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Trichloro(tetrahydrofuran)gallium(III)

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.005 Å
R factor = 0.035
wR factor = 0.093
Data-to-parameter ratio = 28.9

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

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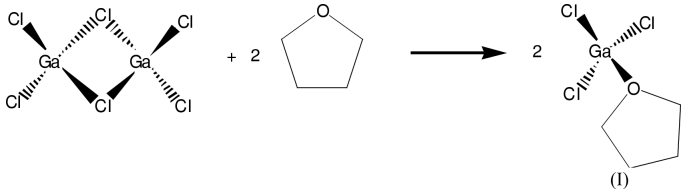
The title compound, $GaCl_3{\cdot}C_4H_8O$, is isostructural with $AlCl_3{\cdot}C_4H_8O$. The molecule is located on a mirror plane, passing through the Ga atom, the O atom and one of the Cl atoms. All other atoms occupy general positions.

Comment

We report here the X-ray crystal structure analysis of $GaCl_3$·thf (thf = C_4H_8O). The halides of Ga(I), Ga(II), and Ga(III) have been used in the synthesis of new gallium clusters (Donchev *et al.*, 2001). The synthesis of these clusters depends on the oxidation state of Ga in the halides on the one hand and the solvent on the other. $GaCl_3$ features a cyclic, dimeric arrangement in non-donor solvents such as alkanes and benzene. By contrast, monomeric adducts of $GaCl_3$ are formed in the presence of Lewis bases. Given this background, we became interested in the reaction of $GaCl_3$·thf with the bulky siloxides ^{t}Bu$_3$SiONa and ^{t}Bu$_2$PhSiONa (Lerner *et al.*, 2002). Therefore, we prepared a solution of $GaCl_3$·thf in pentane. Colourless crystals of the title compound, (I), were grown from this solution at 267 K. $GaCl_3{\cdot}C_4H_8O$ is isostructural with $AlCl_3{\cdot}C_4H_8O$ (Engelhardt *et al.*, 1996). The molecule is located on a mirror plane; the Ga atom, the O atom and one of the Cl atoms lie in this plane. All other atoms occupy general positions.



(I)

Experimental

Colourless crystals of the title compound were obtained from a solution of 0.07 g (0.4 mmol) $GaCl_3$ and 0.5 ml tetrahydrofuran in 20 ml pentane at 267 K. The NMR spectra were recorded on a Bruker AM 250 spectrometer. $GaCl_3$·thf: ^{1}H-NMR (C_6D_6, internal TMS): δ 0.82 (*m*; CH_2), 3.42 (*m*; OCH_2). ^{13}C{^{1}H}NMR (C_6D_6, internal TMS): δ 24.6 (*s*; CH_2), 71.8 (*s*; OCH_2).

Crystal data

$[Ga(C_4H_8O)Cl_3]$
M_r = 248.17
Monoclinic, $P2_1/m$
a = 6.2048 (9) Å
b = 10.3980 (11) Å
c = 7.3089 (10) Å
β = 106.085 (11)°
V = 453.09 (10) Å^3
Z = 2
D_x = 1.819 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 7266 reflections
θ = 3.8–29.8°
μ = 3.85 mm^{-1}
T = 173 (2) K
Plate, colourless
0.30 × 0.20 × 0.10 mm

Data collection

Stoe IPDS II two-circle diffractometer
ω scans
Absorption correction: multi-scan (*MULABS*; Spek, 1990; Blessing, 1995)
T_{min} = 0.392, T_{max} = 0.700
6794 measured reflections
1356 independent reflections
1200 reflections with $I > 2\sigma(I)$
R_{int} = 0.070
θ_{max} = 29.8°
$h = -8 \rightarrow 7$
$k = -14 \rightarrow 14$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.093
S = 1.04
1356 reflections
47 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0482P)^2 + 0.2393P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.66 e Å^{-3}
$\Delta\rho_{min}$ = −1.01 e Å^{-3}
Extinction correction: *SHELXL*
Extinction coefficient: 0.051 (6)

Table 1
Selected geometric parameters (Å, °).

Ga1—O1	1.919 (3)	Ga1—Cl1	2.1405 (11)
Ga1—Cl2	2.1388 (8)		
O1—Ga1—Cl2	104.36 (5)	O1—Ga1—Cl1	106.13 (10)
Cl2—Ga1—Cl2^{i}	114.63 (6)	Cl2—Ga1—Cl1	113.08 (3)

Symmetry codes: (i) $x, \frac{1}{2} - y, z$.

All H atoms were located in a difference Fourier synthesis. They were refined with fixed individual displacement parameters [U(H) = 1.2 U_{eq}(C)], using a riding model with C—H = 0.99 Å.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL*97.

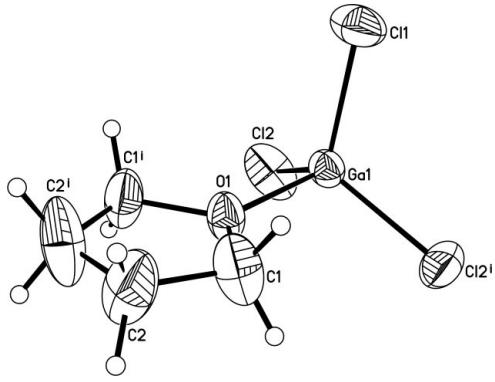


Figure 1
Perspective view of the molecule, with the atom numbering; displacement ellipsoids are drawn at the 50% probability level. Symmetry operator for generating equivalent atoms: (i) $x, -y + 1/2, z$.

References

Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.
Donchev, A., Schnepf, A., Stößer, G., Baum, E., Schnöckel, T., Blank, N. & Wiberg, N. (2001). *Eur. J. Inorg. Chem.* pp. 3348–3353.
Engelhardt, L. M., Junk, P. C., Raston, C. L., Skelton, B. W. & White, A. H. (1996). *J. Chem. Soc. Dalton Trans.* pp. 3297–3301.
Lerner, H.-W., Scholz, S. & Bolte, M. (2002). *Organometallics*, **21**, 3827–3830.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1991). *SHELXTL-Plus.* Release 4.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.
Spek, A. L. (1990). *Acta Cryst.* A**46**, C-34.
Stoe & Cie (2001). *X-AREA*. Stoe & Cie, Darmstadt, Germany.