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Dichloro(tetrahydrofuran-*O*)-[(trimethylsilyl)amido-*N*]gallium(III)

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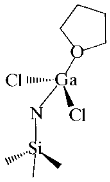
The title compound, [$GaCl_2(C_3H_{10}NSi)(C_4H_8O)$], is the tetrahydrofuran-coordinated monomer of the previously structurally characterized dimer [$GaN(H)SiMe_3Cl_2$]$_2$. The title compound consists of discrete monomeric molecules with two crystallographically independent molecules within the unit cell. The crystal structure is composed of a Ga atom in a four-coordinate tetrahedral arrangement, with Ga—N distances of 2.025 (3) and 2.026 (3) Å in the two independent molecules.

Comment

In the course of examining [$GaN(H)SiMe_3Cl_2$]$_2$ (Wiberg & Schmid, 1966) as a potential synthon for the preparation of a novel zinc amide, it was mixed with a solution of Me_2Zn in toluene. However, the only compound to be isolated from the reaction, after crystallization from tetrahydrofuran (THF) and work-up, was the title compound, (I), which is the tetrahydrofuran-coordinated monomer of the starting material, the previously structurally characterized dimer [$GaN(H)SiMe_3Cl_2$]$_2$, (II) (Nutt *et al.*, 1985).



(I)

Compound (I) has two crystallographically independent molecules within the unit cell. The crystal structure consists of discrete monomers, with the molecular structure consisting of a Ga atom bonded to two Cl atoms, an O atom from the coordinated tetrahydrofuran molecule, which shows some disorder, and the N atom of the amino group. The N atom is further bonded to an Si atom from the trimethylsilyl ligand and to an H atom.

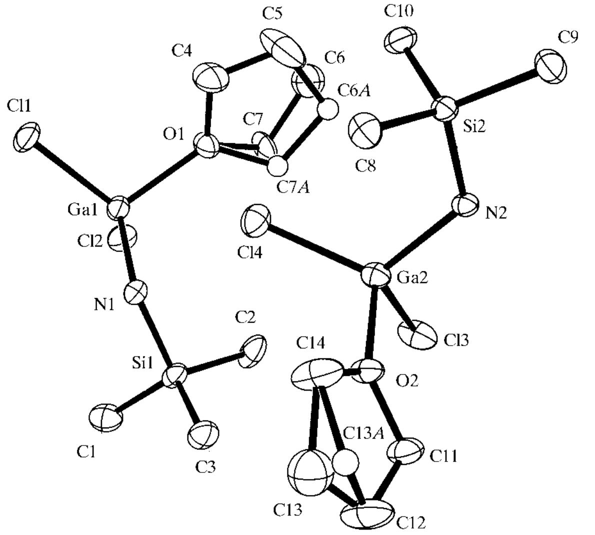


Figure 1
An *ORTEP* (Johnson, 1968) representation of (I) showing the disorder in the tetrahydrofuran ligands. Displacement ellipsoids are at the 30% probability level and all H atoms have been omitted for clarity.

The bond distances in (I) are comparable with those in the starting compound, (II), which is a dimer consisting of a four-membered $(GaN)_2$ ring, with the trimethylsilyl ligands on adjacent N atoms *trans* to one another. The Ga—Cl and N—Si interatomic distances in (I) are Ga1—Cl1 2.2178 (10), Ga1—Cl2 2.2182 (9), Ga2—Cl3 2.2226 (9) and Ga2—Cl4 2.2218 (10) Å, and N1—Si1 1.791 (3) and N2—Si2 1.800 (2) Å (for the crystallographically independent molecules 1 and 2). The corresponding values in (II) are Ga—Cl 2.150 (2) and 2.136 (2) Å, and N—Si 1.805 (4) Å. The Ga—N interatomic distances in (I) are slightly longer than those found in (II): Ga1—N1 2.026 (3) and Ga2—N2 2.025 (2) Å in (I), and Ga—N 1.974 (4) and 1.964 (4) Å in (II), with this bond lengthening most likely occurring due to the electron-releasing effect of the coordinated tetrahydrofuran molecule.

The Ga—O distances [Ga1—O1 2.048 (2) and Ga2—O2 2.041 (2) Å] are within the range found in the other two structually characterized gallium complexes possessing a coordinated tetrahydrofuran molecule and a Ga—Cl interaction, *i.e.* [$GaSi(SiMe_3)_3Cl_2(C_4H_8O)$] [2.010 (3) Å; Linti *et al.*, 1996] and $PhClGaSi(SiMe_3)\cdot THF$ [2.061 (3) Å; Pickett *et al.*, 2000]. The interatomic angles about the four-coordinate Ga atom deviate from those of an ideal tetrahedron, with the most acute being O1—Ga1—Cl1 102.87 (7) and O2—Ga2—Cl3 101.55 (7)° (in molecules 1 and 2, respectively), and the most obtuse being Cl2—Ga1—Cl1 119.27 (4) and Cl4—Ga2—Cl3 119.23 (4)° (in molecules 1 and 2, respectively).

Experimental

A 2 *M* toluene solution of Me_2Zn (1.1 ml, 2.186 mmol) was added dropwise to a vigorously stirred ether solution of (II) (1.00 g, 2.18 mmol) at 195 K. The resulting reaction mixture was warmed to room temperature and stirred for 18 h. The solvent was removed using a vacuum pump and the remaining white residue extracted with

THF. Filtration through Celite, reduction of the solvent volume and overlayering with pentane afforded, after solvent diffusion over a period of days at 238 K, compound (I) as white colourless crystals. Crystals suitable for an X-ray structure determination were grown by solvent diffusion, by keeping saturated THF solutions of (I) overlayered with pentane at 238 K for several days (yield 40%). Spectroscopic analysis: ^{1}H NMR (3000 MHz, C_6D_6, δ, p.p.m.): 0.234 (*s*, $SiMe_3$), 1.214 (*m*, furan), 2.785 (*s*, NH), 3.626 (*m*, furan); ^{13}C NMR (500 MHz, C_6D_6, δ, p.p.m.): 5.593 ($SiMe_3$), 19.404 (furan), 63.964 (furan).

Crystal data

$[GaCl_2(C_3H_{10}NSi)(C_4H_8O)]$
$M_r = 300.93$
Triclinic, $P\overline{1}$
$a = 9.4539$ (2) Å
$b = 11.8134$ (3) Å
$c = 12.8999$ (3) Å
$\alpha = 86.918$ (1)°
$\beta = 83.194$ (1)°
$\gamma = 77.308$ (1)°
$V = 1395.02$ (6) Å^3
$Z = 4$
$D_x = 1.433$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 7048 reflections
$\theta = 1.59$–28.36°
$\mu = 2.41$ mm^{-1}
$T = 193$ (2) K
Cube, colourless
0.44 × 0.29 × 0.24 mm

Data collection

Siemens SMART 1 K CCD diffractometer
ω scans
Absorption correction: multi-scan (Blessing, 1995)
$T_{\min} = 0.392$, $T_{\max} = 0.563$
12 575 measured reflections
6380 independent reflections
4826 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.024$
$\theta_{\max} = 28.36°$
$h = -12 \rightarrow 12$
$k = -15 \rightarrow 15$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
$R(F) = 0.038$
$wR(F^2) = 0.108$
$S = 1.03$
6380 reflections
261 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0514P)^2 + 0.8048P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.96$ e Å^{-3}
$\Delta\rho_{\min} = -0.51$ e Å^{-3}
Extinction correction: *SHELXL*97 (Sheldrick, 1997)
Extinction coefficient: 0.0004 (5)

H-atom positions were calculated for the ordered C atoms and were refined as riding. H atoms on N were located from difference Fourier maps and their coordinates were fixed during refinement. All H-atom displacement parameters were refined isotropically. Both of the tetrahydrofuran ligands exhibit disorder; atoms C6, C7 and C13 were split into two positions and refined with occupancies of 0.5. H atoms were not calculated on atoms C5, C6, C7, C12, C13 or C14 of the disordered tetrahydrofuran ligands.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SAINT* (Siemens, 1995); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXL*97.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BK1507). Services for accessing these data are described at the back of the journal.

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