

## ILLITE FROM ELDORADO, SASKATCHEWAN

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### ABSTRACT

Chemical composition, X-ray diffraction and electron microscopy indicate that the 0.07–2  $\mu\text{m}$  fraction of a mudstone from Eldorado, Saskatchewan consists in an exceptionally pure illite. The illite has a 1M structure and the chemical formula  $(\text{K}_{0.50}\text{Ca}_{0.03}\text{Na}_{0.02}) (\text{Al}_{1.48}\text{Fe}^{3+}_{0.26}\text{Mg}_{0.23}\text{Fe}^{2+}_{0.13}) (\text{Si}_{3.38}\text{Al}_{0.62})\text{O}_{10}(\text{OH})_2$  with a cation-exchange capacity of 10.6 meq/100 g moisture-free clay. Most illite crystals are lath-shaped averaging  $1 \times 0.2 \mu\text{m}$ . Twins are also observed though less frequently than random aggregates of lath-shaped crystals. Total surface-area was found to be 63  $\text{m}^2/\text{g}$  moisture-free clay by the methylene-blue adsorption method. Infrared-absorption data show a weak band at 750  $\text{cm}^{-1}$  but none at 830  $\text{cm}^{-1}$ . This evidence may be used for the identification of illite, since both absorption bands are well defined in muscovites. In many respects, standard illites, much more closely than muscovites, represent the 10 Å phyllosilicates in soils and sediments. The Eldorado illite in particular should serve as an excellent reference mineral for clay micas in these rocks.

**Keywords:** illite, Eldorado, Saskatchewan, mudstone, reference clay mineral, soils, sediments.

### SOMMAIRE

On montre, par analyse chimique, diffraction X et microscopie électronique, que la fraction 0.07–2  $\mu\text{m}$  d'une argillite provenant d'Eldorado (Saskatchewan) consiste en une illite exceptionnellement pure. De structure 1M, celle-ci répond à la formule  $(\text{K}_{0.50}\text{Ca}_{0.03}\text{Na}_{0.02}) (\text{Al}_{1.48}\text{Fe}^{3+}_{0.26}\text{Mg}_{0.23}\text{Fe}^{2+}_{0.13}) (\text{Si}_{3.38}\text{Al}_{0.62})\text{O}_{10}(\text{OH})_2$  et possède une capacité d'échange cationique de 10.6 milliéquivalents pour 100 g d'argile sèche. La plupart des cristaux d'illite sont en forme de latte de dimensions moyennes  $1 \times 0.2 \mu\text{m}$ . Parfois ils sont maclés, mais ils constituent le plus souvent des agrégats irréguliers orientés au hasard. La surface totale est de 63  $\text{m}^2$  par gramme d'argile sèche, d'après la méthode d'absorption du bleu de méthylène. L'absorption infra-rouge

révèle l'absence de bande d'absorption à 830  $\text{cm}^{-1}$ , mais en montre une très faible à 750  $\text{cm}^{-1}$ . Ces caractéristiques distinguent l'illite de la muscovite, où les deux bandes sont bien nettes. À plusieurs points-de-vue, les illites normales, plutôt que les muscovites, sont représentatives des phyllosilicates à 10 Å dans les sols et les sédiments. L'illite d'Eldorado, en particulier, semble destinée à jouer le rôle de spécimen de référence caractéristique des micas argileux de ces roches.

(Traduit par la Rédaction)

**Mots-clés:** illite, Eldorado, Saskatchewan, argillite, mica argileux, spécimen de référence, sols, sédiments.

### INTRODUCTION

*Illite* was originally proposed by Grim *et al.* (1937) as a general term for the mica-type clay minerals that occur in argillaceous sediments. These minerals have  $d(001) = 10 \text{ \AA}$  and lack substantial proportions of expandable-layer components (Grim 1968). Although the term has been widely used, much confusion has developed regarding its meaning (Wentworth 1970), largely because many investigators have attempted to attach more precision to the original definition. This situation seemed to have improved when Mankin & Dodd (1963) described an exceptionally pure illitic clay from Beavers Bend, Oklahoma, proposing it as a reference clay-mica mineral. Subsequently, Gaudette (1965) reported another illitic clay from Marblehead, Wisconsin. These illites were further supplemented by Gaudette *et al.* (1966) who described an illitic clay from Rock Island, Illinois. Chemical analyses of the three illites gave as a general structural formula  $(\text{K} \gg \text{Na, Ca})_x (\text{Al} > \text{Fe, Mg})_y (\text{Si}_{4-z}, \text{Al}_z) \text{O}_{10}(\text{OH})_2$  with rather narrow ranges of the parameters:  $x = 0.43\text{--}0.66$ ,  $y = 2.00\text{--}2.12$  and  $z = 0.61\text{--}0.76$ . Their findings suggest that these clay micas

may be defined as a specific mineral species distinct from muscovite.

Dean (1978) dealt primarily with the mineralogy of a uraniferous mudstone from Eldorado, Saskatchewan, and pointed out the presence of illite as a predominant component of the mudstone. This offered us a rare opportunity to characterize an exceptionally pure illite sample from fracture fillings. To our knowledge, this is the first report on the mineralogical details of an illite from Canada.

## EXPERIMENTAL

### *Material*

The mudstone sample studied was that previously examined by Dean (1978); it had been collected from the Bolger pit of Eldorado Nuclear Limited, approximately 5 km northeast of Eldorado, Saskatchewan. Although initially reported as Precambrian, evidence now indicates a much more recent origin for the mudstone, which occurs as fracture fillings within ferruginous quartzite (D.L. Dick, pers. comm.). This quartzite, in turn, occurs as layers within the finely cleaved, epidote-bearing argillites of the Tazin metasedimentary unit of Precambrian age. Mudstones very similar to that described here also occur as apparent alteration products within fault zones, and as a fairly extensive surficial deposit that occupied a bedrock depression at this locality (Tremblay 1972, p. 191). All the mudstones are associated with supergene uranium mineralization and contain some of the highest uranium values found in the area.

The mudstone has a uniform texture except for several spots, where very small black or dark brown crystals are concentrated. These were identified as mixtures of uraninite, coffinite and possibly schoepite by X-ray-diffraction methods. After these impurities were carefully removed by hand-picking, a portion of the sample was gently crushed to pass through a 100-mesh nylon sieve (whole sample). The minus 100-mesh fraction was then soaked in water overnight and subsequently dispersed by ultrasonic vibration. Minus 0.07  $\mu\text{m}$  and 0.07–2  $\mu\text{m}$  size fractions were separated by a combination of sedimentation and high-speed centrifugation. The 0.07–2  $\mu\text{m}$  fraction, which constitutes a major portion of the mudstone, was transferred to a porous ceramic plate and dried in an oven at 40°C. A millipore filter was used to remove the minus 0.07  $\mu\text{m}$  fraction from suspension. This very fine fraction was recovered as a thin,

self-supporting clay film after drying at room temperature.

In addition to these two fractions, the un-fractionated whole sample and the 100-mesh–2  $\mu\text{m}$  fraction were also examined during certain phases of this investigation.

### *Analytical methods*

A total chemical analysis was made of the 0.07–2  $\mu\text{m}$  fraction that had been dried overnight at 110°C. Silica was determined gravimetrically following decomposition by the  $\text{Na}_2\text{CO}_3$  fusion method. All metal elements other than Si were determined using a Techtron atomic-absorption spectrophotometer after the sample had been dissolved by the  $\text{HF-HClO}_4$  digestion method. The amount of ferrous iron was measured by titration with  $\text{K}_2\text{Cr}_2\text{O}_7$ , and S and C were determined on a Coleman micro-analyzer using  $\text{V}_2\text{O}_5$  flux. From the latter values  $\text{SO}_2$  and  $\text{CO}_2$  were estimated, and  $\text{H}_2\text{O}$  (+) was subsequently found by subtracting these from the loss on ignition at 1000°C for 1 hour.

X-ray analysis was carried out by three different techniques using Co  $K\alpha$  radiation ( $\lambda$  1.7902 Å). The layer-silicate basal reflections before and after various treatments were obtained with a Philips diffractometer from oriented aggregates prepared by drying 1 ml of clay suspension containing 40 mg of sample on a 25 x 37.5 mm glass slide. A Guinier-de Wolff focusing camera (transmission type) was used for recording the nonbasal layer-silicate reflections, and a 114.6 mm diameter Debye-Scherrer camera was used in the identification of the small black crystals.

Thermogravimetric data were recorded with a Cahn electrothermobalance having a constant heating-rate of 5°C per min. Differential thermal analysis was carried out over the temperature range 25–1000°C with an R.L. Stone DTA apparatus operating at a heating rate of 10°C per min. Infrared-absorption spectra were obtained with a Beckman IR-4250 infrared spectrophotometer using both self-supporting film and KBr disc techniques.

Electron-optics and electron-diffraction observations were made on specimens prepared by allowing drops of very dilute clay suspension to dry at room temperature on copper grids coated with a thin film of carbon. An energy-dispersive spectroscopic method was utilized to supplement chemical data of the 0.07–2  $\mu\text{m}$  fraction and to determine the qualitative chemical composition of the small black crystals. For this purpose, an S4 Cambridge Stereoscan

TABLE 1. INTERPLANAR SPACINGS, RELATIVE INTENSITIES AND INDICES (AS 1 *M*-STRUCTURE) OF NON-BASAL REFLECTIONS FROM ELDORADO ILLITE\*

$d(\text{\AA})$	I	hkl	$d(\text{\AA})$	I	hkl
4.46	100	020	2.36	39	$11\bar{4}$
4.39	43	$11\bar{1}$	2.23	18	040
4.06	16	021	2.20	12	220
3.84	9	?	2.17	8	$13\bar{3}$
3.60	26	$11\bar{2}$	2.12	25	202
3.31	23	022 + 003	1.688	16	$11\bar{6}$
3.04	14	112	1.645	8	151
2.87	7	?	1.625	19	204
2.56	37	$130 + 13\bar{1}$	1.492	89	060
2.54	81	$200 + 004$	1.474	16	330
2.44	21	131			

\* Guinier-camera data

electron microscope was used with a Kevex energy-dispersive X-ray spectrometer attachment.

Surface-area and cation-exchange-capacity measurements were carried out on the 0.07–2  $\mu\text{m}$  fraction using the methylene-blue-absorption method described by Hang & Brindley (1970).

## RESULTS AND DISCUSSION

### X-ray-diffraction data

An inspection of X-ray-diffraction patterns

obtained by the Guinier-de Wolff camera confirmed that the 0.07–2  $\mu\text{m}$  fractions consist almost entirely of clay mica, whereas the coarser fractions contain a moderate amount of quartz, some marcasite, minor feldspars and a trace of pyrite. The diffraction patterns of the finer fractions revealed a single reflection at 1.49  $\text{\AA}$  within the range 1.48 to 1.54  $\text{\AA}$ , indicating that the clay mica is dioctahedral. Its overall pattern closely resembles that of 1*M* muscovite (Smith & Yoder 1956), except for weak reflections at 3.84 and 2.87  $\text{\AA}$ . The latter suggests a trace of a 3*T*-structure mica (Table 1). As can be seen in Figure 1, the reflections with indices  $11\bar{l}$  and  $02l$  are as discrete as those with indices  $13\bar{l}$  and  $20l$ , indicating that the layer-stacking arrangement is fairly regular.

Figure 2 shows a diffractogram of an oriented aggregate of the 0.07–2  $\mu\text{m}$  fraction at 40% r.h. (relative humidity), exhibiting a well-developed integral series of  $00l$  reflections. Measurements of the spacings of eight basal orders gave a mean  $d(001)$  of  $10.01 \pm 0.02$   $\text{\AA}$ . Changes in humidity failed to displace any of the reflections but significantly affected the intensity of the first-order basal reflection. The relative-intensity ratio  $I_{001}(10 \text{\AA})/I_{002}(5 \text{\AA})$  decreased from 4.6 at 0% r.h. to 3.8 at 100% r.h. (Table 2). Variations in the intensity of the 001 peak

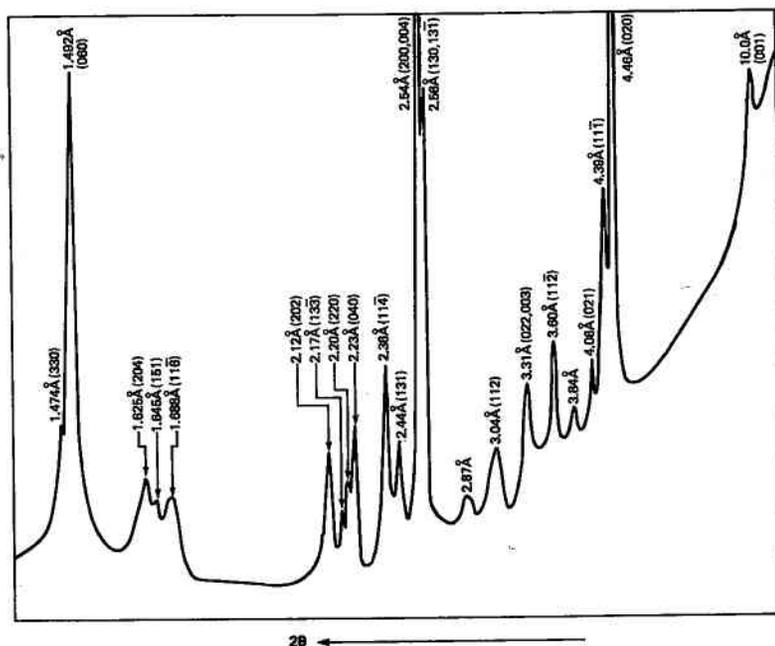


FIG. 1. Microdensitometer trace of a Guinier-de Wolff powder-diffraction pattern of Eldorado illite.

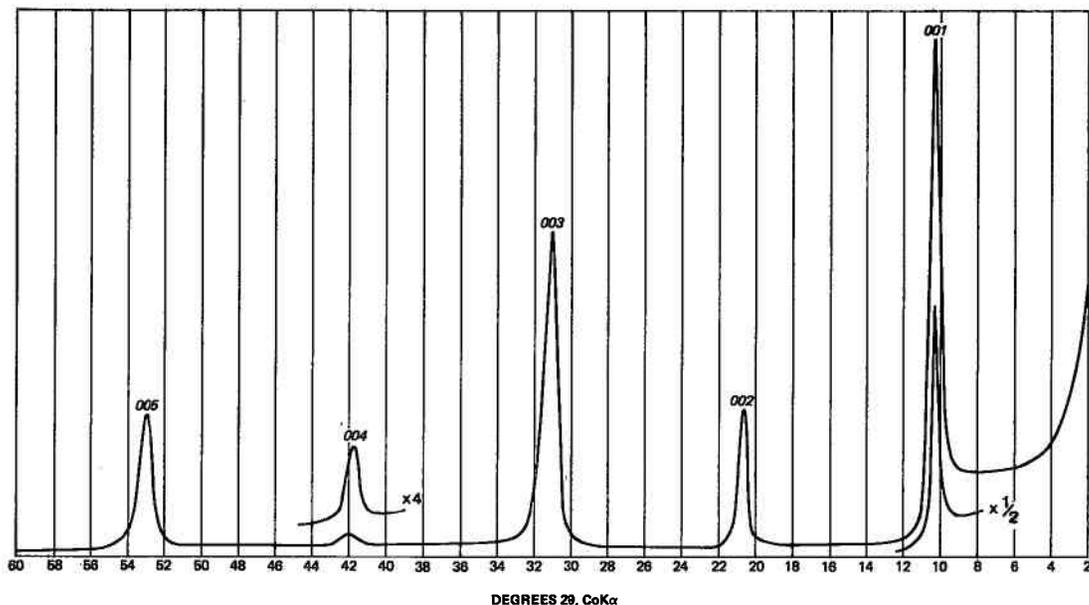


Fig. 2. X-ray diffractogram of a sample of Eldorado illite having preferred orientation. Full scale = 2345 c.p.s.

TABLE 2. INTERPLANAR SPACINGS, INTENSITIES AND INTENSITY RATIOS OF 001, 002 AND 003 BASAL REFLECTIONS OF ELDERADO ILLITE, UNDER VARIOUS EXPERIMENTAL CONDITIONS

Conditions	001		002		003	
	$d(\text{\AA})$	$I_1^*$ ( $I_1/I_2$ ) (cps)	$d(\text{\AA})$	$I_2^*$ ( $I_2/I_2$ ) (cps)	$d(\text{\AA})$	$I_3^*$ ( $I_3/I_2$ ) (cps)
AIR-DRY (40% r.h.)	10.0	2064 (3.4)	4.99	616 (1.0)	3.33	1376 (2.2)
HUMID (100% r.h.)	9.97	2208 (3.8)	5.00	584 (1.0)	3.33	1336 (2.3)
DRY (0% r.h.)	9.97	2736 (4.6)	5.00	592 (1.0)	3.33	1272 (2.2)
GLYCEROL	9.92	1488 (3.4)	4.99	440 (1.0)	3.33	1384 (3.2)
300°C	9.99	1696 (3.0)	4.99	560 (1.0)	3.32	1676 (2.8)
450°C	10.08	1280 (1.7)	5.03	760 (1.0)	3.34	1848 (2.4)
550°C	10.04	1696 (2.1)	5.01	816 (1.0)	3.34	1544 (1.9)
700°C	9.88	760 (2.4)	4.96	320 (1.0)	3.32	800 (2.5)

\* Intensities are expressed in counts per second by peak height, based on the following experimental conditions: 50 Kv 10mA Fe-filtered  $\text{CoK}\alpha$  radiation and a slit system of  $1^\circ-0.024^\circ-1^\circ$ .

induced by changing humidity were reversible, provided that the sample mount was equilibrated at the desired relative humidity for one hour immediately prior to analysis and that the same conditions were maintained during the diffractometer scan. Upon glycerolation (Table 2), the 10 Å reflection showed a significant intensity loss, but no substantial reflection shifts were observed. These diffraction effects are compatible with the presence of a small proportion

of expandable-layer components within the clay-mica sample, probably as little as 5%.

At 40% relative humidity, the width of the illite 10 Å reflection, as expressed by the angular width at half maximum intensity, was  $0.5^\circ$  ( $2\theta$ ). This is much broader than the value of  $0.2-0.3^\circ$  for the microcrystalline muscovites reported by Kodama & Brydon (1968). The angular width varied little with hydration, dehydration and glycerolation. This is a further indication of the presence of only small amounts of expandable-layer components.

Following heat treatment at  $300^\circ\text{C}$  (cf., Table 2), there was little change in the diffraction pattern except for a slight reduction in the 10 Å peak intensity. The  $450^\circ$  and  $550^\circ\text{C}$  heat treatments resulted in a slight increase in the basal spacings, yielding a mean  $d(001)$  of 10.04 Å. The diffraction pattern of the specimen that had been heated to  $700^\circ\text{C}$  showed a major reduction of the 001 basal-reflection intensities and contraction of  $d(001)$  to 9.92 Å.

Dean (1978) reported that a 30-minute treatment of the  $-5\ \mu\text{m}$  fraction of the mudstone with boiling concentrated hydrochloric acid almost completely eliminated the mica reflections and left only a trace of the doublet at 2.54 and 2.56 Å. This indicated that for a dioctahedral mica, the illite was unusually susceptible to acid attack. Following the hot-acid treatment,

TABLE 3. CHEMICAL COMPOSITION AND CALCULATION OF STRUCTURAL FORMULA

	wt. % <sup>*</sup>	wt. % -sulfur and carbon free basis-	wt. % -Ignited basis-	Numbers of cations on the basis of total charges 22 (=O <sub>11</sub> )	
SiO <sub>2</sub>	48.95	48.95	53.35		
TiO <sub>2</sub>	tr	tr	-	Si	3.38
Al <sub>2</sub> O <sub>3</sub>	25.89	25.89	28.22	Al	0.62
Fe <sub>2</sub> O <sub>3</sub>	5.02	5.02	5.47		
FeO	2.40	2.27 <sup>**</sup>	2.47	Al	1.48
MnO	0.08	0.08	0.09	Fe <sup>3+</sup>	0.26
MgO	2.25	2.25	2.45	Fe <sup>2+</sup>	0.13
CaO	0.45	0.45	0.49	Mg	0.23
K <sub>2</sub> O	6.70	6.70	7.30		
Na <sub>2</sub> O	0.15	0.15	0.16	K	0.59
H <sub>2</sub> O (+)	7.60	7.60	-	Na	0.02
SO <sub>2</sub>	0.24	-	-	Ca	0.03
CO <sub>2</sub>	0.95	-	-		
Total	100.68	99.36	100.00		

\* Apart from these major and minor metal elements, an energy dispersive X-ray spectrometer detected a trace of vanadium but not uranium.

\*\* Since the coarser fraction contained pyrite and marcasite, SO<sub>2</sub> was assumed to have originated from these sulfides which might also be present in the 0.07-2 μm fraction sample. Consequently, the amount of ferrous iron required for the amount of S from SO<sub>2</sub> to construct FeS<sub>2</sub> was calculated and its oxide equivalent was subtracted from 2.40%.

only minute traces of quartz and anatase could be detected in X-ray powder patterns of the decomposed illite residue.

#### Chemical data

The chemical analysis of the 0.07-2 μm fraction is shown in Table 3. As in many other clay micas (Weaver & Pollard 1973), low K<sub>2</sub>O, high H<sub>2</sub>O (+) and moderate MgO contents are indicated. The analyzed fraction also contains fairly large amounts of Fe<sub>2</sub>O<sub>3</sub> + FeO. In calculating a structural formula, the H<sub>2</sub>O (+) content was ignored because of the uncertainty in accurately determining it, as pointed out in the discussion of the thermal-analysis data. Calculations of the ionic populations were then based on a total anionic charge of 22, i.e., O<sub>10</sub>(OH)<sub>2</sub>, and yielded a structural formula (K<sub>0.59</sub>Ca<sub>0.03</sub>Na<sub>0.02</sub>)(Al<sub>1.48</sub>Fe<sup>3+</sup><sub>0.26</sub>Mg<sub>0.23</sub>Fe<sup>2+</sup><sub>0.13</sub>)(Si<sub>3.38</sub>Al<sub>0.62</sub>)O<sub>10</sub>(OH)<sub>2</sub>, similar to that of the Beavers Bend illite described by Mankin & Dodd (1963). The ionic population of the octahedral layer is 2.10, which is slightly higher than the ideal dioctahedral-mica value of 2.00. This reflects the presence

of divalent cations, such as Mg and Fe<sup>2+</sup>, substituting for Al.

The total interlayer-cation population of 0.65 is less than the theoretical value of 1.00 for muscovite. Similarly, the substitution of Al for Si in the tetrahedral layer of the illite is low relative to that of muscovite, and most of the excess negative charge created by the latter substitutions is compensated by the positive charge of the interlayer cations.

Although the octahedral ionic population is 2.10, the total octahedral-layer charge is 5.94, giving -0.06 (= 5.94-6.00) as the net charge on the octahedral layer. Assuming that available cation-exchange capacity arises only from the net negative charge originating within the octahedral layer, the cation exchange capacity (CEC) was calculated to be 15.8 meq/100 g dry clay. Experimental determination of CEC by the methylene-blue method yielded a value of 11.5 meq/100 g dry clay. The discrepancy suggests that the net negative charge may be partly compensated by nonexchangeable interlayer cations.

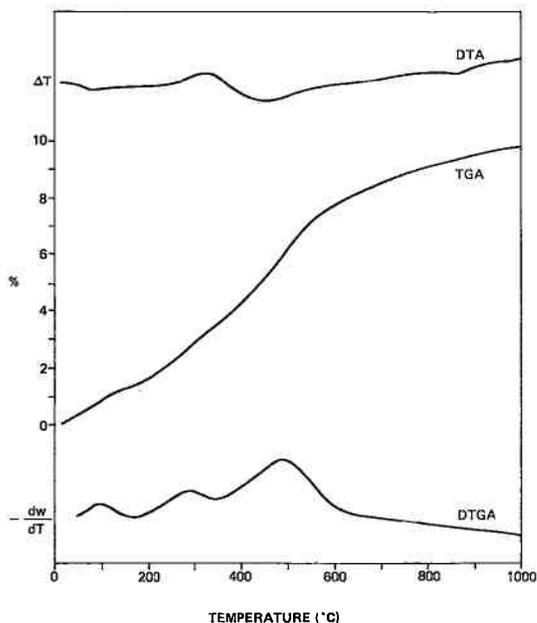


Fig. 3. Thermal data for Eldorado illite.

#### Thermal data

The DTA curve (Fig. 3) shows an exothermic peak at 320°C and three very broad endothermic peaks at about 100, 460 and 860°C. The exothermic reaction may be attributed partly to the oxidation of  $\text{Fe}^{2+}$  to  $\text{Fe}^{3+}$  within the structure. The second endothermic peak is the largest and is due to dehydroxylation. The peak maximum temperature of 460°C is considerably lower than the dehydroxylation temperature commonly reported for illitic clays (about 550°C: Bradley & Grim 1961), indicating unusual thermal instability for the Eldorado illite. The low-temperature endotherm is due to loss of adsorbed water, and the third endotherm at 860°C is probably related to the formation of new phases.

The TGA curve (Fig. 3) exhibits a rather continuous weight loss in the temperature range investigated, with a slightly more pronounced weight loss in the vicinity of 450–550°C. Total weight loss was 9.88%. Because the TGA curve, as it stood, was not very informative, a DTGA curve was plotted from the TGA data (Fig. 3). The DTGA curve indicates a medium-size maximum at 280°C, adjacent to a major one at 480°C, which corresponds to the endotherm at 460°C in the DTA curve. The weight

loss related to the 280°C maximum (2.06%) was calculated from TGA data between 180 and 350°C, where the corresponding minima occurred on the DTGA curve. This weight loss is thought to reflect the decomposition of small amounts of organic matter and the loss of some water molecules trapped in interlayer sites. According to the DTA curve, it may be assumed that dehydroxylation was complete at 650°C, in which case the corresponding weight-loss was 4.7%. The loss above 650°C was 1.62%. From the evidence available, it could not be decided whether 4.7% or the combined value of 6.32% was the real weight-loss resulting from dehydroxylation. Total weight loss as determined by TGA is 8.87% on a moisture-free basis (110–1000°C), in good agreement with the value of 8.79% determined as ignition loss (Table 3).

#### Infrared-absorption spectra

The infrared-absorption spectra of the 0.07–2  $\mu\text{m}$  fraction of the Eldorado illite are shown in Figure 4 (a, b and b'). The absorption band due to the OH-stretching vibration, which appears at 3600  $\text{cm}^{-1}$ , is nearly 40  $\text{cm}^{-1}$  below that in muscovite (Fig. 4, c). This displacement may result from the substitution of Mg and Fe for Al in the octahedral sites, which can affect the OH-stretching frequencies, as has been shown in the series muscovite–phengite–Al-celadonite (Farmer 1974). No substantial dichroic effect was observed in the OH-stretching absorption band at 3600  $\text{cm}^{-1}$  (Fig. 4, b and b') indicating a dominantly dioctahedral structure. A rather strong, broad absorption band at 3400  $\text{cm}^{-1}$  is ascribed to hydroxyl groups involved in water-water hydrogen bonds, probably originating from water molecules adsorbed on the surfaces of clay particles.

As in muscovite (Fig. 4, c), the in-plane Si–O–Si-stretching and Si–O-bending vibrations appear as strong absorption-bands in the 970–1070  $\text{cm}^{-1}$  and 400–550  $\text{cm}^{-1}$  regions, respectively. In the latter region, there are maxima at 430, 470 and 510  $\text{cm}^{-1}$  in the Eldorado illite. In comparison with those of muscovite, the three illite bands are grouped more closely together, and absorption at 430  $\text{cm}^{-1}$  is greater.

The distinct absorption-bands observed in muscovite at 750 and 830  $\text{cm}^{-1}$  are very weak or virtually absent in the illite. Both of these bands are due to the substitution of Al for Si in the tetrahedral layers (Farmer 1974). The

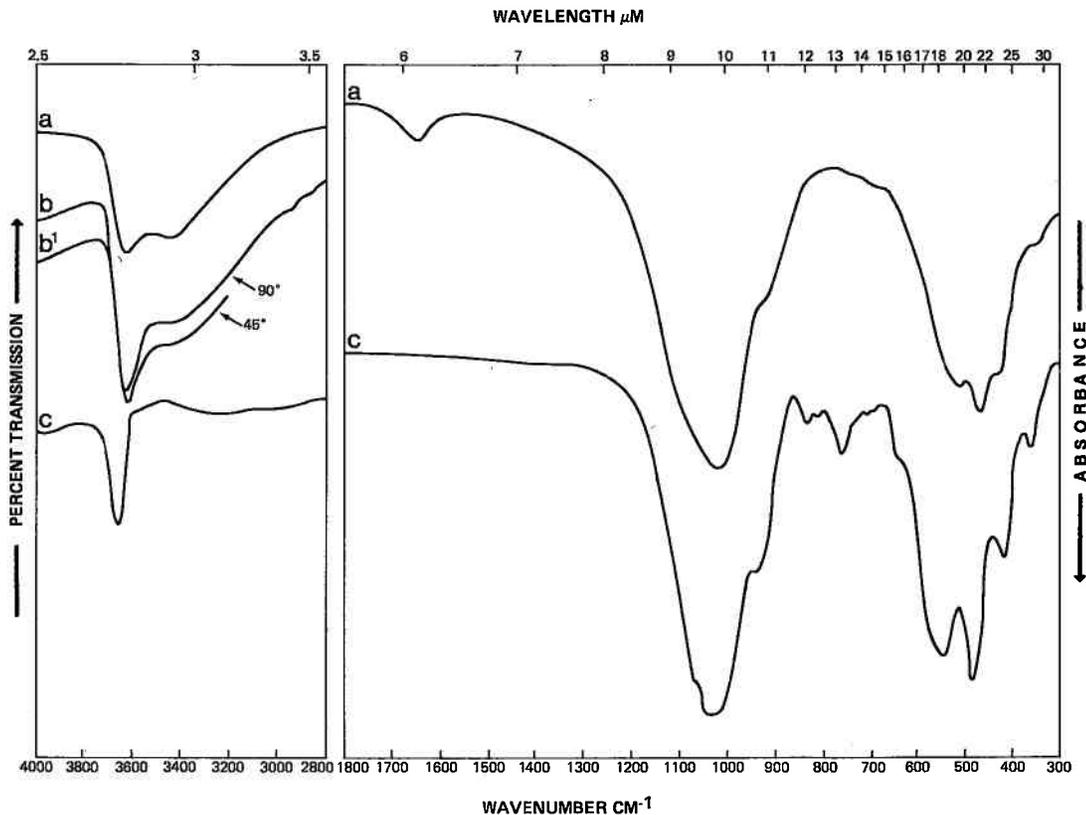


FIG. 4. Infrared-absorption spectra (KBr disc method) of Eldorado illite (a) in comparison with that of a hydrothermal microcrystalline muscovite (c). Infrared-absorption spectra b and b' were obtained from a thin illite film at angles of 90 and 45°, respectively, with the incident infrared beam.

chemical data indicate that the illite has a moderate amount of Al substitution for Si, but less than that in muscovite. This may be the reason for the weakening of the absorption band at  $750\text{ cm}^{-1}$ , which corresponds to Al-O-Si in-plane vibrations. The band at  $830\text{ cm}^{-1}$  was assigned to absorption caused by Al-O out-of-plane vibrations. It was, therefore, expected to be influenced by the presence of other octahedral cations substituting for Al. By comparison with muscovite, the illite shows a lower substitution of Al for Si in the tetrahedral layer and more Mg and Fe atoms substituting for Al in the octahedral layer. The latter situation may lead to a less-ordered cation distribution within the octahedral layer. These are probably the reasons for the decrease in intensity of the absorption band at  $830\text{ cm}^{-1}$ . Librational vibrations involving OH in the range  $915\text{--}950\text{ cm}^{-1}$ , typical of dioctahedral species, were also observed at approximately  $920\text{ cm}^{-1}$ .

#### *Electron-optics and electron-diffraction observations*

Most of the Eldorado illite particles consist of lath-shaped crystallites, as shown in Figure 5a. Their sizes, which range from 2 to  $0.15\text{ }\mu\text{m}$  in length and from 0.3 to  $0.06\text{ }\mu\text{m}$  in width, suggest total surface-areas considerably larger than the  $6\text{ m}^2/\text{g}$  value common among microcrystalline muscovites (Kodama & Brydon 1968). The illite surface area was, in fact, found to be  $63\text{ m}^2/\text{g}$ , which is comparable to that of  $68.1\text{ m}^2/\text{g}$  reported for the Marblehead illite by Gaudette (1965). Lath-shaped crystallites may form twinned aggregates in which groups of parallel, lath-shaped crystals are stacked together at an angle of  $n \times 60^\circ$  relative to each other (Fig. 5b), so that the electron-diffraction patterns obtained from the crystal aggregates still appear unaffected. These observations are similar to those reported for authigenic illites

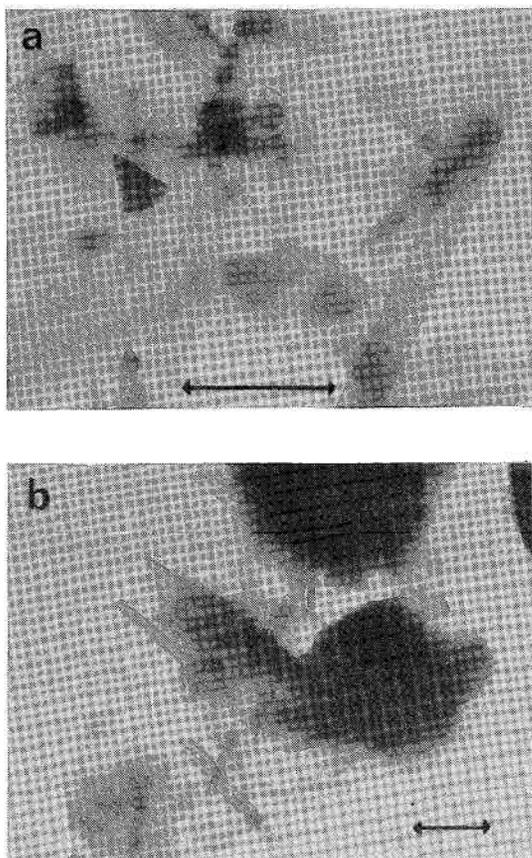


FIG. 5. Electron micrographs of Eldorado illite. Scale indicates 1  $\mu\text{m}$ .

in sandstones by Rex (1966) and Marblehead illite by Güven (1972). Güven suggested that lath-shaped illites may originate through the disintegration of pre-existing mica flakes by parting along the (110) plane. This would result in the exposure of structural hydroxyls and interlayer K atoms situated on this plane. Subsequent chemical alterations, therefore, could greatly affect those cations and anions lying on the plane, and might cause the release of K atoms and the further attachment of hydroxyls or water to octahedral cations exposed by loss of the original hydroxyls. The morphology, low K and high  $\text{H}_2\text{O}$  (+) contents of the Eldorado illite suggest that Güven's hypothesis may apply equally to our sample.

#### *Importance of illite to sedimentary mineralogy*

Although illite may be heterogeneous to some degree, as it contains a proportion of expansible

layers, the presence of the latter cannot account for the observed compositional differences between illites and muscovites. The similarity of the Eldorado illite to the high-purity sedimentary-rock illites from Beavers Bend, Marblehead and Rock Island is unlikely to be fortuitous, as would be the case if illite consisted of mixtures of muscovite and other 2:1 layer-silicate minerals, as envisioned by some workers. Rather, the data appear to support the findings of Hower & Mowatt (1966), who noted the relative deficiency of tetrahedral Al and interlayer K in all size fractions of illites, relative to muscovite. Within broad limits, the compositions of dioctahedral micas in rocks should reflect the conditions under which these were formed.

The Eldorado illite is also of interest as a standard for the quantitative analysis of clay micas by X-ray-diffraction procedures. Although the choice of standards is always a major problem in mineral quantification, it is particularly acute where clay minerals are involved. This

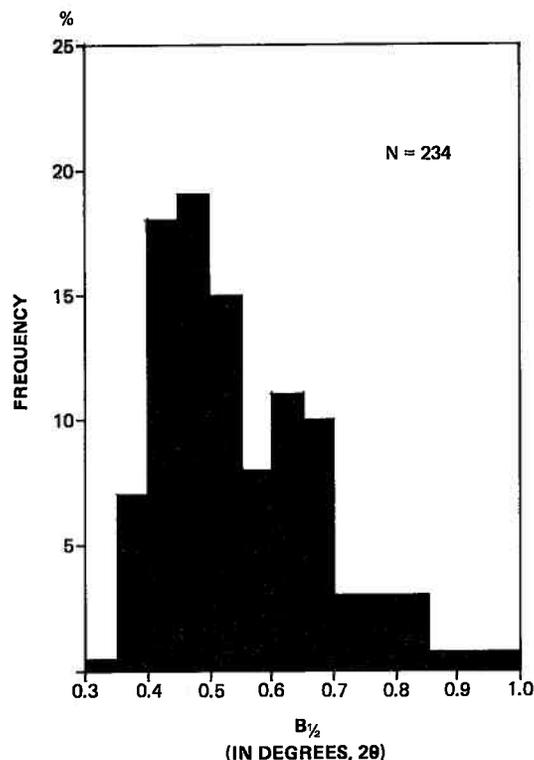


FIG. 6. Histogram of 10 Å-phase minerals in 234 Canadian soil samples showing occurrence frequency versus 10 Å diffraction-peak broadness as expressed by angular width at half height of the peak maximum ( $B_{1/2}$ ).

difficulty is largely due to the compositional variations within mineral species and to the wide variety of crystallinity and particle size of minerals occurring in soils and sediments. Statistics based on diffraction-line-broadness observations of 10 Å reflections in 234 Canadian soil samples (Kodama, unpubl. data) reveal two maxima: 0.45 and 0.65° (2θ), as expressed by the angular width at half height of the peak maximum (Fig. 6). The second maximum is known to be the result of interstratification. Hence, the model angular reflection-width among common soil illites (0.45°) is much closer to the value of 0.5° obtained from the Eldorado illite than to the 0.3° of the 1–2 μm fraction of the microcrystalline muscovite previously used as a standard in our laboratory (Kodama *et al.* 1977). K<sub>2</sub>O values also have been used to estimate mica composition in soils and sediments. The estimate, however, depends on what value is assumed for the K<sub>2</sub>O content of the mica in question. The K<sub>2</sub>O content of the hydrothermal microcrystalline muscovite standard is 9.42% (Kodama *et al.* 1971), higher than the average value of 8% proposed by Weaver (1965) for illitic clay-micas. Both still exceed the 6.70% K<sub>2</sub>O of the present sample. Since it is known that the intensities of mica basal reflections vary with K content (Brown 1955), the illite sample should provide basal-reflection intensity ratios that are more accurate, if these are used in the quantification of illitic micas in soils and sediments. This may be illustrated by a difference between the ratios  $I_{001}$  (10 Å):  $I_{002}$  (5 Å):  $I_{003}$  (3.33 Å) of 4.65:1.00:2.15 for the illite and of 2.16:1.00:1.92 for the standard used previously. The Eldorado illite should serve as an excellent reference-specimen and an appropriate standard.

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