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MINERALOGICAL EXAMINATION OF SIX SOIL FRACTIONS
SUBMITTED BY ATOMIC ENERGY OF CANADA, LIMITED,
CHALK RIVER, ONTARIO

by

R. M. Buchanan

Industrial Minerals Division

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SUMMARY OF RESULTS

Six fractions of a soil from the vicinity of Chalk River, Ontario, were submitted by the Environmental Research Branch, Atomic Energy of Canada, Limited for mineralogical investigation. The presence of attapulgite had been postulated as a possible reason for certain anomalous chemical properties of the soil and it was requested that the samples be examined by electron microscopy, differential thermal analysis and X-ray diffraction to determine if attapulgite is present. Dr. F. W. Boswell, Physical Metallurgy Division, carried out the electron microscopy and reported the results directly. This report contains the results of differential thermal analyses, X-ray diffraction analyses and a microscopic examination. Attapulgite was not detected in any of the six samples. The constituents which were identified are: amorphous soil colloids, quartz, feldspar, chlorite, mica, tourmaline and calcite. In one fraction there is a high proportion of calcite which is considered to be anomalous. Photomicrographs and differential thermograms are used to illustrate most of the observed features.

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INTRODUCTION

For about two and one-half years a soil from the vicinity of Chalk River has been under investigation by Dr. E. J. Evans, Environmental Research Branch, Atomic Energy of Canada, Limited. During the investigation it was observed that the amount of H^+ released to neutral ammonium acetate, after HCl treatment, exceeded the cation exchange capacity by a considerable amount. To explain this property, the presence of attapulgite has been postulated, the inference being that the excess H^+ was released from the OH_2 groups of Bradley's structural scheme of attapulgite (1)*. It was requested that an examination be made of several samples by means of electron microscopy, differential thermal analysis (DTA) and X-ray diffraction in order to determine if attapulgite is present in the soil.

Samples of six fractions of the soil were submitted to the Mines Branch through Mr. V. A. Haw, Acting Chief, Industrial Minerals Division, who forwarded them to Dr. F. W. Boswell, Head, Metal Physics Section, Physical Metallurgy Division, for electron microscopy. The results of his examination were forwarded by Boswell to Evans in a letter dated November 20, 1958, along with contact prints of typical electron micrographs, and the samples were returned to this Division. Later, chemical analyses of the six samples were submitted by Dr. Evans to assist the mineralogical investigations.

* Numbers in brackets are references listed at end of report.

This report contains the results of the differential thermal analyses, which were performed and interpreted by Mr. J. G. Brady, Ceramic Section, along with the results of the X-ray diffraction analyses and the microscopic examination.

DESCRIPTION OF SAMPLES

The samples were received in the form of aqueous dispersions contained in small plastic bottles and labelled as follows: Coarse Silt (CS); Medium Silt (MS); Fine Silt (FS); Coarse Clay (CC); Medium Clay (MC); and Fine Clay (FC). Only a small quantity of solid material was contained in the MC fraction but it was augmented by a later shipment, which was added to the original portion, to make a sample large enough for DTA and X-ray diffraction analysis.

Four of the samples, CC, FS, MS and CS, were found to be composed, when dried, of similar, yellowish-brown, powdery materials. The MC fraction is a darker, greyish-brown substance and the FC fraction is a lighter, brownish-yellow colour.

Chemical Analyses (Submitted by Dr. E. J. Evans)

	<u>SiO₂</u>	<u>Al₂O₃</u>	<u>Fe₂O₃</u>	<u>TiO₂</u>	<u>CaO</u>	<u>MgO</u>	<u>Na₂O</u>	<u>K₂O</u>	<u>L. O. I.</u>	<u>Total</u>
SC	61.0%	11.5%	6.0%	0.9%	3.6%	3.2%	3.9%	2.2%	3.5%	95.8%
MS	56.5	11.3	6.9	1.0	2.2	3.0	3.0	2.0	14.1	100.0
FS	42.4	13.6	7.8	1.1	0.7	2.5	1.9	1.7	25.3	97.0
CC	28.7	16.4	9.2	1.0	3.6	1.4	0.8	0.9	30.0	92.0
MC	17.1	16.0	9.4	0.8	10.7	2.2	0.5	0.4	36.0	93.1
FC	3.1	10.3	0.2	0.2	42.0	2.6	nil	0.1	44.3	102.8

PROCEDURE

Solid material was recovered from the dispersions by evaporating the water at about 90°C. It was felt that this procedure would give the maximum recovery of soil and not bring about any appreciable decomposition of any of the possible constituents.

A preliminary X-ray examination was made of each of the soil fractions in small (57.3 mm diameter) Debye-Scherrer cameras. This served to identify the major non-clay constituents in the coarser fractions and to show that several of the fractions are essentially non-crystalline. Packed powder mounts and oriented aggregates for clay mineral identification were prepared for examination in a Norelco X-Ray Diffractometer. Specimens were heated to various temperatures and treated by vapour-pressure glycolation, where necessary, to establish the identity of the 14A° material. The X-ray results were compared with those produced by a sample of standard attapulgite, which had been purchased from Wards Natural Science Establishment Inc. and was described as being from the same locality as the attapulgite described in the report of the A. P. I. Project 49 (2).

A microscopic examination was made of each of the fractions in order to determine the nature of the non-crystalline material. The samples were mounted in appropriate index liquids and examined under the highest magnification available (about 600X).

RESULTS

X-Ray Diffraction Analysis

1) Standard Attapulgite: The sample of standard attapulgite from Attapulgis, Georgia, produced a pattern with a prominent (110) diffraction peak

at $10.5A^\circ$, but the secondary peak, representing the (400) spacing of $3.23A^\circ$, was diffuse and much weaker than would be expected from the published data of Bradley (1). However, the pattern is in agreement with the data published by Kerr et al (3), which represents material from the same locality. It was evident that only the $10.5A^\circ$ peak would be of any use in identifying attapulgite in mixtures. There did not appear to be any doubt that the attapulgite peak could be distinguished from the $10.0A^\circ$ illite (mica) peak, with our experimental arrangement, unless one constituent was greatly in excess of the other.

2) Coarse Silt (CS): The diffractometer patterns and films show that the dominant constituents, quartz and potash feldspar, are accompanied by some chlorite and, possibly, a small amount of mica.

3) Medium Silt (MS): The proportions of quartz and feldspar in this fraction appear to be smaller, and those of chlorite and mica larger, than in the CS fraction.

4) Fine Silt (FS): This fraction contains much less crystalline material than the previous two, as shown by a weakening of all the diffraction lines. Quartz and feldspar can still be distinguished and the chlorite pattern is distinct. There is a faint suggestion of a peak at the $10.6A^\circ$ position but it is not strong enough to establish, definitely, the presence of attapulgite.

5) Coarse Clay (CC): The quartz and feldspar patterns are very weak and even the diffractometer record of the oriented aggregate shows only a weak, diffuse indication of the $14A^\circ$ chlorite peak. No $10A^\circ$ material was detected.

6) Medium Clay (MC): This fraction must consist predominantly of

non-crystalline material because the only diffraction pattern observable is a small quartz peak at the 3.34\AA position.

7) Fine Clay (FC): This material produced a clear, strong diffraction pattern of calcite. There appears to be little or no dilution by amorphous material.

Microscopic Examination

1) Fine Clay (FC): This sample is a pure, well-crystallized calcite with no observable contaminants. The calcite crystals appear as single grains up to 15 microns in size and as loose aggregates of smaller grains, as shown in Figure 1. They are unusual in that they are slightly elongated in outline with an angular or subangular shape and, in some cases, a noticeable constriction at the middle.

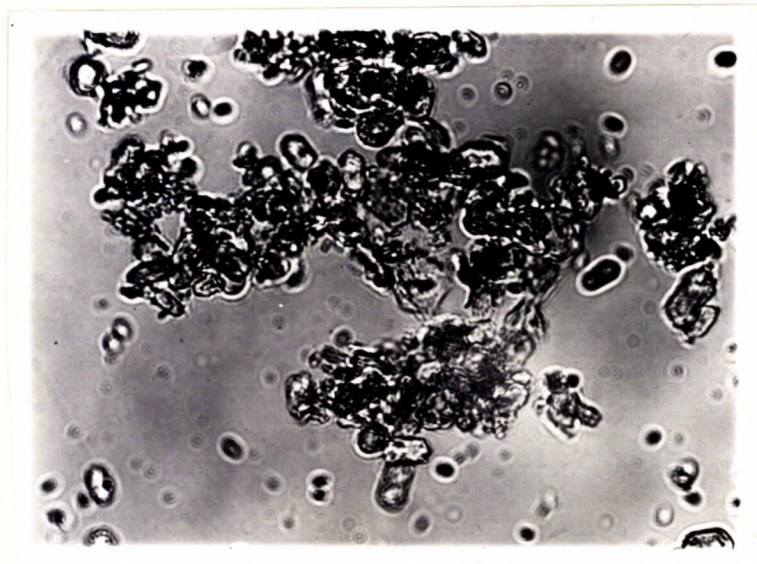


FIGURE 1. - Photomicrograph of Fine Clay fraction in polarized, transmitted light; index of immersion liquid = 1.600, magnification 600X.

2) Medium Clay (MC): With the exception of a few grains with low birefringence (quartz) and some with high birefringence (calcite?), this fraction is composed of irregular, granular aggregates of isotropic material which is yellowish or reddish-brown in colour and translucent to opaque in the larger masses, as shown in Figure 2. The refractive index is variable but appears to be about 1.61. The larger masses are up to 75 microns in size and the sample likely represents colloidal material that was aggregated during drying and was not readily redispersed in the immersion liquid. This seems to be in agreement with the description of Fry (4), who stated that some soils may contain up to 80% of colloidal material (p. 76).

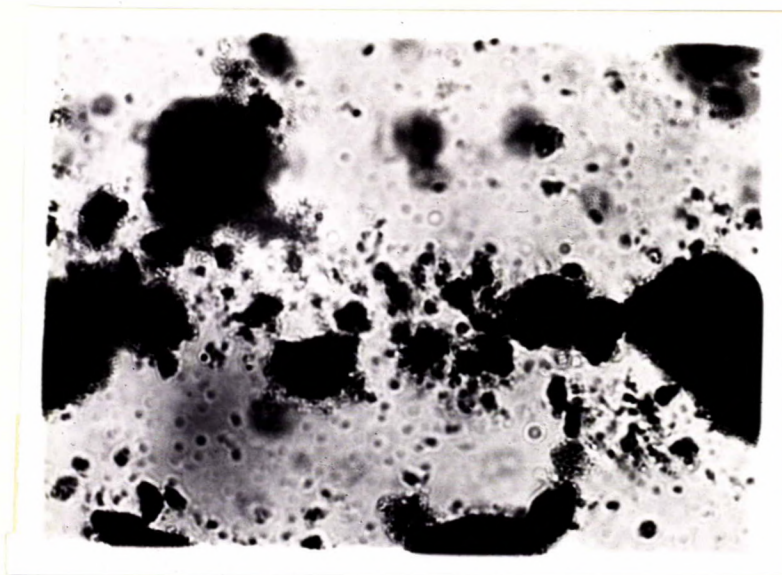


FIGURE 2. - Photomicrograph of Medium Clay fraction in polarized, transmitted light; index of immersion liquid = 1.600, magnification 600X.

3) Coarse Clay (CC): This fraction is similar to MC but there is a higher proportion of birefringent grains, likely quartz and/or feldspar, which are too small (less than about 5 microns) to be identified by optical means. The isotropic material illustrated in Figure 3 is similar to that in MC but is generally more transparent.

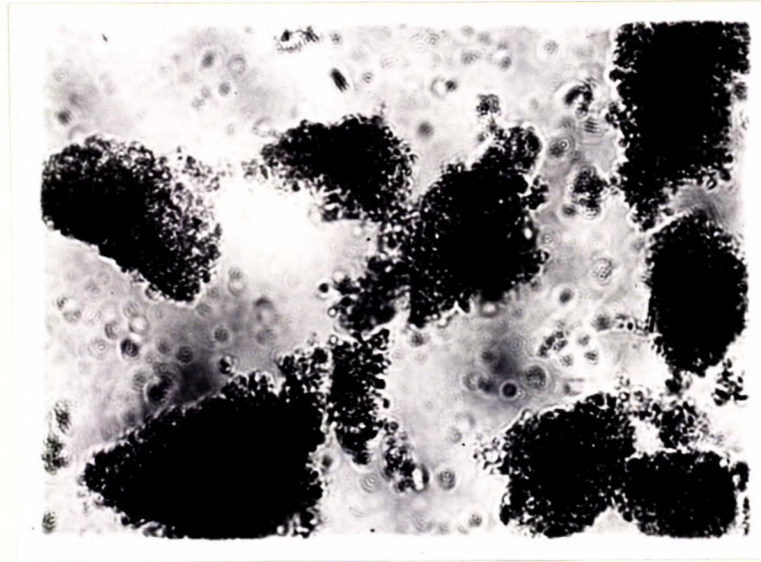


FIGURE 3. - Photomicrograph of Coarse Clay fraction in polarized, transmitted light; index of immersion liquid = 1.600, magnification 600X.

4) Fine Silt (FS): The material in this fraction was more easily dispersed and the maximum particle size is about 10 microns. The proportion of crystalline material is higher and the number of masses of isotropic material lower, than in the finer fractions. Irregular green, pleochroic flakes of chlorite and/or mica appear as the low-relief, grey constituents in Figure 4 and colourless quartz and feldspar grains appear as the high-relief components. A few small masses of amorphous material seem to be almost opaque.

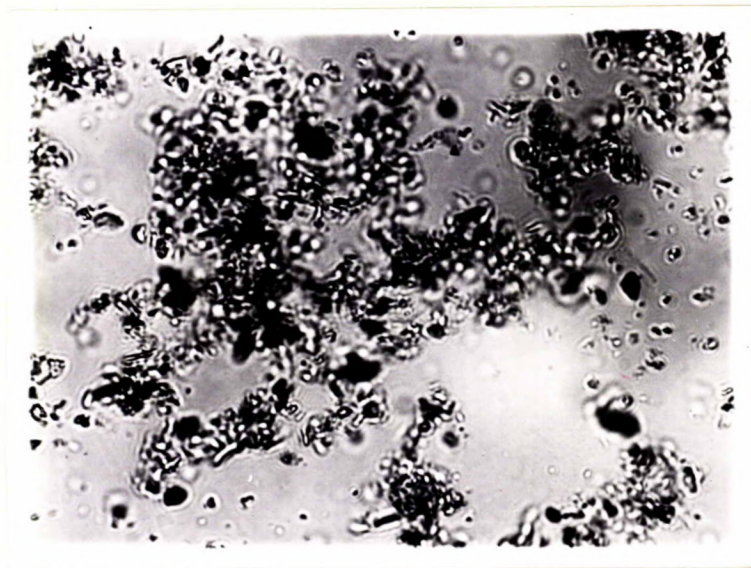


FIGURE 4. - Photomicrograph of Fine Silt fraction in polarized, transmitted light; index of immersion liquid = 1.600, magnification 600X.

5) Medium Silt (MS): The composition of this fraction is similar to that of FS but there is a higher proportion of birefringent grains and the maximum particle size is about 25 microns, as shown in Figure 5.

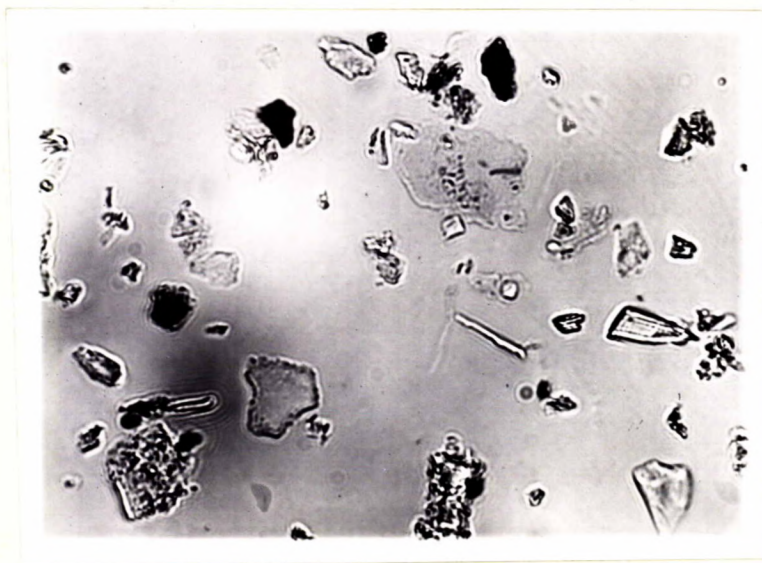


FIGURE 5. - Photomicrograph of Medium Silt fraction in polarized, transmitted light; index of immersion liquid = 1.600, magnification 600X.

6) Coarse Silt (CS): In addition to the constituents observed in MS and FS there are a few, well-formed, pleochroic, green grains of tourmaline, but they cannot be distinguished in Figure 6.

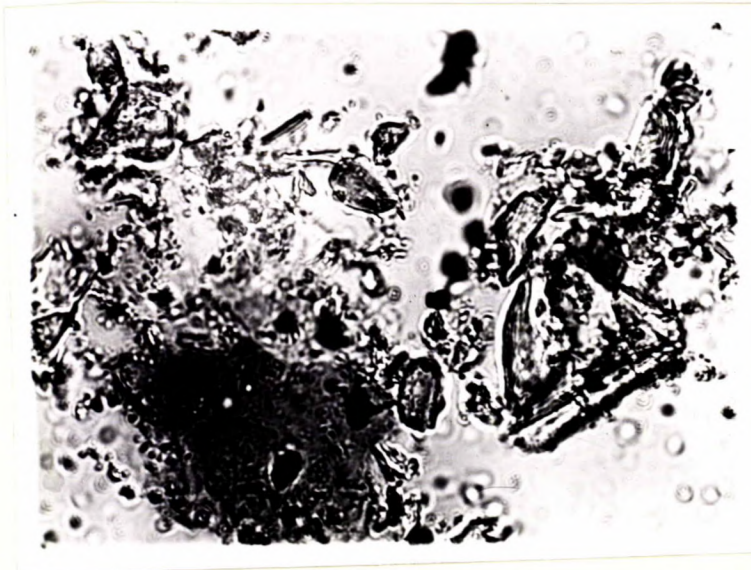


FIGURE 6. - Photomicrograph of Coarse Silt fraction in polarized, transmitted light; index of immersion liquid = 1.600, magnification 600X.

Differential Thermal Analysis

The DTA curves for the silt and clay fractions are shown on pages 10 and 11, along with the curve of the standard attapulgite from Attapulgis, Georgia, described by Kerr et al (5). It was necessary to use a furnace atmosphere of nitrogen to inhibit the oxidation of organic matter so that the inorganic reactions could be observed. It is evident that most of these curves are too indefinite to be useful in establishing the presence of attapulgite and, in the case of CS and of FC, there is no similarity at all. The low-temperature peaks in the range 100° to 150° C likely represent adsorbed water. Exothermic peaks in the range 600° to 700°C in FC, MS and CS may be due to leakage of oxygen into the furnace causing oxidation of the organic

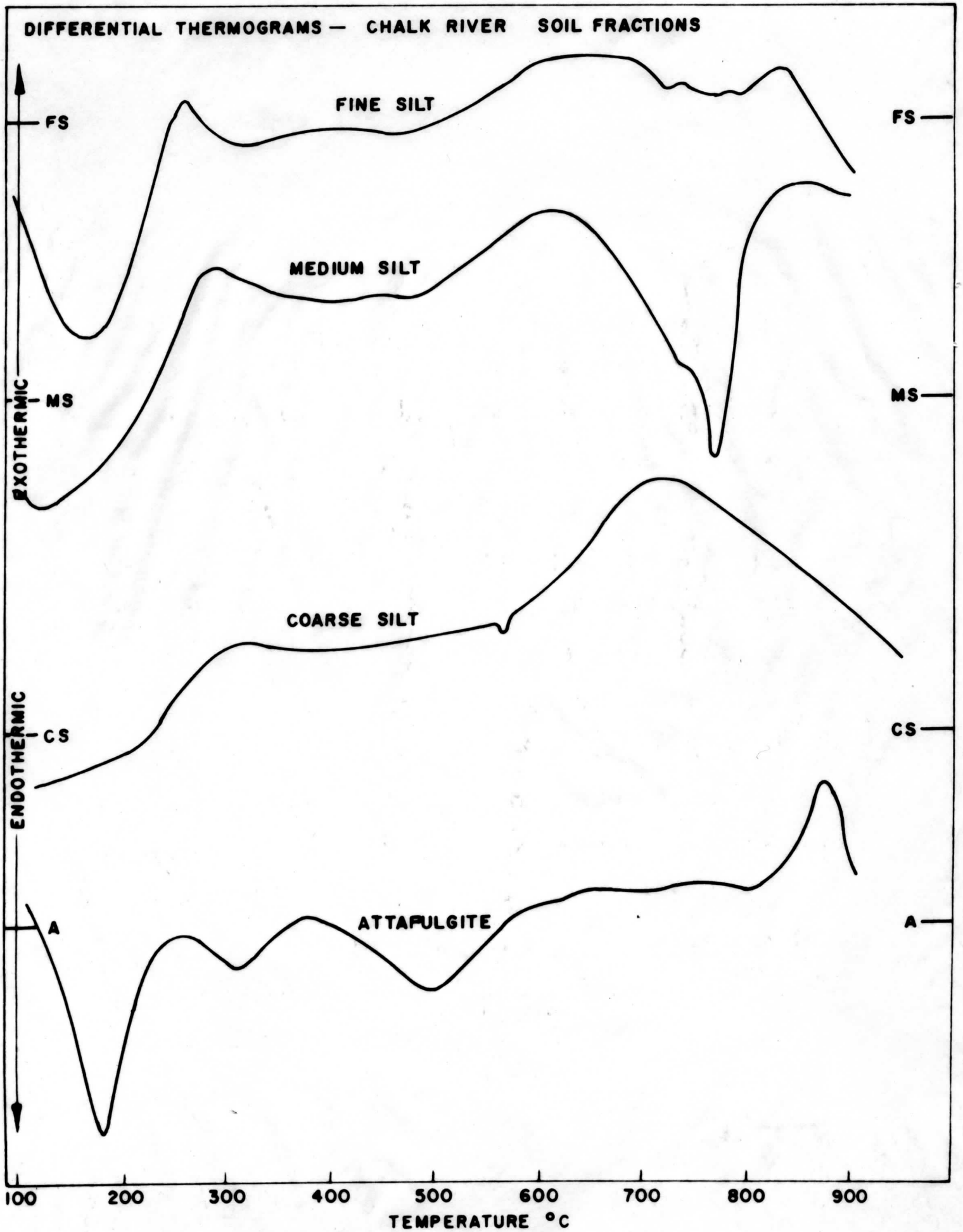


FIGURE 7

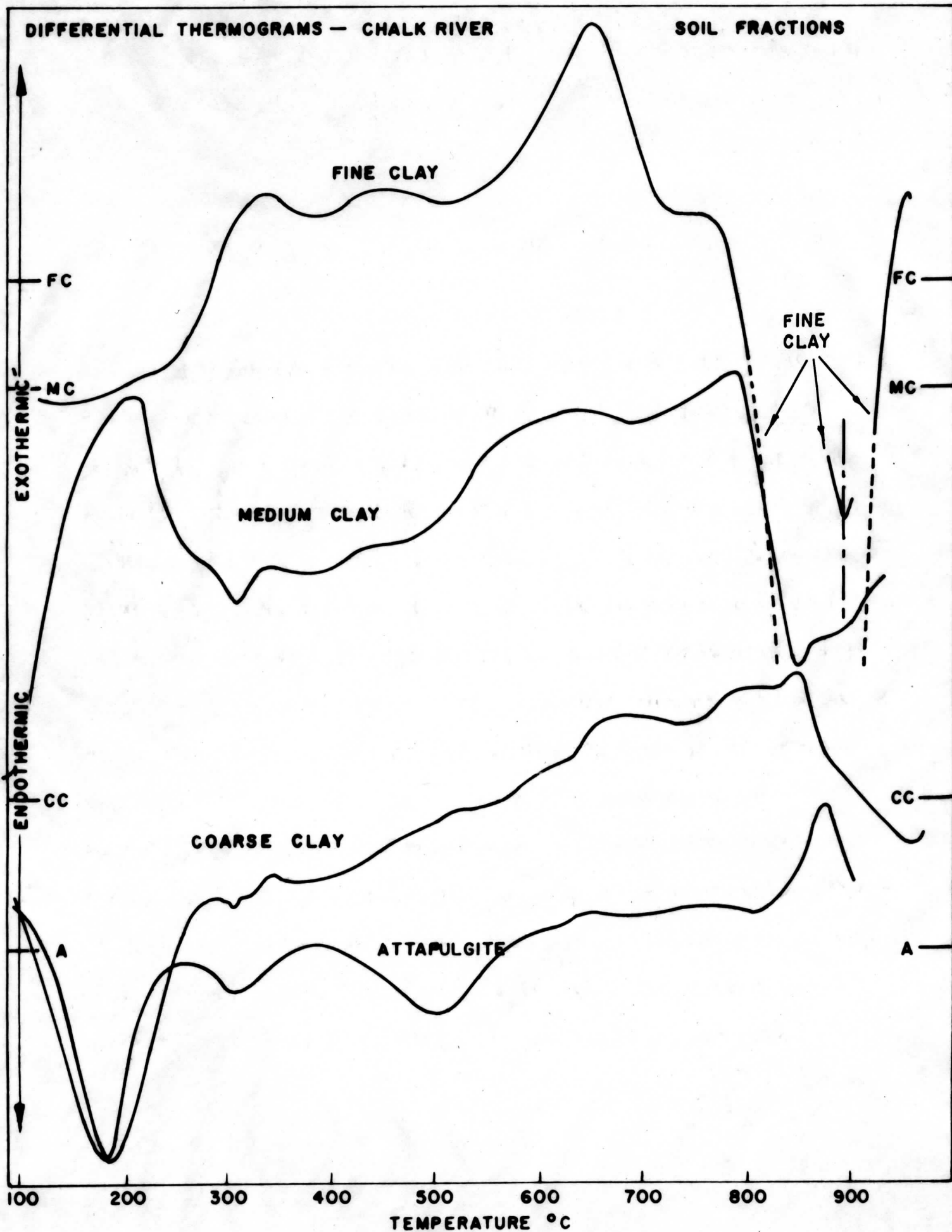


FIGURE 8

matter. Small endothermic peaks at 570°C in CS and MS show the presence of quartz. A large endothermic peak at 930°C in FC indicates a large proportion of calcite and the endothermic peaks at 880°C in MC and at 785°C in MS indicates the presence of small amounts of calcite. The only other identification that was made is the possibility of a mica mineral in FS.

DISCUSSION OF RESULTS

X-ray diffraction analysis, microscopic examination and differential thermal analysis are in general agreement in not showing the presence of attapulgite in any of the samples. The calcite in MC, and MS, indicated by DTA, is apparently below the limit of detection of the X-ray method. All three techniques show that FC is composed, principally, of calcite. The presence of quartz, feldspar, chlorite and mica in the coarser fractions and the occurrence of progressively larger proportions of the aggregated soil colloids in the finer sizes can be considered to be a normal distribution of constituents in a soil that has been developed on transported material rather than on a residual mantle. The only feature that appears to be anomalous is the presence of rather large crystals of calcite as the main constituent of FC. With so much amorphous material of unknown composition present, the chemical analyses could not be used in the mineralogical analysis to any appreciable extent. If all the MgO and CaO in FC are calculated as carbonates they represent about 80% of the sample.

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