CANADA

DEPARTMENT OF ENERGY, MINES AND RESOURCES

OTTAWA

MINES BRANCH INVESTIGATION REPORT IR 67-28

MINERALOGICAL EXAMINATION OF SANDSTONE AND SILTSTONE, THELON FORMATION (PROTEROZOIC), NORTHWEST TERRITORIES

by

R. S. DEAN

MINERAL PROCESSING DIVISION

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1867 1967

APRIL 18, 1967

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PART I

INTRODUCTION

On October 14, 1966, a sample of micaceous material, labelled DF-A298-65, was submitted to the Mineral Processing Division for mineralogical examination by Dr. J.A. Donaldson of the Regional Geology Division, Geological Survey of Canada Branch. The project was assigned Laboratory Number MP-MIN-970.

The sample consisted of micaceous "books" which closely resembled typical muscovite. The largest of these was 5 mm in diameter and several millimetres thick. The material had been collected from an outcrop on the north shore of Schultz Lake, Keewatin District, N.W.T., where it occurred as a fracture-filling in quartzose sandstones of the Thelon Formation (Dubawnt Group). This unit consists of unmetamorphosed sedimentary rocks of Helikian age.

In a preliminary survey to determine the suitability of the sample for potassium-argon age determination, the micaceous flakes were identified as a kaolin mineral by the Petrological Sciences Division, Geological Survey of Canada Branch. The sample was subsequently forwarded to the Mineral Processing Division.

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PROCEDURE AND RESULTS

A portion of the sample was finely ground in an agate mortar, and the resultant powder examined with a Guinier-deWolff 4-sample X-ray powder diffraction camera (1) before and after heat treatment for one-half hour at 580°C.

As shown in Table 1 (Part II), the micaceous grains were found to consist almost entirely of the uncommon kaolin polymorph nacrite (6M kaolin). No nacrite reflections were recorded following the heat treatment.

PART II

INTRODUCTION

The occurrence of nacrite as a fracture-filling in the Thelon sandstone raised questions regarding the detailed mineralogy of the clay-size fraction within this unit. A preliminary study of eight samples by the Petrological Sciences Division had revealed that illite and kaolin were important constituents of the Thelon sandstone matrix. As an initial step toward the resolution of this question two additional samples were examined (as project MP-MIN-973):

(1) DF-A170-63 Green, fissile siltstone, from an outcrop of the upper silty member of the Thelon formation at Lookout Point, on the north shore of the Thelon River, District of MacKenzie.

(2) DF-J120-63 Light pinkish-grey, strongly cross-bedded, medium- to coarse-grained, loosely cemented sandstone, containing a rounded inclusion of white clay measuring approximately 3.5 x 1.5 x 1.0 cm. This sample was collected from an outcrop on the north shore of the Thelon River, northwest of Beaverhill Lake, District of MacKenzie, and is typical of the quartzose sandstones which underlie the siltstones (such as DF-A170-63) and constitute the bulk of the Thelon formation.

PROCEDURE

Sample DF-A170-63

A portion of the sample weighing approximately 30 gm was gently crushed until no aggregates larger than about 1 cm in diameter remained. The fragments were placed in a 300 ml polyethylene bottle containing 100 ml of demineralized distilled water. The cap was sealed and the bottle shaken for 6 hours in an 'Eberbach' reciprocating shaker. The minus 5-micron size fraction was separated from the resultant suspension by centrifugation.

A portion of this material was examined with the Guinier powder camera.

An oriented mount was prepared on a borosilicate glass slide from part of the remaining minus 5-micron suspension. This was scanned with a North American Philips High Angle X-ray Diffractometer under conditions approximating 0% and 100% relative humidity. The differing humidity conditions were obtained by sealing the sample chamber window with thin polyethylene film and introducing a stream of nitrogen gas which had either been passed through a 'Drierite' column or bubbled through hot (60°C) distilled water. Prior to each examination, the mount was allowed to equilibrate for several hours under humidity conditions approximating those under which the analysis was to be made. The same mount was subsequently scanned when saturated with ethylene glycol and following heat treatments for one-half hour at 500°C and 600°C.

Another portion of the minus 5-micron suspension was boiled for 20 minutes in an excess of concentrated hydrochloric acid. An oriented mount prepared from the washed residue was scanned with the diffractometer.

Sample DF-J120-63 (Clay Pebble Inclusion) -

The white clay inclusion was removed from the sandstone by means of a dental drill and gently crushed in an agate mortar. The clay was subsequently transferred to a 150 ml beaker, 50 ml of demineralized distilled water were added, and the resultant slurry was agitated for 3 hours with a magnetic stirrer. The minus 5-micron size fraction was separated by centrifugation.

Oriented mounts of this fine-grained material were scanned with the X-ray diffractometer under conditions approximating 0% and 100% relative humidity, following glycol saturation, after heat treatments for one-half hour at 500°C and 600°C, and following treatment with boiling concentrated hydrochloric acid.

The minus 5-micron fraction of the clay pebble was examined with the Guinier camera and a series of reflections observed which were tentatively identified as being due to the presence of a complex phosphate mineral. Further studies were undertaken in order to verify the identification of this X-ray powder pattern as belonging to a single crystalline phase. A Guinier powder pattern was obtained from the portion of the clay slurry containing particles between 0.005 mm and 0.147 mm (minus 100 mesh) in order to determine the effect of particle size fractionation upon the relative intensities of the reflections in question. Also examined were portions of the minus 5-micron fraction which had either been boiled for 20 minutes in an excess of concentrated hydrochloric acid, stirred for 20 minutes in an excess of 5% acetic acid at room temperature, heated for one hour at 400°C, or heated for one hour at 500°C. The results of these tests supported the hypothesis that the pattern in question represented a single phase.

Additional information concerning the mineralogical composition of the minus 5-micron size fraction of the clay pebble was sought by means of the differential dissolution analysis technique of Hashimoto and Jackson (2). Following heat treatment for one hour at 650°C, dehydroxylated kaolin minerals were dissolved by boiling for 2½ minutes in a large excess of 0.5N sodium hydroxide solution. The residue was collected by centrifugation, washed, and examined with the Guinier camera. The relatively high temperature (650°C) employed during the dehydroxylation phase of this procedure was required by the high thermal stability of one of the kaolin components (dickite).

Sample DF-J120-63 (Sandstone Matrix) -

Fragments totaling approximately 20 gm were removed from the sandstone sample. These loosely cemented aggregates were gently broken down in a mullite mortar. The disaggregated material was transferred to a 150-ml beaker, 50 ml of demineralized distilled water were added, and the resultant slurry was agitated for 6 hours with a magnetic stirrer. The suspension was decanted and the minus 5-micron size fraction separated by centrifugation.

An oriented mount of the minus 5-micron material was scanned with the X-ray diffractometer under conditions approximating 0% and 100% relative humidity, following glycol saturation, and after heat treatment for one-half hour at 500°C. Another portion of the same size fraction was examined with the Guinier camera.

RESULTS

Table 1 lists the results of the X-ray diffraction analyses of the Thelon formation samples.

The minus 5-micron size fraction of the siltstone (DF-J170-63) consists largely of dioctahedral mica (Figure 1 Scan 1). In spite of its fine-grained character, the siltstone mica differed from the common 'illite' of younger sedimentary rocks in that no expanded layers (above 10Å) were detected. A similarity to the clay micas was evident, however, in that the siltstone mica exhibited a high degree of layer stacking disorder. Only very weak, diffuse lines of the 1M polymorph were observed, indicating that the bulk of the mica was 1Md.

A weak, diffuse reflection at 7.10Å was observed in diffractograms of the minus 5-micron siltstone (Figure 1-Scan 1). Scans made at maximum recorder sensitivity disclosed that this reflection was unaffected by heating at 500°C but disappeared completely following the 600°C heat treatment and after the sample was boiled in concentrated hydrochloric acid. The absence of a 14Å reflection from the thermally modified sample suggests that the 7.10Å material is a septechlorite (3, p.339), such as chamosite.

TABLE 1

Mineralogy of Thelon Formation Samples

Mineral	Sample			
	DF-J120-63*		DF-A170-63*	DF-A298-65
	Clay Pebble	Sandstone Matrix	Siltstone	Micaceous "Books"
Nacrite	-	-	-	A
Dickite	B	B	-	-
Kaolinite	B	B	-	-
Mica	C	C	A	-
Septechlorite	-	-	E	-
Quartz	E	D	F	G
Phosphate Mineral	D	F	-	-
K-Feldspar	-	-	D	-
Anatase	F	F	-	-
Hematite	-	-	-	G (?)

Abundance of minerals estimated from "A" (very abundant) to "G" (trace).

* Minus 5-micron size fraction.

The K-feldspar X-ray powder pattern obtained from sample DF-A170-63 consisted of broad lines and bands suggesting, perhaps, that this material is in a highly weathered state.

The mineralogical composition of the minus 5-micron fractions of the clay pebble and sandstone matrix (sample DF-J120-63) were very similar as Table 1 indicates. The X-ray diffractogram of the sandstone matrix illustrated in Figure 1 Scan 2 is essentially identical to those obtained from the fine fraction of the clay pebble.

The X-ray powder photographs indicated that the kaolin within both the sandstone matrix and the clay pebble consisted of dickite (2M kaolin) and kaolinite (1Tc kaolin) in roughly equal proportions. The kaolinite was very well crystallized; the $11\bar{1}$ reflection at 4.18A was well resolved from the $1\bar{1}1$ line at 4.13A (the latter coincided with the 111 dickite reflection) and no incoherent 02ℓ scattering was detected. This would indicate a minimum of b-axis disorder (4, p.62).

X-ray powder data for the phosphate mineral within the clay pebble are listed in Table 2. The degree of coincidence between this pattern and that of goyazite ($SrAl_3(PO_4)_2(OH)_5 \cdot H_2O$), a phosphate of the plumbogummite group, suggested that these minerals, although not identical, were similar.

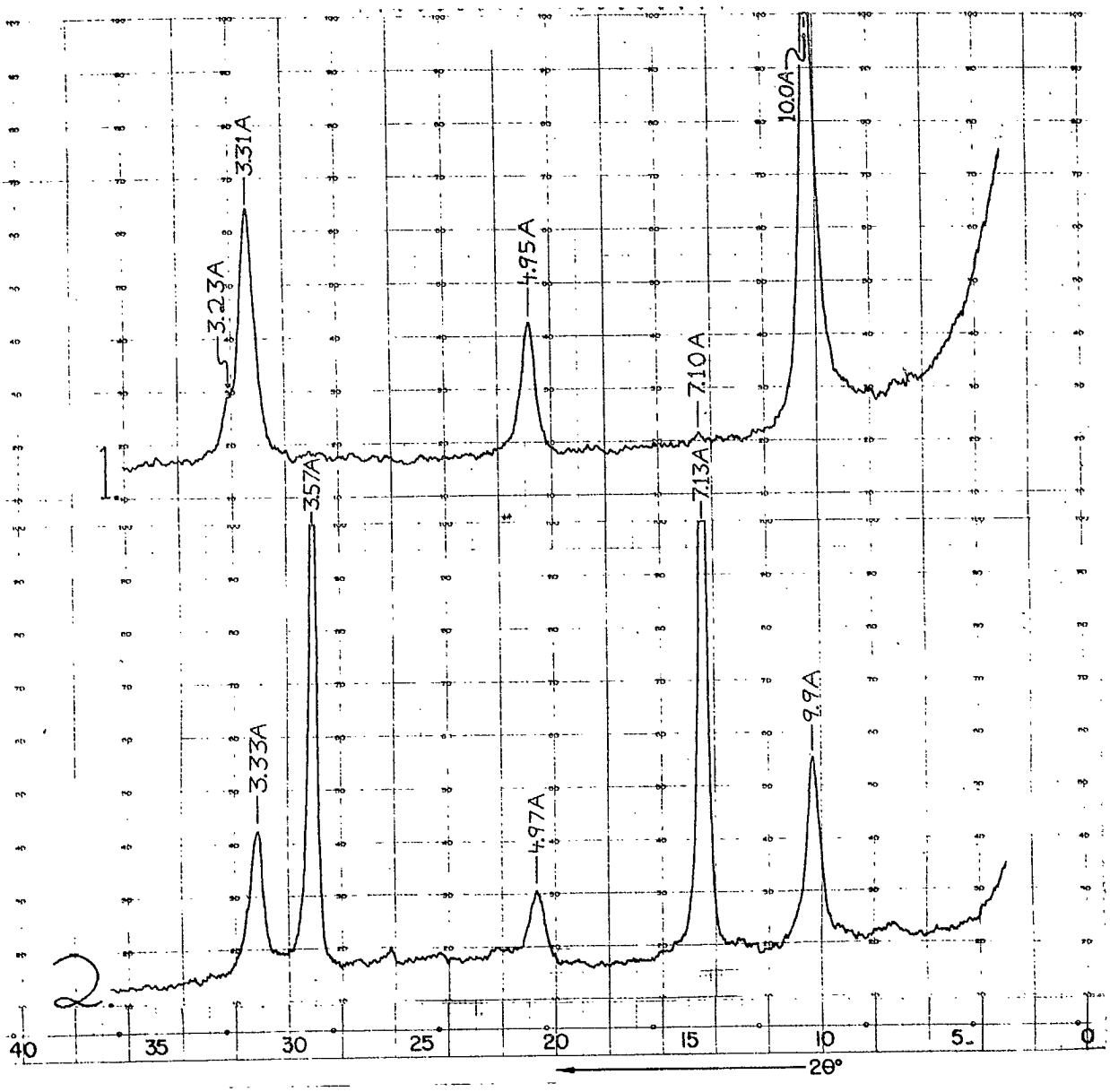

Figure 1 - X-ray diffractograms of oriented mounts of minus 5-micron fraction of Thelon formation samples.

Relative humidity - 0%; Co/Fe radiation; recorder Scale Factor-4; Time Constant-8; scanning speed 1°2θ/minute; 1° slits.

Scan 1 - DF-A170-63 (Siltstone)
Mica - 10.0A; 4.95A; 3.31A
K-Feldspar - 3.23A
Septechlorite - 7.10A

Scan 2 - DF-J120-63 (Sandstone Matrix)
Kaolin (Dickite and Kaolinite) - 7.13A; 3.57A
Mica - 9.9A; 4.97A; 3.33A.

TABLE 2

X-ray Powder Diffraction Data for Goyazite and Phosphate Mineral within Fine Fraction of Sample DF-J120-63 (Clay Pebble)

Goyazite (5, p.31)		DF-J120-63 Phosphate Mineral*	
d (A)	I/I_1	d (A)	Intensity
5.74	100	5.69	s
4.92	20	--	
3.50	80	3.51	ms
-		2.987	w
2.97	100	2.954	vs
2.84	20	2.845	w - diffuse
2.76	20	-	
2.45	20	2.449	w - diffuse
2.20	30	2.212	m
2.16	20	2.188	w - diffuse
2.00	20	-	
1.89	60	1.898	s
1.75	40	1.752	m

* Guinier powder camera; cobalt radiation; quartz standard

The available data did not permit a more precise identification of the Thelon sandstone phosphate mineral to be made. It was noted that the phosphate was completely dissolved (or decomposed) by boiling concentrated hydrochloric acid but was unaffected by 5% acetic acid at room temperature. The 400°C heat treatment resulted in the extinction of some reflections and the displacement of others to larger Bragg angles. Heating at 500°C resulted in a severe decrease in the intensities of all remaining phosphate reflections and none were observed following the 650°C-NaOH differential dissolution analysis. The latter treatment, however, allowed recognition of several of the more prominent anatase reflections, including that at 3.51A, which had previously been masked by the phosphate.

CONCLUSIONS

The mineralogy of the Thelon quartzose sandstone indicates that this unit was deposited during a period of tectonic stability, low relief, and climatic conditions favourable to the development of kaolin within the source area. The rounded clay pebble in sample DF-J120-63 possibly originated as part of a phosphatic mud deposit which became partially indurated before being broken up by currents.

The high clay mica content of the green siltstone (sample DF-A170-63) suggests a decrease in the intensity of weathering within the source area. The presence of K-feldspar and a trioctahedral clay mineral (septechlorite) support this view.

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