

M. S. Dharmaprakash,^a
S. Thamocharan,^{b*}
Gururaj M. Neelgund^c and
S. A. Shivashankar^c

^aDepartment of Chemistry, B.M.S. College of Engineering, Bangalore 560 019, India,

^bMolecular Biophysics Unit, Indian Institute of Science, Bangalore 560 012, India, and

^cMaterials Research Centre, Indian Institute of Science, Bangalore 560 012, India

Correspondence e-mail: thamu_as@yahoo.com

Key indicators

Single-crystal X-ray study

$T = 153$ K

Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å

R factor = 0.058

wR factor = 0.157

Data-to-parameter ratio = 15.4

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

Tris(*tert*-butyl 3-oxobutanoato- $\kappa^2\text{O}^1, \text{O}^3$)-aluminium(III) at 153 K

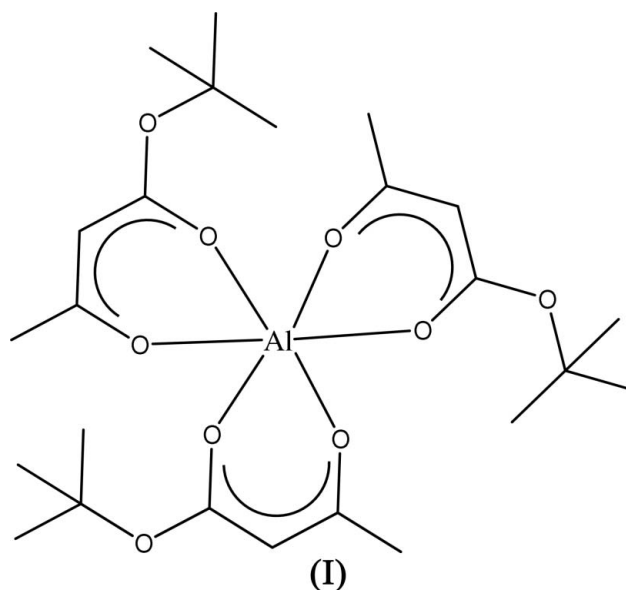
In the title complex, $[\text{Al}(\text{C}_8\text{H}_{13}\text{O}_3)_3]$, a potential metal-organic chemical vapour deposition (MOCVD) precursor, three bidentate ligand molecules are bound to the metal atom, forming an octahedral geometry. Two non-planar six-membered chelate rings adopt screw-boat conformations, while the third ring has a conformation that lies about halfway between an envelope and a screw-boat.

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Comment

We have been investigating the use of metal-organic (MO) complexes as precursors for chemical vapour deposition (CVD) for thin films (Patnaik *et al.*, 1996; Urs *et al.*, 2000, 2003*a,b*; Urs, Anitha *et al.*, 2001; Urs, Shalini *et al.*, 2001). The structure determination of the title complex, (I), a possible MOCVD precursor, is part of our ongoing project. Complex (I) is isostructural with the Fe complex, (II), *viz.* tris(*tert*-butyl 3-oxobutanoato)iron(III) (Urs *et al.*, 2000).



In (I), the Al atom is coordinated by the ketone O atoms of three bidentate ligands, forming an octahedral geometry (Fig. 1). Two of the non-planar six-membered chelate rings, Al/O1/C3/C2/C1/O2 and Al/O4/C11/C10/C9/O5, adopt screw-boat conformations, while the third non-planar chelate ring, Al/O7/C17/C18/C19/O8, has a conformation that lies about halfway between an envelope and a screw-boat. In (II), all three non-planar chelate rings adopt half-chair conformations (Urs *et al.*, 2000). Each chelate ring is approximately perpendicular to each of the other two chelate rings. The $M-\text{O}$ bond

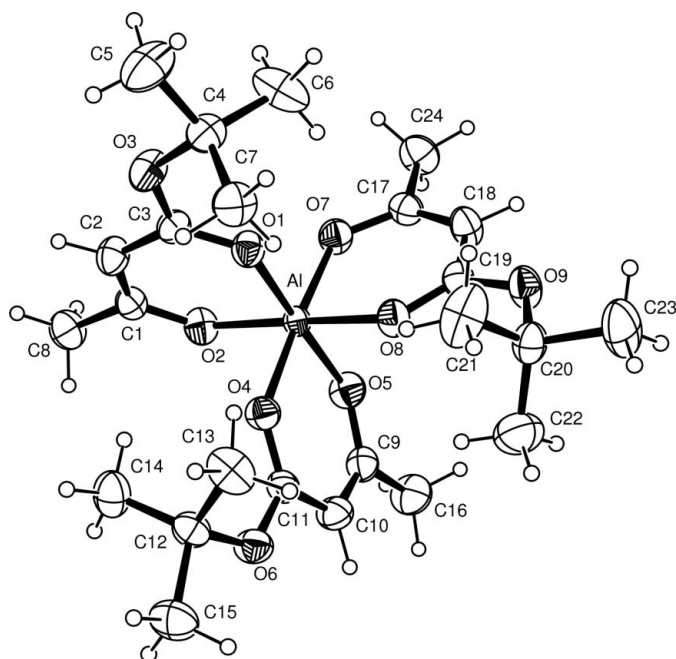


Figure 1
The molecular structure of (I), showing 30% probability displacement ellipsoids and atom-numbering scheme.

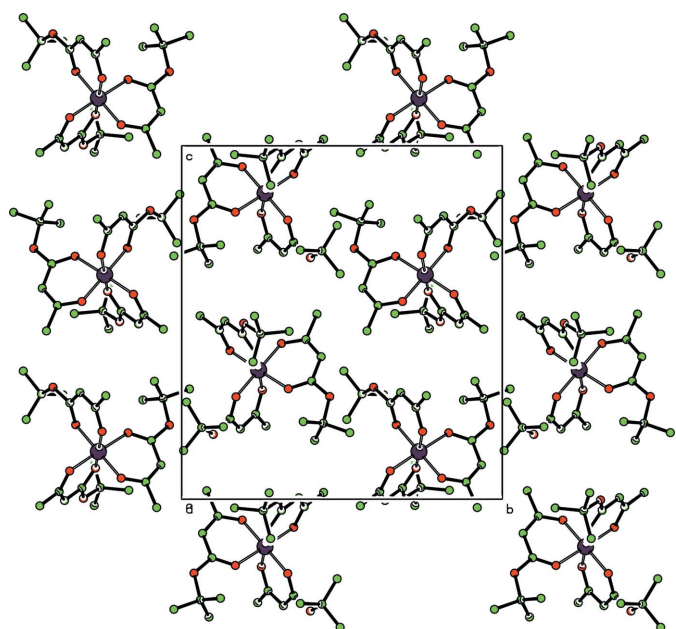


Figure 2
The crystal packing of (I), viewed along the *a* axis. All H atoms have been omitted for clarity.

lengths and O—M—O bond angles in (I) and (II) are different, perhaps as a result of the steric constraint imposed by different ionic radii: 0.79 Å for Fe³⁺ and 0.68 Å for Al³⁺ (Shannon, 1976). In (I), the Al—O bond lengths range from 1.8572 (19) to 1.951 (2) Å, and O—Al—O angles within the chelate rings vary from 86.9 (1) to 88.0 (1)°. In (II), the Fe—O bond lengths range from 1.945 (3) to 2.048 (3) Å and O—Fe—O angles within the chelate rings vary from 86.9 (1) to 88.0 (1)°.

In (I), two of the three methyl groups in each *tert*-butyl group act as donors for weak intramolecular interactions with keto O atoms within the chelate rings (Table 2). Each interaction has a graph-set motif *S*(6) (Bernstein *et al.*, 1995). Similar interactions were observed in (II) (Urs *et al.*, 2000). As in (II), the crystal packing in (I) (Fig. 2) is dominated by van der Waals interactions.

Experimental

The title complex was synthesized by dissolving potassium acetate (2.94 g, 30 mmol) and *tert*-butyl 3-oxobutanoate (4.9 ml, 30 mmol) in 30% ethanol. This was followed by the addition of Al(NO₃)₃·9H₂O (3.75 g, 10 mmol). The resultant mixture was stirred under ice-cold conditions for 30 min and the precipitate formed was filtered off, washed with distilled water, suction-dried and recrystallized from cyclohexane. Single crystals suitable for X-ray analysis were obtained by slow evaporation at room temperature.

Crystal data

[Al(C₈H₁₃O₃)₃]
M_r = 498.53
 Monoclinic, *P*2₁/*n*
a = 9.5660 (16) Å
b = 16.241 (3) Å
c = 18.211 (3) Å
 β = 100.464 (3)°
V = 2782.2 (8) Å³
Z = 4

D_x = 1.190 Mg m⁻³
 Mo *K*α radiation
 Cell parameters from 7479 reflections
 θ = 1.0–25.0°
 μ = 0.12 mm⁻¹
T = 153 (2) K
 Block, colourless
 0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
T_{min} = 0.936, *T_{max}* = 0.977
 22128 measured reflections

4912 independent reflections
 3533 reflections with *I* > 2σ(*I*)
R_{int} = 0.041
 θ_{\max} = 25.0°
h = -11 → 11
k = -19 → 17
l = -21 → 21

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.058
wR (*F*²) = 0.158
S = 1.06
 4912 reflections
 319 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0797P)^2 + 0.4155P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.57 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.16 \text{ e } \text{Å}^{-3}$

Table 1
Selected geometric parameters (Å, °).

Al—O7	1.8572 (19)	O1—C3	1.251 (3)
Al—O2	1.860 (2)	O2—C1	1.286 (3)
Al—O5	1.8675 (19)	O4—C11	1.250 (3)
Al—O1	1.9299 (19)	O5—C9	1.290 (3)
Al—O4	1.9414 (18)	O7—C17	1.287 (3)
Al—O8	1.951 (2)	O8—C19	1.253 (3)
O2—Al—O1	90.02 (8)	O5—Al—O4	89.90 (8)
O5—Al—O1	175.18 (9)	O7—Al—O8	90.49 (8)
O7—Al—O4	175.23 (9)	O2—Al—O8	172.20 (8)

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C6–H6A···O1	0.98	2.33	2.883 (4)	115
C7–H7C···O1	0.98	2.46	2.999 (3)	114
C13–H13C···O4	0.98	2.41	2.923 (3)	112
C14–H14A···O4	0.98	2.36	2.936 (4)	117
C21–H21C···O8	0.98	2.38	2.930 (4)	115
C22–H22A···O8	0.98	2.43	2.980 (4)	115

The structure was refined with the atomic coordinates of non-H atoms taken from the isostructural Fe complex (Urs *et al.*, 2000). All methyl H atoms were constrained to an ideal geometry (C–H = 0.98 Å), with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$, but were allowed to rotate freely about the C–C bonds. All remaining H atoms were placed in geometrically idealized positions (C–H = 0.95 Å) and constrained to ride on their parent atoms, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINTE* (Bruker, 2001); data reduction: *SAINTE*; method used to solve structure: atomic coordinates of non-H atoms taken from isostructural Fe complex (Urs *et al.*, 2000); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *PLATON* (Spek, 2003).

The diffraction data were collected using the CCD facility at the Institute under the IRFA program of the Department of Science & Technology.

References

- Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
- Bruker (2001). *SMART* and *SAINTE*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- 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.
- Shannon, R. D. (1976). *Acta Cryst.* **A32**, 751–767.
- Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
- Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
- Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
- Urs, U. K., Anitha, K. C., Raghunathan, K. L., Shivashankar, S. A., Robinson, W. T. & Guru Row, T. N. (2001). *Acta Cryst.* **E57**, m242–m243.
- Urs, U. K., Dharmaparakash, M. S., Shivashankar, S. A. & Guru Row, T. N. (2003a). *Acta Cryst.* **E59**, m1–m2.
- Urs, U. K., Dharmaparakash, M. S., Shivashankar, S. A. & Guru Row, T. N. (2003b). *Acta Cryst.* **E59**, m83–m84.
- Urs, U. K., Shalini, K., Cameron, T. S., Shivashankar, S. A. & Guru Row, T. N. (2001). *Acta Cryst.* **E57**, m457–m458.
- Urs, U. K., Shalini, K., Shivashankar, S. A. & Guru Row, T. N. (2000). *Acta Cryst.* **C56**, e448–e449.