SHORT COMMUNICATION

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Structure of oxonium pentafluorozirconate dihydrate. Corrigendum.

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Abstract

The structure of this compound, $ZrF_5^-.H_3O^+.2H_2O$, was described [Charpin, Lance, Nierlich, Vigner & Lambard (1988). *Acta Cryst.* C**44**, 1698–1701] as monoclinic, space group $C2$, with $a = 10{\cdot}171\ (9)$, $b = 6{\cdot}603\ (1)$, $c = 18{\cdot}156\ (10)$ Å, $\beta = 106{\cdot}29\ (7)°$, $Z = 8$. It is properly described as orthorhombic, space group *Fdd*2, with $a' = 10{\cdot}171$, $b' = 34{\cdot}854$, $c' = 6{\cdot}603$ Å, $Z = 16$. Deviations from the centrosymmetric space group *Fddd* are small.

The vectors defining the new cell edges are [100], [102] and [0$\bar{1}$0]. When the F(obs.) values, obtained from SUP 51026, were averaged according to Laue symmetry *mmm*, the R index between pairs of equivalent reflections was 0·040 for 447 such pairs ('R_{av}' = 0·020). The total number of resulting reflections was 530, of which seven of the very weakest violated the extinction conditions of *Fdd*2; these seven obviously lay close to the $I < 3\sigma(I)$ cut-off (Charpin *et al.*, 1988). After atom coordinates were transformed according to the relationships $x' = x - z/2$, $y' = z/2$, $z' = -y$ and averaged over equivalent atoms, refinement in *Fdd*2 led to a final $R = 0{\cdot}035$, compared to the R of 0·045 reported ' y Charpin *et al.* (1988). Final coordinates are given in Table 1.† The resulting bond lengths and angles are little changed from the earlier values. Difference maps at the end of the refinement provided no clues as to the positions of H atoms, as also noted earlier.

Table 1. *Coordinates, space group Fdd*2.

x, y, z and $U_{eq} \times 10^4$.

	x	y	z	U_{eq}†(Å²)
Zr	1794 (0·8)	−5 (0·5)	0	79 (2)
F(10,20)	1474 (20)	−17 (3)	−3063 (24)	175 (35)
F(11,23)	2368 (10)	552 (4)	−195 (30)	199 (38)
F(12,21)	2359 (10)	−543 (4)	139 (29)	207 (32)
F(13,22)	−4 (12)	337 (1)	20 (35)	156 (12)
F(14,24)	3670 (18)	−21 (2)	−1922 (25)	107 (28)
O(11,21)	3 (16)	711 (2)	−5370 (27)	301 (28)
O(12,22)	4033 (11)	1179 (3)	−4796 (25)	402 (26)
O(13,23)	5855 (11)	1034 (3)	−7195 (22)	348 (25)

† $U_{eq} = \frac{1}{3}\sum_i\sum_j[U_{ij}(a_i^* a_j^*)(\mathbf{a}_i.\mathbf{a}_j)]$.

A somewhat surprising difference between the *Fdd*2 results and those reported in the $C2$ description is in the e.s.d.'s, some of which are *larger* in *Fdd*2 despite the fewer number of parameters and the better agreement. I suspect that these differences are the result of large correlations, since the structure nearly conforms to the centrosymmetric space group *Fddd*. Indeed, block-matrix refinement (each set of atom coordinates or of U_{ij}'s constituting a block) led to effectively the same e.s.d.'s as reported by Charpin *et al.* (1988) (who, however, reported 'full-matrix' refinement). Refinement in *Fddd* led to an R of 0·062, but the intensities of 28 reflections which violate the additional extinction condition are unexplained and the O atoms (but not Zr or F) must either be disordered or have highly anisotropic U_{ij} values. There can be little doubt that *Fdd*2 is the most appropriate space group.

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† Tables of structure factors and U_{ij}'s have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51684 (4 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Reference

CHARPIN, P., LANCE, M., NIERLICH, M., VIGNER, J. & LAMBARD, J. (1988). *Acta Cryst.* C**44**, 1698–1701.