

EVALUATION OF KAOLIN IN THE WHITEMUD FORMATION OF ALBERTA FOR USE AS PAPER FILLER

by

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DISCLAIMER

This study was undertaken at the request of Alberta Forestry, Lands and Wildlife, and sponsored jointly by Alberta Forestry, Lands and Wildlife and the Alberta Research Council. Every possible effort was made to ensure that the work conforms to accepted scientific practice. However, neither the Alberta Research Council, nor any of their employees, makes any warranty, expressed or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favouring by the Alberta Research Council. The views and opinions of the authors expressed herein do not necessarily state or reflect those of the Alberta Research Council.

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1. EXECUTIVE SUMMARY

1.1. Background

This study is a follow-up to an earlier study of kaolin prospects in Alberta and adjacent regions. to evaluate the resource potential of the kaolin for use as paper filler. Its objective is to provide an unequivocal evaluation of Alberta kaolins, by comprehensive filler quality testing of the best deposit identified from the previous study. That deposit is in the Whitemud Formation, in the Cypress Hills area of southeastern Alberta.

1.2. Fieldwork and testing

The purest, whitest kaolins in the Whitemud Formation occur in kaolinitic sand units. For this study the thickest sand unit in the Alberta Cypress Hills was located and sampled. This followed a thorough review and analysis of the geology of the Cypress Hills area, with particular reference to the Whitemud Formation.

In the next stage, the kaolin was separated from the sand samples and fractionated to filler-grade particle sizes, then analyzed and tested for various properties related to filler use. These tests were conducted in Alberta Research Council laboratories. Arrangements were made also to have the kaolin tested by an established kaolin producer, for an independent evaluation according to industry standards and procedures. The industry tests, by Georgia Kaolin Company, Inc., have not yet been done; when completed, the results will be appended to this report.

In addition to the Whitemud kaolin, a sample of brown carbonaceous clay from coal measures at Wabamun, Alberta is being sent to Georgia Kaolin for testing. Bleaching test will determine whether the carbonaceous matter can be removed to yield a white kaolin product.

1.3. Summary of results and a summary of resul

Essential results of filler quality tests in this study are summarized in the table below. This table compares the properties of optimum Alberta kaolin (in a filler-grade size blend) to those of typical commercial filler clays.

	Whitemud 34E-T/B	"Acme" Filler ECCI (USA)	"Grade C" Filler ECCI (UK)
+10 micron %	2	6	5.4
-2 micron %	62	74	50
Brightness	78.3	82.4/7.0	81.0/5.5
SiO,	52.6	46	47.2
A1 ₂ 0 ₃	30.4	38	37.4
Fe ₂ 0 ₃	1.7	0.89	0.96
TiO ₂	1.0	1.5	0.14
CaO Canada	0.1	0.08	0.11
Mg0	0.7	0.10	0.18
K ₂ 0	2.3	0.42	1.41
Na ₂ 0	0.3	0.20	0.07
L.0.I.	9.8	13.4	12.5
Kaolinite	62	95 <u>- 1 - 1 - 1 - 1 - 1 - 1 - 1 - 1 - 1 - </u>	90
Illite	20	3	9
Quartz	13	-	1
Feldspar	4	¥ <u>-</u>	_ 8
Anatase and/or Ilmenite + Rutile	1	1.5	-

1.4. Conclusions

Test results confirm that Whitemud kaolins in Alberta are of submarginal quality for paper filler use, due to excessive amounts of free quartz, feldspar and illite. The quartz-feldspar content (~18%)

would cause high abrasiveness - unacceptable for modern paper machines. The illite content (20-26%) is a source of discolouration that would likely resist chemical bleaching methods for whitening the clay.

Apart from quality, the major shortcoming of Alberta kaolins is in geological extent. Whitemud kaolinitic sands are not thickly or extensively developed in Alberta, as they are in Saskatchewan. Thus, no sizeable reserves of Whitemud kaolin can be identified in Alberta.

1.5. Recommendations

- (1) No further testing of Whitemud kaolin is recommended.
- (2) If bleaching tests on the Wabamun kaolinitic clay prove encouraging, evaluate this clay as a potential source of paper filler.
- (3) Conduct further studies of Alberta limestones with emphasis on their use for production of precipitated calcium carbonate (PCC). Inventory and evaluate limestone resources specifically in the areas of Alberta with potential sites for paper plant establishment. Concurrently, conduct specific deposit studies recommended previously (Hamilton, 1987) to cover prospects for ground calcium carbonate.

2. INTRODUCTION

2.1. Background

This study is a follow-up to an earlier regional study of kaolin prospects in Alberta and adjacent regions, to evaluate the resource potential of kaolin deposits in the province for use as paper filler. It is a continuation of effort to locate suitable mineral raw materials for paper making in Alberta.

The previous study (Scafe and Hamilton, 1985) established the Whitemud Formation as the best potential source of kaolin in Alberta. Results of that study, which included field sampling and tests of selected deposits in southeastern Alberta and southern Saskatchewan, indicated that the Whitemud kaolins would be of submarginal quality for paper filler use. High content of -2 micron quartz (leading to abrasiveness), and the presence of other clay mineral contaminants with the kaolinite (leading to colour and rheological defects) were the determining factors. However, no actual filler quality tests were performed. Previous tests on the Whitemud kaolins in Saskatchewan show substandard reflectance (Brightness) values for use as paper filler, and previous attempts at bleaching the kaolin were unsuccessful.

Since completion of the study by Scafe and Hamilton, a private company conducting beneficiation studies on Whitemud kaolins in Saskatchewan has reported success in upgrading Saskatchewan kaolin to filler quality. The company, Ekaton Industries Inc., has undertaken preliminary development of its deposit for pilot plant testing, toward potential production of kaolin filler clay. In view of this development, a re-examination of the Whitemud Formation in Alberta was recommended, with more intensive testing, to provide an unequivocal evaluation of the Alberta kaolin deposits. This was the objective of the present study.

2.2. Previous work

Scafe and Hamilton (1985) reviewed previous work on the regional character of the Whitemud Formation as a source of kaolin. Hudson (1987) provided new data from beneficiation experiments with the Saskatchewan kaolins. Master (1987) described the geology and resource aspects of Ekaton's kaolin deposit at Wood Mountain in Saskatchewan, although no data are given on extraction/beneficiation of the kaolin or quality of the recovered product.

Crockford (1951) provides the most detailed geological mapping of the Whitemud Formation in southeastern Alberta. Crockford's work is used extensively in this report, supplemented by unpublished information from IXL Industries Ltd. IXL has explored the region in much detail, seeking kaolinitic clays for ceramic use.

2.3. Method and scope of study

The work plan for this study was to sample the best deposit of Alberta kaolin identified by Scafe and Hamilton (1985), and to conduct comprehensive testing of the samples for kaolin paper filler quality. Sample site selection was based on a thorough geological analysis of the Whitemud Formation in the Cypress Hills area of Alberta, using published geological mapping along with field examination of selected outcrops and quarry exposures, with assistance from IXL Industries Ltd. which operates several clay quarries in the region. Fieldwork was performed in October, 1987. From field observations and on-site discussions with IXL personnel, the selected sample site was confirmed as the best available deposit of kaolin in the region.

The deposit is a kaolinitic sand unit comprising about 30 percent kaolin. Samples were processed initially to separate the kaolin from the sand. The kaolin was fractionated into fine $(-2\mu\text{m})$ and coarse $(-12+2\mu\text{m})$ grades and analyzed and tested for various properties related to filler use, to provide baseline analytical data for the Alberta kaolins. Blending tests were then conducted, combining the kaolin

fractionates with commercial high-grade (Georgia) kaolin into various blends and making preliminary tests of each blend for filler quality. All work was performed in Alberta Research Council laboratories.

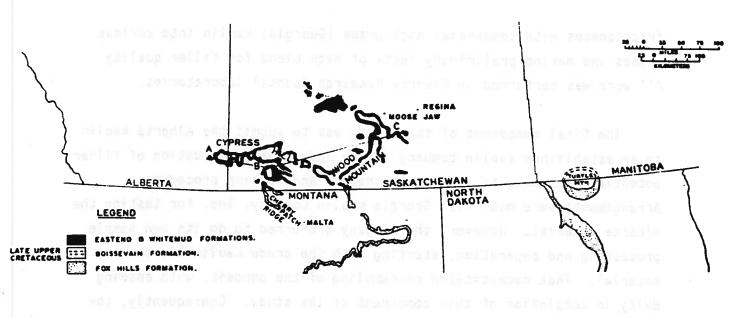
The final component of this study was to submit the Alberta kaolin to an established kaolin company for an independent evaluation of filler potential according to industry standards and testing procedures. Arrangements were made with Georgia Kaolin Company, Inc. for testing the Alberta material. However, the company preferred to do its own sample processing and separation, starting with the crude kaolinitic sand material. That necessitated re-sampling of the deposit, with ensuing delay in completion of this component of the study. Consequently, the company's test results are not embodied in this report, but will be appended later.

Included with the Whitemud material being sent to Georgia Kaolin is a sample of kaolinitic clay from coal measures being mined at Wabamun, Alberta. This clay is dark brown in colour but white-burning, the brown colour being due to carbonaceous matter. Bleaching tests on this clay will attempt to determine whether the carbonaceous matter can be removed to yield a white kaolin product.

3. GEOLOGY AND DISTRIBUTION OF KAOLIN-BEARING FORMATION

3.1 Regional setting of the Whitemud Formation

The kaolin source investigated in this study is a kaolinitic sand unit in the Upper Cretaceous Whitemud Formation. The Whitemud is a succession of non-marine kaolinitic sands, silts and clays, ranging in thickness up to 18 m (Byers, 1969). The formation outcrops extensively in the Cypress Hills area of southeastern Alberta and southwestern Saskatchewan, and also a large area of south-central Saskatchewan (figure 1). Equivalent beds outcropping in the western Alberta Plains are mapped also as Whitemud Formation (Irish and Havard, 1968), but are much less kaolinitic and bear little resemblance to typical Whitemud



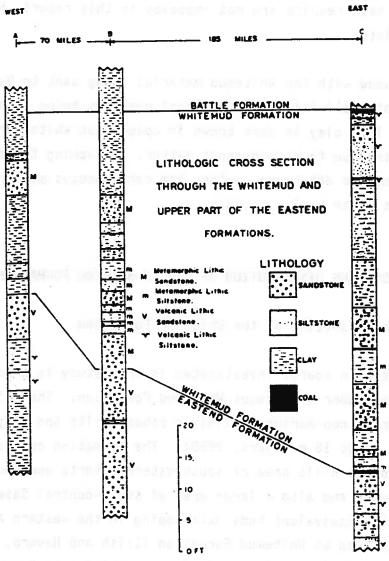


Figure 1. Regional outcrop distribution and lithologies of Whitemud and Eastend Formations (after Byers, 1969).

lithologies: these outcrops are not shown in figure 1 and discussions on the Whitemud Formation in this report do not apply to the western Alberta Plains equivalent.

The Whitemud is thickest in south-central Saskatchewan (figure 1), thinning westward to a maximum of about 9 m in the Alberta Cypress Hills. Lithologies are highly variable, both laterally and vertically within the formation. In Saskatchewan, a general three-fold division of the Whitemud is recognized: a lower "white sandy clay zone"; a middle "brown shale zone"; and an upper "white clay zone". However, this division does not hold throughout the region.

In south-central Saskatchewan the succession is dominated by kaolinitic sand - the "white sandy clay zone" - which comprises the whole of the formation at many localities (Hudson, 1987). The kaolin content reaches 50 to 55 percent in the Wood Mountain area, but decreases westward. The proportion of sand in the formation also decreases westward, and the sediments generally become finer grained (figure 1). In Alberta, the dominant lithologies are clays and silts.

The whitest sediments in the Whitemud Formation are the kaolinitic sands. These sands consist of grains of quartz, feldspar, lithic fragments, mica and chert, with white clay in the matrix. Kaolinite is the dominant clay mineral in the matrix, and in the Wood Mountain area, practically the sole clay mineral (Scafe and Hamilton, 1985). Illite (mica) and fine particle quartz are the minor constituents, increasing in proportion westward.

Clay and silt units in the Whitemud are also kaolinitic, but include significant proportions of illite, mica and quartz, and in some areas, smectite. They vary in colour from white to dark grey. Smectite is most common in the Alberta exposures of the Whitemud; it decreases eastward and is absent or present only as a trace in the sediments of south-central Saskatchewan (Scafe and Hamilton, 1985; Brady, 1962).

The Whitemud Formation was subjected to regional erosion soon after its deposition (and deposition of the overlying Battle Formation shales). Consequently, some or all of the succession was removed from many localities. This erosion is a very significant, controlling factor in the distribution of the Whitemud sediments.

3.2. Alberta Cypress Hills

The outcrop distribution of the Whitemud Formation in Alberta is shown in the geological map of the Alberta Cypress Hills, figure 2. Variations in lithologic character of the formation are also indicated on the map. It is apparent from figure 2 that the dominant Whitemud lithologies in Alberta are clay and silt, and that the successions are highly variable from locality to locality. Sand members that occur are relatively thin and discontinuous; no lower "white sandy clay zone" is recognized.

Kaolinitic sand units are best developed in the vicinity of Eagle Butte, on the northwest flank of the hills. The thickest sand found in the area is in quarry 34 of IXL Industries Ltd (NW-20-8-3-W4). As indicated (figure 2), this sand unit is only 2 to 3 m thick and has limited lateral extent (compare this to the Saskatchewan kaolinitic sands, up to 10 m thick in the Wood Mountain area and extending over 30,000 hectares - Master, 1987). IXL is currently quarrying this sandy unit for ceramic use (as white burning clay for structural ware), but will soon have exhausted its reserves in quarry 34. The company has prospected and test drilled extensively in the Alberta Cypress Hills in search of a similar mineable occurrence, but thus far has been unsuccessful.

The Whitemud Formation is absent over a major part of the Alberta Cypress Hills, as seen from the discontinuous outcrop along the flanks. Crockford (1951) noted definite evidence of removal by erosion on the northern slopes where Whitemud outcrop is lacking. Along the gentler

southern slopes, where lack of outcrop could be due to concealment, the absence of Whitemud has been confirmed by IXL test drilling (pers. comm., IXL staff).

4. KAOLIN ANALYSES AND TESTS

4.1. Sampling

The sampling site selected for this study is quarry 34 of IXL Industries Ltd. This quarry exposes the thickest occurrence of white kaolinitic sand in the Alberta Cypress Hills. The sand unit is layer "E" in the quarry (figure 3). Samples were taken by channel sampling across the bed, sample 34E-Top representing the upper 1.5 m of the bed (figures 4, 5), 34E-Bottom representing the lower 1.5 m.

A sample was also taken from the "E" layer stockpile at the IXL plant in Medicine Hat. This sample, 34E-Stock, represents material quarried from layer "E" but includes also a small amount of the underlying layer "F". This lower bed is a feldspathic, non-kaolinitic sand, similar to sands in the Eastend Formation (which underlies the Whitemud).

Finally, for comparison purposes another sample was taken from the stockpile of equivalent "E" layer sand in IXL's quarry near the town of Ravenscrag, Saskatchewan; this sample is designated RavE. These four samples were used for the kaolin evaluation in this study.

4.2. Procedures

The evaluation steps followed in this study are outlined in figure 6. The essential first step was to separate the kaolin from the sand. The next was to fractionate the kaolin into selected particle size fractions, then to obtain baseline analytical data for each fraction. Two distinct sizes were selected for the fractionation; a fine $(-2\mu\text{m})$ fraction, and a coarse $(-12+2\mu\text{m})$. Sample separations and

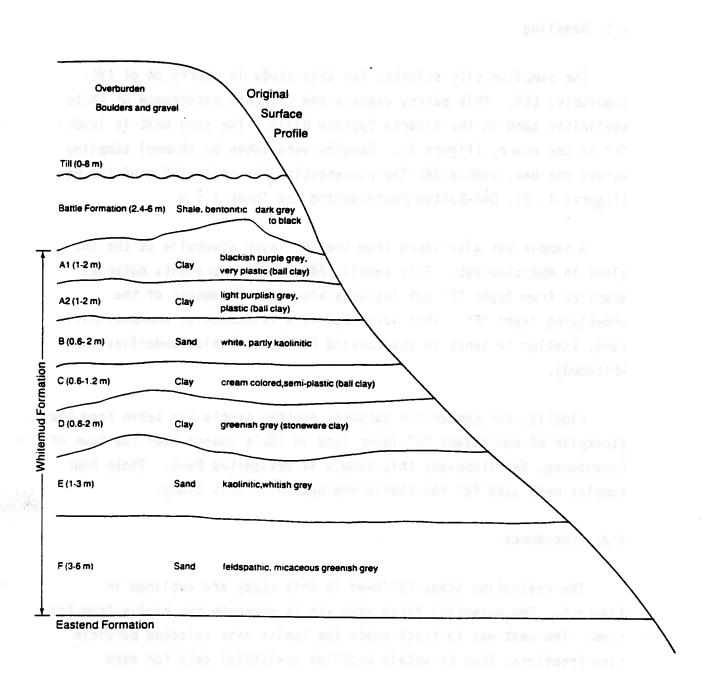


Figure 3. Schematic cross section of IXL quarry 34, Cypress Hills area - sample site for this study.

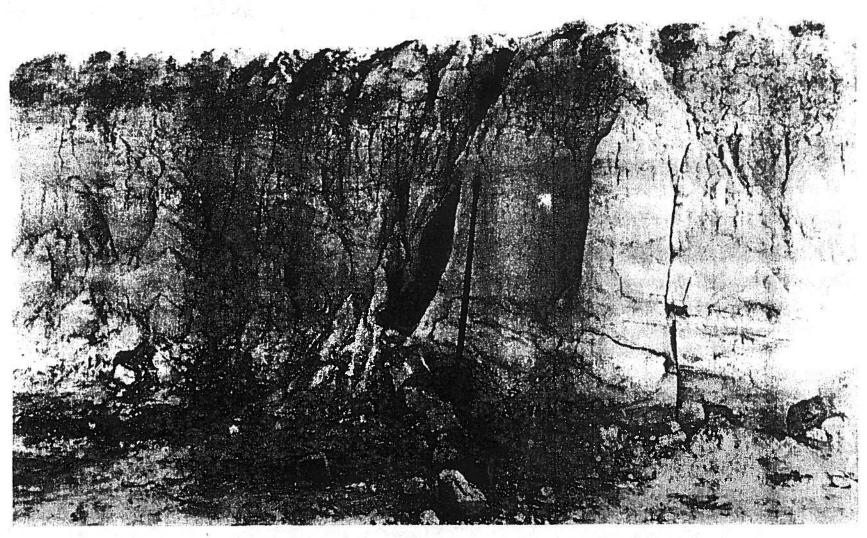


Figure 4. Bed "E" in IXL quarry 34. The sample (34E-Top) from the upper part of Whitemud kaolinitic sand layer "E" in quarry 34 (NW-20-8-3-W4) was taken over the 1.5m distance illustrated by the shovel. The top end of shovel handle is at a line of kaolinite nodules deposited contemporaneously with the sand. The contact with the overlying kaolinitic clay is appproximately 25cm above the tip of the shovel handle.

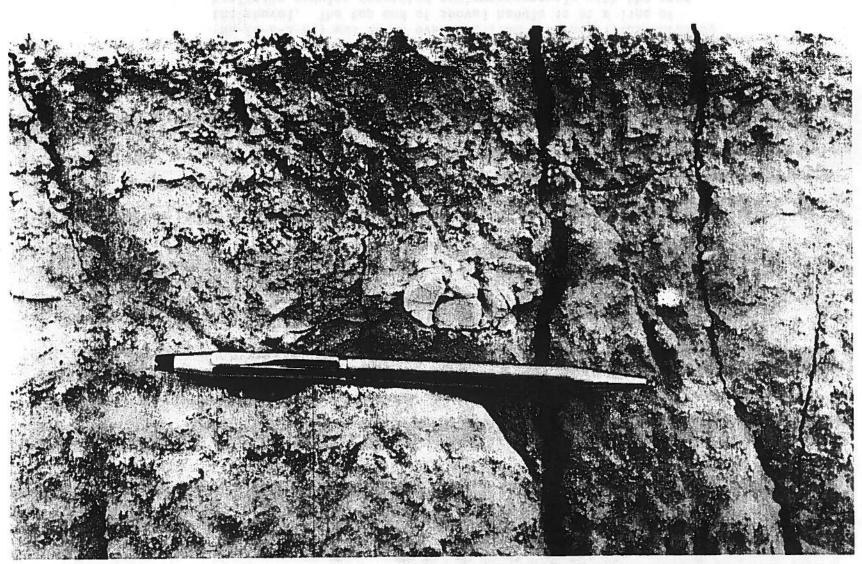


Figure 5. Kaolinite nodules in Whitemud kaolinitic sand, bed "E" of IXL quarry 34. A nodule of pure kaolinite 36mm long projects from the face near the top of the kaolinitic sand. A smaller nodule is directly above the clip of the pen and a third one is at the same elevation as the first at the far left of the photo. Most of the kaolinite is dispersed in the sand in the form of flakes or "books" as shown in scanning electron micrographs in figures 7 to 10 of this report.

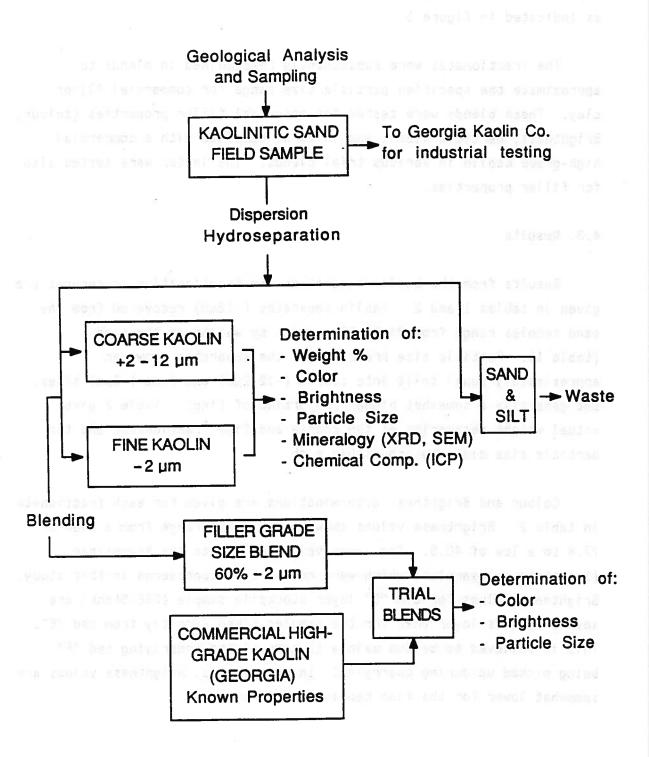


Figure 6. Flow chart of kaolin analyses and tests for this study.

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fractionations were performed by hydroseparation methods as described in appendix A. The analyses and tests performed on the fractionates 1 are as indicated in figure 6.

The fractionates were subsequently re-combined in blends to approximate the specified particle size range for commercial filler clay. These blends were tested for essential filler properties (colour, Brightness, particle size), and in turn, combined with a commercial high-grade kaolin in various trial blends. The latter were tested also for filler properties.

4.3. Results

Results from the kaolin separation and fractionation procedures are given in tables 1 and 2. Kaolin separates $(-12\mu\text{m})$ recovered from the sand samples range from 24 to 28 percent by weight of the sand (table 1). Particle size breakdown of the separates shows an approximately equal split into coarse $(-12+2\mu\text{m})$ and fine $(-2\mu\text{m})$ sizes, but generally a somewhat higher proportion of fines. Table 2 gives actual weight recoveries of the coarse and fine fractionates and the particle size distribution within each.

Colour and Brightness determinations are given for each fractionate in table 2. Brightness values show considerable range from a high of 77.8 to a low of 40.9. The lower values belong to the Ravenscrag (Saskatchewan) samples, which were not further considered in this study. Brightness values for the "E" layer stockpile sample (34E-Stock) are several points lower than for the samples taken directly from bed "E". This is believed to be due mainly to some of the underlying bed "F" being picked up during quarrying. In all samples, Brightness values are somewhat lower for the fine kaolin fractionates.

¹In this report the term "fractionate" refers to the <u>separated</u> fraction (of the particle size range indicated).

TABLE 1. CLAY (KAOLIN) CONTENT OF WHITEMUD KAOLINITIC SANDS SELECTED FOR STUDY

Sample	Initial Sample Weight (kg)	Kaolin (-12μm) Separates (kg)		in Separat Size Breakd -12+2μm		Residual Sand/Silt (kg)	Percent Kaolin
34E-Top ¹	15.26	3.70	0.11	1.67	1.92	11.55	24.2
34E-Bottom ²	11.51	3.23	0.10	1.45	1.68	8.18	28.1
34E-Stock ³	13.17	3.58	0.12	1.75	1.71	9.11	27.2
RavE ⁴	14.71	4.06	0.11	1.47	2.48	10.60	27.6

Channel sample, 1.5 m interval, upper part of bed "E" in IXL quarry no. 34 (NW 1/4 Sec 20, Tp 8, R 3, W4M).

²Channel sample, 1.5 m interval, lower part of bed "E" above.

Sample from stockpile at IXL plant, quarried from bed "E" above (includes small amount of underlying bed "F" - non kaolinitic feldspathic sand).

⁴Sample from stockpile at IXL plant, quarried from equivalent bed "E" at Ravenscrag, Saskatchewan (included for comparison).

⁵Calculated from sedigraph particle size analyses of separates (Appendix A).

TABLE 2. WEIGHT RECOVERIES, PARTICLE SIZE ANALYSES AND BRIGHTNESS/COLOR VALUES OF KAOLIN FRACTIONATES*

Sample	Fraction	Weight Recoveries		Particle +12µm	Size Dist -12+2µm	ribution (% -2µm) C o	1 o a	u r b	Brightness
34E-Top	-12+2μm	1.43	eaby.	1.5	85.0	13.5	88.20	-1.05	4.68	77.8
	-2µm	1.74		-	3.5	96.5	86.77	-1.24	6.88	75.3
34E-Bottom	-12+2µm	1.39		2.0	84.5	13.5	87.55	-1.16	5.40	76.7
	-2µm	1.55		-	3.0	97.0	85.82	-0.94	6.62	73.7
34E-Stock	-12+2µm	1.38		1.0	83.0	16.0	84.35	-1.11	6.73	71.2
	-2μm	1.75		-	5.5	94.5	80.52	-0.89	9.96	64.8
RavE	-12+2μm	1.59		1.0	86.0	13.0	74.15	0.09	II E 6.70	55.0
	-2µm	2.18		(gd) -	3.0	97.0	63.94	1.42	8.07	40.9

^{*}In this report the term "fractionate" refers to the <u>separated</u> fraction (of the particle size range indicated).

The results of chemical and mineral analyses of the kaolin fractionates are presented in table 3. All have kaolinite as the principal mineral constituent, with illite and quartz as the main contaminants. Feldspar is present also in significant amounts, but only in the coarse fractionates. Kaolinite ranges from 68 to 75 percent in the fine fractionates, 36 to 48 percent in the coarse.

All the kaolin fractionates were examined in the scanning electron microscope, to gain an apppreciation of the grain morphology of kaolinite. Salient observations are presented in the SEM micrographs, figures 7 to 10. The observations indicate that kaolinite in the fine fraction occurs mostly as irregular flakes or agglomerates. In the coarse fraction, kaolinite is commonly present as "books" or stacked agglomerates; quartz and feldspar grains are also common.

5. DISCUSSION OF RESULTS

The essential results of analyses and tests in this study are given in table 4. The first two columns of table 4 summarize the properties of Alberta kaolins determined in samples 34E-Top/Bottom and 34E-Stock. The samples, designated BB1 and BB4, have had the fine and coarse fractionates re-combined in blends that simulate a filler-grade particle size distribution, to allow a direct comparison of the properties with those of typical kaolin filler clays (table 5). Particle size distribution and Brightness were determined directly for these blends (appendix B). The chemical and mineral analyses were determined from the baseline data obtained for the fractionates.

The particle size blend selected for BB1 and BB4 $(60\%-2\mu\text{m})$ is average for a filler clay (Scafe and Hamilton, 1985). It comes close to the natural size breakdown of the kaolin separates (table 1) and yields properties that seem to be about optimum for this clay. Increasing the fines content would reduce the level of mineral contaminants, but would also lower the Brightness values.

TABLE 3. CHEMICAL AND MINERAL ANALYSES OF KAOLIN FRACTIONATES

												Mi	n e r	alo	o q v*
Sample	Fraction	SiO ₂	A1 ₂ 0 ₃	Fe ₂	TiO ₂	Ca0	Mg0	K ₂ 0	Na ₂ 0	L.O.I.	Total	K	Igg	Q	F
34E-Top	-12+2µm	58.35	26.73	1.40	0.54	0.04	0.21	3.95	0.44	7.78	99.45	48	21	20	11
-	-2µm	48.15	34.00	1.52	1.64	0.08	0.81	1.43	0.19	11.43	99.25	75	19	6	N PA
34E-Bottom	-12+2µm	59.38	25.21	1.66	0.56	0.04	0.64	3.71	0.29	7.06	98.55	44	23	24	9
	-2µm	48.57	32.64	2.12	0.97	0.10	0.92	1.28	0.19	11.21	98.12	74	19	7	2
34E-Stock	-12+2µm	60.95	24.71	1.79	0.59	0.06	0.68	4.04	0.59	6.78	100.20	36	30	25	9
	-2µm	49.89	31.75	2.55	1.04	0.13	1.07	1.60	0.20	10.68	98.91	68	23	9	
	ten all ga						31 15								

^{*}Mineral analyses from XRD data and chemical balance, assuming compositions as follows:

K - Kaolinite; SiO_2 -46.5%, Al_2O_3 -39.5%, L.O.I.-14.0% (ideal).

I - Illite; $Si0_2-51.2\%$, $Al_20_3-34.5\%$, $K_20-6.8\%$, L.O.I.-7.5% (generalized for hydrous micas - Grimshaw, 1971; Grim, 1953).

Q - Quartz; SiO₂-100%.

F - Feldspar (mainly Orthoclase); $SiO_2-64.7\%$, $Al_2O_3-18.4\%$, $K_2O-16.9\%$ (ideal for orthoclase).



Figure 7. Scanning electron micrograph, $-2\mu m$ fraction, sample 34E-Top. Agglomerates of kaolinite flakes are the only minerals seen in this size fraction from Whitemud kaolinitic sand.



Figure 8. Scanning electron micrograph, $-12+2\mu m$ fraction, sample 34E-Top. Feldspar (F), quartz (Q) and "books" of kaolinite (K) are seen in this size fraction from Whitemud kaolinitic sand.

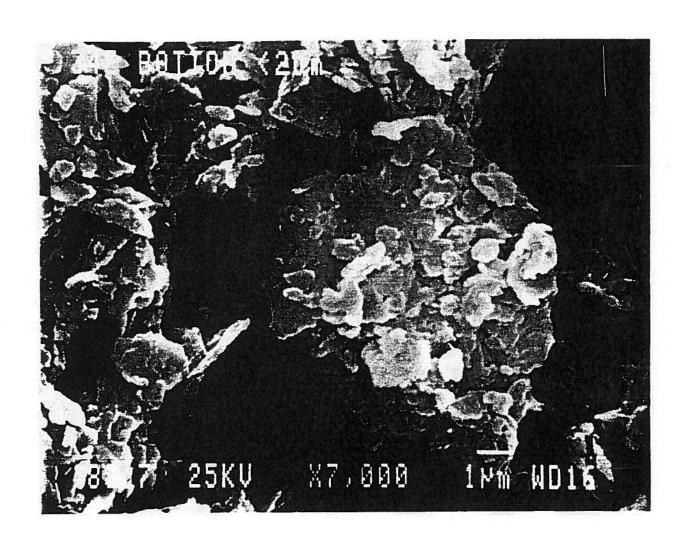


Figure 9. Scanning electron micrograph, $-2\mu m$ fraction, sample 34E-Bottom. Only flakes of kaolinite are present in this $-2\mu m$ size fraction from Whitemud kaolinitic sand.

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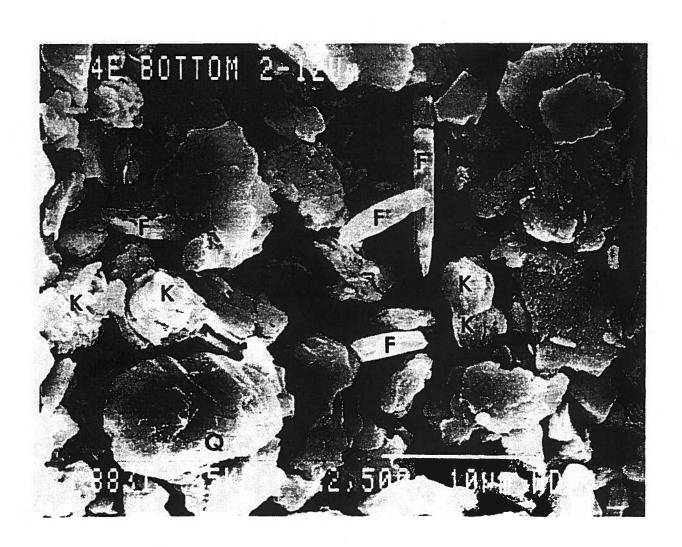


Figure 10. Scanning electron micrograph, $-12+2\mu m$ fraction, sample 34E-Bottom. Agglomerates and "books" of kaolinite (K), cleaved crystals of feldspar (F) and rounded quartz grains (Q) are abundant in this fraction from Whitemud kaolinitic sand.

TABLE 4. PROPERTIES OF ALBERTA KAOLIN, GEORGIA KAOLIN "STANDARD" AND BLENDS, IN PAPER FILLER SIZE GRADES

Properties	Whitem	BB1 ud 34E-T/B ed 60%-2μm)		BB4 aud 34E-: aded 60%		TK Georgia Kaolin "Standard"	BB2 Blend BB1:TK 50:50	BB3 Blend BB1:TK 34:66	BB5 Blend BB4:TF 40:60
V-ME			A.F		Bal	- 21	89	48	
+10 micron t	6_11_	2		2		5	3	4	4
-2 micron %		62		64		68	64	64	65
Brightness		78.3		72.5		86.0	81.9	82.9	80.4
SiO ₂		52.6		54.3		46.9	49.8	48.8	49.9
A1 ₂ 0 ₃		30.4		28.9		38.2	34.3	35.5	34.5
Fe ₂ 0 ₃		1.7		2.2		0.35	1.0	0.8	1.1
TiO,		1.0		0.8		1.42	1.2	1.3	1.2
CaO _		0.1		0.1		0.43	0.3	0.3	0.3
Mg0		0.7		0.9		0.58	0.6	0.6	0.7
K ₂ 0	yle t dura un	2.3		2.6		0.20	1.3	0.9	1.2
Na ₂ O		0.3		0.4		0.04	0.2	0.1	0.4
L.O.I.		9.8		9.1		13.9	11.9	12.5	11.9
Kaolinite		62		54		97	80	85	80
Illite				26		12/12 (FO ()	10	1	10
Quartz		13		15		1.5	7	5	7
Feldspar		4		4		FIRST THIRD STATE A	2	1.5	2
Anatase*		1		1		1.5	1	1	1

^{*}and/or Ilmenite + Ruttle

TABLE 5. PROPERTIES OF TYPICAL FILLER CLAYS (FROM BRISTOW, 1987)

Grade Producer/Country	Grade C ECCI (UK)	Arvors 20B Arvors (France)	Acme ECCI (USA)	Pittong ECCI (Australia)	Alphafill ECCI (Brazil)) 16 E
+10 micron %	5.4	15	6	3	41	
- 2 micron %	50	49	74	78	30	
ISO Brightness*	81.0/5.5	82.7/6.7	82.4/7.0	80.4/9.5	81.6/8.2	
de ora	43.0	48	46	47	46	
SiO ₂	47.2 37.4	37	38	38	39	
A1 ₂ 0 ₃	0.96	0.78	0.89	0.62	0.52	
Fe ₂ 0 ₃ TiO ₂	0.14	0.09	1.5	0.63	0.04	
Can	0.11	0.04	0.08	0.03	0.05	
Mg0	0.18	0.02	0.10	0.1	0.06	
K_0	1.41	1.3	0.42	0.16	0.94	
Na ₂ 0	0.07	0.05	0.20	0.11	0.17	
L.O.I.	12.5	12.3	13.4	13.7	13.5	
Kaolinite	90	89	95	97	95	
Mica (Illite)	9 1110	8	3 CH 3 C	20.22	29.299 5	
Quartz	1	2	Complete Spirit	A Lean SHILL STR	friance of tr	
Feldspar Anatase	MUPLATIES OF A	THE CHARGE AND THE ATOM	1.5	D. WW BUELDS, IN I	April 1 121.12 Fig.	en white

^{*}ISO Brightnesses are about 1-2 units lower than the GE Brightness widely used in the U.S.A. See "English China Clays, Products for the Paper Industry - Test Methods".
All results from English China Clays International Ltd. (ECCI) Laboratories.

In the third column of table 4, properties are given for a commercial high-grade (Georgia) kaolin, designated as sample TK.

Although not strictly marketed as a filler product, this kaolin has properties very similar to a typical filler clay (table 5) and was used as a "standard" for this study.

It is clear from comparison with the properties of this "standard", and with the kaolins in table 5, that the Alberta kaolins have major defects in filler properties. Although Brightness values are only 3 or 4 points below the minimum, the content of mineral contaminants in the Alberta kaolins is far in excess of the limits in typical filler clays. The critical contaminants are quartz and feldspar, both highly abrasive minerals. Illite, although less deleterious, has a negative effect both on rheological properties and Brightness.

The last three columns in table 4 give results of blending trials to determine how filler properties may be enhanced. Blends of the "standard" and the Alberta kaolins were prepared in various proportions as indicated for samples BB2, BB3 and BB5. In BB2, a 50:50 blend yields adequate Brightness, but the quartz-feldspar contaminant remains high (9%). In BB3, a blend of 1 part Alberta kaolin to 2 parts "standard" reduces the abrasives content to 6.5 percent, still well above the maximum (3%) in typical filler clays (table 5). It appears, therefore, that blending with high-grade kaolin filler clay as a means of upgrading Alberta kaolin has limited practicality.

6. CONCLUSIONS

From the foregoing considerations, it must be concluded that Whitemud kaolins in Alberta are of submarginal quality for paper filler use. Some encouraging results were obtained from the tests: good particle size distribution in the kaolin separates; Brightness values up to 78 percent (without any bleaching); absence of smectite. However, excessive amounts of free quartz, feldspar and illite disqualifies the kaolin for paper filler use. The quartz-feldspar content (~18%) would cause high abrasiveness that is unacceptable for modern paper machines.

The illite content (20 to 26%) is a source of discolouration that would most likely resist chemical bleaching methods for whitening the clay.

Apart from quality, the major shortcoming of Alberta kaolin is in geological extent. Whitemud kaolinitic sands are not thickly or extensively developed in Alberta, as they are in Saskatchewan. The thickest sand unit that could be found is the 2-3 m thick bed sampled for this study. That bed currently is quarried for brick clay and will be mined out within five years. Thorough geological survey of the region has failed to locate any other similar prospects. Thus, no sizeable reserves of Whitemud kaolin can be identified in Alberta.

7. INDUSTRIAL KAOLIN TESTING

Part of the work plan for this study was to have tests of Alberta kaolin made by an established producer. Companies producing kaolin paper filler clays (mainly from Georgia) maintain state-of-the-art testing facilities and procedures for their own raw material evaluation and quality control. In addition to the analyses and tests performed in this study, a comprehensive industrial testing program could include:

- abrasion tests.
- bleach response tests.
- viscosity determinations (at solids contents up to 70%).

Abrasion and bleach response tests are critical tests for evaluation of Alberta kaolins. These tests could not be done readily in Alberta Research Council laboratories.

Viscosities are not as critical for filler clays as for coating clays, but are part of a kaolin evaluation. To be valid, the determinations must be under controlled sample processing conditions with respect to the use of dispersants, freeze-thaw treatments, etc. The kaolin separation procedures used in this study probably would have invalidated any viscosity measurements.

For this study, arrangements have been made with Georgia Kaolin Company, Inc. to test the Alberta kaolins. The company will be provided with a freshly dug, bulk sample of the crude kaolinitic sand, on which a full range of tests will be run. Results of the tests when completed will be appended to this report.

Included with the Whitemud material being sent to Georgia Kaolin is a sample of kaolinitic clay from coal measures being mined at Wabamun, Alberta. This clay is dark brown in colour but white-burning, the brown colour being due to carbonaceous matter. Bleaching tests on this clay will attempt to determine whether the carbonaceous matter can be removed to yield a white kaolin product.

8. RECOMMENDATIONS

1. No further testing of the Whitemud kaolin is recommended. The results from this study, with those to come from the testing arranged with Georgia Kaolin Company Inc., provide all the information required for the evaluation.

It was considered sending the kaolin separates and blends from this study to an independent commercial laboratory for third-party evaluation of the material. Contacts were made and quotations on testing costs were obtained; however, the conclusiveness of results in hand makes further testing expense difficult to justify.

- 2. If bleaching tests on the Wabamun clay prove encouraging, it is recommended that this clay be evaluated as a potential source of paper filler.
- 3. Given the importance of mineral fillers to a paper industry, it is recommended that further effort to locate sources in Alberta be focussed on limestone, as an alternative to kaolin worthy of follow-up. Limestone evaluations should be directed primarily

toward precipitated calcium carbonate (PCC); however, ground calcium carbonate should be covered as well, following previous recommendations (Hamilton, 1987).

Assuming the probability of PCC production from on-site precipitators at future paper plants, it is recommended that limestone raw material resources be inventoried and evaluated specifically for the areas of Alberta with potential sites for paper plant establishment. This study could be concurrent with the specific deposit studies recommended previously (Hamilton, 1987).

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APPENDIX A KAOLIN CLAY SEPARATIONS

Industrial Services Laboratory Report

KAOLIN CLAY SEPARATIONS

Prepared for

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OBJECTIVES

The objective of this project was to determine the required conditions to seperate the kaolin clay fractions from samples provided by the Geological Survey Department, to provide particle size analysis of the final clay fractions and to provide brightness measurements of these fractions.

INTRODUCTION

Four samples of clay and sand mixtures were received by the Industrial Services Laboratory on Nov. 3, 1987. These samples were designated as 34E, 34E Top, 34E Bottom and RAV E.

A meeting was held on Nov. 17 with S. Abboud of Terrian Sciences, M. Baaske of Geology and Dr. E. Bertram and J. Laidler of ISL present. It was determined at this meeting that a portion of one of the samples would be gently ground to break up the larger clumps of material, dry sieved, and submitted for particle size analysis on the -325 mesh fraction. It was also decided that the 12 micron and smaller portion would be removed from the bulk of the samples, particle size analysis measurements done after flocculation, the clays then redispersed and the 2 micron and smaller portion removed and particle size analysis repeated. The particle size analysis was to be done by M. Baaske.

The initial work began on Nov. 18 with the large scale separations started on Nov. 24. The final separations were done on Dec. 19 and all the individual portions of each fraction were combined for final particle size analysis and drying on Jan. 25, 1988. The dried products with a copy of their particle size analyses were delivered to Geological Survey on Feb. 16, 1988.

PRELIMINARY WORK

A portion of the 34 E Top (approx. 1 kg) was passed through a disc grinder with a gap of 3 mm between plates. It was then split to about 300 g and this was then passed though a sieve series consisting of 12, 35, 100, 140, 200 and 325 mesh sieves on a shaker for 15 mins (initial sample was 311.24 g). The results of this are shown in Table 1.

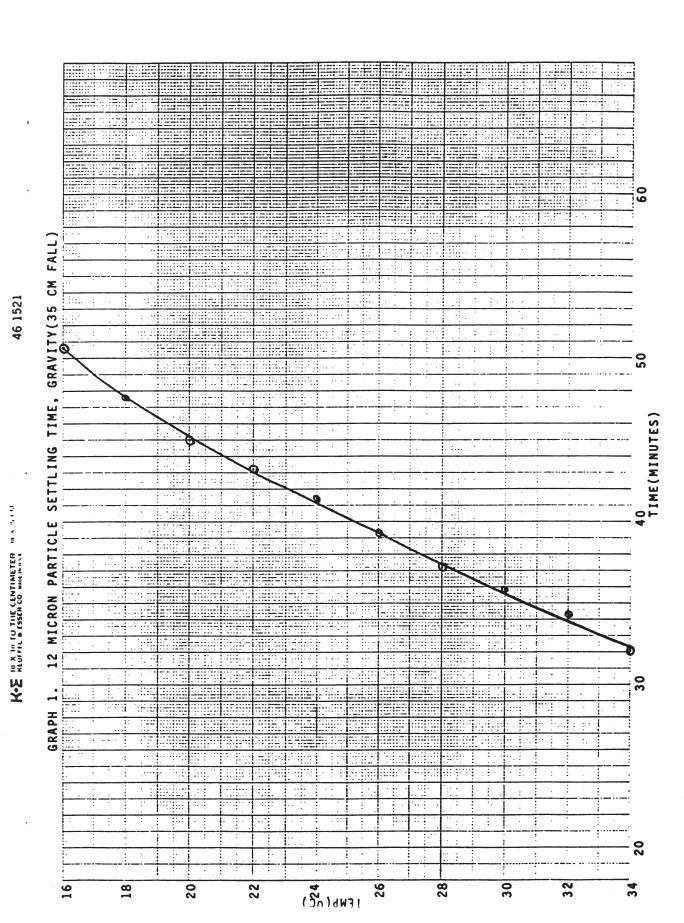
TABLE 1. SIEVE ANALYSIS

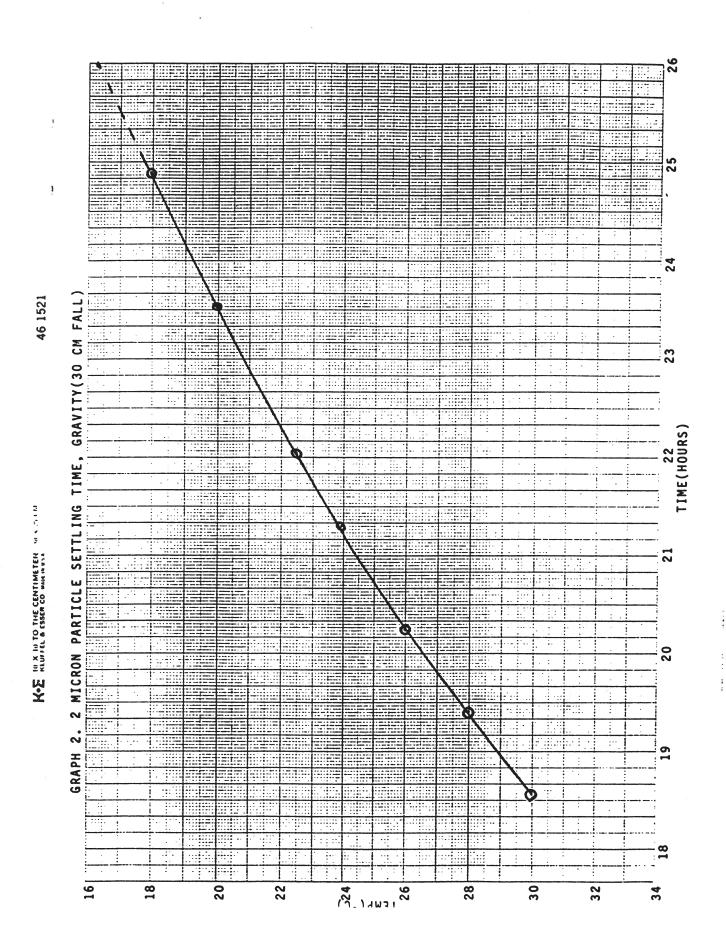
Sieve	Gross	Tare	Net	Per-	
	Wt.(g)	Wt.(g)	Wt.(g)	cent	
-325	382.68	359.13	23.55	7.57	ri ir i
325/200	373.15	358.87	14.28	4.59	
200/140	392.34	373.85	18.49	5.94	
140/100	274.31	251.03	23.28	7.48	
100/35	523.15	375.54	147.61	47.44	
35/12	482.38	412.14	70.24	22.57	
+12	538.69	524.99	13.70	4.40	
			18 1213		
			311.15	99.99	

The particle size analysis of the -325 fraction showed that 67% of this fraction (5.1% of the total sample) was 12 micron and smaller and that 27.5% (2.1% of the total sample) was 2 micron and smaller.

A series of tests were then done on dispersing clay samples in water containing 0.05% Calgorite. Two batches of 34E Top, one of 50 g/L and one of 100 g/L were allowed to soak overnight and then dispersed and allowed to settle according to the data in "NOMOGRAPHS OF SEDIMENTATION TIMES FOR SOIL PARTICLES UNDER GRAVITY OR CENTRIFUGAL ACCELERATION" C.B. Tanner and M.L. Jackson; Soil Sci. Soc. Amer. Proc., vol. 12, 1948, pp. 60-65. Graphs were plotted from the

nomograph on page 61 to determine the settling times of 12 and 2 micron patricles for a 30 cm fall (graphs 1 and 2). The samples were then allowed to settle for 43 min (the calculated settling time for a 35 cm settling of 12 micron particles at 22°C based on the curve from graph 1). The suspended clay was then siphoned off at a depth of 35 cm from the top of the water. This was repeated on both test runs two times and then both were allowed to settle for 2.5 min (settling rate for 50 micron particles for 35 cm at 22°C). All four suspensions of each batch were then treated with enough 20% MgCl₂ solution to give a final MgCl₂ conc. of 0.06%. These were then allowed to settle overnight. The heights of the flocculated clays indicated that the bulk (approx. 80%) of the clay was removed in the first dispersion. It also showed that this settling technique worked just as well on the 100 g/L batch as it did for the 50g/L batch.





LARGE SCALE PREPARATIONS

TWELVE MICRON SETTLINGS

All the clay samples were soaked with enough 0.05% Calgonite in water to cover for at least 16 h. The samples were split into portions to give an equivalent ratio of water to solids of 10:1 (20 L:2 kg) and suspended in calgonite solutions which had been previously allowed to stabilize to room temperature before using. The temperature was monitored during each settling and did not change by more than 0.20c. Over the course of the settlings of the four samples the temperature range was 20-22°C. All settlings were done for a 35 cm fall (times of settling were 45 min at 20°C, 44 min at 21°C, and 43 min at 22°C). The suspended material was then decanted off and set aside. The settled portion was again resuspended to 20 L (35 cm) with 0.05% Calognite in water and allowed to resettle at the previous conditions. The water from this suspension was then decanted and used to resuspend the next 2 kg portion with additional 0.05% Calgonite solution added as necessary to bring it to the proper height. This was done for each 2 kg portion of the sample. All of the suspended material was then flocculated with 0.06% ${\rm MgCl}_2$ (26 g of ${\rm MgCl}_2$ 6 ${\rm H}_2$ 0/20 L of suspension).

After the 12 micron settlings were done the residues from each settling were combined and resuspended to 20 L and allowed to settle for 12-12.5 min (settling time for a 10 cm fall of 12 micron material) and then decanted. This was done twice on each sample followed by flocculation with 0.06% MgCl₂. After allowing the flocculated material to settle overnight the excess liquid was siphoned off and all of the material was combined, with that from the 10 cm settlings being kept seperate from that from the 35 cm settlings.

Samples of each material were taken to determine the solids content and for particle size analysis. The residual material was dried at 80° C, to determine the amount of clays removed. The particle size analysis (Table 2) was done on a Micromeritics Sedigraph 5000ET in

TABLE 2. PARTICLE SIZE ANALYSIS - TWELVE MICRON SETTLINGS

ISL CODE #	SAMPLE	INITIAL	SETTLING	PARTIC	PARTICLE SIZE ANALYSIS	IALYSIS	TOTAL	FINAL	WT. DIFF.
		WT.(kg)	DIST.(cm)	+12 u WT.(kg)	12-2 u WT.(kg)	-2 u WT.(kg)	WT.(kg)	WT.(kg)	(kg)
11-87-102-15-1	34 E	13.17	35		1.409	1.527	2.936	9 11	4 06
			10	0.122	0.342	0.181	0.645	•	
	*			0.122	1.751	1.708	3.581		
17-87-102-15-3	34 E	15.26	35	!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!	1.353	1.837	3.190	11.55	3.71
	T0P		10	0.110	0.312	0.084	0.506		!
				0.110	1.665	1.921	3.696		
IT-87-102-IS-4	34 E	11.51	35	!	1.150	1.540	2.690	8.18	3.33
	B0T.		10	0.098	0.299	0.146	0.543		
				0.098	1.449	1.686	3.233		
11-87-102-15-2	RAV E	14.71	35	8 8 8 8	1.198	2.432	3.630	10.60	4.11
			10	0.106	0.272	0.053	0.431		
				0.106	1.470	2.485	4.061		

0.05% sodium hexametaphosphate in water containing 2 drops of Photoflo.

TWO MICRON SETTLINGS

Before any of the material from the 12 micron settlings was used it was resuspended in reverse osmosis (R.O.) water in order to remove any excess MgCl₂. As the total volume of the material was almost 20 L the suspensions were frozen and then allowed to thaw slowly at room temperature. The clays settled to approximately 5 L total volume each.

The bulk of the clays from the 12 micron separations (from the 35 cm settling) were split into two portions with each being suspended to a total height of 40 cm in R.O. water containing 0.05% Calgorite. These were then allowed to settle for the necessary time as determined from Graph 2. The suspended clays were then siphoned off at a depth of 30 cm. This was repeated a total of 4 times for each clay sample.

Throughout these separations the amounts of -2 micron clays removed and their particle size were mointored. All suspensions were made up to a final concentration of 0.01% MgCl₂ and frozen. They were then thawed at room teperature, the excess liquid siphoned off, combined and allowed to resettle. Any excess liquid was again siphoned off.

The clays from the 10 cm settlings of the 12 micron separations were treated in the same manner for three suspensions to remove the -2 micron clays. The residual material was then suspended twice to remove any 12-2 micron material. These were treated the same as for the previous suspensions.

All portions of the -2 micron clays and all portions of the -12 micron clays were combined for each sample. Samples were then taken for final particle size analysis. All -2 micron clays were concentrated to approx. 50% by drying at 50° C and then freeze dried. The -12 micron clays were dried at 80° C. The particle size analysis and weights of

each are shown in Table 3. There was a small amount of +12 micron material recovered from each sample and these are also included in Table 3.

TABLE 3. FINAL PARTICLE SIZE ANALYSIS

ISL CODE	SAMPLE	FRACTION	WT(kg)-	% D	istribut	ion
				+12u	12-2u	-2u
IT-87-102-IS-1	34 E	+12u	0.348	36.5	50.0	13.5
		12-2u	1.382	1.0	83.0	16.0
		-2u	1.746		5.5	94.5
IT-87-102-IS-3	34 E	+12u	0.310	31.5	59.5	9.0
	TOP	12-2u	1.431	1.5	85.0	13.5
		-2u	1.736		3.5	96.5
IT-87-102-IS-4	34 E	+12u	0.312	31.0	57.5	11.5
	BOT	12-2u	1.389	2.0	84.5	13.5
		-2u	1.548		3.0	97.0
IT-87-102-IS-2	RAV E	+12u	0.277	31.0	62.0	7.0
		12-2u	1.586	1.0	86.0	13.0
		-2u	2.182		3.0	97.0

COLOR AND BRIGHTNESS ANALYSIS

The color and brightness measurements were made with a HunterLab Tristimulus Colorimeter Model D25M-9 (serial #9060) consisting of a Model D25M Optical Sensor and a Model D25-9 Signal Processor according to ASTM C 110-87 "PHYSICAL TESTING OF QUICKLIME, HYDRATED LIME, AND LIMESTONE" and TAPPI 646 os-75 "BRIGHTNESS OF CLAY AND OTHER MINERAL PIGMENTS". This was calibrated at zero with a black plate and then with white standard C2-11680. The white standard coordinates are:

$$X = 83.01$$
, $Y = 85.25$, $Z = 98.19$
 $L = 92.33$, $a = -1.10$, $b = 1.60$

with the Y value being the brightness calibration as compared to ${\rm BaSO}_A$. The results are given in Table 4.

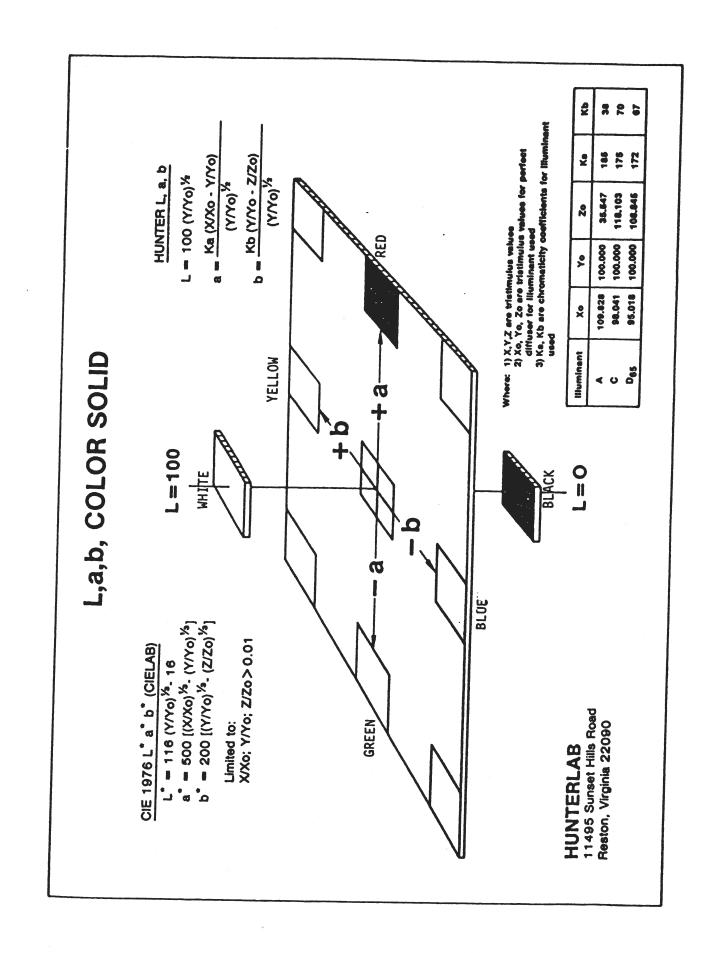
OBSERVATIONS

The two major observations during these separations were that:

- 1. The RAV E sample was quite gray in color as compared to the other samples with an oily odor and all the liquids removed after flocculation were quite reddish in color.
- 2. After each settling to separate the -2 micron clays from the -12 micron clays it was very difficult to resuspend the settled material due to its tendency to form a very dense paste.

TABLE 4. COLOR AND BRIGHTNESS ANAYLSIS

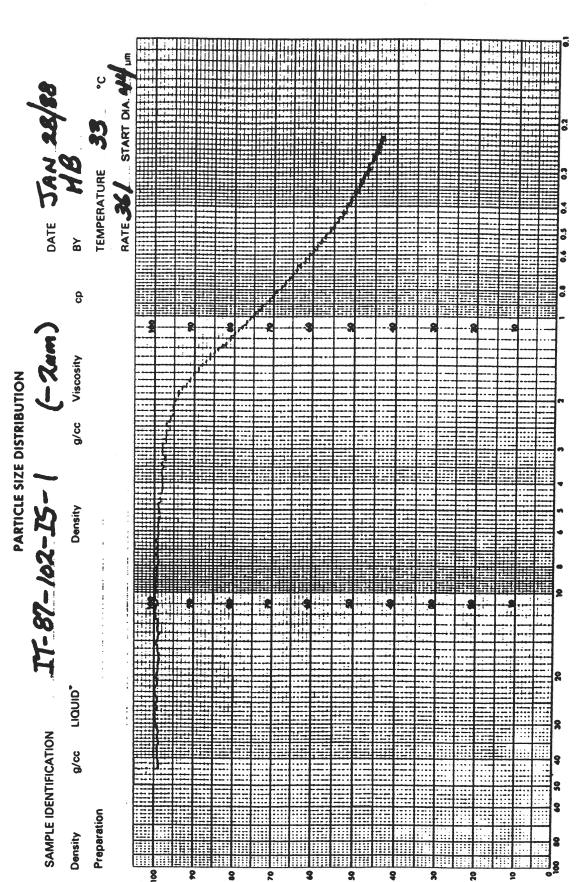
ISL CODE	SAMPLE	FRACTION	•		COLOR	.0R	•		BRIGH	BRIGHTNESS
			AV.	ST.D.	AV.	ST.D.	AV.	ST.D.	(Y _{CI}	(^Y CIE) AV. ST.D.
C2-11680	WHITE STD.		92.31	92.31 ±0.02	-1.10 ±0.00	00.0∓	1.61	1.61 ±0.02	85.25	+0.04
IT-87-102-IS-1	34 E	-2n	80.52	90.0∓	-0.89	+0.01	9.96	±0.18	64.83	+0.10
.		12u-2u	84.35	₹0.03	-1.11	00.0∓	6.73	+0.00	71.15	₹0.03
IT-87-102-IS-3	34 E	-2u	86.77	+0.35	-1.24	₹0.05	6.88	6.88 ±0.14	75.28	+ 0.60
:	T0P	12u-2u	88.20	±0.20	-1.05	€0.03	4.68	00.0∓	77.79	±0.35
IT-87-102-IS-4	34 E	-2u	85.82	€0.0€	-0.94	±0.02	6.62	±0.01	73.66	1 0.08
2	B0T	12u-2u	87.55	±0.01	-1.16	00.0∓	5.40	00.0∓	76.65	₹0.05
IT-87-102-IS-2	RAV E	-2u	63.94	90.0₹	1.42	+0.01	8.07	+0.05	40.89	+0.08
		12u-2u	74.15	±0.16	0.09	±0.02	6.70	±0.02	54.98	+0.23



APPENDIX 1

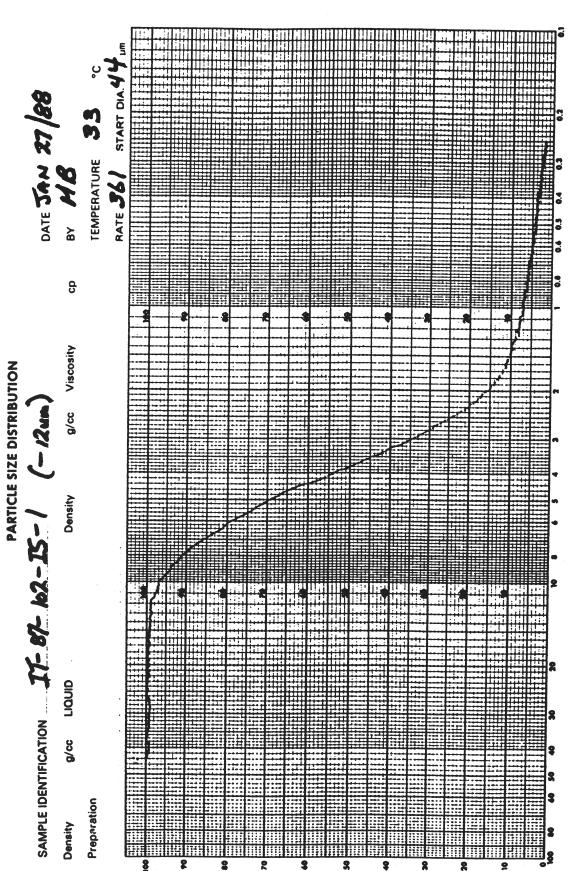
FINAL PARTICLE SIZE ANALYSES

GRAPHS



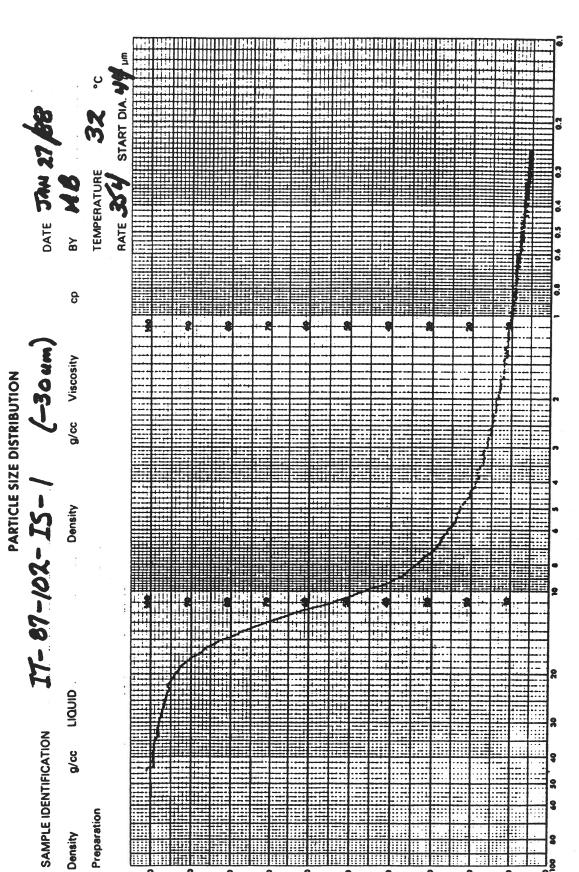
EQUIVALENT SPHERICAL DIAMETER, 11ml

micromeritics •



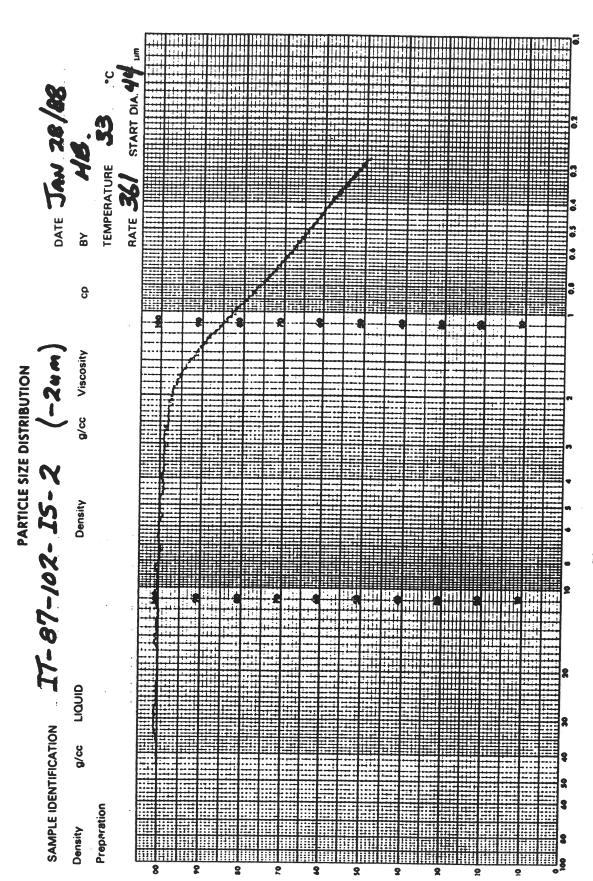
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micromeritics • instrument corporation



EQUIVALENT SPHERICAL DIAMETER, UM

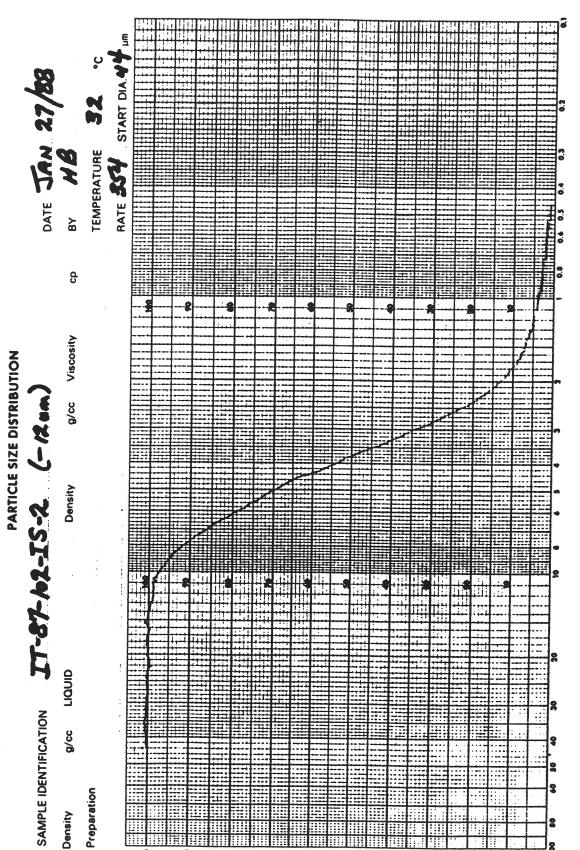
micromeritics •



EQUIVALENT SPHERICAL DIAMETER, um

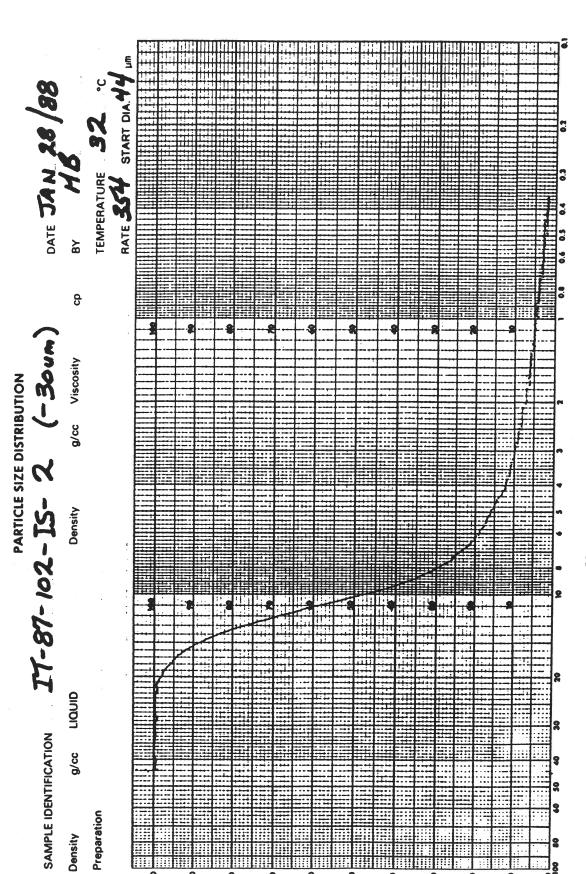
micromeritics •

E :



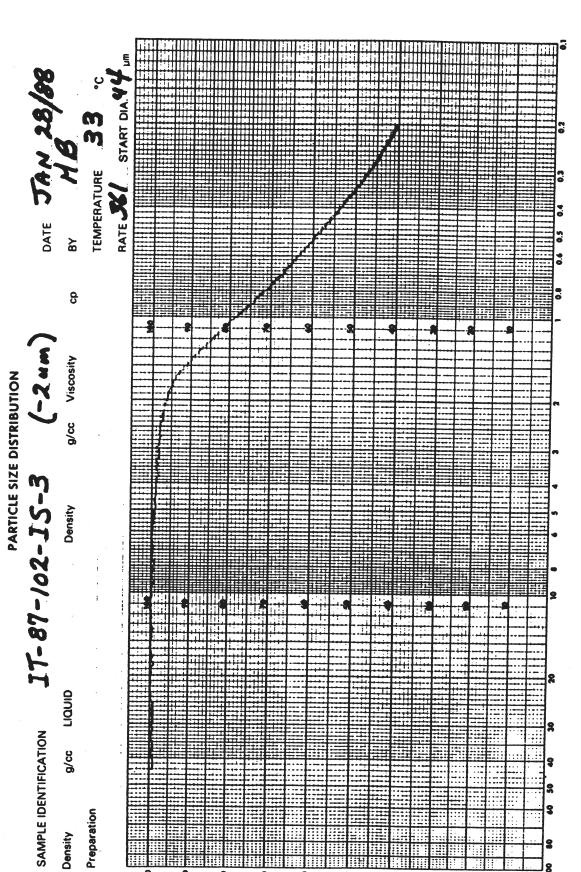
EQUIVALENT SPHERICAL DIAMETER, 1,111

micromeritics "



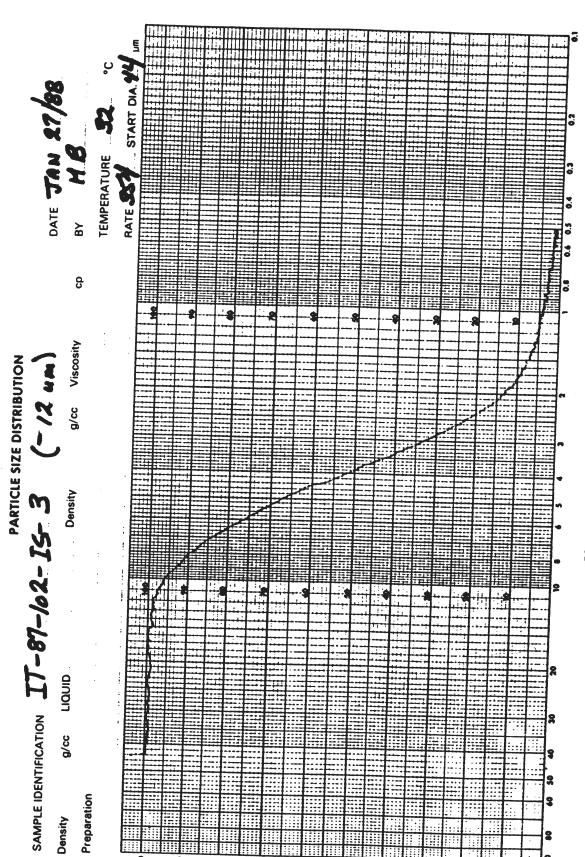
EQUIVALENT SPHERICAL DIAMETER, UM

micromeritics •



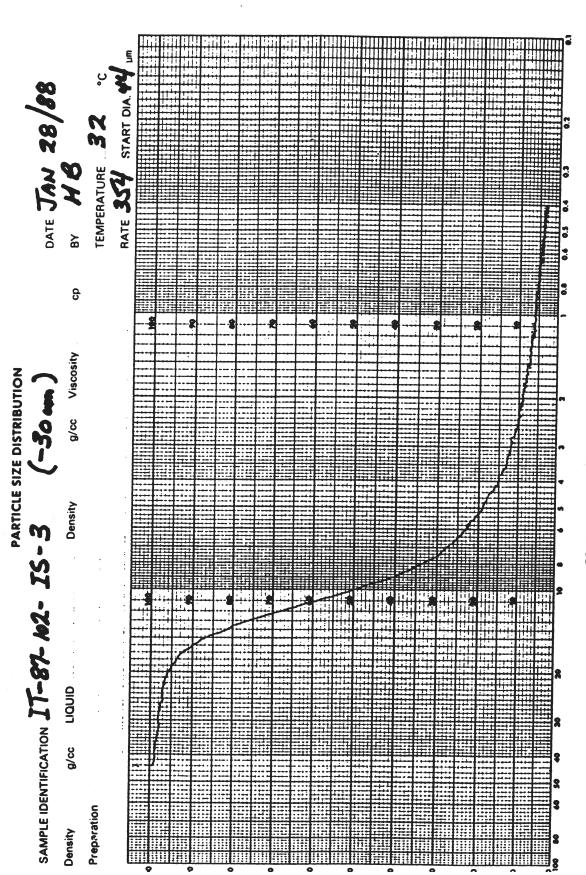
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micromerices • instrument corporation



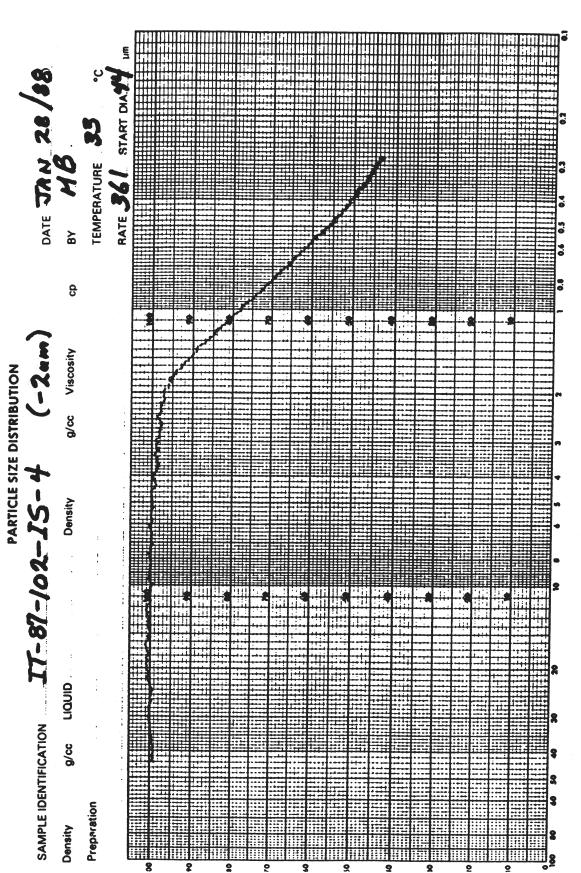
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micromeritics •



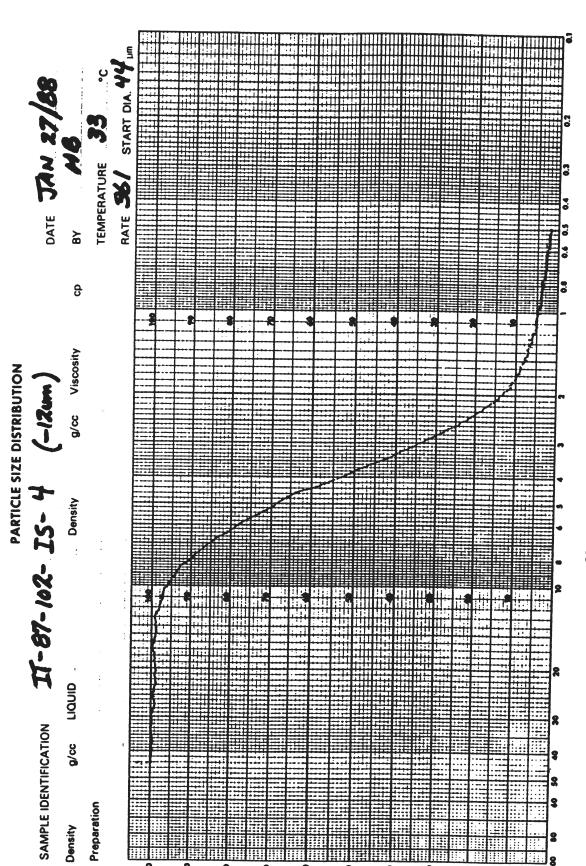
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micromeritics •

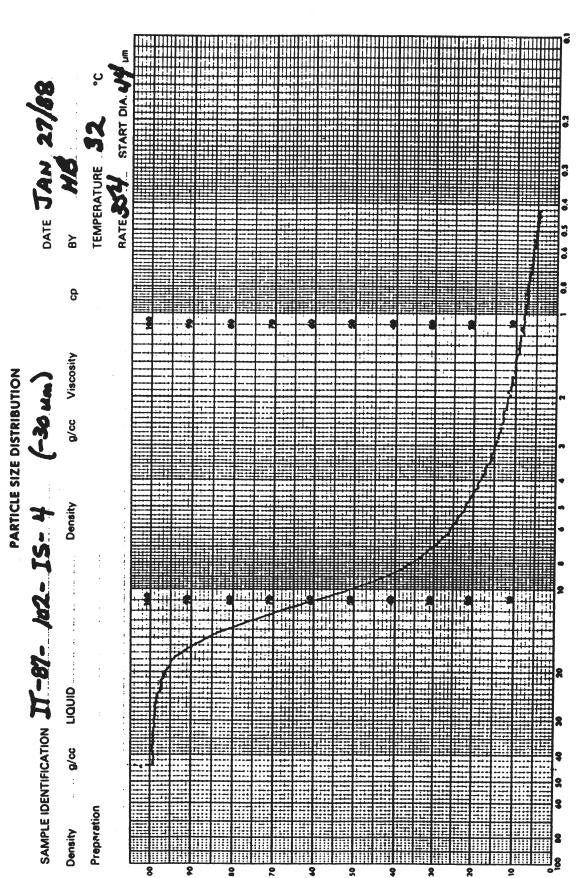


EQUIVALENT SPHERICAL DIAMETER, um

micromeritics •



EQUIVALENT SPHERICAL DIAMETER, um



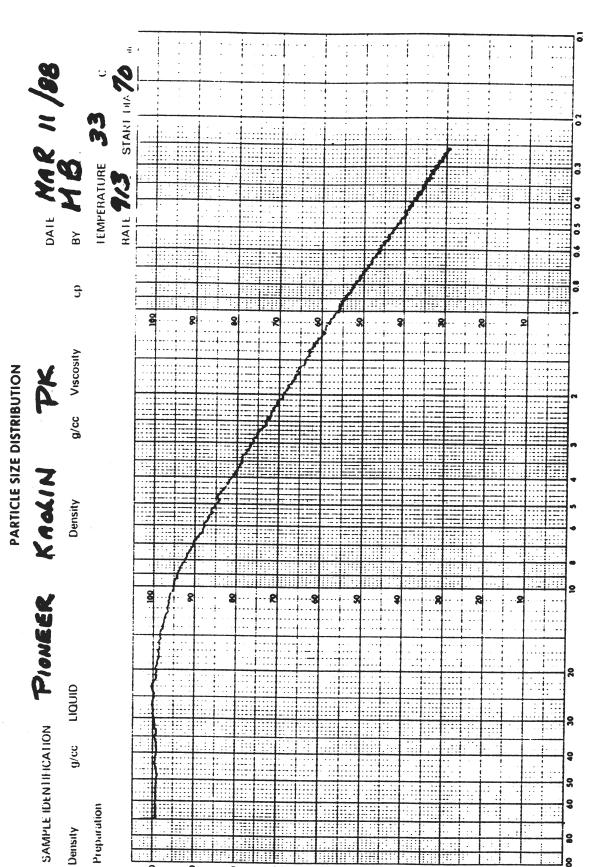
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APPENDIX B

ANALYTICAL DATA FOR KAOLIN "STANDARDS" AND BLENDS

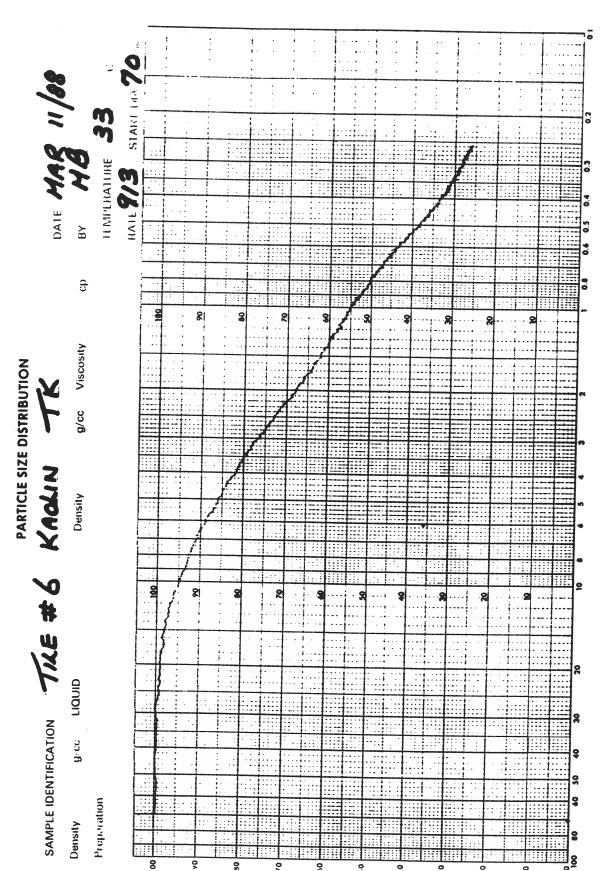
APPENDIX B1

Particle Size Analyses Charts



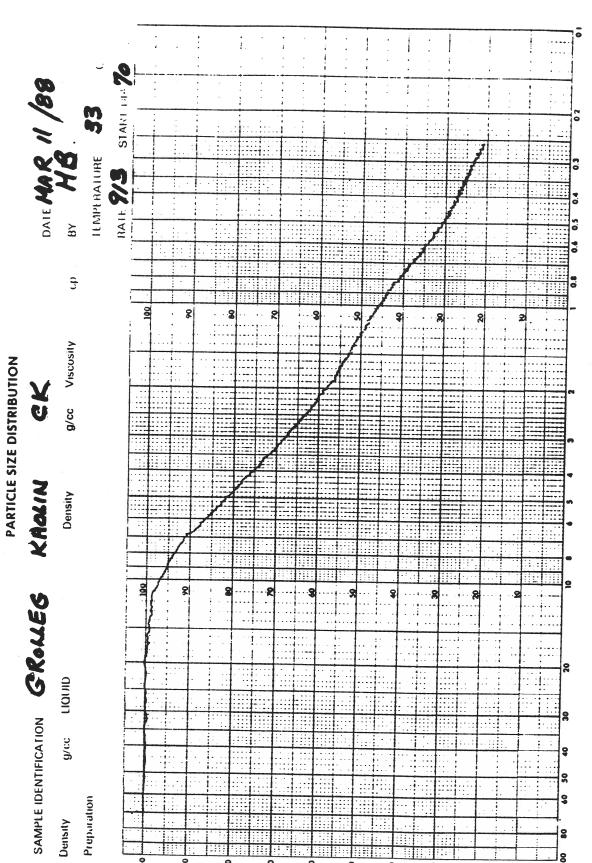
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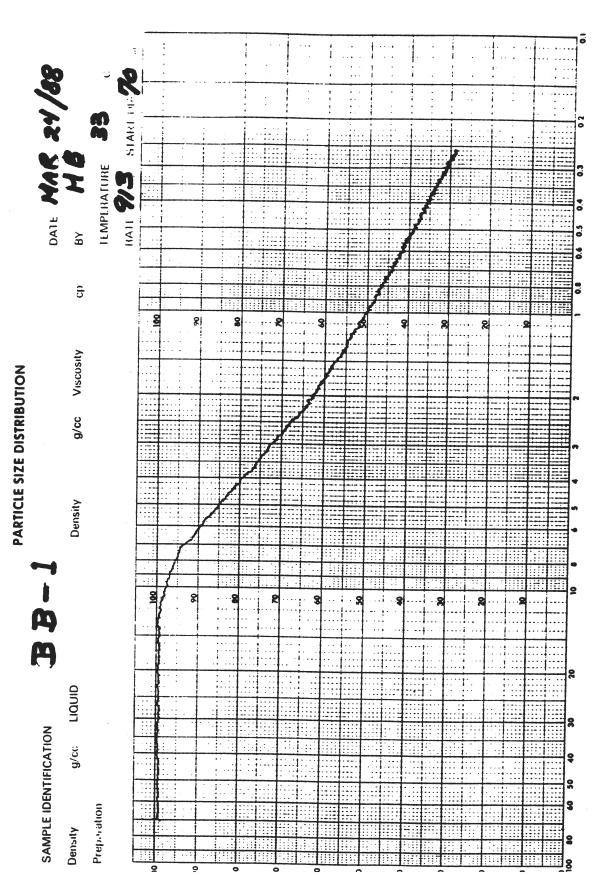
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micromerities '



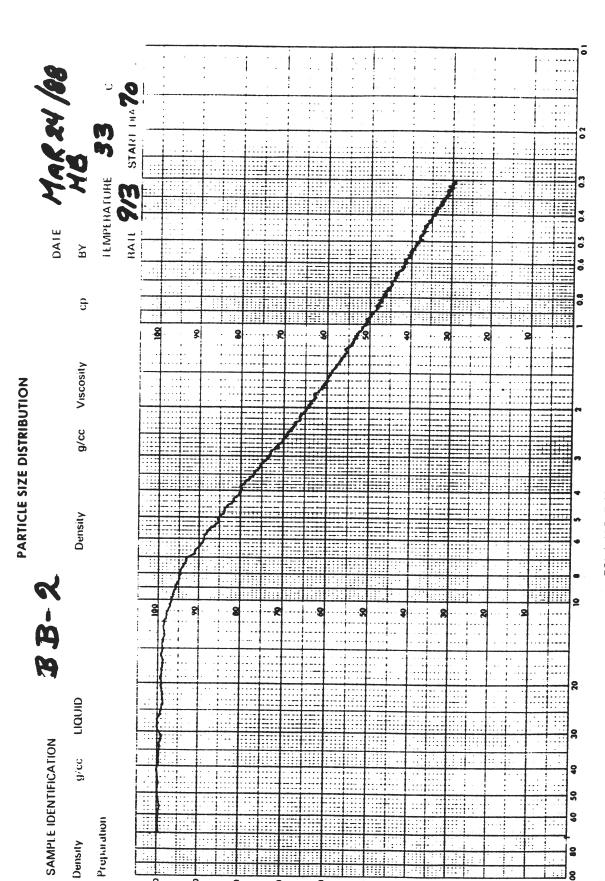
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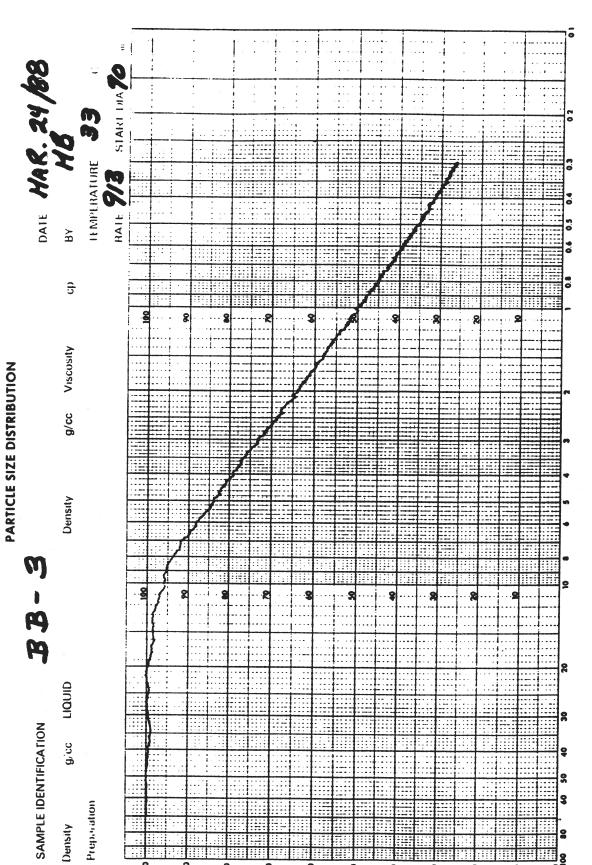
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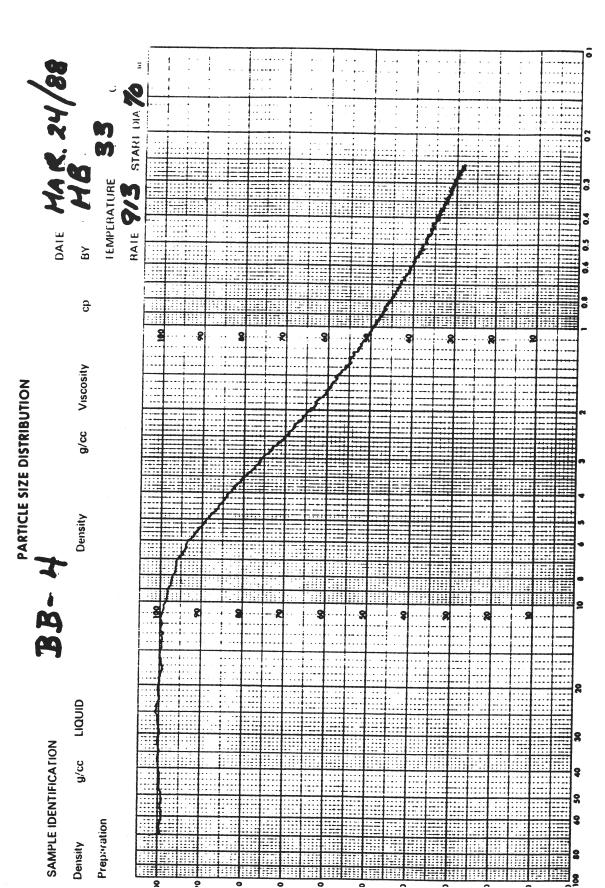
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micromerities (



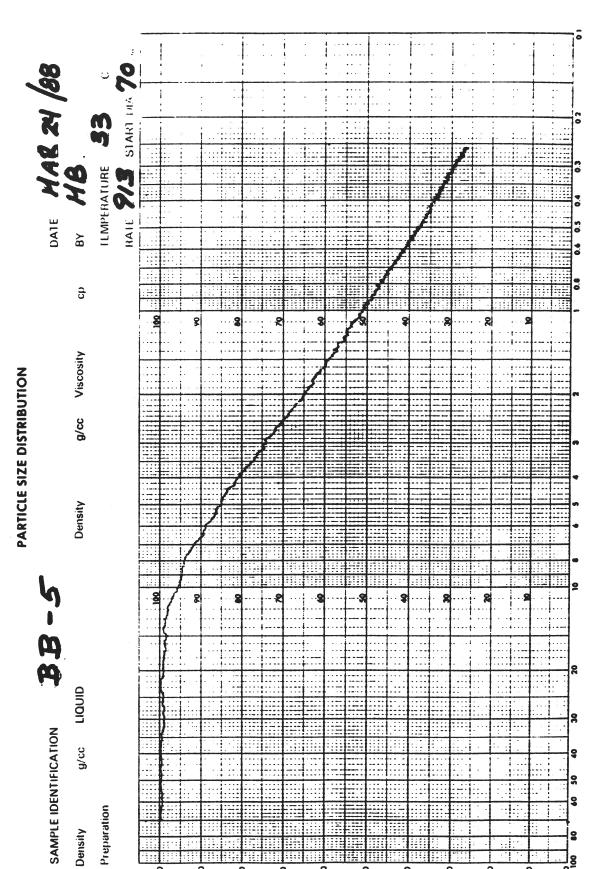
EQUIVALENT SPHERICAL DIAMETER, 11111

M micromeritics



EQUIVALENT SPHERICAL DIAMETER, 11711

micromeritics '



EQUIVALENT SPHERICAL DIAMETER, 11111

micromeritics '

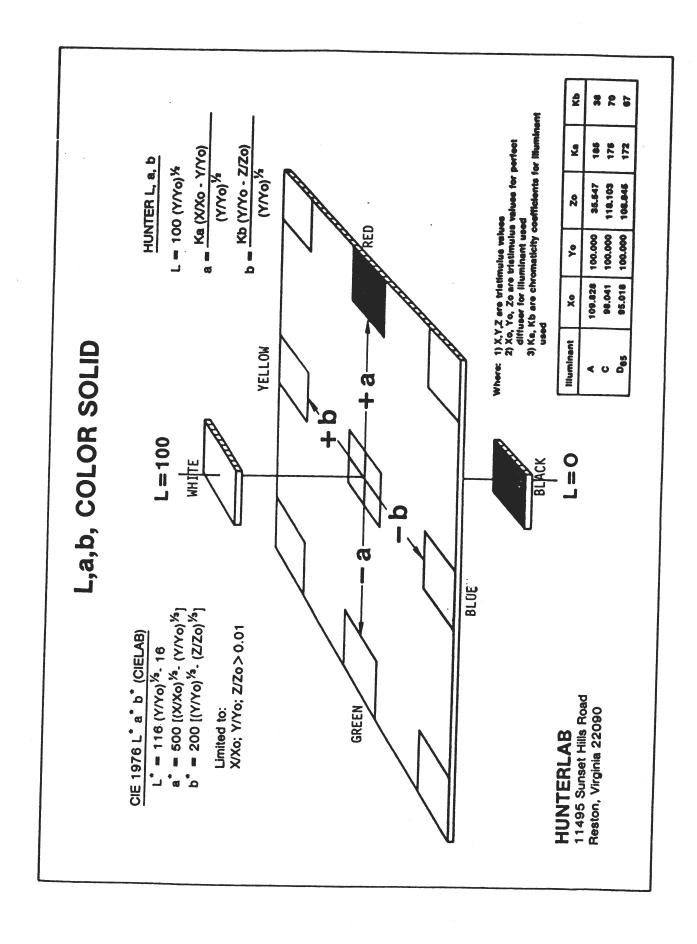
APPENDIX B2

Color and Brightness Determinations

CLAYS-COLOR AND BRIGHTNESS

1000	SAMPLE			00	COLOR			BRIGH	BRIGHTNESS
					ro	q		γ,	ĺ
		•	ST.D.	AV.	ST.D.	AV.	ST.D.	(ULE) AV. ST.	1E) ST.D.
C2-11680	WHITE STD.	92.35	±0.01	-1.12	+0.01	1.61	+0.01	85.25	+0.02
IT-88-30-IS-1	88-1	88.50	€0.03	-1.01	€0.00	5.07	00.0∓	78.32	+0.04
IT-88-30-IS-2	BB-2	90.52	+0.04	-0.94	±0.01	5.62	±0.01	81.95	+0.07
IT-88-30-IS-3	88-3	91.05	€0.03	-0.84	±0.01	6.00	±0.01	82.90	+0.05
IT-88-30-IS-4	88-4	85.13	±0.03	-1.09	±0.01	68.9	±0.01	72.48	+0.05
IT-88-30-IS-5	88-5	89.68	+0.07	-0.86	±0.01	6.35	±0.01	80.43	+0.01
REF-1 REF-2 REF-3	PK-PIONEER TK-TILE #6 CK-GROLLEG	92.31 92.76 91.68	±0.04 ±0.17 ±0.01	-0.24 -0.54 -0.44	±0.04 ±0.04 ±0.02	6.65 6.98 6.74	±0.15 ±0.08 ±0.01	85.21 86.04 84.05	±0.07 ±0.31 ±0.19

Industrial Services Laboratory Industrial Technologies Department Alberta Research Council



APPENDIX B3

Kaolin "Standards" - Suppliers Analyses

KAOLIN "STANDARDS" - SUPPLIERS ANALYSES

(wt.%)	Si0 ₂	(wt.%) Si0 ₂ A1 ₂ 0 ₃ Fe ₂ 0 ₃ Ti0 ₂ Ca0 Mg0 K ₂ 0 Na ₂ 0 L.O.I.	Fe ₂ 03	Ti02	Ca0	Mg0	K ₂ 0	Na ₂ 0	١.0.١.
PK- PIONEER (Georgia kaolin)	45.68	45.68 38.51	.44	.44 1.43 .24 .14 .14	. 24	. 14	. 14	.04	.04 13.51
TK- #6 TILE CLAY (Georgia kaolin)	46.9 38.2	38.2	.38	.35 1.42 .43 .58	.43	.58	1	.04	.04 13.9
CK-GROLLEG (English china clay) 47.7 37.2 .7 0.3 .10 .25 1.95 .10	47.7	37.2	.	0.3	. 10	. 25	1.95	.10	1