What Is the True Space Group of Weberite?

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Analysis of X-ray diffraction evidence obtained from a single crystal of natural weberite, Na_2MgAlF_7, at room temperature and at $-140°C$ rules out all space groups but two, the noncentrosymmetric $Imm2$ (originally proposed by Byström) and $I2_12_12_1$. However, comparison of structure refinements in these two groups and in $Imma$ shows that the departure from centrosymmetry is so slight, and the positional and thermal parameters of some of the F atoms in the $Imm2$ and $I2_12_12_1$ refinements are so highly correlated, that the descriptions of the weberite structure in the three space groups must be regarded as practically indistinguishable. In the absence of a proof of achirality Byström's space group assignment is provisionally accepted as valid, and Na_2MgAlF_7 is considered as isostructural with the recently refined Na_2NiFeF_7.

It took over thirty years for Byström's (*1*) determination of the crystal structure of weberite, Na_2MgAlF_7, to be repeated by modern techniques. Then, by a curious coincidence, *two* determinations were reported in the same year, both designed to verify Byström's space group assignment: one (GT) on the type material from Ivigtut (*2*) and another (HVBDT) on a synthetic analog, Na_2NiFeF_7, assumed to be isostructural (*3*). It is therefore ironical that the two investigations should disagree in their conclusions and that oddly enough the work on Na_2NiFeF_7 should *confirm* Byström's *Imm2*, while the work on weberite itself resulted in a reassignment to *Imma*.

The history of the crystallography of weberite and its synthetic analogs has been briefly reviewed elsewhere (*4*), so only detail directly relevant to our present purpose will be repeated in the following. All the reports agree that weberite is orthorhombic and that the systematic absences are $h + k + l = 2n + 1$, i.e., the unit cell is body centered. The nine body-centered orthorhombic space groups and their relations are presented in Tables I and II; Table I also shows the critical diagnostic classes of reflections allowed by the individual space groups. The reflections $h0l$ and $0kl$ with l odd (designation of axes as in Table III) have been observed for both weberite and Na_2NiFeF_7, many of them strong or very strong. This rules out $Iba2$, $Ibam$, and $Ibca$ as possible space groups. The choice of the correct space group then depends on the observability of $hk0$ reflections with h odd. If these reflections are absent, the space group is $Imma$ or its noncentrosymmetric subgroup $I2mb$. If they are present, the space group is one of $Immm$, $Imm2$, $I222$, and $I2_12_12_1$. GT reported as observed systematic absences hkl with $h + k + l = 2n + 1$ and $hk0$ with $h + k = 2n + 1$, without

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General condition: $h + k + l = 2n$
Supplementary conditions:

None	$I222$, $I2_12_12_1$, $Imm2$, $Immm$
$hk0$ $(h = 2n)$	$I2mb$, $Imma$
$h0l$ $(h = 2n)$ } $0kl$ $(k = 2n)$ }	$Iba2$, $Ibam$
$hk0$ $(h = 2n)$ } $h0l$ $(l = 2n)$ } $0kl$ $(k = 2n)$ }	$Ibca$

commenting on the presence or absence of the $hk0$ class with h odd, and they interpreted their E statistic as favoring a centrosymmetric space group. This they took as an indication that the space group of weberite is $Imma$ and refined the structure accordingly (Table III). HVBDT, on the other hand, who had set out expressly to look for the presence of the diagnostic reflections, did observe a number of $hk0$ with h odd, all of them very weak. They thus concluded that the applicable space group was $Imm2$ and refined the structure of Na_2NiFeF_7 in this space group to an R of 0.029.

The conclusion from these results is that either weberite and Na_2NiFeF_7 have different space groups, even though the structures are essentially the same, or that the space group of both compounds is $Imm2$, but the $hk0$ reflections with h odd are even weaker in weberite than in Na_2NiFeF_7 and thus likely to escape detection in a routine intensity collection by automatic diffractometry, i.e. the $hk0$ reflections simulate the systematic absences of $Imma$ and $I2mb$. A third possibility, not mentioned by GT and HVBDT, is that Byström's structure is not the only one which gives a chemically reasonable atomic arrangement and a low R, and that another structure (or other structures) exists which is as compatible or more so with the observed diffraction intensities.

In view of the continuing interest in fluo-

rides and oxides with structures of the weberite type (the former because of their magnetic properties, the latter as structural alternatives to $A_2B_2O_7$ pyrochlores) it seemed worthwhile to reexamine the structure of the mineral afresh. It was clear that, to resolve the existing ambiguities, a special effort would have to be made to determine whether or not $hk0$ reflections with h odd can be observed.

Routine intensity collection (Table III) from a weberite crystal of good quality (for details, see Experimental) confirmed the orthorhombic body-centered unit cell and produced several very weak $hk0$ reflections with h odd and $I > 2\sigma(I)$. The existence of this class of reflections was confirmed by slow scans across some of them (a sample scan is shown in Fig. 1). The routine intensity collection and the slow scans were repeated at $-140°C$ with much the same results.

The presence of the diagnostic reflections points to one of the four space groups $Immm$, $Imm2$, $I222$, and $I2_12_12_1$. A resonant-circuit test for piezoelectricity gave a negative, and thus inconclusive, result both at room and liquid-nitrogen temperatures. Looking for evidence of chirality by optical measurements seemed unpromising, as demonstration of optical activity in biaxial crystals is notoriously difficult. Predictably, a three-dimensional Patterson map was

TABLE II

SUBGROUP RELATIONS AMONG THE NINE
ORTHORHOMBIC BODY-CENTERED SPACE GROUPS[a]

Group	Maximum subgroup with the same lattice translations				
	$I222$	$I2_12_12_1$	$Imm2$	$Iba2$	$Ima2$
$Immm$	x		x		
$Ibam$	x			x	x
$Ibca$		x		x	
$Imma$		x	x		x

[a] Disregarding orientation; standard settings.

TABLE III

CRYSTAL DATA AND DETAILS OF REFINEMENT OF THE STRUCTURE OF IVIGTUT WEBERITE AND Na_2NiFeF_7[a]

| | Weberite[b] | | | Na_2NiFeF_7 |
| | GT (2) | This work | | HVBDT (3) |
	R.T.	R.T.	−140°C	R.T.
Radiation	Mo $K\alpha$[c]	Mo $K\alpha$		Mo $K\alpha$
μ_{Mo} (cm^{-1})	8.0	7.8		63
a (Å)	7.060(1)	7.051(1)	7.050(4)	7.245(1)
b (Å)	10.000(1)	9.968(2)	9.979(6)	10.320(1)
c (Å)	7.303(1)	7.285(1)	7.297(3)	7.458(1)
V (Å^3)	515.6(2)	512.1(3)	513.4(8)	557.6(2)
θ range (°)	2–30	2–25		6–31
Total no. of reflections	1812	1016	1376	1355
Refinement in $Imma$				
No. of unique reflections	425	306	244	
R; R_w	0.036;—	0.019; 0.018	0.021; 0.020	
Refinement in $Imm2$				
No. of unique reflections		313	254	527[d]
R; R_w		0.019; 0.017	0.021; 0.021	0.029; 0.029[e]
Refinement in $I2_12_12_1$				
No. of unique reflections		510	410	
R : R_w		0.021; 0.021	0.022; 0.024	
Refinement in $I2mb$				
No. of unique reflections		551	441	
R; R_w		0.020; 0.020	0.021; 0.020	

[a] The uncertainties quoted are single esd's in units of the last place. The number of unique reflections is that used in the refinement; the numbers quoted for GT and for this work are for $I > 1\sigma(I)$.

[b] Na_2MgAlF_7 (taken as completely ordered, see text). FW = 230.25.

[c] Cu $K\alpha$ was used in the determination of the unit-cell dimensions.

[d] Includes 14 reflections with $I < 3\sigma(I)$.

[e] $R = R_w = 0.027$ when the 14 reflections with $I < 3\sigma(I)$ are omitted.

compatible with Byström's structure but insensitive to differences between the possible space groups, and application of direct
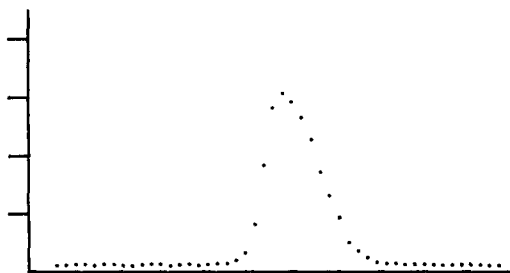
FIG. 1. Profile of one of the diagnostic $hk0$ reflections (530) of weberite at room temperature (superposition of ten slow θ scans, arbitrary counts scaling).

methods to this crystal of isoelectronic atoms resulted in merely reproducing the features of the Patterson map.

Keeping in mind the chemical and electrostatic reasonableness of Byström's structure and the very low R values obtained in the GT and HVBDT refinements based essentially on Byström's model, there appeared no obvious way of establishing the possible existence of an alternative, homometric or otherwise, chemically reasonable structure in these circumstances. The problem then resolved itself into a comparison of refinements of the Byström model in the admissible space groups. Because of the extreme weakness

of the observed $hk0$ reflections with h odd $Imma$ and $I2mb$ were also included in the comparison.

The six space groups are related as follows: $I222 \subset Immm \supset Imm2 \subset Imma \supset (I2mb, I2_12_12_1)$. However, although $Imm2$ is a subgroup of $Immm$, the Byström structure cannot be described in the latter without having to place Na and Mg in the same equipoint (and similarly Na and Al). This rules out $Immm$ and $I222$. Refinements in the remaining four space groups are com-pared in Tables III and IV. It is seen that the differences between corresponding refined fractional coordinates are almost all within three esd's; some of the differences outside these limits, e.g., $x(Imm2)$ and $x(I2mb)$, can be traced to the necessity of having to fix one of the coordinates at a particular (arbitrary) value. Moreover, the refined coordinate values in $Imma$ are indistinguishable, within two esd's, from those reported by GT; in $Imm2$ they are reasonably similar to those of Na_2NiFeF_7 if allowance is made

TABLE IV

REFINEMENT OF THE WEBERITE STRUCTURE IN DIFFERENT SPACE GROUPS: POSITIONAL PARAMETERS[a]

Atom		$Imm2$ + (000)			$I2_12_12_1$ − (0¼¼)			$Imma$ − (0¼¼)			$I2mb$ − (0¼¼)	
Na(1)	4(c)	2492(8)	6(8)	4(b)	¼		4(d)	¼		4(b)	2544(6)	−8(8)
		0			0003(7)	−2(5)		0			0	
		4986(11)	−22(13)		¼			¼			4954(7)	4(10)
Na(2)	4(d)	0		4(a)	−0027(6)	−23(6)	4(a)	0		4(a)	[0]	
		2534(5)	−10(6)		¼			¼			¼	
		7503(10)	−24(14)		¼			¾			¾	
Mg	4(c)	2499(6)	−11(7)	4(b)	¼		4c)	¼		4(b)	2522(5)	−2(7)
		0			0006(5)	−3(5)		0			0	
		[0]			0			0			−0010(5)	2(8)
Al	4(d)	0		4(a)	0000(4)	−11(4)	4(b)	0		4(a)	0025(6)	−11(8)
		2499(3)	13(4)		¼			¼			¼	
		2503(7)	−12(11)		¼			¼			¼	
F(1)	2(a)	0		4(c)	0		4(e)	0		4(b)	0043(7)	−30(10)
		0			0			0			0	
		8844(9)	59(11)		8863(1)	−2(2)		8863(2)	−1(3)		8864(1)	−2(2)
F(2)	2(b)	0										
		¼										
		6119(8)	58(12)									
F(3)	4(d)	0		8(d)	−0016(6)	−15(5)	8(h)	0		8(c)	0042(7)	−19(10)
		1663(6)	−13(5)		1648(1)	−4(1)		1648(1)	−4(1)		1649(1)	−5(1)
		4700(8)	52(10)		4709(1)	4(2)		4709(1)	4(2)		4709(1)	4(2)
F(4)	4(d)	0										
		3367(6)	−4(5)									
		0282(8)	43(11)									
F(5)	8(e)	1838(5)	24(4)	8(d)	1830(3)	8(3)	16(j)	1841(1)	4(1)	8(c)	1874(6)	1(9)
		1378(4)	−6(4)		1359(3)	3(3)		1374(1)	0(1)		1376(3)	6(3)
		1805(7)	40(10)		1818(4)	1(6)		1804(1)	0(1)		1813(4)	9(6)
F(6)	8(e)	3156(4)	17(5)	8(d)	3150(3)	−2(4)				8(c)	3192(6)	−7(9)
		1371(4)	6(4)		1388(3)	−3(3)					1372(3)	−5(3)
		8198(8)	38(9)		8209(4)	0(6)					1794(4)	3(6)

[a] Nonspecial parameter values have been multiplied by 10^4. Values in square brackets are *defined*. The first entry for each parameter refers to the room-temperature structure; the second entry is the low-temperature value presented as deviation from the room-temperature value. The values in parentheses are the single esd's (in units of the last place) for the room-temperature and the low-temperature values, respectively. The room-temperature entries may be compared directly with the HVBDT values for Na_2NiFeF_7. To compare with GT, add (0¼¼) to the $Imma$ values for room temperature and find symmetry-equivalent position where required. Note that Na(1) and Na(2) are interchanged and that our F(1) + F(2) → F(1)$_{GT}$, F(3) + F(4) → F(2)$_{GT}$, and F(5) + F(6) → F(3)$_{GT}$.

for the constant difference in z between the two refinements (which is due to the indefiniteness of the reference point) and for the differences in the effective ionic sizes.

The thermal parameters U_{ij} (Table V) vary little from one refinement to another, although the variation (and uncertainty) in the cross terms is greater than in the U_{ii} terms. The reduction on cooling is appreciable for some of the U_{ij} but statistically insignificant for others. The largest difference between refinements is shown by U_{23} of Na(2), which assumes a large negative value in $Imm2$. Our refinement in $Imma$ reproduces, qualitatively, the thermal parameters of GT. In particular, U_{13} of Na(1) and U_{23} of F(5) are both negative, and U_{33} of Na(1) is by far the largest of all the U_{ij}. A similar qualitative agreement is observed for our refinement in $Imm2$ and the HVBDT B_{ij} values of Na_2NiFeF_7, except that for Na_2NiFeF_7 only the *isotropic* temperature factor of F(4) was refined because of the tendency of the B_{ij} of F(4) to assume negative values.

Examination of the correlation matrix elements for the individual refinements (Table VI) reveals that for F(5) and F(6), which are in the same equipoint in $Imma$ but in separate general equipoints in the other three space groups, the positional and thermal parameters are highly correlated. Similarly, strong correlations exist between the positional (and to a lesser extent the thermal) parameters of F(1) and F(2), and of F(3) and F(4), in $Imm2$, where these atoms occupy separate equipoints.

The conclusion thus is that the space groups compatible with the evidence and symmetry arguments presented here are the noncentrosymmetric $Imm2$ and $I2_12_12_1$. Furthermore, since $hk0$ reflections with h odd were unmistakably observed for Na_2NiFeF_7, these two space groups are the applicable alternatives also for this compound. Na_2NiFeF_7 should therefore be regarded as isostructural with weberite, with

the residual uncertainty that the one might belong to $Imm2$ and the other to $I2_12_12_1$ (the last-named space group not considered as a possibility in HVBDT). However, the existence of the strong F(5)–F(6), etc., correlations, the weakness of the $hk0$ reflections with h odd, and the indifference of R to the choice of space group all indicate that the difference between the noncentrosymmetric description and the centrosymmetric description in $Imma$, which results on ignoring the existence of the critical reflections, is very slight. The atomic positions resulting from the refinements in the three noncentrosymmetric groups are very close to those of the $Imma$ model: the deviations from the respective special values in $Imma$ are within three esd's for 19 of the 31 positional parameters of refinement. Much the same result applies to the refinements in $I2mb$.

Until conclusive evidence of chirality has been presented, we shall regard the intuitively more natural $Imm2$ as the valid space group symmetry of weberite. The metal–fluorine distances resulting from the above refinements (Table VII) are all compatible with the distances expected from sums of the effective ionic radii. The possibility of partial disorder, strictly speaking, cannot be excluded, but it should be noted that a HVBDT refinement assuming a statistical distribution of Ni and Fe gave a somewhat higher R than the ordered structure; a refinement of the room-temperature weberite intensities in $Imm2$ with Mg and Al interchanged resulted in $R = 0.023$ compared with 0.019 for the "normal" distribution. Examination of a synthetic Na_2NiAlF_7 might shed further light on the weberite problem.

Experimental

The material for our structure determination was specially selected for this purpose

TABLE V

REFINEMENT OF THE WEBERITE STRUCTURE IN DIFFERENT SPACE GROUPS: THERMAL PARAMETERS ($\times 10^4$)[a]

Each parameter cell lists the two values as they appear in the table.

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Na(1)	181(5) / 122(6)	196(5) / 130(7)	556(7) / 416(14)	0 / 0	−49(5) / −38(8)	0 / 0
	177(5) / 119(5)	192(5) / 122(6)	553(7) / 440(11)	0 / 0	−45(4) / −29(5)	0 / 0
	174(6) / 111(6)	194(6) / 119(6)	551(9) / 443(11)	0 / 0	−46(4) / −33(6)	0 / 0
	180(4) / 121(5)	191(4) / 122(5)	539(6) / 432(9)	0 / 0	−45(4) / −33(5)	0 / 0
Na(2)	229(5) / 153(7)	332(7) / 254(8)	150(5) / 135(10)	0 / 0	0 / 0	−97(5) / −62(7)
	219(5) / 148(6)	333(6) / 251(7)	149(4) / 120(8)	0 / 0	0 / 0	95(3) / 69(5)
	222(6) / 143(6)	333(11) / 247(7)	143(5) / 123(8)	0 / 0	0 / 0	95(4) / 72(5)
	213(4) / 142(5)	335(5) / 253(5)	146(4) / 123(6)	0 / 0	0 / 0	95(3) / 70(4)
Mg	78(3) / 53(5)	67(3) / 54(5)	75(3) / 89(7)	0 / 0	7(2) / 2(5)	0 / 0
	74(3) / 48(4)	63(3) / 48(4)	71(3) / 76(6)	0 / 0	−8(2) / −5(3)	0 / 0
	72(4) / 45(4)	60(4) / 45(4)	68(3) / 75(6)	0 / 0	8(2) / 5(4)	0 / 0
	73(2) / 52(3)	59(2) / 47(3)	68(3) / 78(5)	0 / 0	8(2) / 5(3)	0 / 0
Al	82(3) / 59(4)	73(3) / 52(5)	76(3) / 90(7)	0 / 0	0 / 0	−18(2) / −5(4)
	78(3) / 56(4)	70(3) / 50(4)	75(3) / 76(5)	0 / 0	0 / 0	−18(2) / −10(3)
	76(3) / 49(4)	67(3) / 47(4)	70(3) / 75(6)	0 / 0	0 / 0	19(2) / 13(3)
	76(2) / 53(3)	67(2) / 52(3)	71(3) / 78(4)	0 / 0	0 / 0	−18(2) / −12(3)
F(1)	109(11) / 46(15)	195(11) / 159(16)	164(11) / 73(16)	0 / −2(27)	0 / 0	0 / 0
	78(5) / 62(6)	191(6) / 143(7)	127(5) / 116(9)	−29(24) / 0	0 / 0	0 / 0
	73(5) / 51(7)	192(6) / 134(8)	123(5) / 111(9)	0 / 0	0 / 0	0 / 0
	77(4) / 54(5)	187(5) / 140(6)	126(4) / 118(7)	0 / 0	15(10) / 13(14)	0 / 0
F(2)[b]	56(10) / 70(15)	196(11) / 139(16)	96(11) / 170(17)	0 / 0	0 / 0	0 / 0
F(3)	222(11) / 178(14)	153(10) / 78(13)	113(10) / 65(15)	35(17) / 0	0 / 0	6(10) / 19(14)
	184(4) / 123(5)	159(4) / 109(5)	95(3) / 102(6)	0 / −57(17)	4(18) / −20(25)	14(3) / 3(4)
	177(5) / 120(6)	152(4) / 105(6)	92(4) / 94(7)	0 / 0	0 / 0	14(3) / 2(2)
	180(3) / 127(4)	156(3) / 111(4)	94(3) / 100(5)	−11(9) / −12(12)	−9(9) / −10(13)	14(3) / 0(4)
F(4)[b]	151(10) / 84(13)	174(10) / 152(15)	84(10) / 151(16)	0 / 0	0 / 0	22(10) / −15(15)
F(5)	129(9) / 72(11)	159(10) / 95(12)	154(10) / 182(15)	−1(9) / 21(12)	14(9) / 0(13)	−94(9) / −52(12)
	145(10) / 83(16)	128(16) / 104(19)	180(16) / 166(31)	98(10) / −12(10)	9(13) / −1(20)	−61(13) / −33(20)
	124(3) / 85(4)	128(3) / 88(4)	163(3) / 141(5)	34(2) / 32(3)	14(2) / 14(4)	−59(2) / −38(9)
	143(7) / 87(10)	135(7) / 88(10)	154(7) / 134(11)	18(7) / 2(8)	21(7) / −1(10)	−64(7) / −30(9)

F(6)[b]	136(9)	122(13)	109(9)	104(12)	189(10)	115(14)	−67(9)	−24(13)	22(9)	25(13)	26(9)	24(12)
c	115(10)	103(5)	135(17)	85(17)	155(14)	118(28)	−32(10)	53(10)	25(12)	32(19)	−83(13)	−38(19)
d	108(7)	95(10)	117(7)	95(10)	178(8)	154(11)	48(6)	41(9)	12(7)	30(10)	−51(7)	−45(10)

[a] Lines 1, 2, 3, and 4 correspond, respectively, to refinements in $Imm2$, $I2_12_12_1$, $Imma$, and $I2mb$, except as under[b-d]. The first entry is U_{ij}(R.T.), the second, U_{ij}(−140°C). The U_{ij} are defined from $T = \exp[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{23}klb^*c^* + 2U_{13}hla^*c^* + 2U_{12}hka^*b^*)]$.
[b] $Imm2$.
[c] $I2_12_12_1$.
[d] $I2mb$.

by Professor H. Pauly of the Mineralogical Institute, Technical University of Denmark, from his extensive collection of the Ivigtut material. It was a fragment of a 2-cm crystal with cleavage faces from the pegmatite overlying the Ivigtut deposit in the western half. The clear, colorless crystal ($0.1 \times 0.2 \times 0.4$ mm) used for diffraction was cleaved off this fragment.

Because of the smallness of the fragment and the general scarcity of homogeneous weberite samples an accurate chemical analysis was not attempted. However, the results of X-ray fluorescence analyses (pure fluoride standards) on samples of Ivigtut weberite and the remarkable constancy of the weberite optics indicate that homogeneous weberite may be regarded as stoichiometric Na_2MgAlF_7 (H. Pauly, personal communication; cf. also Ref. (6)).[1]

The piezoelectric tester was of the Giebe–Scheibe type (Crystal Structures Ltd., Bottisham, Cambridge, England).

X-Ray intensities were collected with a CAD-4 four-circle diffractometer (cf. Table III) and reduced by routine procedures (7). A conventional cold-gas-flow cryogenic device was employed in the collection at −140°C. Correction for Lorentz and polarization factors were applied but not for absorption. The ionic scattering factors were taken from Ref. (8). Weighting schemes of the type $w^{-1} = \sigma^2|F_0| + k|F_0|^2$ (σ = individual esd calculated from diffractometer counting statistics) and full-matrix least-squares refinements (9) were used throughout.

Tables of structure factors for the individual refinements will be supplied by the authors on request.

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[1] The original analysis of Ivigtut weberite (5) included 1.19% K, 0.08% Ca, and 0.37% Fe. The author comments that the Ca probably originated in a fluorite impurity, but the K and Fe were thought possibly to replace Na and Al or Mg, respectively, in the weberite itself.

TABLE VI

CORRELATION MATRIX ELEMENTS ($\times$ 100) GREATER THAN $|0.5|^a$

Atoms	Refinement in space group			
	$Imm2$	$I2_12_12_1$	$Imma$	$I2mb$
Na(1)–Mg	x -50; -61	y —; 57 U_{33} —; 56	U_{11} 51; — U_{33} 51; —	
Na(1)–Al				x 50; 59
Na(2)–Mg			U_{33} 51; —	
Na(2)–Al		U_{33} —; 54	U_{11} 51; —	
Mg–Al			U_{11} 53; —	x 63; 65
F(1)–F(2)	z 90; 88 U_{11} -56; -62 U_{22} —; -52 U_{33} -53; —			
F(3)–F(4)	y 93; 89 z 95; 93 U_{11} -65; -66 U_{22} -65; -62 U_{33} -72; -58 U_{23} -73; -72			
F(5)–F(6)	x 90; 85 y -92; -89 z 97; 96 U_{11} -79; -71 U_{22} -81; -75 U_{33} -80; -71 U_{12} 85; 88 U_{13} -85; -82 U_{23} 80; 80	x 86; 83 y 88; 90 z 93; 92 U_{11} -83; -85 U_{22} -93; -90 U_{33} -92; -95 U_{12} -89; -88 U_{13} -94; -95 U_{23} -94; -95		x 97; 98 y 94; 91 z 95; 94 U_{11} -80; -80 U_{22} -78; -80 U_{33} -78; -74 U_{12} -85; -86 U_{13} -84; -84 U_{23} -81; -82

^{a} Since all the correlations are between corresponding parameters, e.g., x–x or U_{11}–U_{11}, the parameter symbol is listed only once. First entry, room temperature; second entry, $-140°C$.

TABLE VII

COMPARISON OF Mg–F AND Al–F DISTANCES (Å) IN THE WEBERITE STRUCTURE REFINED IN DIFFERENT SPACE GROUPS

Space group	Mg–F		Al–F	
	Range	Mean	Range	Mean
$Imm2$	1.943–1.958	1.951(7)	1.785–1.835	1.804(22)
$I2_12_12_1$	1.948–1.952	1.949(3)	1.788–1.820	1.799(18)
$I2mb$	1.931–1.963	1.950(17)	1.789–1.819	1.799(18)
$Imma$	1.948–1.954	1.952(4)	1.789–1.820	1.799(18)
$Imma$ (GT)	1.951–1.959	1.956(5)	1.792–1.823	1.802(18)

Acknowledgments

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