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The centrosymmetric dimer of dichloro(trimethylsiloxy)aluminium

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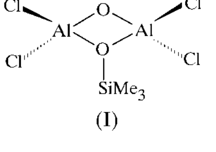
The title molecule is dimeric, *i.e.* di-μ-trimethylsiloxy-bis(dichloroaluminium), $[Al_2Cl_4(C_3H_9Si)_2]$, and possesses exact crystallographic inversion symmetry. The O atoms of the trimethylsiloxy groups bridge the two Al atoms to form a four-membered ring. The Si—O bond distance [1.711 (3) Å], the Al—O mean bond distance [1.806 (4) Å] and the mean Si—C bond distance [1.875 (6) Å] appear to agree well with standard data. Mean values for C—Si—C, O—Si—C, and Si—O—Al angles are 112.9 (3), 105.8 (2), and 131.8 (2)° repectively. The two ring angles O—Al—O and Al—O—Al are 84.43 (16) and 95.57 (16)°, respectively.

Comment

We are interested in the dimer, (I), of dichlorotrimethylsiloxyaluminium, because we intend to explore its reactions with a variety of nucleophiles, such as amides and azides, to prepare new precursors to Sialons (silicon–aluminium–oxygen–nitrogen ceramics) or related alloys. Previous work (Schmidbaur *et al.*, 1964) indicated that the molecule is dimeric and it was proposed that the O atoms of the trimethylsiloxy group, rather than the Cl ligands, bridge the Al atoms to form the dimer. Our X-ray analysis confirmed the suggested structure. At a later time, we discovered that the structure of $(Me_3SiOAlBr_2)_2$, the bromine analogue, has been determined (Bonamico & Dessy, 1967) and is essentially identical to $(Me_3SiOAlCl_2)_2$.



(I)

Experimental

A solution of $(SiMe_3)_2O$ (1.74 g, 10.7 mmol) in CH_2Cl_2 was added to a suspension of $AlCl_3$ (1.43 g, 10.7 mmol) in CH_2Cl_2 (40 ml). The resulting solution was stirred for 12 h and then filtered. Upon distillation of the solvent, the compound was isolated as a colourless precipitate (1.062 g, 53.1% yield). The sample used for the diffraction experiments was prepared by slow sublimation under vacuum. The calculated powder diffraction pattern of the crystal was identical with that of the bulk material.

Crystal data

$[Al_2Cl_4(C_3H_9Si)_2]$
$M_r = 374.14$
Monoclinic, $P2_1/n$
$a = 6.7860$ (14) Å
$b = 9.1740$ (18) Å
$c = 14.982$ (3) Å
$\beta = 101.42$ (3)°
$V = 914.2$ (3) Å^3
$Z = 2$
$D_x = 1.359$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 24 reflections
$\theta = 13$–15°
$\mu = 0.861$ mm^{-1}
$T = 176$ (2) K
Cleaved fragment, colourless
0.25 × 0.20 × 0.15 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
θ–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.806$, $T_{max} = 0.879$
1602 measured reflections
1602 independent reflections
1128 reflections with $I > 2\sigma(I)$
$\theta_{max} = 24.97°$
$h = 0 \rightarrow 8$
$k = 0 \rightarrow 10$
$l = -17 \rightarrow 17$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.155$
$S = 1.097$
1602 reflections
73 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0658P)^2 + 1.6734P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.63$ e Å^{-3}
$\Delta\rho_{min} = -0.35$ e Å^{-3}

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 *Software*; data reduction: *CAD*-4 *Software*; program(s) used to solve structure: *XS* in *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *XL* in *SHELXTL*; software used to prepare material for publication: *XL* in *SHELXTL*.

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