Canadian Mineralogist
Vol. 21, pp. 503-508 (1983)

WEDDELLITE FROM BIGGS, OREGON, U.S.A.

J. A. MANDARINO

Department of Mineralogy and Geology, Royal Ontario Museum, 100 Queen's Park, Toronto, Ontario M5S 2C6

and

Department of Geology, University of Toronto, Toronto, Ontario M5S 1A1

NOBLE V. WITT

1319 N.E. 117th Street, Vancouver, Washington 98665, U.S.A.

ABSTRACT

The rare mineral weddellite, $CaC_2O_4{\cdot}(2+x)H_2O$, has been identified from an occurrence near Biggs, Oregon, U.S.A. Crystals up to 5 × 5 × 40 mm occur in cavities in nodules of the so-called "Biggs jasper". The nodules are in lake-bottom sediments sandwiched between basalt flows of Miocene age. Associated minerals are quartz and whewellite ($CaC_2O_4{\cdot}H_2O$). The whewellite appears to replace some of the weddellite. The weddellite occurs as tan, euhedral crystals and as white, fibrous aggregates surrounding some of the euhedral crystals. The euhedral crystals are dull to vitreous in lustre and often have a dark brown, resinous core containing an undetermined organic material; the white material has a silky lustre. The hardness is about 4 and the density is 2.02(2) g/cm^3 for the tan outer shell of the euhedral crystals. Weddellite is tetragonal, space group *I*4/*m*, *a* 12.33(2), *c* 7.353(3) Å, *V* 1117.9 Å^3, *Z* = 8. Analysis of the white material gave CaO 35.4, C_2O_3 43.2, H_2O 24.7, total 103.3 wt. %. The empirical formula derived from these data is $Ca_{1.04}C_{1.97}O_{4.00}{\cdot}2.26H_2O$ or, ideally, $CaC_2O_4{\cdot}2.26H_2O$; the calculated density is 2.020(4) g/cm^3. Optically, the white material is uniaxial (+), with ω = 1.524 and ϵ = 1.544. The compatibility index is 0.008, indicating *superior* compatibility of the density, optical and chemical data.

Keywords: weddellite, Oregon, oxalate, calcium, whewellite.

SOMMAIRE

Le minéral rare weddellite, $CaC_2O_4{\cdot}(2+x)H_2O$, a été identifié dans un gîte près de Biggs, en Orégon, É-U. Il se présente en cristaux pouvant atteindre 5 × 5 × 40 mm dans des cavités de nodules du soi-disant "jaspe de Biggs". Les nodules se trouvent dans des sédiments lacustres intercalés entre des coulées basaltiques d'âge miocène. Les minéraux associés sont le quartz et la whewellite $CaC_2O_4{\cdot}H_2O$. La whewellite semble remplacer une partie de la weddellite. Celle-ci se présente en cristaux idiomorphes brun pâle et en agrégats fibreux blancs entourant certains cristaux idiomorphes. Les fibres ont un éclat soyeux; les cristaux ont un éclat mat à vitreux et montrent souvent un coeur résineux brun foncé renfermant un matériau organique indéterminé. Le matériau brun pâle possède une dureté d'environ 4 et une densité de 2.02(2). La weddellite est tétragonale, de groupe spatial *I*4/*m*, *a* 12.33(2), *c* 7.353(3) Å, *V* 1117.9 Å^3, *Z* = 8. L'analyse chimique du matériau blanc donne CaO 35.4, C_2O_3 43.2, H_2O 24.7, somme 103.3% en poids. La formule empirique obtenue à partir de ces données est $Ca_{1.04}C_{1.97}O_{4.00}{\cdot}2.26H_2O$ ou, idéalement, $CaC_2O_4{\cdot}2.26H_2O$; la densité calculée est 2.020(4). Le matériau blanc est optiquement uniaxe (+), d'indices principaux ω 1.524 et ϵ 1.544. L'indice de compatibilité, 0.008, indique une compatibilité *supérieure* de la densité avec données optiques et chimiques.

(Traduit par la Rédaction)

Mots-clés: weddellite, Orégon, oxalate, calcium, whewellite.

INTRODUCTION

Although the substance now called *weddellite* has been known for many years, its natural occurrence was restricted to plants and animals. There has been much confusion in the literature as to the chemical composition of the compound. For many years, it was considered to be calcium oxalate trihydrate, $CaC_2O_4{\cdot}3H_2O$. It was not until Bannister & Hey (1936) identified the compound from the Weddell Sea, near Antarctica, that many of the mysteries surrounding this material were solved. The material that Bannister & Hey described was found as transparent, colorless, tetragonal bipyramidal crystals in bottom sediments from the central part of the Weddell Sea. The samples in which they occur were dredged from depths between 4434 and 5008 metres. The extremely sharp crystals range in size from 0.2 × 0.1 to 0.3 × 0.15 mm. They are uniaxial (+), with ω = 1.523 and a birefringence of about 0.02. The density could not be measured because of the small size of the crystals. A single-crystal X-ray study showed that they are tetragonal, space group *I*4/*m*, *a* 12.40, *c* 7.37 Å, *c*:*a* = 0.594:1. The only form noted is the tetragonal bipyramid {011}. Although there was not sufficient material for a complete chemical analysis, Bannister & Hey found that the mineral gave an X-ray powder-diffraction pattern identical to that of some of the calcium oxalate crystals present in human renal calculi. A study of the latter crystals gave more complete data concerning the compound. Its hardness is about 4, and the crystals are uniaxial (+), ω 1.523, ϵ 1.544. The measured density is 1.99 g/cm^3; after a correction introduced to account for 4% "dahlite" (carbonate-hydroxylapatite), the true density is found to be 1.94 g/cm^3. Two chemical analyses were carried out and gave practically the same results, and the formula was established as $CaC_2O_4 \cdot 2H_2O$. Because Bannister & Hey felt that there was still some doubt about the water content in the Weddell Sea crystals, they decided not to name the mineral. The sharpness of the crystals and the absence of any signs of abrasion indicated that they are authigenic.

In a discussion of the composition of human urinary calculi, in particular those made up of carbonate-hydroxylapatite and hydroxylapatite, Frondel & Prien (1942) used the name *weddellite* for the first time for $CaC_2O_4 \cdot 2H_2O$, but gave no data. Prien & Frondel (1947) noted that the compound was very abundant in human urinary calculi. The material also is produced pathologically by other mammals, such as the hippopotamus, as cited by Bannister *et al.* (1947), and dogs, as noted by Milton & Axelrod (1951). The first nonpathological occurrence of the compound in an animal was reported by Lowenstam (1968), who found that the gizzard plates of the deepwater gastropod, *Scaphander cylindrellus,* consist of weddellite. Traill (1970) reported weddellite as colorless crystals in sponge spicules from Fisher Strait, Hudson Bay area, N.W.T.

The compound is common in some plants and was observed in the common onion, *Allium cepa,* by Honneger (1952). Knobloch & Kalina (1968) identified the compound in the plant *Spyrogyra.* The extensive association of weddellite and other oxalates with lichens and fungi is summarized by Mandarino (1983).

A second occurrence of weddellite in ocean-bottom sediments was described by Hutton & Taft (1965). Their samples were dredged from a depth of about one fathom (about 2 metres) off the southwestern coast of Florida. Hutton & Taft cast doubt on the authigenic nature of their material as well as that from the Weddell Sea because they felt that the oxalate could have formed in both sets of samples during the long period of storage between the dates of collection and examination. During his study of the deepwater gastropod mentioned earlier, Lowenstam (1968) also examined a sample of bottom sediment dredged from a depth of 4196 metres in the Weddell Sea. In this sample, which had been preserved in a 70% alcohol solution, were some small tetragonal bipyramids that are uniaxial (+), with ω = 1.5237 and ϵ = 1.5434. They gave an X-ray powder-diffraction pattern identical to that of weddellite. Thus, the original authigenic nature of the Weddell Sea crystals was confirmed. Further confirmation is given by Marlowe (1970), who identified weddellite in bottom sediments from the St. Lawrence and Saguenay rivers in Quebec. His samples were preserved in alcohol, so that the weddellite could not have formed through the decomposition of organic material in the samples. It is interesting to note that the original Weddell Sea samples were preserved in alcohol at the time that they were collected. Unfortunately, during the 30-year storage before they were studied by Bannister & Hey (1936), some of the samples had dried up because of defective corks. It is not known whether the samples in which weddellite crystals were found were still preserved in alcohol or were some of those that had lost the fluid.

In addition to its occurrence in animals and plants and in bottom sediments, weddellite has been reported from older rocks. Slovenec & Sinkovec (1973, 1974) identified very fine-grained whewellite and weddellite among the insoluble residues of an Upper Cretaceous limestone in Yugoslavia. Weddellite and whewellite have been found on Cretaceous ophiolitic rocks in Italy by Tirelli (1977). Mandarino (1983) noted the presence of weddellite on calcium-rich rocks of Precambrian age in Ontario. The occurrences noted by both Tirelli (1977) and Mandarino (1983) probably are related to oxalic acid produced by lichens. Decaying vegetable fibres probably are the cause of the weddellite and whewellite present in a Chinese wall painting at the Royal Ontario Museum (pers. comm., the Canadian Conservation Institute, National Museums of Canada).

In September 1979, a specimen of an unidentified mineral from Biggs, Oregon, U.S.A., was submitted for identification to the senior author by the junior author. An X-ray powder-diffraction pattern of the mineral proved to be the same as that of weddellite. Because the weddellite in all the previously described occurrences is very fine-grained, with crystals rarely reaching an overall size of ¼ mm, the specimens from Oregon provided an opportunity to describe what are now the largest crystals of weddellite known.

APPEARANCE AND PHYSICAL PROPERTIES

The Biggs weddellite occurs as large, euhedral crystals (up to 5 × 5 × 40 mm) in cavities in the so-called "Biggs jasper". The euhedral crystals are tan and have a dull to vitreous lustre. They commonly have a dark brown, resinous core that contains an unidentified organic material. Some of the euhedral crystals are coated with a secondary overgrowth of white, fibrous weddellite that has a silky lustre. The mineral is translucent to transparent, has a white streak and fluoresces yellow under long-wave ultraviolet radiation. The Biggs mineral breaks with a conchoidal fracture and, unlike other reported crystals of weddellite, has a good {010} cleavage. Because of this good cleavage, the mineral breaks into fibres parallel to [001]. The hardness is about 4. The density of the tan outer shell of one of the crystals, determined with the Berman balance, is 2.02(2) g/cm^3. The density calculated from the unit-cell parameters and the chemical data, following the method outlined by Mandarino (1981b), is 2.020(4) g/cm^3. The Biggs weddellite is optically uniaxial (+), with ω 1.524(2) and ϵ 1.544(2).

CRYSTALLOGRAPHY

The weddellite crystals from the Weddell Sea were reported by Bannister & Hey (1936) to display only the tetragonal bipyramid {011}. Prien & Frondel (1947) observed no additional forms among several hundred urinary calculi. The Biggs weddellite crystals, however, are primarily prismatic in habit and show the forms {010}, {011} and {001}. The prismatic nature of the Biggs material (Figs. 1–3) casts doubt on the statement given by Palache *et al.* (1951) that the tetragonal crystals of prismatic habit described by Nakano (1923) and by Hammarsten

FIG. 1. Microcrystals of weddellite showing {010} and {011}. Length of larger crystal about 5 mm. ROM specimen M38916.

FIG. 2. Large crystal (about 20 mm long) of weddellite. ROM specimen M38917.

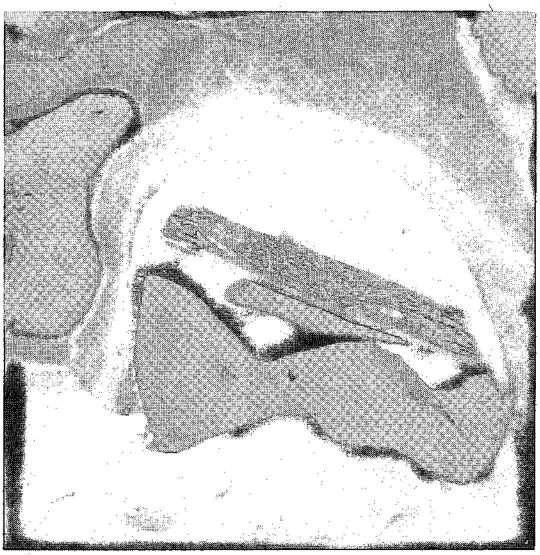

FIG. 3. Large crystal (about 30 mm long) of weddellite. ROM specimen M38918.

(1929) may represent another hydrate. In fact, the data presented by these two investigators tend to confirm that they were dealing with synthetic weddellite. In his study of synthetic tetragonal calcium oxalate, Nakano (1923) admittedly had insufficient material to determine the amount of water in his crystals; he was under the impression that he was dealing with calcium oxalate trihydrate. Because of the crystallographic data that he presented, however, it is clear that he was dealing with the dihydrate, synthetic weddellite. He identified the forms {111}, {110}, {100}, {012}, {201} and {403}. Based on the angle between {110} and {111}, he calculated a *c:a* ratio of 1.1316. This is approximately twice the axial ratio of weddellite calculated from the unit-cell parameters determined by various workers (the average of nine determinations is 0.5960 or 1.1920/2). Using the smaller axial ratio and following the convention $k>h$, Nakano's forms become: {221}, {110}, {010}, {011}, {041} and {083}. Nakano (1923) noted that all but the first two forms are rare.

The exhaustive work by Hammarsten (1929) was concerned mainly with determining the conditions necessary for the formation of calcium oxalates. She synthesized three calcium oxalates: the mono-, di- and trihydrates. Although she carried out no rigorous crystallographic studies, some of her excellent drawings of the dihydrate make it clear that she synthesized weddellite. It was possible to measure the ρ angle for a pyramidal face from one of these drawings. The measured angle of 40° compares closely with the angle 40.15° calculated for the form {111}. Several different habits of synthetic weddellite were produced by Hammarsten. Some of these are pseudo-octahedra and others are long prisms terminated by bipyramidal faces. Both the first- and second-order prisms are present as well as the first- and second-order bipyramids. Some of her drawings indicate the absence of any symmetry planes parallel to [001]. From the foregoing observations, it appears that the following forms were developed on Hammarsten's weddellite crystals: {110}, {111}, {010}, {011}, {001} and an unidentified {*hkl*}.

TABLE 1. MORPHOLOGICAL DATA FOR BIGGS WEDDELLITE

Tetragonal bipyramidal 4/m
$a:c$ = 1 : 0.5964 $p_0:r_0$ = 0.5964 : 1

Forms	ϕ	ρ	A	$\overline{M}$
* {001}	----	0.00°	90.00°	90.00°
* {010}	0.00°	90.00	90.00	45.00
{110}	45.00	90.00	45.00	90.00
{011}	0.00	30.81	90.00	68.77
{083}	0.00	57.84	90.00	53.23
{041}	0.00	67.26	90.00	49.30
{111}	45.00	40.15	62.88	90.00
{221}	45.00	59.34	52.54	90.00

Note. Only the forms marked with an asterisk (*) have been observed on Biggs weddellite. The other forms have been observed on crystals of synthetic weddellite. All angles have been calculated using the unit-cell parameters of the Biggs weddellite.

The calculated angles and other morphological data for the Biggs weddellite are given in Table 1. In order to assist future workers to identify forms on weddellite crystals, angles are given for forms noted on synthetic crystals also.

X-ray powder-diffraction patterns were prepared from the white, fibrous material and from the tan, euhedral crystals. The patterns were produced in a Debye–Scherrer camera of diameter 114.6 mm using Cu*Kα* radiation and were corrected for shrinkage. The powder data are not given here because they do not differ significantly from the data published by other workers. The unit-cell parameters refined from the powder-diffraction data for the white, fibrous Biggs weddellite are given in Table 2, where they are compared with the values obtained from other studies. In addition to values of *a* and *c*, the unit-cell volumes and axial ratios calculated from them are also given in Table 2.

CHEMICAL COMPOSITION

A thermogravimetric analysis (TGA) (see next section) of 7 mg of the white, fibrous weddellite provided quantitative data for the H_2O and C_2O_3 contents. Another sample weighing about 15 mg was analyzed for calcium by neutron-activation analysis at the SLOWPOKE facility, University of Toronto. The results of the analyses are given in Table 3. The empirical formula derived from these data is:

TABLE 2. UNIT-CELL PARAMETERS REPORTED FOR WEDDELLITE

a	c	V	$c:a$	Reference
12.33 (2)	7.353 (3)	1117.9	0.5964	TS
12.371 (3)	7.357 (2)	1125.9	0.5947	T&D
12.352 (14)	7.350 (2)	1121.4	0.5950	T
12.34	7.37	1122.3	0.5972	S&S
12.360 (5)	7.340 (5)	1121.3	0.5939	H&T
12.30 (2)	7.34 (2)	1110.5	0.5967	S
12.302 (7)	7.381 (3)	1117.0	0.6000	H
12.375	7.377	1129.7	0.5961	K
12.40 (2)	7.37 (2)	1133.2	0.5944	B&H

Notes: TS This study, T&D Tazzoli & Domeneghetti (1980), T Tirelli (1977), S&S Slovenec & Sinkovec (1973), H&T Hutton & Taft (1965), S Sterling (1965), H Honegger (1952), K Klasens *et al.* (1937), B&H Bannister & Hey (1936). Unit-cell parameters were used to calculate the values of $c:a$.

TABLE 3. CHEMICAL COMPOSITION OF BIGGS WEDDELLITE

	wt.%	mol. ratio	anionic ratio	cationic ratio	cell contents
CaO (1)	35.4	0.6312	0.6312	0.6312	Ca 1.039
C_2O_3 (2)	43.2	0.5998	1.7995	1.1997	C 1.974
H_2O (2)	24.7	1.3707	1.3707	2.7414	H 4.511
total	103.3		3.8014		O 6.256

Notes: (1) Neutron activation by Dr. R. G. V. Hancock. (2) TGA by Mr. R. Ramik. Molecular weight: 170.00 . Cell contents calculated on the basis of four oxygen ions in the "anhydrous" portion.

$Ca_{1.04}C_{1.97}O_{4.00}\cdot 2.26H_2O$ or, ideally, $CaC_2O_4\cdot 2.26H_2O$. Tazzoli & Domeneghetti (1980) confirmed the finding of Sterling (1964, 1965) that water cannot exceed 2.5 mols per formula unit and that water in excess of two mols is zeolitic.

A partial analysis by TGA was carried out on a 5-mg sample of the resinous, dark core of one of the larger crystals. Only C_2O_3 (46.7%) and H_2O (23.8%) were determined. The molecular ratio $H_2O : C_2O_3$ is 2.04 compared to 2.29 for the white, fibrous material. This is much closer to the theoretical value of 2, but the C_2O_3 figure probably is too high because of the presence of an unknown hydrocarbon; a lower value for C_2O_3 would give a higher $H_2O : C_2O_3$ ratio.

THERMAL ANALYSIS

A thermogravimetric analysis was carried out on 6.888 mg of the white, fibrous material in a Mettler Thermoanalyzer equipped with a high-vacuum system and a quadrupole mass-spectrometer. After a 50-hour static period in the high vacuum at room temperature, the sample was heated at 2°/min. to 875° and then at 8°/min. to 1050°C. All heating was done in high vacuum (10^{-7}torr residual atmosphere). The following weight losses occurred:

22.4 wt.% at 22°C in the high vacuum with no heating,
2.3 wt.% between 22° and 210°C,
17.0 wt.% between 210° and 515°C,
25.2 wt.% between 515° and 1000°C, and
1.0 wt.% after heating the residue to 1050°C in air.

The first weight-loss represents the bulk of the H_2O; because it was evolved at room temperature in a high vacuum, it probably is very loosely bound. This 22.4 wt.% of H_2O represents 2.046 molecules per formula. The second weight-loss also consists of H_2O, as monitored by the mass spectrometer. This amounts to 0.210 molecules of H_2O per formula. The third weight-loss consists of CO with minor CO_2 and the fourth weight-loss involved mainly CO_2 with minor CO. The final weight-loss is assumed to be loss of elemental carbon and is included with the total C_2O_3 value. An X-ray powder-diffraction pattern shows that the air-heated residue consists almost entirely of well-crystallized CaO plus some poorly crystallized $Ca(OH)_2$.

It is interesting to note that the water lost at room temperature under high vacuum amounts to about two mols of H_2O, whereas that lost at the higher temperatures amounts to about ¼ mol. These figures are in direct contradiction to the findings of Sterling (1964, 1965) and Tazzoli & Domeneghetti (1980), who referred to the two water molecules as "non-zeolitic" and the rest as "zeolitic".

OCCURRENCE AND ASSOCIATED MINERALS

The weddellite occurs in nodules of jasper found in lake-bottom sediments associated with some of the most recent (Miocene) flows of the Columbia River Basalt Plateau. The locality is near Biggs, Oregon, in the west-central part of the plateau. A vertical section exposed at the locality indicates that the particular bottom-sediment in which the jasper occurs has been disturbed by a basalt flow that invaded the lake. The leading edge of this flow exhibits the typical features of a pillow lava. A later flow covered the bottom sediments. Fish scales and bones were found in several of the jasper nodules. These and other organic material in the sediments probably provided the calcium and the oxalic acid necessary for the formation of the weddellite.

Some of the crystals of "weddellite" gave X-ray powder-diffraction patterns identical to that of whewellite, $CaC_2O_4\cdot H_2O$. In these particular cases, the whewellite has replaced the weddellite pseudomorphously. Some small, euhedral crystals were observed in a few specimens; these may represent primary whewellite. A grey, vermiform mineral was proved, by X-ray diffraction, to be quartz. A thin section of the jasper showed that this host material for the weddellite, whewellite and quartz is made up of very small grains of quartz. An X-ray-fluorescence scan of this material showed only major silicon with a trace of iron.

CONCLUSIONS

This study, in addition to presenting full data for large weddellite crystals from Biggs, Oregon, also reviews the other occurrences of the mineral. It is clear that several earlier workers synthesized the mineral, although there has been much confusion in the literature about the identities of these synthetic products.

The results of thermogravimetric analysis shows that most of the water (2 mols) is very loosely bound and is evolved at 22°C when subjected to a high vacuum. The remaining ¼ mol is given off between 22° and 210°C. It is difficult to reconcile these results with previous crystal-structure studies.

Application of the Gladstone–Dale equations and the compatibility concept as defined by Mandarino (1979, 1981a) results in a compatibility index of 0.008 for the Biggs weddellite. Thus, the compatibility of the mean refractive index, density and chemical data is considered *superior*.

ACKNOWLEDGEMENTS

The authors are indebted to several people who assisted in this study. Mr. Blaine A. Schmeer of Tulatin, Oregon, brought the first weddellite crystal to the attention of N. V. Witt. Mr. Fred Dormaier allowed Mr. Witt to collect the material that formed the basis of this study. Mr. R. A. Ramik, of the Royal Ontario Museum, carried out the thermal analyses. He and Mrs. C. Peat prepared the X-ray patterns. Dr. R. G. V. Hancock performed the neutron-activation analysis at the SLOWPOKE facility, University of Toronto. Mrs. Violet Anderson took the photograph of the microcrystal shown in Figure 1. The other photographs were taken by Mr. B. Boyle of the ROM Photography Department. Messrs. R. Falls and L. Groat tracked down many of the references. This study was financially assisted, in part, by a grant (#A7998) from the Natural Sciences and Engineering Research Council to the senior author.

REFERENCES

BANNISTER, F. A. & HEY, M. H. (1936): Report on some crystalline components of the Weddell Sea deposits. *Discovery Reports* **13**, 60-69.

______, ______ & OAKLEY, K. P. (1947): Identification of a calculus from a hippopotamus. *Nature* **160**, 470.

FRONDEL, C. & PRIEN, E. L. (1942): Carbonate–apatite and hydroxyl-apatite in urinary calculi. *Science* **95**, 431.

HAMMARSTEN, G. (1929): On calcium oxalate and its solubility in the presence of inorganic salts with special reference to the occurrence of oxaluria. *C. r. trav. lab. Carlsberg* **17** (11), 1-85.

HONEGGER, R. (1952): The polyhydrates of calcium oxalate. *Viertelsjahrsschr. naturforsch. Ges. Zürich Beih.* **97**, 1-44.

HUTTON, C. O. & TAFT, W. H. (1965): Weddellite in modern sediments, Florida. *Mineral. Mag.* **34**, 256-265.

KLASENS, H. A., PERDOK, W. G. & TERPSTRA, P. (1937): Crystallography of strontium oxalate. *Z. Krist.* **96**, 227-230.

KNOBLOCH, K. & KALINA, T. (1968): Kreuzformige Oxalat-kristalle in *Spyrogyra. Ber. Deutsch. Bot. Ges.* **81**, 286-288.

LOWENSTAM, H. A. (1968): Weddellite in a marine gastropod and in Antarctic sediments. *Science* **162**, 1129-1130.

MANDARINO, J. A. (1979): The Gladstone–Dale relationship. III. Some general applications. *Can. Mineral.* **17**, 71-76.

______ (1981a): The Gladstone–Dale relationship. IV. The compatibility concept and its application. *Can. Mineral.* **19**, 441-450.

______ (1981b): Comments on the calculation of the density of minerals. *Can. Mineral.* **19**, 531-534.

______ (1983): Weddellite from Lutterworth Township, Haliburton County, Ontario. *Can. Mineral.* **21**, 509-511.

MARLOWE, J. I. (1970): Weddellite in bottom sediment from the St. Lawrence and Saguenay rivers. *J. Sed. Petrology* **40**, 499-506.

MILTON, C. & AXELROD, J. M. (1951): Calculi and other stones found in mammals. *J. Mammal.* **32**, 139-154.

NAKANO, H. (1923): Beitrage zur Kenntnis der in den Harnsteinen enthaltenen Substanzen. *J. Biochem.* **2**, 437-441.

PALACHE, C., BERMAN, H. & FRONDEL, C. (1951): *The System Of Mineralogy (7th ed.).* J. Wiley & Sons, New York.

PRIEN, E. L. & FRONDEL, C. (1947): Studies in urolithiasis. I. The composition of urinary calculi. *J. Urology* **57**, 949-991.

SLOVENEC, D. & SINKOVEC, B. (1973): Whewellite and weddellite from the Upper Cretaceous limestone near Zminj in Istra. *Bull. Sci., Cons. Acad. Sci. Arts RSF Yougoslavie* **18A**, 228-229.

______, & ______ (1974): Whewellite ($CaC_2O_4 \cdot H_2O$) and weddellite ($CaC_2O_4 \cdot 2H_2O$) from the Upper Cretaceous limestone near Zminj in Istra. *Geol. Vjesn.* **27**, 209-216.

STERLING, C. (1964): Crystal structure of weddellite. *Science* **146**, 518-519.

______ (1965): Crystal structure analysis of weddellite, $CaC_2O_4 \cdot (2+x)H_2O$. *Acta Cryst.* **18**, 917-921.

TAZZOLI, V. & DOMENEGHETTI, C. (1980): The crystal structures of whewellite and weddellite: re-examination and comparison. *Amer. Mineral.* **65**, 327-334.

TIRELLI, G. (1977): Weddellite e whewellite dell' Appennino modenese. *Mineral. Petrog. Acta* **21**, 93-100.

TRAILL, R. J. (1970): A catalogue of Canadian minerals. *Geol. Surv. Can. Pap.* **69-45**.

Received September 8, 1982, revised manuscript accepted February 3, 1983.