

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

 Tris(methyl 3-oxobutanoato- κ^2O,O')-aluminium(III)

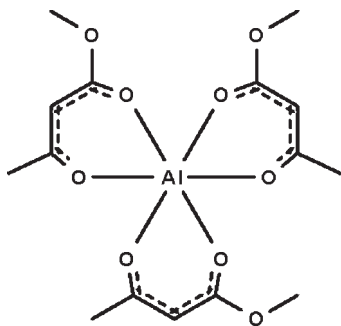
 Gururaj M. Neelgund,^a S. A. Shivashankar,^a
 T. Narasimhamurthy^a and Seik Weng Ng^{b*}
^aMaterials Research Centre, Indian Institute of Science, Bangalore 560 012, India, and ^bDepartment of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia
 Correspondence e-mail: seikweng@um.edu.my

Received 19 November 2009; accepted 20 November 2009

 Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.006$ Å; R factor = 0.068; wR factor = 0.189; data-to-parameter ratio = 13.8.

 In the title compound, $[\text{Al}(\text{C}_5\text{H}_7\text{O}_3)_3]$, three acac-type ligands (methyl 3-oxobutanoate anions) chelate to the aluminium(III) cation in a slightly distorted AlO_6 octahedral coordination geometry. Electron delocalization occurs within the chelating rings.

Related literature

 For the crystal structure of tris(acetylacetonato)aluminium, see: von Chrzanowski *et al.* (2007).


Experimental

Crystal data

 $[\text{Al}(\text{C}_5\text{H}_7\text{O}_3)_3]$
 $M_r = 372.30$

 Triclinic, $P\bar{1}$
 $a = 6.476$ (1) Å
 $b = 9.986$ (2) Å
 $c = 14.368$ (2) Å
 $\alpha = 90.478$ (2)°
 $\beta = 92.229$ (2)°
 $\gamma = 99.337$ (2)°

 $V = 916.1$ (2) Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 0.15$ mm⁻¹
 $T = 293$ K
 $0.5 \times 0.4 \times 0.2$ mm

Data collection

 Bruker SMART APEX
 diffractometer
 Absorption correction: multi-scan
 SADABS (Sheldrick, 1996)
 $T_{\min} = 0.927$, $T_{\max} = 0.970$

 8801 measured reflections
 3207 independent reflections
 2304 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.041$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.068$
 $wR(F^2) = 0.189$
 $S = 1.02$
 3207 reflections

 232 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 1.26$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.28$ e Å⁻³

Table 1

Selected bond lengths (Å).

Al1—O1	1.905 (3)	Al1—O6	1.849 (3)
Al1—O3	1.859 (3)	Al1—O7	1.904 (3)
Al1—O4	1.909 (3)	Al1—O9	1.869 (3)

Data collection: SMART (Bruker, 2004); cell refinement: SAINT (Bruker, 2004); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: X-SEED (Barbour, 2001); software used to prepare material for publication: publCIF (Westrip, 2009).

We acknowledge the use of the X-ray CCD facility at the Indian Institute of Science, Bangalore under the IRHPA/DST program, and we thank the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2684).

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supplementary materials

Acta Cryst. (2009). E65, m1681 [doi:10.1107/S1600536809049812]

Tris(methyl 3-oxobutanoato- κ^2O,O')aluminium(III)

G. M. Neelgund, S. A. Shivashankar, T. Narasimhamurthy and S. W. Ng

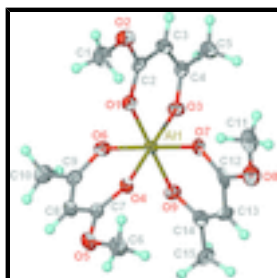
Experimental

Aluminium isopropoxide (10 mmol, 2.04 g) was dissolved in toluene (25 ml) under a nitrogen atmosphere. Methyl acetoacetate (30 mmol, 3.2 ml) was added. The mixture turned yellow. The solution was stirred for 6 h. The solvent was removed by fractional distillation under vacuum to yield the product, which was purified by repeated recrystallization from cyclohexane.

Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.93 to 0.96 Å) and were included in the refinement in the riding model approximation, with $U(H)$ set to 1.2 to 1.5 $U(C)$. The final difference Fourier map had a peak in the vicinity of the C13 and C14 atoms.

Figures



supplementary materials

$$V = 916.1 (2) \text{ \AA}^3$$

Data collection

Bruker SMART APEX diffractometer	3207 independent reflections
Radiation source: fine-focus sealed tube graphite	2304 reflections with $I > 2\sigma(I)$
ω scans	$R_{\text{int}} = 0.041$
Absorption correction: multi-scan SADABS (Sheldrick, 1996)	$\theta_{\text{max}} = 25.0^\circ$, $\theta_{\text{min}} = 1.4^\circ$
$T_{\text{min}} = 0.927$, $T_{\text{max}} = 0.970$	$h = -7 \rightarrow 7$
8801 measured reflections	$k = -11 \rightarrow 11$
	$l = -17 \rightarrow 17$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.068$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.189$	H-atom parameters constrained
$S = 1.02$	$w = 1/[\sigma^2(F_o^2) + (0.0907P)^2 + 1.477P]$
3207 reflections	where $P = (F_o^2 + 2F_c^2)/3$
232 parameters	$(\Delta/\sigma)_{\text{max}} = 0.001$
0 restraints	$\Delta\rho_{\text{max}} = 1.26 \text{ e \AA}^{-3}$
	$\Delta\rho_{\text{min}} = -0.28 \text{ e \AA}^{-3}$

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
All	0.61876 (18)	0.34609 (11)	0.25756 (8)	0.0287 (3)
O1	0.8373 (4)	0.4902 (2)	0.29494 (17)	0.0298 (6)
O2	1.0420 (4)	0.6906 (3)	0.2872 (2)	0.0407 (7)
O3	0.4846 (4)	0.4644 (3)	0.18716 (19)	0.0348 (7)
O4	0.7671 (4)	0.2253 (2)	0.32512 (17)	0.0279 (6)
O5	0.8034 (4)	0.0612 (3)	0.42532 (19)	0.0388 (7)
O6	0.4689 (4)	0.3726 (2)	0.36122 (18)	0.0317 (6)
O7	0.7870 (4)	0.3167 (3)	0.15592 (17)	0.0342 (7)
O8	0.9151 (5)	0.1916 (3)	0.0516 (2)	0.0472 (8)
O9	0.4156 (4)	0.1989 (3)	0.21949 (19)	0.0375 (7)
C1	1.1890 (6)	0.6352 (5)	0.3464 (3)	0.0419 (10)
H1A	1.3137	0.7011	0.3560	0.063*
H1B	1.2235	0.5551	0.3176	0.063*
H1C	1.1283	0.6125	0.4052	0.063*
C2	0.8676 (6)	0.6084 (4)	0.2614 (3)	0.0302 (9)
C3	0.7318 (7)	0.6615 (4)	0.1987 (3)	0.0364 (10)
H3	0.7674	0.7509	0.1799	0.044*

C4	0.5509 (6)	0.5875 (4)	0.1646 (3)	0.0324 (9)
C5	0.4062 (7)	0.6453 (4)	0.0957 (3)	0.0418 (11)
H5A	0.2692	0.6377	0.1203	0.063*
H5B	0.3978	0.5957	0.0378	0.063*
H5C	0.4602	0.7391	0.0854	0.063*
C6	0.9613 (7)	0.0242 (5)	0.3656 (3)	0.0423 (10)
H6A	1.0342	-0.0400	0.3968	0.063*
H6B	1.0590	0.1039	0.3518	0.063*
H6C	0.8953	-0.0157	0.3088	0.063*
C7	0.7055 (6)	0.1615 (4)	0.3967 (3)	0.0280 (8)
C9	0.4357 (6)	0.2936 (4)	0.4312 (3)	0.0317 (9)
C8	0.5413 (6)	0.1888 (4)	0.4513 (3)	0.0336 (9)
H8	0.5038	0.1345	0.5021	0.040*
C10	0.2692 (7)	0.3274 (4)	0.4948 (3)	0.0433 (11)
H10A	0.1454	0.3372	0.4583	0.065*
H10B	0.3205	0.4109	0.5279	0.065*
H10C	0.2364	0.2557	0.5385	0.065*
C11	1.0873 (7)	0.2966 (5)	0.0492 (3)	0.0464 (11)
H11A	1.1911	0.2695	0.0104	0.070*
H11B	1.1462	0.3157	0.1112	0.070*
H11C	1.0424	0.3766	0.0244	0.070*
C12	0.7677 (7)	0.2051 (5)	0.1104 (3)	0.0380 (10)
C13	0.5996 (7)	0.0964 (4)	0.1146 (3)	0.0403 (10)
H13	0.6048	0.0183	0.0796	0.048*
C14	0.4322 (7)	0.1000 (4)	0.1665 (3)	0.0379 (10)
C15	0.2460 (7)	-0.0173 (4)	0.1643 (3)	0.0434 (11)
H15A	0.1178	0.0182	0.1545	0.065*
H15B	0.2434	-0.0639	0.2225	0.065*
H15C	0.2605	-0.0795	0.1145	0.065*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Al1	0.0348 (7)	0.0243 (6)	0.0268 (6)	0.0046 (5)	0.0003 (5)	0.0002 (4)
O1	0.0298 (14)	0.0295 (14)	0.0295 (14)	0.0021 (11)	0.0035 (11)	-0.0032 (11)
O2	0.0372 (17)	0.0351 (15)	0.0484 (18)	0.0012 (13)	0.0016 (14)	0.0019 (13)
O3	0.0390 (16)	0.0275 (14)	0.0395 (16)	0.0099 (12)	0.0002 (13)	0.0030 (12)
O4	0.0289 (14)	0.0320 (14)	0.0233 (13)	0.0055 (11)	0.0028 (11)	0.0030 (11)
O5	0.0450 (17)	0.0441 (17)	0.0313 (15)	0.0179 (14)	0.0079 (13)	0.0078 (12)
O6	0.0277 (14)	0.0289 (14)	0.0385 (16)	0.0035 (11)	0.0059 (12)	0.0016 (12)
O7	0.0491 (17)	0.0345 (15)	0.0217 (14)	0.0136 (13)	0.0050 (12)	-0.0011 (11)
O8	0.0407 (18)	0.058 (2)	0.0421 (18)	0.0050 (15)	0.0080 (14)	-0.0004 (15)
O9	0.0457 (17)	0.0283 (14)	0.0366 (16)	0.0027 (12)	-0.0069 (13)	-0.0013 (12)
C1	0.030 (2)	0.051 (3)	0.044 (3)	0.0045 (19)	0.0002 (19)	-0.001 (2)
C2	0.030 (2)	0.027 (2)	0.032 (2)	-0.0037 (16)	0.0129 (17)	-0.0102 (16)
C3	0.047 (3)	0.027 (2)	0.037 (2)	0.0094 (18)	0.0084 (19)	0.0068 (17)
C4	0.040 (2)	0.033 (2)	0.028 (2)	0.0124 (18)	0.0097 (17)	-0.0013 (16)
C5	0.055 (3)	0.037 (2)	0.038 (2)	0.022 (2)	0.004 (2)	0.0085 (18)

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C6	0.044 (3)	0.047 (3)	0.041 (2)	0.023 (2)	0.006 (2)	0.007 (2)
C7	0.030 (2)	0.0283 (19)	0.0240 (19)	0.0028 (16)	-0.0050 (16)	0.0005 (15)
C9	0.029 (2)	0.029 (2)	0.034 (2)	-0.0040 (16)	0.0020 (17)	-0.0061 (17)
C8	0.038 (2)	0.035 (2)	0.026 (2)	-0.0002 (18)	0.0076 (17)	0.0030 (16)
C10	0.039 (2)	0.044 (2)	0.047 (3)	0.002 (2)	0.021 (2)	-0.006 (2)
C11	0.030 (2)	0.074 (3)	0.036 (2)	0.008 (2)	0.0128 (19)	0.003 (2)
C12	0.044 (2)	0.058 (3)	0.0174 (19)	0.026 (2)	0.0062 (17)	0.0084 (18)
C13	0.058 (3)	0.022 (2)	0.039 (2)	0.0031 (19)	-0.007 (2)	-0.0040 (17)
C14	0.049 (3)	0.036 (2)	0.030 (2)	0.0126 (19)	-0.0016 (19)	0.0033 (18)
C15	0.051 (3)	0.033 (2)	0.044 (3)	-0.001 (2)	0.009 (2)	-0.0113 (19)

Geometric parameters (Å, °)

A11—O1	1.905 (3)	C4—C5	1.518 (6)
A11—O3	1.859 (3)	C5—H5A	0.9600
A11—O4	1.909 (3)	C5—H5B	0.9600
A11—O6	1.849 (3)	C5—H5C	0.9600
A11—O7	1.904 (3)	C6—H6A	0.9600
A11—O9	1.869 (3)	C6—H6B	0.9600
O1—C2	1.267 (5)	C6—H6C	0.9600
O2—C2	1.322 (4)	C7—C8	1.406 (5)
O2—C1	1.432 (5)	C9—C8	1.367 (6)
O3—C4	1.284 (5)	C9—C10	1.517 (5)
O4—C7	1.258 (4)	C8—H8	0.9300
O5—C7	1.329 (4)	C10—H10A	0.9600
O5—C6	1.450 (5)	C10—H10B	0.9600
O6—C9	1.286 (5)	C10—H10C	0.9600
O7—C12	1.273 (5)	C11—H11A	0.9600
O8—C12	1.322 (5)	C11—H11B	0.9600
O8—C11	1.403 (5)	C11—H11C	0.9600
O9—C14	1.263 (5)	C12—C13	1.411 (6)
C1—H1A	0.9600	C13—C14	1.345 (6)
C1—H1B	0.9600	C13—H13	0.9300
C1—H1C	0.9600	C14—C15	1.538 (6)
C2—C3	1.401 (6)	C15—H15A	0.9600
C3—C4	1.352 (6)	C15—H15B	0.9600
C3—H3	0.9300	C15—H15C	0.9600
O6—A11—O3	92.13 (12)	H5B—C5—H5C	109.5
O6—A11—O9	90.45 (13)	O5—C6—H6A	109.5
O3—A11—O9	91.19 (13)	O5—C6—H6B	109.5
O6—A11—O1	91.76 (12)	H6A—C6—H6B	109.5
O3—A11—O1	90.98 (12)	O5—C6—H6C	109.5
O9—A11—O1	176.85 (13)	H6A—C6—H6C	109.5
O6—A11—O7	176.44 (13)	H6B—C6—H6C	109.5
O3—A11—O7	90.98 (12)	O4—C7—O5	118.6 (3)
O9—A11—O7	91.22 (13)	O4—C7—C8	125.3 (3)
O1—A11—O7	86.45 (12)	O5—C7—C8	116.1 (3)
O6—A11—O4	90.71 (12)	O6—C9—C8	124.9 (4)
O3—A11—O4	177.05 (13)	O6—C9—C10	114.6 (3)

O9—A11—O4	89.59 (12)	C8—C9—C10	120.5 (4)
O1—A11—O4	88.13 (12)	C9—C8—C7	121.4 (3)
O7—A11—O4	86.16 (12)	C9—C8—H8	119.3
C2—O1—A11	126.1 (3)	C7—C8—H8	119.3
C2—O2—C1	116.9 (3)	C9—C10—H10A	109.5
C4—O3—A11	129.5 (3)	C9—C10—H10B	109.5
C7—O4—A11	125.6 (2)	H10A—C10—H10B	109.5
C7—O5—C6	116.8 (3)	C9—C10—H10C	109.5
C9—O6—A11	127.3 (2)	H10A—C10—H10C	109.5
C12—O7—A11	123.6 (3)	H10B—C10—H10C	109.5
C12—O8—C11	117.6 (4)	O8—C11—H11A	109.5
C14—O9—A11	129.2 (3)	O8—C11—H11B	109.5
O2—C1—H1A	109.5	H11A—C11—H11B	109.5
O2—C1—H1B	109.5	O8—C11—H11C	109.5
H1A—C1—H1B	109.5	H11A—C11—H11C	109.5
O2—C1—H1C	109.5	H11B—C11—H11C	109.5
H1A—C1—H1C	109.5	O7—C12—O8	117.0 (4)
H1B—C1—H1C	109.5	O7—C12—C13	126.0 (4)
O1—C2—O2	118.0 (4)	O8—C12—C13	116.9 (4)
O1—C2—C3	125.9 (3)	C14—C13—C12	123.3 (4)
O2—C2—C3	116.1 (3)	C14—C13—H13	118.4
C4—C3—C2	122.4 (4)	C12—C13—H13	118.4
C4—C3—H3	118.8	O9—C14—C13	122.9 (4)
C2—C3—H3	118.8	O9—C14—C15	115.5 (4)
O3—C4—C3	124.0 (4)	C13—C14—C15	121.6 (4)
O3—C4—C5	114.0 (4)	C14—C15—H15A	109.5
C3—C4—C5	122.0 (4)	C14—C15—H15B	109.5
C4—C5—H5A	109.5	H15A—C15—H15B	109.5
C4—C5—H5B	109.5	C14—C15—H15C	109.5
H5A—C5—H5B	109.5	H15A—C15—H15C	109.5
C4—C5—H5C	109.5	H15B—C15—H15C	109.5
H5A—C5—H5C	109.5		
O6—A11—O1—C2	-102.8 (3)	C1—O2—C2—C3	-176.1 (3)
O3—A11—O1—C2	-10.6 (3)	O1—C2—C3—C4	-2.0 (6)
O7—A11—O1—C2	80.3 (3)	O2—C2—C3—C4	178.1 (4)
O4—A11—O1—C2	166.6 (3)	A11—O3—C4—C3	-8.0 (6)
O6—A11—O3—C4	102.4 (3)	A11—O3—C4—C5	172.5 (3)
O9—A11—O3—C4	-167.1 (3)	C2—C3—C4—O3	1.4 (6)
O1—A11—O3—C4	10.6 (3)	C2—C3—C4—C5	-179.1 (4)
O7—A11—O3—C4	-75.8 (3)	A11—O4—C7—O5	166.7 (2)
O6—A11—O4—C7	22.3 (3)	A11—O4—C7—C8	-14.6 (5)
O9—A11—O4—C7	-68.1 (3)	C6—O5—C7—O4	-6.2 (5)
O1—A11—O4—C7	114.0 (3)	C6—O5—C7—C8	175.0 (3)
O7—A11—O4—C7	-159.4 (3)	A11—O6—C9—C8	14.4 (5)
O3—A11—O6—C9	158.5 (3)	A11—O6—C9—C10	-167.3 (3)
O9—A11—O6—C9	67.3 (3)	O6—C9—C8—C7	2.3 (6)
O1—A11—O6—C9	-110.5 (3)	C10—C9—C8—C7	-175.8 (4)
O4—A11—O6—C9	-22.3 (3)	O4—C7—C8—C9	-1.8 (6)
O3—A11—O7—C12	-110.4 (3)	O5—C7—C8—C9	176.9 (3)

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O9—A11—O7—C12	-19.2 (3)	A11—O7—C12—O8	-169.6 (2)
O1—A11—O7—C12	158.7 (3)	A11—O7—C12—C13	12.8 (5)
O4—A11—O7—C12	70.3 (3)	C11—O8—C12—O7	4.5 (5)
O6—A11—O9—C14	-157.2 (3)	C11—O8—C12—C13	-177.6 (4)
O3—A11—O9—C14	110.6 (3)	O7—C12—C13—C14	2.2 (7)
O7—A11—O9—C14	19.6 (3)	O8—C12—C13—C14	-175.4 (4)
O4—A11—O9—C14	-66.5 (3)	A11—O9—C14—C13	-11.6 (6)
A11—O1—C2—O2	-171.6 (2)	A11—O9—C14—C15	168.8 (3)
A11—O1—C2—C3	8.6 (5)	C12—C13—C14—O9	-3.4 (7)
C1—O2—C2—O1	4.1 (5)	C12—C13—C14—C15	176.2 (4)

Fig. 1

