After anisotropic refinement of this model all H atoms except those belonging to water molecules in the coordination sphere and to $O(5W)$ were located in a difference Fourier map and added to the model for final calculation with fixed isotropic thermal parameters of 0.08 Å^2, benzene H left riding on the C atoms with C—H bond lengths fixed to 1.08 Å, and constraints of O—H bond lengths being not shorter than 0.85 Å and angles H—O—H in the water molecule not smaller than $104°$. Full-matrix refinement converged at $R = 0.074$. Unusually high thermal parameters resulted for water O atoms in the coordination sphere [$O(1W)$: $B_{eq} = 21.45$, $O(2W)$: $B_{eq} = 10.09 \text{ Å}^2$] and the crystal water on the centre of symmetry [$O(5W)$: $B_{eq} = 17.71 \text{ Å}^2$]. Therefore a full-matrix refinement in Cc was tried, which resulted in R values of $R = 0.037$, $wR = 0.033$ [$w = 1/\sigma^2(F_o)$] and thermal parameters of $B_{eq} = 5.38$ [$O(1W)$], 2.95 [$O(2W)$], 4.57 [$O(3W)$], 8.84 Å^2 [$O(5W)$]. On the other hand, severe distortions in the geometry of the anions were observed, e.g. bond lengths in the benzene rings ranging from 1.352 to 1.448 Å. This was taken as a strong indication for Cc not being the correct space group. Thus refinement in $C2/c$ was performed treating part of the atoms as disordered (s.o.f. $= 0.5$) by untying the coordination water O atoms from the twofold axis, and the crystal water $O(5W)$ from the centre of symmetry. This refinement converged at $R = 0.065$, $wR = 0.061$ [$w = 1/\sigma^2(F_o)$]. Max. Δ/σ on final cycle $= 0.007$, $\Delta\rho$ fluctuations within $+0.60$ and -0.61 e Å^{-3}. All calculations

were performed with SHELX76 (Sheldrick, 1976) using scattering factors of Cromer & Mann (1968); drawings by PLUTO (Motherwell & Clegg, 1976).

Atomic positions and equivalent isotropic thermal parameters are given in Table 1,* bond lengths and angles in Table 2. Fig. 1 shows the surrounding of the Cu atom, Fig. 2 a perspective view of the unit cell.

Related literature. An analogous coordination sphere was observed in copper(II) 1,2,4,5-benzenetetra-carboxylate decahydrate (Usubaliev, Shnulin & Mamedov, 1982) and in catena-triaqua-μ-[1,3,5-benzenetricarboxylato(2−)]-copper(II) (Pech & Pickardt, 1988).

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Structure of an Iron(II) Bromide Complex of 15-Crown-5 at 163 K

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Abstract. (1,4,7,10,13-Pentaoxacyclopentadecane)-iron(II) bromide dichloromethane solvate (I), $[Fe(C_{10}H_{20}O_5)]^{2+}.2Br^-.CH_2Cl_2$, $M_r = 520.87$, orthorhombic, $P2_12_12_1$, $a = 12.088 (9)$, $b = 13.425 (7)$, $c = 11.235 (6) \text{ Å}$, $V = 1823 (3) \text{ Å}^3$, $Z = 4$, $D_x = 1.897 \text{ g cm}^{-3}$, $\lambda(\text{Mo } K\alpha) = 0.71069 \text{ Å}$, $\mu = 54.791 \text{ cm}^{-1}$, $F(000) = 1032$, $T = 163 \text{ K}$, $R = 0.0436$ for 1875 reflections ($F \geq 4\sigma_F$). The Fe^{2+} ion is coordinated to the five oxygens of the crown and to the two Br^- ions. The range of $Fe\cdots O$ distances is $2.175 (6)$–$2.265 (6) \text{ Å}$. The average C—O and C—C bond lengths in the crown are $1.437 (8)$ and $1.502 (11) \text{ Å}$. The O atoms are nearly planar [r.m.s. deviation: $0.0947 (3) \text{ Å}$], with the cation only $-0.0122 (12) \text{ Å}$ from the mean plane. The average O—Fe—O angle for adjacent O atoms is $72.1 (16)°$. All C—O and C—C torsion angles in the crown are *anti* and *gauche*, respectively. The Fe—Br distances are $2.6228 (15)$ and $2.6281 (14) \text{ Å}$. The CH_2Cl_2 molecule is well behaved.

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Experimental. The title compound was prepared by the reaction of 15-crown-5 with $FeBr_2$ in methylene chloride in an inert atmosphere. Crystals, due to moisture sensitivity, were mounted in a Lindemann capillary tube with grease using a Schlenk apparatus. The grease prevented an accurate measurement of the crystal size. The maximum dimension was 0·5 mm. A summary of data collection and structural refinement is given in Table 1.

(1)
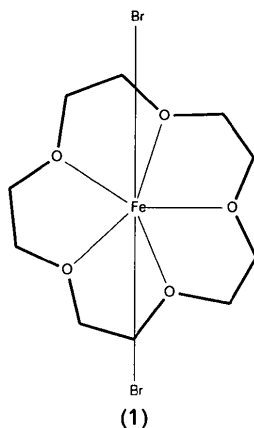
The Fe and Br atomic positions were deduced from a sharpened Patterson map. The structure was developed from five subsequent Fourier and difference Fourier maps. Although some H atoms were located in a difference map, all H atoms were allowed to ride on their respective C atoms in ideal positions (C—H = 1·00 Å and H—C—H = 109·5°). Only non-H atomic positional and anisotropic thermal parameters and a single isotropic thermal parameter for H atoms were refined by full-matrix least squares (*SHELX*76; Sheldrick, 1976). Twenty reflections were not used because of lopsided backgrounds. Scattering factors and anomalous-dispersion corrections for all non-H atoms were taken from International Tables for X-ray Crystallography (1974); H-atom scattering factors were taken from Stewart, Davidson & Simpson (1965). Atomic parameters are listed in Table 2* and coordination geometry, bond lengths and bond angles are given in Table 3. Atom labeling is given on projections in Fig. 1. Packing of the unit cell is illustrated in Fig. 2. Principal computer programs are given by Gadol & Davis (1982).

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* Tables of anisotropic thermal parameters, H-atom parameters, bond lengths and angles involving H atoms, least-squares planes and structure factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52862 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Summary of data collection and refinement for* (1)

(A) Data collection (163 K)*

Mode	ω scan
Scan range, °	1·0
Background	Stationary counts at scan extremities for total scan time
Scan rate, ° min⁻¹	2·5–6·0
Exposure time, h	20
Check reflections	002, 020, 200, 1Ī1
Stability correction range on *I*	Not applied (< 1% variation)
2θ range, °	4·0–55·0
Range in *hkl*, min.	0,0,0
max.	17,19,16
Total reflections, measured, unique	2375, 2375
Crystal dimensions, mm	< 0·5 (in grease in capillary)
Transmission factor range	Not applied

(B) Structure refinement†

Reflections used, m $(F \geq 4\sigma_F)$	1875
No. of variables, n	192
Extinction parameter	2·5 (14) × 10⁻⁸
Goodness of fit, S	1·104
R, wR	0·0436, 0·0412
R for all data	0·0675
Max. shift/e.s.d.	0·003
Max., min. density in diff. map, e Å⁻³	0·77, −0·84

* Syntex $P2_1$ autodiffractometer with a graphite monochromator and a Syntex LT-1 inert-gas (N_2) low-temperature delivery system. Data reduction was carried out as described by Riley & Davis (1976). Unit-cell parameters were obtained by least-squares refinement of the setting angles of 15 reflections with $20·7 < 2\theta < 28·1°$.

† Function minimized was $\sum w(|F_o| - |F_c|)^2$, where $w = \sigma_F^{-2}$. $\sigma_F = F\sigma_I/2I$; $\sigma_I = [N_{pk} + N_{bg1} + N_{bg2} + (0·04I)^2]^{1/2}$.

Table 2. *Positional and equivalent isotropic thermal parameters for* non-H *atoms in* (1)

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

	x	y	z	U_{eq} (Å²)
Fe	0·24196 (9)	0·24409 (9)	0·26645 (10)	0·0161 (3)
Br1	0·36150 (7)	0·37374 (8)	0·38460 (7)	0·0217 (2)
Br2	0·11051 (7)	0·12526 (8)	0·14582 (7)	0·0228 (3)
C	0·8319 (7)	0·2099 (7)	0·2958 (8)	0·026 (3)
C11	0·7558 (2)	0·0999 (2)	0·2654 (2)	0·0280 (7)
C12	0·7768 (2)	0·3119 (2)	0·2138 (2)	0·0395 (9)
O1	0·3477 (5)	0·2631 (5)	0·1039 (5)	0·023 (2)
C2	0·3387 (7)	0·3616 (8)	0·0545 (7)	0·028 (3)
C3	0·2170 (6)	0·3852 (8)	0·0501 (7)	0·022 (3)
O4	0·1796 (4)	0·3740 (5)	0·1721 (4)	0·017 (2)
C5	0·0673 (7)	0·4021 (7)	0·1961 (8)	0·023 (3)
C6	0·0554 (7)	0·3937 (6)	0·3291 (7)	0·021 (3)
O7	0·0870 (5)	0·2934 (4)	0·3581 (5)	0·019 (2)
C8	0·0882 (7)	0·2695 (6)	0·4821 (6)	0·019 (3)
C9	0·1233 (8)	0·1624 (7)	0·4921 (8)	0·026 (3)
O10	0·2293 (5)	0·1525 (4)	0·4352 (5)	0·022 (2)
C11	0·2617 (8)	0·0508 (6)	0·4140 (9)	0·031 (3)
C12	0·3705 (7)	0·0506 (7)	0·3523 (9)	0·030 (3)
O13	0·3735 (5)	0·1325 (5)	0·2722 (5)	0·028 (2)
C14	0·4488 (7)	0·1261 (9)	0·1739 (8)	0·033 (3)
C15	0·4596 (7)	0·2298 (7)	0·1246 (9)	0·030 (3)

Related literature. The structure of a bis(12-crown-4)–iron(II) sandwich complex has been reported (Meier & Rihs, 1985) but no distances were given. The Fe—O distances are longer than observed in less

$$[Fe(C_{10}H_{20}O_5)]^{2+}.2Br^-.CH_2Cl_2$$

rigid systems such as the bis(tartrato-$O,O',$-O'')iron(II) complex reported by Ivanov & Kosoy (1975) (1·983–2·149 Å) and the aquacitratoiron(II) hexaaquairon(II) complex of Strouse, Layten & Strouse (1977) (2·085–2·178 Å). In fact, the Fe—O distances observed here are similar to the Fe—N distances (2·23 Å) observed in some 2,13-dimethyl-3,6,9,12,18-pentaazabicyclo[12.3.1]octadeca-1(18),2,-12,14,16-pentaeneiron(III) complexes (Fleischer & Hawkinson, 1967).
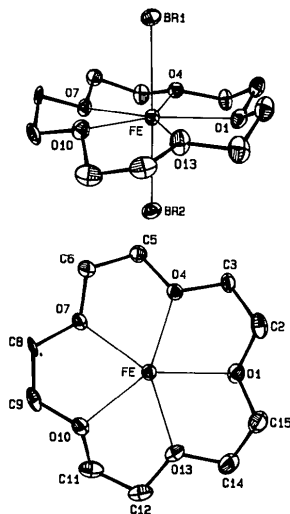
Fig. 1. Perspective drawings of (1) showing atom labeling. In both views, H atoms and the methylene chloride solvate have been omitted for clarity. In the bottom view, the Br atoms have also been omitted. Thermal ellipsoids are drawn at the 50% probability level.
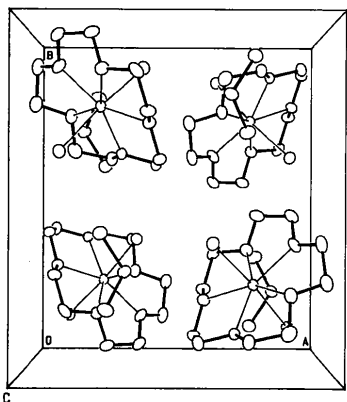
Table 3. *Bond lengths* (Å), *bond angles* (°) *and torsion angles* (°) *in* (1)

1	2	3	1—2	1—2—3
Br1	Fe	Br2	2·6228 (15)	175·50 (6)
Br1	Fe	O1		91·3 (2)
Br1	Fe	O4		84·6 (2)
Br1	Fe	O7		91·84 (15)
Br1	Fe	O10		88·5 (2)
Br1	Fe	O13		92·2 (2)
Br2	Fe	O1	2·6281 (14)	89·6 (2)
Br2	Fe	O4		91·5 (2)
Br2	Fe	O7		84·87 (15)
Br2	Fe	O10		93·5 (2)
Br2	Fe	O13		92·2 (2)
O1	Fe	O4	2·244 (6)	73·1 (2)
O4	Fe	O7	2·175 (6)	72·4 (2)
O7	Fe	O10	2·237 (6)	73·7 (2)
O10	Fe	O13	2·265 (6)	69·6 (2)
O13	Fe	O1	2·186 (6)	71·8 (2)
Cl1	C	Cl2	1·773 (9)	110·3 (5)
Cl2	C		1·781 (9)	
C2	O1	C15	1·438 (12)	114·7 (6)
C3	C2	O1	1·506 (12)	106·3 (7)
O4	C3	C2	1·451 (9)	104·5 (6)
C5	O4	C3	1·435 (10)	116·4 (6)
C6	C5	O4	1·506 (12)	104·9 (7)
O7	C6	C5	1·437 (10)	105·6 (7)
C8	O7	C6	1·430 (9)	115·6 (6)
C9	C8	O7	1·503 (13)	106·8 (6)
O10	C9	C8	1·438 (11)	107·9 (7)
C11	O10	C9	1·441 (10)	113·8 (7)
C12	C11	O10	1·487 (13)	108·6 (7)
O13	C12	C11	1·421 (11)	108·4 (7)
C14	O13	C12	1·435 (11)	117·2 (7)
C15	C14	O13	1·50 (2)	106·4 (8)
O1	C15	C14	1·443 (10)	105·4 (7)

C2—O1—C15—C14	−179·2 (7)	C15—O1—C2—C3	−175·1 (7)
O1—C2—C3—O4	56·9 (9)	C2—C3—O4—C5	173·9 (8)
C3—O4—C5—C6	−173·9 (7)	O4—C5—C6—O7	−55·7 (8)
C5—C6—O7—C8	176·6 (7)	C6—O7—C8—C9	−179·9 (7)
O7—C8—C9—O10	57·3 (8)	C8—C9—O10—C11	−166·7 (7)
C9—O10—C11—C12	178·5 (7)	O10—C11—C12—O13	−36·4 (10)
C11—C12—O13—C14	−156·9 (7)	C12—O13—C14—C15	−164·5 (7)
O13—C14—C15—O1	−52·4 (9)		

Fig. 2. Packing diagram of (1) as viewed along the c axis. H atoms have been omitted for clarity.

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