American Mineralogist, Volume 75, pages 1415–1420, 1990

Montesommaite, $(K,Na)_9Al_9Si_{23}O_{64}\cdot 10H_2O$, a new zeolite related to merlinoite and the gismondine group*

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Abstract

The new zeolite montesommaite, $(K,Na)_9Al_9Si_{23}O_{64}\cdot 10H_2O$, occurs with dolomite, calcite, chabazite, and natrolite in vesicles in scoria from Pollena, Monte Somma-Vesuvius, Italy. Montesommaite forms transparent, colorless, dipyramidal crystals, having a vitreous luster, a white streak, and no cleavage. The observed and calculated densities are 2.34 ± 0.04 and 2.30 g/cm^3, respectively. Optically, it is biaxial negative, with $2V = 35 \pm 5°$ (observed) and 39° (calculated), $\alpha = 1.498$, $\beta = 1.506$, $\gamma = 1.507$ (all ±0.001), and no dispersion. The orientation is $X = c$, $Y = a$ or b, and $Z = a$ or b (a and b are indistinguishable). An electron microprobe analysis gave K_2O 16.7, Na_2O 0.2, Al_2O_3 19.8, SiO_2 55.7, H_2O 7.6 (by difference), sum 100.0 wt%. This yields $K_{8.8}Na_{0.2}Al_{9.7}Si_{23.1}O_{75.6}H_{21.0}$ or, ideally, $(K,Na)_9Al_9Si_{23}O_{64}\cdot 10H_2O$ for the unit-cell contents. Montesommaite is orthorhombic, $Fdd2$, with $a = b = 10.099(1)$, $c = 17.307(3)$ Å, and $Z = 1$. The mineral is very nearly tetragonal, $I\bar{4}2d$ or $I4_1md$ (pseudo-$I4_1/amd$), with $A = a/\sqrt{2} = 7.141$ and $C = c = 17.307$ Å. The strongest powder X-ray diffraction lines (d_{obs},I_{obs},hkl) are 6.589(75)111; 4.334(43)004; 3.299(100)222; 3.130(100)311,115; 2.797(30)313; 2.513(18)400,206; 2.347(22)315; 1.784(22)440; 1.720(16)515,337; and 1.558(18)2·2·10. Similarities in cell parameters, symmetry, and chemical formulae strongly imply that montesommaite is related to merlinoite ($K_5Ca_2Al_9Si_{23}O_{64}\cdot 24H_2O$) and to the members of the gismondine group. This suggestion is verified by a determination of the idealized substructure of montesommaite in $I4_1/amd$ symmetry using powder diffraction intensities.

Introduction

In December of 1987 we received from Ezio Bernabé of Riccione, Italy, some very small, colorless, euhedral crystals of an unknown mineral from the town of Pollena in the Monte Somma-Vesuvius volcanic complex. Preliminary study suggested that the crystals might represent a new species, and subsequent investigation has confirmed that hypothesis. The new mineral has been named montesommaite after Monte Somma, the high ridge that constitutes the remains of the volcanic cone that preceded Vesuvius. Both the species and name have been approved in advance of publication by the Commission on New Minerals and Mineral Names, IMA. Holotype material has been deposited in the National Museum of Natural History, Smithsonian Institution, under catalogue number NMNH 165440.

All of the original (holotype) crystals were extracted from a single 3-mm vesicle, which occurs in a small specimen of scoria. The associated minerals are dolomite, calcite, chabazite, and natrolite. The holotype specimen was unique until the recent discovery of additional, similar specimens from the same locality. However, even with this new material, montesommaite remains an exceedingly rare mineral.

Physical and optical properties

Montesommaite occurs as transparent, colorless, euhedral crystals up to 0.1 mm in size. The point group, as determined from the combined optical and X-ray diffraction studies (see later), is $mm2$, and the crystals have an orthorhombic or distorted tetragonal aspect (Fig. 1). Given the acentric point symmetry, the forms present must be described as two pedions $\{001\}$ and $\{00\bar{1}\}$ and two pyramids $\{hkl\}$ and $\{\bar{h}\bar{k}\bar{l}\}$ of unknown indices. The streak is white, the luster is vitreous, and there are no

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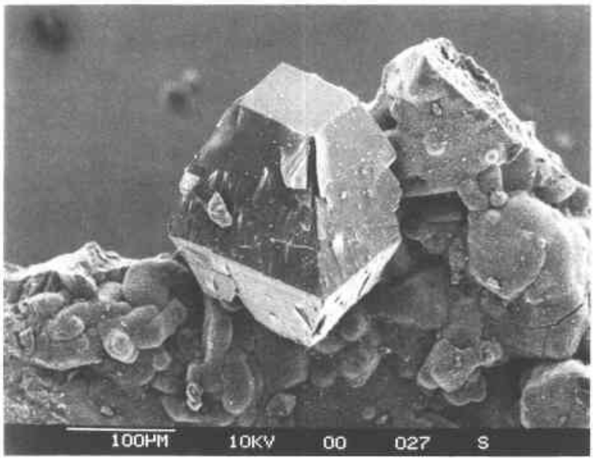

Fig. 1. A scanning electron micrograph of a single montesommaite crystal attached to the surface of a scoria vesicle.

apparent cleavages. The density, measured using a heavy liquid technique, is 2.34 ± 0.04 g/cm^3 compared to a value of 2.30 calculated from the refined unit-cell parameters and the idealized formula $(K,Na)_9Al_9Si_{23}O_{64}\cdot 10H_2O$. Hardness could not be determined because of the fragility and minute size of the crystals.

Optically, montesommaite is biaxial negative, with $\alpha = 1.498(1)$, $\beta = 1.506$, $\gamma = 1.507$ (all ±0.001), and $2V = 35 \pm 5°$ measured in Na light. The calculated value of $2V$ is 39°, and there is no dispersion. The optical orientation is $X = c$, $Y = a$ or b, and $Z = a$ or b, the ambiquity arising from the strongly pseudotetragonal character of the mineral, which causes a and b to be equal within error of measurement. There is no fluorescence in long- or shortwave ultraviolet radiation.

CHEMICAL COMPOSITION

As the amount of available material was very small, montesommaite was chemically analyzed using an ARL-SEMQ electron microprobe operated at 15 kV, with a 0.025-μA sample current and a 20-μm beam spot. The analytical standards used were microcline for K, Al, and Si and hornblende for Na. A wavelength-dispersive microprobe scan revealed no other elements with an atomic number greater than 9. The resulting analysis is K_2O 16.7, Na_2O 0.2, Al_2O_3 19.8, SiO_2 55.7, H_2O (7.6), sum 100.0 wt%. H_2O could not be directly determined, again because of the very small amount of material available, and is calculated here by difference.

When one uses the above analysis, the refined orthorhombic unit-cell parameters, and the measured density, the unit-cell contents of montesommaite for the orthorhombic cell are $K_{8.8}Na_{0.2}Al_{9.7}Si_{23.1}O_{75.6}H_{21.0}$, which, if normalized to 32 (Al + Si), become $(K_{8.6}Na_{0.2})_{\Sigma 8.8}(Al_{9.4}Si_{22.6})_{\Sigma 32}O_{73.9}H_{20.5}$. The idealized formula is $(K,Na)_9Al_9Si_{23}O_{64}\cdot 10H_2O$ with $Z = 1$, assuming all H_2O to be present as molecular H_2O. As will be shown below, this interpretation is supported by a crystal structure analysis of the montesommaite substructure, except that the number of H_2O molecules per cell may be slightly less than the ten indicated here.

CRYSTALLOGRAPHY

The most salient feature of the crystallography of montesommaite is its several levels of pseudosymmetry. Precession and Weissenberg photographs show, with very small and problematical exceptions, a tetragonal intensity distribution. The interpretation of the photographs is complicated by the fact that all crystals examined were highly imperfect, consisting of one major and several minor individuals and showing some streaking of reflections indicative of structural disorder. Nevertheless, a tetragonal (or pseudotetragonal) unit cell can be recognized, which has parameters $A = 7.1410(9)$ and $C = 17.307(3)$ Å and symmetry $I\bar{4}2d$ or $I4_1md$. In addition, the reflections of the class $hk0$ with (h,k) odd are, with few exceptions, unobserved or of low intensity, and the symmetry of the structure therefore approaches $I4_1/amd$. However, the optical study shows that montesommaite is decidedly biaxial, and this, taken with the apparent deviations from $4/mmm$ symmetry on the diffraction photographs, indicates that the mineral is actually orthorhombic, $Fdd2$, with $a = b = 10.099(1)$ and $c = 17.307(3)$ Å. The orthorhombic cell is rotated 45° around the pseudofourfold axis of the tetragonal subcell so that $a = b = A\sqrt{2}$ and $c = C$, with a concomitant change in lattice type from body centered to face centered. The magnitudes of the supercell translations a and b, as determined from the single-crystal photographs and refined from the powder diffraction data (Table 1), are equal within error of measurement. The powder data are fully indexable on the pseudotetragonal cell.

The strongly pseudotetragonal character of the crystals makes twinning likely, but the X-ray diffraction and optical studies produced no clear evidence of this phenomenon. Twinning, if present, would probably be by 90° rotation around the pseudofourfold axis, c, but this would give rise to uniaxial optics in contradiction to the pronounced biaxial character observed for this mineral. On the other hand, if montesommaite were really tetragonal and twinned so as to simulate a biaxial mineral, it would require twinning by reflection on $\{100\}$ with an obliquity of $\sim 17° (= 1/2 \times 2V)$. Such a large angle between the two individuals of the twin should make the twinning obvious under the microscope, but none was observed.

A TETRAHEDRAL FRAMEWORK MODEL FOR MONTESOMMAITE

The known chemical and crystallographic data for montesommaite are consistent with the new mineral being a member of the zeolite family. The pseudotetragonal orthorhombic cell and the a and b lattice parameters of 10 Å suggest a framework with a 4.8^2 net (Smith, 1978), which occurs in merlinoite and gismondine.

Merlinoite is orthorhombic, $Immm$, with $a = 14.116$, $b = 14.299$, and $c = 9.964$ Å, and it has the idealized

TABLE 1. Powder X-ray diffraction data for montesommaite indexed on both its orthorhombic and pseudotetragonal cells

I_{obs}	d_{obs}	d_{calc}	*hkl*	*HKL*
75	6.589	6.601	111	101
0.7	4.488	4.488	113	103
43	4.334	4.327	004	004
100b	3.299	3.301	222	202
100b	3.130	3.140	311	211
		3.115	115	105
30	2.797	2.794	313	213
18b	2.513	2.525	400	220
		2.505	206	116
22	2.347	2.347	315	215
11	2.256	2.258	420	310
16b	2.178	2.181	404	224
		2.163	008	008
2	2.005	2.002	424	314
5	1.959	1.961	335	305
		1.955	317	217
1b	1.865	1.873	513	323
		1.857	119	109
22	1.784	1.785	440	400
16	1.720	1.719	515	325
		1.715	337	307
9	1.655	1.652	602	332
0.7	1.595	1.597	620	420
11	1.571	1.570	622	422
18	1.558	1.557	2·2·10	2·0·10
13	1.546	1.546	517	327
0.7	1.497	1.498	624	424
2	1.442	1.442	0·0·12	0·0·12
5	1.420	1.423	711	431
		1.419	537	417
3	1.397	1.397	626	426
3	1.381	1.382	642	512
11	1.320	1.320	555, 715	505, 435
6	1.291	1.292	733	523
3	1.262	1.262	800	440
8	1.253	1.252	4·0·12	2·2·12
4	1.237	1.237	557, 717	507, 437
0.7b	1.227	1.232	5·1·11	3·2·11
		1.229	3·1·13	2·1·13
		1.225	820	530
3	1.212	1.213	822	532
		1.212	804	444
5	1.174	1.174	6·2·10	4·2·10

Note: 114.6 mm Gandolfi camera, polycrystalline specimen, Cu$K\alpha$ radiation, I_{obs} estimated by comparison with a calibrated intensity strip, *b* = slightly broadened line. Indexed with the aid of the single-crystal photographs. The first index given (*hkl*) is for the true, orthorhombic cell, and the second (*HKL*) is for the pseudotetragonal subcell. In the orthorhombic case *hkl* and *khl* are nonequivalent, but since they have the same *d* values only *hkl* is listed.

formula $K_5Ca_2Al_9Si_{23}O_{64}\cdot 24H_2O$ (Galli et al., 1979). It is pseudotetragonal, with $a \approx b \approx 2A$ of montesommaite, and the Al/Si/O ratio is identical for both minerals. Gismondine and its isotypes are monoclinic or orthorhombic and pseudotetragonal, with $a = b = 10$ Å and $c = 9.8$ Å (or vice versa). The topological symmetry of the gismondine-type framework is $I4_1/amd$, which for an ordered distribution of Al and Si reduces to *Fddd* with orthorhombic cell parameters $a_o \approx b_o \approx a_t\sqrt{2}$ and $c_o = c_t$ (Alberti and Vezzalini, 1979). This situation is analogous to that in montesommaite, which is orthorhombic, *Fdd*2, and pseudotetragonal, $I4_1/amd$, and which has the same geometrical relationship between its orthorhombic and pseudotetragonal cells. Chemically, the gismondine group may be represented by the formula $(Ca,K,Na)_{4-8}$-$(Al,Si)_{16}O_{32}\cdot nH_2O$, which resembles that of montesommaite when the latter is written as $2[(K,Na)_{4.5}(Al,Si)_{16}O_{32}\cdot 5H_2O]$. Although the above data suggest that montesommaite is allied to merlinoite and the gismondine group, there is the conspicuous dissimilarity of the 17.3 Å cell translation, which occurs in montesommaite but not in the latter minerals.

Smith and Rinaldi (1962) and Smith (1968, 1978) have systematically derived four-connected three-dimensional frameworks that can be obtained by adding a perpendicular branch to each node of a 4.8^2 net. Their work was restricted to nets that repeat after two layers. Among these frameworks is Smith model number 38 (untwisted UDUD, $P4_2/mmc$), with $a = b = 7.03$ Å and $c = 8.97$ Å. The latter parameters were obtained by distance least squares (DLS) refinement. A possible framework model for montesommaite may be obtained from Smith model 38 by translating successive 4.8^2 sheets by $a/2$ and $b/2$,

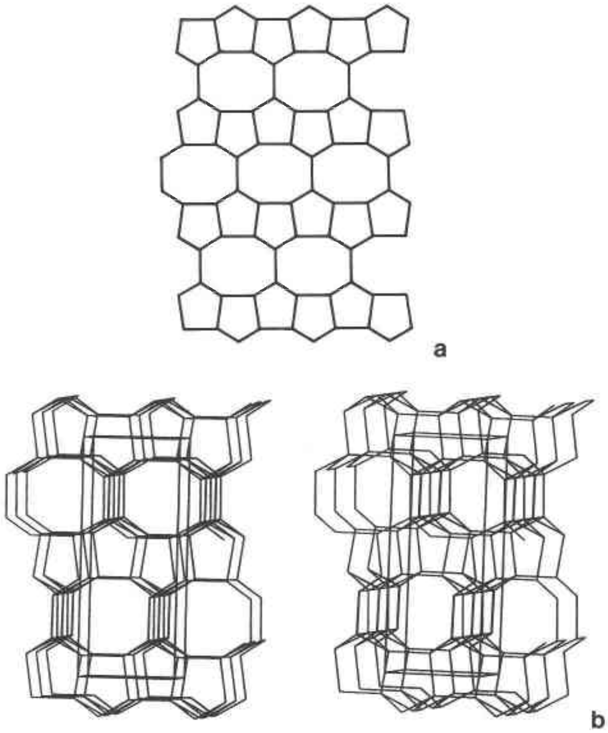

Fig. 2. Tetrahedral node diagrams of the aluminosilicate framework proposed for montesommaite. The framework is shown in its ideal, tetragonal (symmetry $I4_1/amd$) form, but its actual configuration in montesommaite should differ little from this, as the mineral is strongly pseudotetragonal. (**a**) A (100) projection, showing the $(5.^28)_2(5.8^2)_1$ net. (**b**) A stereographic image looking approximately toward (100) of the tetragonal unit cell.

TABLE 2. Atomic parameters from the DLS refinement and a calculated powder X-ray diffraction pattern for the idealized montesommaite substructure

Atom	Equipoint	*x*	*y*	*z*
T	16*h*	0	0.4673	0.0898
O(1)	8*e*	0	¼	0.1163
O(2)	16*g*	0.1857	0.4357	⅞
O(3)	8*c*	0	0	0

$2\theta_{calc}$	d_{calc}	*H*	*K*	*L*	I_{calc}	$2\theta_{calc}$	d_{calc}	*H*	*K*	*L*	I_{calc}
13.40	6.601	1	0	1	93.5	46.41	1.955	2	1	7	0.0
19.77	4.488	1	0	3	0.4	48.56	1.873	3	2	3	5.3
20.35	4.361	1	1	2	16.7	49.02	1.857	1	0	9	2.7
20.51	4.327	0	0	4	53.8	49.21	1.850	2	0	8	0.0
24.92	3.570	2	0	0	0.2	51.12	1.785	4	0	0	21.5
26.99	3.301	2	0	2	100.0	51.34	1.778	3	1	6	1.8
28.40	3.141	2	1	1	68.8	52.28	1.748	4	0	2	0.0
28.64	3.115	1	0	5	33.0	53.10	1.723	4	1	1	1.3
32.01	2.794	2	1	3	33.1	53.24	1.719	3	2	5	3.2
32.49	2.754	2	0	4	0.1	53.39	1.715	3	0	7	3.8
35.53	2.525	2	2	0	26.5	55.34	1.659	4	1	3	0.9
35.82	2.505	1	1	6	1.6	55.58	1.652	3	3	2	4.6
38.13	2.358	3	0	1	1.2	55.65	1.650	4	0	4	1.0
38.32	2.347	2	1	5	18.0	55.76	1.647	2	1	9	0.0
38.50	2.336	1	0	7	4.9	55.93	1.643	2	2	8	0.0
40.16	2.244	2	0	6	3.6	56.13	1.637	1	1	10	0.0
40.98	2.200	3	0	3	0.4	57.69	1.597	4	2	0	0.6
41.29	2.185	3	1	2	0.9	58.75	1.570	4	2	2	14.4
41.37	2.181	2	2	4	4.2	59.09	1.562	3	1	8	1.9
41.72	2.163	0	0	8	8.8	59.29	1.557	2	0	10	12.3
45.26	2.002	3	1	4	3.7	59.65	1.549	4	1	5	2.5
46.09	1.968	3	2	1	0.6	59.78	1.546	3	2	7	8.8
46.25	1.961	3	0	5	0.6						

Note: The intensities are calculated based on the idealized ($I4_1/amd$) substructure and using the DLS coordinates for the framework atoms and the peak positions from the difference synthesis for K and H_2O.

respectively. The resulting four-layer framework has $I4_1/amd$ symmetry, with DLS cell parameters of $a = b = 7.09$ Å and $c = 17.33$ Å. A stereographic drawing of the framework is illustrated in Figure 2. The (100) projection in Figure 2 is a $(5.^28)_2(5.8^2)_1$ net. Other frameworks based on this net have been enumerated by Smith and Bennett (1984).

The DLS atomic coordinates and a calculated powder X-ray diffraction pattern for the idealized montesommaite substructure are listed in Table 2. The calculation is based upon the DLS coordinates for framework cations and O atoms and upon the locations of the extraframework species K and H_2O obtained from the substructure solution (to be discussed later). Although $I4_1/amd$ is the maximum topological symmetry of the framework, both ordering of tetrahedral Si/Al and the presence of extraframework cations and H_2O could reduce the symmetry to *Fdd*2. Disordered intergrowths with Smith framework 38 are also possible and might explain the X-ray peak broadening observed in montesommaite.

SOLUTION OF THE IDEALIZED SUBSTRUCTURE

The framework proposed for montesommaite is compatible with the subcell parameters and $I4_1/amd$ pseudosymmetry observed for this mineral. A further check for compatibility may be made by calculating structure factors from the framework model and comparing them with those derived from the powder diffraction pattern. Accordingly, the intensities of 22 single-index lines in the Gandolfi pattern were estimated as carefully as possible by comparison with a calibrated intensity film strip. The resulting I_{obs} were converted to $|F|_{obs}$ using Lorentz polarization (Lp) and multiplicity (*m*) factors for the Debye-Scherrer method. Although Lp is the same for both the Gandolfi and Debye-Scherrer methods, the value of *m* for the former may, for some reflections, be half that of the latter (Zhang, 1980). However, this should not be a problem in the present case, as the Gandolfi photograph of montesommaite was obtained from a polycrystalline specimen. The satisfactory agreement between $|F|_{obs}$ and $|F|_{cal}$ supports the validity of the data-reduction procedure. It was not possible to obtain a Debye-Scherrer photograph because of the very small amount of material available for study, and attempts to use the polycrystalline Gandolfi specimen for this purpose yielded only spotty and incomplete Debye rings because of the large crystallite size.

The subcell of montesommaite is pseudotetragonal, ideally $I4_1/amd$, with $A = 7.141$ Å, $C = 17.307$ Å, and its calculated cell contents are $(K_{4.3}Na_{0.1})_{\Sigma 4.4}(Al_{4.7}Si_{11.3})_{\Sigma 16}O_{32}\cdot 5.0H_2O$. A calculation of structure factors based upon this cell (with the origin chosen at $2/m$ in $I4_1/amd$) and the framework atoms T (= Al,Si) on 16*h*, O(1) on 8*e*, O(2) on 16*g*, and O(3) on 8*c* yielded an unweighted residual of 0.28. This and subsequent calculations were carried out with the SHELX-76 package (Sheldrick, 1976) under the following constraints: (1) The contents of the T site were fixed at $(Al_{4.7}Si_{11.3})$ to conform to the results of the chemical analysis. (2) The atomic coordinates were fixed at those from the DLS refinement for framework atoms and at those from the difference syntheses for all other atoms. (3) Isotropic temperature factors (*U*) were fixed at 0.005 $Å^2$ and 0.025 $Å^2$ for framework and extraframework cations, respectively, and at 0.015 Å for all anions. Only the overall scale factor was refined until the final stage of the structure solution process, at which time atomic coordinates were allowed to vary.

A difference synthesis was then prepared using the framework atoms as input. The largest peak on the resulting difference map had a height of $3e^-/Å^3$ and was located on 8*d* at (0,0,1/2), which lies within the eight-membered-ring channels of the framework. Adding this peak to the model as 4.3 K atoms, the minor Na in the chemical analysis being neglected, produced a large decrease in the residual from 0.28 to 0.16. The 8*d* site is coordinated by two O(1) at 2.93 Å and four O(2) at 3.15 Å, both values falling within the 2.65–3.27 Å range given by Baur (1970) for K-O bond distances. Since the 8*d* site lies within the channels, forms reasonable K-O distances, and produces a substantial reduction in the residual when added to the model as K, (0,0,1/2) is accepted as the position of the extraframework cation in montesommaite.

The three criteria just noted were also used in the search for the last missing chemical component of the structure, namely the H_2O molecules. The second largest peak in the difference synthesis had a height of 2e⁻/Å³ and was situated on 8*e* at (0,1/4,0.342). Four H_2O molecules were assigned to this site, and this produced a further reduction in the residual from 0.16 to 0.11. (The 8*e* site must have a maximum occupancy of 50% in order to avoid H_2O-H_2O distances of 1.14 Å.) The H_2O molecules are located in the channels, 2.39 and 3.27 Å from the extraframework cation K^+. This places K in eightfold coordination by one H_2O at 2.39 Å, two O(1) at 2.93 Å, four O(2) at 3.15 Å, and one H_2O at 3.27 Å. The coordination polyhedron may be regarded as a strongly distorted cube but more nearly resembles a distorted orthorhombic prism.

The short K-O distance of 2.39 Å deserves further comment. It is slightly shorter than the 2.65-Å minimum given by Baur (1970) but is not unprecedented among K-O distances; e.g., a 2.36-Å distance occurs in the structure of $K_2Ti_6O_{13}$ (Cid-Dresdner and Buerger, 1962). Alternatively, the 2.39-Å distance may be an artifact resulting from an error in the *z* coordinate of H_2O taken from the difference map. A small error in *z* will produce a relatively large error in the K-H_2O bond distance because of the rather large magnitude of the cell translation along *C* (17.3 Å). Moving H_2O from 8*e* at (0,1/4,0.342) to 4*b* at (0,1/4,3/8) produces two equal K-H_2O distances of 2.80 Å but also leads to an unacceptable increase in the residual from 0.11 to 0.14.

Since the chemical analysis of montesommaite indicates five H_2O per subcell and only four can be accommodated in the 8*e* site, a second difference synthesis was prepared in the hope of finding the missing H_2O molecule. Of the four largest peaks in the synthesis, all had heights of less than 1 e⁻/Å³ and could be dismissed from consideration on crystal chemical grounds; e.g., they did not occur on channel sites, or they occurred at unreasonably short distances from framework atoms. Additional H_2O molecule sites may indeed exist in montesommaite, but their detection is beyond the capacity of the very limited data at hand. A second possibility is that the subcell really does contain only four H_2O. Owing to the minute amount of material available for analysis, H_2O had to be determined by difference rather than by direct measurement. Four rather than five H_2O per subcell would reduce the analytical total by only 1.8% to 98.2%, which is within the error of ordinary electron microprobe analyses.

Table 3. Refined atomic coordinates and structure factors for the idealized montesommaite substructure

Atom	Equipoint	*x*	*y*	*z*	U_{iso} (Å²)
K	8*d*	0	0	½	0.025
T	16*h*	0	0.463(4)	0.090(2)	0.005
O(1)	8*e*	0	¼	0.116(8)	0.015
O(2)	16*g*	0.186(7)	0.436	⅞	0.015
O(3)	8*c*	0	0	0	0.015
H_2O	8*e*	0	¼	0.342(15)	0.015

HKL	F_{obs}	F_{cal}	*HKL*	F_{obs}	F_{cal}
101	74	81	2·0·10	177	161
103	11	6	327	106	89
004	172	177	424	26	24
202	175	184	0·0·12	129	107
213	80	71	426	57	28
215	83	79	512	60	57
314	31	47	523	89	90
400	235	228	440	132	147
332	117	110	2·2·12	152	163
240	34	25	4·2·10	92	109
422	97	106			

Note: Calculations are based upon a cell having *A* = 7.141 Å, *C* = 17.307 Å, and symmetry $I4_1/amd$. Atom T represents (4.7 Al + 11.3 Si). The K and H_2O sites contain 4.3 K and 4 O, respectively. Esd in parentheses.

Refinement of coordinates for all atoms produced a residual of 0.10 and the coordinates and structure factors in Table 3. An attempt was also made to refine isotropic temperature factors, but this produced improbably high and low values, reflecting the errors in the semiquantitative intensity data. The isotropic *U*s in Table 3 are the same assumed values left constant throughout the substructure solution process. The refined coordinates in Table 3 are, however, credible for two reasons: First, they yield reasonable T-O and K-O bond parameters. The only exception to this rule is a T-O(3)-T angle of 180°, but this improbably large angle is one more reason why the ideal $I4_1/amd$ structure must be unstable relative to the real orthorhombic configuration. Second, the coordinates in Table 3 differ from their DLS counterparts in Table 2 by no more than one esd. Some selected interatomic distances and angles calculated from the parameters in Table 3 are listed in Table 4.

The solution of the substructure of montesommaite could be taken one step further by working in the actual subcell symmetry, $I\bar{4}2m$ or $I4_1md$. That one of these space groups (rather than $I4_1/amd$) is the true subcell symmetry is demonstrated by the existence of weak *HK*0 reflections with *H* odd on the precession photographs and by the medium intensity reflection 310 in the powder pattern. There is, however, nothing to be gained by attempting to

Table 4. Selected interatomic distances (Å) and angles (°) for the idealized montesommaite substructure

T-O(3)	1.58(3)	O(2)-T-O(3)	107(1) × 2
O(1)	1.58(5)	O(1)-T-O(2)	109(4) × 2
O(2)	1.63(3) × 2	O(2)-T-O(2)	109(5)
Mean	1.61	O(1)-T-O(3)	116(5)
		Mean	109
T-T	3.04(6)	T-O(1)-T	147(10)
T	3.14(4) × 2	T-O(2)-T	150(5)
T	3.16(7)	T-O(3)-T	180(9)
Mean	3.12		
K-H_2O	2.39(17)	K-K	3.571(1) × 2
O(1)	2.93(12) × 2	K-O(3)	3.571(1) × 2
O(2)	3.15(4) × 4	K-T	3.63(3) × 4
H_2O	3.27(21)		
Mean	3.02		

Note: Esd in parentheses. Two and four equivalent distances or angles are indicated by ×2 and ×4, respectively.

work in a lower symmetry because a refinement of positional parameters would be necessary to determine how the real substructure deviates from its ideal $I4_1/amd$ configuration. Such a refinement is not practical here, given the large number of variables in the lower symmetry space groups and the small number of available reflections. Despite the latter limitation, the structure described here represents a good first approximation to the crystal structure of montesommaite. A complete determination of the structure in its true orthorhombic symmetry must await the discovery of single crystals of a quality suitable for such analysis.

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MANUSCRIPT RECEIVED JULY 13, 1989
MANUSCRIPT ACCEPTED OCTOBER 12, 1990