

HOWLITE AND ULEXITE FROM THE CARBONIFEROUS GYPSUM AND ANHYDRITE BEDS IN WESTERN NEWFOUNDLAND

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ABSTRACT

The Carboniferous anhydrite and gypsum beds in southwestern Newfoundland contain small quantities of ulexite ($\text{NaCaB}_5\text{O}_9 \cdot 8\text{H}_2\text{O}$) and howlite ($\text{Ca}_2\text{SiB}_5\text{O}_9(\text{OH})_5$). The ulexite occurs as irregular patches 1-3 cm in diameter, consisting of white fibrous crystals; the howlite forms composite framboidal nodules about 0.5 to 2 cm in diameter, consisting of thin platy crystals less than 0.1 mm long with prominent (100) planes. The two minerals were also found at two localities in Nova Scotia not previously reported — the Gays River Pb-Zn deposit and a gypsum quarry near Milford. The howlite is believed to have been formed from a gel; the formation of the borates was contemporaneous with the precipitation of calcium sulphate.

Borate minerals, in small quantities, seem to be common in the anhydrite and gypsum of the Carboniferous Basin of the Atlantic Provinces, but no reports of similar occurrences elsewhere are known at this time. The normal amounts of boron in seawater are sufficient to account for the observed quantity of borates. It is possible that similar small quantities of borates are present in other anhydrite-gypsum deposits but have been overlooked. If they are truly absent from marine evaporite beds elsewhere, the difference may be caused by differences in pH, solubility, rate of evaporation *etc.* rather than by the presence or absence of external sources of boron.

RÉSUMÉ

Les lits de gypse et d'anhydrite de la période Carbonifère du sud-ouest de Terre-Neuve contiennent de petites quantités d'ulexite ($\text{NaCaB}_5\text{O}_9 \cdot 8\text{H}_2\text{O}$) et de howlite, $\text{Ca}_2\text{SiB}_5\text{O}_9(\text{OH})_5$. L'ulexite se présente sous forme de taches irrégulières de 1 à 3 cm de diamètre; celles-ci sont composées de cristaux blancs fibreux; la howlite, elle, se présente sous

forme de nodules composés en forme de framboises de 0.5 à 2 cm de diamètre; elle est composée de minces cristaux en plaquettes d'une longueur de moins de 0.1 mm avec des plans (100) proéminents. Ces deux minéraux ont aussi été trouvés dans deux régions non-localisées auparavant de la Nouvelle-Ecosse — le gisement Pb-Zn de Gays River et une carrière de gypse près de Milford. La howlite semble avoir été formée d'un gel; la formation des borates était contemporaine avec la précipitation de sulfate de calcium.

La présence des minéraux de borate (en petites quantités) semble être courante dans l'anhydrite et le gypse du Bassin Carbonifère des provinces de l'Atlantique; par contre on ne rapporte aucune autre présence similaire ailleurs pour l'instant. Les quantités normales de bore dans l'eau de mer sont suffisantes pour expliquer la quantité de borates observée. Il est fort possible que de semblables petites quantités de borates soient présentes dans d'autres gisements d'anhydrite-gypse mais elles auraient été négligées. Si elles sont vraiment absentes de d'autres lits marins évaporés, la différence peut être causée par les variances dans le pH, la solubilité, le taux d'évaporation, *etc.* plutôt que par la présence ou l'absence des sources extérieures de bore.

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INTRODUCTION

The occurrence of borate minerals in the gypsum deposits of Lower Carboniferous (Windsor) age in the Atlantic Provinces of Canada was first discovered more than 100 years ago by Henry How, then Professor of Chemistry and Natural History at King's College in Windsor, Nova Scotia. How described "natro-borocalcite" (now ulexite, $\text{NaCaB}_5\text{O}_9 \cdot 8\text{H}_2\text{O}$) from "a bed of a very extensive formation of gypsum at Windsor, on the Clifton estate, lately the property of Judge Haliburton" (How 1857), and "cryptomorphite" (now ginorite, $\text{Ca}_2\text{B}_{14}\text{O}_{28} \cdot 8\text{H}_2\text{O}$; Hey & Bannister 1952) from "the same quarry, at a distance of about 100 yards and

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at about 20 feet lower level" (How 1861). Both minerals were associated with glauberite, $\text{Na}_2\text{Ca}(\text{SO}_4)_2$. Later, How reported a new mineral which he named "silicoborocalcite" (now howlite, $\text{Ca}_2\text{SiB}_5\text{O}_9(\text{OH})_2$) from a locality at Noel, Hants Co., Nova Scotia, "in nodules sometimes as large as a man's head" (How 1868, 1877).

How's original localities were later re-examined and his discoveries confirmed by N. R. Goodman, who also found howlite as nodules up to 5 cm in diameter at Wentworth, Hants County, as "small nodules" at Cheverie (Hants Co.) and Bevis Point (Victoria Co., Cape Breton Island), and as nodules more than 15 cm in diameter "in a clay horizon in the National Gypsum Co. quarries at Dingwall (Cape Breton Island)". Goodman further reported ulexite at Wentworth, Dingwall, Bevis Point, and at White Head near Cheverie, and in addition described the occurrence of danburite ($\text{CaSi}_2\text{B}_2\text{O}_8$) at White Head and "in the shore outcrop 2 miles SW of Iona, Victoria Co. (Cape Breton Island)", and inyoite, $\text{Ca}_2\text{B}_6\text{O}_{11} \cdot 13\text{H}_2\text{O}$, as "beautiful colourless crystals" from the Wentworth area, Hants Co., all in Nova Scotia (Goodman 1957). Earlier, T. L. Walker had reported the occur-

rence of ulexite and inyoite from the gypsum quarry at Hillsborough, south of Moncton, New Brunswick (Walker 1921). More recently, Sabina (1965) found crystals of howlite up to 1.3 cm long, associated with fine-grained howlite and danburite, in gypsum near Iona, Cape Breton Island (see Fig. 1 for all localities).

In this paper, we report for the first time the occurrence of howlite and ulexite in the Lower Carboniferous evaporite deposits in western Newfoundland. In addition, during a recent field trip organized by the Nova Scotia Department of Mines, we found howlite and ulexite in anhydrite in drill-cores from the newly explored Gays River Pb-Zn deposit about 8 km southeast of Shubenacadie, and ulexite nodules in the large gypsum quarry near Milford, both in Halifax Co., Nova Scotia. We further obtained from Mr. W. Lewis, Senior Geologist of the Fundy Gypsum Co., a specimen of gypsum from Hillsborough, New Brunswick, containing several ulexite nodules, thus confirming Walker's earlier report. It is clear therefore that borate minerals, in small quantities, are widespread in the Lower Carboniferous evaporite basin of the Atlantic Provinces.

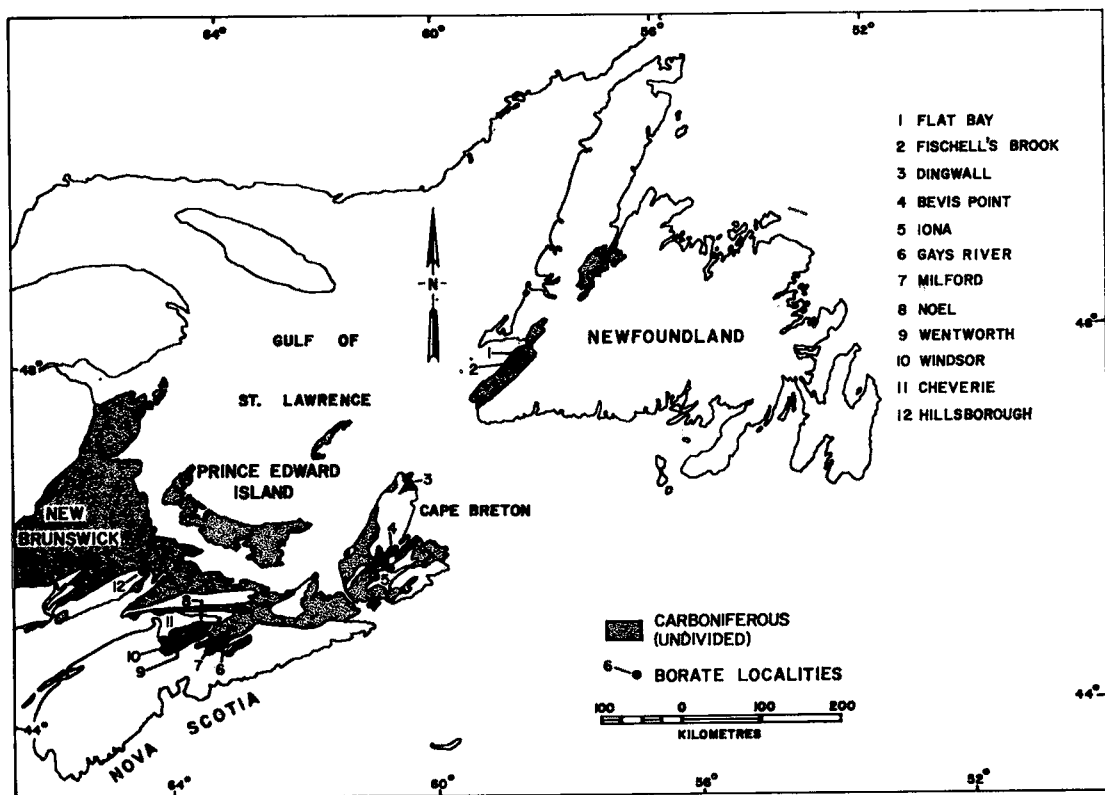


FIG. 1. Borate localities in the Carboniferous basin of the Atlantic region.

BORATES IN NEWFOUNDLAND

Geological setting

The Carboniferous rocks of Newfoundland form the northeasterly part of a large Carboniferous basin extending across the Gulf of St. Lawrence through Prince Edward Island to New Brunswick and Nova Scotia. In Newfoundland, Carboniferous rocks are found in two separate basins within a relatively narrow, elongate, northeasterly-trending belt in the western part of the island, extending from Cape Anguille in the southwest to White Bay in the northeast. Rocks of the southwesterly Carboniferous basin are exposed along the shore of St. George's Bay and in the adjacent Anguille Mountains, forming a belt 20-25 km wide and about 100 km long; they are separated by a major fault of regional extent from the igneous and metamorphic rocks of the Long Range Complex to the southeast.

The Carboniferous sediments of this basin have been subdivided into three stratigraphic units. The lowest, the Anguille Group, consists of a thick sequence of terrestrial clastic sediments, mainly sandstone, shale and conglomerate; it is of Mississippian age, and is believed to be equivalent to the Upper Horton Group of Nova Scotia. The middle unit, the Codroy Group, comprises limestone, siltstone and thick Ca sulphate beds, followed by siltstone, sandstone and minor limestone of non-marine origin. The Codroy Group, of Upper Mississippian age, has been correlated with the Windsor Group of Nova Scotia. The upper unit, mainly Pennsylvanian but including locally rocks of Late Mississippian age, is the Barachois Group, consisting of sandstone, siltstone and conglomerate with minor limestone and some coal beds. It is equivalent in part to the Canso Group and partly to the Pictou Group of Nova Scotia (Riley 1962).

Evaporite sequences, comprising mainly anhydrite and gypsum with a few salt beds, are confined to the Codroy Group. The occurrences of gypsum at numerous localities within the area have been described by several authors (e.g. Hayes & Johnson 1938; Baird 1957). A large gypsum quarry south of Flat Bay, owned by the Flintkote Co. of Canada, is now in operation; in 1973, it produced about 735,000 tons of gypsum (Stonehouse 1973).

Borate localities

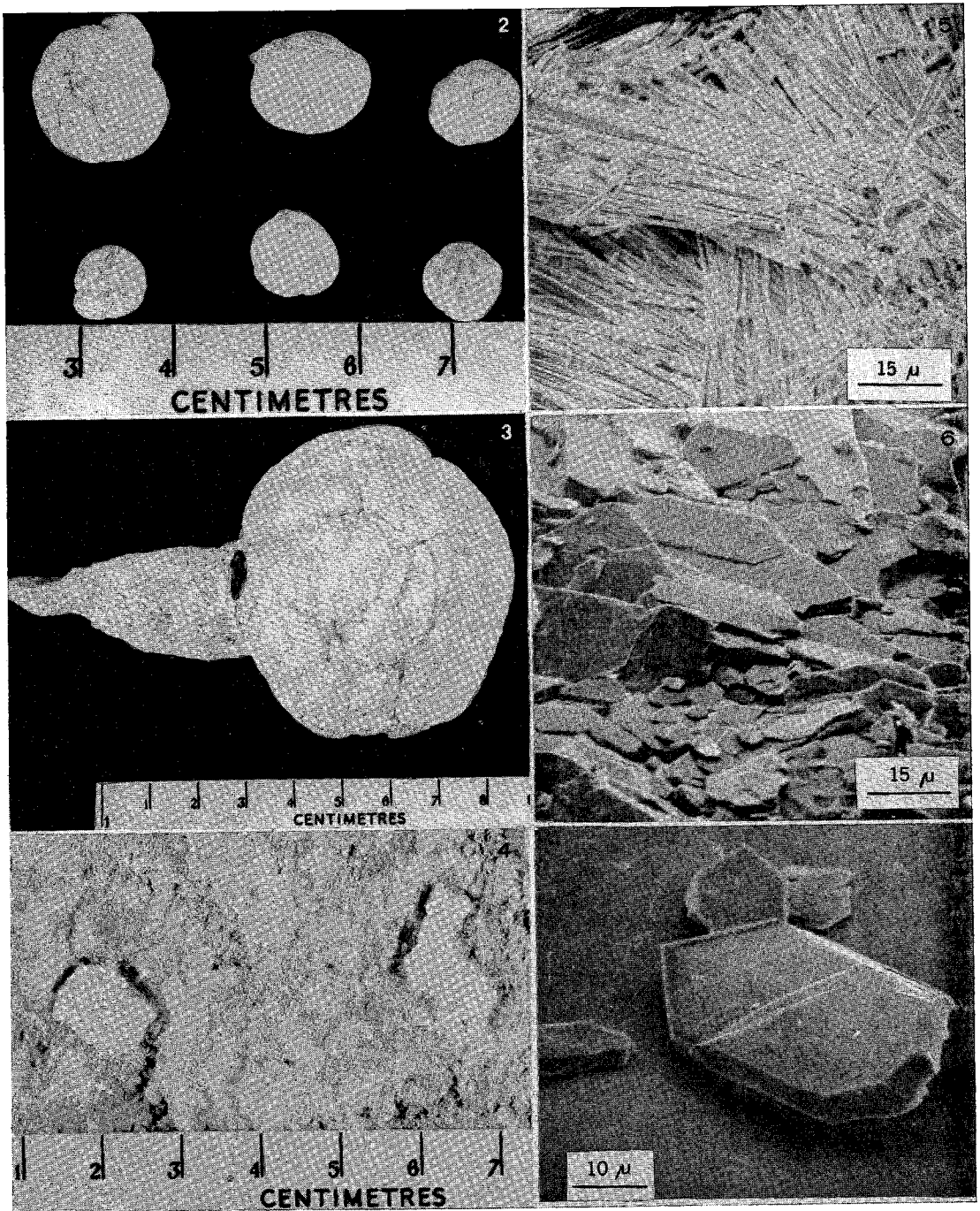
Small quantities of borate minerals have been found so far at two localities within the gypsum deposits in Newfoundland — at Fischell's

Brook and in the Flat Bay gypsum quarry. The rocks of the Anguille and Codroy Groups are folded here into an elongate anticline, the axis of which runs NE-SW, almost parallel to the coastline. A nearly continuous section of these beds is exposed in Fischell's Brook. The Anguille Group is here represented by about 200 ft. (61 m) of sandy conglomerate that grades towards the top of the group into a 10-foot (3 m) quartzitic sandstone. This is conformably overlain by a 60-foot (18 m) thick formation consisting of alternating limestone and shale, the Ship Cove Formation, which marks the base of the Codroy Group. The Ship Cove Formation is followed by an approximately 3000-foot (915 m) thick sequence of red, finely cross-bedded siltstone, sandstone, green shale and evaporites, overlain by a 1200-foot (366 m) sequence of alternating dark grey shale and carbonate; the carbonates consist of marine fossiliferous limestone, algal limestone, small algal reefs and red, dense, non-fossiliferous dolomite.

The evaporite horizons in the Fischell's Brook section attain a thickness of 150 ft. (46 m) and 200 ft. (61 m) respectively. Howlite was found in the higher of the two horizons, which crops out by the railway bridge on the southern bank of the stream (48°18'30"N, 58°40'30"W) in a vertical cliff. The outcrop consists of blue anhydrite at the cliff base and white gypsum on top; the anhydrite is bedded, with grey mud stringers marking the bedding.

The howlite appears as white nodules in the anhydrite; being somewhat more resistant to erosion than the anhydrite, the nodules are most conspicuous on weathered surfaces. Most of the nodules are from 0.5 cm to 1.5 cm in diameter and have a characteristic crenulated surface resembling a cauliflower; when clean, the surface has a vitreous to pearly luster (Fig. 2, 3). Although the nodules are small, they are fairly abundant at this locality — about one nodule per square foot of exposed surface (10-11 nodules per square meter). The white gypsum in the upper part of the cliff is inaccessible, and thus the total thickness of the howlite-bearing horizon is not known. So far, howlite is the only boron-containing mineral found at this locality.

The most abundant borate mineral in the Flat Bay gypsum quarry is ulexite, which forms irregular to oval masses 1-3 cm across, consisting of very fine felted fibers. The ulexite masses are somewhat crumbly and less resistant to weathering than the howlite nodules described above, but are conspicuous on light grey weathered surfaces by their pure white colour (Fig. 4); their main host rock is gypsum, but



FIGS. 2-7. (2) Howlite nodules from Fischell's Brook, southwestern Newfoundland; (3) howlite nodule from Dingwall, Nova Scotia (don. by E. R. W. Neale); (4) ulexite patches in weathered gypsum, Flat Bay quarry, S. W. Newfoundland; (5) scanning electron microscope image of ulexite, Flat Bay; (6) scanning electron microscope image of howlite, Fischell's Brook; (7) scanning electron microscope image of single crystal of howlite, Fischell's Brook.

some have been found also in the underlying anhydrite. The occurrence of ulexite is restricted to some parts of the quarry, suggesting that the mineral is concentrated within a relatively narrow stratigraphic horizon; but as the structure of the evaporite beds in this area, which may be very complex in detail, has not yet been worked out, no more precise statement can be made at this time.

No howlite has been observed by either author in the surface exposures of the Flat Bay gypsum quarry, but a short piece of a drill-core from the quarry, collected by C. Fong, contains a typical howlite nodule 1.5 cm in diameter, enclosed in anhydrite. The presence of the silicoborate in the Flat Bay area is thus established, but a more systematic search of both surface exposures and drill-core will be needed before the distribution of the borate minerals can be determined.

CRYSTAL HABIT OF HOWLITE AND ULEXITE

Several typical nodules of howlite and ulexite from various localities were examined by scanning electron microscope (Cambridge Scientific Instruments "Stereoscan" Mark 2A, operated by Dr. V. C. Barber, Department of Biology, Memorial University of Newfoundland).

The howlite nodules were found to consist of tightly-packed thin platy crystals. The size of the crystals ranges from 10 to about 50 microns. In the nodules from Fischell's Brook and Dingwall, the average size is about 30-40 microns and the largest crystal observed was 110 microns long; in dense porcelain-like nodules from California the crystals are smaller, about 10-20 microns on the average, but their habit and parallel packing are very similar to the Canadian specimens. The thickness of the plates ranges from 2 to about 6 microns (Fig. 6). One of the nodules from Fischell's Brook disaggregated in water to a fine white powder. Scanning electron microscope showed that this powder consisted of single crystals and small crystal aggregates of howlite (Fig. 7), closely resembling an illustration of a "typical crystal habit of howlite" presented by Murdoch (1957, p. 521); the platy habit of the mineral is the result of a shortening along the crystallographic *a*-axis and the corresponding development of a prominent (100) face (Murdoch 1957).

The "cottonballs" of ulexite consist of thin needle-like crystals, some with almost square cross-sections, others somewhat flattened. The crystals are about 1-3 microns thick and from 50 to 80 microns long (Fig. 5); they are arranged in loosely-packed, randomly-oriented

overlapping bundles. This simple habit of ulexite, with its characteristic square to rectangular cross-sections formed apparently by equal or almost equal development of two pinacoids in the vertical zone, differs somewhat from the highly modified crystals reported by Murdoch (1940, Figs. 3 to 5). The crystals appear to be terminated by simple pinacoidal planes, but these may be cleavage planes or fracture surfaces rather than true crystal terminations. Considerable difficulties were encountered in observing ulexite crystals at high magnifications, probably due to their transparency combined with a relatively loose packing.

X-RAY DIFFRACTION STUDY

The borate minerals from the Newfoundland localities were studied by standard methods on a Philips x-ray diffractometer, together with a specimen of howlite from Dingwall, Nova Scotia, and a specimen of porcelainous howlite from California, both kindly provided by G. R. Grantham, Curator of Geology of the Nova Scotia Museum in Halifax. Cell dimensions for the two minerals were calculated by R. G. Cawthorn of Memorial University from the diffractometer data, using a computer program of Appleman *et al.* (1972).

The x-ray study shows a close similarity between specimens of howlite from Fischell's Brook, Dingwall and California, and an equally good correspondence between the investigated howlite and ulexite and published x-ray data on both minerals (Murdoch 1940, 1957; Clark & Christ 1959; Finney *et al.* 1970; Stojanovic 1967; Traill 1969). The narrow range of variation can probably be ascribed to the purity of chemical composition of the borate minerals, with only a small range of substitution in the lattice.

A much greater discrepancy was noted in the relative intensities of the diffraction peaks of howlite from different localities (Table 1). This is particularly evident when our results and those given in several previous publications are compared to intensities calculated from structural factors (Smith *et al.* 1973). The anomaly consists mainly of the enhancement of the *h*00 peaks and a relative suppression of others. This appears to be related to the morphology of the crystallites with the prominent (100) face (Figs. 6, 7) and possibly to (100) cleavage. Although the anomalous variation in peak intensities may thus be caused simply by preferred orientation produced during the packing of diffractometer holders, it persisted in our patterns even after its cause had been identified

TABLE 1. VARIATION IN LINE INTENSITIES IN POWDER PATTERNS OF HOWLITE

$d(\text{\AA})$	avg.	(a)	(b)	(c)	(d)	(e)	(f)	(g)
12.4	54	52	33	40	60	28		
6.19	100	97	81	100	90	100	100	
4.36	*	*	*	30	*	19		
4.12	91	100	64	40	100	20		
3.59	34	16	44	80	18	95	90	
3.09	94	93	100	90	75	65	80	
3.01	23	*	25	30	16	37		
2.94	63	86	81	40	50	28		
2.90	16	*	*	-	*	29		40
2.78	17	*	*	*	*	25		
2.66	*	*	*	20	*	27		
2.47	37	49	38	-	50	*		
2.44	*	*	*	40	*	23		
2.16	*	23	*	*	10	*		
2.07	*	13	23	50	*	19		
2.06	35	44	36	-	50	*		
2.03	23	*	25	70	10	41		40
2.01	18	29	28	20	20	*		
2.00	*	20	*	-	-	*		
1.97	*	*	*	30	10	18		
1.92	*	14	31	30	*			
1.79	16	25	27	50	16			

- line absent * line present but not among the 15 strongest lines
 (a)Fischell's Brook, Newfoundland (this study); (b)Dingwall, Nova Scotia
 Scotia (this study); (c)"Califormita" porcelaneous howlite (this study);
 (d) Murodoch 1957, JCPDS Card 10-410; (e) Stojanovic 1967, JCPDS Card 18-
 618; (f)Smith *et al.* 1973; (g)Trat11 1969.

and an attempt had been made to correct it; even repeated careful grinding and repacking (by the back-filling method) did not succeed in bringing the observed intensities appreciably closer to the calculated values. Similar variation is evident in powder patterns published by others. Of the five strongest lines in each powder pattern, only two (6.19 and 3.09Å) are common to all seven patterns compared in Table 1. As this enhancement effect could have adverse influence on a routine identification of howlite by any method using a limited number of "strongest" lines, it should be kept in mind.

DISCUSSION

Although the presence of small quantities of borate minerals in the gypsum deposits of Nova Scotia has been known for a long time, it is interesting to note that no similar occurrences in other localities in North America or elsewhere have been reported in any major geological publication. (The authors would welcome any reference to published papers that they may have missed). Both ulexite and howlite are well-known constituents of the borate deposits of the western USA, but these deposits are part of a continental evaporite sequence in which the boron is believed to be of volcanic (fumarolic) origin (Foshag 1921; Schaller 1930). A little-known Yugoslav occurrence of a completely different type, described by Stojanovic (1967), is located near Raska on the river Ibar, about 150 km south of Belgrade. Fine-grained white howlite forms two lenses, 10 meters and 3 meters long and 20-30 cm thick, lying conformably in an interbedded sequence of volcanics, clastic sediments, carbonates and marls of Tertiary age; locally the mineral is found also in

the matrix of conglomerates, sandstones and tuffites of the same sequence. Stojanovic (1967, p. 40) believes that the howlite is of pneumatolytic origin.

The occurrence of howlite and ulexite in marine evaporites, particularly in anhydrite and gypsum deposits, thus appears to be restricted to the Carboniferous basin of the Atlantic provinces of Canada. The problem is whether the absence of borates from other gypsum deposits elsewhere is real, in which case special conditions should be sought for the Carboniferous evaporites of Atlantic Canada, or whether small quantities of borates are present in a similar setting at other localities but have been either overlooked or not reported in literature.

The nodular form and framboidal surface of the howlite suggest formation from a gel, probably in the form of coalescing globules. At Fischell's Brook, where the silicoborate nodules are found in relative abundance, the sulphate sequence is interbedded with shales and siltstones, where colloidal silica may be assumed to be freely available; in contrast, siliceous beds are lacking in the Flat Bay area where howlite is rare and the main borate minerals is ulexite.

The formation of composite globules in the colloidal state assumes lateral mobility of the gel. Although the fibrous ulexite probably crystallized directly from a solution, the concentration of ulexite fibers in small irregular patches again requires some mobility of the crystals. Thus we postulate that the formation of the borate minerals was contemporaneous with the precipitation of the sulphate. The question of which of the two Ca sulphates formed first does not seem relevant to this discussion.

Simple calculations based on the average composition of sea-water (SO₄ 0.2649%, H₃BO₃ 0.0026%; Mason 1966, p. 194) indicate that the normal amounts of boron in sea-water are more than sufficient to account for the occurrence of borates in the observed amounts in the marine evaporites of the Atlantic provinces. In view of this, and of the wide distribution of small quantities of borates throughout the Carboniferous basin, no special external source of the boron needs to be sought. This would suggest further that similar small quantities of borate minerals ought to be present in anhydrite and gypsum deposits at other localities outside the Atlantic provinces. If a systematic search of such deposits shows that borates are truly absent, the difference would have to be explained by variation in other critical factors such as pH, temperature, solubility, and rate of evaporation. Clearly, further work will be needed to solve the problem.

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