Structure of Aqua-hexakis(μ_2-benzoato)-bis(methanol)-μ_3-oxo-triiron(III) Benzoate Ethanol Methanol Solvate

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Abstract. [Fe₃(O)(C₇H₅O₂)₆(CH₃OH)₂(H₂O)]-[C₇H₅O₂]·C₂H₅OH·CH₃OH (1/1/1), M_r = 1191·56, triclinic, $P\bar{1}$, a = 11·591 (3), b = 13·308 (2), c = 20·154 (3) Å, α = 96·910 (12), β = 96·95 (2), γ = 114·10 (2)°, V = 2767·1 (9) Å³, Z = 2, D_x = 1·43 g cm⁻³, μ = 8·471 cm⁻¹, Mo $K\alpha$ radiation, λ = 0·7107 Å, $F(000)$ = 1234, T = 198 K, R = 0·0522 for 6252 reflections [$F_o \geq 4\sigma(F_o)$]. The complex has nearly D_{3h} symmetry with two coordinated CH₃OH molecules and one coordinated H₂O molecule. The coordination around the Fe atoms is essentially octahedral. Each Fe atom lies slightly out of the plane of the O atoms of the bridging benzoates and is directed towards the μ_3-O atom. The average Fe—O distances are 1·907 (2) Å for the μ_3-O atom and 2·010 (1) Å for the benzoate O atoms.

Experimental. As a by-product of ongoing research related to the respiratory protein hemerythrin (Sessler, Sibert & Lynch, 1990; Sessler, Hugdahl, Lynch & Davis, 1990), complex (1), [Fe₃(O)-(O₂CC₆H₅)₆(CH₃OH)₂(H₂O)][O₂CC₆H₅]·CH₃OH–C₂H₅OH, was synthesized. Orange crystals were obtained by slow evaporation of a methanol–ethanol (5:1) solution. The crystals quickly decomposed when taken out of the mother liquor, presumably due to the loss of methanol and/or ethanol solvate molecules.
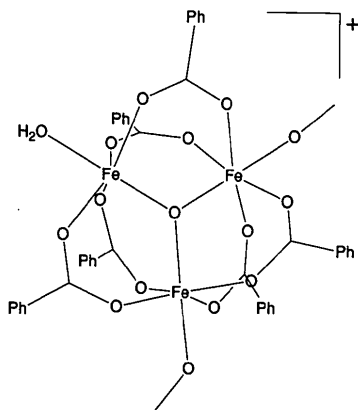
The data crystal was an orange needle of approximate dimensions 0·13 × 0·20 × 0·41 mm. The data

were collected on a Nicolet *P3* diffractometer, using graphite-monochromatized Mo $K\alpha$ radiation, and a Nicolet LT-2 low-temperature delivery system. Lattice parameters were obtained from the least-squares refinement of 42 reflections with 24·4 < 2θ < 30·0°. Data were collected using the ω-scan technique (10 318 reflections, 9788 unique, R_{int} = 0·0134), with a 2θ range from 4·0–50° in 2θ, over a 1·3° ω scan at 4–8° min⁻¹ (h = 0→12, k = –15→15, l = –23→23). Four reflections ($2\bar{3}3$; $\bar{2}32$; $\bar{3}3\bar{2}$; $\bar{3}01$) were remeasured every 96 reflections to monitor instrument and crystal stability. A smoothed curve of the intensities of these four reflections was used to scale the data. The scaling factor ranged from 0·968–1·03. The data were also corrected for Lp effects and absorption. The absorption correction was based on crystal-face measurements with a minimium and maximum transmission factor range of 0·8291–0·9147. The data reduction, absorption and decay correction were performed using *SHELXTL-Plus* (Sheldrick, 1987). Reflections having F_o < $4\sigma(F_o)$ were considered unobserved (3536 reflections). The structure was solved by the direct methods and refined by full-matrix least squares (Sheldrick, 1987). The non-H atoms were refined with anisotropic thermal parameters. The H atoms were obtained from a ΔF map and refined with isotropic thermal parameters. During the latter stages of refinement, the H atoms of the methanol C atoms (C60, C62, C2B) and the C atoms of the ethanol molecule (C2A, C3A) were idealized (C—H 0·96 Å) with isotropic thermal parameters fixed at 1·2 × U_{eq} of the relevant C atom. In all, a total of 858 paremeters were refined in blocks of 394 and 465 parameters with the scale factor being refined in each block. The function $\sum w(|F_o| - |F_c|)^2$ was minimized, where $w = 1/[\sigma(F_o)]^2$ and $\sigma(F_o) = 0\cdot5kI^{-1/2}\{[\sigma(I)^2 + (0\cdot02I)^2\}^{1/2}$. The intensity, I, is given by $(I_{peak} - I_{background})$ × (scan rate); 0·02 is a factor to downweight intense reflections and to account for instrument instability and k is the correction due to Lp effects, absorption and decay. $\sigma(I)$ was estimated from counting statistics; $\sigma(I) = [(I_{peak} + I_{background})^{1/2}$ × (scan rate)]. Final R = 0·0522 for 6252 reflections, wR = 0·0533 (R_{all} = 0·0863, wR_{all} = 0·0583) and a goodness of fit = 1·43.

Table 1. *Fractional coordinates and equivalent iso-tropic thermal parameters* ($\mathring{A}^2$) *for the non-H atoms of* (1)

For anisotropic atoms, U is U_{eq}, where
$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	*U*
Fe1	−0·80124 (7)	0·20168 (6)	0·77919 (3)	0·0248 (3)
Fe2	−0·57396 (7)	0·14695 (6)	0·72108 (3)	0·0242 (3)
Fe3	−0·56507 (7)	0·40018 (6)	0·73015 (3)	0·0225 (3)
O4	−0·6468 (3)	0·2495 (2)	0·74382 (14)	0·0235 (13)
O5	−0·8872 (3)	0·2743 (3)	0·7218 (2)	0·034 (2)
C6	−0·8426 (5)	0·3577 (4)	0·6932 (2)	0·030 (2)
O7	−0·7254 (3)	0·4115 (3)	0·6908 (2)	0·036 (2)
C8	−0·9343 (5)	0·3965 (4)	0·6589 (2)	0·029 (2)
C9	−0·0626 (6)	0·3469 (5)	0·6625 (3)	0·044 (3)
C10	−0·1474 (7)	0·3832 (7)	0·6309 (4)	0·071 (4)
C11	−0·1025 (8)	0·4695 (7)	0·5957 (3)	0·069 (4)
C12	−0·9755 (8)	0·5184 (6)	0·5916 (3)	0·056 (4)
C13	−0·8910 (6)	0·4822 (5)	0·6227 (3)	0·039 (3)
O14	−0·7350 (3)	0·3342 (3)	0·8557 (2)	0·033 (2)
C15	−0·6453 (5)	0·4312 (4)	0·8655 (2)	0·026 (2)
O16	−0·5737 (3)	0·4683 (3)	0·8233 (2)	0·037 (2)
C17	−0·6205 (5)	0·5078 (4)	0·9311 (2)	0·026 (2)
C18	−0·6950 (6)	0·4733 (5)	0·9797 (3)	0·045 (3)
C19	−0·6715 (7)	0·5431 (6)	1·0406 (3)	0·055 (3)
C20	−0·5732 (6)	0·6483 (5)	1·0545 (3)	0·045 (3)
C21	−0·4995 (7)	0·6827 (5)	1·0072 (3)	0·045 (3)
C22	−0·5230 (6)	0·6129 (5)	0·9459 (3)	0·041 (3)
O23	−0·6045 (3)	0·0753 (3)	0·8029 (2)	0·036 (2)
C24	−0·6767 (5)	0·0690 (4)	0·8444 (3)	0·028 (2)
O25	−0·7499 (3)	0·1182 (3)	0·8461 (2)	0·039 (2)
C26	−0·6770 (5)	−0·0016 (4)	0·8974 (2)	0·030 (2)
C27	−0·6010 (7)	−0·0579 (5)	0·8973 (4)	0·055 (3)
C28	−0·6058 (9)	−0·1278 (7)	0·9438 (4)	0·077 (5)
C29	−0·6834 (8)	−0·1387 (6)	0·9907 (4)	0·069 (4)
C30	−0·7562 (7)	−0·0802 (6)	0·9930 (3)	0·056 (3)
C31	−0·7532 (6)	−0·0103 (5)	0·9460 (3)	0·045 (3)
O32	−0·7422 (3)	0·0292 (3)	0·6659 (2)	0·037 (2)
C33	−0·8574 (5)	0·0099 (4)	0·6674 (3)	0·031 (2)
O34	−0·8952 (3)	0·0602 (3)	0·7101 (2)	0·040 (2)
C35	−0·9577 (5)	−0·0838 (4)	0·6137 (3)	0·038 (2)
C36	−0·9230 (7)	−0·1463 (5)	0·5680 (3)	0·052 (3)
C37	−0·0150 (8)	−0·2317 (6)	0·5170 (4)	0·066 (4)
C38	−0·1419 (8)	−0·2531 (7)	0·5145 (4)	0·080 (4)
C39	−0·1793 (7)	−0·1939 (6)	0·5633 (4)	0·074 (4)
C40	−0·0855 (6)	−0·1087 (5)	0·6127 (3)	0·055 (3)
O41	−0·5241 (3)	0·1989 (3)	0·6351 (2)	0·037 (2)
C42	−0·5197 (5)	0·2805 (4)	0·6081 (2)	0·028 (2)
O43	−0·5413 (3)	0·3602 (3)	0·6347 (2)	0·033 (2)
C44	−0·4841 (5)	0·2832 (4)	0·5392 (2)	0·030 (2)
C45	−0·4722 (6)	0·1931 (5)	0·5046 (3)	0·044 (3)
C46	−0·4396 (7)	0·1963 (6)	0·4406 (3)	0·061 (3)
C47	−0·4210 (7)	0·2893 (6)	0·4114 (3)	0·056 (3)
C48	−0·4306 (6)	0·3798 (6)	0·4462 (3)	0·050 (3)
C49	−0·4636 (5)	0·3764 (5)	0·5092 (3)	0·037 (3)
O50	−0·3945 (3)	0·2536 (3)	0·7723 (2)	0·034 (2)
C51	−0·3355 (5)	0·3586 (4)	0·7806 (2)	0·026 (2)
O52	−0·3856 (3)	0·4226 (3)	0·7650 (2)	0·035 (2)
C53	−0·1959 (5)	0·4119 (4)	0·8106 (3)	0·030 (2)
C54	−0·1320 (6)	0·3483 (5)	0·8271 (3)	0·050 (3)
C55	−0·0008 (6)	0·3997 (6)	0·8541 (4)	0·065 (3)
C56	0·0636 (7)	0·5130 (6)	0·8657 (4)	0·064 (3)
C57	0·0029 (6)	0·5772 (5)	0·8491 (4)	0·059 (3)
C58	−0·1269 (5)	0·5270 (5)	0·8211 (3)	0·044 (3)
O59	−0·9675 (4)	0·1518 (3)	0·8205 (2)	0·035 (2)
C60	−0·9739 (7)	0·1527 (5)	0·8920 (3)	0·064 (4)
O61	−0·4801 (4)	0·5649 (3)	0·7158 (2)	0·034 (2)
C62	−0·3536 (6)	0·6264 (5)	0·7041 (4)	0·067 (3)
O63	−0·4985 (4)	0·0368 (3)	0·6949 (2)	0·034 (2)
O1A	−0·5630 (4)	−0·2880 (3)	0·7633 (2)	0·060 (2)
C2A	−0·6866 (8)	−0·3089 (6)	0·7758 (4)	0·084 (4)
C3A	−0·7570 (12)	−0·2400 (9)	0·7339 (5)	0·164 (8)
O1B	−0·2906 (4)	0·0863 (5)	0·6380 (3)	0·078 (3)
C2B	−0·2384 (8)	0·0770 (7)	0·5799 (4)	0·090 (5)
O1C	−0·2011 (4)	0·0339 (3)	0·7528 (2)	0·055 (2)
C2C	−0·2775 (6)	−0·0430 (5)	0·7790 (3)	0·044 (3)
O3C	−0·3960 (4)	−0·0692 (3)	0·7739 (2)	0·048 (2)
C4C	−0·2185 (5)	−0·1033 (4)	0·8213 (3)	0·038 (2)
C5C	−0·2706 (6)	−0·1449 (5)	0·8754 (3)	0·051 (3)
C6C	−0·2095 (7)	−0·1939 (6)	0·9166 (4)	0·061 (4)
C7C	−0·1058 (7)	−0·2061 (5)	0·9017 (4)	0·058 (3)
C8C	−0·0524 (6)	−0·1653 (5)	0·8463 (4)	0·053 (3)
C9C	−0·1082 (5)	−0·1120 (5)	0·8070 (3)	0·043 (3)

The maximum $|\Delta/\sigma| < 0\cdot1$ in the final refinement cycle and the minimum and maximum peaks in the final ΔF map were $-0\cdot64$ and $0\cdot70$ e $\mathring{A}^{-3}$, respectively. Scattering factors for the non-H atoms were taken from Cromer & Mann (1968), with anomalous-dispersion corrections from the work of Cromer & Liberman (1970). The scattering factors
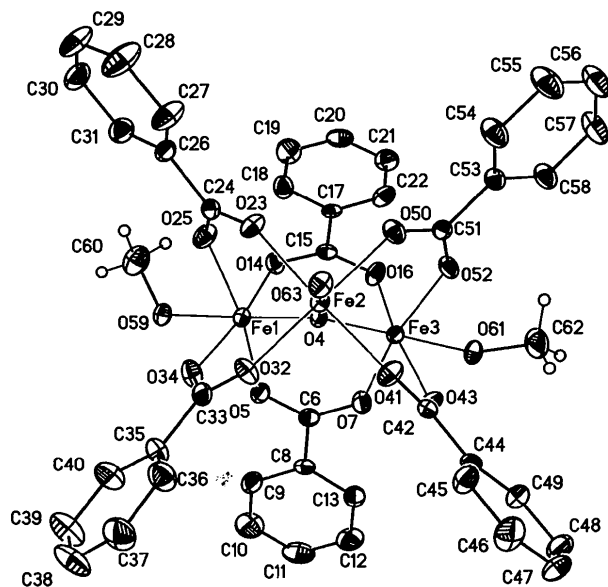

Fig. 1. View of (1) showing the atom-labelling scheme. Ellipsoids are scaled to the 30% probability level. Most H atoms are omitted for clarity.
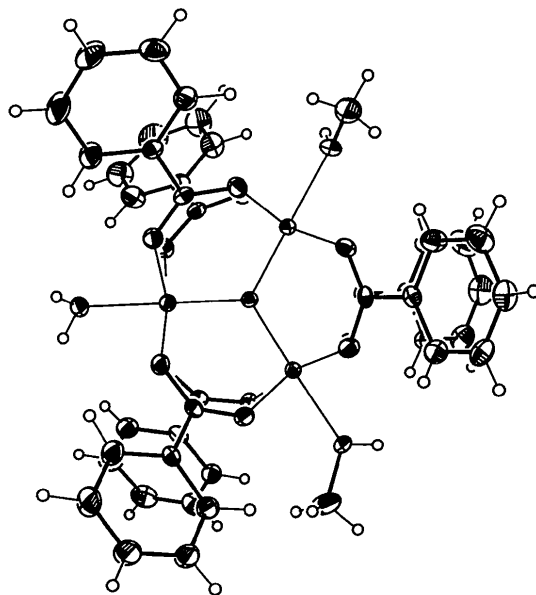

Fig. 2. View of (1) looking down the pseudo-threefold axis of the complex. The benzoates bridge the Fe atoms. The water Fe—O bond [Fe2—O63 2·042 (5) Å] is slightly shorter than the methanol Fe—O bonds [Fe1—O59 2·080 (4), Fe3—O61 2·077 (4) Å].

Table 2. Bond lengths (Å) and angles (°) for the non-H atoms of (1)

Table 2 (cont.)

1	2	3	1—2	1—2—3
O4	Fe1	O5	1·899 (3)	96·86 (15)
O4	Fe1	O14		94·89 (13)
O4	Fe1	O25		95·7 (2)
O4	Fe1	O34		95·19 (14)
O5	Fe1	O14	2·011 (4)	91·0 (2)
O5	Fe1	O25		167·4 (2)
O5	Fe1	O34		88·3 (2)
O5	Fe1	O59		83·9 (2)
O14	Fe1	O25	2·008 (3)	87·5 (2)
O14	Fe1	O34		169·9 (2)
O14	Fe1	O59		83·71 (14)
O25	Fe1	O34	2·029 (4)	91·0 (2)
O25	Fe1	O59		83·5 (2)
O34	Fe1	O59	1·999 (3)	86·2 (2)
O59	Fe1	O4	2·080 (4)	178·43 (13)
O4	Fe2	O23	1·915 (4)	96·1 (2)
O4	Fe2	O32		92·84 (15)
O4	Fe2	O41		95·8 (2)
O4	Fe2	O50		94·05 (14)
O23	Fe2	O32	1·999 (4)	91·55 (13)
O23	Fe2	O41		168·1 (2)
O23	Fe2	O50		88·70 (14)
O23	Fe2	O63		84·7 (2)
O32	Fe2	O41	2·013 (3)	89·00 (13)
O32	Fe2	O50		173·0 (2)
O32	Fe2	O63		86·3 (2)
O41	Fe2	O50	2·004 (4)	89·32 (14)
O41	Fe2	O63		83·5 (2)
O50	Fe2	O63	2·030 (3)	86·8 (2)
O63	Fe2	O4	2·042 (5)	178·82 (14)
O4	Fe3	O7	1·908 (3)	96·75 (14)
O4	Fe3	O16		94·40 (13)
O4	Fe3	O43		95·65 (13)
O4	Fe3	O52		95·80 (15)
O7	Fe3	O16	2·002 (4)	90·1 (2)
O7	Fe3	O43		88·7 (2)
O7	Fe3	O52		167·42 (14)
O7	Fe3	O61		82·0 (2)
O16	Fe3	O43	2·017 (4)	169·95 (14)
O16	Fe3	O52		89·8 (2)
O16	Fe3	O61		84·78 (15)
O43	Fe3	O52	2·015 (3)	89·2 (2)
O43	Fe3	O61		85·17 (15)
O52	Fe3	O61	1·999 (4)	85·4 (2)
O61	Fe3	O4	2·077 (4)	178·5 (2)
Fe1	O4	Fe2		120·4 (2)
Fe1	O4	Fe3		119·6 (2)
Fe2	O4	Fe3		120·0 (2)
C6	O5	Fe1	1·260 (6)	132·0 (3)
O7	C6	C8	1·261 (6)	116·8 (5)
O7	C6	O5		125·0 (6)
C8	C6	O5	1·493 (9)	118·3 (5)
Fe3	O7	C6		133·3 (4)
C9	C8	C13	1·375 (8)	119·3 (6)
C9	C8	C6		120·6 (5)
C13	C8	C6	1·381 (8)	120·2 (5)
C10	C9	C8	1·383 (13)	120·5 (6)
C11	C10	C9	1·376 (12)	119·5 (7)
C12	C11	C10	1·363 (11)	120·2 (9)
C13	C12	C11	1·377 (12)	120·5 (7)
C8	C13	C12		120·1 (6)
C15	O14	Fe1	1·256 (5)	133·4 (3)
O16	C15	C17	1·263 (6)	117·3 (4)
O16	C15	O14		124·2 (4)
C17	C15	O14	1·487 (7)	118·5 (4)
Fe3	O16	C15		133·1 (3)
C18	C17	C22	1·380 (8)	117·7 (5)
C18	C17	C15		120·7 (4)
C22	C17	C15	1·359 (7)	121·7 (5)
C19	C18	C17	1·372 (9)	120·9 (5)
C20	C19	C18	1·365 (8)	120·5 (6)
C21	C20	C19	1·353 (9)	118·9 (6)
C22	C21	C20	1·378 (8)	120·7 (5)
C17	C22	C21		121·3 (6)
C24	O23	Fe2	1·239 (7)	134·4 (4)
O25	C24	C26	1·267 (8)	117·1 (5)
O25	C24	O23		125·1 (5)
C26	C24	O23	1·505 (8)	117·8 (6)
Fe1	O25	C24		131·3 (4)
C27	C26	C31	1·369 (11)	119·8 (6)
C27	C26	C24		119·6 (5)
C31	C26	C24	1·380 (9)	120·6 (6)
C28	C27	C26	1·389 (12)	119·9 (8)
C29	C28	C27	1·361 (13)	120·3 (10)
C30	C29	C28	1·362 (14)	120·5 (8)
C31	C30	C29	1·400 (10)	119·8 (7)
C26	C31	C30		119·6 (6)
C33	O32	Fe2	1·258 (7)	132·6 (3)
O34	C33	C35	1·255 (7)	117·9 (5)
O34	C33	O32		125·7 (4)
C35	C33	O32	1·505 (6)	116·4 (5)
Fe1	O34	C33		131·9 (3)
C36	C35	C40	1·371 (10)	120·3 (5)
C36	C35	C33		120·7 (5)
C40	C35	C33	1·376 (9)	119·0 (6)
C37	C36	C35	1·395 (8)	121·1 (7)
C38	C37	C36	1·372 (13)	118·3 (8)
C39	C38	C37	1·402 (13)	121·2 (6)
C40	C39	C38	1·385 (8)	119·1 (8)
C35	C40	C39		119·9 (7)
C42	O41	Fe2	1·258 (7)	134·7 (4)
O43	C42	C44	1·260 (7)	117·8 (5)
O43	C42	O41		125·3 (5)
C44	C42	O41	1·496 (7)	117·0 (5)
Fe3	O43	C42		130·1 (3)
C45	C44	C49	1·378 (9)	119·3 (5)
C45	C44	C42		120·3 (5)
C49	C44	C42	1·391 (9)	120·4 (5)
C46	C45	C44	1·388 (10)	119·9 (7)
C47	C46	C45	1·382 (12)	119·8 (8)
C48	C47	C46	1·371 (12)	120·4 (7)
C49	C48	C47	1·369 (9)	119·8 (7)
C44	C49	C48		120·8 (7)
C51	O50	Fe2	1·259 (6)	130·6 (4)
O52	C51	C53	1·260 (8)	117·0 (4)
O52	C51	O50		125·0 (4)
C53	C51	O50	1·487 (7)	118·0 (5)
Fe3	O52	C51		134·3 (3)
C54	C53	C58	1·377 (10)	118·7 (5)
C54	C53	C51		121·1 (5)
C58	C53	C51	1·382 (7)	120·2 (6)
C55	C54	C53	1·392 (9)	120·2 (6)
C56	C55	C54	1·356 (10)	119·8 (8)
C57	C56	C55	1·355 (12)	120·9 (6)
C58	C57	C56	1·383 (8)	119·8 (6)
C53	C58	C57		120·5 (7)
C60	O59	Fe1	1·450 (8)	126·2 (4)
C62	O61	Fe3	1·424 (7)	128·3 (4)
C2A	O1A		1·405 (11)	
C3A	C2A	O1A	1·69 (2)	114·1 (7)
C2B	O1B		1·396 (11)	
C2C	O1C		1·270 (7)	
O3C	C2C	C4C	1·259 (8)	118·9 (5)
O3C	C2C	O1C		124·5 (7)
C4C	C2C	O1C	1·521 (10)	116·6 (5)
C5C	C4C	C9C	1·376 (9)	120·0 (6)
C5C	C4C	C2C		120·2 (6)
C9C	C4C	C2C	1·391 (10)	119·7 (5)
C6C	C5C	C4C	1·407 (12)	119·3 (7)
C7C	C6C	C5C	1·342 (13)	121·0 (7)
C8C	C7C	C6C	1·401 (11)	120·3 (8)
C9C	C8C	C7C	1·391 (11)	119·3 (7)
C4C	C9C	C8C		120·0 (6)

for the H atoms are from Stewart, Davidson & Simpson (1965). The linear absorption coefficient was calculated from values found in *International Tables for X-ray Crystallography*, (1974, Vol. IV, p. 55).* The figures were generated using *SHELXTL-Plus* (Sheldrick, 1987). Positional and thermal parameters for non-H atoms are listed in Table 1,

while the bond lengths and angles for the non-H atoms are listed in Table 2. The atom-labelling scheme is shown in Fig. 1 while a view showing the nearly D_{3h} symmetry of the complex is shown in Fig. 2.

Related literature. A review the chemical and physical properties of related triangular bridged structures has been reported (Cannon & White, 1988).

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The 1/2 Complex of Mercury(II) Trifluoroacetate and Pyridine *N*-Oxide

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Abstract. Bis(pyridine *N*-oxide)bis(trifluoroacetate)mercury(II), [Hg(C$_2$F$_3$O$_2$)$_2$(C$_5$H$_5$NO)$_2$], M_r = 616·8, monoclinic, $C2/c$, a = 12·52 (1), b = 21·33 (2), c = 7·62 (1) Å, β = 107·9 (1)°, V = 1936·4 Å^3, Z = 4, D_x = 2·12 g cm^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, μ = 77·06 cm^{-1}, $F(000)$ = 1160·0, T = 290 K, R = 0·022 for 887 unique observed reflections. Distorted octahedral coordination around the Hg atom on a twofold axis with Hg—O 2·167 (6) Å to trifluoroacetate, Hg—O 2·447 (5) Å to pyridine oxide and Hg—O 2·548 (5) Å to a further centrosymmetrically related pyridine oxide. Centrosymmetrically related Hg atoms are thus bridged along **c** by pyridine oxide. The CF$_3$ group shows disorder, occupying two alternative orientations.

Experimental. Crystals grown from an equimolar solution of mercury(II) trifluoroacetate and pyridine *N*-oxide in dichloromethane were acicular along **c**. Cell dimensions, initially from Weissenberg photographs [λ(Cu $K\alpha$) = 1·542 Å], refined from setting angles of 20 reflexions in the range $10 < 2\theta < 40°$ on STADI-2 two-circle diffractometer (graphite-monochromatized Mo $K\alpha$). This was also used for measurement of 952 unique intensities from a crystal

$0·2 \times 0·09 \times 0·05$ mm for the layers $hk0$ to $hk7$, standard measured every 20 reflexions, < 5% intensity variation, h −11 to 10, k 0 to 20, max. $(\sin\theta)/\lambda$ = $0·55$ Å^{-1}; variable ω scan, $2\theta'$ fixed, stationary background count. Lp correction and absorption correction (point by point integration, max. and min. transmission factors 0·501, 0·271) using the programs of *SHELX*76 (Sheldrick, 1976). The same programs were also used in the structure determination involving 887 reflexions for which $I > 3\sigma(I)$. Hg-atom position and confirmation of space group from Patterson maps. Positions of non-H atoms were from ΔF map which indicated disorder of the CF$_3$ group with occupation of two alternative orientations. During initial least-squares refinement, based on F, two sets of F-atom positions with complementary site occupations were refined alternately, the minor set [F(4), F(5), F(6)] with site occupation 0·25 were subsequently fixed in positions with refineable isotropic vibrations. Refinement of: Hg, position and U_{ij}; C, N, O and F(1), F(2), F(3), positions and U_{iso}; H, common U_{iso} with calculated positions (C—H = 1·08 Å) gave R = 0·039. Refinement of anisotropic U_{ij} for C, N, O and F atoms reduced R to 0·022 and significantly improved the e.s.d.'s, the results quoted refer to this refinement. Interlayer scale factors were refined at an intermediate stage. Final $\Delta/\sigma < 0·04$, variations in final F map +0·43 to −0·46 e Å^{-3}. R

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