

Gottardiite, a new high-silica zeolite from Antarctica: the natural counterpart of synthetic NU-87

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Abstract: Gottardiite has been discovered in the Jurassic Ferrar dolerites of Mt. Adamson (Northern Victoria Land, Antarctica). The new zeolite occurs as subparallel aggregates of transparent pseudo-hexagonal lamellae. The mineral is orthorhombic, space group *Cmca*, with $a = 13.698(2)$, $b = 25.213(3)$ and $c = 22.660(2)$ Å.

The strongest X-ray diffraction lines are (in Å): 11.34(100), 4.37(79), 4.01(57), 3.282(68). The gottardiite framework topology is the same as that of synthetic NU-87. The chemical formula is: $\text{Na}_{2.5}\text{K}_{0.2}\text{Mg}_{3.1}\text{Ca}_{4.8}\text{Al}_{18.8}\text{Si}_{117.2}\text{O}_{272} \cdot 93\text{H}_2\text{O}$. The Si/Al ratio is 6.2, the highest found up to now in a natural zeolite. Gottardiite is optically biaxial (-), with $\alpha = 1.480(2)$, $\beta = 1.485(2)$, $\gamma = 1.486(2)$, $2V < 60^\circ$, where $X = b$, $Y = a$ and $Z = c$. The density is $2.14(4) \text{ g/cm}^3$ (obs.), and 2.16 g/cm^3 (calc.). Both thermal stability and rehydration capacity are very high. The name is in honour of Glauco Gottardi (1928–1988).

Key-words: Gottardiite, zeolite, Antarctica, chemical and physical properties, powder diffraction.

Introduction

In the course of a systematic investigation of zeolites from Northern Victoria Land (Antarctica) (Vezzalini *et al.*, 1994; Bigi *et al.*, 1994) a new natural zeolite has been discovered in the Jurassic Ferrar dolerites of Mt. Adamson.

The rock samples were collected along the ridge of Mt. Adamson by Claudio Ghezzi, Roberto Lanza and Antonio Rossi during field work carried out in the austral summer 1990/91, as part of the Italian Antarctic Research Program (PNRA). The rocks are highly vesicular and contain several zeolitic species, some of which, tschernichite and boggsite, very rare (Galli *et al.*, 1995).

The authors dedicate this zeolite to their mentor and guide, Glauco Gottardi (1928–1988), in recognition of his pioneering work into the structure and crystal chemistry of natural zeolites. The name Gottardiite is a fitting tribute to the memory of a great teacher and is a token of the esteem in which he was held by all who followed in his footsteps. Both the mineral and its name have

been approved by the Commission on New Minerals and Mineral Names of the IMA (Ref. 94-054); a type specimen has been deposited at the British Museum of Natural History, London, with accession number BM 1994,100.

Occurrence

Victoria Land in Antarctica was the site of very extensive mesozoic-to-recent magmatic activity. The area (Carmignani *et al.*, 1987) is characterized by a crystalline basement (Paleozoic granitoids and Precambrian metamorphic rocks) capped by thin layers of continental sediments (the upper part of the late Paleozoic-Early Triassic Beacon Supergroup) overlaid and intruded by Jurassic Tholeiites as thick lava flows (Kirkpatrick basalts) or sills (Ferrar dolerites).

At Mt. Adamson, in Northern Victoria Land, the crystalline basement is covered by a 35 m thick layer of sandstone (Beacon sediments), capped by about 450 m of basaltic rocks (Ferrar

Table 1. Chemical analyses (wt.%) of host rock and smectite.

	Host rock*	Smectite**
SiO ₂	51.77	43.74
TiO ₂	0.70	n.d.
Al ₂ O ₃	17.56	12.65
Fe ₂ O ₃	9.95	13.58
MnO	0.16	n.d.
MgO	5.74	3.13
CaO	10.79	0.96
BaO	tr.	tr.
SrO	tr.	0.03
Na ₂ O	1.94	0.14
K ₂ O	0.52	0.11
P ₂ O ₅	0.09	n.d.
H ₂ O	0.78	25.66
CO ₂	tr.	n.d.
Total	100.00	100.00

* X-ray fluorescence analysis; H₂O from ignition loss.

** Electron microprobe analysis; H₂O content from thermogravimetric analysis.

Chemical formula on the basis of O₂₀(OH)₄ p.f.u.:

^[4] [Si_{7.34}Al_{0.66}] $\Sigma = 8.0$ ^[6] [Al_{1.84} Fe_{1.72} Mg_{0.78}] $\Sigma = 4.34$

^[12] [Ca_{0.17} Na_{0.05} K_{0.02}] $\Sigma = 0.24$ O₂₀ (OH)₄ · 12.36 H₂O

dolerites). About ten layers of sandstone of different thickness are recognizable in these basalts. The igneous rocks occurring along the ridge show textures varying from doleritic or subophitic to medium-grained ophitic. The phenocrysts are idiomorphic Ca-rich plagioclase, normally zoned and twinned, allotriomorphic clinopyroxene, often zoned and twinned, and occasional crystals of olivine, sometimes embedded in a thin layer of hiddingsite. The groundmass consists of microcrysts of Ca-plagioclase, clinopyroxene, relict olivine, traces of vitreous material and opaque minerals (Fe-Ti oxides) as microlites. X-ray fluorescence analysis of the host rock was carried out by wavelength dispersive X-ray fluorescence (Philips PW 1480) on pressed pellets, using the method of Franzini *et al.* (1975). The X-ray fluorescence data (Table 1) lie in the field of the basaltic andesites of the T.A.S. diagram (Le Bas *et al.*, 1986).

The alteration of the rock, the number and size of vesicles and cavities, and the abundance of zeolites vary in the samples collected. The walls of the vesicles and cavities are always covered by a thin layer of highly-disordered Fe-smectite (Table 1), which is crumbly and varies in colour

from pale yellow to dark orange. The growth of this mineral precedes the widespread zeolite crystallization in this area; chemical data reported in Table 1 do not suggest any clear relationship between rock and smectite.

Many zeolitic species were found, namely, in order of abundance, mordenite, heulandite, erionite, phillipsite, stilbite, levyne, epistilbite, tschermichite, boggsite, and cowlesite (Vezzalini *et al.*, 1994; Galli *et al.*, 1995). The other neogenic minerals present in the cavities are quartz, cristobalite, apophyllite, gypsum, and very occasionally, calcite.

Many geodes are filled, partially or completely, by only one zeolitic species; in the other cases, the most common associations, in paragenetic sequence, are mordenite-heulandite, erionite-phillipsite, erionite-heulandite, chabazite-mordenite and levyne-erionite. In many geodes, the zeolites are covered by minute milky-white spheres of cristobalite, which are completely empty. Prismatic plagioclase crystals, ranging in composition from Ab32 to Ab45, are often wedged between the zeolites which fill the geodes.

Gottardiite was found in cavities and small fractures in only one of the Ferrar dolerites samples collected. The specimen was from the SW crest, just under the summit of Mt. Adamson (lat. 73°56' S, long. 162°56' E).

Appearance and physical properties

Gottardiite occurs as thin lamellae, pseudo-hexagonal in shape or elongated along [100]. The crystals rarely occur isolated; more frequently they form subparallel (001) or wedge-shaped aggregates of a few individual crystals (Fig. 1a, b). They are transparent and colourless, or at most light-straw coloured. In only one geode did we find slightly milky crystals, which assume a bright iridescent appearance under oblique light.

The maximum crystal sizes are 0.3 × 0.2 × 0.02 mm. The observed forms are: {001} major and {0-10}, {1-40} and {110} minor (Fig. 2). No doubly well-terminated crystals were found. Both the isolated lamellae and the aggregates occur as irregular intergrowths leaving voids between them, where minute, spherical, spongy aggregates of straw-coloured clay material can be found (Fig. 1b).

No fluorescent effects were observed under either long- or short-wavelength ultraviolet radia-

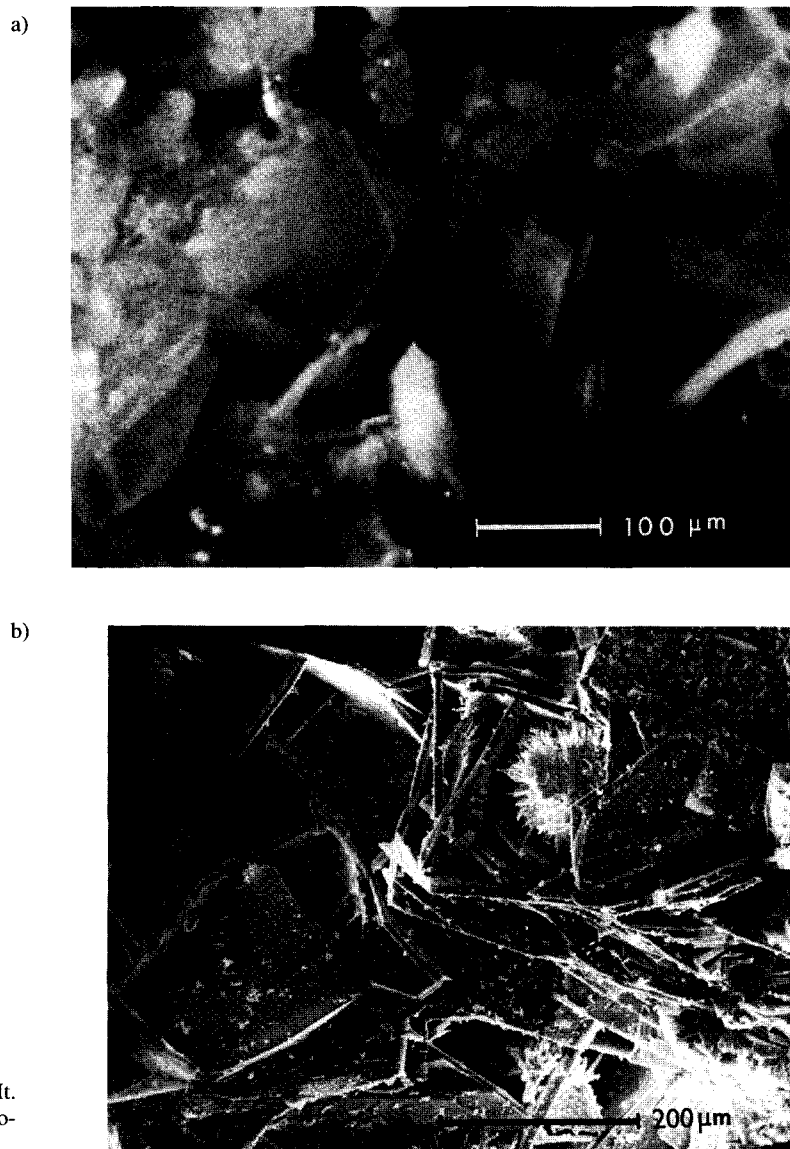


Fig. 1. Gottardiite from Mt. Adamson: a) pseudo-hexagonal crystals; b) SEM photo.

tion. Owing to the small size of the crystals it was impossible to determine their hardness. Gottardiite is brittle, with conchoidal to irregular fracture and exhibits a perfect {001} cleavage. The measured density is $2.14(4) \text{ g/cm}^3$, determined by a torsion microbalance and toluene, according to Berman (1939); this value agrees satisfactorily with that calculated (2.16 g/cm^3). Calculations using the Gladstone-Dale relationship and the constants reported by Mandarino (1981) indicate compatibility indices $(1-K_p/K_c) = 0.023$ (excel-

lent) and $= 0.015$ (superior) for calculated and measured densities, respectively. On the universal stage, in Na-parallel light ($\lambda = 589 \text{ nm}$), gottardiite is biaxial negative, with $2V_{\text{meas}} < 60^\circ$ and $2V_{\text{calc}} = 48^\circ$. The refractive indices are $\alpha = 1.480(2)$, $\beta = 1.485(2)$, $\gamma = 1.486(2)$, with optical orientation $X = b$, $Y = a$ and $Z = c$, as shown in Fig. 2. Extinction is parallel.

An infrared absorption spectrum was recorded between 4000 and 500 cm^{-1} with a IR Bruker IFS 113 V spectrometer, using the KBr pellet

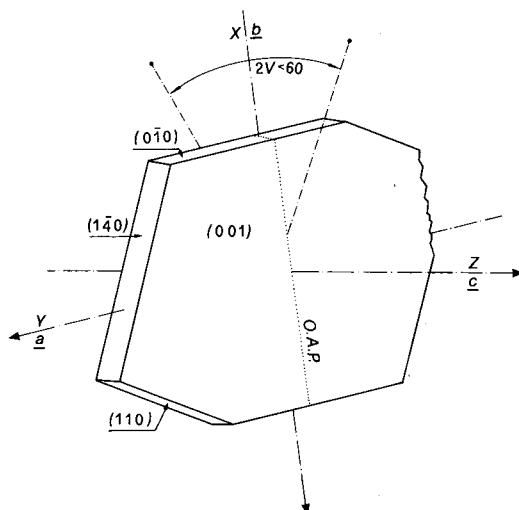


Fig. 2. Drawing of the habit of gottardiite crystals, showing the optical orientation of the refractive indices.

method (Fig. 3). The peaks in the zone $3600\text{--}3300\text{ cm}^{-1}$ and 1600 cm^{-1} can be reasonably attributed to the structural water. The main peaks in the $1200\text{--}900\text{ cm}^{-1}$ region are due to absorption by tetrahedral bonds.

Chemical composition and thermal behaviour

The chemical analyses were performed with an ARL-SEMQ electron microprobe operating in wavelength dispersive mode at 15 kV, with a 20 nA sample current. The standards used were: natural albite (Si, Al, Na), microcline (K), paracelsian (Ba), synthetic An_{70} glass (Ca), Sr-anorthite (Sr),

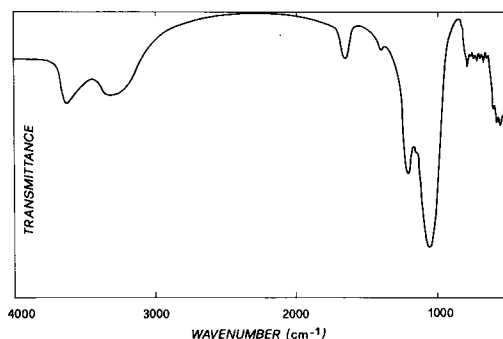


Fig. 3. Infrared spectrum of gottardiite.

P140 olivine (Fe), DI85-JD15 (Mg). Because of possible ion mobility and H_2O loss, as frequently occurs in zeolites, a beam diameter of $20\text{ }\mu\text{m}$ was used. A total of 13 point analyses were performed on a few crystals and averaged; the low variability in the point analyses suggests good compositional homogeneity for gottardiite.

Water content was determined by thermogravimetric analysis on 3.91 mg of sample using a Seiko SSC/5200 operating at $10^\circ\text{C}/\text{min}$ from 18 to 1000°C in air. The weight loss so determined is 16.44%, which is greater than the H_2O content that can be deduced from the percentage difference to 100 of the microprobe analysis values (12.76). This behaviour is typical of zeolites with wide channels (*e.g.* boggsite, faujasite, mordenite) (Howard *et al.*, 1990) and so the averaged weight percentages of oxides were normalized to a total of 83.56. The analytical results are summarized in Table 2, together with the chemical formula based on 272 oxygens, being the number of framework oxygens in the unit cell found by structure analysis (Alberti *et al.*, 1996). The number of tetrahedral atoms, which is very close to half that of the oxygen atoms, and the very low balance error (Passaglia, 1970) $E = +0.6\%$, support the reliability of the chemical analysis. Worth noting is the Si/Al ratio, which is the highest among the natural zeolites found up to now. The divalent extraframework cations (8.03) prevail over the monovalent ones (2.70). The Mg content is particularly high; though lower than that of ferrierite, it is comparable with that of mazzite, faujasite and offretite. The low Mg content found in smectite (see Table 1) and in the zeolites present in the samples collected in this area (Vezzalini *et al.*, 1994; Galli *et al.*, 1995) suggests a Mg-selectivity of gottardiite. Ca and Na are also abundant, whereas K, Sr and Ba are very low.

The TG and DTG curves of gottardiite are reported in Fig. 4. The DTG curve shows a broad band with two peaks at 127 and 175°C respectively; this feature suggests a practically continuous loss of weakly bonded water molecules. To test the reversible dehydration capacity of gottardiite, 3.5 mg of powder were heated to 110, 280, 500, 650, 800, and 1100°C each time being allowed to cool to room temperature before reheating. The mineral quickly and completely regained its weight loss at temperatures of up to 800°C , whereas at 1100°C its rehydration capacity became zero, probably due to the framework destruction occurring in this temperature range.

Table 2. Chemical analysis and unit cell content (on the basis of 272 oxygens) of gottardiite.

Oxide	wt. %	Atoms per unit cell	
SiO ₂	69.23 ± 0.51	Si	117.17
Al ₂ O ₃	9.40 ± 0.30	Al	18.75
Fe ₂ O ₃	0.10 ± 0.03	Fe ³⁺	0.13
MgO	1.24 ± 0.10	Mg	3.13
CaO	2.67 ± 0.14	Ca	4.84
SrO	0.04 ± 0.04	Sr	0.04
BaO	0.03 ± 0.04	Ba	0.02
Na ₂ O	0.77 ± 0.28	Na	2.53
K ₂ O	0.08 ± 0.04	K	0.17
		O	272
H ₂ O**	16.44	H ₂ O	92.80
R [†]	0.862		
M/D [‡]	0.336		

* Electron microprobe analysis: mean of 13 point-analyses and standard deviations.

** Water content from TG analysis.

† R = Si/(Si + Al).

‡ M = Na + K; D = Mg + Ca + Sr + Ba.

The high thermal stability of gottardiite is strictly related to the very high silicon content; besides, its fast and complete rehydration is characteristic of zeolites in which the breaking of T-O-T bridges does not take place during dehydration, as occurs in barrerite, stellerite, stilbite, heulandite, clinoptilolite, losod and zeolite EAB (Alberti & Vezzalini, 1984).

X-ray crystallography

Table 3 reports the X-ray powder diffraction patterns of gottardiite carried out using a Philips PW 2253/20 diffractometer and a Gandolfi camera (diameter 114.6 mm) with Cu K α radiation and Pb(NO₃)₂ as internal standard. The angular and densitometric measurements of the Gandolfi pattern were obtained with the AS/880 System (Officina Elettrotecnica di Tenno). The measured intensities of the Gandolfi pattern are more reliable than those obtained with a powder diffractometer, which are affected by isoorientation effects. Least-squares refinement of 75 reflections from the diffractometer pattern, was performed using Alberti's method (1976), which takes into account the diffraction intensities obtained from single crystal. The unit cell parameters obtained are: $a = 13.698$ (2), $b = 25.213$ (3) and $c = 22.660$ (2) Å.

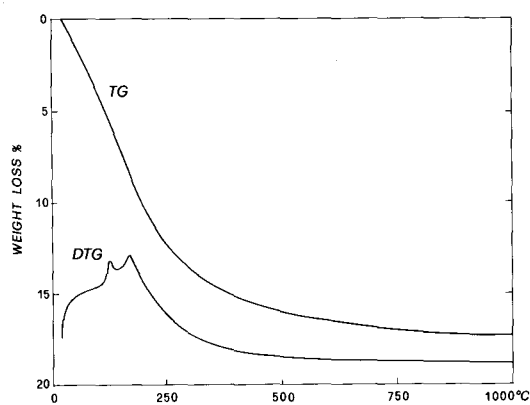


Fig. 4. Thermal curves of gottardiite; heating rate 10°C/min, operated in air.

The topological and the real symmetry of Gottardiite are *Fmmm* and *Cmca*, respectively (Alberti *et al.*, 1996). The framework topology has not been found in other natural zeolites, but is the same as that of the synthetic zeolite NU-87 (structure type code NES); however the H-form of the latter highly siliceous synthetic zeolite has a $P2_1/c$ monoclinic symmetry with $a = 14.32$, $b = 22.38$, $c = 25.09$ Å and $\beta = 151.5^\circ$ (Shannon *et al.*, 1991).

Conclusions

The discovery of natural analogues of known synthetic zeolites is becoming a fairly common occurrence; to date this has happened in the case of the following minerals:

gottardiite $\Leftrightarrow$ NU-87,
mazzite $\Leftrightarrow$ omega,
perlialite $\Leftrightarrow$ Type-L,
tschernichite $\Leftrightarrow$ beta,
pahasapaite $\Leftrightarrow$ RHO,
bellbergite $\Leftrightarrow$ EAB,
gaultite $\Leftrightarrow$ VPI-7.

The discovery of the natural counterpart of the NU-87 structure type is important because it implies that an organic template, used as directing agent, [e.g. bistrimethylammonio-alkane (Casci & Stewart, 1990) or diquaternary cations of formula (CH₃)₃N⁺(CH₂)₁₀N⁺(CH₃)₃ (Moini *et al.*, 1994)], may not be essential for its synthesis. Furthermore, gottardiite has a Si/Al ratio (6.2) lower than that of synthetic NU-87 (in the range 21–50, after Moini *et al.*, 1994; and Nakagawa, 1994), in-

Table 3. X-ray powder diffraction data for gottardiite.

h k l	d _{calc}	d _{obs} *	I/I ₀ *	d _{obs} **	I/I ₀ **	h k l	d _{calc}	d _{obs} *	I/I ₀ *	d _{obs} **	I/I ₀ **
0 2 0	12.61	12.63	<1	12.63	9	5 5 3	2.294	2.293	<1	2.295	3
0 0 2	11.33	11.33	100	11.34	100	1 5 9	2.223	2.223	1	2.228	3
1 1 1	10.63	10.65	2	10.64	31	3 1 9	2.196	2.197	1	2.200	2
0 2 2	8.43	8.44	1	8.44	11	2 0 10	2.151	2.151	2	2.153	4
2 0 0	6.85	6.85	1	6.86	11	6 4 0	2.146	2.144	1	2.142	5
0 0 4	5.67	5.67	2	-	-	6 0 4	2.117	2.119	1	2.115	7
0 4 2	5.51	5.51	1	5.53	7	0 12 0	2.101	2.100	1	2.102	3
2 2 2	5.31	5.32	<1	5.31	2	5 5 6	2.030	2.032	1	2.034	3
1 3 3	5.20	5.21	<1	5.20	2	3 5 9	2.020	2.020	1	2.024	4
1 5 1	4.63	4.63	5	4.64	35	6 6 0	2.006	2.006	1	2.010	3
3 1 1	4.41	4.41	5	4.37	79	1 3 11	1.9798	1.9782	<1	1.9774	<1
2 0 4	4.37	4.37	20	-	-	4 8 6	1.9762	1.9535	1	1.9541	3
2 4 2	4.29	4.29	1	4.21	19	6 0 6	1.9538	1.9315	<1	1.9317	2
0 4 4	4.21	4.21	4	4.13	12	5 5 7	1.9317	1.9135	1	1.9136	4
2 2 4	4.12	4.12	1	4.01	57	2 6 10	1.9150	1.8821	1	-	-
1 5 3	4.01	4.01	11	3.938	36	1 13 1	1.9135	1.8721	2	1.8730	5
0 6 2	3.940	3.940	5	3.855	9	4 10 4	1.9113	1.8330	1	-	-
3 1 3	3.861	3.861	1	-	-	0 10 8	1.8833	1.8326	1	-	-
1 3 5	3.830	3.829	2	3.776	14	3 1 11	1.8726	1.8090	1	-	-
0 0 6	3.777	3.776	40	3.613	5	5 5 8	1.8343	1.7984	1	-	-
0 2 6	3.618	3.620	1	-	-	3 3 11	1.8326	1.7734	1	1.7739	3
2 4 4	3.589	3.586	1	-	-	0 4 12	1.8089	1.7486	1	1.7480	2
2 6 0	3.582	3.424	4	3.428	29	7 1 5	1.7920	1.6923	<1	-	-
4 0 0	3.424	3.425	4	3.363	19	7 5 3	1.7733	1.6698	1	-	-
0 6 4	3.375	3.376	2	3.282	68	2 4 12	1.7490	1.6253	1	-	-
3 5 1	3.347	3.349	3	3.107	5	8 0 2	1.6930	1.6244	1	-	-
2 0 6	3.307	3.308	10	3.025	2	6 10 0	1.6923	1.6045	<1	-	-
1 5 5	3.273	3.277	10	-	-	2 6 12	1.6704	1.5885	<1	-	-
3 5 2	3.243	3.242	2	2.929	6	8 2 4	1.6253	1.5747	1	-	-
3 1 5	3.191	3.191	5	-	-	3 1 13	1.6251	1.5586	<1	-	-
0 8 0	3.152	3.152	1	-	-	6 10 4	1.6215	1.5490	1	-	-
1 1 7	3.126	3.126	4	2.659	2	0 2 14	1.6054	1.5276	1	-	-
3 5 3	3.089	3.089	2	2.586	7	1 15 4	1.6004	1.5100	1	-	-
2 6 4	3.027	3.029	1	2.523	12	7 5 7	1.5893	1.4984	1	-	-
3 3 5	3.004	3.004	1	-	-	3 11 9	1.5890	1.4742	<1	-	-
1 5 6	2.952	2.953	1	-	-	2 0 14	1.5752	1.4675	1	-	-
2 4 6	2.929	2.929	2	-	-	3 15 1	1.5736	1.4518	1	-	-
4 4 2	2.908	2.906	1	-	-	3 9 11	1.5598	1.4307	<1	-	-
3 5 4	2.906	2.853	1	-	-	3 5 13	1.5497	1.4299	<1	-	-
4 2 4	2.854	2.806	1	2.807	2	2 4 14	1.5282	1.4153	3	-	-
3 7 1	2.806	2.763	3	2.766	3	8 6 4	1.5270	1.4072	1	-	-
1 7 5	2.762	2.713	<1	-	-	1 15 6	1.5261	1.3872	1	-	-
3 5 5	2.712	2.654	1	-	-	4 10 10	1.5122	1.3863	1	-	-
4 6 0	2.665	2.618	1	2.586	7	0 6 14	1.5104	1.3866	1	-	-
2 0 8	2.617	2.536	1	2.463	6	1 1 15	1.4989	1.3869	1	-	-
0 4 8	2.584	2.536	1	2.415	8	2 6 14	1.4750	1.3869	1	-	-
4 0 6	2.537	2.521	2	2.390	<1	5 1 13	1.4682	1.3869	1	-	-
0 10 0	2.521	2.487	1	-	-	3 11 11	1.4526	1.3869	1	-	-
4 2 6	2.487	2.462	1	-	-	9 5 3	1.4307	1.3869	1	-	-
0 10 2	2.461	2.417	3	-	-	5 15 1	1.4299	1.3869	1	-	-
2 4 8	2.417	2.394	1	-	-	7 1 11	1.4165	1.3869	1	-	-
5 5 1	2.394	2.372	1	-	-	0 0 16	1.4163	1.3869	1	-	-
3 9 1	2.375	2.346	8	-	-	7 11 5	1.4140	1.3869	1	-	-
1 7 7	2.349	2.333	1	-	-	5 15 3	1.4076	1.3869	1	-	-
0 6 8	2.349	2.333	1	-	-	9 5 5	1.3872	1.3869	1	-	-
5 1 5	2.334	2.333	1	-	-	2 0 16	1.3869	1.3869	1	-	-
				-	-	3 17 3	1.3866	1.3869	1	-	-

Table 3. (Legend)

* Philips automated diffractometer PW 2253/20; CuK α monochromated radiation ($\lambda = 1.54051 \text{ \AA}$) using a curved pyrolytic graphite single crystal; slits 1° , 0.2 mm, 0.5° ; step scan (2θ) 0.02° , time per step (s) 18; internal standard Pb(NO $_3$) $_2$ (cubic: $a = 7.8568 \text{ \AA}$).

** Gandolfi camera; angular and densitometric measurements by AS/880 System (by courtesy of Officina Elettrotecnica di Tenno, Italy).

dicating that the range of composition required for the crystallization of this structural type is greater than that reported in literature.

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