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The crystal chemistry of nolanite, $(V,Fe,Ti,Al)_{10}O_{14}(OH)_2$, from Kalgoorlie, Western Australia

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Abstract

Nolanite crystals associated with gold mineralization in the "green leader" deposits of Kalgoorlie, Western Australia, show large local variations in vanadium, titanium and iron contents. The structures have been refined for two compositions with V/ΣM atomic fractions of 0.69 and 0.52. Cell parameters (space group $P6_3mc$, Z = 1) are $a = 5.890(4)$, $c = 9.255(5)$Å and $a = 5.897(2)$, $c = 9.256(3)$Å respectively. Final weighted R factors were 0.039 and 0.049 for 737 and 604 reflections with $F > 4\sigma(F)$, collected on an automatic diffractometer using Mo$K\alpha$ radiation. The structure is based on a closest-packed anion framework with a double hexagonal stacking sequence, ABCBA, *i.e.*, (*ch*) and with cations ordered into 2 tetrahedral and 8 octahedral sites. Valence sum calculations are consistent with nolanite being an oxyhydroxide, $(V,Fe,Ti,Al)_{10}O_{14}(OH)_2$, and with predominately trivalent vanadium and mixed divalent and trivalent iron. Based on a comparison of refined site occupancies and bond lengths for the two structures, plausible cation ordering schemes have been proposed and the intergrain compositional variations have been confirmed to be due to substitutions of the type $V^{3+} \leftrightarrow Fe^{3+}$ and $2(V^{3+},Fe^{3+}) \leftrightarrow (Fe^{2+}+Ti^{4+})$ in the octahedral sites.

Introduction

A new iron vanadate with hexagonal symmetry, $a = 5.854$ and $c = 9.295$Å from Goldfields, Saskatchewan, was first reported by Barnes and Qurashi (1952). The mineral was subsequently described in detail and named nolanite by Robinson *et al.* (1957). Chemical analyses led to inconclusive results regarding the formula because of the difficulty of removing impurities, but Robinson *et al.* used crystal-chemical arguments to propose that the structure was based on a closest-packed anion lattice with 16 anions and 10 cations per unit cell. They suggested unit cell compositions in the range $Fe^{2+}_{2.5}V^{3+}_{1.7}V^{4+}_{5.5}O_{16}$ to $Fe^{2+}_{2.8}V^{3+}_{1.5}V^{4+}_{5.5}O_{16}$. Subsequently their model was confirmed by Hanson (1958) from a single-crystal structure analysis using Weissenberg intensity data. He showed that the structure comprised a closest-packed anion framework with a stacking sequence ABCBA, and with metal ions occupying one 6-fold octahedrally coordinated site and two 2-fold sites, one of octahedral and one of tetrahedral coordination. He proposed an ordering of cations $(V^{4+})_6{}^{[6]}(0.78Fe^{2+} + 0.22V^{3+})_2{}^{[6]}(0.47Fe^{2+} + 0.53V^{3+})_2{}^{[4]}O_{16}$ but noted that the unit cell composition was unbalanced by 1.5 electrons.

A new occurrence of nolanite, in metamorphosed greenstones associated with native gold and tellurides at Kalgoorlie, was reported by Taylor and Radtke (1967). They carried out electron microprobe analyses, supported by wet chemical analysis on purified material which gave the valence states of vanadium as V^{4+} and V^{5+} with $V_2O_5/V_2O_4 = 1.57$, and proposed a unit cell composition based on 18 anions and a cation to anion ratio of 1:2 *i.e.*, $Al_{0.17}Si_{0.05}Ti_{0.27}V_{5.16}Fe_{3.31}Zn_{0.04}Sn_{0.01}O_{18}$. This composition was in conflict with the results of the structure refinement of Hanson (1958).

Our interest in this material arose from a systematic study of högbomite–nigerite polytypes (Grey and Gatehouse, 1979; Gatehouse and Grey, 1982), which we have

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Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Crystal 1						
M(1)	51 (3)	51 (3)	45 (2)	20 (2)	-1 (3)	1 (3)
M(2)	48 (5)	48 (5)	36 (3)	24 (3)	0	0
M(3)	58 (5)	58 (5)	68 (3)	29 (3)	0	0
O(1)	60(20)	60(20)	60(15)	30(10)	0	0
O(2)	64(20)	64(20)	31(16)	32(10)	0	0
O(3)	81(20)	81(20)	96(11)	46(13)	-2(11)	2(11)
O(4)	65(16)	65(16)	62 (9)	36(10)	-4 (8)	4 (8)
Crystal 2						
M(1)	49 (3)	49 (3)	49 (3)	24 (3)	-7 (4)	7 (4)
M(2)	44 (8)	44 (8)	41 (7)	22 (4)	0	0
M(3)	55 (7)	55 (7)	70 (7)	27 (4)	0	0
O(1)	139(35)	139(35)	41(33)	69(17)	0	0
O(2)	-37(22)	-37(22)	112(38)	-19(11)	0	0
O(3)	116(23)	116(23)	89(21)	23(32)	-10(15)	10(15)
O(4)	25(15)	25(15)	95(21)	12(18)	3 (9)	-3 (9)

* The temperature factors are of the form

$$\exp\left[-2\pi^2\left(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{12}hka^*b^* + U_{13}hla^*c^* + 2U_{23}klb^*c^*\right)\right]$$

Am Min Gatehouse et al Table 4 1905 29

TABL

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR CRYSTAL 1

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
0	0	-18	39	42	-3	6	-14	9	9	-1	7	-12	8	8	-2	6	-10	35	35	0	2	-8	17	17
0	1	-18	8	7	-2	6	-14	10	11	0	7	-12	10	10	-1	6	-10	14	13	-1	3	-8	24	24
-1	2	-18	17	15	0	6	-14	23	23	-4	8	-12	18	18	0	6	-10	53	53	0	3	-8	41	43
0	2	-18	22	22	-2	7	-14	8	11	-3	8	-12	9	7	-3	7	-10	11	11	-2	4	-8	61	60
0	1	-17	10	12	0	1	-13	25	25	-2	8	-12	14	15	-2	7	-10	21	21	-1	4	-8	24	23
0	2	-17	24	24	0	2	-13	42	42	-1	8	-12	18	18	-1	7	-10	11	12	0	4	-8	14	14
-1	3	-17	14	14	-1	3	-13	21	22	0	1	-11	11	14	0	7	-10	10	10	-2	5	-8	22	21
-1	4	-17	10	11	-1	4	-13	23	22	0	2	-11	40	39	-4	8	-10	49	48	-1	5	-8	37	35
0	0	-16	33	34	0	4	-13	38	38	-1	3	-11	15	13	-3	8	-10	12	13	0	5	-8	15	17
0	1	-16	9	8	-2	5	-13	23	23	-1	4	-11	13	12	-2	8	-10	28	28	-3	6	-8	32	32
-1	2	-16	15	17	0	5	-13	17	18	0	4	-11	34	34	-1	8	-10	15	16	-2	6	-8	13	13
0	2	-16	12	10	-2	6	-13	34	34	-2	5	-11	11	12	0	8	-10	25	24	-1	6	-8	18	18
-1	3	-16	9	7	-1	6	-13	16	18	0	5	-11	14	13	-4	9	-10	10	9	0	6	-8	42	42
0	3	-16	16	17	-3	7	-13	15	16	-2	6	-11	29	31	-3	9	-10	16	17	-3	7	-8	15	15
-2	4	-16	33	31	-2	7	-13	8	5	-1	6	-11	10	10	0	1	-9	17	16	-2	7	-8	30	31
-1	4	-16	9	8	-1	7	-13	20	19	-3	7	-11	11	11	0	2	-9	34	33	-1	7	-8	18	17
0	4	-16	10	9	0	7	-13	20	19	-1	7	-11	9	10	-1	3	-9	15	14	0	7	-8	15	16
-1	5	-16	14	15	-3	8	-13	16	17	0	7	-11	9	10	-1	4	-9	13	13	-4	8	-8	36	37
0	5	-16	8	5	0	0	-12	26	24	-3	8	-11	9	9	0	4	-9	29	28	-3	8	-8	16	17
0	1	-15	23	24	0	1	-12	13	12	-2	8	-11	24	25	-2	5	-9	12	13	-2	8	-8	11	12
0	2	-15	38	39	-1	2	-12	30	28	-1	8	-11	8	1	0	5	-9	12	11	-1	8	-8	24	24
-1	3	-15	19	21	0	2	-12	27	27	0	8	-11	23	24	-2	6	-9	25	25	0	8	-8	9	11
-1	4	-15	21	21	-1	3	-12	10	11	-4	9	-11	8	9	-1	6	-9	11	10	-4	9	-8	15	15
0	4	-15	34	36	0	3	-12	27	27	0	0	-10	78	77	-3	7	-9	9	10	-3	9	-8	22	24
-2	5	-15	21	21	-2	4	-12	23	22	0	1	-10	17	17	-1	7	-9	8	10	-2	9	-8	12	11
0	5	-15	16	18	-1	4	-12	12	11	-1	2	-10	29	30	0	7	-9	13	11	-1	9	-8	12	12
-2	6	-15	31	32	0	4	-12	22	23	0	2	-10	51	50	-3	8	-9	8	8	0	9	-8	22	23
-1	6	-15	17	18	-2	5	-12	11	11	-1	3	-10	15	15	-2	8	-9	21	21	-5	10	-8	19	19
0	0	-14	34	33	-1	5	-12	24	24	0	3	-10	28	28	0	8	-9	19	19	-4	10	-8	12	11
0	1	-14	8	5	0	5	-12	11	9	-2	4	-10	66	66	-4	9	-9	9	9	-3	10	-8	12	13
-1	2	-14	15	13	-3	6	-12	22	23	-1	4	-10	15	15	-2	9	-9	8	9	0	1	-7	51	49
0	2	-14	12	13	-2	6	-12	19	20	0	4	-10	42	41	-1	9	-9	8	7	0	2	-7	37	35
0	3	-14	14	13	-1	6	-12	9	8	-2	5	-10	13	13	-4	10	-9	17	17	-1	3	-7	38	37
-2	4	-14	29	29	0	6	-12	19	19	-1	5	-10	24	24	0	0	-8	83	81	0	3	-7	6	7
0	4	-14	10	12	-3	7	-12	7	8	0	5	-10	12	12	0	1	-8	29	28	-1	4	-7	38	37
-1	5	-14	12	11	-2	7	-12	22	21	-3	6	-10	19	21	-1	2	-8	49	48	0	4	-7	28	28

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR CRYSTAL 1

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-2	5	-7	39	38	-4	9	-6	7	3	0	4	-4	41	41	-2	8	-3	20	21	-4	9	-2	17	17
-1	5	-7	7	5	0	9	-6	8	4	-2	5	-4	10	9	0	8	-3	19	19	-3	9	-2	30	30
0	5	-7	25	25	-4	10	-6	16	16	-1	5	-4	13	13	-4	9	-3	23	22	-2	9	-2	14	13
-2	6	-7	25	23	-3	10	-6	8	2	0	5	-4	6	5	-2	9	-3	31	32	-1	9	-2	14	12
-1	6	-7	25	26	-2	10	-6	29	29	-3	6	-4	15	14	-1	9	-3	27	27	0	9	-2	25	26
-3	7	-7	23	22	0	1	-5	36	34	-2	6	-4	32	31	-4	10	-3	18	17	-5	10	-2	27	27
-1	7	-7	28	27	0	2	-5	134	132	-1	6	-4	6	2	-3	10	-3	23	23	-4	10	-2	11	9
0	7	-7	28	29	-1	3	-5	32	31	0	6	-4	30	29	-1	10	-3	18	19	-3	10	-2	11	12
-3	8	-7	24	23	0	3	-5	5	7	-2	7	-4	8	9	0	10	-3	14	14	-2	10	-2	15	14
-2	8	-7	18	18	-1	4	-5	25	24	-1	7	-4	6	4	-5	11	-3	24	25	-1	10	-2	13	12
0	8	-7	14	15	0	4	-5	95	94	0	7	-4	7	8	-3	11	-3	16	17	0	10	-2	9	7
-4	9	-7	25	25	-2	5	-5	21	21	-4	8	-4	27	27	0	0	-2	68	67	-5	11	-2	11	11
-3	9	-7	8	5	0	5	-5	24	23	-3	8	-4	6	1	0	1	-2	52	53	-4	11	-2	22	24
-2	9	-7	17	16	-2	6	-5	76	76	-2	8	-4	22	22	-1	2	-2	90	90	-3	11	-2	12	13
-1	9	-7	16	17	-1	6	-5	20	19	-1	8	-4	11	10	0	2	-2	61	59	0	1	-1	13	12
0	9	-7	9	6	-3	7	-5	19	19	0	8	-4	19	19	-1	3	-2	38	38	0	2	-1	79	82
-4	10	-7	14	14	-1	7	-5	15	15	-4	9	-4	8	6	0	3	-2	71	71	-1	3	-1	8	8
-3	10	-7	16	17	0	7	-5	12	14	-3	9	-4	10	8	-2	4	-2	37	38	0	3	-1	11	12
0	0	-6	91	89	-3	8	-5	14	15	0	9	-4	8	6	-1	4	-2	33	32	-1	4	-1	6	6
0	1	-6	6	5	-2	8	-5	56	57	-5	10	-4	11	10	0	4	-2	30	30	0	4	-1	58	58
-1	2	-6	12	11	0	8	-5	51	52	-4	10	-4	17	17	-2	5	-2	29	29	-2	5	-1	10	10
0	2	-6	41	41	-4	9	-5	12	13	-2	10	-4	22	22	-1	5	-2	52	51	-1	5	-1	6	7
-1	3	-6	5	5	-2	9	-5	11	15	0	1	-3	77	76	0	5	-2	23	23	0	5	-1	8	7
0	3	-6	9	9	-1	9	-5	14	13	0	2	-3	62	61	-3	6	-2	46	46	-2	6	-1	45	45
-2	4	-6	68	65	-4	10	-5	47	47	-1	3	-3	71	70	-2	6	-2	20	20	-1	6	-1	7	3
-1	4	-6	6	2	-3	10	-5	13	12	0	3	-3	8	7	-1	6	-2	20	20	-2	7	-1	8	9
0	4	-6	31	30	-1	10	-5	11	11	-1	4	-3	51	51	0	6	-2	22	22	0	7	-1	8	8
-1	5	-6	9	8	-5	11	-5	10	12	0	4	-3	38	38	-3	7	-2	19	18	-2	8	-1	33	34
-3	6	-6	10	10	0	0	-4	48	45	-2	5	-3	41	39	-2	7	-2	38	38	0	8	-1	28	29
-2	6	-6	23	25	0	1	-4	15	13	0	5	-3	49	49	-1	7	-2	19	19	-2	9	-1	7	4
0	6	-6	45	45	-1	2	-4	21	21	-2	6	-3	29	29	0	7	-2	18	20	0	9	-1	9	7
-2	7	-6	6	6	0	2	-4	63	63	-1	6	-3	38	39	-4	8	-2	19	19	-4	10	-1	26	27
-4	8	-6	40	39	-1	3	-4	7	7	-3	7	-3	41	40	-3	8	-2	17	17	0	10	-1	23	24
-3	8	-6	7	1	0	3	-4	15	16	-1	7	-3	27	28	-2	8	-2	12	12	0	1	0	22	24
-2	8	-6	18	19	-2	4	-4	41	38	0	7	-3	25	24	-1	8	-2	31	31	-1	2	0	43	43
0	8	-6	15	17	-1	4	-4	7	6	-3	8	-3	29	28	0	8	-2	11	10	0	2	0	64	66

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR CRYSTAL 1

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-1	3	0	17	18	-2	6	1	45	44	-4	9	2	16	17	-1	10	3	18	19	0	5	5	22	24
0	3	0	32	31	-2	7	1	7	9	-3	9	2	28	30	0	10	3	13	13	-2	6	5	76	76
-2	4	0	172	179	-1	7	1	7	5	-2	9	2	14	14	-5	11	3	24	25	-1	6	5	21	19
-1	4	0	11	11	-3	8	1	7	2	-1	9	2	12	12	-3	11	3	16	17	-3	7	5	20	20
0	4	0	48	49	-2	8	1	34	33	0	9	2	25	26	0	0	4	47	49	-2	7	5	6	6
-2	5	0	11	11	0	8	1	28	28	-5	10	2	27	28	0	1	4	13	13	-1	7	5	15	15
-1	5	0	25	25	-2	9	1	7	5	-4	10	2	11	10	-1	2	4	20	21	0	7	5	13	13
0	5	0	12	13	0	9	1	9	7	-3	10	2	13	12	0	2	4	63	63	-3	8	5	13	15
-3	6	0	24	25	-4	10	1	26	26	-2	10	2	16	14	-1	3	4	9	7	-2	8	5	56	57
-2	6	0	39	39	-3	10	1	7	1	-1	10	2	12	12	0	3	4	16	16	0	8	5	51	52
-1	6	0	6	7	0	10	1	23	23	0	10	2	8	8	-2	4	4	42	40	-4	9	5	13	12
0	6	0	98	99	0	0	2	66	67	-5	11	2	12	11	-1	4	4	7	6	-2	9	5	13	16
-3	7	0	12	9	0	1	2	51	53	-4	11	2	23	24	0	4	4	41	41	-1	9	5	14	13
-2	7	0	15	16	-1	2	2	90	90	-3	11	2	14	12	-2	5	4	10	9	-4	10	5	47	47
-4	8	0	82	84	0	2	2	62	60	0	1	3	75	76	-1	5	4	14	14	-3	10	5	11	12
-2	8	0	29	31	-1	3	2	38	38	0	2	3	61	61	0	5	4	6	5	-5	11	5	14	12
-1	8	0	17	18	0	3	2	71	70	-1	3	3	71	71	-3	6	4	12	14	0	0	6	90	90
0	8	0	26	28	-2	4	2	37	38	0	3	3	7	7	-2	6	4	31	31	0	1	6	5	5
-3	9	0	14	14	-1	4	2	31	32	-1	4	3	51	51	0	6	4	30	31	-1	2	6	11	11
-2	9	0	7	8	0	4	2	30	31	0	4	3	38	38	-2	7	4	8	9	0	2	6	40	39
0	9	0	9	10	-2	5	2	30	29	-2	5	3	41	39	-4	8	4	30	28	-1	3	6	7	5
-5	10	0	13	18	-1	5	2	52	51	0	5	3	49	50	-2	8	4	21	22	0	3	6	10	9
-4	10	0	26	26	0	5	2	23	23	-2	6	3	29	29	-1	8	4	10	10	-2	4	6	66	65
-3	10	0	7	2	-3	6	2	47	46	-1	6	3	40	39	0	8	4	17	19	0	4	6	29	28
-2	10	0	56	59	-2	6	2	20	21	-3	7	3	40	40	-4	9	4	7	6	-1	5	6	9	8
0	10	0	22	22	-1	6	2	21	20	-2	7	3	6	4	-3	9	4	10	8	0	5	6	6	5
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0	1	1	13	12	-3	7	2	19	18	0	7	3	24	23	-4	10	4	15	17	0	6	6	44	45
0	2	1	79	80	-2	7	2	38	38	-3	8	3	28	28	-2	10	4	21	22	-2	7	6	7	6
-1	3	1	8	8	-1	7	2	17	19	-2	8	3	20	20	0	1	5	35	34	0	7	6	8	3
0	3	1	12	12	0	7	2	18	20	0	8	3	20	19	0	2	5	131	132	-4	8	6	41	39
-1	4	1	6	6	-4	8	2	19	19	-4	9	3	21	21	-1	3	5	33	32	-2	8	6	19	18
0	4	1	57	57	-3	8	2	17	16	-2	9	3	30	32	0	3	5	8	7	0	8	6	17	16
-2	5	1	9	10	-2	8	2	13	13	-1	9	3	25	27	-1	4	5	26	24	-4	10	6	16	15
-1	5	1	6	7	-1	8	2	30	32	-4	10	3	18	17	0	4	5	95	94	-2	10	6	29	29
0	5	1	8	8	0	8	2	10	11	-3	10	3	25	23	-2	5	5	20	20	0	1	7	50	49

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR CRYSTAL 1

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
0	2	7	36	37	-1	6	8	17	18	-4	9	9	10	9	0	4	11	34	34	0	1	13	24	25
-1	3	7	37	37	0	6	8	42	42	-2	9	9	10	9	-2	5	11	13	11	0	2	13	41	42
0	3	7	8	7	-3	7	8	16	15	-1	9	9	9	7	0	5	11	13	13	-1	3	13	21	22
-1	4	7	37	37	-2	7	8	31	30	-4	10	9	17	18	-2	6	11	30	30	-1	4	13	22	22
0	4	7	28	29	-1	7	8	18	17	0	0	10	76	76	-1	6	11	11	10	0	4	13	36	38
-2	5	7	38	38	0	7	8	17	16	0	1	10	17	17	-3	7	11	12	12	-2	5	13	21	23
0	5	7	24	25	-4	8	8	36	37	-1	2	10	30	30	-1	7	11	10	9	0	5	13	18	17
-2	6	7	24	24	-3	8	8	17	16	0	2	10	50	51	0	7	11	9	9	-2	6	13	35	34
-1	6	7	25	26	-2	8	8	11	11	-1	3	10	16	16	-3	8	11	9	9	-1	6	13	18	18
-3	7	7	22	22	-1	8	8	23	24	0	3	10	28	28	-2	8	11	23	24	-3	7	13	16	16
-2	7	7	9	7	0	8	8	11	11	-2	4	10	65	65	0	8	11	23	24	-1	7	13	19	19
-1	7	7	27	27	-4	9	8	14	15	-1	4	10	15	15	-4	9	11	8	8	0	7	13	20	20
0	7	7	28	29	-3	9	8	24	24	0	4	10	42	42	0	0	12	25	26	-3	8	13	17	17
-3	8	7	24	23	-2	9	8	10	11	-2	5	10	12	13	0	1	12	12	12	0	0	14	34	34
-2	8	7	19	20	-1	9	8	13	12	-1	5	10	23	24	-1	2	12	29	28	0	1	14	7	5
0	8	7	17	16	0	9	8	22	22	0	5	10	10	12	0	2	12	28	27	-1	2	14	13	14
-4	9	7	26	25	-5	10	8	20	20	-3	6	10	22	21	-1	3	12	12	11	0	2	14	11	11
-2	9	7	15	16	-4	10	8	10	10	-2	6	10	36	36	0	3	12	26	26	0	3	14	14	14
-1	9	7	18	17	-3	10	8	14	13	-1	6	10	13	13	-2	4	12	23	24	-2	4	14	28	30
0	9	7	7	6	0	1	9	16	16	0	6	10	53	52	-1	4	12	10	10	-1	4	14	9	6
-4	10	7	15	15	0	2	9	33	34	-3	7	10	11	12	0	4	12	23	23	0	4	14	11	10
-3	10	7	16	17	-1	3	9	14	14	-2	7	10	20	21	-2	5	12	11	11	-1	5	14	12	11
0	0	8	82	82	0	3	9	7	5	-1	7	10	13	12	-1	5	12	24	24	-2	6	14	9	10
0	1	8	28	28	-1	4	9	13	13	0	7	10	9	10	0	5	12	10	9	-1	6	14	7	5
-1	2	8	48	48	0	4	9	28	29	-4	8	10	46	48	-3	6	12	22	22	0	6	14	22	24
0	2	8	16	15	-2	5	9	14	13	-3	8	10	12	13	-2	6	12	20	20	-2	7	14	11	11
-1	3	8	24	24	-1	5	9	8	4	-2	8	10	28	28	-1	6	12	9	8	0	1	15	21	24
0	3	8	44	43	0	5	9	13	12	-1	8	10	15	16	0	6	12	21	21	0	2	15	37	39
-2	4	8	61	61	-2	6	9	26	25	0	8	10	26	25	-3	7	12	9	8	-1	3	15	21	21
-1	4	8	22	23	-1	6	9	10	10	-4	9	10	9	9	-2	7	12	21	21	-1	4	15	20	21
0	4	8	15	14	-3	7	9	10	11	-3	9	10	15	16	-1	7	12	9	8	0	4	15	34	36
-2	5	8	22	21	-1	7	9	9	10	-2	9	10	9	9	0	7	12	11	9	-2	5	15	20	22
-1	5	8	36	35	0	7	9	10	11	0	1	11	13	13	-4	8	12	18	19	0	5	15	17	17
0	5	8	17	17	-3	8	9	8	8	0	2	11	38	39	-3	8	12	9	8	-2	6	15	32	32
-3	6	8	31	32	-2	8	9	22	21	-1	3	11	14	13	-2	8	12	14	15	-1	6	15	16	18
-2	6	8	14	13	0	8	9	19	19	-1	4	11	10	11	-1	8	12	18	18	0	0	16	31	33

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR CRYSTAL 1

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
0	1	16	9	8	0	3	16	17	17	0	5	16	8	5	-1	3	17	13	14	0	1	18	10	7
-1	2	16	15	17	-2	4	16	29	29	0	1	17	12	12	-1	4	17	14	11	-1	2	18	15	16
0	2	16	10	11	0	4	16	11	10	0	2	17	24	24	0	0	18	40	41	0	2	18	21	23
-1	3	16	8	7	-1	5	16	12	14															

TABLE

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR CRYSTAL 2 O

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-1	2	-18	21	17	0	1	-12	10	10	-1	5	-10	22	22	-1	5	-8	36	37	-1	9	-7	15	16
0	2	-18	20	21	-1	2	-12	28	26	-3	6	-10	16	20	0	5	-8	19	19	-3	10	-7	19	17
0	2	-17	21	23	0	2	-12	26	26	-2	6	-10	36	36	-3	6	-8	32	34	-2	10	-7	13	2
0	1	-16	13	8	-1	3	-12	11	9	0	6	-10	52	53	-1	6	-8	18	19	0	1	-6	8	6
-1	2	-16	20	20	0	3	-12	27	25	-2	7	-10	21	21	0	6	-8	43	41	-1	2	-6	10	13
0	3	-16	23	19	-2	4	-12	23	25	-1	7	-10	15	10	-3	7	-8	17	17	0	2	-6	41	39
-2	4	-16	32	30	0	4	-12	25	22	-4	8	-10	51	48	-2	7	-8	31	32	0	3	-6	9	10
-2	5	-16	17	6	-1	5	-12	22	22	-2	8	-10	27	28	-1	7	-8	16	17	-2	4	-6	68	69
-1	5	-16	18	17	-3	6	-12	21	21	-1	8	-10	18	16	0	7	-8	18	15	0	4	-6	29	29
0	1	-15	19	21	-2	6	-12	16	19	0	8	-10	27	25	-4	8	-8	32	36	0	5	-6	11	6
0	2	-15	41	39	0	6	-12	21	21	-3	9	-10	19	17	-3	8	-8	20	17	-2	6	-6	25	24
-1	3	-15	23	18	-3	7	-12	14	8	-2	9	-10	13	9	-2	8	-8	14	11	0	6	-6	46	48
-1	4	-15	21	19	-2	7	-12	17	19	0	1	-9	16	18	-1	8	-8	26	26	-3	7	-6	13	4
0	4	-15	35	35	-4	8	-12	25	20	0	2	-9	32	32	-4	9	-8	16	14	-2	7	-6	12	5
-2	5	-15	19	20	-2	8	-12	16	15	-1	3	-9	12	16	-3	9	-8	22	26	-4	8	-6	42	43
-2	6	-15	29	32	-1	8	-12	15	17	-1	4	-9	13	15	-1	9	-8	16	13	-2	8	-6	19	19
0	3	-14	14	15	0	1	-11	12	16	0	4	-9	29	28	0	9	-8	22	23	0	8	-6	17	17
-2	4	-14	29	29	0	2	-11	43	41	-2	5	-9	17	15	-5	10	-8	18	22	-4	10	-6	15	16
-1	5	-14	17	13	-1	3	-11	13	14	-2	6	-9	25	25	-3	10	-8	16	14	-2	10	-6	31	31
-2	6	-14	14	11	-1	4	-11	11	14	-1	6	-9	13	13	0	1	-7	48	48	0	1	-5	32	33
-1	6	-14	13	6	0	4	-11	36	36	-3	7	-9	15	13	0	2	-7	36	38	0	2	-5	131	133
0	6	-14	20	24	-2	5	-11	14	14	-1	7	-9	13	12	-1	3	-7	35	35	-1	3	-5	30	32
-2	7	-14	16	12	0	5	-11	15	12	0	7	-9	14	13	-1	4	-7	35	36	0	3	-5	9	8
0	1	-13	26	27	-2	6	-11	32	33	-3	8	-9	14	11	0	4	-7	27	30	-1	4	-5	25	24
0	2	-13	42	41	-1	6	-11	16	11	-2	8	-9	24	20	-2	5	-7	37	37	0	4	-5	96	95
-1	3	-13	23	23	-3	7	-11	13	11	0	8	-9	16	19	0	5	-7	22	23	-2	5	-5	21	20
-1	4	-13	21	24	-2	8	-11	28	27	-4	10	-9	17	17	-2	6	-7	24	25	0	5	-5	24	24
0	4	-13	38	36	0	8	-11	23	27	0	1	-8	29	29	-1	6	-7	24	24	-2	6	-5	76	77
-2	5	-13	24	25	-4	9	-11	13	10	-1	2	-8	52	50	-3	7	-7	19	20	-1	6	-5	20	20
0	5	-13	17	18	0	1	-10	15	16	0	2	-8	17	15	-1	7	-7	27	27	-3	7	-5	17	20
-2	6	-13	33	33	-1	2	-10	30	28	-1	3	-8	25	26	0	7	-7	29	29	-1	7	-5	15	15
-1	6	-13	18	19	0	2	-10	51	49	0	3	-8	47	45	-3	8	-7	24	23	0	7	-5	12	13
-3	7	-13	15	17	-1	3	-10	15	15	-2	4	-8	58	59	-2	8	-7	23	20	-3	8	-5	13	16
-1	7	-13	21	20	0	3	-10	24	26	-1	4	-8	23	24	0	8	-7	17	16	-2	8	-5	58	58
0	7	-13	19	21	-2	4	-10	66	65	0	4	-8	12	13	-4	9	-7	26	26	0	8	-5	50	53
-3	8	-13	15	18	0	4	-10	39	41	-2	5	-8	20	21	-2	9	-7	13	14	-4	9	-5	14	13

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-2	9	-5	14	17	0	5	-3	51	50	-4	8	-2	23	20	-1	5	0	26	26	-1	3	2	40	40
-1	9	-5	16	15	-2	6	-3	30	28	-3	8	-2	15	16	-3	6	0	27	26	0	3	2	71	70
-4	10	-5	47	48	-1	6	-3	41	40	-2	8	-2	16	14	-2	6	0	41	40	-2	4	2	38	38
-5	11	-5	19	14	-3	7	-3	40	41	-1	8	-2	31	32	0	6	0	100	101	-1	4	2	31	32
0	1	-4	14	13	-1	7	-3	30	29	-4	9	-2	18	15	-2	7	0	18	16	0	4	2	29	30
-1	2	-4	21	23	0	7	-3	25	24	-3	9	-2	31	29	-1	7	0	13	8	-2	5	2	28	27
0	2	-4	61	62	-3	8	-3	30	30	-2	9	-2	18	16	0	7	0	14	12	-1	5	2	52	51
-1	3	-4	11	10	-2	8	-3	22	20	0	9	-2	27	25	-4	8	0	86	86	0	5	2	25	25
0	3	-4	18	18	0	8	-3	20	19	-5	10	-2	28	28	-2	8	0	34	31	-3	6	2	49	46
-2	4	-4	38	39	-4	9	-3	22	22	-4	10	-2	14	10	-1	8	0	18	20	-2	6	2	21	20
-1	4	-4	9	7	-2	9	-3	32	32	-2	10	-2	15	15	0	8	0	26	29	-1	6	2	22	22
0	4	-4	41	41	-1	9	-3	24	27	-5	11	-2	16	13	-3	9	0	18	16	0	6	2	21	23
-2	5	-4	11	8	-4	10	-3	21	17	-4	11	-2	26	23	-5	10	0	20	20	-3	7	2	21	21
-1	5	-4	15	15	-3	10	-3	27	24	-3	11	-2	16	11	-4	10	0	28	27	-2	7	2	38	38
0	5	-4	11	9	-1	10	-3	19	20	0	1	-1	12	12	-2	10	0	62	61	-1	7	2	16	18
-3	6	-4	16	17	-5	11	-3	27	26	0	2	-1	82	82	0	10	0	20	24	0	7	2	16	18
-2	6	-4	31	31	-3	11	-3	16	18	-1	3	-1	10	8	-3	11	0	14	7	-4	8	2	21	19
-1	6	-4	11	4	0	1	-2	51	52	0	3	-1	9	10	0	1	1	12	12	-3	8	2	17	17
0	6	-4	30	30	-1	2	-2	89	89	-1	4	-1	9	8	0	2	1	83	84	-2	8	2	17	13
-3	7	-4	11	7	0	2	-2	62	61	0	4	-1	61	59	-1	3	1	9	8	-1	8	2	33	32
-4	8	-4	28	28	-1	3	-2	40	40	-2	6	-1	47	46	0	3	1	9	10	0	8	2	13	11
-2	8	-4	21	22	0	3	-2	72	70	-2	7	-1	14	7	-1	4	1	9	8	-4	9	2	16	15
-1	8	-4	15	13	-2	4	-2	39	39	-2	8	-1	37	35	0	4	1	60	60	-3	9	2	28	30
0	8	-4	19	19	-1	4	-2	32	31	0	8	-1	30	29	-2	5	1	12	12	-2	9	2	15	16
-3	9	-4	12	10	0	4	-2	30	32	-4	10	-1	26	27	-2	6	1	45	47	-1	9	2	16	14
-5	10	-4	16	14	-2	5	-2	27	27	0	10	-1	25	25	-3	7	1	11	6	0	9	2	25	26
-4	10	-4	22	16	-1	5	-2	51	51	-4	11	-1	14	5	-2	7	1	11	7	-5	10	2	27	27
-2	10	-4	22	23	0	5	-2	27	26	0	1	0	23	25	-3	8	1	13	5	-2	10	2	15	14
0	1	-3	78	79	-3	6	-2	48	46	-1	2	0	44	44	-2	8	1	33	36	-5	11	2	13	13
0	2	-3	60	60	-2	6	-2	21	22	0	2	0	66	67	0	8	1	27	30	-4	11	2	26	24
-1	3	-3	71	72	-1	6	-2	20	21	-1	3	0	17	17	-4	10	1	28	28	0	1	3	78	79
0	3	-3	9	10	0	6	-2	23	23	0	3	0	32	32	0	10	1	26	26	0	2	3	61	61
-1	4	-3	53	53	-3	7	-2	22	21	-2	4	0	180	182	0	0	2	65	68	-1	3	3	72	71
0	4	-3	37	37	-2	7	-2	39	37	-1	4	0	13	13	0	1	2	52	52	0	3	3	8	9
-2	5	-3	42	41	-1	7	-2	16	18	0	4	0	51	49	-1	2	2	90	89	-1	4	3	53	53
-1	5	-3	10	6	0	7	-2	18	18	-2	5	0	16	15	0	2	2	62	60	0	4	3	37	38

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR CRYSTAL 2

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-2	5	3	42	41	-4	8	4	28	27	-2	6	6	25	26	0	4	8	12	14	-2	9	9	13	11
-1	5	3	10	6	-2	8	4	20	22	0	6	6	49	48	-2	5	8	21	21	-4	10	9	18	17
0	5	3	50	49	-1	8	4	14	13	-4	8	6	39	43	-1	5	8	37	37	0	0	10	77	78
-2	6	3	28	29	0	8	4	16	19	-2	8	6	22	20	0	5	8	21	19	0	1	10	16	15
-1	6	3	40	40	-4	10	4	17	16	0	8	6	20	19	-3	6	8	33	34	-1	2	10	25	28
-3	7	3	40	40	-2	10	4	19	22	-4	10	6	18	17	-2	6	8	12	13	0	2	10	51	49
-1	7	3	31	29	0	1	5	33	33	-2	10	6	31	32	-1	6	8	21	19	-1	3	10	14	15
0	7	3	23	25	0	2	5	133	133	0	1	7	47	48	0	6	8	39	41	0	3	10	26	26
-3	8	3	30	29	-1	3	5	32	31	0	2	7	36	36	-3	7	8	19	17	-2	4	10	65	66
-2	8	3	19	21	-1	4	5	24	24	-1	3	7	35	35	-2	7	8	31	32	-1	4	10	13	14
0	8	3	21	19	0	4	5	97	95	-1	4	7	36	36	-1	7	8	19	17	0	4	10	41	41
-4	9	3	21	22	-2	5	5	19	20	0	4	7	29	29	0	7	8	15	15	-2	5	10	12	11
-2	9	3	31	32	0	5	5	23	24	-2	5	7	37	37	-4	8	8	35	36	-1	5	10	21	23
-1	9	3	27	27	-2	6	5	76	77	0	5	7	25	23	-3	8	8	17	17	0	5	10	11	12
-4	10	3	19	17	-1	6	5	17	20	-2	6	7	23	23	-2	8	8	13	12	-3	6	10	16	20
-3	10	3	24	24	-3	7	5	17	20	-1	6	7	26	25	-1	8	8	26	26	-2	6	10	33	35
-1	10	3	20	20	-1	7	5	19	16	-3	7	7	18	20	0	8	8	13	11	-1	6	10	12	13
0	10	3	15	14	0	7	5	13	14	-2	7	7	11	6	-3	9	8	26	26	0	6	10	51	53
-5	11	3	25	25	-3	8	5	13	16	-1	7	7	26	27	-1	9	8	15	13	-2	7	10	17	21
-3	11	3	15	18	-2	8	5	57	58	0	7	7	29	29	0	9	8	23	24	-1	7	10	12	10
0	0	4	44	43	0	8	5	52	53	-3	8	7	22	23	-5	10	8	19	22	-4	8	10	46	49
0	1	4	13	13	-1	9	5	15	15	-2	8	7	19	19	-3	10	8	14	14	-2	8	10	28	27
-1	2	4	22	23	-4	10	5	48	48	0	8	7	17	15	0	1	9	19	17	-1	8	10	15	16
0	2	4	63	62	-3	10	5	15	14	-4	9	7	25	26	0	2	9	32	32	0	8	10	23	25
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H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
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