Canadian Mineralogist
Vol. 15, pp. 14-21 (1977)

NONTRONITE AND FERRUGINOUS OPAL FROM THE PEACE RIVER IRON DEPOSIT IN ALBERTA, CANADA[1]

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ABSTRACT

Nontronite from the Peace River iron deposit in Alberta occurs as minute flakes in ooliths, and contains about 40 wt.% submicroscopic goethite. The nontronite composition, determined after leaching to remove the goethite, is Fe_2O_3 25.9, FeO 4.6, SiO_2 43.1, Al_2O_3 10.5, and H_2O 15.4 wt.%. Most flakes are amorphous to X-rays, but some contain zones (~0.2 μm in diameter) that are polycrystalline. Consequently, X-ray diffractometer tracings of powder mounts display a very weak peak at 4.5Å. The "amorphous" flakes have the basic nontronite structure but the octahedral layers are distorted. Thus, peaks due to OH coordinated with cations in the octahedral layers are absent from infrared patterns, the Mössbauer values for quadrupole splitting of doublets due to Fe^{3+} are large, and the structure holds large amounts of interlayer water.

Ferruginous opal occurs as matrix between the ooliths in the Peace River deposit. The mineral contains about 10 wt.% submicroscopic goethite, and in humid conditions absorbs large amounts of water which is expelled on drying. The composition of ferruginous opal, determined after leaching to remove the goethite, is Fe_2O_3 29.2, SiO_2 36.8, Al_2O_3 4.4, CaO 0.4, and H_2O 18.1 wt.%.

SOMMAIRE

Dans le gisement de fer de Peace River, en Alberta, la nontronite se présente dans les oolites sous forme de paillettes minuscules qui contiennent environ 40 pour cent en poids de goethite cryptocristalline. Après élimination de la goethite par lixiviation, la nontronite donne à l'analyse: Fe_2O_3 25.9, FeO 4.6, SiO_2 43.1, Al_2O_3 10.5, H_2O 15.4 (pourcentages en poids). La plupart des paillettes sont amorphes aux rayons X, mais certaines d'entre elles renferment des zones polycristallines d'environ 0.2 μm de diamètre. Par conséquent, les diagrammes de poudre enregistrés au diffractomètre à rayons X montrent un maximum très faible à 4.5Å. Les paillettes dites amorphes possèdent essentiellement la structure de la nontronite, avec couches octaédriques déformées. En effet, les spectres infrarouges ne montrent aucune bande assignable aux hydroxyles coordonnés aux cations des couches octaédriques, les valeurs Mössbauer du dédoublement quadripolaire de Fe^{3+} sont élevées, et la structure contient entre les couches une grande quantité d'eau interstitielle.

L'opale ferrugineuse forme un ciment entre les oolites du gîte. Elle contient environ 10 pour cent en poids de goethite cryptocristalline et, exposée à l'humidité, elle absorbe en grande quantité de l'eau qui s'élimine au séchage. Sa composition centésimale, en poids, après séparation de la goethite par lixiviation, est la suivante: Fe_2O_3 29.2, SiO_2 36.8, Al_2O_3 4.4, CaO 0.4, H_2O 18.1.

INTRODUCTION

About 50% of the material in the Peace River oolitic iron deposits of northwestern Alberta is amorphous to X-ray diffraction. Some occurs in ooliths and some as matrix between the ooliths. The main amorphous phase in the ooliths is a nontronite-like mineral, hereafter referred to as Peace River nontronite, and a minor one is a phosphate. The main amorphous matrix material is ferruginous opal and a minor phase is a micaceous mineral similar to illite. The Peace River nontronite and ferruginous opal were studied by a variety of techniques including ore microscopy, electron microprobe, X-ray diffraction, infrared spectroscopy, transmission electron microscopy, Mössbauer spectroscopy, and wet-chemical analyses. Data on the phosphate and illite are reported elsewhere (Petruk 1977).

PEACE RIVER NONTRONITE

Peace River nontronite accounts for about 30 wt.% of the material and for 38% of the iron in the Peace River deposit. The nontronite occurs as concentric layers in ooliths (Fig. 1), and some contains discrete goethite inclusions 0.5 to 100 microns in diameter. The mineral crushes into very minute flakes. Rohrlich (1974) found that mineral flakes in ooliths are oriented

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tangential to the ooliths, and it is likely that the Peace River nontronite is similarly oriented.

In reflected light the Peace River nontronite in some oolith layers is dark grey, in others it is medium grey, and in some it is light grey approaching that of goethite. Combined X-ray diffraction and ore microscope studies show that the Peace River nontronite consists of a mixture of submicroscopic goethite and nontronite. The variation in color under the ore microscope is due to the variable amounts of goethite and nontronite: the dark grey layers contain less goethite than the light grey layers.

X-ray diffraction

X-ray powder diffraction and diffractometer studies of separate grains, concentrates, and residues from concentrates treated with Na-dithionite to dissolve the goethite (Mehra & Jackson 1960), show that most of the nontronite is amorphous at room temperature, at 100% humidity, and after heating to 1000°C. However, a very weak peak was observed at 4.48Å at room temperature, and a very weak one was evident at 18.6Å after heating to 350°C for 30 minutes in a dry nitrogen atmosphere. The peaks are presumed to be the strongest reflections for nontronite.

Transmission electron microscopy

A Peace River nontronite concentrate that was treated with Na-dithionite was studied by transmission electron microscopy to further define the degree of structural ordering. It was found that part of one grain (< 25 microns in diameter) was single-crystal nontronite; parts of 6 grains, a few hundred angstroms or less in diameter, were polycrystalline nontronite; 18 grains were amorphous, and 4 were designated as highly faulted. Table 1 shows that nearly all the nontronite diffraction patterns obtained by transmission electron diffraction had spots for the main nontronite *d*-values of 4.50, 1.51, and 2.61Å, as well as for other lines that are consistent with reported nontronite X-ray powder diffraction patterns (Mikheev 1957; Isophording 1975). The electron diffraction patterns were measured as described by Laufer *et al.* (1975) and by Jubb & Laufer (1976). The highly faulted grains are characterized by spots that remain visible while the crystal is tilted, but the spots move to give the impression that the pattern is being distorted. Such diffraction patterns result from crystals that consist of platelets only one or two atomic layers thick, or that have stacking faults with similar spacings. Flaky minerals that cleave readily would be expected to fall into this group (Hirsch *et al.* 1965).

FIG. 1. Polished section of a sample from the Peace River iron deposit. Shown are concentric nontronite layers in ooliths (med. grey), and the ferruginous opal matrix. The white areas are geothite, and the well-polished grey grains are quartz.

Infrared spectra

Infrared spectra were obtained from a Peace River nontronite concentrate that was prepared

TABLE 1. NONTRONITE DIFFRACTION PATTERNS

TEM of Peace River Grains							X-ray**	
Single Crystal	Pollycrstalline 1	2	3	4	5	6	d(Å)	I
4.38	4.51	4.39	4.55	4.50	4.54	--	4.56	S
--	--	--	--	--	--	4.32*	--	
--	--	--	--	--	--	4.20*	4.12	W
3.49	--	--	--	--	--	--	3.52	W
3.06	--	--	--	--	--	--	--	
--	2.60	--	2.63	2.61	--	2.58	2.62	M
--	--	2.54	--	--	--	2.54	2.56	S
2.48	--	--	--	--	--	2.42	2.44	W
--	2.25	--	2.26	2.26	--	--	2.24	W
--	--	--	--	--	2.18*	--	2.12	W
--	--	--	--	--	1.97	--	--	
--	1.71	--	--	--	--	--	1.74	M
--	--	1.66	--	--	--	--	1.69	M
1.53	1.51	--	1.51	1.50	1.51	1.53	1.53	S

* very weak ** X-ray (Mikheev 1957).

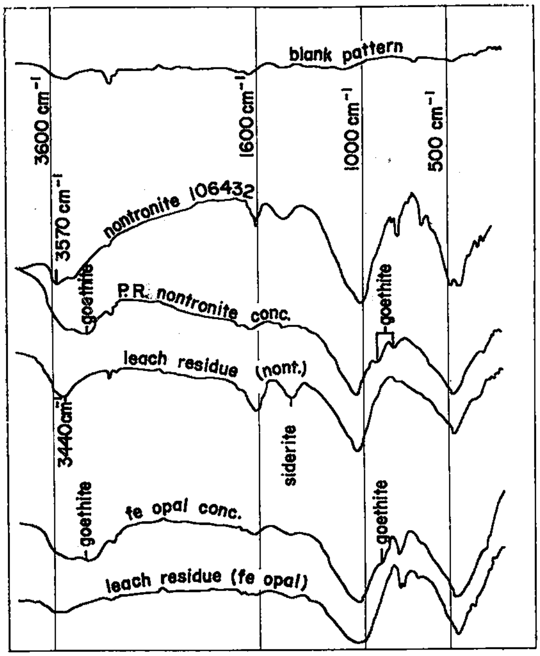

FIG. 2. Infrared patterns for nontronite and ferruginous opal.

in the ore-dressing laboratory, and on a leach residue from the same concentrate treated with Na-dithionite to dissolve the iron oxides. The concentrate showed goethite peaks superimposed on a nontronite pattern, whereas the leach residue showed a siderite peak superimposed on a stronger nontronite pattern (Fig. 2). The nontronite pattern is similar to that of nontronite 106432 from the National Museum of Natural History, Smithsonian Institution as reported in the catalogue of Sadtler Research Laboratory (1973), except that the peaks at 3570 cm^{-1} and at 818 and 850 cm^{-1} are missing for the nontronite from the Peace River deposit. Farmer (1974) reported that interlayer water in smectites gives a broad maximum near 3400 cm^{-1} due to hydroxyl groups involved in water–water hydrogen bonds, a very weak peak at 3610 cm^{-1} due to OH stretching between interlayer water and the very weak surface oxygen bonds, and a peak at 1630 cm^{-1} due to water bending vibration. He further reported that OH groups coordinated to divalent and trivalent cations give a broad peak near 3550 cm^{-1} due to stretching frequency, and peaks at 818 and 850 cm^{-1} due to libration frequencies of Fe^{3+} to OH. The strong peaks around 500 and 1000 cm^{-1} are the main Si-O absorption peaks. The peak at 1410 cm^{-1} is due to siderite. Figure 2 shows that nontronite from the Peace River deposit contains the peaks due to interlayer water but not to the OH coordinated with cations. This suggests that the mineral structure is developed well enough to hold interlayer water, but the octahedral layers of cations with associated OH are not fully developed. This explains why the mineral is largely amorphous to X-ray diffraction.

Microprobe and chemical analysis

Peace River nontronite grains that have a low reflectivity were analyzed with a MAC Model 400 electron microprobe by D. R. Owens, using as standards: Fe_2O_3 for Fe, apatite for Ca and P, Al_2O_3 for Al, and SiO_2 for Si. The data were processed with the program of Rucklidge & Gasparrini (1969). The results show that the mineral contains much more Fe than a normal nontronite (Table 2), obviously due to the presence

TABLE 2. MICROPROBE AND CHEMICAL ANALYSES FOR NONTRONITE AND FERRUGINOUS OPAL

Oxide or Element	Nontronite						Ferruginous Opal				
	P.R. non.** Probe	Conc., Chem.	L. Res., Chem.	Liquor, Chem.	nont. Calc.	Normal* nont.	Probe	Conc., Chem.	L. Res., Chem.	Liquor, Chem.	Calc.
Fe_2O_3†	48.3	63.24	28.17	51.97	25.93	27.53	34.1	42.48	28.32	18.38	29.24
FeO†	3.8	-	-	-	4.61	-	-	-	-	-	-
SiO_2	21.0	16.34	44.26	1.52	43.10	40.02	35.3	33.72	39.38	0.15	36.77
Al_2O_3	4.9	5.20	9.07	-	10.49	10.55	5.2	3.86	4.08	-	4.36
CaO	0.2	1.27	2.69	-	-	-	2.1	0.81	0.41	-	0.44
P_2O_5	0.8	0.73	1.86	-	-	-	-	-	-	-	-
H_2O^+	-	3.02	8.98	-	10.38	21.83	-	5.74	8.68	-	9.28
H_2O^-	-	8.51	4.32	-	4.99	-	-	9.61	8.25	-	8.82
TOTAL		98.31	99.35		99.50	99.93		96.22	89.12		88.91
Fe	36.7	44.23	19.70	36.35	21.72	19.25	23.9	29.74	19.81	10.05	20.45

†Calculated *Analyses 13 (Isophording 1975); **Semi-quantitative transmission electron microscope analysis show that a few nontronite grains also contain small amounts of K and Mg; H_2O^+ = water loss above 105°C; H_2O^- = water loss between 20 and 105°C; L.Res. = leach residue; conc. = concentrate.

of submicroscopic goethite. To determine the Peace River nontronite composition, a concentrate containing some goethite and minor amounts of siderite, quartz, and phosphate was prepared by heavy-liquid separations and treated with Na-dithionite according to the method of Mehra & Jackson (1960) to dissolve the iron oxides but not iron combined with silicates and

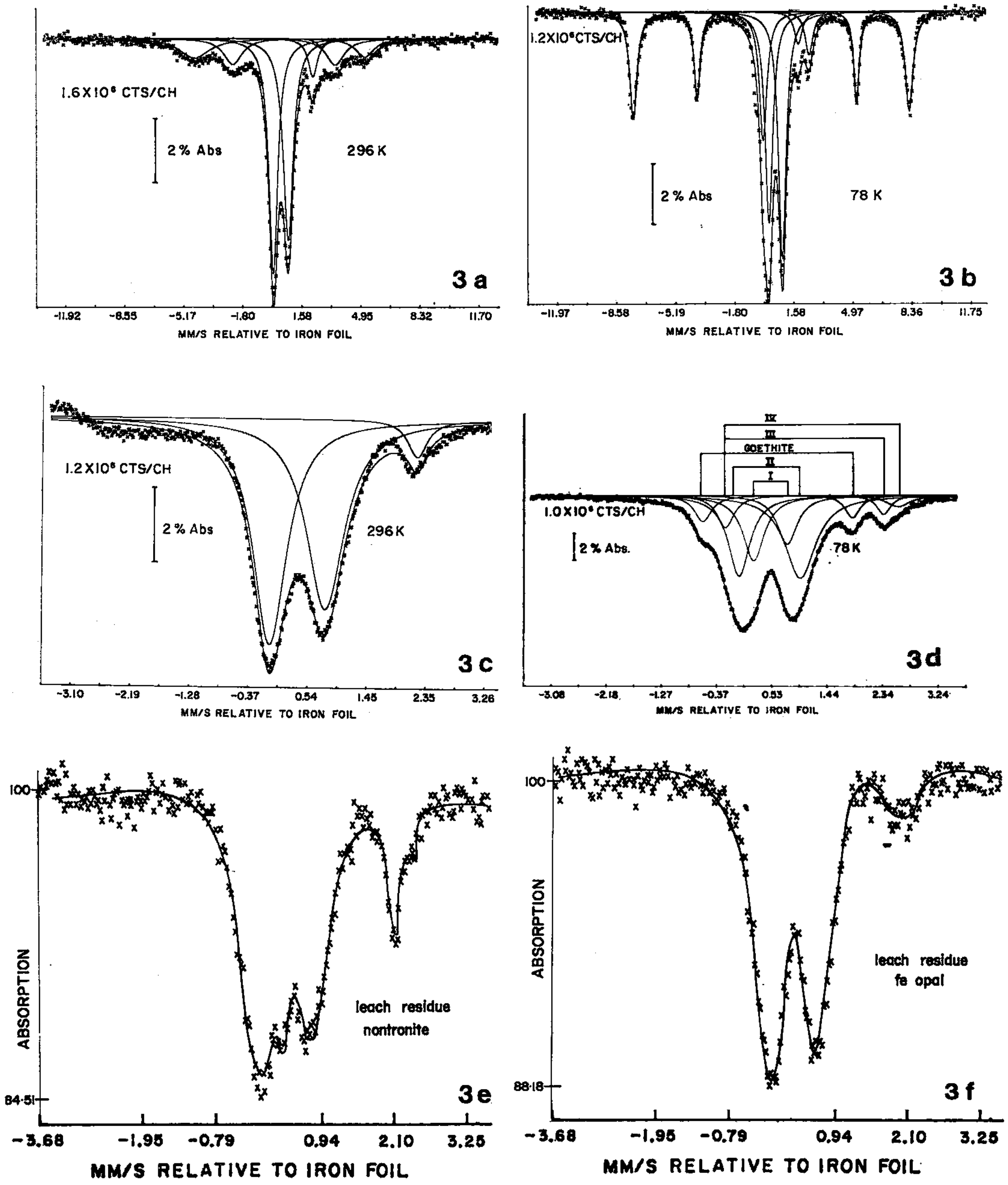

FIG. 3. Mössbauer patterns (a) nontronite, Sample 7A, room temperature, velocity range ≃±15 mm/s; (b)/ nontronite, Sample 7A, liquid nitrogen, velocity range ≃±15 mm/s; (c) nontronite, Sample 7A, room temperature, velocity range ≃±6 mm/s; (d) nontronite, Sample 7A, liquid-nitrogen temperature, velocity range ≃-6 to +6 mm/s, doublets resolved; (e) nontronite, Sample 3, room temperature, velocity range ≃-6 to +6 mm/s. (f) ferruginous opal, room temperature, velocity range ≃-6 to +6 mm/s.

siderite. The Peace River nontronite concentrate, leach residue, and leach liquor were analyzed chemically (Table 2); 56.97 wt. % of the sample dissolved, 51.97 wt. % as Fe_2O_3, and 1.52 wt. % as SiO_2. The remaining 3.48 wt. % probably consisted of other impurities and water that were released when the goethite dissolved. X-ray diffraction studies showed that the leach residue contained 2 wt. % siderite and 7 wt. % quartz. In addition, $CaO:P_2O_5$ was the same as for the phosphate of this ore (Petruk 1977), and, therefore it is assumed that the CaO and P_2O_5 are constituents of a very fine-grained phosphate and not of nontronite. The leach residue analysis was recalculated to account for the quartz, siderite, and phosphate and the calculated values are considered to be the composition of the nontronite (column 6, Table 2). This composition is consistent with reported nontronite compositions (Isophording 1975). It was also calculated that the Peace River nontronite with the lowest reflectivity (P.R. nont.–Table 2) is a mixture of 60 wt. % poorly crystallized nontronite and 40 wt. % submicroscopic goethite.

Mössbauer spectra

Mössbauer spectra were obtained from two residues from Peace River nontronite concentrates treated with Na-dithionite. The spectra for one residue, Sample 7A, were obtained and measured in CANMET laboratories according to the method described by Kodama *et al.* (in press), and experimental envelopes were fitted to a series of Lorentzian line shapes using a least-squares approximation method. The spectrum for the other residue (Sample 3), consisting of nontronite and about 3 wt. % siderite (X-ray diffractometer analysis) and containing 19.3 wt. % Fe (chemical analysis), was obtained and measured as described by Manning & Tricker (1975). Experimental envelopes were fitted to series of Lorentzian line shapes using Stone's (1967) program on the University of Manchester Regional Computer Centre CDC 6600 computer.

Sample 7A was run at room temperature (≃296°K) over a velocity range of about −15 to + 15 mm/s (Fig. 3a), and rerun at liquid-nitrogen temperature (≃78°K) over the same velocity range (Fig. 3b) to eliminate the effects of superparamagnetism on goethite due to very small grain size. The Mössbauer spectrum obtained at liquid-nitrogen temperature displays the four outer goethite peaks (≃−7.7, −4.1, +5.0, and +8.0 mm/s) and a central envelope. The central portion of the spectrum was rerun over a smaller velocity range (≃−6 to +6 mm/s) at room and liquid-nitrogen temperatures (Figs. 3c, d) to identify the nontronite peaks and the remaining inner peaks of the goethite spectrum. Figures 3c and 3d display an envelope with peaks at ≃0.0 and ≃+0.8 mm/s and a peak at ≃+2.2 mm/s. The Mössbauer spectrum for Sample 3, composed of nontronite with minor siderite, shows an envelope with peaks at ≃0.0, ≃+0.3 and ≃+0.8 mm/s and peaks at ≃+2.11 and ≃+2.25 mm/s (Fig. 3e). The relatively sharp absorption peak at ≃2.11 mm/s and its low velocity partner at ≃0.3 mm/s are due to ferrous iron in siderite (Weaver *et al.* 1973). It is concluded, therefore, that the envelope with peaks at ≃0.0 and 0.8 mm/s and the peak at ≃2.2 mm/s are due to nontronite.

TABLE 3. MÖSSBAUER DATA FOR PEACE RIVER NONTRONITE

	Peak Positions		IS	QS mm/s	HW mm/s	ion	%Fe
	1	2					
Doublet I	+0.24	+0.79	0.51	0.55	0.41	Fe^{3+}	31
Doublet II	0.00	+1.00	0.50	1.00	0.51	Fe^{3+}	54
Doublet III	-0.23	+2.35	1.07	2.58	0.31	Fe^{2+}	7
Doublet IV	-0.23	+2.58	1.18	2.81	0.41	Fe^{2+}	8

(IS Relative to Fe foil. Nontronite peak at liquid nitrogen temp.)

The peaks at ≃0.0 and 0.8 mm/s are caused mainly by octahedrally bonded ferric ions (Bancroft 1973). The Fe^{3+} doublet, however, is extremely broad (overall half-width ≃1.5 mm/s) indicating that ferric ions are distributed over several octahedral sites. At least two different sites are expected for nontronite, corresponding to *cis* and *trans* configurations of OH groups. Brunot (1973) reported that a spectrum for hydrothermal nontronite is comprised of a broad doublet (overall half-width ≃0.85 mm/s) with peaks at +0.15 and +0.4 mm/s. He resolved the spectrum into two doublets corresponding to Fe^{3+} ions in *cis* and *trans* sites. For the *cis* sites he calculated isomer shifts (IS), relative to his source (Co diffused into a Pd foil), and quadrupole splitting (QS) as 0.17 and 0.24 mm/s respectively, and for *trans* sites 0.18 and 0.69 mm/s. In a review paper on Mössbauer studies on clay minerals, Coey (1975) lists, for a series of eleven montmorillonites and nontronites, IS (relative to iron metal) = 0.38 and QS = 0.5 mm/s.

The spectrum for the Peace River nontronite is more complex than any nontronite spectra reported in the literature (Weaver *et al.* 1967; Taylor *et al.* 1968; Brunot 1973; Coey 1975). Attempts to resolve the main envelope of the spectra obtained at room temperature (Figs. 3c & 3e) into two or more doublets were unsuccessful. However, the main envelope and peak at

+2.2 mm/s of the spectrum run at liquid-nitrogen temperature ($\simeq$78°K) over a small velocity range ($\pm$6 mm/s) were resolved into five sets of doublets (doublets I, II, III, IV, and the inner goethite peaks) (Fig. 3d). The peak positions and calculated IS (relative to iron foil), QS, and HW values for the doublets are given in Table 3.

The values for the quadrupole splitting (QS = 0.55 and 1.00 mm/s) of the doublets are larger than values reported previously for this mineral, indicating distorted octahedral sites (Bancroft, 1973). The half-widths of individual peaks of the doublets (0.51 and 0.41) are broader than half-widths of individual peaks for ordered materials (usually <0.3 mm/s), which suggests that the octahedral layers of Peace River nontronite are poorly developed. The interpretation that octahedral sites are distorted and octahedral layers poorly developed confirm results obtained by infrared analysis.

The IS values (0.51 and 0.50 mm/s) for Fe^{3+} seem high, but, when converted to a comparable base, are in agreement with the reported IS values for nontronite. Coey (1975) reported 0.38 relative to iron metal and Brunot (1973) reported 0.17 and 0.18 for the *cis* and *trans* doublets relative to Pd–foil. Brunot's values, converted relative to Fe foil by adding 0.185 mm/s (Muir *et al.* 1966), are 0.355 and 0.365 respectively. The IS values of this study were obtained at liquid-nitrogen temperature. Using the classical temperature dependence limit of $\simeq$–0.07 mm/s per 100°K, calculated for iron (Frauenfelder 1963), the values at room temperature are $\simeq$0.35 to 0.40 mm/s relative to iron foil for the two doublets.

The peaks for doublets III and IV (Table 3) are due to ferrous ions in the octahedral layers of nontronite. In this respect the Peace River nontronite is similar to a ferrous nontronite from the Red Sea whose Mössbauer spectrum has a ferroan absorption peak at 2.3 mm/s (Bischoff 1972).

Intensity data, calculated by assuming that the Mössbauer fraction is the same for all iron sites, give the Fe^{3+}:Fe^{2+} ratio in nontronite from Sample 3 as 84:16 and for Sample 7A as 85:15. It can be calculated, from this Fe^{3+}:Fe^{2+} ratio and from the amount of goethite dissolved by Na-dithionite treatment, that Peace River nontronite, which contains 36.7 wt. % Fe, has 8% of the iron as Fe^{2+} in nontronite, 40% as Fe^{3+} in nontronite, and 52% as Fe^{3+} in submicroscopic goethite.

FERRUGINOUS OPAL

Ferruginous opal is the main constituent of the matrix and accounts for about 18 wt. % of the material in the deposit and for 10% of the iron. The mineral is pale yellow and isotropic in transmitted light. It absorbs water in humid conditions and expels it upon drying, causing shrinkage cracks which probably account for the ore becoming crumbly when exposed to dry atmospheric conditions. The ferruginous opal crushes into large rounded grains. The specific gravity of the mineral, determined with heavy liquids, is about 2.0.

TABLE 4. X-RAY DIFFRACTION PATTERN* OF OPAL FROM AUSTRALIA

hkl	*I*(obs.)	*d*(obs.)	*d*(calc.)
1 0 0	10	4.32	4.33
0 0 6	8	4.08	4.10
1 1 0	9	2.50	2.50
2 0 3	4	2.10	2.09
0 0 12	5	2.05	2.04
2 0 9	1	1.71	1.70
0 1 14	7	1.62	1.62
1 2 6	1	1.53	1.52
3 0 0	6	1.44	1.44
2 1 9	3	1.41	1.40
0 0 18	4	1.36	1.36
1 0 18	1	1.30	1.30
2 2 0	3	1.25	1.25
1 1 18	5	1.20	1.20

a = 5.00±0.01, c = 24.52±0.01Å

* indexed on a hexagonal cell by J.M. Stewart, CANMET.

Ferruginous opal is largely amorphous to X-ray diffraction, but J. T. Jubb, CANMET, found by electron transmission microscope studies of a ground ferruginous opal concentrate, which was treated to dissolve the goethite, that about 2 to 5% of the grains are hexagonal, $a \cong$ 5Å and $c \cong$ 25Å. A similar hexagonal structure was obtained for an opal purchased from a mineral collector in Australia by A. E. Johnson. The indexed X-ray diffraction pattern for the Australian opal is given in Table 4 because the ASTM card file does not include an opal pattern.

Infrared patterns were obtained for a ferruginous opal concentrate before and after dissolving goethite with Na-dithionite (Fig. 2). The patterns are typical opal patterns but the one with goethite (ferruginous opal concentrate) shows weak goethite peaks and the one without goethite (leach residue) shows a weak quartz peak (between 500 and 1000 cm^{-1}). There is no infrared evidence of water held by the ferruginous opal.

The composition of the ferruginous opal exposed to atmospheric conditions was determined with an electron microprobe using the same standards as for the Peace River nontronite (Table 2). To determine whether the ferruginous opal contains submicroscopic goethite, a ferruginous opal concentrate was prepared by heavy liquids and treated with Na-dithionite to dissolve the goethite but not the ferruginous opal. The concentrate, leach residue, and leach liquor were analyzed chemically (Table 2) and by X-ray diffractometer; 18.51 wt. % of the sample dissolved, 18.38 wt. % as Fe_2O_3, and 0.15 wt. % as SiO_2. X-ray diffractometer analyses showed that the leach residue contained 1.5 wt. % siderite and 5 wt. % quartz. The partial composition of the leach residue was corrected for the siderite and quartz and the corrected data are included in Table 2 (last column) as the probable composition for opal without submicroscopic goethite. It was calculated from these data that the ferruginous opal in the deposit consists of an intimate mixture of 90 wt. % opal and 10 wt. % submicroscopic goethite.

Mössbauer spectrum for the ferruginous opal, obtained at room temperature (Fig. 3f) shows that nearly all the iron is ferric as indicated by the doublet with peaks at 0 and +0.8 mm/s. This doublet is similar to the main doublet observed for Fe^{3+} in nontronite.

DISCUSSION

The data obtained in this study confirm that the main amorphous phases are nontronite in the ooliths and ferruginous opal in the matrix; both contain submicroscopic goethite. The nontronite is partly crystalline; manifestations of the structure can be recognized on X-ray diffraction patterns, and individual crystallized nontronite grains can be found with transmission electron diffraction. Infrared and Mössbauer studies show that the undeveloped part of the nontronite structure is the octahedral layers of cations coordinated to the OH groups. Grains with undeveloped layers appear to be faulted. The mineral structure is sufficiently developed, however, to hold large amounts of interlayer water. Unlike most nontronites, that from Peace River contains some ferrous iron, and in this respect the mineral is like the ferroan nontronite from the Red Sea (Bischoff 1972). Petruk (1977) postulated that nearly all the iron in nontronite *in situ* in the lower horizon of the Peace River deposit is ferrous, but upon exposure to the atmosphere it is progressively oxidized.

The ferruginous opal has the X-ray diffraction and infrared properties of opal, breaks into large pieces on drying, and is soft. It contains large amounts of iron and other impurities and holds large amounts of absorbed water. Some of the absorbed water escapes when the opal is heated to 105°C, but much remains. Thermogravimetric analysis of the ore (Petruk 1977) suggests that absorbed water continues to escape as the ore is heated to 600°C. It is possible that the hardness of the mineral is related to the amount of absorbed water.

ACKNOWLEDGEMENTS

The authors thank all those who contributed to this study by performing special analyses; included are D. R. Owens for electron microprobe analyses; J. C. Hole, J. C. Cloutier, R. S. Kobus, and J. A. Graham for the chemical analyses; J. T. Jubb and K. M. Pickwick for some transmission electron microscope analyses; J. M. Stewart and E. J. Murray for the X-ray diffraction analyses; and R. S. Dean for X-ray diffraction at 100% humidity and in the dry nitrogen atmosphere. Acknowledgements are also extended to Y. Bourgoin for preparing polished and thin sections and for sample preparation; to I. B. Klymowsky for sample preparation, to R. G. Pinard for sample preparation and general technical assistance rendered during the course of the investigation; and Professor J. M. Thomas, University College of Wales, Abersystwyth, for the use of Mössbauer equipment and for computing facilities.

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Manuscript received July 1976, emended October 1976.