CANADA

DEPARTMENT OF MINES AND TECHNICAL SURVEYS

OTTAWA

MINES BRANCH INVESTIGATION REPORT IR 62-55

AN INVESTIGATION OF BENTONITES FROM SOUTHERN MANITOBA

by

J. S. ROSS & R. M. BUCHANAN

MINERAL PROCESSING DIVISION

COPY NO. 36

AUGUST 3, 1962

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J. S. Ross* and R. M. Buchanan**

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SUMMARY OF RESULTS

Seventy-one bentonite samples from Upper Cretaceous strata in southern Manitoba were submitted by the Mines Branch, Manitoba Department of Mines and Resources, for mineralogical examination and determination of possible industrial applications. These were combined to form nine composite samples identified by letters A to I, which were compared with commercial bentonites. Samples G, H, and I consisted of montmorillonite, whereas samples A to F contained, in addition to montmorillonite, smaller amounts of cristobalite and/or quartz. In some of clays "A" to "F", mica, kaolin or chlorite, feldspar (?) and cristobalite (?) were minor constituents.

Bentonites A to G require more intensive evaluation for iron foundary use but the economics of producing bentonite for this use only is questionable. Sample G was the best of the group for well drilling purposes but even when improved by the use of additives it did not have the quality of the better commercial bentonites. Bentonites H and I, which represent non-swelling bentonite from the producing operation of Pembina Mountain Clays Ltd., at Morden, Manitoba, had, when activated, petroleum oil bleaching qualities far superior to Filtrol 13, a standard activated bentonite. Except for finding new markets for the Morden product, current economics do not warrant further investigation of bentonite from these locations at this time.

Because of Canada's future requirements for high-quality swelling bentonite, further exploration and investigation is warranted for deposits of swelling bentonite in Manitoba.

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*Senior Scientific Officer, Non-Metallic Minerals Section and **Head, Ore Mineralogy Section, Mineral Processing Division, Mines Branch, Department of Mines and Technical Surveys, Ottawa, Canada.

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INTRODUCTION

A survey of bentonite deposits in Manitoba was conducted during the summer of 1959 by the Mines Branch, Manitoba Department of Mines and Resources. The Mines Branch, Department of Mines and Technical Surveys, was invited to participate in the project, to conduct mineralogical examinations on the samples collected and to test some of the properties that are important in the commercial evaluation of bentonite.

The work conducted in Ottawa was greatly delayed by unforeseen circumstances. However, an attempt was made to preserve the original condition of the samples in the sealed plastic bags in which they were shipped to Ottawa. Undoubtedly the water content changed during storage but the determination of mineral constituents and the testing of various physical and chemical properties were not significantly affected.

Several members of the Mines Branch provided data included in various sections of this report. Details of their contributions, which are gratefully acknowledged, may be found in their individual reports listed below:

X-ray diffraction analyses	(1)	R. S. Dean, Mineral Processing Division
Differential thermal analyses	(2)	J. G. Brady, Mineral Processing Division
Chemical analyses	(3, 4)	D. J. Charette, et al, Mineral Sciences Division
Foundry testing	(5)	A. E. Murton, Physical Metallurgy Division

DESCRIPTION OF SAMPLES

Two shipments containing a total of seventy-one specimens were received. Each specimen weighed from 3 to 10 pounds and, except for F and G, represented approximately one vertical foot of formation. They were combined to form nine test samples, at the suggestion of the Manitoba Mines Branch, as shown in Table 1. The four localities sampled were:

Locality 1.　　Twin Sisters Butte,
　　　　　　　S E Section 25, Township 4, Range 7 W.

Locality 2.　　Pit of Pembina Mountain Clays Limited,
　　　　　　　W 1/2 Section 25, Township 4, Range 7 W.

Locality 3.　　East of Brown, S E Section 18,
　　　　　　　Township 1, Range 5 W.

Locality 4. East bank of Pembina river 1/2 mile east of road
bridge north of town of Mowbray in S W Section 27,
Township 1, Range 8 W.

Table 1

Samples Submitted for Investigation

Test Sample	Field Sample Numbers	Locality	Thickness of Strata Sampled
A	1-10	1	9 ft 10 in. (9.8 ft)
B	11-20	1	9 ft 6 in. (9.5 ft)
C	21-30	1	11 ft 5 in. (11.4 ft)
D	31-40	1	12 ft 3 in. (12.2 ft)
E	41-51	1	13 ft 11 in. (13.9 ft)
			Total: 56.8 ft
F	52-57	2	9 ft
G	58-62	3	10 in. (0.8 ft)
H	63-68	2	?
I	69-71	4	?

GEOLOGICAL RELATIONSHIP OF SAMPLES

The following brief descriptions are quoted from information
supplied by Mr. Barry B. Bannatyne, Industrial Minerals Geologist, Mani-
toba Mines Branch (6).

Locality 2

"The main zone of non-swelling bentonite occurs at the base of the Pembina
member of the Cretaceous Vermilion River formation. Several thin beds

(8 to 12 inches thick) occur interbedded with black carbonaceous shales. The non-swelling bentonites are being strip-mined by Pembina Mountain Clays Limited 4 miles southwest of Miami, Manitoba, on W 1/2 Section 25, Township 4, Range 7 W. Samples #63 to #68, inclusive, are taken from the east-central part of these workings".

"Above the non-swelling bentonite and carbonaceous shale section, there is exposed at the Miami pit a chocolate-brown shale of which a thickness of 9 feet was sampled. Swelling tests of this material showed a thin suspension with some fine sediment settling out. These beds constitute the upper part of the Pembina member. Samples No. 52 to No. 57 are of this material".

Locality 1

"Overlying the Pembina member are the Millwood beds, composed mainly of highly bentonitic shales which exhibit swelling properties; they are part of the Riding Mountain formation. The rock is referred to here as 'semi-swelling bentonite'.

"A well exposed and easily reached section of the Millwood beds is at Twin Sisters Butte, S E Section 25, Township 4, Range 7 W, a few hundred yards southeast of the Pembina Mountain Clays Limited pit. The upper part of the 65-foot section exposed there may be part of the Odanah beds, also of the Riding Mountain formation. Samples No. 1 to No. 51 were taken at 1 to 1 1/2 foot intervals from top to bottom of this section. There is a gradational break in the section around sample No. 20 with the upper part lighter weathering and the lower part darker weathering with numerous ironstone concretions, some with a manganese stain. Near the top of the section is a seven-inch bed of a greenish waxy bentonite, which is included in sample No. 11."

Locality 3

"Another deposit of the greenish bentonite, which shows good swelling properties, occurs in S E Section 18, Township 1, Range 5 W, east of Brown. The stratigraphic sequence there has not been definitely determined, but the occurrence is on the hummocky edge of the escarpment, and an exposure of the Boyne member occurs 1/2 mile to the north at a lower elevation. Thus this bed, which is 10 inches thick, may occur within the Millwood or the lower part of the Odanah beds, similar to the occurrence at Twin Sisters Butte. Samples No. 58 to 62 are from the 10-inch bed east of Brown".

Locality 4

"The bentonite is the non-swelling type from the base of the Pembina member, similar to test sample H. Test sample I was taken from an

exposure in S W 27-1-8 W along the east bank of the Pembina River 1/2 mile east of the road bridge north of the town of Mowbray. The section is described by Wickenden (7)".

INVESTIGATION

The samples were prepared for examination by drying followed by coarse crushing. Each sample was then divided into two parts when riffled in a Jones sample splitter. One part was kept for reference and the other was combined to form nine samples as suggested by the Manitoba Mines Branch. These were then pulverized in a Micro-Pulverizer and thoroughly mixed. All subsequent test work was conducted on portions of the pulverized products.

X-Ray Diffraction Analysis

The nine samples of bentonite (A to I) were analyzed and compared with a commercial swelling bentonite (Volclay).

A portion of each sample was dispersed in distilled water. Small representative portions of the suspensions were placed on glass microscope slides and evaporated to dryness below the boiling point. No preliminary size fractionation or cation-exchange treatment was attempted. The sedimentation procedure was designed to give the maximum preferred orientation of the platy minerals parallel to the glass slide. A partial segregation may have taken place with the coarser particles concentrated near the surface of the glass. Only a very thin layer was deposited, however, and it was probably irradiated completely so that the diffracted X-ray beam represented the whole sample.

The minerals detected in each sample are listed in the approximate order of abundance in Table 2. Significant features in the X-ray diffractograms are illustrated by photographs of the strip charts. A quantitative analysis was not attempted because of the great variation in the diffraction effects of montmorillonite minerals.

Table 2

X-Ray Diffraction Analyses

Sample	Major Constituents	Intermediate Constituents	Minor Constituents
A	montmorillonite	cristobalite, quartz	–
B	montmorillonite	quartz, cristobalite	–
C	montmorillonite	quartz, cristobalite	–
D	montmorillonite	quartz, cristobalite	feldspar (?)
E	montmorillonite	quartz	cristobalite (?) mica mineral (?)
F	montmorillonite	quartz	kaolin or chlorite, mica mineral
G	montmorillonite	–	–
H	montmorillonite	–	–
I	montmorillonite	–	–
Volclay	montmorillonite	–	quartz, kaolin or chlorite, feldspar (?)

Montmorillonite was the major constituent in every sample and in three of
them, (G, H and I), it was the only mineral detected. With the exception of
the Volclay sample, and possibly sample G, which gave an integral series
of high-order reflections from a collapsed (9.6A°) structure, the air-dry
montmorillonites showed broad, non-integral basal reflections.

Saturation with ethylene glycol caused the (001) peaks of all samples to
be sharpened markedly and to be shifted into the 16.5-17.0 A° range. In
each case an integral, or approximately integral, series of basal reflections
was recorded after glycolation (Figure 6) indicating that uniform expansion
had occurred throughout the montmorillonite layers. Hence the montmorill-
onite is essentially free from collapsed (illitic) layers. The non-integral

basal reflections observed in air-dried samples are probably the result of random interstratification due to unequal hydration of the individual sheets.

The apparent first-order basal reflections of all air-dried samples, except Volclay, lie within the range 10.9-12.8A°. No control over the relative humidity of the atmosphere surrounding the sample during analysis was available and probably this caused some variation from one sample to another.

Montmorillonite in sample G showed a strong superstructure line (d=23A°) which is approximately twice the normal apparent basal spacing of 11.9A° (Figure 5).

Cristobalite - was identified in samples A, B, C, D, and suspected in E. The cristobalite diffraction pattern consisted of a fairly sharp peak at 4.07A° (Figures 1 and 2) and broad peaks at about 2.82 and 2.49A° (not shown in figures). This probably is indicative of very fine-grained and/or poorly crystallized material.

Quartz - gave sharp diffraction maxima at 4.26 and 3.34A° (Figures 1, 2, 3, 4).

Kaolin/Chlorite - Sample F and Volclay both showed weak reflections within the region 7.13-7.15A° which were unaffected by glycol treatment. In sample F a possible weak higher order reflection was observed at 3.56A° (Figure 3). This is indicative of the presence of minerals in kaolin and/or chlorite groups.

Mica Mineral - Weak reflections remained at 9.9A° in the diffractograms of sample F and, possibly E, after the first order basal montmorillonite peak had been shifted to a larger d-spacing by glycol treatment. This suggests the presence of a mica mineral.

Feldspar - Weak reflections at 3.18 and 3.21A° in sample D and Volclay, respectively were tentatively assigned to minerals of the feldspar group.
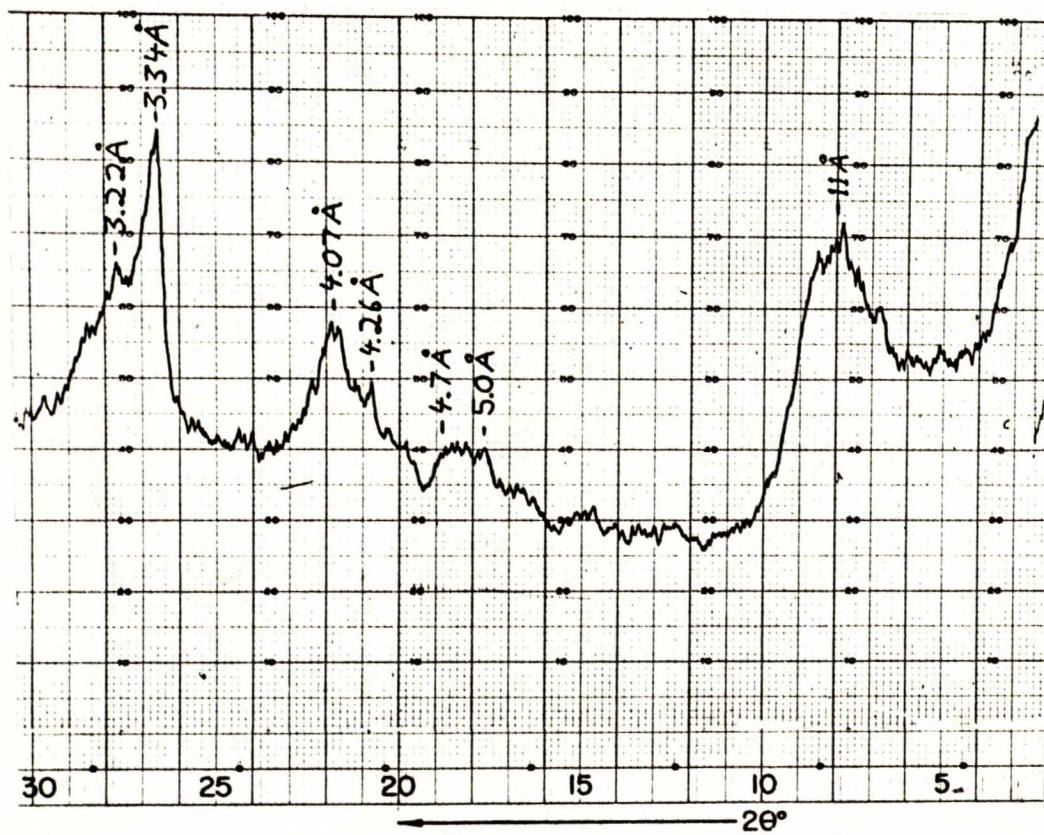
Figure 1. X-ray diffractogram of sample C, air-dried,
oriented specimen.

Montmorillonite – 11;5.0-4.7; 3.22A°
Quartz – 4.26; 3.34A°
Cristobalite – 4.07A°
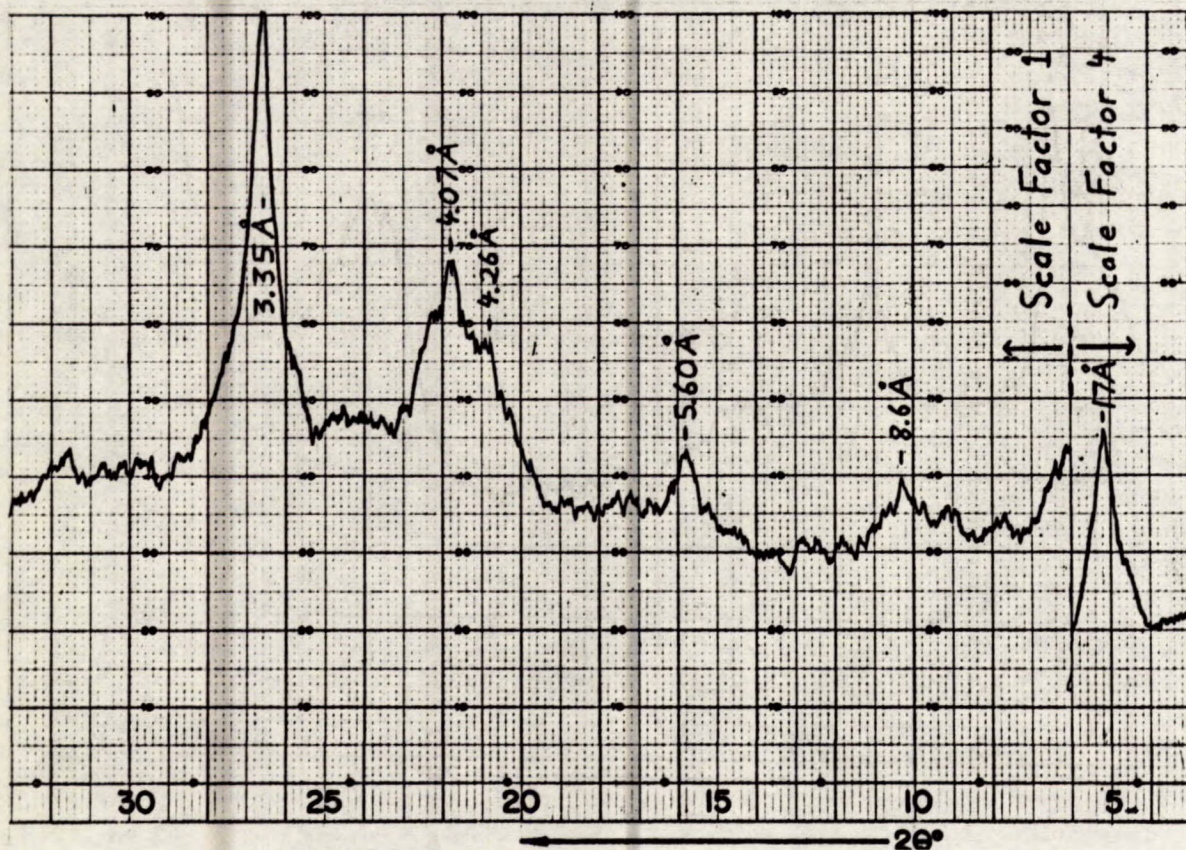
Figure 2. X-ray diffractogram of sample C, oriented
specimen, saturated with ethylene glycol.

Montmorillonite - 17;8. 6; 5. 60; 3. 35A°
Quartz - 4. 26; 3. 35A°
Cristobalite - 4. 07A°
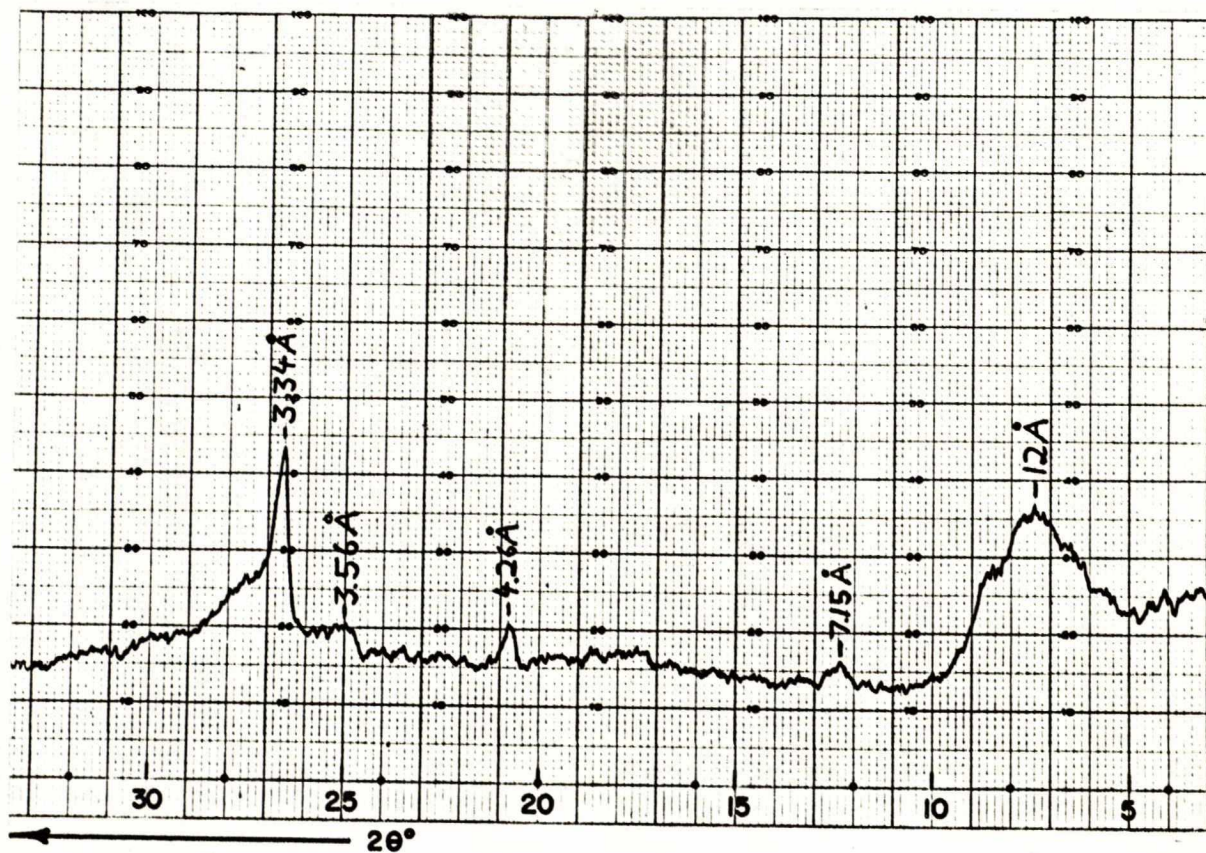
Figure 3. X-ray diffractogram of sample F, air-dried,
oriented specimen.

Montmorillonite - 12A°
Quartz - 4.26; 3.34A°
Kaolin/Chlorite - 7.15; 3.56A°
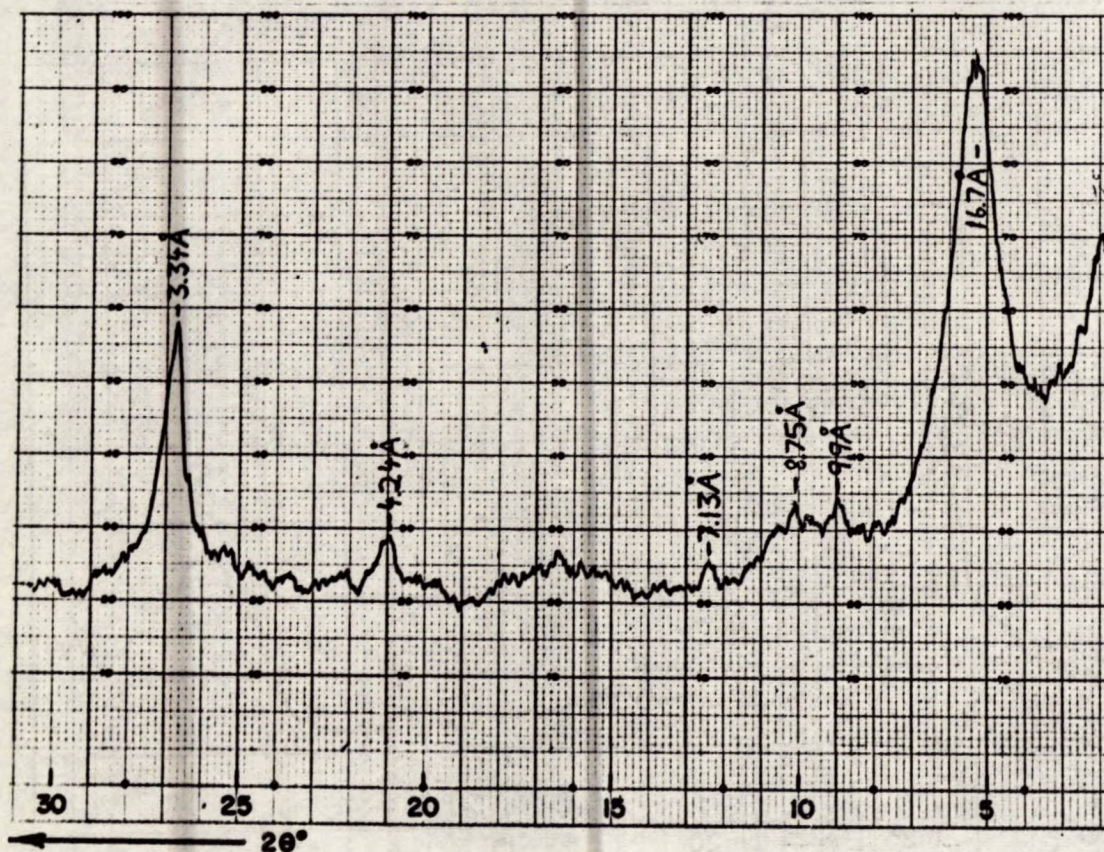
Figure 4. X-ray diffractogram of sample F, oriented
specimen, saturated with ethylene glycol.

Montmorillonite - 16. 7; 8. 75; 4. 24; 3. 34A°
Quartz - 4. 24; 3. 34A°
Kaolin/Chlorite - 7. 13A°
Mica Mineral - 9. 9A°
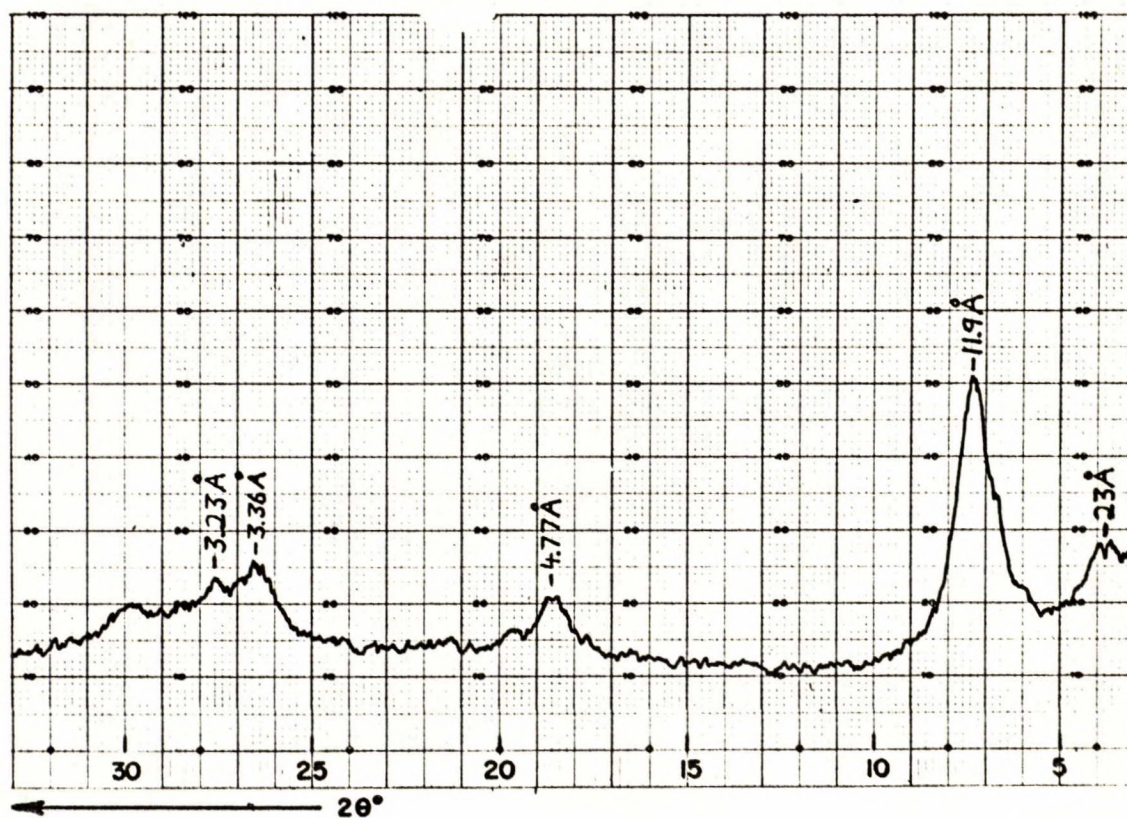
Figure 5. X-ray diffractogram of sample G, air-dried,
oriented specimen.

Montmorillonite - 23; 11.9; 4.77; 3.36; 3.23A°
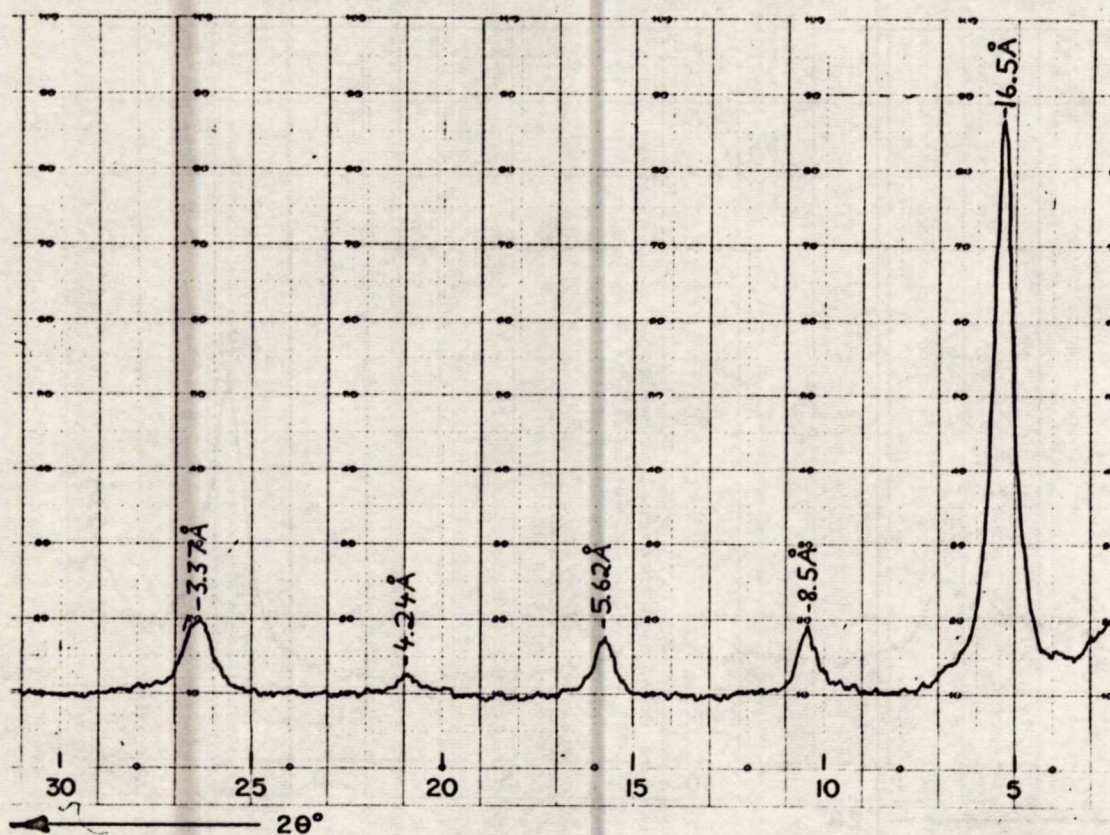
Figure 6. X-ray diffractogram of sample G, oriented
specimen, saturated with ethylene glycol.

Montmorillonite - 16.5; 8.5; 5.62; 4.24; 3.37A°

Microscopic Examination

Coarse fractions (+200-mesh) obtained during the determination of 'Grit Content' (see page 19) were examined under a stereomicroscope and representative grains of each constituent were identified by X-ray diffraction analysis.

The 'grit' fractions represented various proportions of the samples (Table 5); they were further divided by hand screening on small laboratory sieves to give the particle size analyses shown in Table 3.

Table 3

Screen Analysis of Grit

Sample	Size (mesh)	Weight (grams)	Proportion of +200 mesh grit (%)	Proportion of whole sample (%)
A	+28	4.981	34.9	5.0
	-28+48	3.559	25.0	3.6
	-48+100	2.622	18.4	2.6
	-100+200	3.088	21.7	3.1
		14.250	100.0	14.3
B	+28	1.073	15.1	1.1
	-28+48	0.419	5.9	0.4
	-48+100	1.276	18.0	1.3
	-100+200	4.328	61.0	4.4
		7.096	100.0	7.2
C	+28	0.470	15.9	0.5
	-28+48	0.417	14.1	0.4
	-48+100	0.619	21.0	0.6
	-100+200	1.449	49.0	1.5
		2.955	100.0	3.0
D	+28	1.263	20.0	1.3
	-28+48	0.513	8.1	0.5
	-48+100	1.208	19.1	1.2
	-100+200	3.345	52.8	3.3
		6.329	100.0	6.3

Table 3 (cont'd)

Sample	Size (mesh)	Weight (grams)	Proportion of +200 mesh grit (%)	Proportion of whole sample (%)
E	+28	0.626	8.4	0.6
	-28+48	0.273	3.7	0.3
	-48+100	0.913	12.3	0.9
	-100+200	5.600	75.6	5.4
		7.412	100.0	7.2
F	+28	0.477	45.8	0.5
	-28+48	0.120	11.5	0.1
	-48+100	0.146	14.0	0.1
	-100+200	0.299	28.7	0.3
		1.042	100.0	1.0
G	+28	1.756	33.7	2.8
	-28+48	0.760	14.5	1.2
	-48+100	0.903	17.3	1.5
	-100+200	1.798	34.5	2.9
		5.217	100.0	8.4
H	+28	4.363	18.1	4.9
	-28+48	8.304	34.4	9.2
	-48+100	6.827	28.3	7.6
	-100+200	4.623	19.2	5.1
		24.117	100.0	26.8
I	+28	0.056	0.9	0.1
	-28+48	0.072	1.2	0.1
	-48+100	0.415	7.0	0.4
	-100+200	5.417	90.9	4.7
		5.960	100.0	5.3

In samples A to E, inclusive, the most common constituents of the 'grit' material were white, non-calcareous, opaline microfossils that gave no distinct X-ray diffraction pattern. These features are found in the fossil Radiolaria. Only a small proportion of these were in the +100 mesh fractions although rare aggregates of several individual fossils loosely cemented with calcite, were observed. Soft, porous, black organic material was also common. Minerals identified, usually in fine-grained aggregates, were goethite, dolomite, siderite, calcite, quartz, mica and a poorly crystallized zeolite mineral, probably heulandite. Rare, soft white masses of bassanite

($CaSO_4 \cdot 1/2 \ H_2O$) probably were formed by the partial dehydration of gypsum during drying of the samples.

Sample F contained fewer microfossils but a higher proportion of them were in the -48+100M fraction. Other constituents were waxy green montmorillonite, which frequently cemented small grains of other materials: dolomite, goethite, quartz, mica, apatite and a zeolite material similar to that observed in samples A to E.

Microfossils were most common constituents in the -100 M fraction of G, which made up most of the grit of sample G. Mineral grains identified were dolomite, quartz, calcite and a zeolite similar to that observed in previous samples.

No microfossils were observed in the grit from sample H. All size fractions had a similar assemblage with predominant white aggregates of montmorillonite, smaller amounts of yellowish green aggregates of jarosite, rare mica flakes and some black, carbonaceous grains.

Montmorillonite was the predominant constituent in the grit from sample I which also contained quartz, feldspar and jarosite.

Differential Thermal Analysis

Equipment and procedures similar to those described by Brady (8) were used to carry out differential thermal analyses (DTA) on the nine Manitoba samples and on one sample of Volclay, a commercial swelling bentonite. Thermal effects were recorded at a sensitivity suitable for clay minerals. Small amounts of other minerals such as quartz and cristobalite could not be detected. DTA curves, reduced in size, are reproduced in Figures 7 and 8.

A sharp endothermic peak in the DTA curve shows that Volclay contains a typical sodium montmorillonite.

Another sharp endothermic-exothermic doublet at 860°C and 940°C suggests that Volclay is similar to sample H-26 in the report of American Petroleum Institute (API) Research Project 49 by Kerr, et al (9), which was described as sodium bentonite from Clay Spur, Wyoming.

DTA curves for samples G, H, and I are similar to that for Volclay. The large low-temperature endothermic peaks (150°-180°C) suggest that these samples contain more calcium in the exchange positions than Volclay. Endothermic-exothermic doublets in the 900°C region differ from the corresponding peaks in Volclay. This is to be expected because they are sensitive to changes in composition in both the octahedral and tetrahedral layers. The heating programme was not carried high enough to record the completion of the high-temperature exothermic reaction in samples H and I.

EXOTHERMIC

ENDOTHERMIC

A

B

C

D

E

100 200 300 400 500 600 700 800 900 1000

TEMPERATURE - °C
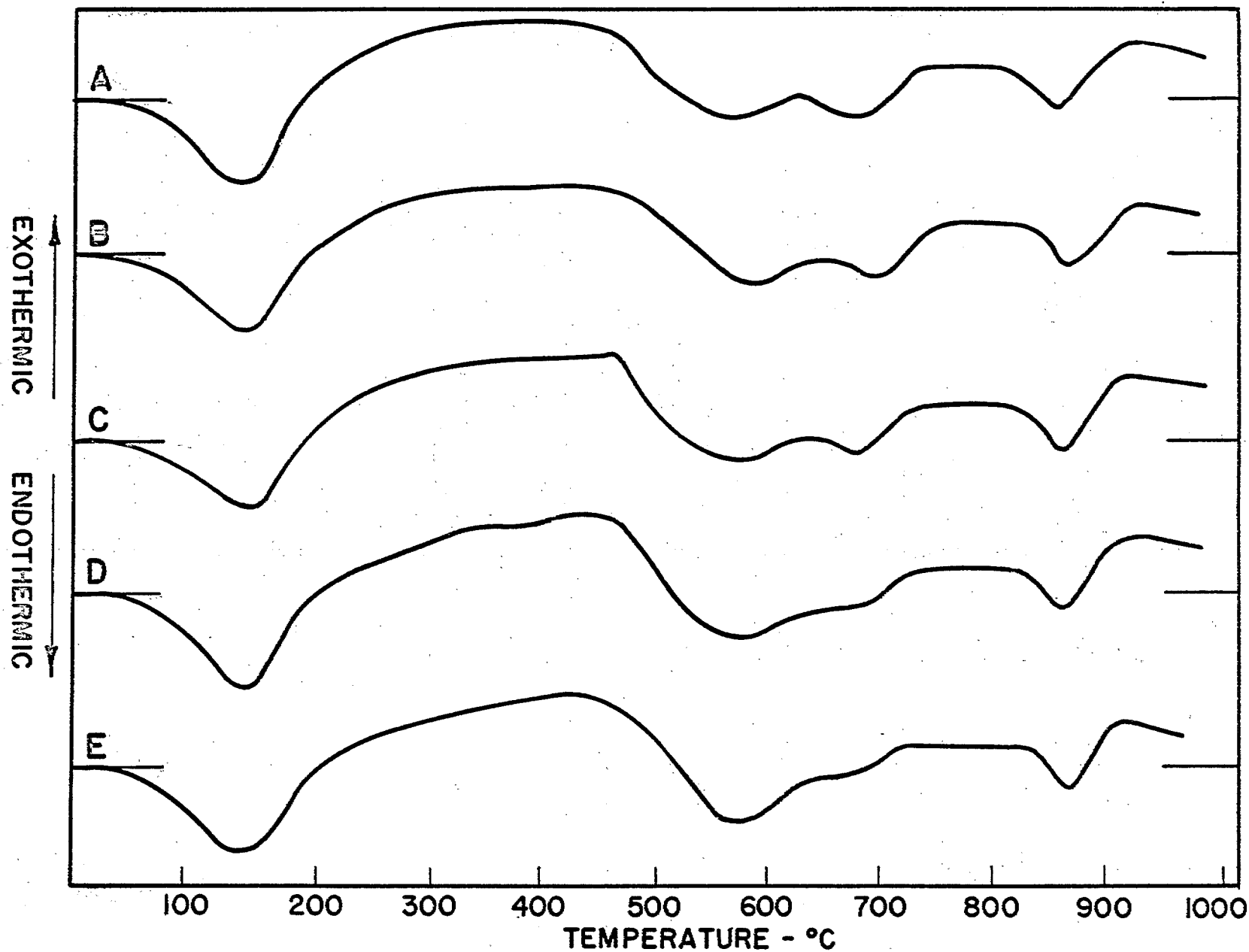
Figure 7 - DTA curves of bentonite samples A to E.

EXOTHERMIC

ENDOTHERMIC

F

G

H

I

VOLCLAY

TEMPERATURE - °C

100   200   300   400   500   600   700   800   900   1000
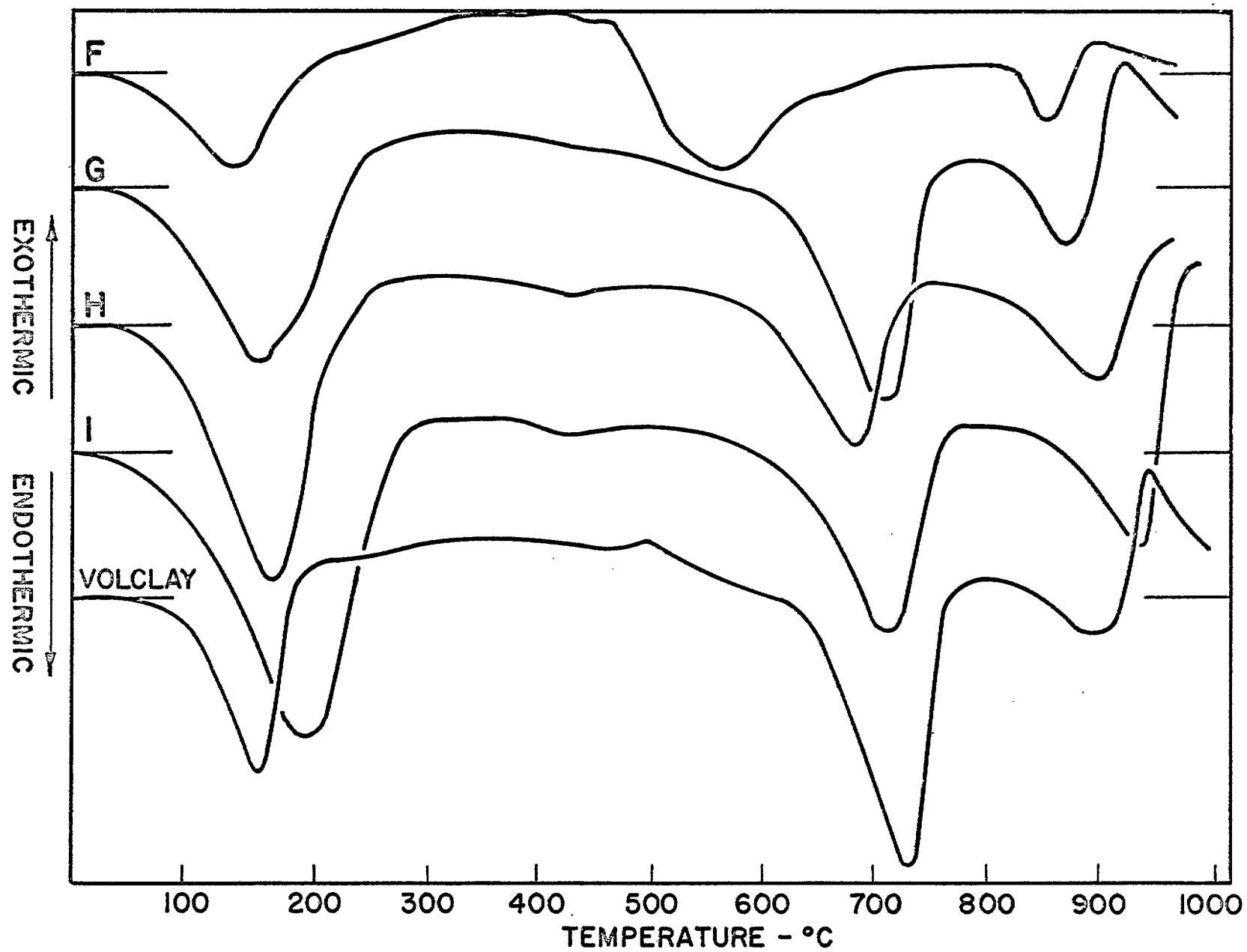
Figure 8 - DTA curves of bentonite samples F to I and Volclay

DTA curves for samples A, B, C, D, E and F differ markedly from those for G, H, I and Volclay. Less prominent endothermic peaks at 150-180°C show that they contain smaller proportions of montmorillonite. The double endothermic peaks in the 850 to 925°C range are typical of materials containing a small amount of montmorillonite, a small amount of illite, or a mixture of montmorillonite and illite. The principal difference between the two groups, however, is in the 500 to 700°C range where the peaks are somewhat similar to the curves for API samples H-31 and H-32a (Kerr, et al, (9)), which are described as mixed-layer montmorillonite-illite. Illite (or chlorite) is indicated by the first of the double endothermic peaks at about 570°C and the second is indicative of montmorillonite. On the basis of the general appearance of the curves, montmorillonite-illite is more probable than montmorillonite-chlorite. Samples E and F probably contain less clay material than A, B, C and D, which appear to be almost identical in this respect.

Moisture Content

Although it was apparent that more moisture had escaped from at least some of the samples, the moisture content was determined for all as received. One quarter of each sample was dried in the oven at 105 to 110°C for 48 hours and the weight loss recorded. These weight losses varied considerably and were grouped and averaged for the nine composites as shown in Table 1. Table 4 indicates the average moisture content of each group of samples.

Table 4

Moisture Content of Composite Samples

Composite Sample	Total Original Weight (grams)	Total Weight Dried at 105-110°C (grams)	% Moisture
A	5424	4547	16.2
B	2160	2030	6.0
C	1651	1552	6.0
D	1972	1859	5.7
E	2344	2182	6.9
F	1509	1405	6.9
G	1203	815	32.2
H	1426	1244	12.8
I	1253	815	35.0

The amount of free moisture in the original samples generally varied with the montmorillonite content.

Grit Content

The grit content, which is a measure of the non-clay fraction of a material, was determined as follows.

Dried bentonites resulting from the moisture determinations were recombined with the respective samples. The original samples were then dried, crushed in a jaw crusher to minus 3/16 inch, riffled into two parts, and one part combined into composite samples as indicated by Table 1.

Two-pound portions were riffled from each composite and 8-to 10-mesh fractions were screened from each. The remainder was returned to the respective samples. These fractions were then tested according to the American Petroleum Institute procedure (10).

They were dried overnight at 105 to 110°C and 100 grams of each were combined with 1,000 ml of distilled water and vigorously mixed for 10 minutes.

After aging for 2 hours, the mixtures were again stirred for 5 minutes and wet-screened with a 200-mesh sieve. The per cent oversize, or grit, is shown in Table 5.

Table 5

Grit Content of Crude Bentonites

Composite Sample	+200 M Grit %
A	14.3
B	7.2
C	3.0
D	6.3
E	7.2
F	1.0
G	8.4
H	26.8
I	5.3

With the exception of A and H, less than 9 per cent of each sample was over 200 mesh in size. Although the grit content is an inverse indication of the clay content, it has little bearing on bleaching quality. Descriptions of the grit constituents of each sample are included in the section 'Microscopic Examination'.

Milling

  The remainder of the minus 3/16 inch portion of the composite samples, was pulverized by three passes through a Micro-Pulverizer, to a particle size comparable with that of commercial bentonite. The well-known commercial bentonites used in this investigation are listed in Table 6. Except for the Filtrol variety, kindly donated by the producer, these products were purchased from retail outlets.

Table 6

Commercial Bentonites Used For Comparison

Trade Name	Producing Company	Origin	General Type	Main Use
Beaverbond	Magcobar Mining Company Limited	Alberta	Swelling	Foundry
Autobond	Magcobar Mining Company Limited	Alberta	Swelling	Foundry
SPV and Foundry Volclay	American Colloid Company	Black Hills region, USA	Swelling	Foundry
Filtrol, Grade 13	Filtrol Corporation	California	Non-Swelling	Bleaching
Panther Creek	American Colloid Company	Mississippi	Non-Swelling	Foundry

  Dry screen analyses of the pulverized Manitoba samples and of the commercial bentonites are given in Table 7. Each sample was oven-dried prior to screening for 30 minutes. Owing to the hygroscopic properties of bentonite, the results obtained with a 325-mesh screen were not reproducible and are therefore omitted from Table 7.

Table 7

Screen Analyses of Manitoba and Commercial Bentonites

Sample	+100M %	-100+150M %	-150+200M %	-200M %
A	1.0	2.6	3.0	93.4
B	0.5	2.8	7.5	89.2
C	0.3	1.0	1.8	96.9
D	0.3	1.1	3.7	94.9
E	0.2	1.1	3.6	95.1
F	0.1	0.1	1.0	98.8
G	0.2	2.0	8.3	89.5
H	0.1	1.1	5.0	93.8
I	0.1	1.0	5.4	93.5
Panther Creek	8.1	12.7	13.7	65.5
Autobond	0.5	1.7	5.8	92.0
Beaverbond	0.1	0.7	2.3	96.9
Filtrol 13	0.7	2.5	3.3	93.5
Volclay	3.4	3.4	8.8	84.4

To compare the properties and potential uses of bentonites, it is essential that the particle-size distribution above 200 mesh be similar for all samples. Table 5 indicates that the milling process produced particle sizes closely approximating those of four of the five commercial bentonites.

The method for determining grit content (p. 19) is standard for unground samples. However, to compare grit content with those of commercial bentonites, it was necessary to implement a modified procedure for pulverized material. As a result, the percentage of grit was determined for each commercial and each milled Manitoba sample using a 200-mesh screen. Results are given in Table 8.

Table 8

Grit Content of Commercial and Manitoba Bentonites

Sample	+200M Grit %	Total % over 200M
A	1.4	6.6
B	2.4	10.8
C	0.3	3.1
D	1.5	5.1
E	2.2	4.9
F	0.2	1.2
G	1.4	10.5
H	5.3	6.2
I	5.3	6.2
Panther Creek	27.1	34.5
Autobond	0.8	8.0
Beaverbond	0.1	3.1
Volclay	2.3	15.6

The milled samples A to G contain about the same proportion of plus 200-mesh clay as commercial swelling bentonites. Their grit content is slightly higher. Samples H and I have a much lower proportion of plus 200-mesh clay than the commercial non-swelling bentonite (Panther Creek) and a slightly higher grit content than Volclay.

Chemical Analysis

Samples were riffled from those listed in Table 7 and submitted for chemical analysis. All samples had been exposed previously to the atmosphere for at least three weeks. The results are given in Table 9 on a moisture-free basis.

Of the Manitoba samples, G, H, and I had a greater affinity for water over a three-week period. This is in accordance with the results of the mineralogical investigation which indicates that these contain higher proportions of montmorillonite. In general, the loss on ignition for samples G, H, I and the commercial bentonites was higher than for those with more impurities. Bentonites A to F contain appreciably more potassium than the others and, as expected, the only activated clay, Filtrol, contains much more sulphur than the others.

Table 9

Chemical Analyses (%, Moisture-free basis)

Sample	H₂O Below 110°C	H₂O 110 to 925°C	L.O.I. 110 to +925°C	SiO₂	Al₂O₃	Fe as Fe₂O₃	CaO	MgO	K₂O	Na₂O	TiO₂	MnO	Total S as SO₃	Total
A	3.80	4.89	5.49	66.84	16.10	4.13	0.94	2.82	1.83	1.00	0.44		0.18	99.77
B	3.85	4.57	5.60	66.66	14.88	4.27	1.14	2.75	2.08	1.21	0.48	0.54	0.15	99.76
C	4.08	4.37	5.57	68.08	13.32	4.13	1.24	2.89	2.11	1.26	0.50		0.48	99.58
D	3.88	4.51	6.12	66.20	14.02	4.49	1.22	3.02	2.28	1.36	0.51	0.86	0.60	100.09
E	3.57	6.37	5.61	69.92	11.52	4.42	1.38	2.84	2.45	1.49	0.53		0.56	100.16
F	3.60	4.33	5.26	68.08	12.96	5.13	0.88	2.57	3.35	1.18	0.65		0.31	100.06
G	6.16	6.00	6.71	62.88	18.67	4.56	1.48	3.74	0.51	0.94	0.22	0.13	0.10	99.84
H	10.75	6.91	8.09	63.72	19.86	1.42	0.16	4.67	0.26	0.77	0.52	0.12	0.62	99.59
I	11.76	7.00	7.86	59.64	21.82	2.59	1.52	3.92	0.25	0.14	0.50	0.02	1.52	99.78
Panther Creek	3.74	7.62	8.73	67.57	13.13	5.45	0.58	1.57	1.40	0.09	0.70		0.17	99.22
Autobond	3.25	5.00	7.49	67.10	14.21	4.54	2.37	1.48	0.52	1.58	0.31		0.10	99.50
Beaver-bond	5.86	4.26	6.32	69.90	14.17	4.09	2.02	1.40	0.53	1.48	0.29		0.17	100.20
Filtrol 13	12.87	9.55	11.55	66.05	13.06	1.52	2.88	3.79	0.20	0.10	0.23		3.54	99.38
Volclay	5.87	5.70	5.90	63.40	19.34	4.41	1.55	2.21	0.57	2.87	0.15		0.62	100.40

Of the Manitoba samples, G, H, and I, had a greater affinity for water over a three-week period. This is in accordance with the results of the mineralogical investigation.

Bulk Density

The bulk densities of the commercial and milled Manitoba bentonites, determined with a Scott volumeter, are listed in Table 10. All samples were dried for 16 hours prior to testing.

Table 10

Bulk Densities of Commercial, and Milled Manitoba Bentonites

Sample	Bulk Density (moisture-free basis) g/cm^3
A	0.40
B	0.46
C	0.39
D	0.40
E	0.41
F	0.51
G	0.70
H	0.59
I	0.71
Panther Creek	0.43
Autobond	0.62
Beaverbond	0.54
Filtrol 13	0.45
Volclay	0.50

There is no appreciable variation in bulk density among these samples. In general, those bentonites with a higher content of montmorillonite have a slightly higher bulk density.

Light Reflectivity

Using a Densichron light reflectivity meter with no filter, the light reflectivities of the Manitoba samples and some of the commercial clays were determined.

Table 11

Light Reflectivity

Sample	% Light Reflectivity
Standard-magnesium basic carbonate	100
A	52 1/2
B	52 1/2
C	51 1/2
D	51
E	51
F	51
G	60 1/2
H	84
I	75
Panther Creek	53 1/2
Autobond	58 1/2
Beaverbond	57 1/2
Volclay	69

Light reflectivity is one indication of the quality of a material for use as a filler. The reflectivities of samples H and I are comparable to those of other types of clays used in the manufacture of rubber and linoleum. They do not have the reflectivity required for most pharmaceuticals and paints.

Hydrogen-Ion Concentration

The pH, or hydrogen-ion concentration, indicates the presence of alkalies in a clay and considerably influences the properties of bentonite used in well drilling. It is normally desirable to maintain the pH of a drilling fluid above 7.0 and seldom is it allowed to go below 6.0. As shown in Table 12, all samples, with the exception of the non-swelling H, I, and Panther Creek bentonites, had pH values above 7.0.

These values were obtained by thoroughly mixing 7 grams of dried sample in 100 millilitres of distilled water and reading the scale of a Beckman glass-electrode pH meter every 30 seconds until a constant value was obtained.

Table 12

Hydrogen-Ion Concentration

Sample	Hydrogen-Ion Concentration
A	9. 1
B	9. 0
C	8. 1
D	7. 9
E	7. 7
F	8. 0
G	9. 0
H	4. 3
I	3. 1
Panther Creek	5. 4
Autobond	9. 5
Beaverbond	9. 1

Swelling Properties

An indication of the rate and extent of bentonite hydration can be determined by swelling tests. These properties have a direct bearing on a clay's suitability in well drilling in particular, and in any application where swelling bentonite is desired. Crude, pulverized, and chemically treated samples were investigated. The crude samples consisted of dried 8-to 10-mesh fractions riffled from those previously prepared for the grit tests. The chemically treated bentonites were prepared by the addition of sodium carbonate to milled products and subsequent thorough mixing.

In each case, the procedure was identical. Two grams of dried bentonite were added in 0.1-gram lots to a 100-millilitre glass graduate filled with distilled water. Each addition was made as soon as the previous one had settled to the bottom. The volume of gel was noted one hour after the last portion had been added.

Sample G has swelling properties equivalent to those of Autobond and Beaverbond but inferior to those of Volclay and of standard drilling-purpose bentonite. The addition of 8 per cent sodium carbonate notably increased the swelling of sample G to a volume less than that for natural Volclay. In summary, even with a sodium carbonate additive, the swelling properties of these Manitoba bentonites are well below those of foundry-purpose Volclay.

Table 13

Swelling Properties

Sample	Volume of Gel, (cc)				
	-8+10 mesh	Milled	Milled with 2% Na_2CO_3	Milled with 3% Na_2CO_3	Milled with 8% Na_2CO_3
A	8	9	8	7	9
B	11	11	11	11	11
C	13	11	12	11	10
D	12	10	11	10	10
E	12	10	11	11	11
F	12	10	10	10	10
G	12	15	16	18	21
H	5	3	5	6	6
I	7	6	7	6	7
Panther Creek		5			
Autobond		14			
Beaverbond		15			
Volclay		26			

Colloidal Content

The colloidal content is another means by which bentonite may be ev-aluated, particularly for use in well drilling.

Ten grams of dried sample were mixed for 20 minutes with 500 milli-litres of distilled water, poured into a graduate and allowed to settle for 24 hours. After decantation, the remaining residue was dried and weighed. The weight loss represents the colloidal fraction. The test results are pres-ented in Table 14.

Table 14

Colloidal Fraction

Sample	% Colloidal Fraction
A	41
B	48
C	44
D	34
E	34
F	38
G	52
H	9
I	4
Panther Creek	13
Autobond	56
Beaverbond	80
Volclay	77

Beaverbond and Volclay, with lower colloidal contents than some standard bentonites used in well drilling, have colloidal properties far sup-erior to those of the Manitoba samples.

Gelling Properties

Gelling characteristics are the most important in evaluating bentonite for use in well drilling. These are determined by evaluating the gel strength and the yield (the number of Imperial barrels of mud having and apparent viscosity of 15 centipoises than can be produced from one ton of clay). Stand-ard bentonites used for well drilling normally produce yields of 100 barrels or more. Minimum preferred initial and 10-minute gel strength are, respecti-vely, 2 and 10 grams. The procedure used was that recommended by the American Petroleum Institute (10).

Suspensions were prepared by agitation of known proportions of bentonite and distilled water with a high-speed mixer for 20 minutes. At least three samples with viscosities of about 8, 15 and 25 centipoises were prepared. After ageing 24 to 36 hours, they were stirred for an additional 5 minutes and their viscosities determined at 25°C with a Stormer viscometer. The initial and 10-minute gel strengths were determined also. The results, from more than 200 tests, are listed in Table 15. In addition, slurries with from 1/2 to 3 per cent sodium carbonate were tested.

The addition of sodium carbonate to sample B did not sufficiently increase the slurry viscosity. By graphical analysis, a content of 2.7 per cent sodium carbonate can produce the maximum viscosity that can be obtained from sample G under these conditions. The yield of sample G containing 2.7 per cent sodium carbonate was then determined with the Stormer viscometer. It amounted to 101 Imperial barrels per ton with an initial gel strength of 3 grams and a 10-minute gel strength of 14 grams.

In summary, none of the Manitoba bentonites in their natural state can produce yields competitive with those of commercial clays used in well drilling. Sample G, with a yield of 59, has the highest of the Manitoba group. However, with the addition of 2.7 per cent sodium carbonate, the yield of sample G was increased to the competitive value of 101 and it retained suitable gel strength. Various other additives would probably be as effective. However, this aspect was not pursued further because of the small amount of sample remaining, the dubious economics of the production of a chemically activated drilling clay in Manitoba, and the thin horizon from which the sample came.

Evaluation For Foundry Use

The chief use for bentonite in Canada is in foundries. The technical requirements are more stringent for use in steel than in iron foundries. Those for steel as specified by the Steel Founders' Society of America Tentative Specification for Western Bentonite, SFSA Designation: 13T-61 (11), are as follows:

Property	Minimum	Maximum
Moisture	6%	12%
pH	8.2	
CaO		0.70%
Liquid limit	600	850

The moisture content of the Manitoba milled samples (Table 4, p. 18) was less than the maximum required, and for samples A to F could be increased easily to within the desired limits. Although the pH values (Table 10) for samples C, D, E and F were slightly below the minimum required, they were not increased with the use of sodium carbonate because of the limited amount

Table 15

Yield and Gel Strength

Sample	Initial Gel Strength (grams)	10-Minute Gel Strength (grams)	Yield (Imp. bbl per ton)	Viscosity* (centipoises)					
				plus 1/2% Na_2CO_3	plus 1% Na_2CO_3	plus 1.1/2% Na_2CO_3	plus 2% Na_2CO_3	plus 2.1/2% Na_2CO_3	plus 3% Na_2CO_3
A	12	20	34						
B	5	14	35	21	26	26	26	26	26
C	13	13	29						
D	13	20	16						
E	7	7	21						
F	14	19	26						
G	4	16	59	24	42	66	75	86	83
H	9	10	18						
I	9	10	21						
Panther Creek	9	12	18						
Autobond	3	3	60						
Beaverbond	3	2	78						
Volclay	2	4	119	11	12	12	12	12	12

*Using the weight of bentonite required to produce both a 15-centipoise slurry and the yield indicated in the fourth column.

of the sample on hand. With the exception of sample H, all samples contained more calcium oxide than allowable (Table 9).

Four of the commercial bentonites investigated are used in foundries. Volclay and Beaverbond are used in Canadian steel foundries and Autobond and Panther Creek are used in iron foundries. From Table 9 it can be seen that their moisture content requires adjustment to within the limits of the specifications for steel-foundry use and that, except for the Panther Creek clay, their calcium oxide content is higher than that of the Manitoba clays and the specifications of the Steel Founders' Society.

Laboratory tests were undertaken by A. E. Murton, Ferrous Metals Section, Physical Metallurgy Division, Mines Branch, to compare the moulding properties and liquid limits of the commercial and Manitoba bentonites. Tables 16 and 17 show his results. Sample G-1 consisted of clay from sample G intimately mixed with 2. 7 per cent sodium carbonate.

Table 16

Liquid Limit Tests

Sample	Liquid Limit (per Steel Founders' Society of America Tentative Standard 13T-61)
A	173
B	196
C	184
D	172
E	170
F	154
G	367
G-1	578
H	137
I	133
Volclay	651
Panther Creek	162
Beaverbond	632
Autobond	337

The moulding properties (Table 17) were tested using 3,500 grams of Ottawa "45" sand and 225 grams of sample. The mixture was mulled for 1 minute, brought to the desired consistency by the addition of water, and wet-mulled for 6 minutes.

Table 17

Moulding Properties

Sample	Moisture Added %	Green Compressive Strength psi	Green Deformation %	Green Shear Strength psi	Green Tensile Strength $oz/in.^2$	Dry Compressive Strength psi	Jolts to Break Overhang Specimen
A	3.5	7.9	2.50	2.25	16.0	112	12
B	3.6	7.3	2.75	2.23	18.6	133	13
C	3.5	7.7	2.55	2.58	22.0	103	15
D	3.5	7.9	2.55	2.53	19.4	93	13
E	3.5	8.2	2.43	2.23	19.4	93	12
F	3.5	6.4	2.59	1.88	18.8	105	10
G	3.5	10.5	2.41	2.90	25.4	115	20
G-1	3.7	11.1	2.54	3.20	32.4	139	16
H	4.2	14.1	2.39	3.75	34.6	51	25
I	4.0	13.0	2.26	3.75	34.8	84	28
Volclay	3.5	8.6	2.54	2.50	24.4	163	22
Panther Creek	4.0	8.6	2.06	2.30	2.0	96	14
Beaverbond	4.0	10.2	2.8	3.10	26.0	104	25
Autobond	3.6	11.4	2.53	3.05	27.8	95	23

Mr. Murton indicated that increased moisture results in decreased green strength and increased green deformation. His conclusions regarding the samples tested are as follows:

"1.   Samples H and I appear to be too low in dry strength for foundry use.

2.   The properties of the other clays indicate that they are too low in dry strength to be acceptable for steel.

3.   The more promising samples (probably all except H and I, but depending also on the differential thermal analyses), should be further tested by using the sand mixtures for casting metal. "

Pelletizing

The effectiveness of bentonite for pelletizing mineral concentrates is very difficult to assess by general investigation of a few small samples. Each mineral concentrate has different characteristics and the resulting pellets are subjected to dissimilar temperature and mechanical treatment. In addition, test equipment and procedures, as well as specifications, vary among most companies.

Because of these numerous variables, it is preferable to submit milled bentonite to a potential consumer for assessment. In any event, the samples submitted were not large enough for pelletizing tests.

In the pelletizing of iron ore concentrates and their subsequent treatment, there are some conditions which are about similar to those in foundry moulding. Thus, the test results in Tables 16 and 17 suggest the effectiveness of these bentonites in iron ore pelletizing. It should be understood that these suggestions are not conclusive and are the results of tests on minus 45-mesh silica. Of the Manitoba samples, H and I formed the strongest green cubes but when dried, these cubes had the least compressive strength but also the least tendency to shatter. All Manitoba samples, when bonded with silica sand, gave dry compressive strengths well below those for Volclay indicating that dried pellets containing these Manitoba clays may not be as resistant to compression as pellets containing Volclay.

Therefore, except for in the green state, there is no suggestion from the limited tests performed, that these Manitoba bentonites, when in an unpeptized and milled state, would be competitive in quality for use in pelletizing iron ore concentrates.

Bleaching Properties

Naturally and artificially activated non-swelling bentonites are used for decolorizing mineral, animal, and vegetable oils and other food products. Normally, the artificially activated clays have superior adsorptive properties. The Filtrol brand of activated bentonite is in standard use in the United States and Filtrol, Grade 13, was used as a standard in comparing the bleaching properties of samples B, D, E, G, H, and I when activated.

In activation, the effects of various quantities of different chemicals can be investigated over various periods of time. However, it was decided that sulphuric acid would probably be the most economical activating agent for use in Manitoba and that, initially, a 10 per cent solution of sulphuric acid would be used. The recognized procedure of the United States Bureau of Mines (12) was followed in activating these clays. One hundred grams of clay, 60 millilitres of concentrated sulphuric acid and 540 millilitres of water were placed in a 1,000-millilitre beaker and boiled and stirred for 1 hour. After settling, the clear liquid was decanted. Six hundred millilitres of distilled water were then added and the mixture stirred thoroughly and allowed to settle. This decanting and washing process was repeated five times and then the residue was filtered and finally washed five times with jets of distilled water. The residue was dried for 48 hours at 110°C and ground with pestle and mortar to pass a 150-mesh screen. The process was repeated using additional bentonite and an activation period of 4 hours.

The weight loss during activation was determined and the products were analysed for iron and loss on ignition (Table 18).

Bleaching reduced the iron content of these bentonites appreciably. Bleaching for 4 hours removed more iron than for a 1-hour activation period except with samples H and I with which a 1-hour period gave maximum iron removal. Acidulation notably increased the light reflectivity of samples G and I to above 80 but did not affect sample H which initially was above 80. In most cases, the weight loss was greater during a 4-hour activation period. For both periods it was appreciably greater for samples H and I.

For the bleaching tests, 12 grams of dried activated sample and 300 grams of Imperial Oil Limited unbleached MCT 30 stock motor lubricating oil were placed in a 1,000-millilitre stainless steel beaker. A metal lid was placed on the beaker and the ingredients were rapidly agitated while kept in an atmosphere of carbon dioxide and heated. Heating was regulated to raise the oil temperature to 230°C in 15 minutes. The mixture was then rapidly cooled to 93°C and filtered at 15 inches of mercury. The filtration time was noted and the transmittance of the filtrate was measured with a Bausch and Lomb "Spectronic 20" colorimeter set at a diffraction grating of 5,100 Angstrom units. The colorimeter was calibrated with distilled water. Results are listed in Table 19.

Table 18

Chemical Analyses, Reflectivity,
And Weight Loss of Activated Bentonites

Sample	Total Fe as Fe_2O_3 Dry-basis %	Loss of Total Fe Dry-basis %	Loss on Ignition Dry-basis %	Reflectivity %	Weight Loss, %
B original	4.27		5.60	52 1/2	
D "	4.49		6.12	51	
E "	4.42		5.61	51	
G "	4.56		6.71	60 1/2	
H "	1.42		8.09	84	
I "	2.59		7.86	75	
Activated Filtrol (as received)	1.52				
B activated 1 hr	2.81	1.46	5.36		13.4
D " "	2.90	1.59	5.96		13.0
E " "	3.18	1.24	6.03		12.8
G " "	3.77	.79	7.06	81	17.7
H " "	0.96	.46	8.12	83 1/2	21.9
I " "	1.46	1.13	8.27	82 1/2	24.8
B activated 4 hr	1.96	2.31	4.48		17.0
D " "	2.04	2.45	4.93		20.3
E " "	2.14	2.28	5.02		18.6
G " "	3.56	1.00	7.24	81 1/2	15.9
H " "	0.98	.44	6.87		24.7
I " "	1.64	.95	7.93		23.7

Table 19

Oil-Filtrate Transmittance and Filtering Time

Sample	Filtering Time min.	Transmittance %
Undecolorized MCT 30 stock		30
B-1 hr		53
B-4 hr	7.9	41
D-1 hr		40
D-4 hr	6.3	54
E-1 hr		36
E-4 hr	11.9	51
G-1 hr		57
G-4 hr	3.7	54
H-1 hr	2.0	92
H-4 hr	1.8	95
I -1 hr	2.1	96
I -4 hr	1.9	100
Filtrol, Grade 13	3.5	80

It was initially intended to investigate the effects of activation periods of 1/2, 2, and 3 hours and of increased concentrations of sulphuric acid. However, this initial test series produced, with samples H and I, results that were exceptionally desirable and that more closely approached the ultimate under the conditions of this experimental procedure than did the commercial bentonite. Because highly favourable results were obtained with H and I using a relatively low acid concentration and activation period, it was not necessary to determine the optimum bleaching conditions at this stage. This could be done after detailed exploration and sampling.

As compared with the 1-hour period, acid activation for a 4-hour interval increased light transmittance by the oil with four of the six samples and increased it only slightly with samples H and I. Samples H and I produced filtering times and light transmittance far superior to those of commercial Filtrol, Grade 13. The results in Table 19 for the Filtrol, H and I samples are averages of several tests.

SUMMARY AND CONCLUSIONS

Seventy-one samples submitted were combined into nine composites lettered A to I.

Montmorillonite was the major constituent of every sample and the only detectable one in G, H and I. In samples A to F, quartz and/or cristobalite were intermediate constituents and in some, mica, kaolin or chlorite, feldspar (?), and cristobalite (?) were the minor constituents. Grit in samples A, B, C, D, E, F and G, was composed mainly of siliceous microfossils whereas that in H and I was mainly montmorillonite.

Differential thermal analysis supported X-ray diffraction results and indicated that bentonites G, H, and I were comprised of montmorillonite.

Concerning possible industrial applications for this commodity, it can be seen from Table 20 that the domestic consumption of bentonite falls into three main groups (well drilling, foundry use, and pelletizing) in addition to smaller-consuming groups which use it mainly for bleaching and as a filler.

The Manitoba samples, pulverized to a grain size comparable to that of the commercial bentonites, had a grit content slightly higher than most of the commercial clays. Generally samples A to F contained less montmorillonite and had less affinity for moisture; higher potassium content and gel strength; and lower bulk density, light reflectivity, swelling effect, colloidal content, and yield than sample G and their commercial counterparts. They had better gel strengths but were not suitable for steel-foundry use nor is there any indication from the limited test work that they would be competitive in quality for pelletizing iron ore concentrates.

Table 20

Canadian Consumption of Bentonite, 1960

Use	Short tons
Well drilling	39,144
Iron and steel foundries	13,283
Pelletizing	8,500*
Petroleum refining	1,871
Paper	277
Miscellaneous chemicals	568
Miscellaneous non-metallic products	1,228
	64,871

* Estimated.

Sample G, which contained more montmorillonite than clays A to F, had better swelling, colloidal, yield and foundry-use characteristics than samples A to F, but was inferior to commercial swelling bentonites. With additives, these properties can be improved but the clay would still not be competitive in quality with typical Wyoming bentonite for steel foundry, drilling-mud, and probably pelletizing uses. With additives, it would compete in quality with lower grades of drilling-purpose bentonite. Further testing of samples A to G would be required before it is known whether they are suitable for use in iron foundries.

The non-swelling bentonites, H and I, have light reflectivities which are comparable to those of other types of clays used as fillers. However, the current Canadian market for non-swelling bentonite as a filler is small. Samples H and I, when activated with 10 per cent sulphuric acid for 1 hour, produced, in a short filtering interval, petroleum oil having superior light transmittance to that derived with a standard commercial bleaching clay. Annually, about 1,900 tons of activated bentonite are consumed in Canada for bleaching petroleum oils. In addition, there is a foreign market for about 5,000 tons of such high-quality products.

In conclusion, Manitoba samples A to G are not suitable for steel-foundry use and require further testing before their use in iron foundries can be determined. Without being able to supply bentonite to steel foundries, a company would find it difficult to market a clay for application only in iron foundries. Chemicals can be added to sample G to improve some of the properties of importance in well drilling, but the bentonite would still not be competitive in quality for this use. Samples H and I could be used as low-grade fillers but the market for this type is small. Above all, this investigation has substantiated the fact that samples H and I, which represent bentonite from the pit being operated by Pembina Mountain Clays Ltd. at Morden, Manitoba can be activated to have petroleum-oil-bleaching qualities far superior to (activated) Filtrol 13. Because of current marketing problems, the relative abundance of high-quality non-swelling bentonite at Morden, and of high-quality swelling bentonite in north-central United States, further testing of bentonite from these particular locations, is not warranted at this time.

There would be, however, a large demand for high-quality swelling bentonite from Manitoba. As a result, and except for finding new markets for the Morden production, development of the Manitoba bentonite industry should be towards the exploration for, and investigation of, high-quality swelling bentonite deposits.

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