

Low-temperature tris(*tert*-butyl 3-oxobutanoato)iron(III)

Usha K. Urs *et al.*

Electronic paper

This paper is published electronically. It meets the data-validation criteria for publication in Acta Crystallographica Section C. The submission has been checked by a Section C Co-editor though the text in the 'Comments' section is the responsibility of the authors.

Low-temperature tris(*tert*-butyl 3-oxobutanoato)iron(III)

Usha K. Urs,^a K. Shalini,^b S. A. Shivashankar^b and T. N. Guru Row^{a*}

^aSolid State and Structural Chemistry Unit, Indian Institute of Science, Bangalore 560 012, India, and ^bMaterials Research Centre, Indian Institute of Science, Bangalore 560 012, India
Correspondence e-mail: ssctng@sscu.iisc.ernet.in

Received 28 August 2000

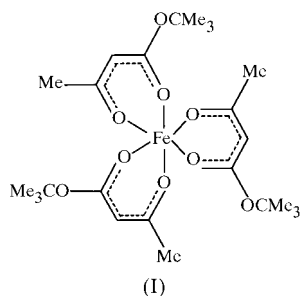
Accepted 11 September 2000

Data validation number: IUC0000258

The low-temperature (173 K) structure of the title complex, [Fe(C₈H₁₃O₃)₃], a metal–organic chemical vapour deposition (MOCVD) precursor, has been analyzed. The Fe atom is octahedrally coordinated and the three chelate rings are found to be significantly non-planar, adopting a half-chair conformation with the Fe atom out of the plane.

Comment

We have been exploring the use of metal–organic (MO) complexes as precursors for chemical vapour deposition (CVD) of thin films (Patnaik *et al.*, 1996). In the title compound, (I), an iron complex, the ligand has *tert*-butoxy as the terminal group on one side and methyl on the other. The bulky groups render the complex more volatile, a very desirable quality for CVD.



The coordination geometry is essentially octahedral. The Fe–O bond lengths range from 1.945 (3) to 2.048 (3) Å and the O–Fe–O bond angles within the chelate rings range from 86.9 (1) to 88.0 (1)°. The bite O···O distances are comparable to those observed in similar iron complexes (Pfluger & Haradem, 1983; Soling, 1976). The six-membered chelate rings are significantly non-planar and adopt a half-chair conformation with the Fe atom out of the plane by about 0.3 Å, unlike the boat conformation observed in other iron

complexes (Clegg, 1985; Calogero *et al.*, 1997). It is noted that the average Fe–O lengths in each chelate ring are 1.948 and 2.039 Å, the shorter length being on the same side as the methyl group.

The angles between the best planes fitted through the six atoms of each ring are 100, 91 and 105°. The intramolecular short contacts are only of the C–H···O type, involving the *tert*-butyl C atom and the chelate-ring O atom. The distances range from 2.881 (7) to 2.991 (6) Å and the C–H···O angles vary from 112.3 (3) to 118.1 (3)°. There are no significant intermolecular C–H···O interactions and the packing is dominated by van der Waals interactions.

Experimental

The complex was synthesized by dissolving *tert*-butyl acetoacetate (9.8 ml, 60 mmol) and potassium acetate (5.88 g, 60 mmol) in ethanol (30 ml). This was followed by the addition of iron(III) chloride (5.4 g, 20 mmol) dissolved in water (20 ml). The mixture was stirred under ice-cold conditions for 6 h and the precipitate which formed was filtered off, washed with water and recrystallized from cyclohexane. Single crystals were obtained by slow evaporation from a solution in cyclohexane.

Crystal data

[Fe(C ₈ H ₁₃ O ₃) ₃]	$D_x = 1.278 \text{ Mg m}^{-3}$
$M_r = 527.4$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 10 463 reflections
$a = 9.598 (3) \text{ \AA}$	$\theta = 2.5\text{--}28.5^\circ$
$b = 16.056 (4) \text{ \AA}$	$\mu = 0.596 \text{ mm}^{-1}$
$c = 18.112 (4) \text{ \AA}$	$T = 173 (2) \text{ K}$
$\beta = 100.88 (2)^\circ$	Prism, red
$V = 2741 (1) \text{ \AA}^3$	$0.15 \times 0.15 \times 0.10 \text{ mm}$
$Z = 4$	

Data collection

Rigaku AFC-8 coupled with Mercury CCD diffractometer	$R_{\text{int}} = 0.06$
ω scans	$\theta_{\text{max}} = 30.59^\circ$
31 072 measured reflections	$h = 0 \rightarrow 13$
7261 independent reflections	$k = -22 \rightarrow 22$
3190 reflections with $I > 2\sigma(I)$	$l = -24 \rightarrow 23$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.062$	$w = 1/[\sigma^2(F_o^2) + (0.0144P)^2]$
$wR(F^2) = 0.173$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.906$	$(\Delta/\sigma)_{\text{max}} = 0.007$
7261 reflections	$\Delta\rho_{\text{max}} = 0.54 \text{ e \AA}^{-3}$
319 parameters	$\Delta\rho_{\text{min}} = -0.56 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (Å, °).

Fe1–O5	1.945 (3)	Fe1–O4	2.030 (3)
Fe1–O7	1.946 (3)	Fe1–O1	2.039 (3)
Fe1–O2	1.952 (3)	Fe1–O8	2.048 (3)
O5–Fe1–O4	87.7 (1)	O2–Fe1–O1	86.9 (1)
O7–Fe1–O4	172.1 (1)	O7–Fe1–O8	88.0 (1)
O5–Fe1–O1	173.4 (1)	O2–Fe1–O8	168.9 (1)

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

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
C14—H14A \cdots O4	0.98	2.34	2.928 (5)	117.6 (3)
C7—H7C \cdots O1	0.98	2.47	2.991 (6)	113.2 (3)
C13—H13C \cdots O4	0.98	2.42	2.932 (6)	112.3 (3)
C21—H21C \cdots O8	0.98	2.38	2.930 (6)	114.6 (3)
C22—H22A \cdots O8	0.98	2.44	2.988 (6)	114.6 (3)
C6—H6A \cdots O1	0.98	2.29	2.881 (7)	118.1 (3)

The positional parameters of all the H atoms were calculated geometrically and fixed with $U(H) = 1.2U_{eq}$ (parent atom).

Data collection: *CrystalClear* (Rigaku Corporation, 1999); cell refinement: *CrystalClear* (Rigaku, 1999); data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1998).

We thank Dr T. E. Concolino of the Molecular Structure Corporation, Texas, USA, for data collection. KU thanks IISc for a research associateship. KS thanks CSIR for a research fellowship.

References

- Calogero, S., Stievano, L., Diamandescu, L., Mihaila-Tarabasanu, D. & Valle, G. (1997). *Polyhedron*, **16**, 3953–3966.
- Clegg, W. (1985). *Acta Cryst.* **C41**, 1164–1166.
- Farrugia, L. J. (1998). *WinGX*. University of Glasgow, Scotland.
- Patnaik, S., Guru Row, T. N., Lakshmi, R., Anjana, D., Goswami, J., Shivashankar, S. A., Chandrasekaran, S. & Robinson, W. T. (1996). *Acta Cryst.* **C52**, 891–894.
- Pflugger, C. E. & Haradem, P. S. (1983). *Inorg. Chim. Acta*, **69**, 141–146.
- Rigaku (1999). *CrystalClear*. Rigaku Corporation, Tokyo, Japan.
- Sheldrick, G. M. (1985). *SHELXS86*. In *Crystallographic Computing 3*, edited by G. M. Sheldrick, C. Kruger & R. Goddard, pp. 175–189. Oxford University Press.