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THE IDENTIFICATION OF SEPIOLITE IN THE PRAIRIE EVAPORITE FORMATION OF SASKATCHEWAN

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ABSTRACT

During a study of the mineralogy of clay seams that occur in the potash ore zones of the Prairie Evaporite Formation, Saskatchewan, sepiolite was detected by X-ray diffraction. Identification of well-crystallized sepiolite is confirmed by transmission-electron microscopy and diffraction. Assuming the sepiolite to be diagenetic, the occurrence is one of the oldest known.

Keywords: sepiolite, clay, electron microscopy, electron diffraction, Prairie Evaporite Formation, Saskatchewan.

SOMMAIRE

Nous avons décelé de la sépiolite par diffraction X au cours d'une étude minéralogique des niveaux d'argile qui se présentent dans les zones minéralisées en potasse de la formation Prairie Evaporite (Saskatchewan). La microscopie électronique par transmission et la diffraction électronique en confirment l'identité sur spécimens bien cristallisés. Si cette sépiolite est diagénétique, c'est l'une des plus anciennes que l'on connaisse.

(Traduit par la Rédaction)

Mots-clés: sépiolite, argile, microscopie électronique, diffraction électronique, formation Prairie Evaporite, Saskatchewan.

During the course of a detailed study of the clay mineralogy at several potash mines in the Middle Devonian Prairie Evaporite Formation near Saskatoon, Saskatchewan (Mossman *et al.*, 1982), X-ray diffractograms of one sample (Vanscoy) were found to show a strong line at about 12 Å. The presence of sepiolite was suspected. In view of the rarity of this species in ancient Phanerozoic sediments, it was decided to seek confirmation using electron microscopy and diffraction.

Specimens for transmission-electron microscopy (TEM) were prepared from a sepiolite standard sample (GSC standard 16299) and from the Vanscoy sample. In each case the sample was simply ground, dispersed in distilled water, and deposited on a carbon film supported on a standard 3.2 mm copper TEM specimen-support grid.

Transmission electron-diffraction patterns were obtained from the standard and from the Vanscoy sample. The lines observed are recorded in Table 1, together with the principal lines listed on the Joint Committee on Powder Diffraction Standards card 13–595, and other data of Brindley (1959), as cited by Caillère & Hénin (1961).

The 4.49 and 4.30 Å rings of the GSC standard appear as a doublet on the TEM plate (Figs. 1, 2), but are not resolved on the Vanscoy sample, where a single diffuse line at about 4.42 Å is observed instead (Figs. 3,4). The lines at 7.45 and 5.01 Å for the standard are weak, and were not observed for the Vanscoy sample. Otherwise, the agreement between the Vanscoy sample and sepiolite is good, corresponding most closely to Brindley's Eski Chehir sample, in which lines at 4.5 and 4.3 Å also were not resolved. X-ray-emission spectroscopy from individual ribbons of the Vanscoy sample showed the presence of magnesium and silicon, supporting the identification of sepiolite, which is, ideally, $Si_{12}Mg_8O_{32} \cdot nH_2O$ or $(Si_{12-x}M_x)(Mg_{8-y}N_1)\,O_{32} \cdot nH_2O$, where *M* and *N* are isomorphous substitutions for Si and Mg (Rautureau & Tchoubar 1974, 1976).

Morphological information supports the diffraction and X-ray-emission data, when the ribbons seen in Figure 3 are compared to sepiolite (Fig. 1). Finally, in Figure 5, which shows part of the field represented by the diffraction pattern of Figure 4, we see an individual ribbon that appears dark because it is diffracting strongly into the row of 12 Å spots. When this same ribbon is viewed in dark field, using

TABLE 1. X-RAY-DIFFRACTION DATA (BRINDLEY) AND ELECTRON-DIFFRACTION DATA (PRESENT WORK) FOR SEPIOLITE

	1		2		3		4	
h k l	d(Å)	I	d(Å)	I	d(Å)	I	d(Å)	I
110	12.1	100	12	S	12.3	60	12	S
130	7.47	10	7.45	W	7.6	5		
150	5.01	8	5.01	W	4.9	6B		
060	4.50	25	4.49	S	4.5	20NR	4.51	S,NR
131	4.31	40	4.30	S	4.3		4.28	
330	4.02	8						
260	3.75	30	3.71	M	3.75	20B	3.70	M
241	3.53	12	3.49	W	3.49	5	3.51	W
080	3.37	30			3.34	20NR,B		
331	3.20	35						
261	3.05	12						
081	2.83	8			2.98			
510,0.10.0	2.69	20			2.67	2.56 40NR,B		
441,281	2.62	30						
371,191	2.56	55	2.55	S			2.56	S
202,042	2.45	25	2.44	M	2.49		2.45	S
222,461	2.41	16	2.41	M	2.43	10		
062,312	2.26	30	2:24	M	2.24	20B	2.28	M
640,2.12.0	2.13	8					2.15	M
082,601	2.07	20	2.02	W	2.08	6B		
	1.88	8						
	1.70	10	1.70	M	1.69	5B	1.73	W

1. JCPDS Card 13-595, sample from Little Cottonwood, Utah (Brindley).
2. Present work, electron diffraction, GSC sepiolite standard.
3. X-ray diffraction, sepiolite from Eski Chehir (Brindley).
4. Present work, electron diffraction, Vanscoy sample.

Notes: B: broad, NR: not resolved; S: strong; M: moderate; W: weak.

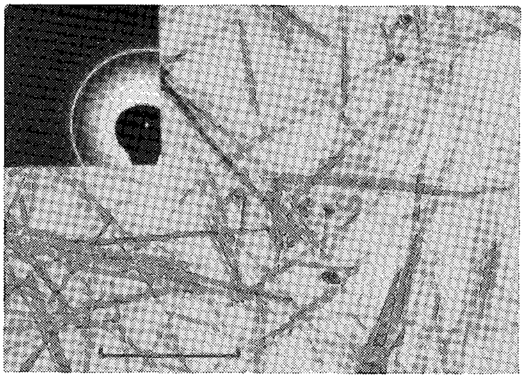

FIG. 1. GSC standard sepiolite. Inset: diffraction pattern. Marked: 1μm.

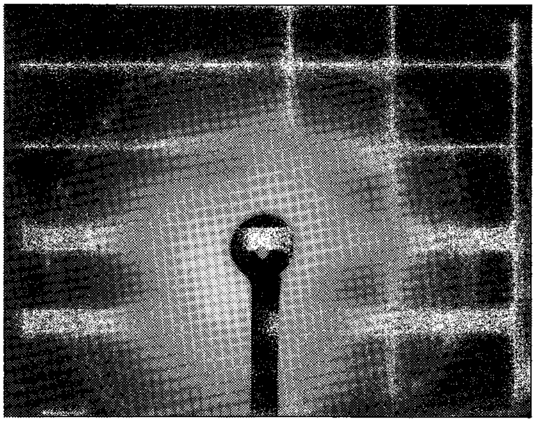

FIG. 2. GSC standard sepiolite. Diffraction pattern of steeply tilted sample, using long camera length. Note 12 Å ring, and well-resolved 4.49 – 4.30 Å doublet. Marker: 1μm.

FIG. 3. Vanscoy sample. Inset: diffraction pattern corresponding to entire field.

one of the 12 Å spots, it appears bright, confirming that it is responsible for these spots. The ribbons observed in the Vanscoy sample are thus shown to correspond to the phase that shows the 12 Å spacing, and whose diffraction pattern generally corresponds to that of sepiolite.

The observations are taken to confirm the identification of sepiolite in the Vanscoy sample. The fact that the 12 Å spots were observed only with the sample strongly tilted is interesting. The sample-preparation technique used would tend to cause the ribbons to lie flat on the supporting film, so that diffraction from planes parallel to the faces of the ribbons would not be observed without tilting steeply enough that a significant number of ribbons were on edge. Thus, this observation suggests that the sepiolites examined grew with a well-developed

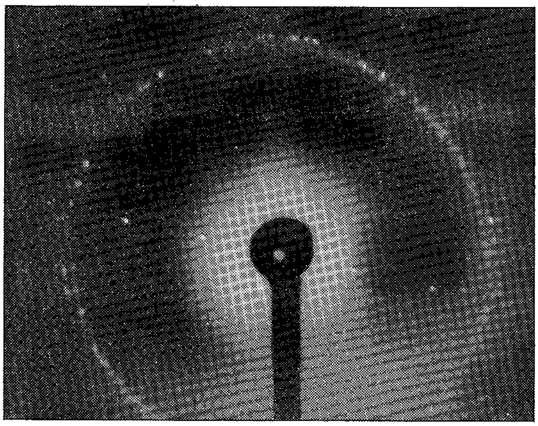

FIG. 4. Vanscoy sample, steeply tilted. Diffraction pattern of ribbon-shaped particles, long camera length. Note 12 Å spots, and broad ring corresponding to unresolved doublet. The inside and outside edges of this ring correspond to spacings of 4.51 and 4.28 Å, respectively.

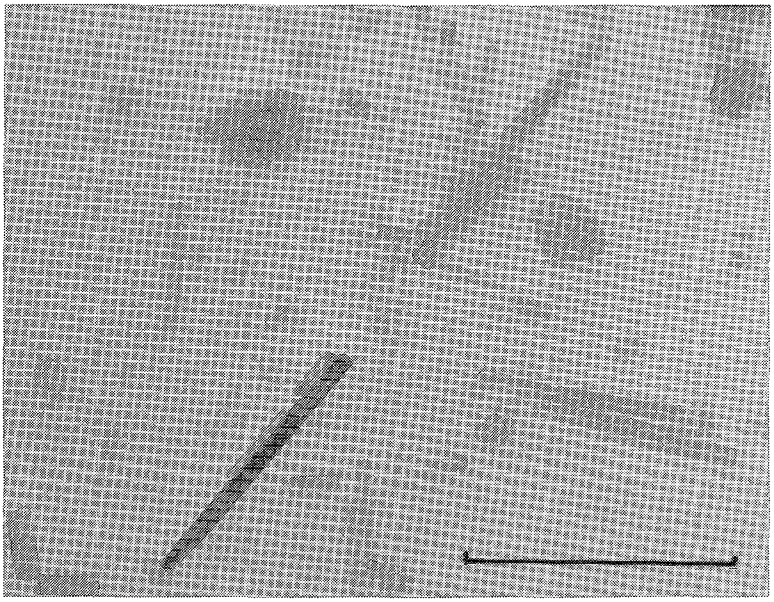

FIG. 5. Part of field responsible for the diffraction pattern of Figure 4. Note diffracting ribbon (dark). Marker: 1μm.

of Rautureau & Tchoubar (1976), who appear to have observed both {100} and {010} forms. A well-developed {010} form would not be consistent with our observations.

Lines given on PDF 13–595 between 3.37 and 2.62 Å were not observed by electron diffraction with either the standard or the Vanscoy sample. In addition, neither the 4.51 and 4.28 Å lines on the Vanscoy sample nor Brindley's lines at 4.5 and 4.3 Å on his Eski Chehir sample were resolved.

These observations may possibly be explained by the observation of Brindley (Caillère & Hénin 1961) that "crystallinity varied from sample to sample". In this connection, we observed that diffraction patterns of sepiolite specimens disappeared on exposure for a few seconds to an intense electron beam, *i.e.*, crystallinity was lost, although long exposure to a less intense beam had no effect. The effect is almost certainly due to heating, possibly resulting in a change from sepiolite to sepiolite II form, either {110} or another form near this. For example, development of the {100} form, at 26.6° to {110}, would be consistent with our results and with some of the observations and then to an amorphous phase, as reported by Caillère & Hénin (1961).

The occurrence of sepiolite in the Prairie Evaporite Formation of Saskatchewan may be one of the oldest known (J.D. Adshead, priv. comm. 1979) if the material formed as a diagenetic phase at an early stage in the development of the evaporite. However, an explanation of this sepiolite as a postdepositional alteration product cannot be ruled out.

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REFERENCES

BRINDLEY, G.W. (1959): X-ray and electron diffraction data for sepiolite. *Amer. Mineral* **44**, 495-500.

CAILLÈRE, S. & HÉNIN, S. (1961): Sepiolite. *In* The X-ray Identification and Crystal Structures of Clay Minerals (G. Brown, ed.). The Mineralogical Society (Clay Minerals Group), London.

MOSSMAN, D.J., DELABIO, R.N. & MACKINTOSH, A.D. (1982): Mineralogy of clay marker seams in some Saskatchewan potash mines. *Can. J. Earth Sci.* **19** (in press).

RAUTUREAU, M. & TCHOUBAR, C. (1974): Précisions concernant l'analyse structurale de la sépiolite par microdiffraction électronique. *C. R. Acad. Sci. Paris* **278B**, 25-28.

——— & ——— (1976): Structural analysis of sepiolite by selected area electron diffraction – relations with physico-chemical properties. *Clays Clay Minerals* **24**, 43-49.

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