

Trichloro(diethyl ether- κ O)aluminium(III) from a twinned crystal

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Key indicators

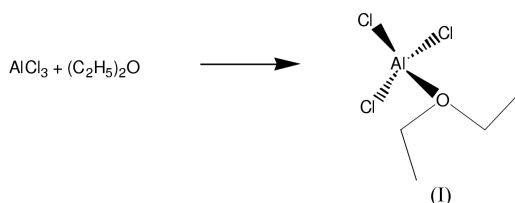
Single-crystal X-ray study
 $T = 100$ K
 Mean $\sigma(\text{C}-\text{C}) = 0.005$ Å
 R factor = 0.041
 wR factor = 0.110
 Data-to-parameter ratio = 26.8

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

The title compound, $[\text{AlCl}_3(\text{C}_4\text{H}_{10}\text{O})]$, forms crystals which appear orthorhombic, but which are actually monoclinic. They are twinned on (100). This is a rather common occurrence when the monoclinic angle β is close to 90° .

Comment

Recently, we have reported the X-ray crystal structure analyses of the tetrahydrofuran adducts of AlBr_3 (Scholz *et al.*, 2003) and GaCl_3 (Scholz *et al.*, 2002). Halides of group 13 elements have found widespread use as starting materials in inorganic chemistry. The synthesis of group 13 element clusters depends, on the one hand, on the oxidation state of the elements in the halides and, on the other hand, on the solvent used. AlCl_3 features a polymeric arrangement in the solid state. In contrast with donor solvents, AlCl_3 is poorly soluble in none of the donor solvents such as alkenes or benzene. We are interested in the synthesis of Al cluster compounds in a weakly polar medium. Therefore, we have prepared the title compound, $\text{AlCl}_3 \cdot \text{Et}_2\text{O}$, (I), which is soluble in pentane. Colourless crystals of (I) were grown from solution in diethyl ether at ambient temperature.



The Al centre in (I) is tetrahedrally coordinated by three Cl ligands and one ether O atom. Bond lengths are in the usual ranges (Cambridge Structural Database, Version 5.24, April 2003; Allen, 2002). The three Cl–Al–Cl bond angles are significantly wider than the remaining three Cl–Al–O angles around the Al centre. The torsion angles of the ether moiety show synclinal values.

Experimental

Colourless crystals of the (I) were obtained from a solution of AlCl_3 (1.14 g, 0.64 mmol) in diethyl ether (30 ml) at ambient temperature.

Crystal data

$[\text{AlCl}_3(\text{C}_4\text{H}_{10}\text{O})]$
 $M_r = 207.45$
 Monoclinic, $P2_1/n$
 $a = 6.8961$ (8) Å
 $b = 11.2426$ (9) Å
 $c = 12.5118$ (14) Å
 $\beta = 90.611$ (9)°
 $V = 969.99$ (17) Å³
 $Z = 4$

$D_x = 1.421$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 23 489 reflections
 $\theta = 3.6$ – 27.6°
 $\mu = 0.97$ mm⁻¹
 $T = 100$ (2) K
 Block, colourless
 $0.36 \times 0.34 \times 0.28$ mm

Data collection

Stoe IPDS II two-circle diffractometer
 ω scans
 Absorption correction: multi-scan (MULABS; Spek, 1990; Blessing, 1995)
 $T_{\min} = 0.716$, $T_{\max} = 0.767$
 17 320 measured reflections

2224 independent reflections
 2015 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.088$
 $\theta_{\text{max}} = 27.8^\circ$
 $h = -8 \rightarrow 8$
 $k = -14 \rightarrow 14$
 $l = -16 \rightarrow 16$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.041$
 $wR(F^2) = 0.110$
 $S = 1.06$
 2224 reflections
 83 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0667P)^2 + 0.357P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.39 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.58 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (\AA , $^\circ$).

Al1—O1	1.830 (2)	Al1—Cl2	2.1422 (13)
Al1—Cl3	2.0872 (12)	O1—C1	1.484 (3)
Al1—Cl1	2.1212 (10)	O1—C3	1.506 (4)
O1—Al1—Cl3	105.30 (8)	Cl1—Al1—Cl2	112.79 (5)
O1—Al1—Cl1	106.06 (8)	Cl1—O1—C3	114.8 (2)
Cl3—Al1—Cl1	110.41 (5)	Cl1—O1—Al1	124.52 (18)
O1—Al1—Cl2	107.87 (7)	C3—O1—Al1	120.25 (16)
Cl3—Al1—Cl2	113.79 (6)		
C3—O1—C1—C2	90.1 (3)	C1—O1—C3—C4	79.7 (3)

The crystals of (I) looked suspicious under polarized light, because they did not turn dark (extinguish) when the microscope stage was turned, but showed the whole spectrum of rainbow colours. Some reflection profiles were excellent, but others were definitely split. The R_{int} and R_{sigma} values for the orthorhombic crystal system were 0.128 and 0.040, respectively. However, the systematic extinction conditions could only be observed for an n -glide plane and for a 2_1 screw axis perpendicular to that glide plane. R_{int} and R_{sigma} for the monoclinic crystal system were 0.088 and 0.046, respectively. Structure solution in space group $P2_1/n$ was possible, but refinement did not proceed satisfactorily. Taking into account our experiences with other twinned

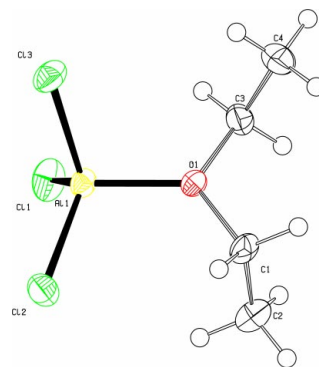


Figure 1

A perspective view of (I) with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

structures, and applying the twin law $(-100/010/001)$, provided ultimate success. The twin ratio refined to 0.424 (2):0.578 (2). H atoms were refined with fixed individual displacement parameters [$U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$ or $U_{\text{iso}}(\text{H}) = 1.5 U_{\text{eq}}(\text{C}_{\text{methyl}})$], using a riding model with $\text{C—H} = 0.99 \text{ \AA}$ or methyl $\text{C—H} = 0.98 \text{ \AA}$.

Data collection: X-AREA (Stoe & Cie, 2001); cell refinement: X-AREA; data reduction: X-AREA; program(s) used to solve structure: SHELXS97 (Sheldrick, 1990); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: PLATON (Spek, 1990); software used to prepare material for publication: SHELXL97.

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