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Aquabis(tetrahydrofuran)hexakis(trichloroacetato)copper(II)diiron(III) hexane solvate

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In the oxo-centered title compound, $[CuFe_2O(C_2Cl_3O_2)_6(C_4H_8O)_2(H_2O)]\cdot C_6H_{14}$, the central O atom is linked to the three metal atoms, each of which is linked to four trichloroacetate anions. The Fe atoms are coordinated by a tetrahydrofuran molecule and the Cu atom by a water molecule. Two trimetallic units are linked by a pair of hydrogen bonds across a center of inversion into a dimeric entity.

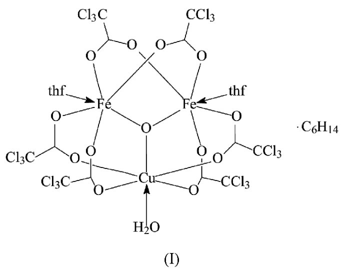
Key indicators

Single-crystal X-ray study
T = 181 K
Mean σ(C–C) = 0.010 Å
Disorder in solvent or counterion
R factor = 0.067
wR factor = 0.151
Data-to-parameter ratio = 17.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

Two attempts at the synthesis of the mixed-metal carboxylates represented by the formula $M^{II}M^{III}_2O(O_2CR)_6L_3$, where R is CF_3 and L is tetrahydrofuran (THF), gave the compound that has two THF and one H_2O molecule (Amini *et al.*, 2004*a*,*b*). The title compound, (I), was obtained in an attempt at duplicating the synthesis of $CuFe_2O(O_2CCCl_3)_6(C_4H_8O)_3$, with trichloroacetic acid as the carboxylic acid source in place of trifluoroacetic acid. The compound crystallizes as a hexane solvate; the $MFe_2O(O_2CCCl_3)(THF)_3$ complexes, where M is Mn, Co and Ni (Wang & Yu, 1990), do not contain water or alkane molecules. The metal-organic entity has a central O atom that is linked to three metal atoms which are arranged in a triangular configuration, with the O atom in the middle and each of the six carboxylate groups bridging two metal atoms; the Fe atoms are each coordinated by a tetrahydrofuran molecule and the Cu atom by the water molecule. Two $CuFe_2O(O_2CCCl_3)_6(C_4H_8O)_2(H_2O)$ units (Fig. 1) are linked by a pair of hydrogen bonds across a center of inversion, forming a hydrogen-bonded dimer. The crystalline compound turns opaque when left in the open, probably owing to the loss of solvent molecules. The compound has a structure similar to that of the trifluoroacetate analog (Amini *et al.*, 2004*a*).



(I)

Experimental

The procedure for preparing the title compound was adapted from that reported for the synthesis of $MFe_2O(O_2CCCl_3)(THF)_3$, where M is Mn, Co and Ni (Wang & Yu, 1990). Crystals were grown from a THF solution of the compound to which several drops of hexane were added; the compound is extremely soluble in THF. The previous study used pentane rather than hexane, and it was not incorporated in the crystal structure.

Crystal data

$[CuFe_2O(C_2Cl_3O_2)_6(C_4H_8O)_2$-
$(H_2O)]\cdot C_6H_{14}$
$M_r = 1413.86$
Monoclinic, $P2_1/n$
$a = 13.2528\ (9)$ Å
$b = 13.6796\ (9)$ Å
$c = 29.358\ (2)$ Å
$\beta = 92.430\ (1)^\circ$
$V = 5317.6\ (6)$ Å^3
$Z = 4$
$D_x = 1.766$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6734 reflections
$\theta = 2.6$–26.3°
$\mu = 1.90$ mm^{-1}
$T = 181\ (2)$ K
Plate, red
$0.50 \times 0.23 \times 0.10$ mm

Data collection

Bruker P4/SMART diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.617$, $T_{max} = 0.833$
36718 measured reflections
10348 independent reflections
6477 reflections with $I > 2\sigma(I)$
$R_{int} = 0.066$
$\theta_{max} = 26.5^\circ$
$h = -5 \rightarrow 16$
$k = -17 \rightarrow 16$
$l = -36 \rightarrow 36$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.067$
$wR(F^2) = 0.151$
$S = 1.10$
10348 reflections
592 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0334P)^2 + 36.2345P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.98$ e Å^{-3}
$\Delta\rho_{min} = -0.78$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cu1—O1	2.046 (5)	Fe2—O11	2.079 (4)
Cu1—O3	2.274 (5)	Fe2—O13	2.120 (5)
Cu1—O5	2.348 (5)	Fe2—O15	1.896 (4)
Cu1—O15	1.956 (4)	Fe3—O15	1.899 (4)
Cu1—O16	1.994 (5)	Fe3—O6	1.999 (5)
Cu1—O7	2.018 (5)	Fe3—O8	2.034 (5)
Fe2—O4	2.009 (5)	Fe3—O10	2.049 (5)
Fe2—O2	2.053 (4)	Fe3—O12	2.069 (5)
Fe2—O9	2.080 (5)	Fe3—O14	2.112 (4)
O1—Cu1—O3	89.5 (2)	O9—Fe2—O11	92.5 (2)
O1—Cu1—O5	82.9 (2)	O9—Fe2—O13	82.4 (2)
O1—Cu1—O7	171.0 (2)	O9—Fe2—O15	96.9 (2)
O1—Cu1—O15	94.8 (2)	O11—Fe2—O13	83.3 (2)
O1—Cu1—O16	86.5 (2)	O11—Fe2—O15	93.1 (2)
O3—Cu1—O5	168.9 (2)	O13—Fe2—O15	176.3 (2)
O3—Cu1—O7	89.4 (2)	O6—Fe3—O8	92.4 (3)
O3—Cu1—O15	94.8 (2)	O6—Fe3—O10	165.2 (2)
O3—Cu1—O16	86.2 (2)	O6—Fe3—O12	87.4 (2)
O5—Cu1—O7	96.8 (2)	O6—Fe3—O14	84.3 (2)
O5—Cu1—O15	93.9 (2)	O6—Fe3—O15	99.4 (2)
O5—Cu1—O16	85.2 (2)	O8—Fe3—O10	85.9 (2)
O7—Cu1—O15	94.2 (2)	O8—Fe3—O12	170.8 (2)
O7—Cu1—O16	84.5 (2)	O8—Fe3—O14	84.6 (2)
O15—Cu1—O16	178.3 (2)	O8—Fe3—O15	95.6 (2)
O2—Fe2—O4	93.1 (2)	O10—Fe3—O12	91.9 (2)
O2—Fe2—O9	166.6 (2)	O10—Fe3—O14	80.9 (2)
O2—Fe2—O11	87.7 (2)	O10—Fe3—O15	95.4 (2)
O2—Fe2—O13	84.3 (2)	O12—Fe3—O15	93.5 (2)
O2—Fe2—O15	96.4 (2)	O12—Fe3—O14	86.3 (2)
O4—Fe2—O9	84.2 (2)	O14—Fe3—O15	176.3 (2)
O4—Fe2—O11	168.9 (2)	Cu1—O15—Fe2	119.3 (2)
O4—Fe2—O13	85.9 (2)	Cu1—O15—Fe3	118.4 (2)
O4—Fe2—O15	97.8 (2)	Fe2—O15—Fe3	122.3 (2)

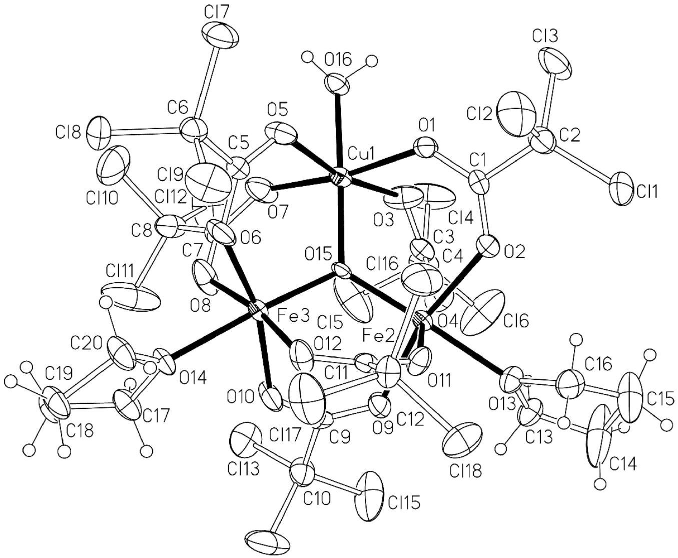


Figure 1
*ORTEP*II (Johnson, 1976) plot of (I); displacement ellipsoids are drawn at the 50% probability level. H atoms are drawn as spheres of arbitrary radii. The disordered hexane solvent molecule is not shown.

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O16—HO16a···O1^{i}	0.848 (10)	2.22 (4)	2.967 (7)	147 (7)
O16—HO16b···O5^{i}	0.849 (10)	2.09 (4)	2.872 (6)	152 (7)

Symmetry code: (i) $1 - x, 1 - y, 1 - z$.

The hexane solvent molecule is disordered; this was regarded as a 1:1 disorder as the refinement of the occupancy led to an unacceptable value. The C—C distances were retrained to 1.54 (1) Å and the 1,3-related C···C distances to 2.51 (1) Å. The displacement parameters of the unprimed and primed atoms were set equal to each other. The H atoms were placed at calculated positions (C—H = 0.95 Å for the aromatic, 0.99 Å for the methylene and 0.98 Å for the methyl H atoms), and were included in the refinement in the riding-model approximation; $U_{iso}(H) = 1.2U_{eq}(C)$, except for the methyl group, for which $U_{iso}(H) = 1.5U_{eq}(C)$. The water H atoms were located and refined with an O—H restraint of 0.85 (1) Å.

The three metal atoms that occupy general positions in the asymmetric unit could, in principle, be statistically disordered in the ratio of 1Cu to 2Fe (as required by the stoichiometry). The structure of solvent-free $NiFe_2O(O_2CCCl_3)(THF)_3$ was refined with 1/3 occupancy for the Ni and 2/3 occupancy for the two Fe atoms (Wang & Yu, 1990). The related trifluoroactetates are statistically disordered, and the disorder was satisfactorily modeled (Amini *et al.*, 2004*a*,*b*). The present structure was initially refined with disorder for the metal atoms. The refinement gave an occupancy of approximately 0.90 for Cu on one site and occupancies of 0.05 for each of the other two sites. As the occupancy of Cu1 was nearly unity whereas those of the atoms Cu2 and Cu3 were nearly zero, the structure was then refined with no disorder for the metal atoms.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

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