Holtite, $(Si_{2.25}Sb_{0.75})B[Al_6(Al_{0.43}Ta_{0.27}\square_{0.30})O_{15}(O,OH)_{2.25}]$: crystal structure and crystal chemistry

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Abstract

Holtite, $(Si_{2.25}Sb_{0.75})B[Al_6(Al_{0.43}Ta_{0.27}\square_{0.30})O_{15}(O,OH)_{2.25}]$, is orthorhombic $a = 4.6914(5)$, $b = 11.896(2)$, $c = 20.383(4)$ Å, space group *Pnma*. Full-matrix least-squares refinement yielded a final $R = 0.030$, for 1664 reflections with $I \geqslant 3\sigma(I)$. Its crystal structure is closely related to that of dumortierite, but with the difference that extensive substitution of Si by Sb occurs in holtite. SbO_3 triangular pyramids replace the SiO_4 tetrahedra arranged around the inside edge of the hexagonally shaped tunnels which are the feature of the dumortierite and holtite structures. Associated with these replacements is the replacement of Al by Ta in the octahedral site centred in these tunnels, and the development of vacancies in this metal site and its attached oxygens.

KEYWORDS: holtite, crystal structure, crystal chemistry, Greenbushes, Australia

Introduction

THE mineral holtite was first identified as a new mineral species when small crystals of it were found associated with stibiotantalite and tantalite in specimens from an alluvial tin lease near the township of Greenbushes, Western Australia (Pryce, 1971). Since then a second occurrence has been recorded in the Kola Peninsula, USSR, by Voloshin *et al.* (1976). The mineral was named after H. E. Holt (1908–67), Prime Minister of Australia (1966–67).

The unit cell parameters and space group, determined from X-ray studies of single crystals of holtite from Greenbushes, indicated a kinship with the mineral dumortierite which is composed mainly of the light element oxides Al_2O_3, B_2O_3, and SiO_2; it was (at that time) generally described as $4[(Al, Fe)_7BSi_3O_{18}]$. Holtite was found to contain significant amounts of the heavier elements Ta and Sb. Using the chemical data obtained from the Australian material, Pryce deduced that holtite contained the full unit cell contents $4(X_{10}O_{17})$ compared with the then accepted dumortierite cell contents of $4(X_{11}O_{18})$. Furthermore he found it difficult to group the constituent elements in holtite after dumortierite, but suggested that the Ta, Nb and Sb ions probably occupied Al sites in the holtite cell. In comparison, the Russian holtite was reported to contain higher Si, less Sb (reported only as Sb_2O_5—the Australian results report both Sb_2O_3 and Sb_2O_5) and higher boron, more in agreement with dumortierite itself (Losert, 1956; Murty, 1971; and Moore and Araki, 1978). The average overall composition of this material was given as $4[X_{10.2}(O,OH)_{17.4}]$ (Table 3; Voloshin *et al.*, 1976) or $4[X_{10.5}(O,OH)_{18}]$ when normalized to 18 anions.

The original determination of the crystal structure of dumortierite by Golovastikov (1965) was followed by a more accurate refinement of the

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structure by Moore and Araki (1978). This later study showed that some aluminium sites in the structure were partially occupied and that the composition of dumortierite is more typically of the form $Si_3B[Al_{6.70}\square_{0.25}O_{17.25}(OH)_{0.75}]$. Notwithstanding an overall satisfactory interpretation of the crystal chemistry of dumortierite based on the single crystal study, it is still discussed in terms of the simpler formula $Al_7BO_3(SiO_4)_3O_3$. The chemical formula used to describe the chemical substitutions in holtite is given as the conformable

$$Al_6(Ta,Sb,Li)[(Si,As)O_4]_3BO_3(O,OH)_3$$

(for example, Fleischer, 1983).

The study of the crystal structure of holtite described here was undertaken to confirm the inferred relationship with dumortierite; to resolve the nature of the complicated chemical substitutions in holtite; and to determine whether the chemistry of holtite might also be influenced by cation vacancies as was known to be the case in the apparently related dumortierite.

Experimental

An excellent, prism shaped, single crystal of holtite measuring 0.12 × 0.07 × 0.07 mm along the three crystallographic axes was obtained from the mineral collection at the Chemistry Centre of Western Australia. The crystal was mounted along the *a* axis; rotation and Weissenberg photographs of the zero and first levels exhibit extremely sharp spots at an approximately 4.7 Å spacing. The rotation films also display diffuse intermediate layers normal to *a* at spacings of 2*a* and 3*a*, as was discussed by Pryce (1971). The crystal was subsequently transferred such that the *a* axis was parallel to the axis of a Enraf-Nonius CAD-4F automated diffractometer. Cell constants were obtained from an orientation matrix based on 25 reference reflections and Mo–Kα radiation (graphite monochromated, λ = 0.710687 Å) which yielded a = 4.6914(5), b = 11.896(2), c = 20.383(4) Å, V = 1137.65 Å^3 and space group alternatives *Pnma* (standard setting) or $Pn2_1a$. The total number of reflections collected was 3359; after the data were merged this reduced to 2132 unique reflections (R_{amal} = 2.15). Absorption corrections were applied using the numerical analytical procedure in SHELX-76 (Sheldrick, 1976).

Solution and refinement

Because of the similarities in the distribution of the intensity of the X-ray data, cell size and overall chemistry, structure refinement of holtite was commenced using the dumortierite structure as the starting model (and space group *Pnma*). Support for this approach was also obtained from the direct methods system MULTAN (Main *et al.*, 1978), because, of the two solutions which resulted, the one with the better combined figure of merit yielded atom positions which were found to lie close to those of dumortierite. Full-matrix least-squares refinement based on dumortierite

Table 1. Atomic coordinates and occupancies for holtite.

Atom	Occupancy	x	y	z
Al 1	.2150Al + .1350Ta	.4052(1)	.75	.2499(0)
Al 2	1.0	.5571(2)	.6107(1)	.4735(0)
Al 3	1.0	.0588(2)	.4899(1)	.4315(0)
Al 4	1.0	.0603(2)	.3593(1)	.2902(0)
Si 1	0.375	.0878(4)	.75	.4077(1)
Sb 1	0.125	.1150(3)	.75	.3823(1)
Si 2	0.75	.5890(2)	.5216(1)	.3294(1)
Sb 2	0.25	.6117(2)	.5632(1)	.3164(0)
O 1	0.5	.3798(6)	.75	.4572(1)
O 2	0.375	.1618(9)	.75	.3305(2)
O 3	1.0	.8906(4)	.6377(2)	.4243(1)
O 4	1.0	.4016(4)	.4348(2)	.2815(1)
O 5	1.0	.3922(4)	.5511(2)	.3942(1)
O 6	1.0	.8837(4)	.4504(2)	.3518(1)
O 7	0.75	.6608(6)	.6333(2)	.2886(1)
O 8	0.5	.1729(6)	.25	.3499(1)
O 9	1.0	.2531(4)	.3508(1)	.4477(1)
O10	0.5	.7620(6)	.25	.2734(1)
O11	1.0	.7498(4)	.4670(1)	.4883(1)
B 1	0.5	.2252(8)	.25	.4158(2)

atom positions led to convergence at $R = 0.030$ and $R_w = 0.033$ for the 1664 reflections with $I \geqslant 3\sigma(I)$. The refinement was complicated to some extent by the effects of the partial occupancy of the Al(1) site and the splitting of the Si(1) and Si(2) sites to accommodate the (unexpected) Sb substitutions which occur near them. Coefficients for scattering factor curves for Al^{3+}, Sb^{3+}, Ta^{5+}, and B, Si, O (neutral) were taken from the International Tables, Volume 4, (1974). Calculations were done using the SHELX-76 library of programs installed on a Vax 11-750 computer. Atomic coordinates are given in Table 1, anisotropic temperature factors in Table 2, and interatomic distances and angles in Table 3. Copies of observed and calculated structure factors are available on request.

Site occupation

Site occupancy refinement was necessary because Al(1) revealed an unusually high electron density. Al(2), Al(3), and Al(4) all have much lower and more equivalent associated electron densities (overall variation about 10%). Al(1) has approximately twice the average of the Al(2), Al(3), and Al(4) values. The occupancy parameters of Al(1) converged to 0.215 Al + 0.135 Ta + 0.15□, very much in accord with the parameters found for Al(1) in dumortierite $\frac{1}{2}$(0.764 Al + 0.236□) by Moore and Araki (1978). The Si(1) and Si(2) sites in the dumortierite structure were found to be the localities of the Sb substitution in holtite. Each Si site splits into a pair of atoms separated by about 0.5 Å, and on refinement each set of occupancy parameters converged to 0.75 Si + 0.25 Sb. The Si atoms are located very close to the fractional coordinates of the two positions occupied by Si in dumortierite. The Sb atoms are shifted towards the Al(1) position to accommodate the longer triangular pyramidal bonds in the SbO_3 groups (average ~1.9 Å). Because the resulting Sb(1)–O(2), and Sb(2)–O(7) interactions (1.078 and 1.035 Å respectively) are too short to allow full occupancy of O(2) and O(7), each oxygen was therefore refined with an occupancy factor of 0.75. The factors obtained for O(2) and O(7), which are determined by the Si to Sb ratio in the Si, Sb(1) and Si, Sb(2) paired sites, are in good agreement with the overall metal occupancy in Al(1) to which O(2) and O(7) are also bonded (although, significantly, for Al(1) this is slightly lower, by 0.05). For this reason there is not the need in holtite (as there is in dumortierite), for OH^- replacement of O^{2+} to provide a reduction in local charge to compensate for the condition of a metal vacancy in Al(1). Because Al(1) must be equally or less occupied than O(2) and O(7), there exists the possibility that any deficiency in local charge due to a relative metal vacancy in Al(1) may be accommodated by OH^- substitution. However, in holtite the effect of the higher charged Ta^{5+} in Al(1) must be considered in the balance. For a particular occupancy of O (and OH^-) in O(2) and O(7) determined by a Si to Sb balance in Si, Sb(1) and Si, Sb(2), any

Table 2. Anisotropic temperature factors for holtite ($\times 10^{-4}$).

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Al 1	0136(2)	0024(2)	0025(2)	0000	-0004(2)	0000
Al 2	0045(3)	0044(3)	0048(3)	0006(2)	-0003(3)	-0004(3)
Al 3	0043(3)	0052(3)	0047(3)	0003(3)	0000(3)	-0002(3)
Al 4	0082(3)	0071(3)	0085(3)	0013(3)	-0010(3)	0000(3)
Si 1	0037(7)	0036(6)	0058(7)	0000	0001(7)	0000
Sb 1	0040(4)	0036(4)	0052(5)	0000	0007(4)	0000
Si 2	0035(4)	0078(5)	0033(4)	-0019(4)	-0005(3)	0013(4)
Sb 2	0043(3)	0032(3)	0028(3)	0011(2)	0009(2)	-0005(3)
O 1	0064(11)	0042(10)	0084(11)	0000	-0008(9)	0000
O 2	0133(17)	0116(16)	0079(15)	0000	0009(14)	0000
O 3	0052(8)	0060(7)	0073(7)	0006(6)	0016(6)	0004(6)
O 4	0062(8)	0063(7)	0076(7)	-0011(6)	0011(6)	-0002(7)
O 5	0051(8)	0074(7)	0057(7)	0003(6)	0011(6)	-0015(6)
O 6	0089(8)	0077(8)	0062(7)	-0007(6)	-0012(6)	0009(7)
O 7	0109(11)	0098(11)	0101(10)	0028(9)	-0016(10)	0001(10)
O 8	0105(12)	0082(11)	0055(11)	0000	-0005(10)	0000
O 9	0083(8)	0053(7)	0055(7)	-0004(6)	0018(6)	0012(7)
O10	0070(12)	0058(11)	0141(12)	0000	-0006(10)	0000
O11	0046(8)	0056(7)	0062(7)	-0008(6)	-0003(6)	-0009(6)
B 1	0029(15)	0026(13)	0068(15)	0000	-0019(12)	0000

Ta^{5+} substitution for Al^{3+} in Al(1) is allied to higher O^{2-} (i.e. lesser OH^-).

The –Al(1)–Al(1)–separation in holtite is an usually short (2.235 Å) for face-sharing octahedra (comparing favourably with 2.350 Å in dumortierite) and relates to the disorder in these face-sharing chains of octahedra. In comparison, the Al(4)–Al(4) separation for face-sharing octahedral pairs in the structure is the more normal distance of 2.600 Å (dumortierite, 2.566 Å).

Table 3. Bond lengths (Å) and angles (°) in holtite.

		Al 1		
2 Al 1 - O 7	1.965	O 7 - O 7	2.777	88.14
1 Al 1 - O 2	1.999	O 7 - O 7	2.777	89.91
2 Al 1 - O 7	1.996	2xO 2 - O 7	2.797	87.85
1 Al 1 - O 2	2.035	2xO 2 - O 7	2.797	89.73
		2xO 7 - O 7	2.825	90.97
		2xO 2 - O 7	2.853	91.12
		2xO 2 - O 7	2.860	91.28
		Al 2		
Al 2 - O 3	1.886	O 5 - O11	2.495	81.90
Al 2 - O11	1.880	O 3 - O11	2.502	81.23
Al 2 - O 9	1.893	O 9 - O11	2.530	82.14
Al 2 - O 1	1.884	O11 - O11	2.517	81.98
Al 2 - O 5	1.926	O 3 - O 5	2.628	87.11
Al 2 - O11	1.957	O 1 - O 5	2.693	89.91
		O 3 - O 9	2.698	91.13
		O 5 - O11	2.738	89.62
		O 9 - O11	2.833	97.34
		O 1 - O 3	2.739	97.0
		O 1 - O11	2.875	99.64
		O 1 - O 9	2.859	98.31
		Al 3		
Al 3 - O 5	1.885	O 5 - O11	2.495	81.57
Al 3 - O11	1.875	O 3 - O11	2.502	82.15
Al 3 - O 6	1.880	O 9 - O11	2.530	82.10
Al 3 - O 9	1.918	O11 - O11	2.517	82.85
Al 3 - O 3	1.933	O 3 - O 5	2.628	87.52
Al 3 - O11	1.934	O 3 - O 6	2.674	89.05
		O 5 - O 9	2.701	90.48
		O 3 - O11	2.752	90.72
		O 5 - O 6	2.738	96.35
		O 9 - O11	2.833	97.80
		O 6 - O11	2.859	99.17
		O 6 - O 9	2.869	98.09
		Al 4		
Al 4 - O 4	1.845	O 8 - O10	2.479	81.49
Al 4 - O 8	1.856	O10 - O10	2.532	78.30
Al 4 - O 4	1.872	O 8 - O10	2.548	80.80
Al 4 - O 6	1.853	O 4 - O10	2.552	81.25
Al 4 - O10	1.941	O 4 - O10	2.552	84.02
Al 4 - O10	2.067	O 4 - O 4	2.674	92.04
		O 4 - O 6	2.725	94.04
		O 6 - O 8	2.743	95.40
		O 4 - O10	2.778	89.59
		O 4 - O 6	2.827	99.74
		O 4 - O 8	2.816	99.07
		O 6 - O10	2.926	100.93

		Si 1		
Si - O 2	1.611	O 3 - O 3	2.672	107.19
2xSi - O 3	1.660	2xO 2 - O 3	2.657	108.61
Si - O 1	1.701	2xO 1 - O 3	2.739	109.14
		O 1 - O 2	2.778	113.93
		Sb 1		
2xSb 1 - O 3	1.904	O 3 - O 3	2.672	89.11
Sb 1 - O 1	1.968	2xO 3 - O 1	2.739	90.01
Sb 1 - Si 1	0.533			
		Si 2		
Si 2 - O 7	1.603	O 4 - O 5	2.682	107.75
Si 2 - O 5	1.649	O 4 - O 6	2.684	106.23
Si 2 - O 4	1.671	O 4 - O 7	2.660	108.64
Si 2 - O 6	1.684	O 5 - O 7	2.679	110.89
		O 5 - O 6	2.738	110.45
		O 6 - O 7	2.736	112.64
		Sb 2		
Sb 2 - O 4	1.952	O 4 - O 6	2.684	85.88
Sb 2 - O 6	1.987	O 5 - O 6	2.738	89.64
Sb 2 - O 5	1.896	O 4 - O 5	2.682	88.35
Sb 2 - Si 2	0.571			
		B1		
2 x B1 - O 8	1.365	2 x O 8 - O 9	2.358	119.0
B1 - O 9	1.371	O 9 - O 9	2.397	121.9

Note: Esd for bond distances, .003 Å; bond angles, 0.1°.

Boron has typically triangular coordination, with an average bond length in the BO_3 triangle of 1.367 Å.

Composition and chemical substitutions

The single crystal refinement results in the formula

$$(Si_{2.25}Sb_{0.75})B[Al_6(Al_{0.43}Ta_{0.27}\square_{0.30})O_{15}(O,OH)_{2.25}].$$

Table 4 compares the formula of holtite derived from the chemical analysis of J. R. Gamble (Pryce, 1971) with the results of the single crystal refinement (both normalized to 17.2500 oxygen atoms). Sb^{5+}, Nb, Fe, Mn, Ti and Be were not considered in the structure refinement. The Sb content derived from the structure refinement is greater than the Sb^{3+} reported in the chemical analysis but less than the combined Sb^{5+}, Sb^{3+} value, suggesting that some Sb^{5+} is present in the structure, but not as much as was originally reported. Otherwise, the overall agreement is satisfactory, except for a major discrepancy in the B content. The B(1) site in holtite behaves normally during the structure refinement, yielding typical temperature factors with the assigned full occupancy. It appears that the chemical analysis for B in Greenbushes holtite is in error and it should be more in agreement with the value obtained for the Kola Peninsular specimen.

In holtite Sb replaces the Si of the original dumortierite structure, not Al. The Sb lone-electron pairs are accommodated in the hexagonal tunnels which otherwise contain the Si tetrahedra and the Al(1) octahedra. The low-Si, high-Sb content of Greenbushes holtite, compared with the high-Si, low-Sb content of the Kola Peninsular material, is explained in terms of the Sb for Si replacement which occurs near Si(1) and Si(2) sites and is accompanied by the introduction of oxygen vacancies.

The overall chemical replacement mechanism cannot be of the form:

$$(OH)^- + Si^{4+} + Al^{3+} = Sb^{3+} + Ta^{5+} + O^{2-}$$

because this does not allow for the introduction of anion vacancies in the structure. Based on an original dumortierite composition of

$$Si_3B[Al_6Al_{0.75}\square_{0.25}O_{15}(O_{2.25}, OH_{0.75})],$$

substitution apparently occurs in two stages. The first stage

$$(1)\quad (OH)^- + Si^{4+} = Sb^{3+} + \square\,(\text{anion}),$$

results in the approximate composition

Table 4. Chemical composition and unit-cell contents of holtite.

	1	2		3	4	5
SiO_2	20.30	26.74	:	Si 9.03	1.17	1.08
Sb_2O_5	4.61	6.49	:	Sb^{5+} 0.76	.10	
Al_2O_3	46.43	43.44	:	Al 24.34	3.16	3.20
Ta_2O_5	11.24	11.70	:	Ta 1.36	0.18	0.15
Nb_2O_5	0.76	0.15	:	Nb 0.16	0.02	
Fe_2O_3	0.27	0.20	:	Fe 0.10	0.01	
MnO	0.05		:	Mn 0.02	0.00	
TiO_2	0.09		:	Ti 0.03	0.00	
BeO	0.05		:	Be 0.05	0.01	
As_2O_5		2.92				
B_2O_3	1.82	4.67	:	B 1.40	0.19	0.50
Sb_2O_3	13.89		:	Sb^{3+} 2.54	0.33	0.43
			:	H 1.12	0.15	
H_2O^+	0.38	1.13	:	Sum 40.91	5.31	5.36
H_2O^-	0.08		:	O 67.02	8.70	8.70
OTHER(*)		2.27	:			
TOTAL	99.97	99.71	:			

(*) Includes MgO, CaO, Na_2O, K_2O, Li_2O, Rb_2O, Cs_2O.

1 Holtite from Greenbushes (Pryce, 1971).

2 Holtite from Kola Peninsula (Voloshin *et al.*, 1976).

3 Unit cell contents calculated from 1, based on D=3.90, V=1136.5 Å^3 (Pryce, 1971).

4 Analysis 3 recalculated to ΣO=8.70.

5 Contents derived from this X-ray analysis, ΣO=8.70.

$$Si_{2.25}Sb_{0.75}B[Al_6(Al_{0.75}\square_{0.25})O_{15}(O_{2.25}\square_{0.75)}].$$

In consideration of the crystal chemistry of Greenbushes holtite revealed by the X-ray study, the second stage appears to be

$$(2)\quad Al^{3+} = \tfrac{3}{5}Ta^{5+} + \tfrac{2}{5}\square\ \text{(cation)}.$$

resulting in a theoretical end composition of

$$Si_{2.25}Sb_{0.75}B[Al_6(Al_{0.43}Ta_{0.19}\square_{0.13}\square_{0.25})O_{15}(O_{2.25}\square_{0.75})].$$

The structure of holtite is essentially the same as that described for dumortierite (Golovastikov, 1965; Moore and Araki, 1978). It is composed of the same three kinds of chains running parallel to *a*. The first is a partly disordered [Al,TaO_3] chain of face-sharing octahedra; the other two types are [Al_4O_{12}] chains, one of which joins to equivalent chains by reflection at the corners of octahedra to form [Al_4O_{11}] sheets. The structure of dumortierite has been discussed elegantly by Moore and Araki (1978) in terms of design on a semi-regular planar net, and the same concept applies for holtite. The main differences between the two structures lie within the hexagonal tunnels formed by the two [Al_4O_{12}] chains, one of which forms the aforementioned sheets, Fig. 1. The Al(1) site, disordered as it is, has partial occupancy of Ta; and the Si tetrahedra in the hexagonal array in the tunnels, which provide the supporting spoke-like bonding to the central octahedra, are partially replaced by SbO_3 groups. The diffuse layers visible on the X-ray films indicate that, although there is an ordering effect in any one individual tunnel, it is not strongly correlated with that of adjacent tunnels. The fact that such an ordering exists in individual tunnels suggests that a complete replacement of SiO_4 rings by SbO_3 rings takes place at ordered intervals, which are probably inter-related to the order of the vacancies in the Al(1) site.

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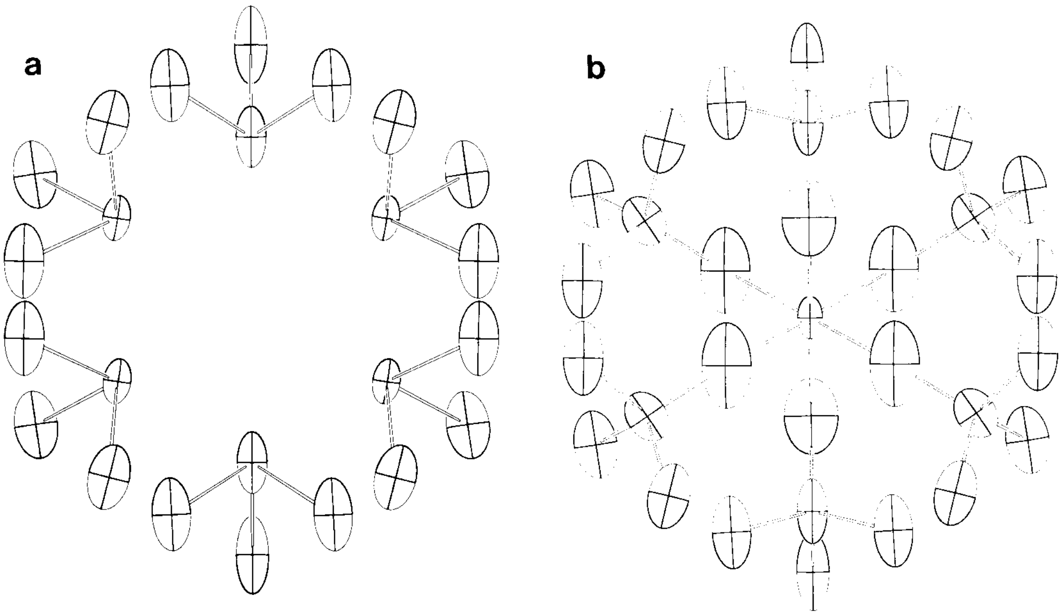

FIG. 1. Disposition of (a) SbO_3 groups, and (b) SiO_4 tetrahedra and the coordinated central (Al,Ta) octahedral site in holtite. ORTEP drawing: projection, (100).

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