS					REMARKS
			Cone No.	Fired Shrinkage %	Absorption %	Colour	Hardness	
A-1	White clay, good workability, very plastic, water of plasticity 25.8%, tendency to crack with rapid drying, drying shrinkage 6.4%.	29 (1659°C)	04	1.3	15.7	Cream	Fairly hard	Medium-duty fire clay or stoneware clay, plastic; V efflor.*
			02	2.2	13.1	Cream	Hard	
			1	3.7	11.2	Darker cream	Hard	
			5	4.3	9.3	Darker cream	Very hard	
A-2	Buff clay, good workability, very plastic, water of plasticity 27.8%, cracks badly with rapid drying, drying shrinkage 6.5%.	19 (1541°C)	04	3.0	9.2	Buff	Hard	White efflorescence; plastic stoneware clay, care required in drying.
			02	4.3	6.0	Buff	Very hard	
			1	5.0	5.4	Buff	Very hard	
			5	6.0	1.3	Buff	Steel hard	
A-3	Grey, sandy clay, good workability, plastic (gritty), water of plasticity 24.8%, cracks badly with rapid drying, drying shrinkage 6.6%.	26 (1621°C)	04	0.9	14.4	Pinkish cream	Fairly soft	Low-duty fire clay; care required in drying; V efflor.*
			02	1.7	12.5	Speckled cream buff	Fairly hard	
			1	2.2	11.3	Speckled cream buff	Fairly hard	
			5	2.9	9.8	Speckled cream buff	Hard	
A-4	Buff clay, fair workability, plastic and sticky, water of plasticity 38.9%, cracks with slow drying, drying shrinkage 10.2%.	17+ (1517°C)	06	0.7	14.3	Salmon buff	Fairly hard	White efflorescence; low-grade stoneware, or common clay, very difficult to dry.
			04	3.2	9.8	Salmon buff	Hard	
			02	5.2	5.3	Brownish buff	Very hard	
			1	5.7	4.2	Brownish buff	Very hard	
A-5	Grey clay, good workability, very plastic, water of plasticity 29.2%, slight tendency to crack with slow drying, drying shrinkage 6.7%.	16+ (1500°C)	06	0.3	14.7	Light salmon	Fairly soft	White efflorescence; low-grade stoneware, or common clay; very difficult to dry.
			04	1.3	11.7	Light salmon buff	Medium hard	
			02	3.0	8.1	Salmon buff	Hard	
			1	3.0	7.7	Salmon buff	Hard	
Avonlea Mixture Suitable for Sewer Pipe	Grey clay mixture, good workability, very plastic, water of plasticity 26.2%, cracks with rapid drying, drying shrinkage 7.5%.	20-23 (1580°C)	06	0.8	13.5	Pink buff	Fairly hard	Typical stoneware clay, has a tendency to scum, care required in drying; V efflor.*
			04	1.8	10.6	Pink buff	Hard	
			02	3.0	8.2	Buff	Very hard	
			1	3.8	6.0	Dark buff	Very hard	
			5	4.6	4.3	Greyish buff	Steel hard	

\* V efflor. indicating vanadium efflorescence was observed after the trial pieces were saturated with water and then dried.



TABLE 4  
Physical Properties of Selected Kaolinized Sands, Sandy Fire Clay,  
Plastic Fire Clays, Ball Clays and Stoneware Clay from South-Central Saskatchewan

Clay No.	UNFIRED CHARACTERISTICS	P.C.E.	FIRED CHARACTERISTICS					REMARKS
			Cone No.	Fired Shrinkage %	Absorption %	Colour	Hardness	
Halbrite Kaolinized Sand	Light grey clay, fairly good plasticity and workability, water of plasticity 20.0%, safe drying, drying shrinkage 4.3%.	30 (1665°C)	02	0.3	14.7	Nearly white	Fairly soft	An open-firing fire clay or kaolinized sand. The fired samples have black specks.
			5	1.0	11.1	Greyish white	Fairly soft	
			10	2.3	9.2	Light grey	Hard	
12-S-4&5 Kaolinized Sand	Nearly white clay, fairly good workability and plasticity, gritty, water of plasticity 23.3%, safe drying, drying shrinkage 5.7%.	31 (1683°C)	02	1.0	16.7	White	Fairly soft	Similar type of clay to the Sandy Fire Clay, fired briquettes have black specks; V efflor.*
			5	2.5	13.4	White	Fairly hard	
			10	3.2	11.9	White	Hard	
Cactus Hills Sandy Fire Clay	Light grey clay, works well, fairly plastic, gritty, water of plasticity 26.5%, very small cracks in rapid drying, drying shrinkage 5.1%.	31	02	1.0	17.6	Greyish white	Fairly soft	Open-firing fire clay. Fired briquettes have black specks; V efflor.* Sample similar to kaolinized sands.
			5	1.8	15.0	Greyish white	Fairly soft	
			10	3.3	17.4	Light grey	Fairly hard	
Cactus Hills Plastic Fire Clay	Light grey clay, good plasticity, works well, tough, water of plasticity 30.0%, cracks with rapid drying, drying shrinkage 6.5%.	32 + (1720°C)	02	1.7	18.1	White	Fairly hard	Fired briquettes have some brown specks. This is a high-quality refractory bond clay.
			5	3.7	9.3	White	Hard	
			10	10.3	5.0	Off white	Very hard	
2622 Ball Clay	Grey to brown clay, plastic (tough and greasy), water of plasticity 26.4%, tendency to crack with rapid drying, drying shrinkage 7.3%.	32 (1717°C)	06	2.0	15.8	White	Fairly hard	There were no discolouring specks, after firing. This is a white-firing ball clay, V efflor.*
			5	6.5	7.2	White	Hard	
			10	7.5	2.0	cream	Very hard	
10-S-1 Ball Clay	Light grey clay, good workability and very plastic, water of plasticity 30.6%, slight cracking with rapid drying, drying shrinkage 8.3%.	32	02	5.5	12.7	Off white	Hard	Brown specks at cone 10 and some V efflor.*; an off-white-firing ball clay which has a white scum.
			5	9.8	3.3	Off white	Steel hard	
			10	10.3	1.2	Off white	Steel hard	
11-S-1 Ball Clay	Light grey clay, good workability and very plastic, water of plasticity 32.3%, cracks in rapid drying, drying shrinkage 9.7%.	30 + (1670°C)	02	4.5	11.5	Off white	Very hard	Same comments as 10-S-1.
			5	7.7	3.6	Off white	Steel hard	
			10	8.3	1.4	Off white	Steel hard	
10 Ball Clay	Cream clay, good workability and plasticity, water of plasticity 29.7%, safe drying, drying shrinkage 7.0%.	30 +	02	2.7	14.2	Nearly white	Hard	This is a white-to-cream firing material which has some specks; V efflor.*
			5	5.5	10.1	Nearly white	Very hard	
			10	7.2	4.4	Very pale grey	Steel hard	
Midland Ball Clay	Grey clay, very plastic, water of plasticity 33.4%, cracks badly in drying, drying shrinkage 10.8%.	32	02	5.7	10.7	White	Very hard	This is a white-firing ball clay; V efflor.*
			5	8.5	4.7	White	Steel hard	
			10	9.3	3.0	White	Steel hard	
Big Muddy Fire Clay 6120	Light grey clay, good workability and plasticity, water of plasticity 28.1%, small cracks with rapid drying, drying shrinkage 7.2%.	31 + (1690°C)	02	6.0	14.5	Light cream	Hard	Fired sample had many specks. This is a refractory, plastic fire clay; V efflor.*. 6123 has the same P.C.E.
			5	7.8	9.6	cream	Very hard	
			10	9.0	5.9	Rusty white	Steel hard	
Big Muddy Fire Clay 6121	Greyish brown clay, good workability, good plasticity, somewhat greasy and tough, water of plasticity 28.8%, cracks rather badly with rapid drying, drying shrinkage 7.9%.	32+	02	7.5	10.8	cream	Very hard	Tendency to scum. This is a very refractory plastic fire clay which is similar to a ball clay; V efflor.*. Big Muddy 6122 is similar to 6121.
			5	8.9	6.0	cream (some specks)	Steel hard	
			10	9.9	3.5	cream (some specks)	Steel hard	
Big Muddy Stoneware Clay 6125	Dark grey clay, good workability and plasticity, water of plasticity 27.8%, slight tendency to crack with rapid drying, drying shrinkage 8.6%.	20-23 (1580°C)	02	4.3	11.8	Light salmon buff	Hard	This is a plastic stoneware clay which scums and has some specks, particularly at cone 10; V efflor.*. Big Muddy 6124 is similar to 6125.
			5	5.5	8.1	Light salmon buff	Very hard	
			10	7.0	3.2	Dark salmon buff (specks)	Steel hard	

\* V efflor. indicating vanadium efflorescence was observed after the trial pieces were saturated with water and then dried.



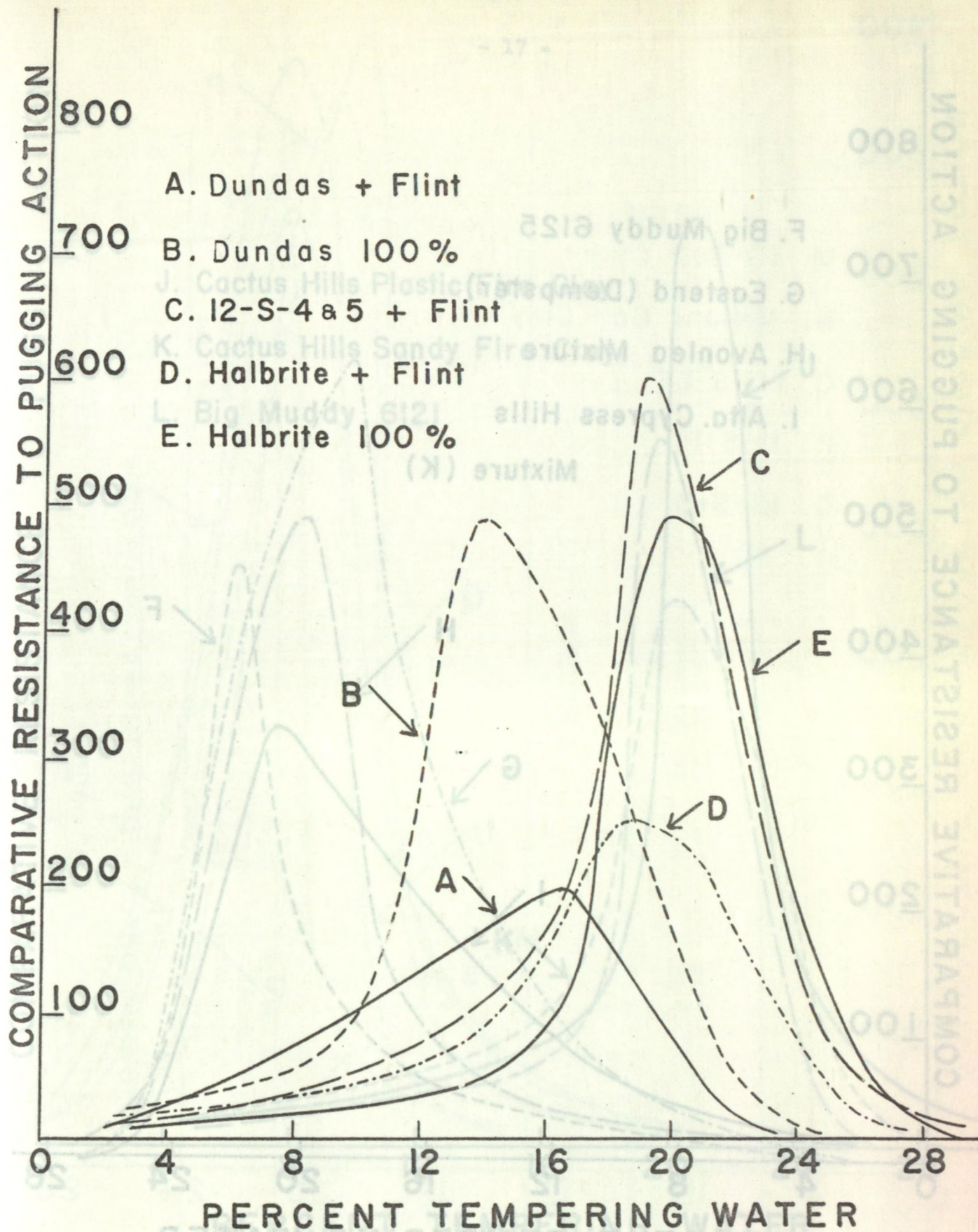
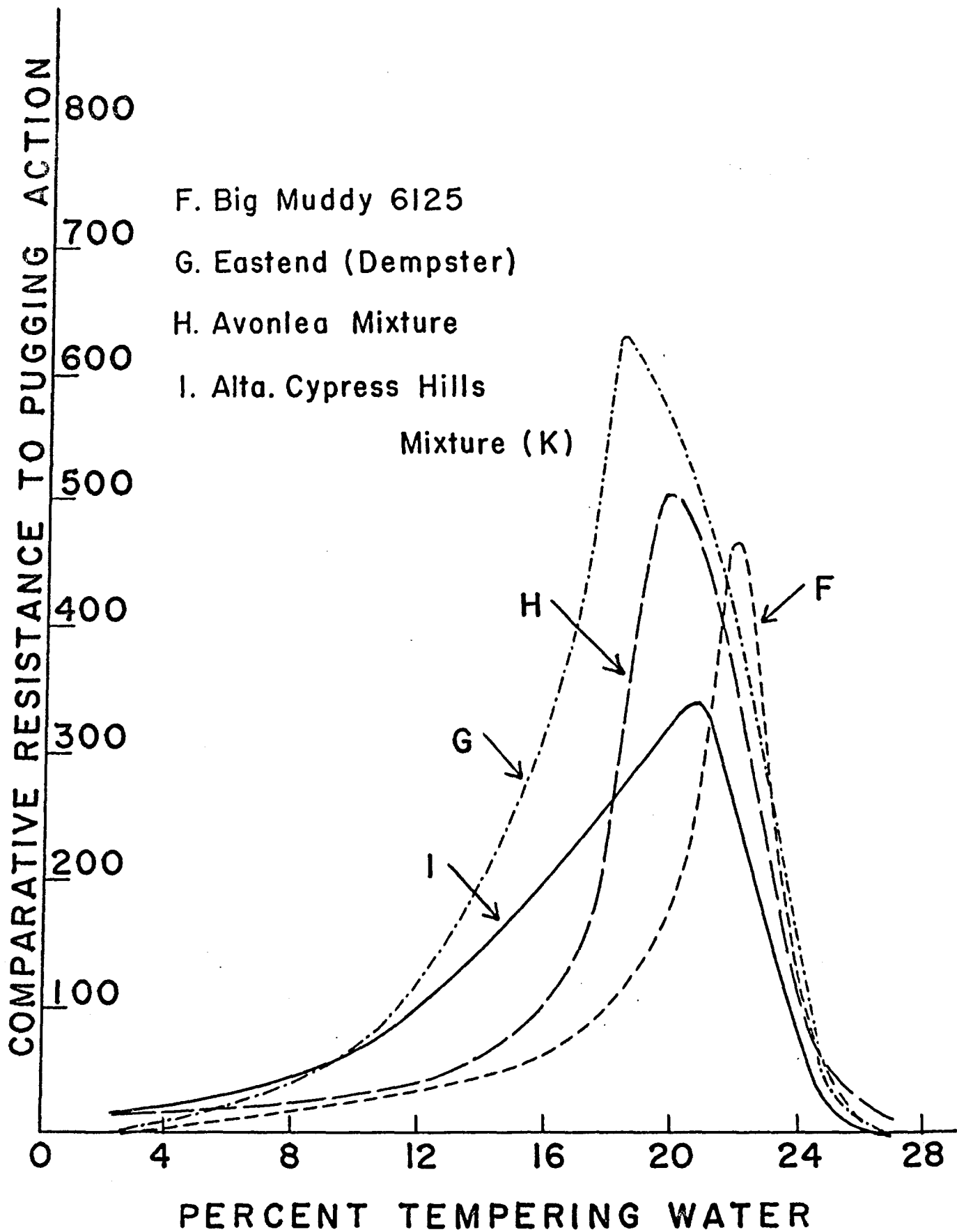
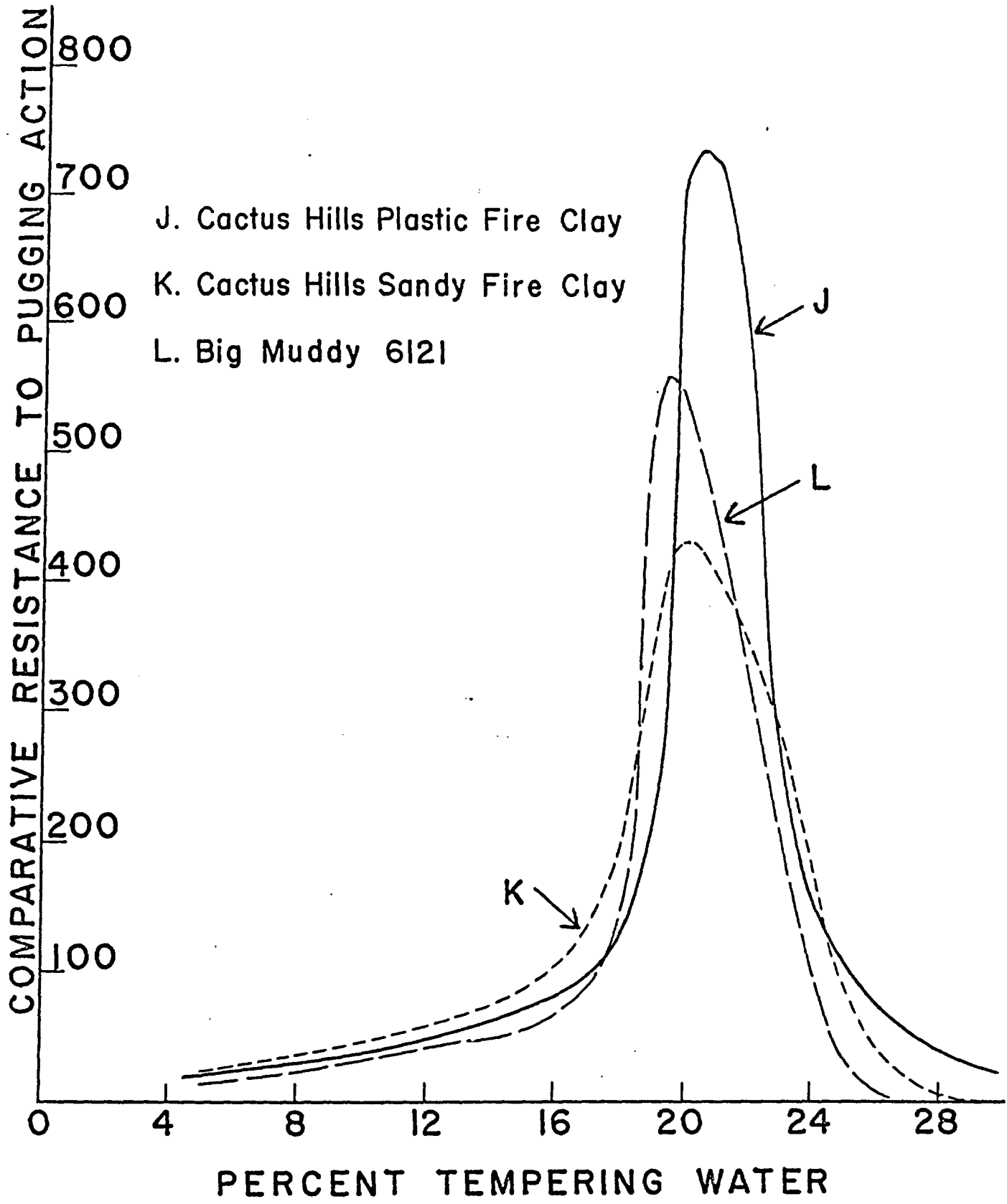


Figure 2. Plasticity Curves of Standard Dundas and Kaolinized Sands.



Note: All Samples Contain 50% Flint

Figure 3. Plasticity Curves of Stoneware Clays.



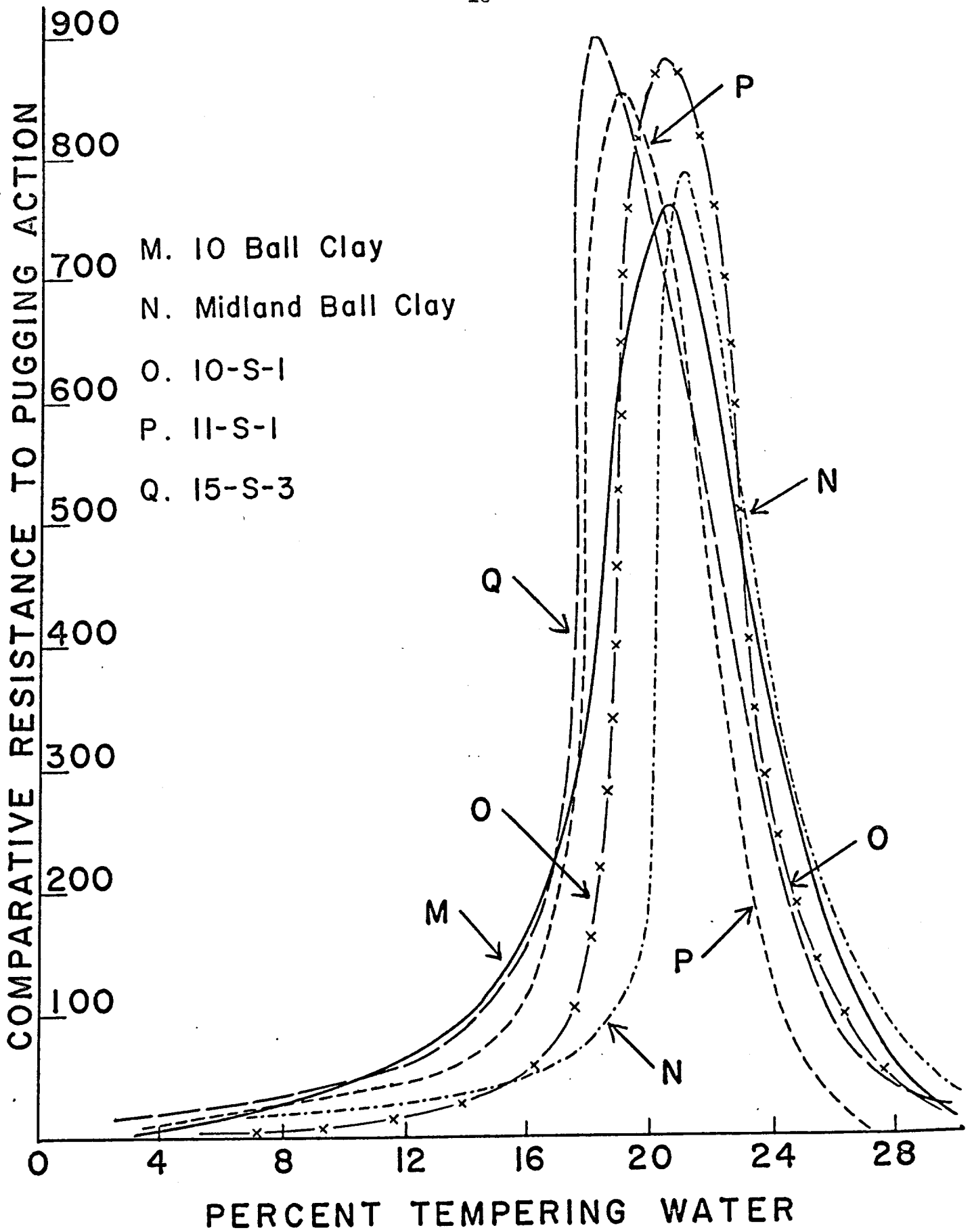
J. Cactus Hills Plastic Fire Clay

K. Cactus Hills Sandy Fire Clay

L. Big Muddy 6121

Note: All Samples Contain 50% Flint

Figure 4. Plasticity Curves of Fire Clays.



- M. 10 Ball Clay
- N. Midland Ball Clay
- O. 10-S-1
- P. 11-S-1
- Q. 15-S-3

Note: All Samples Contain 50% Flint

Figure 5. Plasticity Curves of Ball Clays.

TABLE 5

Relative Plasticity of Typical Whitemud Formation Clays\*

Sample	Area Under Plastograph Curve Sq. In.	Peak Height Chart Units	Sample	Area Under Plastograph Curve Sq. In.	Peak Height Chart Units
<u>Standard Dundas and Kaolinized Sands</u>			<u>Fire Clays</u>		
Dundas 100%	8.50	500	Cactus Hills Plastic	7.70	740
Dundas + Flint	4.55	210	Cactus Hills Sandy	6.10	440
Halbrite 100%	7.30	500	Big Muddy 6121	5.50	560
Halbrite + Flint	4.85	260			
12-S-4&5	3.30	600			
<u>Stoneware Clays</u>			<u>Ball Clays</u>		
Avonlea Mixture	6.15	520	10	11.85	760
Eastend (Dempster)	9.10	560	11-S-1	9.30	850
Alberta Cypress Hills Mixture (K)	6.20	360	15-S-3	14.60	900
Big Muddy 6125	5.30	480	Midland	8.40	790
			10-S-1	11.30	880

\* All plastograph samples are 50% clay - 50% flint except Halbrite 100% and the Standard Dundas 100%.



The various temperature-gradient curves for shrinkage and water-absorption, the significance of which is discussed by McNamara and Dulberg (45), are shown in Figures 6, 7, and 8. A negative shrinkage value indicates some residual expansion (expansion after firing and cooling), which is probably caused by a quartz inversion at 573°C (1062°F) and perhaps the expulsion of combined water (44). The curves in Fig. 6 for Alberta Cypress Hills Mixture (K) and Eastend (Dempster), and the curve in Fig. 8 for Avonlea Mixture, indicate that these materials are vitrifying clays, of the stoneware type, having moderate to high shrinkage values. The plastic fire clay curve in Fig. 7 indicates that the clay vitrifies gradually at a high temperature with a very large amount of shrinkage. The curve of 11-S-1 ball clay (Fig. 8) shows that this is a typical vitrifying clay of the ball clay type as described by McNamara and Dulberg(45). Curves for Cactus Hills Sandy Fire Clay (Fig. 7) and 12-S-4&5 kaolinized sand (Fig. 8) indicate that these clays are non-plastic, low-shrinkage, refractory materials that do not vitrify readily.

### Differential Thermal Analysis

The DTA curves are shown in Figures 9 to 17, inclusive. API Kaolinite H-4, described by Kerr et al.(46), was selected as a standard. Kaolinite -  $\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$  - has a percentage composition of 46.3%  $\text{SiO}_2$ , 39.8%  $\text{Al}_2\text{O}_3$ , and 13.9%  $\text{H}_2\text{O}$ . The DTA curve for the kaolinite standard is shown in Fig. 16. In these figures endothermic peaks point down and exothermic peaks point up. Kaolinite peaks occur on the H-4 standard curve, and on all curves of the test samples, at approximately 150°C (302°F), 580°C (1076°F) and 970°C (1778°F). At approximately 150°C (302°F), the curves of the Whitemud formation samples have as large, or larger, peaks than the standard. This is probably because the clay mineral or clay minerals in the samples contain as much, or more, absorbed moisture than the standard. The test clay curves have smaller peaks than the standard at approximately 580°C (1076°F) and shorter and duller ones at approximately 970°C (1778°) because the clays are more heterogeneous and contain less kaolinite than the standard. In general, the greater the peak area at 580°C (1076°F) and the sharper the peak at 970°C (1778°F), the greater the kaolinite content.

The approximate kaolinite percentage was obtained by determining the area per gram under the 580°C (1076°F) peak and comparing this with a standard kaolinite curve. The very small peak area produced by mica and chlorite in some of the clays at this temperature was negligible. The API H-4 kaolinite standard was not satisfactory for clays having a high percentage of very small clay particles. According to Speil et al.(47), the peak area decreases with particle size. Consequently, sample 10-S-1(3A), which consists of minus 10 micron particles, was selected for a standard to determine the kaolinite percentages of samples known to have, or suspected of having, a large proportion of small kaolinite particles. It was calculated that 10-S-1(3A) contained 86% kaolinite. This standard was used particularly with ball clays, plastic fire clays, and fractionated samples that consisted mainly of kaolinite and quartz. Kaolinite percentages obtained with the 10-S-1(3A) standard were higher than those obtained from the H-4 standard.



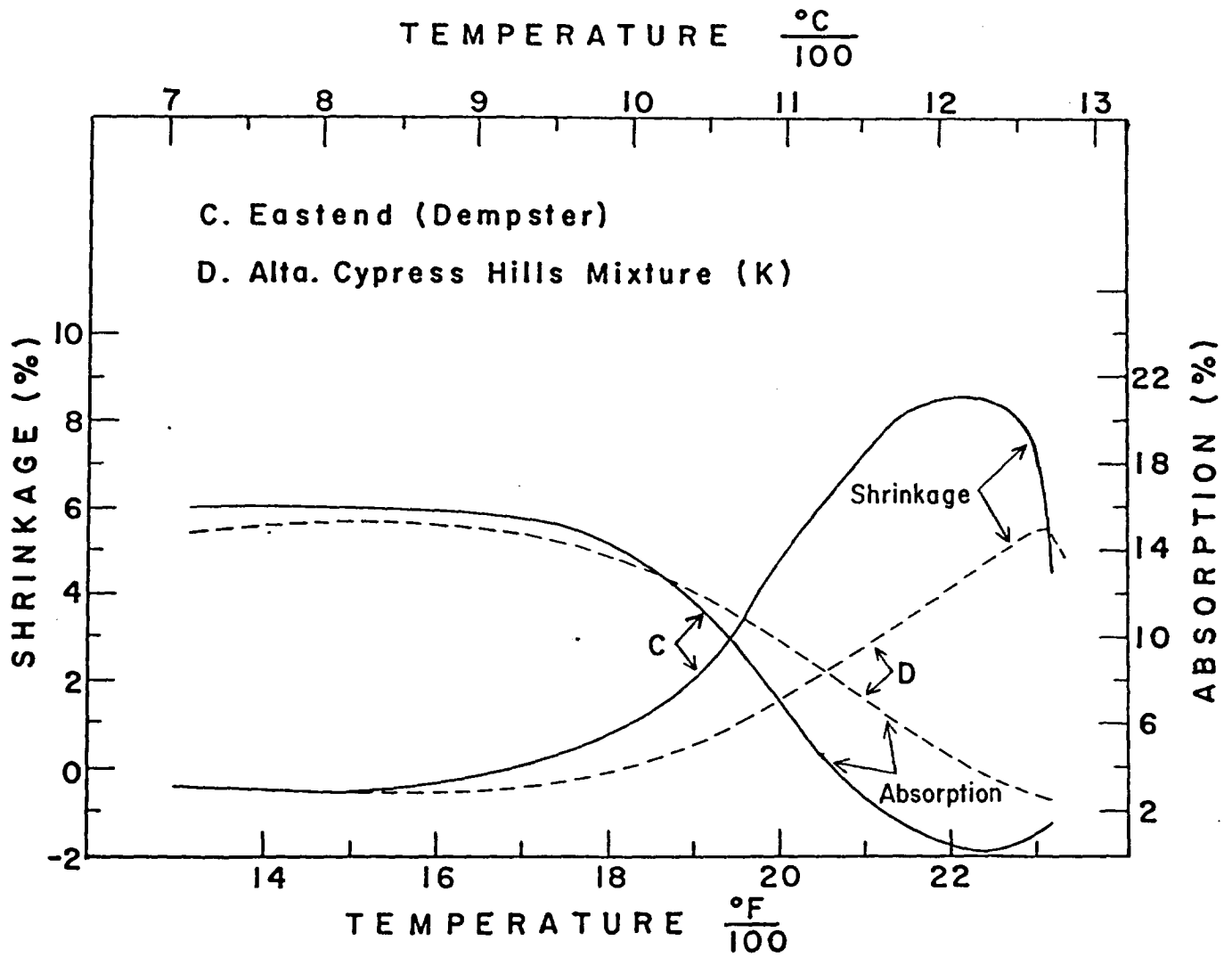


Figure 6. Temperature Gradient Curves of Cypress Hills Stoneware Clays.

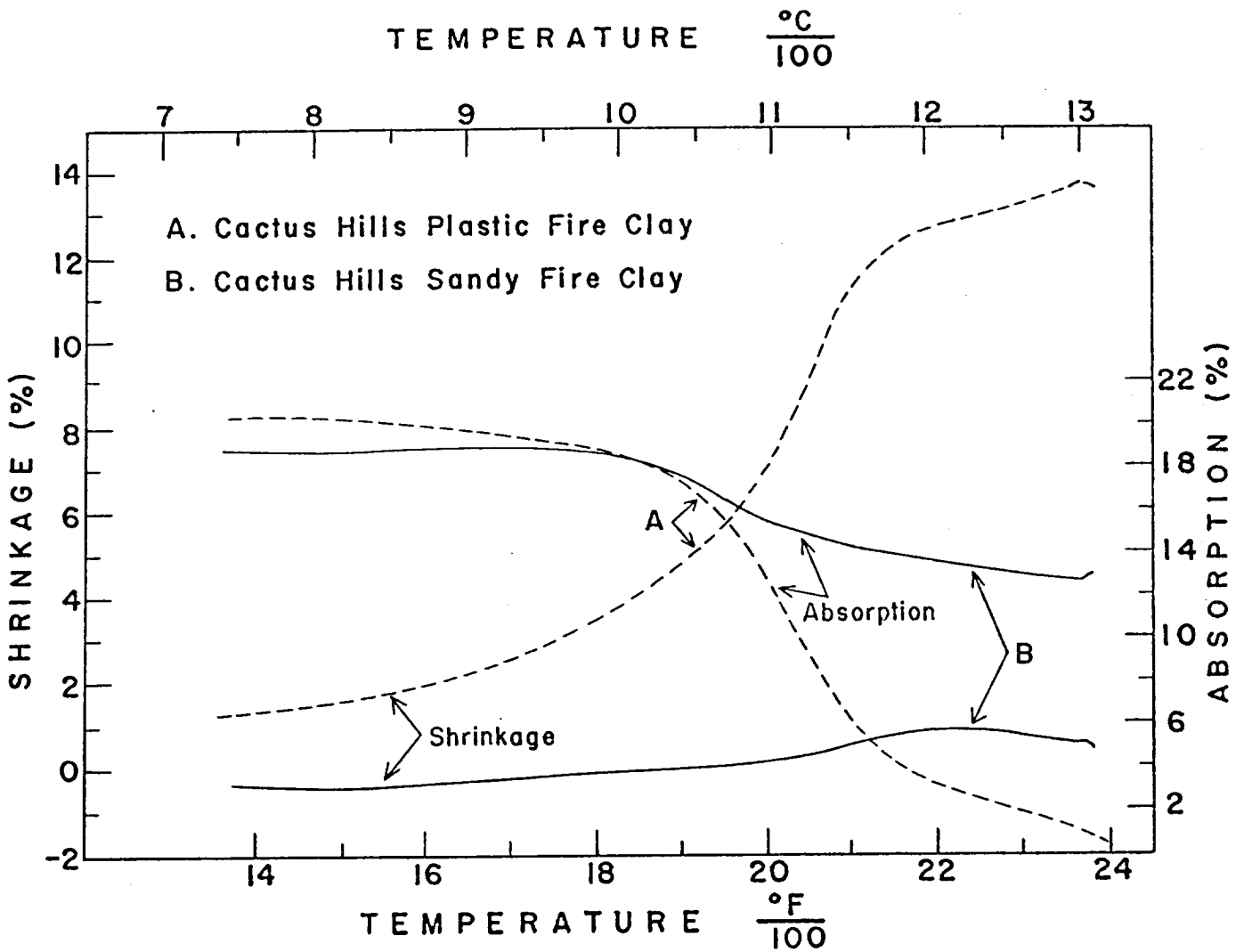


Figure 7. Temperature Gradient Curves of Fire Clays.

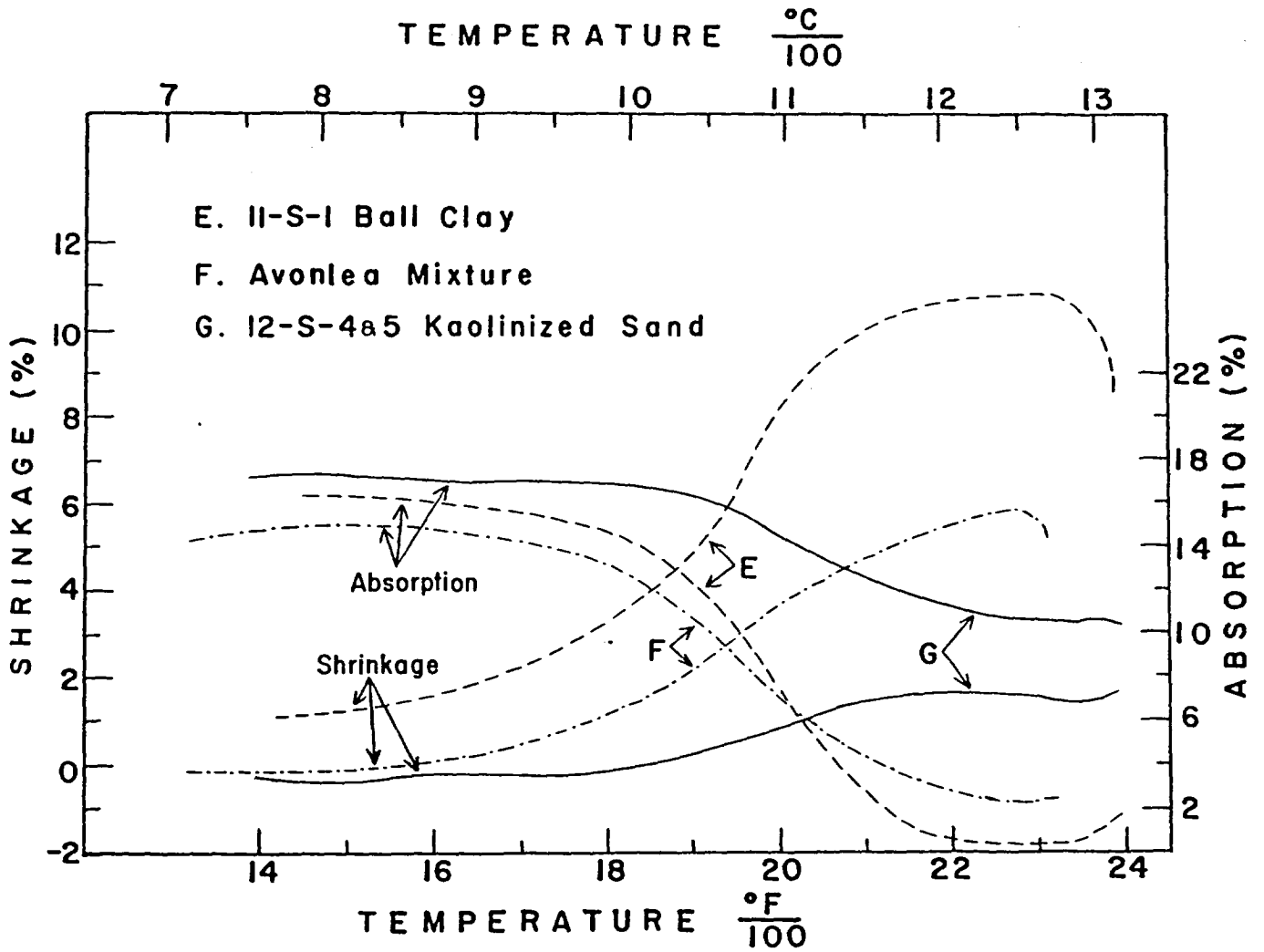


Figure 8. Temperature Gradient Curves of 11-S-1 Ball Clay, Avonlea Mixture, and 12-S-4&5 Kaolinized Sand.

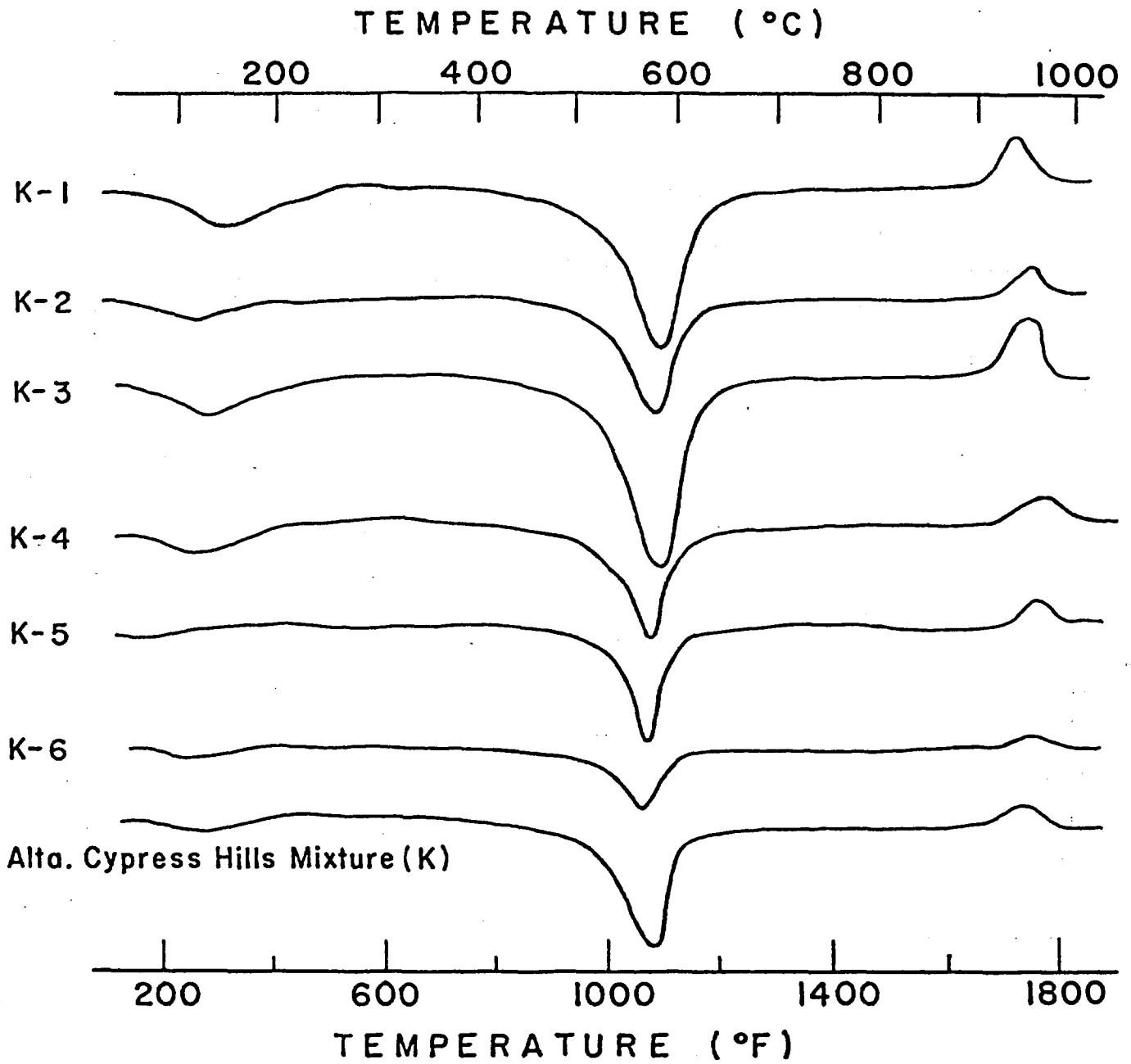


Figure 9. DTA Curves of Area "K" Stoneware Clays,  
Alberta Cypress Hills.

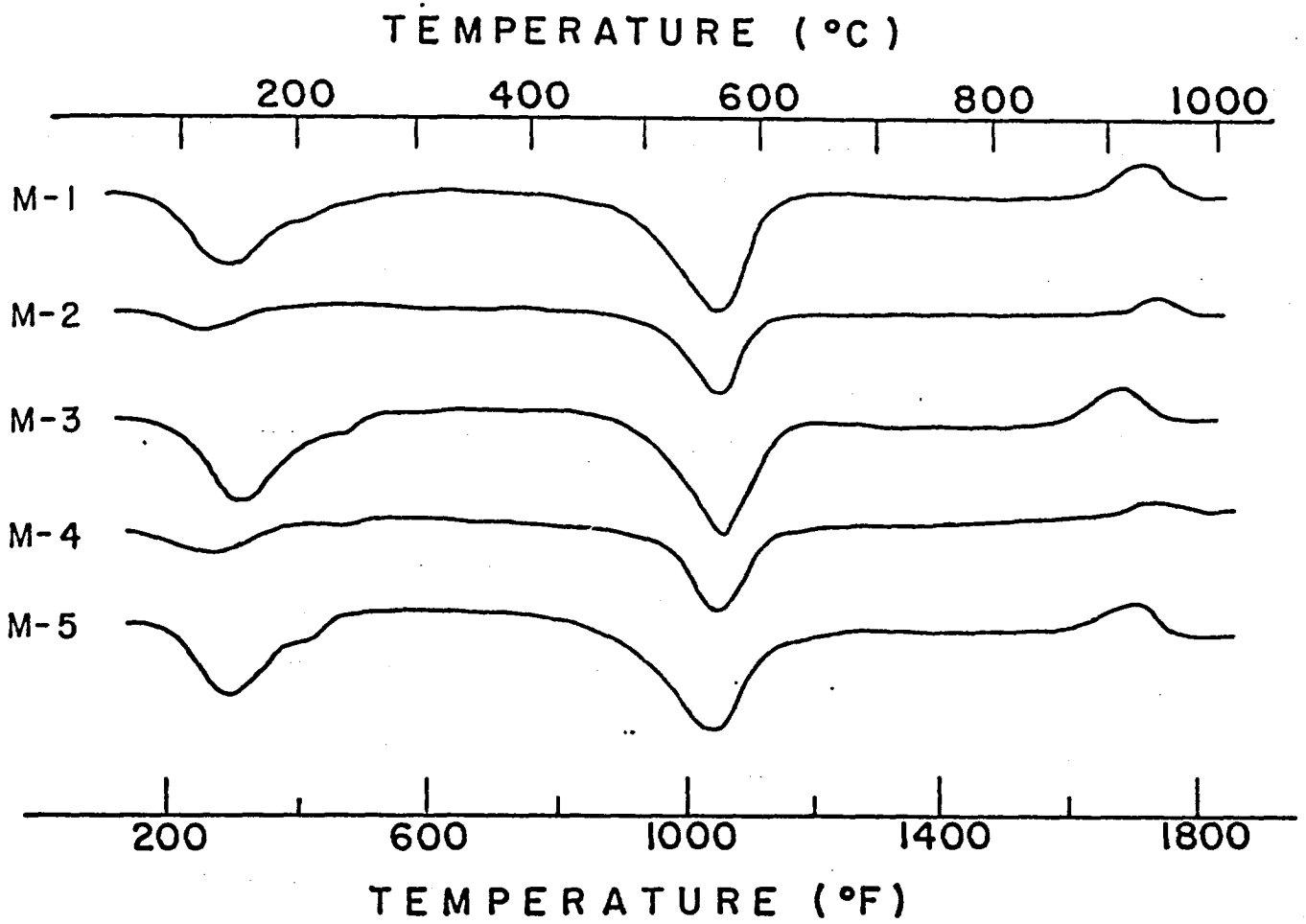


Figure 10. DTA Curves of Area "M" Stoneware Clays,  
Alberta Cypress Hills.

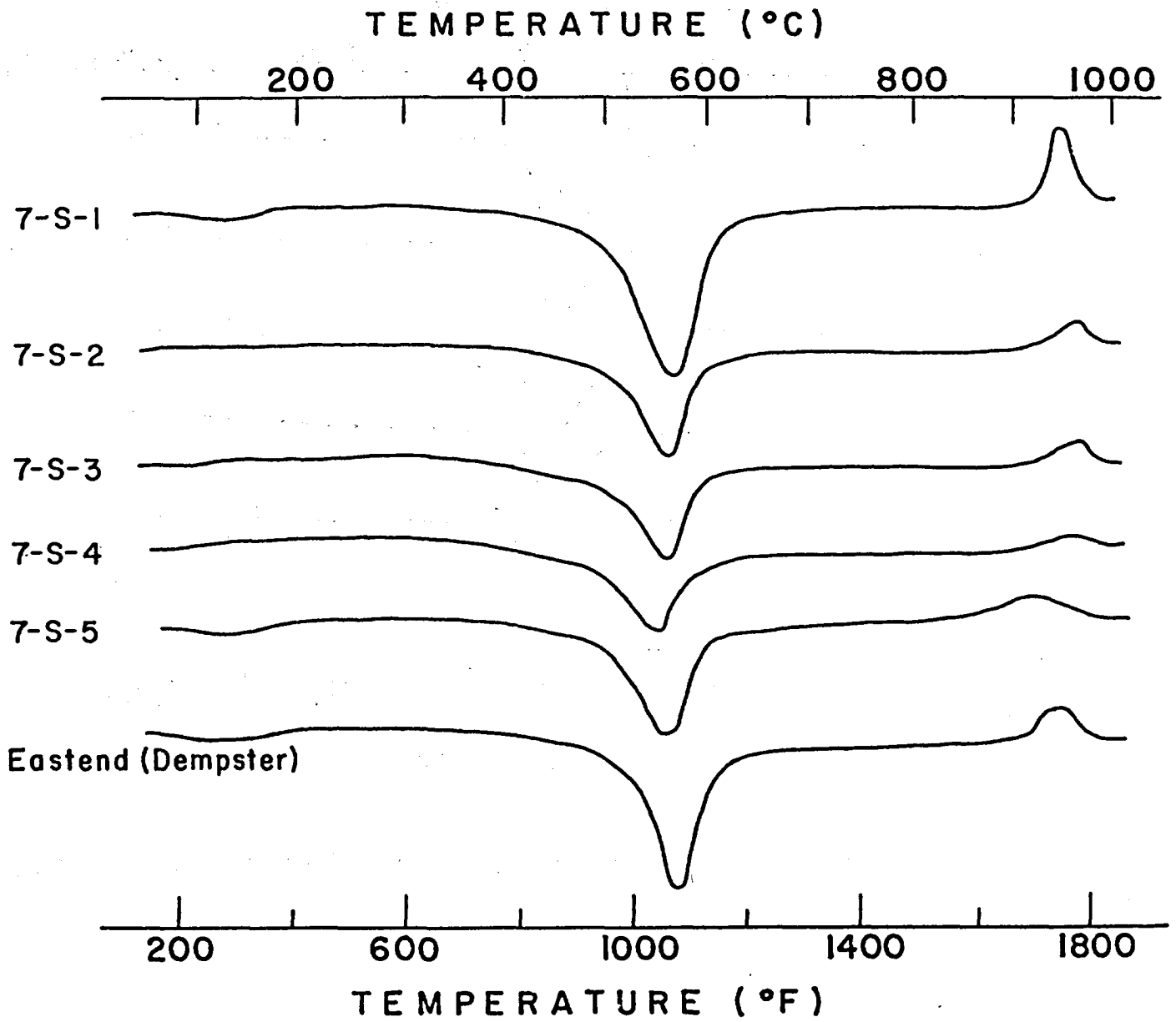


Figure II. DTA Curves of Eastend Stoneware Clays.



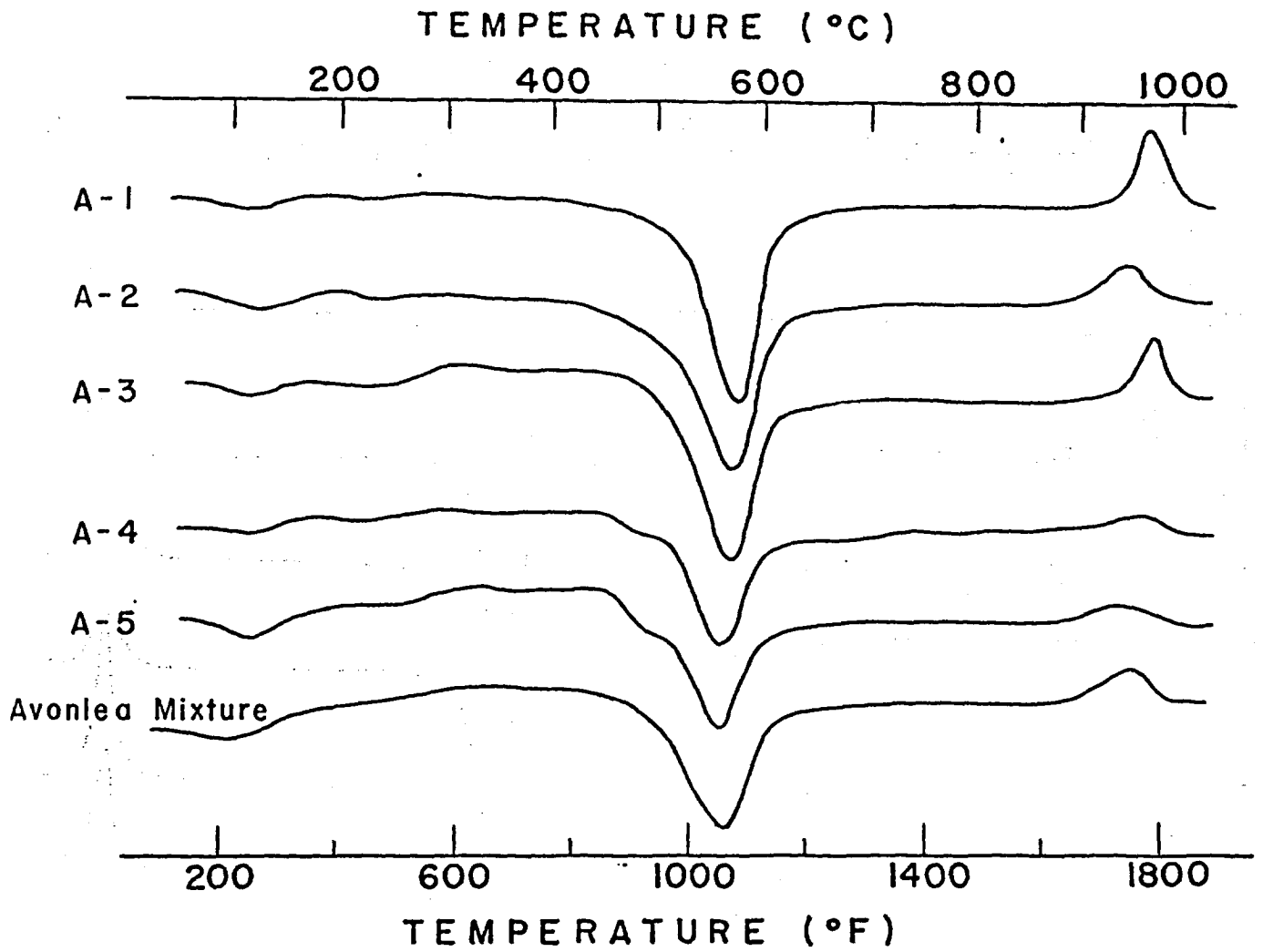


Figure 12. D T A Curves of Avonlea Stoneware Clays.

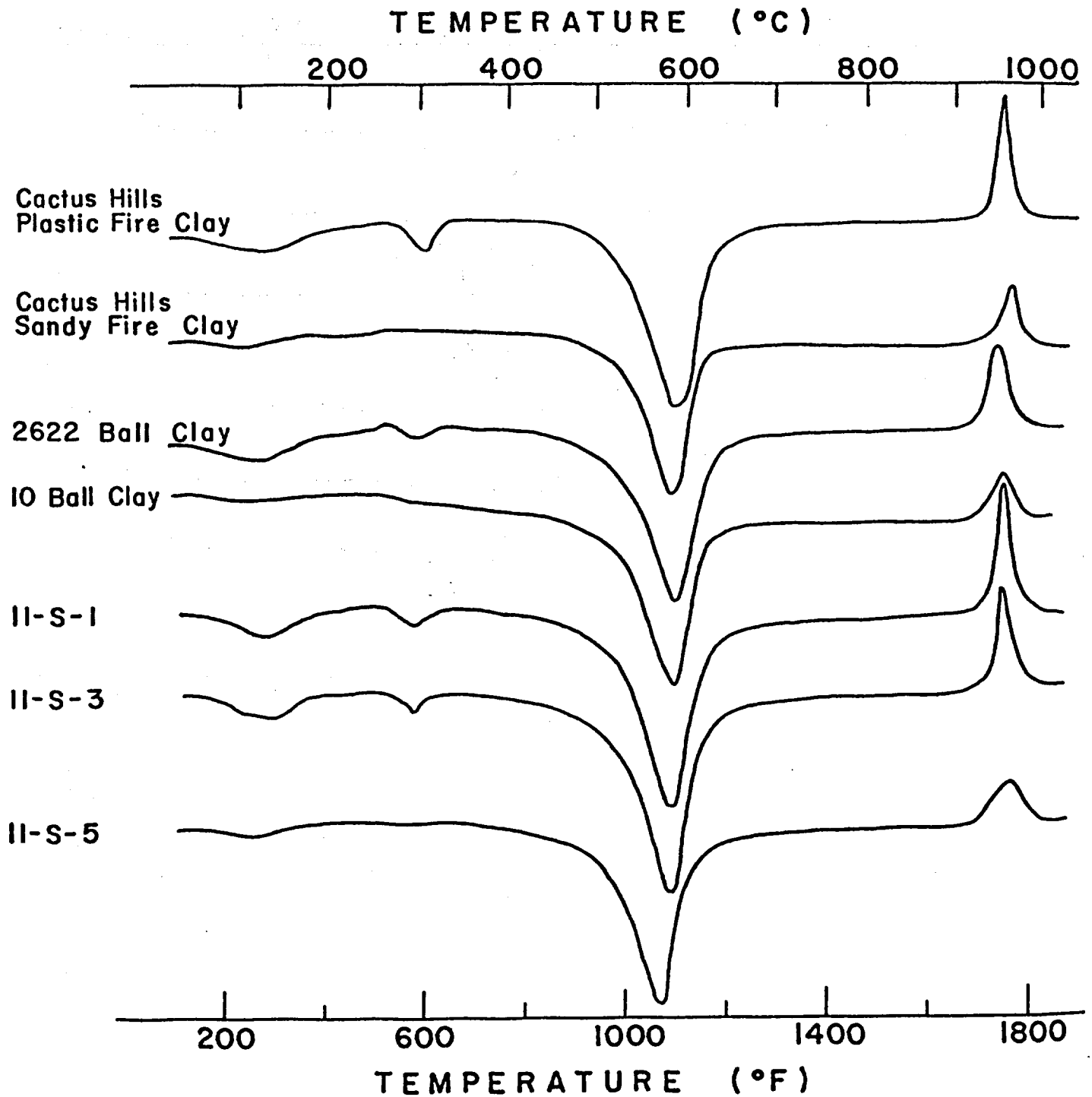


Figure 13. DTA Curves of Cactus Hills Fire Clays, Ball Clays 2622 and 10, and Project II Ball Clays (II-S-1, II-S-3, II-S-5).

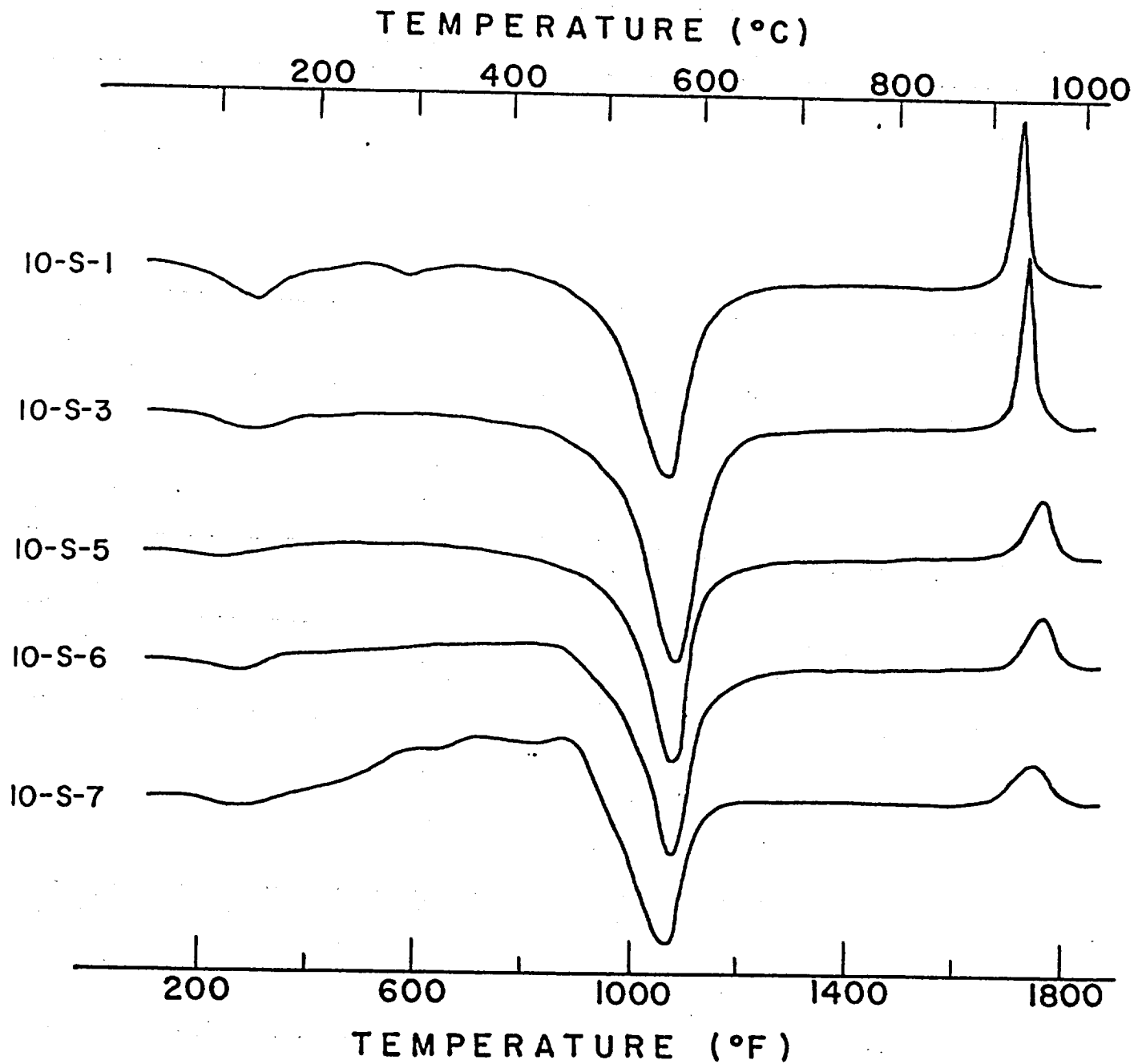


Figure 14. DTA Curves of Project 10 Ball Clays.

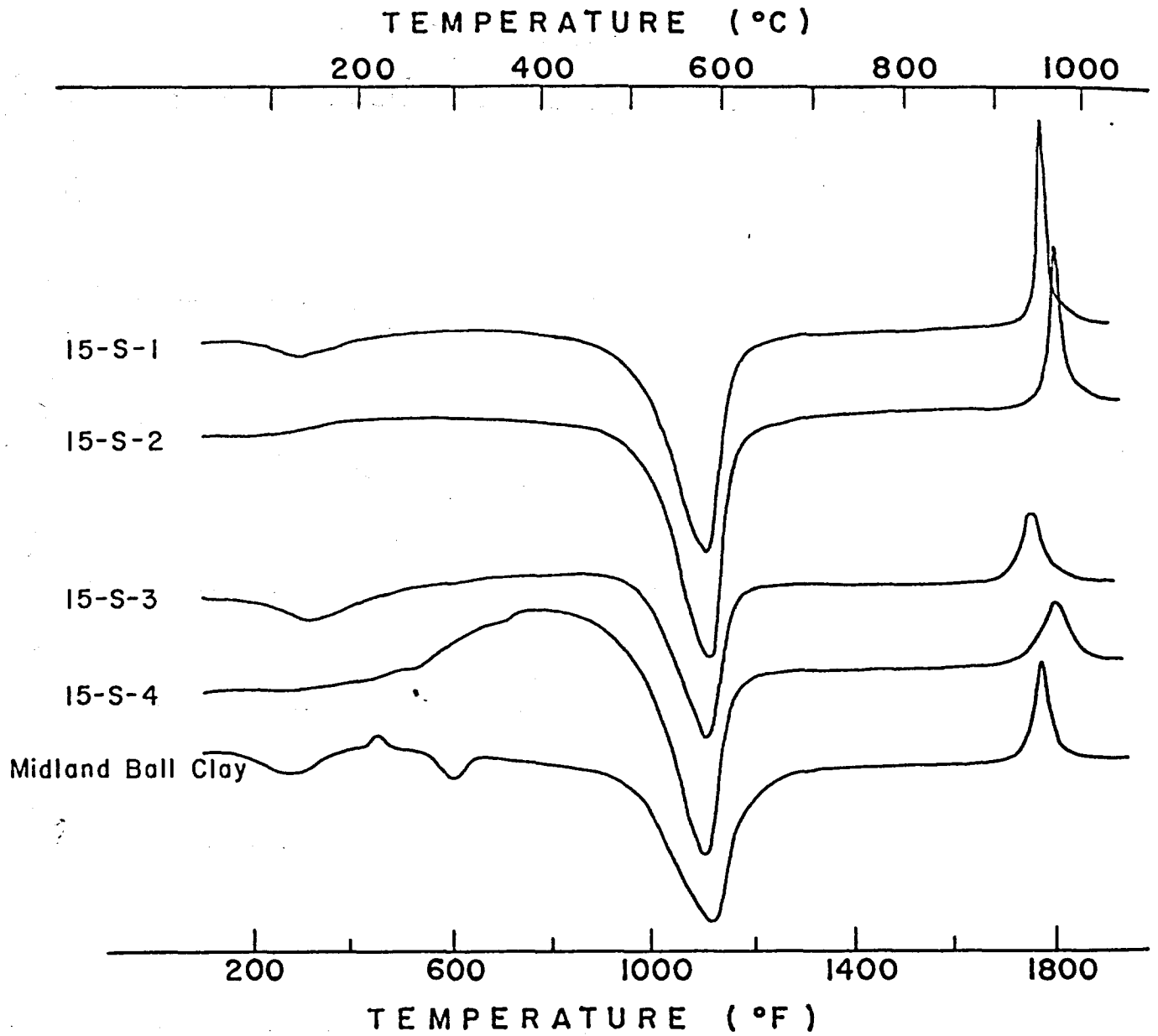


Figure 15. DTA Curves of Project 15 Ball Clays (15-S-1, 15-S-2, 15-S-3, 15-S-4) and Midland Ball Clay.

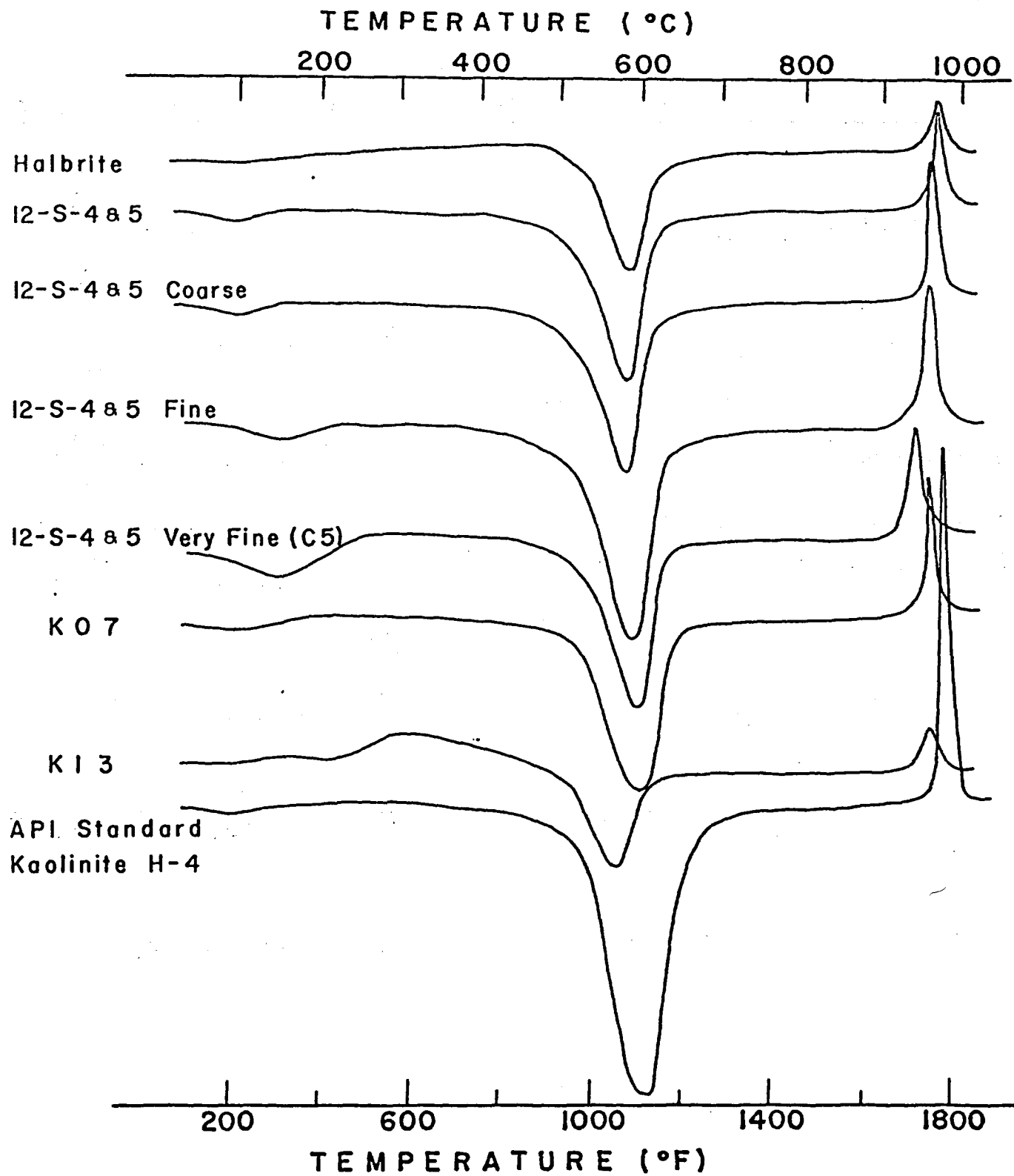


Figure 16. DTA Curves of Kaolinized Sands and API Standard Kaolinite H-4.

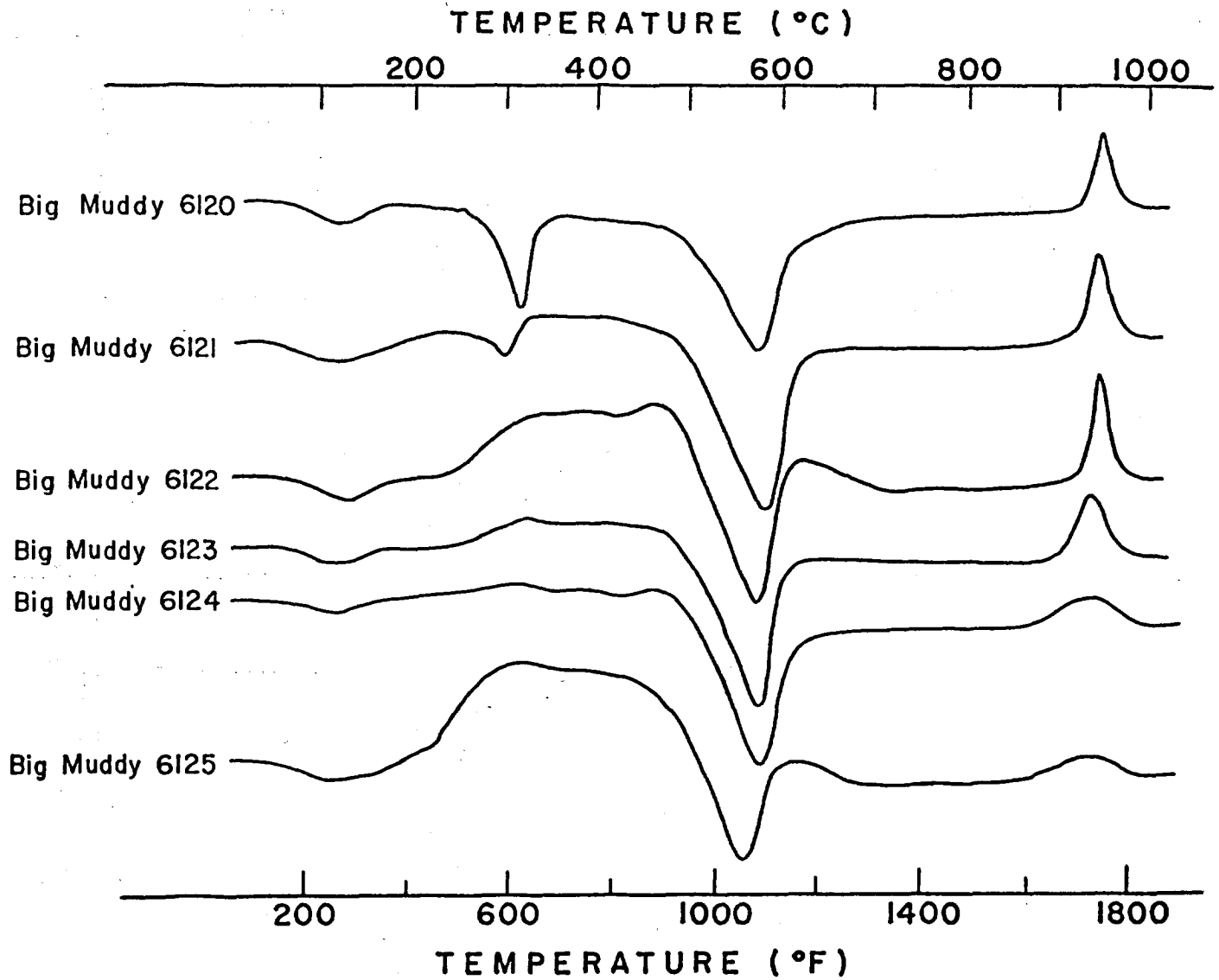


Figure 17. DTA Curves of Clays from Big Muddy Valley.

The area under the quartz ( $\text{SiO}_2$ ) peak at approximately  $570^\circ\text{C}$  ( $1058^\circ\text{F}$ ), which was obtained after eliminating the  $580^\circ$  ( $1076^\circ\text{F}$ ) clay mineral peak by calcining the clay to  $700^\circ\text{C}$  ( $1292^\circ\text{F}$ ), was measured to determine the approximate amount of quartz in each sample. With increasing temperature, the quartz peak is produced during the inversion from the low to the high form of quartz. Potter's flint was used as a standard for the quartz determinations.

The percentages of kaolinite and quartz obtained from DTA curves are shown in Tables 6, 7, 8, and 9. The kaolinite percentages obtained from comparison with kaolinite H-4 are shown without brackets and those obtained from comparison with 10-S-1(3A) are in brackets. Where both figures are reported the bracketed ones, according to X-ray and chemical analyses, are the more accurate. For example, the alumina percentages of samples 12-S-4&5 Coarse and 12-S-4&5 Fine, reported by Spyker et al. (36), indicate that the former sample contains 79% kaolinite and the latter sample contains 94% kaolinite. One exception is 12-S-4&5 Very Fine (C5). This sample, according to X-ray and chemical analysis (Spyker et al. (36), is principally kaolinite. It consists of particles mainly less than 1 micron in size, and consequently the kaolinite value is low when compared with sample 10-S-1(3A) which has coarser particles.

Occasionally, some of the fire clay and ball clay curves have a small endothermic peak at approximately  $320^\circ\text{C}$  ( $618^\circ\text{F}$ ), caused by gibbsite ( $\text{Al}_2\text{O}_3 \cdot 3\text{H}_2\text{O}$ ). This is shown by the Cactus Hills Plastic Fire Clay curve in Fig. 13. The approximate amount of gibbsite was obtained by determining the area per gram of sample under the  $320^\circ\text{C}$  peak and comparing this with a standard gibbsite curve. Other curves, particularly some of the Big Muddy Valley ones in Fig. 17, have broad exothermic peaks from  $320^\circ\text{C}$  ( $618^\circ\text{F}$ ), caused by the oxidation of carbonaceous material.

#### X-Ray Diffraction

The results of the XRD work on the "as received" samples and on some of the fractionated samples are shown, along with the results of the semi-quantitative DTA, in Tables 6, 7, 8, and 9. The results of microscopic and XRD examination of the plus 200 mesh fraction of some of the samples are shown in Table 10. From examination of the above tables, kaolinite and quartz are the principal minerals present in the Whitemud formation clays. Their presence was readily identified by DTA and XRD. In general, kaolinite is highest in the refractory fire clays, ball clays, and fractionated samples, and is slightly lower in the stoneware clays. The greatest percentages of quartz occur in the non-plastic clays, and the least percentages in the very plastic, high-quality clays, such as the ball clays.

Mica was identified by XRD in many of the samples, particularly in the stoneware clays. It occurs also in the lower seams of the ball clay deposits, and in the sandy fire clays and kaolinized sands. Muscovite was observed in small quantities in a few of the plus 200 mesh samples, and illite was identified in small quantities in the very fine fractions of some of the clays. The quantity of mica was frequently small in the samples, and usually no attempt was made to positively identify the form in which it occurred.

TABLE 6

Mineralogical Composition of Alberta Cypress Hills  
Stoneware Clays

Sample	Mineral	X-Ray Diffraction Results	D.T.A. Results %	Sample	Mineral	X-Ray Diffraction Results	D.T.A. Results %
<u>Area K Clays</u>				<u>Area M Clays</u>			
K-1	Kaolinite Quartz Mica Montmorillonite	Major Major Minor Minor	35 38	M-1	Kaolinite Quartz Montmorillonite	Moderate Major Minor	26 40
K-2	Kaolinite Quartz Mica Feldspar	Moderate Major Minor Minor	23 53	M-2	Kaolinite Quartz Mica Montmorillonite Feldspar	Minor Major Minor Minor Minor	16 46
K-3	Kaolinite Quartz	Major Moderate	38 30	M-3	Kaolinite Quartz Montmorillonite	Moderate Moderate Minor	27 27
K-4	Kaolinite Quartz Mica Feldspar	Moderate Major Moderate Minor	23 36	M-4	Kaolinite Quartz Mica Feldspar	Moderate Moderate Moderate Minor	20 29
K-4 (3A) Fraction	Kaolinite Quartz Mica Montmorillonite	Major Trace Minor Minor	52(64)	M-5	Kaolinite Quartz Mica Montmorillonite Feldspar	Moderate Moderate Moderate Minor Minor	24 33
K-5	Kaolinite Quartz Mica Feldspar	Moderate Major Moderate Minor	22 43				
K-6	Kaolinite Quartz Feldspar	Minor Major Minor	15 42				
Alberta Cypress Hills Mixture (K)	Kaolinite Quartz Mica Feldspar Montmorillonite	Moderate Major Minor Minor Trace	27 47				



TABLE 7

Mineralogical Composition of Eastend and Avonlea Stoneware Clays

Sample	Mineral	X-Ray Diffraction Results	D.T.A. Results %	Sample	Mineral	X-Ray Diffraction Results	D.T.A. Results %
<u>Eastend Samples</u>				<u>Avonlea Samples</u>			
7-S-1	Kaolinite Quartz	Major Major	34 36	A-1	Kaolinite Quartz	Major Moderate	38 30
7-S-1 (3B) Fraction	Kaolinite Quartz Montmorillonite	Major Trace Trace	56(58)	A-2	Kaolinite Quartz Mica	Major Moderate Minor	38 26
7-S-2	Kaolinite Quartz Mica Feldspar	Moderate Major Minor Minor	19 44	A-3	Kaolinite Quartz Feldspar	Major Moderate Minor	35 32
7-S-3	Kaolinite Quartz Mica Feldspar	Moderate Major Minor Minor	18 45	A-4	Kaolinite Quartz Mica Feldspar	Moderate Moderate Minor Minor	28 30
7-S-4	Kaolinite Quartz Mica Feldspar	Moderate Moderate Moderate Minor	18 27	A-4 (3A) Fraction	Kaolinite Quartz Mica Montmorillonite	Major Trace Trace Minor	41(47)
7-S-5	Kaolinite Quartz Mica Feldspar	Moderate Moderate Moderate Trace	22 27	A-5	Kaolinite Quartz Mica Feldspar Montmorillonite Chlorite Chlorite- vermiculite mixed layer	Moderate Moderate Minor Moderate Moderate Minor Minor (tentative)	26 22
7-S-5 (3A) Fraction	Kaolinite Quartz Montmorillonite	Major Trace Minor	42(48)	Avonlea Mixture	Kaolin Quartz Mica Feldspar Montmorillonite Chlorites	Major Moderate Minor Minor Minor Insufficient to identify	28 30
Eastend (Dempster)	Kaolinite Quartz Mica Feldspar	Major Major Minor Minor	35 40				
Eastend (Dempster) (3A) Fraction	Kaolinite Quartz Mica Montmorillonite	Major Trace Minor Minor	49(59)				

TABLE 8

Mineralogical Composition of Cactus Hills Fire Clays and Willows Area Ball Clays

Sample	Mineral	X-Ray Diffraction Results	D.T.A. Results %	Sample	Mineral	X-Ray Diffraction Results	D.T.A. Results %
	<u>Cactus Hills Fire Clays</u>				<u>Project 10, Willows, Ball Clays</u>		
Cactus Hills Plastic Fire Clay	Kaolinite Quartz Gibbsite Gypsum	Major Moderate Minor Suggestion	49 (65) 22 2.4	10-S-1	Kaolinite Quartz Gibbsite	Major Minor None detected	48 (63) 19 1.0
Cactus Hills Plastic Fire Clay (3A) Fraction	Kaolinite Gibbsite	Major None detected	82 (96) Small peak	10-S-1 (3A) Fraction	Kaolinite Quartz Gibbsite	Major Minor None detected	70 (86) 10 Small peak
Cactus Hills Sandy Fire Clay	Kaolinite Quartz Mica	Major Major Minor	37 44	10-S-3	Kaolinite Quartz Gypsum	Major Moderate Trace	52 (68) 24
	<u>Project 11 and 2622 Ball Clays from Willows</u>			10-S-5	Kaolinite Quartz Mica	Major Major Minor	42 (52) 42
2622 Ball Clay	Kaolinite Quartz Gibbsite	Major Moderate None detected	45 (56) 28 0.5	10-S-6	Kaolinite Quartz Mica	Major Moderate Moderate	46 (60) 21
11-S-1	Kaolinite Quartz Gypsum Gibbsite	Major Moderate Weak None detected	47 (63) 21 1.20	10-S-7	Kaolinite Quartz Mica Feldspar	Major Moderate Minor Trace	37 23
11-S-1 (3A) Fraction	Kaolinite Quartz Gibbsite	Major Minor None detected	61 (80) 16 Small peak				
11-S-3	Kaolinite Quartz Gibbsite	Major Moderate None detected	49 (60) 21 0.5				
11-S-5	Kaolinite Quartz Mica	Major Moderate Moderate	45 (50) 28				
11-S-5 (3A) Fraction	Kaolinite Quartz Mica	Major Minor Minor	65 (80) 10				
10	Kaolinite Quartz Mica	Major Moderate Minor	44 (52) 24				

TABLE 9

Mineralogical Composition of Flintoft Ball Clays, Kaolinized Sands from Fir Mountain, Eastend and Halbrite, and Midland and Big Muddy Valley Plastic Clays

Sample	Mineral	X-Ray Diffraction Results	D.T.A. Results %	Sample	Mineral	X-Ray Diffraction Results	D.T.A. Results %
<u>Project 15 Ball Clays, Flintoft</u>				<u>Midland and Big Muddy Valley Clays</u>			
15-S-1	Kaolinite Quartz	Major Moderate	51 (70) 22	Midland Ball Clay	Kaolinite Quartz Gibbsite	Major Minor None detected	43 (56) 17 1.0
15-S-2	Kaolinite Quartz Mica	Major Moderate Tentative	50 (65) 25	Big Muddy Fire Clay 6120	Kaolinite Quartz Gibbsite	Major Minor Minor	37 (4) 18 8.0
15-S-3	Kaolinite quartz	Major Moderate	39 (47) 25	Big Muddy Fire Clay 6121	Kaolinite Quartz Gibbsite Montmorillonite	Major Minor Minor Minor	48 (59) 19 2.8
15-S-3 (3A) Fraction	Kaolinite Quartz Montmorillonite	Major Trace Large minor	43 (57)	Big Muddy 6121 (3A) Fraction	Kaolinite Quartz	Major Trace	85(100)
15-S-4	Kaolinite Quartz Mica	Major Moderate Moderate	40 25	Big Muddy Fire Clay 6122	Kaolinite Quartz	Major Minor	43 (55) 17
<u>Kaolinized Sands</u>				Big Muddy Fire Clay 6123	Kaolinite Quartz	Major Moderate	38 (46) 22
12-S-4 & 5	Kaolinite Quartz	Major Major	43 39	Big Muddy Stoneware Clay 6124	Kaolinite Quartz Mica	Major Moderate	37 27
12-S-4 & 5 Coarse Fraction	Kaolinite Quartz Mica	Major Minor Minor	66 (82) 9	Big Muddy Stoneware Clay 6125	Kaolinite Quartz Mica	Major Moderate Minor	33 21
12-S-4 & 5 Fine Fraction	Kaolinite Quartz	Major Trace	74 (91) Trace				
12-S-4 & 5 Very Fine Fraction (C5)	Kaolinite Quartz	Major Rarely detectable	54 (66)				
K 07 Air-Floated	Kaolinite Quartz	Major Minor	84 4				
K 13 Air-Floated	Kaolinite Quartz Mica	Major Minor Moderate	55 14				
Halbrite	Kaolinite Quartz Mica Feldspar	Moderate Moderate Minor Minor	32 28				

TABLE 10  
Mineralogical Composition of Plus 200 Mesh Fractions of  
Selected Whitemud Formation Clays

Sample and Percentage +200 Mesh	Mineral	Comments
Alberta Cypress Hills Mixture (K) 29.4%	Quartz Kaolinite Goethite Muscovite	Rounded or irregular clear or translucent grains, sometimes stained yellowish red. In fine-grained aggregates of quartz from white to dark grey. Reddish-brown, fine-grained aggregates with quartz. Clear, colourless flakes accompanied by green micaceous material.
K-2 Stoneware 41.0%	quartz Muscovite Kaolinite	Clear, colourless, angular grains; some loosely cemented aggregates. Clear, colourless flakes. a. Platy, soft, greyish-green material. b. Soft, fine-grained, grey aggregates with quartz.
M-4 Stoneware 0.2%	Quartz Muscovite Goethite Carbonaceous Amber ? Micrometeorites ?	Clear, angular, and rounded grains. Irregular, fine-grained, greyish-white aggregates with quartz. Rounded, nodular, reddish-brown grains, black interior. Soft, black material. Rare, bright-yellow, clear grains, no diffraction pattern, possibly amber.
Eastend (Dempster) Stoneware 8.0%	Quartz Siderite Goethite Muscovite Kaolinite ?	Clear, angular grains, a few stained reddish, some rare, loosely cemented aggregates. Brownish-grey, euhedral prismatic crystals in aggregates, also spherical, nodular aggregates. Nodular, spherical aggregates, black interior and a thin reddish-brown coating. Rare, thin, colourless flakes. A mineral present in white and yellow-brown aggregates with quartz and a mica mineral.
7-S-1 Stoneware 3.9%	Quartz Goethite Gypsum Calcite Carbonaceous	Clear, colourless, angular grains and rare yellowish-green aggregates. Nodular spherical grains with a black interior and a thin reddish-brown coating. Clear, colourless, platy grains. Rare, translucent, yellowish-green grains. Soft, black material.
7-S-5 Stoneware 6.4%	Quartz Goethite Gypsum	Clear, colourless, angular grains. Nodular spherical granules; black, metallic with a thin, reddish-brown coating. Clear, colourless, platy grains.
Avonlea Mixture Stoneware 19.2%	Quartz Gypsum Goethite Kaolinite Jarosite Carbonaceous Muscovite	Clear, colourless, angular and rounded, grey or greenish-grey grains. Fine-grained soft, white aggregates in hemihydrate form (probably because of drying). Nodular, spherical, reddish-brown aggregates, black interior. Platy and columnar aggregates of soft brownish-grey material. Soft, fine-grained, yellow material and a matrix of aggregates of clear quartz grains. Soft, black material. Rare, colourless flakes.
A-1 Stoneware 15.5%	Quartz Gypsum Goethite Carbonaceous Kaolinite Hematite ? Muscovite	Clear, angular and rounded, frosted grains; a few loosely cemented aggregates. Soft, platy white, in hemihydrate form, probably because of drying sample. Fine-grained, angular, reddish-brown fragments. Black, irregular grains. Brownish-grey, soft, platy and columnar aggregates. Rare, shiny, metallic, blue-black material. Rare, colourless flakes.
A-4 Stoneware 2.0%	Quartz Gypsum Carbonaceous	Clear, rounded and angular grains; yellow brown or grey aggregates. Clear, colourless or yellowish-grey platy grains. Soft, black, irregular grains.
12-S-4&5 Kaolinized Sand 36.3%	Quartz Kaolinite Carbonaceous Muscovite	Clear, white, grey or greenish-grey grains. Brownish-grey, soft, platy and columnar aggregates. Black, soft, irregular grains. Rare, clear, colourless flakes.
Halbrite Kaolinized Sand 46.0%	Quartz Kaolinite Gypsum Carbonaceous	Clear, colourless, angular grains; some translucent grains and rare ferruginous aggregates. Rare, platy and columnar aggregates; soft, brownish-grey. Rare, clear, platy grains. Soft, black material.
Sandy Fire Clay, Cactus Hills 43.7%	Quartz Carbonaceous Kaolinite	Mostly rounded, clear, colourless or milky-grey grains. Soft, black material. a) In soft, platy, or columnar brownish-grey aggregates. b) Soft, fine-grained, white aggregates, with quartz.
Plastic Fire Clay, Cactus Hills 2.2%	Quartz Goethite Carbonaceous Kaolinite Gypsum	Clear, angular grains, also some rounded, milky grains and loosely cemented aggregates. a) Brownish-grey, nodular aggregates, black interior. b) Brown grains, probably pseudomorphic after siderite. c) Fine-grained, reddish-brown aggregates. Soft, black material. Soft, fine-grained, white aggregates, with quartz. A few clear, platy grains.
2622 Ball Clay 0.6%	Quartz Kaolinite Carbonaceous Barite	Clear, colourless, irregular grains; fine-grained white, and a few loosely cemented aggregates. Platy and columnar, brownish-grey, soft material. Soft, black material. Irregular, hard, brownish-grey grains.
11-S-1 Ball Clay 1.7%	Quartz Gypsum Goethite Carbonaceous	Clear, colourless, rounded grains and a few loose aggregates of small grains. Clear, platy grains. Rare, hard nodular granules with fine-grained black interior and a yellow-brown exterior. Soft, black material.
15-S-3 Ball Clay 2.0%	Quartz Gypsum Goethite Carbonaceous	Loosely cemented aggregates and clear, colourless, angular grains. Clear platy grains. Rare nodular granules, black interior, yellow brown exterior. Soft, black.

The percentage composition of ideal muscovite ( $KAl_2(AlSi_3O_{10})(OH)_2$ ) is  $SiO_2$ , 45.2;  $Al_2O_3$ , 38.5;  $K_2O$ , 11.8; and  $H_2O$ , 4.5. The compositions of the clay micas--such as illite--are extremely variable, because of substitution in the crystal lattice. Warsaw(48) recently discussed illite and the micas related to it. In the present report, illite refers to a micaceous clay-grade material which broadly contains less potassium, and more silica and water, than does muscovite.

Feldspar was identified by XRD frequently in the stoneware clays (Tables 6 and 7), and rarely in the fire clays and ball clays (Tables 8 and 9). The potash, soda and lime contents of feldspars vary, but the formula of a typical feldspar (orthoclase) is  $K_2O \cdot Al_2O_3 \cdot 6SiO_2$ , resulting in a percentage composition of  $K_2O$ -16.9,  $Al_2O_3$ -18.4, and  $SiO_2$ -64.7.

Although not all samples containing feldspar were fractionated, this mineral was not identified in the plus 200 mesh fractions of samples K-2, M-4, 7-S-5 and A-4 (Tables 6, 7 and 10) nor in the minus 10 micron fractions of samples K-4, 7-S-5 and A-4 (Tables 6 and 7). Thus it may be concluded that feldspar in the Whitemud formation clays has particle sizes mainly smaller than 200 mesh (74 microns) but greater than 10 microns.

Montmorillonite was readily identified by XRD in some of the clays from the Alberta Cypress Hills (K-1, M-1, M-2, M-3, M-5; see Table 6), particularly from area M. It was also identified in stoneware samples 7-S-1 (3B), 7-S-5(3A), and Eastend (Dempster)(3A), from Eastend (Table 7); in stoneware samples A-4 (3A), A-5 and Avonlea Mixture, from Avonlea (Table 7); and in 15-S-3(3A) ball clay and Big Muddy Plastic Fire Clay 6121 (Table 9).

The composition of montmorillonite may be extremely variable, because of substitutions in the crystal lattice. Ross and Hendricks(49) calculated formulas for various members of the montmorillonoids. They suggested that the clays at the montmorillonite end of the montmorillonite-beidellite series would have  $(Al_{3.34}^{Mg.66} Na_{.66} Si_8O_{20}(OH)_4$  plus interlayer water as their formula.

Gibbsite, which was readily identified in small quantities by DTA in some of the very refractory plastic fire clays and very refractory ball clays (Tables 8 and 9; Figures 13, 14 and 15), was identified in a few of the samples by XRD. Frequently, however, the amount of gibbsite was too small to identify by this method. Gibbsite was observed as small white concretions in Big Muddy Fire Clay 6121.

A chloritic clay mineral and one tentatively identified as a mixed-layer, chlorite-vermiculite clay were identified in sample A-5 from Avonlea, Saskatchewan. The stratum from which this sample was taken is probably a transition from the Whitemud formation to the lower Eastend formation.

Other minerals that are present in extremely small quantities in a few of the clays were identified by XRD in the plus 200 mesh fraction of some of the clays listed in Table 10. Gypsum ( $CaSO_4 \cdot 2H_2O$ ) and goethite ( $Fe_2O_3 \cdot 3H_2O$ ) frequently occur in clays from the majority of the areas from which samples were taken. Carbonaceous material was commonly found in the samples examined. Siderite ( $FeCO_3$ ) was identified in Eastend (Dempster), jarosite ( $KFe_3(SO_4)_2(OH)_6$ ) in the Avonlea Mixture, and calcite ( $CaCO_3$ ) in 7-S-1.

The minus 10 micron samples were made up principally of fine-particle kaolinite. Some of the fine fractions contained montmorillonite or illite, or both. Further, gibbsite and fine particle quartz were identified in some of these samples. Humphries and Brady(27) found that the Whitemud formation ball clays that they examined contained at least approximately 80% minus 10 micron particles.

Although substances such as siderite, calcite, carbonaceous material, and gypsum may, when present in appreciable quantities, cause some difficulty in firing and affect the properties of the fired product, the DTA curves indicate that they do not seriously affect the samples examined. In fact, the DTA curves show that the Whitemud clays should be relatively easy to fire. Substances such as pyrite, high-temperature organic material, dolomite, and calcite (this last was identified in one sample only) are absent from the Whitemud clays. These materials are potential trouble-makers during firing, and provision must be made in the firing cycle, if they are present, to decompose them properly at the correct temperatures.

### Mineralogical Balances

Mineralogical balances of three typical stoneware clay mixtures, two typical sandy kaolinized materials, a sandy fire clay (similar to a kaolinized sand), a plastic fire clay and three ball clays are shown in Table 11. The crystalline silica percentages shown in this table were obtained by the Trostel and Wynne method(50). These values checked closely with the DTA values for quartz, which are shown for the samples in Tables 6,7,8, and 9. Kaolinite and gibbsite percentages were obtained from the DTA curves. The total of the percentages of these minerals was deducted from the total of the chemical analysis for each clay. The remainder has value and composition varying with the complexity of the clay. Part of the composition of the remainders is made up of small percentages of minerals and materials not identified.

Mica occurs in the remainders for all samples shown in Table 11, except possibly in the refractory plastic fire clay from the Cactus Hills and in the ball clays 10-S-1, 11-S-1 and 15-8-3. The substantial amount of +110°C H<sub>2</sub>O (combined water) in the remainders for the Avonlea Mixture, Cactus Hills Sandy Fire Clay and the kaolinized sands suggests that mica is principally in the form of illite. The amount of +110°C H<sub>2</sub>O in the Alberta Cypress Hills Mixture (K) and Eastend (Dempster) remainders is low; possibly these clays contain less mica than the others, or some of the mica is in the form of muscovite. Muscovite was observed in the plus 200 mesh fractions of the stoneware clays and the 12-S-4&5 kaolinized sand. Calculation of the percentage mica in the remainders is unsatisfactory, because other aluminum silicates, such as feldspar and montmorillonite, may also be present.

Feldspar is a constituent of the stoneware clay and the Halbrite kaolinized sand remainders, but does not appear in the remainders for the 12-S-4&5 kaolinized sand, the Cactus Hills fire clays, and the ball clays.

The stoneware clay remainders (Table 11), in addition to containing mica and feldspar, also contain montmorillonite and goethite. Eastend (Dempster) contains siderite and Avonlea Mixture contains gypsum and jarosite.

Montmorillonite makes up part of the remainders for the stoneware clays and ball clay 15-S-3. The remainder for 15-S-3 is estimated to contain

TABLE 11  
Mineralogical Balances of Selected Whitewud Formation Clays

Clay	Minerals	Total %	SiO <sub>2</sub> %	FeO %	Fe <sub>2</sub> O <sub>3</sub> %	TiO <sub>2</sub> %	Al <sub>2</sub> O <sub>3</sub> %	CaO %	MgO %	Organic Carbon %	S %	CO <sub>2</sub> %	Na <sub>2</sub> O %	K <sub>2</sub> O %	H <sub>2</sub> O at 110°C % Moisture	H <sub>2</sub> O at 110°C %
Alberta Cypress Hills Mixture (K) Stoneware	Chemical Analysis	100.53	72.32	0.19	1.64	1.17	16.54	0.12	0.05	0.10	0.012	0.12	0.40	2.59	0.77	4.51
	Crystalline Silica	49.90	49.90													
	Kaolinite	27.00	12.60				10.70									3.70
	Remainder - mica, feldspar, and small amounts of montmorillonite, and goethite	23.63	9.82	0.19	1.64	1.17	5.84	0.12	0.05	0.10	0.012	0.12	0.40	2.59	0.77	0.81
Eastend (Dampster) Stoneware	Chemical Analysis	99.97	67.56	0.64	1.86	0.59	18.78	0.14	0.46	0.08	0.023	0.40	0.17	2.57	0.95	5.75
	Crystalline Silica	41.40	41.40													
	Kaolinite	32.00	16.30				13.80									4.90
	Remainder - mica, feldspar, and small amounts of montmorillonite, siderite and goethite	23.64	9.86	0.64	1.86	0.59	4.98	0.14	0.46	0.08	0.023	0.40	0.17	2.57	0.95	0.85
Avonlea Stoneware	Chemical Analysis	100.18	63.48	0.45	2.57	0.84	19.65	0.52	1.27	0.28	0.087	0.20	0.70	1.81	1.23	7.09
	Crystalline Silica	36.08	36.08													
	Kaolinite	27.50	12.73				10.95									3.32
	Remainder - feldspar, mica, montmorillonite, chlorite, chlorite-vermiculite mixed layer (tentative), and very small amounts of gypsum, jarosite, goethite and carbonaceous material	36.60	14.67	0.45	2.57	0.84	8.70	0.52	1.27	0.28	0.087	0.20	0.70	1.81	1.23	3.27
Halbrite Kaolinized Sand	Chemical Analysis	100.36	71.06	0.32	0.71	0.59	18.48	0.08	0.44	0.19	0.18	0.36	0.12	1.31	0.37	6.15
	Crystalline Silica	48.00	48.00													
	Kaolinite	32.00	14.82				12.73									4.45
	Remainder - principally mica, feldspar, and small amounts of gypsum and carbonaceous material	20.36	8.24	0.32	0.71	0.59	5.75	0.08	0.44	0.19	0.18	0.36	0.12	1.31	0.37	1.70
12-S-445 Kaolinized Sand	Chemical Analysis	100.42	66.32	0.13	1.14	0.88	20.80	0.16	0.61	0.05	0.026	0.66	0.12	0.69	0.78	8.05
	Crystalline Silica	38.20	38.20													
	Kaolinite	43.00	19.91				17.11									5.98
	Remainder - principally mica	19.22	8.21	0.13	1.14	0.88	3.69	0.16	0.61	0.05	0.026	0.66	0.12	0.69	0.78	2.07
Cactus Hills Sandy Fire Clay	Chemical Analysis	99.74	65.44	0.32	0.71	0.93	21.84	0.24	0.74	0.22	0.06	0.08	0.07	0.79	0.52	7.78
	Crystalline Silica	41.20	41.20													
	Kaolinite	37.00	17.13				14.71									5.14
	Remainder - principally mica	21.54	7.11	0.32	0.71	0.93	7.11	0.24	0.74	0.22	0.06	0.08	0.07	0.79	0.52	2.64
Cactus Hills Plastic Fire Clay	Chemical Analysis	100.49	53.76	0.19	1.00	0.93	29.10	0.36	0.37	0.21	0.057	0.70	0.08	0.19	1.56	11.97
	Crystalline Silica	19.48	19.48													
	Kaolinite	62.00	30.10				25.87									9.01
	Gibbsite	2.40					1.07									0.73
Remainder - small amounts of goethite, gypsum, carbonaceous material, and other minerals not accounted for	13.61	4.18	0.19	1.00	0.93	1.56	0.36	0.37	0.21	0.057	0.70	0.08	0.19	1.56	2.21	
10-S-1 Ball Clay	Chemical Analysis	100.55	54.36	0.06	0.86	1.03	28.28	0.46	0.86	0.10	0.49	0.25	0.60	0.08	2.24	10.98
	Crystalline Silica	17.90	17.90													
	Kaolinite	62.00	29.17				25.07									8.76
	Gibbsite	1.00					0.65									0.35
Remainder - small amounts of unidentified minerals, gypsum, goethite and carbonaceous material	18.65	7.29	0.06	0.86	1.03	2.56	0.46	0.86	0.10	0.49	0.25	0.60	0.08	2.24	1.77	
11-S-1 Ball Clay	Chemical Analysis	99.96	56.52	0.13	0.79	1.08	26.98	0.26	0.72	0.25	0.31	n.d.	0.48	0.17	0.59	11.68
	Crystalline Silica	22.92	22.92													
	Kaolinite	60.00	27.78				23.89									8.34
	Gibbsite	1.20					0.78									0.42
Remainder - small amounts of unidentified minerals, gypsum, goethite and carbonaceous material	15.84	5.82	0.13	0.79	1.08	2.32	0.26	0.72	0.25	0.31	n.d.	0.48	0.17	0.59	2.92	
15-S-3 Ball Clay	Chemical Analysis	100.44	60.00	0.06	1.99	1.13	22.92	0.16	1.08	0.91	0.24	n.d.	0.32	0.51	1.89	9.23
	Crystalline Silica	27.16	27.16													
	Kaolinite	47.00	21.76				18.71									6.53
	Remainder - montmorillonite, small amounts of unidentified minerals, and minor amounts of gypsum, carbonaceous material and goethite	26.28	11.08	0.06	1.99	1.13	4.21	0.16	1.08	0.91	0.24	n.d.	0.32	0.51	1.89	2.70



10 to 15 per cent montmorillonite. If this amount of montmorillonite, having an approximate formula suggested by Ross and Hendricks(48), is deducted, the balance has the approximate composition shown for the 11-S-1 remainder in Table 11.

The remainders for 15-S-3 less montmorillonite, for 10-S-1 and 11-S-1, and for the Cactus Hills Plastic Fire Clay make up approximately 12 to 19 per cent of the samples. This total consists of small percentages of gypsum, goethite, carbonaceous material, probably a titanium-bearing mineral or minerals, moisture, a carbonate mineral or an aluminium silicate or silicates. The aluminum silicate minerals were not identified but the most likely are montmorillonite, illite or mica, and feldspar. If these minerals occur, they are not present in sufficient quantities to be identified by the procedures employed.

#### Particle Size

The particle size distribution curves for ball clays 2622 and 92 from the Willows area, and those for kaolinized sands 12-S-4&5, 12-S-4&5 Coarse and 12-S-4&5 Fine, are shown in Figure 18. The curves indicate that the two ball clays and the beneficiated kaolinized sand samples have a high proportion of small particles, and that the unbeneficiated kaolinized sand also contains a substantial quantity.

The amount of plus 200 mesh material for the clays listed in Table 10 gives an indication of their nature. The very fine-grained, plastic ball clays, such as 10-S-1, 11-S-1 and 15-S-3, Cactus Hills Plastic Fire Clay, and Alberta Cypress Hills stoneware sample M-4, have approximately 2% or less of plus 200 mesh particles. Cactus Hills Sandy Fire Clay, the Halbrite sample and 12-S-4&5 -- all of which are refractory kaolinized sands that have, at the best, moderate plasticity -- contain approximately 35 to 45% plus 200 mesh particles. The majority of the plus 200 mesh material is quartz, which is a non-plastic ingredient.

### DISCUSSION AND CONCLUSIONS

In considering the effect of the mineralogical composition of White-mud formation clays on their commercial utilization, it is important to keep in mind how the clays might be used and what the necessary properties of the final product must be. Clays that are used for the production of kaolin for the paper industry should be easy to beneficiate so that a clay having a good white colour, free of grit, can be obtained. Extruded products, such as sewer pipe, require a plastic clay having good workability, low drying shrinkage, good drying qualities, and a long vitrification range. Ball clays and china clays used in the whiteware industry should fire to a white or near-white colour and, in addition, should have certain raw and fired properties necessary for the type of ware which is being manufactured. One of the most important properties of a fire clay is the pyrometric cone equivalent or refractoriness.

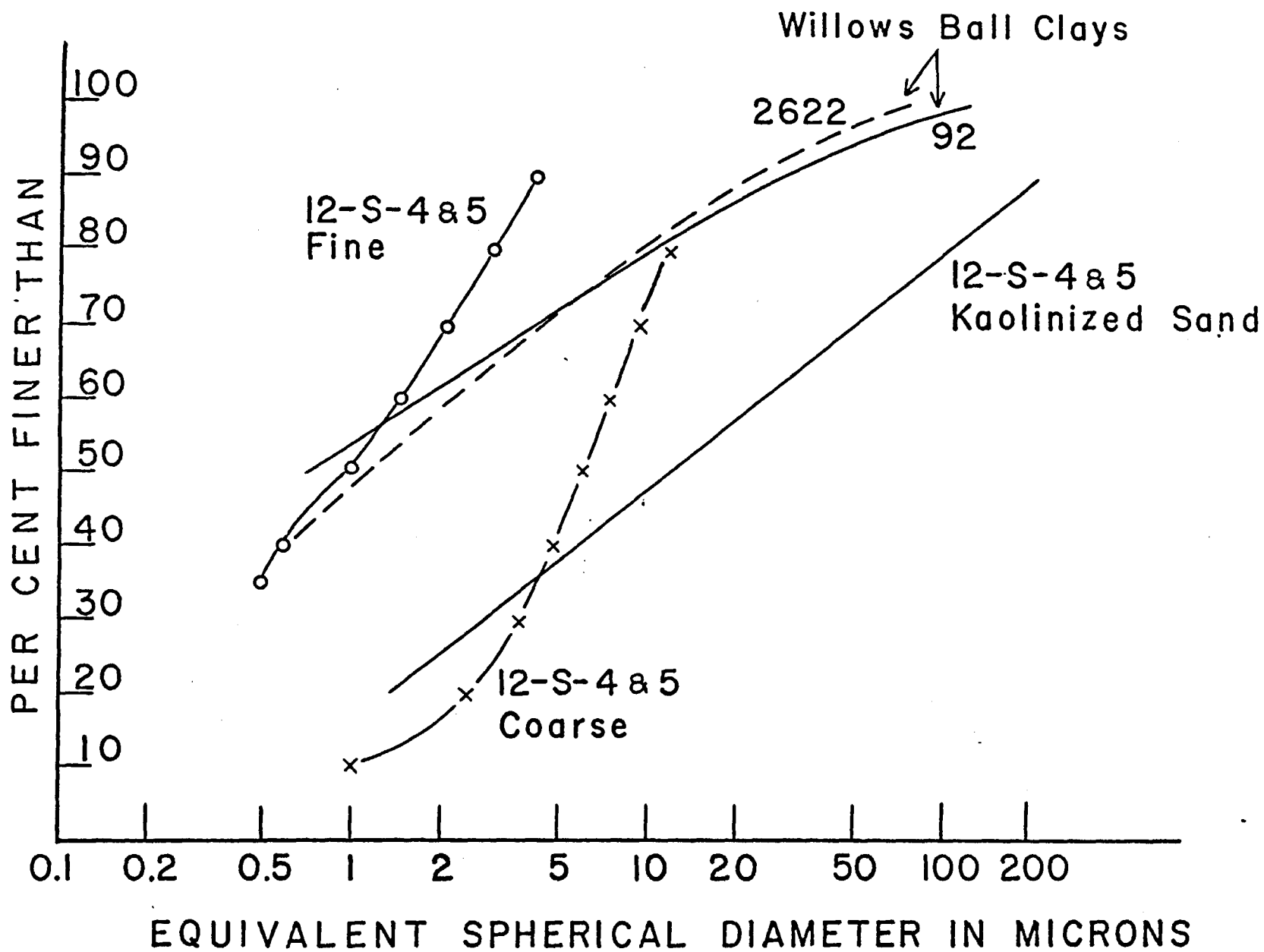


Figure 18. Particle Size Distribution of Some Whitemud Clays.

Differential thermal analysis (DTA) and X-ray diffraction (XRD) studies show that kaolinite is the principal clay mineral in the Whitemud formation clays (Figures 9-17 and Tables 6-9). It is a refractory mineral having a PCE of approximately 34(1763°C) and, consequently, is a desirable constituent of refractory clays. The ball clays of Project 10, 11 and 15, the assorted ball clays such as 2622, 10 and Midland, and the good-quality fire clays (Tables 4,8 and 9), all of which contain high proportions of kaolinite, have PCE's of 30 to 33. Clays from the Avonlea, Eastend and Alberta Cypress Hills areas are less refractory than the ball clays and good-quality fire clays (Tables 1,2,3,4,6,7,8 and 9), because they contain less kaolinite and more low-melting minerals, and are more heterogeneous, than the refractory clays.

The very plastic Whitemud formation clays, such as the ball clays and plastic fire clays that contain a high proportion of fine-particle kaolinite, vitrify readily. The plastic stoneware clays vitrify at a slightly lower temperature than do the plastic fire clays and ball clays because of their higher content of mica, of feldspar and of other minerals, and their lower kaolinite content. The kaolinized sands, sandy fire clays and very sandy stoneware clays do not vitrify easily, because of their high quartz content.

Kaolinite is responsible for a great deal of the plasticity of the Whitemud clays, particularly since it is the mineral which occurs most abundantly in them. Montmorillonite and a fine mica mineral (illite) also occur in many of the clays. According to Marshall(43), montmorillonitic and kaolinitic clays have large-to-medium plastograph peaks, with the montmorillonite peaks having the greatest areas. He showed that the plastograph area for the mica clay mineral "illite" was very small as compared with the other two. Consequently, the Whitemud ball clays and plastic fire clays are very plastic, because all of them contain a substantial amount of fine particle kaolinite and some of them contain montmorillonite. This composition also causes these clays to be tough, greasy, and sticky, and consequently makes them difficult to work. Where kaolinite and mica occur together in these clays, kaolinite is chiefly responsible for increasing plasticity, although fine mica in the form of illite is also plastic. Coarse mica, such as muscovite, decreases their plasticity.

The Whitemud formation clays fire to a white, cream, grey, or buff colour (Tables 1,2,3,4) because of their high kaolinite and quartz contents and the absence of high proportions of minerals high in iron or other colourants. The majority of these clays contain traces of soluble vanadium salts, which may appear as yellow to green efflorescence on the fired product, particularly in porous products.

Quartz is a refractory, non-plastic ingredient that is present in substantial quantities in the Whitemud formation clays. Its particle size varies from coarse to very fine grains. Quartz normally does not greatly lower the refractoriness of those Whitemud clays in which kaolinite is the other principal mineral. The temperature gradient curves of the Cactus Hills Sandy Fire Clay (Fig.7) and 12-S-4&5 kaolinized sand (Fig.8) and the fired results in Tables 1,2,3 and 4, indicate that the clays high in quartz do not vitrify readily.

Quartz has a detrimental effect on plasticity, which, in the Whitemud

clays, is largely offset by the plastic nature of the kaolinite and the small amounts of montmorillonite and illite. Such samples as K-2, K-5 and M-2 (Table 6), which contain over 40% quartz, have moderate to poor plasticity. The plasticity curves of the Halbrite, 12-S-4&5, Alberta Cypress Hills Mixture (K) and Cactus Hills Sandy Fire Clay samples (Figures 2,3 and 4; respectively) indicate that the clays will extrude, although they contain a high proportion of quartz, a great deal of which is plus 200 mesh, and other non-plastic ingredients. These clays produce a weak body when extruded.

The Whitemud formation ball clays, the majority of the fine fractions, and some of the other types contain extremely small particle quartz.

Quartz has a reversible expansion at 573°C (1062°F). Uncombined quartz in the fired material causes severe cracking if the ware is cooled too rapidly through this inversion point. Since the Whitemud formation clays, particularly the stoneware clays and kaolinized sands, contain considerable quartz (Tables 6,7 and 9), some care must be exercised in firing these materials. The temperature gradient shrinkage curves of the Eastend (Dempster), Alberta Cypress Hills Mixture K, Cactus Hills Sandy Fire Clay and 12-S-4&5 samples show that there is a substantial residual expansion at temperatures below 750°C (1382°F), caused principally by the 40% or greater quartz which the samples contain.

It was stated earlier that mica probably does not enhance the plasticity of the Whitemud formation clays to so great an extent as do kaolinite and montmorillonite. Mica acts as a flux in fired ceramics produced from the Whitemud formation clays, and is an undesirable impurity where refractoriness is important. It is a common constituent of the stoneware clays (Tables 6 and 7) where, along with feldspar and sometimes montmorillonite, it reduces the refractoriness of the quartz-kaolinite mixture so that the clays have a PCE of about 20 (Tables 1 and 2).

Coarse mica flakes, which are present in many of the clays, such as Big Muddy Stoneware 6125 (Table 4), cause specking of the fired clay. Mica of this type may cause undesirable specking in some of the white-burning Saskatchewan ball clays such as 10 (Table 4). Removal of most of the coarse mica is necessary in the production of paper-grade or ceramic-grade china clay from the Whitemud formation kaolinized sands, in order to obtain a high quality product. Whitemud clays containing fine mica (illite) may fire to a buff or cream colour because of iron in the crystal structure.

Feldspar was identified frequently in the middle-to-lower beds of the stoneware occurrences, but infrequently in the ball clay, fire clays and kaolinized sand occurrences (Tables 6,7,8, and 9). It was not identified in any of the plus 200 mesh fractions, nor did it appear in any of the very fine fractions. Feldspar is a non-plastic ingredient and an active flux. During firing, it lowers the melting point and vitrification range of the Whitemud clays in which it occurs. The Whitemud stoneware clays are of medium refractoriness and, in general, are good vitrifying clays, partly because of feldspar (Tables 1 and 2, and Figure 6).

Montmorillonite is generally an undesirable component of the Whitemud formation clays, because of its adverse effect on their drying characteristics. The high drying shrinkages and cracking in drying of some of the Whitemud clays, particularly those from Alberta Cypress Hills Area M (Tables 1 and 6), are

caused by montmorillonite. However, a very small amount commonly found in the plastic clays enhances their plasticity and contributes a great deal to their unfired strength. The phenomenally high dry strength of 15-S-3, and possibly of some of the other Saskatchewan ball clays, which were described by Peterson and Tompkins(28) and Worcester(25), is produced by montmorillonite, as well as small-particle kaolinite. Bell(51) has illustrated the effect of 1% of bentonite (montmorillonite) on the plasticity of a non-plastic shale.

The Whitemud clays that contain a substantial amount of montmorillonite frequently fire to a dark buff or light brown colour (Clay M-3, Table 1). This is usually not detrimental to clays used for such products as sewer pipe and flue liners. However, a few of the Saskatchewan ball clays fire to an undesirable off-shade of white, due partly to iron which may occur in montmorillonite.

Montmorillonite is not refractory and, consequently, it helps to reduce the PCE of the kaolinite Whitemud clays. For example, the stoneware clays of Alberta Cypress Hills area M, which contain appreciable quantities of montmorillonite, have low PCE's.

Small quantities of gibbsite occur in such refractory plastic clays as the Cactus Hills Plastic Fire Clay, Big Muddy Fire Clay 6121, and in ball clays 10-S-1 and 11-S-1 (Tables 8 and 9). Gibbsite is an extremely refractory mineral and, consequently, helps to maintain a high PCE in the Whitemud clays in which it occurs. No gibbsite was detected in the stoneware clays or the kaolinized sands.

Goethite, an iron-bearing mineral, was identified in small quantities in the plus 200 mesh fraction, principally in the very plastic clays from the Cypress Hills areas and south-central Saskatchewan (Table 10). Although present in small quantities, this mineral is very undesirable in those Whitemud formation clays that might be used in the whitewares and pottery industries, because it causes the formation of brownish specks on fired ware. It may also be detrimental in the very fine form if it is present in beneficiated kaolin. It is an active flux and lowers the PCE.

Gypsum occurs in very small quantities in the Whitemud clays and, in general, is not harmful to them unless large gypsum pockets are encountered. Gypsum may be a source of soluble salts which cause scumming and efflorescence on dried and fired ceramic products.

Carbonaceous material commonly occurs - usually in very small quantities -- in the Whitemud clays and is easily burned out, according to the DTA curves. It was found, in the plus 200 mesh fraction in the form of soft black flakes, in the majority of the clays shown in Table 10. The very plastic ball clays and fire clays frequently contain a very fine, colloidal type of organic material that colours many of the clays almost black. Carbonaceous material may influence the deflocculation characteristics of slip prepared from these clays. An appreciable amount of it, such as is found in some of the Big Muddy clays, may cause difficulty in firing unless proper provision is made for burning it out during the oxidation period.

Jarosite, calcite, and siderite usually occur in very small quantities, and so they would not frequently cause trouble. Jarosite would probably cause

a scumming or efflorescence problem. If calcite or siderite is encountered in any quantity, some care in firing would be required during the decomposition stage of those carbonates.

In general, an attempt was made to identify the minerals in each sample that were most abundant, because they are the ones which have the greatest influence on the commercial utilization of the clays. It was noted previously that certain minerals, which were present in small quantities and which have an effect on properties, were readily identified by DTA or microscopic means without much detailed work. No concentration and identification of heavy minerals, such as zircon, tourmaline, rutile, apatite, anatase, andalusite and garnet, was attempted. Fraser's work (29,30,31,32) indicated that many of the Whitemud formation clays contain these minerals, particularly zircon, tourmaline and rutile, in small amounts in the minus 270 mesh fraction. Frequently, concentration of the fine fractions of the samples showed that clay minerals were present, such as montmorillonite, illite, or chlorite, which were not identified in the "as received" samples because the amounts were beyond the limits of detection of the methods employed.

The foregoing discussion has dealt with the effect of individual minerals on the properties of the Whitemud formation clays. In an examination of the physical properties of the typical clays, as shown in Tables 1,2,3 and 4, it is apparent that all clays examined from the Western, or Cypress Hills, area are of the stoneware or kaolinized-sand type. Mixtures of the plastic and non-plastic beds are vitrifying clays, which are most suitable for such products as sewer pipe, stoneware, conduits, flue lining, face bricks, building tile, and drain tile. The clays are only moderately refractory, because many contain substantial proportions of mica, and feldspar, sometimes some montmorillonite, and a low to moderate quantity of kaolinite. The amount of quartz is variable, but quartz is generally the most abundant non-plastic ingredient. If montmorillonite, mica and feldspar are absent from the Cypress Hills clays, or present in small quantities, and kaolinite and quartz are the predominant minerals, then the clays become more refractory. For example, sample K-3 (Table 1) has a PCE of 28, and 7-S-1 (Table 2) a PCE of 26. Nevertheless, these PCE values are lower than those of the refractory kaolinized sands, ball clays and fire clays from the south-central area of Saskatchewan.

The properties of the Cypress Hills stoneware clays are not seriously affected by minor amounts of jarosite, siderite, goethite, and gypsum. Vanadium efflorescence on face brick manufactured from these clays should be prevented, since it is usually undesirable.

The Cypress Hills clays owe their plasticity, principally, to kaolinite and to a small amount of montmorillonite and illite. Montmorillonite in very small amounts has a beneficial influence on their plasticity, but in some cases it occurs in sufficient quantities to cause drying difficulties.

The Whitemud clays of the south-central Saskatchewan areas have a much wider range of properties than do the Cypress Hills clays. The stoneware clays from this area generally have the same range of physical properties and mineralogical compositions as the Cypress Hills clays. Kaolinized sand 12-S-4&5 from Fir Mountain, the Cactus Hills Sandy Fire Clay (a kaolinized sand), and Halbrite (Table 4) have PCE's of approximately 30 to 31; they are more refractory than the Cypress Hills sands, such as sample K-2 (Table 1), because the eastern sands contain less mica and feldspar and more kaolinite

than the Cypress Hills samples. The generally greater kaolinite content of the eastern sands is important in a consideration of the amount of kaolinite which can be recovered from the Whitemud sands. A larger amount of kaolinite was recovered from 12-S-4&5 (Sample K07), by air separation, than from the Knollys sample (K13), shown by Spyker et al.(36) and in Table 9. Ball clays and refractory fire clays occur only in the south-central area.

It has been difficult to beneficiate the Saskatchewan kaolinized sands to obtain a good quality paper-grade clay(36). The raw colour of beneficiated samples such as 12-S-4&5 Fine has not been entirely suitable for a high quality paper filler or coater, possibly because of colloidal iron. Fine-particle quartz has been difficult to remove. However, many of these sands contain a substantial proportion of kaolinite, and a solution may be found to improve the raw colour of the clay fraction. According to Spyker et al.(36), 12-S-4&5 Fine has more than 50% of the particles smaller than 1 micron. Thus, they believe this material should be suitable for a filler in linoleum, rubber, and coloured paper. Beneficiated samples such as 12-S-4&5 Coarse are suitable for ceramic-grade kaolin(36).

The very refractory, high-strength, light-firing ball clays and the very refractory plastic fire clays of the south-central Saskatchewan Whitemud formation generally occur in the upper portion of the formation, above kaolinized sands. If gibbsite occurs, it usually appears in the upper seams of the plastic beds. If a mica mineral and/or feldspar occur in the ball clays or plastic fire clays, they usually appear in the lower seams of the plastic beds. Mica (illite) was identified in them more frequently than was feldspar. Kaolinite and quartz are the principal constituents of all seams. Because of the distribution of the minerals in these plastic refractory clays, the most refractory materials generally occur in the upper seams.

Some of the Saskatchewan ball clays have an extremely high dry strength and are very plastic, because of very fine-particle clay minerals, principally kaolinite. Montmorillonite contributes greatly to the high dry strength of some ball clays. Many of these clays fire to a white or near white colour. Occasionally, some of the seams contain coarse goethite or coarse mica which causes the fired clay to have a speckled appearance unless these minerals are removed. Whitemud formation ball clays containing montmorillonite, illite or fine goethite may fire to an off-white colour. The clays that fire to a speckled appearance or to an off-white shade may be, unless suitably treated, detrimental to a whiteware body for which white colour is important.

Plastic and sandy clays from the Whitemud formation, consisting mainly of kaolinite and quartz, can be successfully blended to form high-duty or lower quality refractories. Gibbsite, which occurs in some of the plastic fire clays, raises the PCE.

In general, the Whitemud formation provides good quality stoneware clays, ball clays, fire clays and kaolinized sands, containing principally kaolinite and quartz and frequently mica, feldspar and montmorillonite. Gibbsite occurs in some of the plastic refractory clays. DTA indicates that there should be no problem in firing the Whitemud clays. Contaminating minerals such as carbonaceous material, siderite, jarosite and gypsum should not prove troublesome, because they are present in minor quantities. The clays do not contain dolomite, calcite and pyrite, which are common sources



of firing trouble in many other Canadian clays and shales. Usually, the composition of the Whitemud clays results in a medium to long firing range, whereas in many other Canadian clays the firing range is very short.

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