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γ-Tris(2,4-pentanedionato-κ^2O,O')aluminium(III) at 110 K

Lars S. von Chrzanowski,* Martin Lutz and Anthony L. Spek

Bijvoet Center for Biomolecular Research, Crystal and Structural Chemistry, Utrecht University, Padualaan 8, 3584 CH Utrecht, The Netherlands

Correspondence e-mail: l.vonchrzanowski@chem.uu.nl

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The title compound, $[Al(C_5H_7O_2)_3]$, was reinvestigated at 110 K to provide information on thermal vibrations in the structure, comparing it to the previously published structure by McClelland [*Acta Cryst.* (1975), B**31**, 2496–2498] based on room-temperature data. The γ-polymorph contains four independent molecules in the asymmetric unit with their centers of gravity at pseudo-special positions.

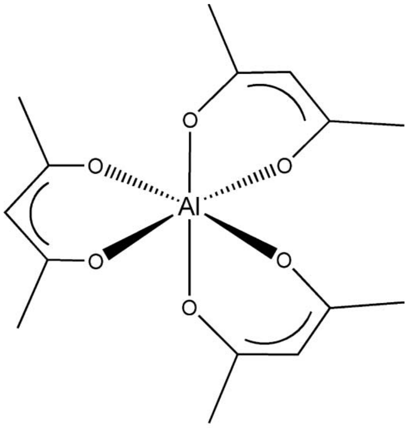
Key indicators

Single-crystal X-ray study
T = 110 K
Mean σ(C–C) = 0.003 Å
R factor = 0.031
wR factor = 0.076
Data-to-parameter ratio = 17.8

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

Comment

The crystal structure of γ-tris(2,4-pentanedionato)aluminium(III), (I), was reported previously by McClelland (1975). In the course of our studies on $Al(acac)_3$ (acac = acetylacetonate or 2,4-pentanedionate) and in order to improve the refinement, we redetermined the structure at low temperature and obtained significantly improved agreement factors; all H atoms were located in a difference Fourier map. The low-temperature structure shows significant atom vibrations which were analysed. The crystal structure consists of four independent molecules (Fig. 1). Molecules 2–4 have an approximate non-crystallographic D_3 symmetry, with r.m.s. deviations of 0.147–0.158 Å from ideal symmetry (Pilati & Forni, 1998). Molecule 1 is described by approximately C_2 symmetry, with an r.m.s. deviation of 0.138 Å from ideal symmetry.



(I)

In the unit cell, molecules are arranged with their centres of gravity close to pseudo-special positions ($\frac{2}{3}$, $\frac{3}{4}$, 0.76), (0.63, $\frac{1}{2}$, 0.77), (0.87, $\frac{3}{4}$, 0.26) and (0.85, 0.51, 0.28). Thus, pseudo-translation symmetry in the directions of the a and b axes occurs (Fig. 2).

A displacement ellipsoid plot (Fig. 1) reveals the anisotropic character of some methyl groups in spite of low-temperature measurements. Indeed, rigid-body analysis

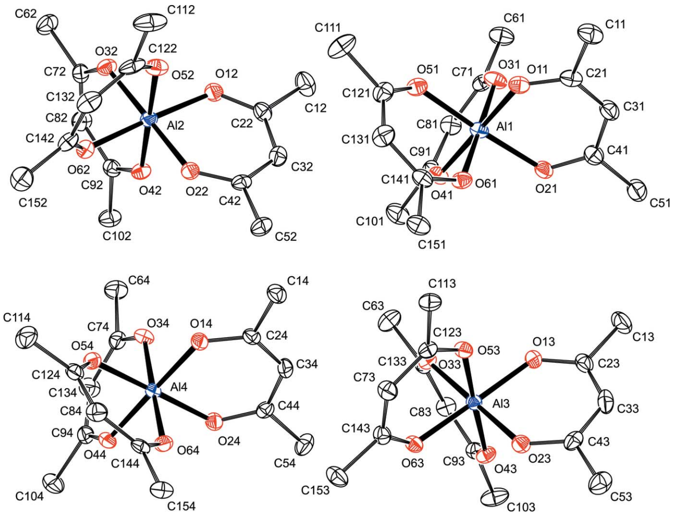


Figure 1
The structures of the four independent molecules of (I). Displacement ellipsoids are drawn at the 50% probability level. H atoms have been omitted for clarity.

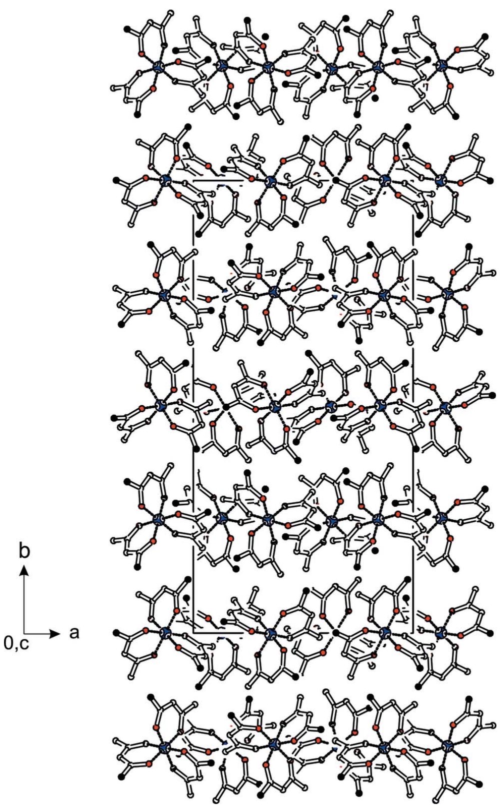


Figure 2
The crystal packing viewed along the *c* axis. H atoms have been omitted for clarity.

(Schomaker & Trueblood, 1998) for the four molecules show high factors of $R = 0.149$–0.159 $[R = \Sigma\{[U(\mathrm{obs}) - U(\mathrm{calc})]^2\}/\Sigma[U(\mathrm{obs})^2]^{1/2}]$. A difference plot (Hummel *et al.*, 1990) between the observed displacement parameters and the rigid body model for molecule 1 (Fig. 3) also indicates large internal motions.

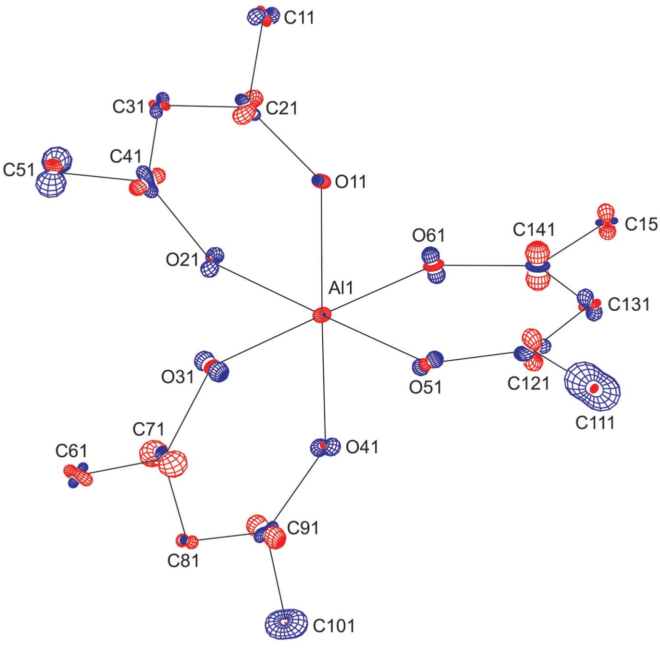


Figure 3
PEANUT plot (Hummel *et al.*, 1990) showing the difference between the measured displacement parameters and the parameters obtained by a rigid body refinement using the program *THMA11* (Schomaker & Trueblood, 1998). A scale factor of 3.08 was used for the root-mean-square surfaces. Blue lines indicate positive differences and red lines negative ones. The largest difference is expressed by the methyl groups.

A normal probability plot (Abrahams & Keve, 1971) of equivalent bond distances (Fig. 4) shows a slope of 2.979; four outliers belong to the Al—O distances. The range of Al—O distances is wide [1.8728 (13)–1.8947 (12) Å]. The plot indicates larger variance than the standard uncertainties of the individual bond lengths. There is no chemical evidence for this large variance. Therefore, this effect can be attributed to the internal motion of the molecules.

Experimental

Crystals were obtained by slow evaporation of a solution of the commercially available material (Aldrich) in ethyl acetate at room temperature. In a batch, plate- and needle-shaped cystals were present. A needle-shaped crystal was selected and cut to an appropriate size for data collection.

Crystal data

$[\mathrm{Al}(\mathrm{C_5H_7O_2})_3]$
$M_r = 324.30$
Orthorhombic, $Pna2_1$
$a = 15.51663\ (18)$ Å
$b = 31.9596\ (7)$ Å
$c = 13.2306\ (2)$ Å
$V = 6561.14\ (19)$ Å^3
$Z = 16$
$D_x = 1.313$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.15$ mm^{-1}
$T = 110\ (2)$ K
Block, colourless
0.45 × 0.36 × 0.24 mm

Data collection

Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)
$T_{\min} = 0.63$, $T_{\max} = 0.97$
83015 measured reflections
14519 independent reflections
12829 reflections with $I > 2\sigma(I)$
$R_{\mathrm{int}} = 0.036$
$\theta_{\max} = 27.5°$

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Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.076$
$S = 1.04$
14519 reflections
817 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0396P)^2 + 1.1373P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}
Absolute structure: Flack (1983), 6667 Friedel pairs
Flack parameter: 0.08 (8)

Table 1
Selected bond lengths (Å).

Al1—O21	1.8790 (12)	Al3—O63	1.8773 (12)
Al1—O51	1.8806 (13)	Al3—O13	1.8794 (12)
Al1—O41	1.8811 (12)	Al3—O33	1.8812 (12)
Al1—O31	1.8819 (13)	Al3—O43	1.8816 (13)
Al1—O61	1.8853 (13)	Al3—O53	1.8874 (13)
Al1—O11	1.8947 (12)	Al3—O23	1.8887 (12)
Al2—O32	1.8728 (13)	Al4—O54	1.8736 (12)
Al2—O62	1.8805 (12)	Al4—O44	1.8744 (12)
Al2—O52	1.8842 (13)	Al4—O24	1.8863 (12)
Al2—O22	1.8888 (12)	Al4—O34	1.8877 (13)
Al2—O42	1.8896 (13)	Al4—O14	1.8878 (12)
Al2—O12	1.8901 (12)	Al4—O64	1.8909 (13)

All H atoms were introduced in geometrically idealized positions, refined with a riding model and subsequently confirmed in a difference Fourier map. Their isotropic displacement parameters were set equal to $U_{iso}(H) = 1.2U_{eq}(C)$ for H atoms of the central C—H units and to $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl H atoms (C—H = 0.95–0.98 Å). The standard uncertainty of the Flack parameter is smaller than 0.1 and thus implies a sufficient inversion-distinguishing power (Flack & Bernardinelli, 2000). Therefore Friedel pairs were not merged prior to the refinement.

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *PEAKREF* (Schreurs, 2005); data reduction: *EVALCCD* (Duisenberg *et al.*, 2003); program(s) used to solve structure: coordinates were taken from the literature (McClelland, 1975); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

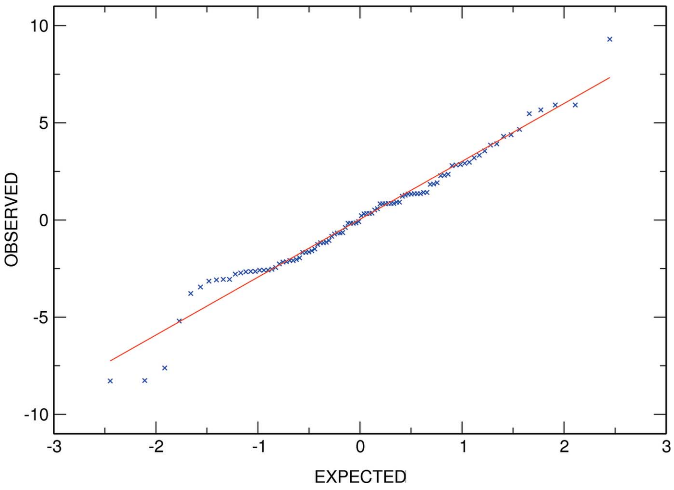

Figure 4
Normal probability plot of all equivalent bond distances. Shown is $[d - d(\text{mean})]/\sigma$ for the ordinate *versus* the expected values for the abscissa. Parameters of the linear regression: $y = 2.979x + 0.038$.

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