respect to the pseudo-cubic axes of 7·4 Å. Fig. 1 represents a view of the structure in the *ab* plane.

$ZnBr_2.2H_2O$ and $MgZn_2Br_6.6H_2O$ are not only isostructural but almost exactly isometric. There are small differences in the unit cell (~0·2%) but all the atomic positional and displacement parameters are the same within the limits of experimental uncertainty. The only change that occurs when Mg is substituted for Zn is a reduction in the (Mg, Zn)—O bond lengths but this is only marginally significant.

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Structures of Oxonium Pentafluorozirconate Mono- and Dihydrates

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Abstract. (I) $[ZrF_5^-.H_3O^+.2H_2O]$, $M_r = 241{\cdot}27$, monoclinic, $C2$, $a = 10{\cdot}171\ (9)$, $b = 6{\cdot}603\ (1)$, $c = 18{\cdot}156\ (10)$ Å, $\beta = 106{\cdot}29\ (7)°$, $V = 1170\ (2)$ Å^3, $Z = 8$, $D_x = 2{\cdot}738$ g cm^{-3}, $\lambda(\text{Mo } K\bar{\alpha}) = 0{\cdot}71073$ Å, $\mu = 19{\cdot}03$ cm^{-1}, $F(000) = 928$, $T = 293$ K, $R = 0{\cdot}045$ for 977 unique observed reflections. (II) $[ZrF_5^-.H_3O^+.H_2O]$, $M_r = 223{\cdot}25$, orthorhombic, $Cmmm$, $a = 6{\cdot}620\ (3)$, $b = 9{\cdot}858\ (3)$, $c = 8{\cdot}066\ (2)$ Å, $V = 526{\cdot}4\ (5)$ Å^3, $Z = 4$, $D_x = 2{\cdot}817$ g cm^{-3}, $\lambda(\text{Mo } K\bar{\alpha}) = 0{\cdot}71073$ Å, $\mu = 20{\cdot}92$ cm^{-1}, $F(000) = 424$, $T = 293$ K, $R = 0{\cdot}022$ for 323 unique observed reflections. Both structures consist of two-dimensional networks of $(ZrF_5)^{n-}$ connected by oxonium ions and water molecules *via* O—H···O and O—H···F hydrogen bonds. The two structures have nearly identical $(ZrF_5)^{n-}$ layers but differ principally in the packing of these layers and in the hydrogen bonding between them.

Introduction. Hydrates of ZrF_4 have been of interest in research work on fluorine glasses; during the synthesis of $ZrF_4.H_2O$ two distinct fluorohydrates have been isolated and crystallized. Several fluorohydrates have already been mentioned (Waters, 1960; Gaudreau, 1965) but, in the absence of crystal structure determinations, the number of water molecules was still questionable. The present paper reports the structure of $ZrF_4.HF.3H_2O$ (I) (given by Gaudreau as $ZrF_4.HF.4H_2O$) and $ZrF_4.HF.2H_2O$ (II) (given by Waters as $ZrF_4.HF.3H_2O$): as was previously shown for the Ti compound, $TiF_5^-.OH_3^+$ (Cohen, Selig & Gut, 1982), we establish the ionic formulation and the presence of oxonium ions.

Experimental. Enraf–Nonius CAD-4 diffractometer, graphite monochromator; unit-cell parameters refined from 25 reflections in the range $8 < \theta < 12°$; three standard reflections monitored at 100 reflections intervals; scans by $\omega/2\theta$ method; sphere up to $\theta = 25°$; Lorentz and polarization corrections; structure solved by Patterson function for Zr, remaining atoms located by difference Fourier technique; full-matrix least-squares refinement on F; scattering factors including anomalous-dispersion terms from *International Tables for X-ray Crystallography* (1974); calculations on a PDP 11/23 Plus using the *SDP Plus* suite of programs (Frenz, 1983). (I) prepared by dissolving ZrF_4 in 40% aqueous HF and evaporating slowly at ambient temperature (Gaudreau, 1965); colourless crystal, 1·0 × 0·6 × 0·4 mm; loss of 19·3% in 16 h, linearly corrected; 1231 reflections collected, 1122 unique, 977 with $I \geq 3\sigma(I)$; $0 \leq h \leq 12$, $0 \leq k \leq 7$, $-21 \leq l \leq 21$; empirical absorption correction using the program *DIFABS* (Walker & Stuart, 1983), max. and min. transmissions, 2·0–0·78; anisotropic thermal parameters for Zr; $R = 0{\cdot}045$, $wR = 0{\cdot}049$ ($w = 1$), $(\Delta/\sigma)_{\max} = 0{\cdot}10$, $S = 4{\cdot}5$. In final difference Fourier synthesis, no peaks greater than 0·9 e Å^{-3}; no correction for extinction. (II) ZrF_4 in 40% aqueous HF and evaporat-

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ing quickly at 303 K (Waters, 1960); colourless crystal, 0·25 × 0·10 × 0·05 mm; loss of 1% in 7 h, no correction; 431 reflections collected, 358 unique, 323 with $I > 3\sigma(I)$; $0 \le h \le 8$, $0 \le k \le 12$, $0 \le l \le 10$; no absorption correction, anisotropic thermal parameters for Zr,F,O; one H_2O [O(2)], located at 8(*q*) positions of the *Cmmm* group, was considered as disordered and given an occupation factor $\frac{1}{2}$; $R = 0{\cdot}022$, $wR = 0{\cdot}028$ ($w = 1$), $(\Delta/\sigma)_{\max} = 0{\cdot}02$, $S = 1{\cdot}73$. In final difference Fourier synthesis, no peaks greater than 0·6 e Å^{-3}; no correction for extinction.

Discussion. Tables 1 and 2 list the final positional and equivalent isotropic thermal parameters for (I) and (II), respectively. Table 3 gives selected comparative bond distances and angles for (I) and (II).*

Table 1. *The final atom coordinates and isotropic thermal parameters of* $ZrF_5^-.H_3O^+.2H_2O$ (I)

	x	*y*	*z*	B_{eq} or B_{iso} (Å^2)
Zr(1)	0·1788 (1)	0·0	−0·00102 (6)	0·54 (2)*
Zr(2)	0·1799 (1)	0·2498 (2)	0·50101 (6)	0·60 (2)*
F(10)	0·1363 (7)	0·308 (1)	−0·0073 (4)	0·6 (1)
F(11)	0·2921 (8)	0·009 (2)	0·1101 (4)	1·4 (1)
F(12)	0·1806 (8)	−0·018 (2)	−0·1092 (4)	1·5 (1)
F(13)	0·0340 (7)	0·005 (2)	0·0671 (4)	1·1 (1)
F(14)	0·3552 (7)	0·193 (1)	−0·0095 (4)	0·8 (1)
F(20)	0·1382 (8)	0·557 (1)	0·4985 (4)	1·3 (1)
F(21)	0·2907 (7)	0·226 (1)	0·6086 (4)	1·3 (1)
F(22)	−0·0329 (7)	0·254 (2)	0·4331 (4)	1·1 (1)
F(23)	0·1816 (8)	0·265 (2)	0·3895 (4)	1·4 (1)
F(24)	0·3600 (8)	0·443 (1)	0·4995 (4)	1·5 (2)
O(11)	0·071 (1)	0·541 (2)	0·1427 (6)	1·8 (2)
O(12)	0·522 (1)	0·482 (3)	0·2369 (7)	3·0 (2)
O(13)	0·689 (1)	0·718 (2)	0·2066 (6)	2·4 (2)
O(21)	0·929 (1)	0·791 (2)	0·3575 (6)	2·0 (2)
O(22)	0·787 (1)	0·230 (2)	0·2638 (6)	2·7 (2)
O(23)	0·983 (1)	0·470 (2)	0·2939 (7)	2·7 (2)

* $B_{eq} = \frac{4}{3}\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j$.

Table 2. *The final atom coordinates and isotropic thermal parameters of* $ZrF_5^-.H_3O^+.H_2O$ (II)

	x	*y*	*z*	B_{eq} or B_{iso} (Å^2)
Zr	0·0	0·18320 (6)	0·0	0·794 (9)*
F(1)	0·3049 (4)	0·1371 (3)	0·0	1·51 (5)*
F(2)	0·0	0·0	0·1463 (5)	1·35 (7)*
F(3)	0·0	0·2463 (3)	0·2392 (4)	2·13 (6)
O(1)	0·0	0·5	0·350 (1)	7·5 (3)*
O(2)	0·105 (2)	0·117 (1)	0·5	5·2 (3)*

* $B_{eq} = \frac{4}{3}\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j$.

Table 3. *Selected distances* (Å) *and angles* (°) *in compounds* (I) *and* (II)

Environment of the Zr atoms: Zr—F distances

	(I)		(II)
	Zr(1)	Zr(2)	
Non-bridging F	1·973 (8)	1·969 (7)	
	2·024 (7)	2·032 (8)	2·027 (3)
Bridging F	2·173 (8)	2·170 (6)	
	2·161 (6)	2·162 (8)	2·157 (3)
	2·075 (8)	2·074 (9)	
	2·072 (8)	2·069 (9)	2·069 (3)
	2·242 (8)	2·242 (8)	
	2·238 (7)	2·240 (9)	2·192 (3)

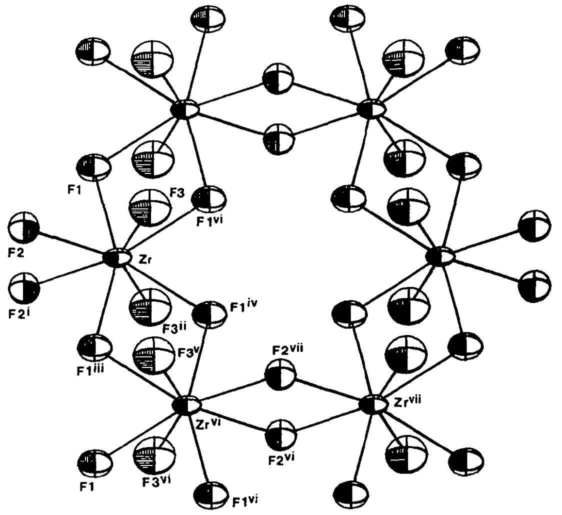

Fig. 1. *ORTEP* (Johnson, 1976) drawing of $(ZrF_5)^{n-}$ in $ZrF_5^-.H_3O^+.H_2O$ (II). Symmetry code: (i) $-x, -y, -z$; (ii) $x, y, -z$; (iii) $-x, y, z$; (iv) $\frac{1}{2}+x, \frac{1}{2}-y, -z$; (v) $\frac{1}{2}-x, \frac{1}{2}-y, z$; (vi) $\frac{1}{2}-x, \frac{1}{2}-y, -z$; (vii) $\frac{1}{2}+x, \frac{1}{2}+y, z$.

In both compounds the structure consists of a two-dimensional polymeric $(ZrF_5)^{n-}$ network, the oxonium ions and water molecules linking these layers *via* O···F and O···O hydrogen bonds. The two structures are nearly identical concerning the structure of the layers; however, the Zr atom is required to possess *mm* symmetry by the space group in (II), whereas no local symmetry is imposed on the sites occupied by atoms Zr(1) and Zr(2) in (I). Fig. 1 shows such a typical layer $(ZrF_5)^{n-}$. Zr is coordinated by eight F atoms: six F act as bridges and are shared with three Zr atoms in the plane, the two non-bridging F participate in hydrogen bonds. The F are unequally shared between Zr atoms, the bridging Zr—F bonds ranging from 2·063 (9) to 2·242 (8) Å in (I) and from 2·070 (3) to 2·157 (2) Å in (II); the non-bridging bonds are shorter: 1·973 (8), 2·024 (7) Å (I) and 2·028 (3) Å (II). These values agree with those found in the two known hydrates of ZrF_4 [$ZrF_4.H_2O$ (Kojić-Prodić, Gabela, Ružic-Toroš &

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* Lists of structure factors, anisotropic thermal parameters and bond distances and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51026 (27 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 4. *Distances* (Å) *and angles* (°) *possibly involved in hydrogen bonds in compounds* (I) *and* (II)

Compound (I)

O(11^{i})–F(11)	2·74 (1)	O(21)–F(21^{i})	2·77 (1)
O(11^{ii})–F(12^{ii})	2·78 (1)	O(21)–F(23)	2·74 (1)
O(11^{i})–O(13^{i})	2·56 (1)	O(21)–O(23)	2·55 (2)
O(11^{ii})–O(22)	2·92 (1)	O(21)–O(12^{i})	2·90 (2)
F(11)–O(11^{i})–F(12^{i})	152·8 (5)	F(21^{i})–O(21)–F(23)	152·8 (5)
F(11)–O(11^{i})–O(13^{i})	111·0 (5)	F(23)–O(21)–O(12^{i})	134·3 (5)
F(11)–O(11^{i})–O(22^{iii})	135·2 (5)	F(23)–O(21)–O(23)	111·8 (5)
O(12)–O(13)	2·48 (2)	O(22)–O(23)	2·49 (2)
O(12)–F(12^{iii})	2·63 (1)	O(22)–F(21^{ii})	2·65 (2)
O(12)–O(21^{i})	2·91 (2)	O(22)–O(11^{ii})	2·92 (2)
O(13)–O(12)–F(12^{iii})	102·7 (6)	F(21^{ii})–O(22)–O(11^{ii})	102·0 (5)
O(13)–O(12)–O(21^{i})	145·9 (6)	F(21^{ii})–O(22)–O(23)	103·5 (5)
F(12^{iii})–O(12)–O(21^{i})	108·9 (5)	O(11^{ii})–O(22)–O(23)	145·7 (6)
O(13)–F(11^{ii})	2·66 (2)	O(23)–F(23^{iii})	2·63 (1)
O(13)–O(11)	2·56 (2)	O(23)–O(21)	2·55 (2)
O(13)–O(12)	2·48 (2)	O(23)–O(22)	2·49 (2)
F(11^{ii})–O(13)–O(11)	110·7 (5)	F(23^{iii})–O(23)–O(21)	110·6 (5)
F(11^{ii})–O(13)–O(12)	104·0 (6)	F(23^{iii})–O(23)–O(22)	103·7 (6)
O(11)–O(13)–O(12)	111·3 (6)	O(21)–O(23)–O(22)	111·5 (6)

Symmetry code: (i) $-x, y, -z$; (ii) $\frac{1}{2}-x, \frac{1}{2}+y, -z$; (iii) $\frac{1}{2}+x, \frac{1}{2}+y, z$.

Compound (II)

O(1)–F(3)	2·656 (4)	O(2)–O(2^{i})	2·69 (1)
O(1)–O(1^{i})	2·42 (1)	O(2)–F(3)	2·557 (7)
O(1^{i})–O(1)–F(3)	109·7 (2)	O(2)–O(2^{i})–F(3^{i})	106·6 (4)
F(3)–O(1)–F(3^{viii})	140·6 (3)	F(3)–O(2)–F(3)	110·7 (4)

Symmetry code: (i) $-x, -y, -z$; (viii) $-x, -y, z$.

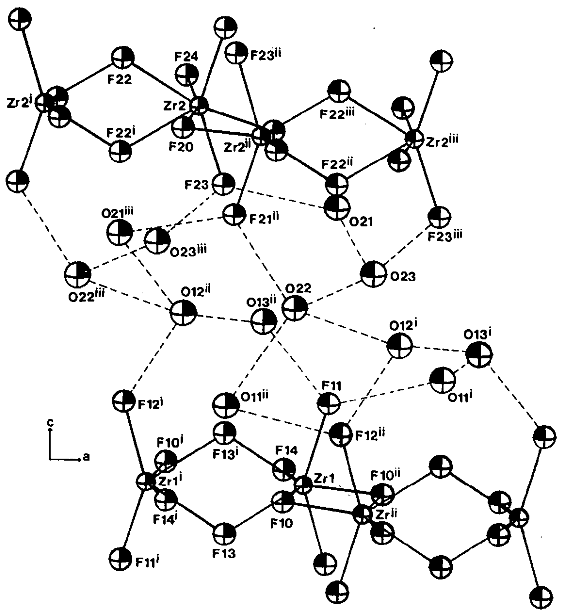

Fig. 2. Packing of compound (I) with distances possibly involved in hydrogen bonds. Symmetry code: (i) $-x, y, -z$; (ii) $\frac{1}{2}-x, \frac{1}{2}+y, -z$; (iii) $\frac{1}{2}+x, \frac{1}{2}+y, z$.

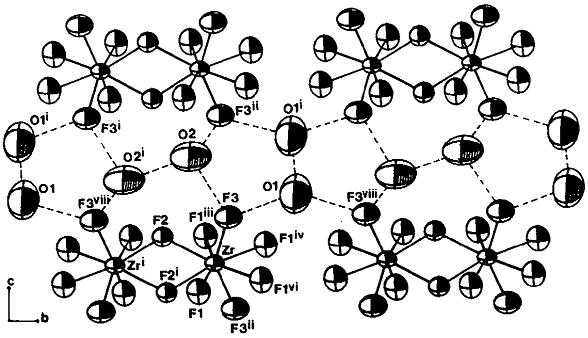

Fig. 3. Packing of compound (II) with distances possibly involved in hydrogen bonds. Symmetry code: (i) $-x, -y, -z$; (ii) $x, y, -z$; (iii) $-x, y, z$; (iv) $\frac{1}{2}+x, \frac{1}{2}-y, -z$; (vi) $\frac{1}{2}-x, \frac{1}{2}-y, -z$; (viii) $-x, -y, z$.

Šljukić, 1981); $ZrF_4.3H_2O$ (Gabela, Kojić-Prodić, Šljukić & Ružic-Toroš, 1977)]. The stereochemistry of Zr corresponds to a dodecahedron, sharing three edges with three other polyhedra: the dihedral angles between the two fluorine trapezoids defining the dodecahedron are respectively 92·8 (2) [Zr(1)] and 86·6 (2)° [Zr(2)] in (I), and 90° in (II). The Zr–Zr distances are 3·601 (2) and 3·649 (2) Å in (I) and 3·563 (1) and 3·612 (1) Å in (II), in a pseudo hexagonal arrangement.

Although H atoms have not been observed, the O···O and O···F distances are indicative of H bonding. Table 4 gives O···O and O···F distances and angles. Packing diagrams with possible H bonds are shown in Figs. 2 and 3 for (I) and (II), respectively. In both structures the two non-bridging F atoms are involved in OH···F hydrogen bonds. In the dihydrate (I), there are short contacts between O connected to F atoms [for example: O(11)–O(13) 2·56 (2), O(12)–O(13) 2·48 (2) Å in layer 1 and O(21)–O(23) 2·55 (2), O(22)–O(23) 2·49 (2) Å in layer 2], whereas the O–O contacts between these two layers are weak: O(12)–O(21) 2·91 (2) and O(22)–O(11) 2·92 (2) Å.

In the monohydrate (II), the interlayer bonding is stronger since one O atom, O(2), is shared by two layers while the other, O(1), belongs to one layer, the two O–O distances being short: O(1)–O(1^{i}) 2·42 (1) and O(2)–O(2^{i}) 2·69 (1) Å. In both compounds, the oxonium ion cannot be clearly distinguished from the water molecules in the absence of localization of the H atoms: neutron diffraction would be necessary to determine the position of the oxonium ions.

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Structure of Tribenzylammonium 1,1,1,1,2,2,2,3,3,3-Decacarbonyl-2,3-μ-hydrido-2,3-μ-sulfonyl-*triangulo*-triosmium

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Abstract. $[NH(CH_2C_6H_5)_3][Os_3(CO)_{10}(\mu\text{-H})(\mu\text{-SO}_2)]$, $M_r = 1204{\cdot}16$, monoclinic, $P2_1$, $a = 9{\cdot}605\,(1)$, $b = 12{\cdot}809\,(4)$, $c = 15{\cdot}116\,(2)$ Å, $\beta = 105{\cdot}1\,(5)°$, $V = 1795{\cdot}82$ Å^3, $Z = 2$, $D_x = 2{\cdot}223$ g cm^{-3}, $\lambda(\text{Mo } K\alpha_1) = 0{\cdot}7093$ Å, $\mu = 107{\cdot}1$ cm^{-1}, $F(000) = 1101$, $T = 298$ K, $R = 0{\cdot}034$ for 1523 reflections with $I \geq 2\sigma(I)$. The anion consists of a triangular array of osmium atoms with two unbridged edges [Os—Os 2·850 (3) and 2·864 (5) Å] and the third edge bridged by the hydride and SO_2 ligands [Os—Os 2·908 (7) Å]. A close contact [2·65 (4) Å] between the nitrogen of the cation and one of the SO_2 oxygens indicates hydrogen bonding between the tribenzylammonium cation and the cluster anion.

Introduction. Our recent investigations of reactions between metal hydride complexes and SO_2 have shown interesting reduction chemistry (Kubas & Ryan, 1986). Previously we reported on the structure of $Os_3(CO)_{10}(\mu\text{-H})_2(\mu\text{-SO}_2)$, one of a rather small number of metal complexes containing hydride and SO_2 ligands (Jarvinen & Ryan, 1984). The complex $Os_3(CO)_{10}(\mu\text{-H})_2(\mu\text{-SO}_2)$ is the first product observed by NMR or IR spectroscopy from the reaction of $Os_3(CO)_{10}(\mu\text{-H})_2$ and SO_2, but additional products form at rates dependent on variables such as the solvent, temperature, and SO_2 concentration. Tertiary amines were added to the reaction mixture in an attempt to bind acidic protons that might be present. Addition of tribenzylamine gave the title compound, which formally results from the transfer of a proton from $Os_3(CO)_{10}(\mu\text{-H})_2(\mu\text{-SO}_2)$ to tribenzylamine.

Experimental. The compound was prepared by dissolving 152·7 mg of $Os_3(CO)_{10}(\mu\text{-H})_2$ and 53·7 mg of tribenzylamine in 28 ml of 3:1 heptane/chloroform under N_2. Sulfur dioxide was bubbled through the purple solution which was then left overnight. The resulting clear yellow solution was placed in a freezer and a small amount of powdery solid formed. The solution was warmed to room temperature and the $CHCl_3$ was partially evaporated by passing N_2 over the solution. The flask was again placed in a freezer where crystals formed, 57·4 mg, 26%. Analysis: calc. for $C_{31}H_{23}NO_{12}Os_3S$: C, 30·92; H, 1·93; N, 1·16; O, 15·94; S, 2·66%. Found: C, 30·84; H, 2·02; N, 1·12; O, 16·62; S, 2·77%. A yellow crystal having a length of about 0·1 mm was mounted on a standard goniometer. Data were collected by θ–2θ scans ($2\theta_{\max} = 40°$) on a CAD-4 automated diffractometer using Mo $K\alpha$ radiation and a graphite monochromator. Cell dimensions were determined by least-squares refinement of 25 centered reflections ($12° \leq \theta \leq 35°$). The space group was chosen on the basis of systematically absent reflections and a subsequent successful refinement. Reflections in a quadrant with Miller indices $h = 0$–9, $k = 0$–12 and $l = 0 \pm 14$ were collected and averaged to yield 1523 reflections with $I \geq 2\sigma(I)$. The R value based on F_o^2 for the merge of equivalent reflections was 0·024. Data correction including a combination of spherical and φ absorption corrections was made (max. transmission 0·484, min. 0·284) and corrections for Lorentz and polarization factors were performed as previously (Ryan & Swanson, 1974). The structure was solved by standard Patterson methods and refined by full-matrix least squares employing the Los Alamos *Generalized Crystal Structure Analysis System* (Larson & Von Dreele, 1986). All atoms except the metals and SO_2 group were modeled as having isotropic thermal motion. The function minimized was $\sum w(F_o^2 - F_c^2)^2$ and weights were determined by $w = 1/(\sigma F_o^2)^2$ and $\sigma F_o^2 = [I + B + 0{\cdot}01I^2]T\text{Lp}$. The final residuals were $R = 0{\cdot}034$ and $wR = 0{\cdot}031$ for the 1523 reflections having $I \geq 2\sigma(I)$. $(\Delta/\sigma)_{\max} = 0{\cdot}51$. Hydrogens were placed several times after successive refinements and

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