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[(Carbazolylmethyl)dimethylamine-*N*]-trimethylgallium

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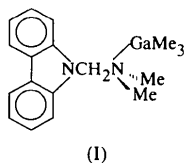
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Abstract

In the title structure, [Ga(CH₃)₃(C₁₅H₁₆N₂)], both the Ga and amine N atoms appear in distorted tetrahedral environments. The Ga—N(2) bond distance is 2.173 (3) Å.

Comment

High-purity trimethylgallium (TMGa) has been used extensively in the preparation of compound semiconductor epitaxy layers and its purity is critical to the quality of the product. One of the best methods of purification is to form a TMGa–ligand adduct, purify this adduct and finally decompose it to liberate TMGa (Bradley, Chudzynska & Frigo, 1988; Bradley, Chudzynska, Factor, Frigo, Hursthouse, Hussain & Smith, 1988; Foster, Rushworth, Cole-Hamilton, Jones & Stagg, 1988). In order to achieve successful purification of TMGa and related precursors, it is essential to select a suitable ligand and we have been investigating 9-(dimethylaminomethyl)carbazole and have used it in the title compound, (I).



As the N(1) atom is part of the aromatic ring, gallium is coordinated by only one N atom, N(2), as expected. The Ga atom is four coordinate and the geometry around it and the N(2) atom can be described as distorted tetrahedral. The Ga—N(2) bond distance of 2.173 (3) Å is comparable to the value of 2.12 (1) Å in [Me₃GaNH₂C(CH₃)₃] (Atwood, Jones & Cowley, 1992). Bond angles of 114.2 (3)–116.9 (3) for C—Ga—C and 101.6 (2)–102.0 (2)° for C—Ga—N(2) are comparable to those in [Me₃GaNH₂C(CH₃)₃] [111.8 (4)–117.1 (4) and 98.5 (4)–110.4 (5)°, respectively]. The obtuse N(2)—C(13)—N(1) angle of 115.9 (3)° can be

attributed to steric repulsion between the carbazole group and the methyl groups on the N(2) atom. The substituents on the Ga and N(2) atoms are mutually staggered along the Ga—N(2) bond. The three fused rings of the carbazole moiety lie approximately in the same plane, the mean deviation from which is only 0.05 Å.

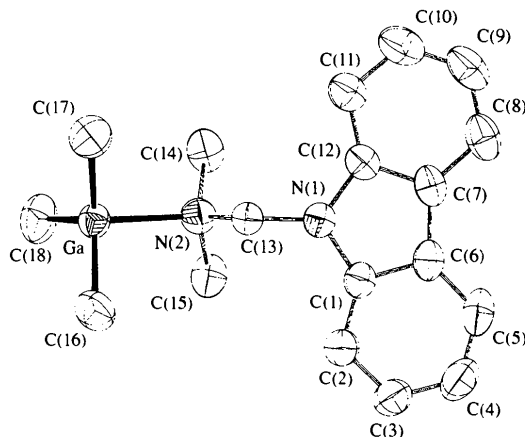


Fig. 1. View of the title structure showing the atom-numbering scheme and 50% probability displacement ellipsoids. H atoms have been omitted for clarity.

Experimental

All manipulations were performed in a glove box under purified nitrogen. Prior to use the solvents were refluxed with sodium benzophenone and distilled under nitrogen. To a benzene solution (10 ml) of 9-(dimethylaminomethyl)carbazole (0.45 g, 2 mmol), a solution of trimethylgallium (0.30 g, 2.6 mmol) in benzene (2 ml) was added dropwise. The resulting mixture was stirred at room temperature for 2 h. The title structure was isolated by removing part of the solvent. A single crystal was obtained by recrystallization from benzene and sealed in a glass capillary tube under nitrogen.

Crystal data

[Ga(CH₃)₃(C₁₅H₁₆N₂)]
M_r = 339.13
 Monoclinic
*P*2₁/*n*
a = 11.932 (2) Å
b = 10.032 (8) Å
c = 15.309 (3) Å
 β = 109.73 (1)°
V = 1725 (1) Å³
Z = 4
D_x = 1.31 Mg m⁻³
D_m not measured

Data collection

Enraf–Nonius CAD-4
 diffractometer

Mo *K*α radiation

λ = 0.71073 Å

Cell parameters from 25
 reflections

θ = 14.01–14.88°

μ = 1.58 mm⁻¹

T = 296 K

Rectangular parallelepiped

0.95 × 0.42 × 0.35 mm

Colourless

2297 reflections with
I > 3σ(*I*)

$\omega/2\theta$ scans
Absorption correction:
 ψ scans (North, Phillips
& Mathews, 1968)
 $T_{\min} = 0.521$, $T_{\max} = 0.575$
3391 measured reflections
3241 independent reflections

$R_{\text{int}} = 0.007$
 $\theta_{\max} = 25^\circ$
 $h = 0 \rightarrow 14$
 $k = 0 \rightarrow 11$
 $l = -18 \rightarrow 18$
3 standard reflections
every 300 reflections
intensity decay: 1.1%

Refinement

Refinement on F
 $R = 0.040$
 $wR = 0.048$
 $S = 1.40$
2176 reflections
291 parameters
H-atom parameters refined
 $w = 1/\sigma^2(F_o)$

$(\Delta/\sigma)_{\max} = 0.08$
 $\Delta\rho_{\max} = 0.40 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.42 \text{ e } \text{Å}^{-3}$
Extinction coefficient:
 3.57×10^{-6}
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. Selected geometric parameters (Å , $^\circ$)

Ga—N(2)	2.173 (3)	N(1)—C(12)	1.396 (4)
Ga—C(16)	1.973 (5)	N(1)—C(13)	1.447 (4)
Ga—C(17)	1.972 (5)	N(2)—C(13)	1.489 (4)
Ga—C(18)	1.968 (5)	N(2)—C(14)	1.471 (5)
N(1)—C(11)	1.397 (4)	N(2)—C(15)	1.464 (5)
C(16)—Ga—N(2)	102.0 (2)	C(14)—N(2)—C(13)	110.7 (3)
C(17)—Ga—N(2)	102.0 (2)	C(15)—N(2)—C(14)	110.5 (4)
C(18)—Ga—N(2)	101.6 (2)	C(15)—N(2)—C(13)	110.8 (3)
C(17)—Ga—C(16)	116.9 (3)	C(13)—N(2)—Ga	108.1 (2)
C(18)—Ga—C(16)	114.2 (3)	C(14)—N(2)—Ga	108.2 (3)
C(18)—Ga—C(17)	116.6 (3)	C(15)—N(2)—Ga	108.5 (2)
C(12)—N(1)—C(11)	108.5 (3)	N(1)—C(12)—C(7)	108.6 (3)
C(12)—N(1)—C(13)	125.8 (3)	N(1)—C(13)—N(2)	115.9 (3)
C(1)—N(1)—C(13)	125.2 (3)		

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *TEXSAN PRO-CESS* (Molecular Structure Corporation, 1987). Program(s) used to solve structure: *MITHRIL* (Gilmore, 1983). Program(s) used to refine structure: *TEXSAN LS*. Molecular graphics: *ORTEPII* (Johnson, 1976).

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Lists of structure factors, anisotropic displacement parameters, atomic coordinates and complete geometry have been deposited with the IUCr (Reference: BM1084). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- Atwood, D. A., Jones, R. A. & Cowley, A. H. (1992). *J. Organomet. Chem.* **434**, 143–150.
- Bradley, D. C., Chudzynska, H., Factor, M. M., Frigo, D. M., Hursthouse, M. B., Hussian, B. & Smith, L. M. (1988). *Polyhedron*, **7**, 1289–1298.
- Bradley, D. C., Chudzynska, H. & Frigo, D. M. (1988). *Chemtronics*, **3**, 159–161.
- Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
- Foster, D. F., Rushworth, S. A., Cole-Hamilton, D. J., Jones, A. C. & Stagg, J. P. (1988). *Chemtronics*, **3**, 38–43.
- Gilmore, C. J. (1983). *MITHRIL. Computer Program for the Automatic Solution of Crystal Structures from X-ray Data*. Department of Chemistry, University of Glasgow, Scotland.

- Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- Molecular Structure Corporation (1987). *TEXSAN. TEXRAY Structure Analysis Package*. Revised. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
- North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.

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Tetrachlorobis(*N,N,N',N'*-tetramethylethylenediamine-*N,N'*)thorium(IV)

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Abstract

The title compound, $[\text{ThCl}_4(\text{C}_6\text{H}_{16}\text{N}_2)_2]$, displays an approximate dodecahedral geometry in the solid state, with the Th atom binding to four Cl ligands and to the four N atoms of two tetramethylethylenediamine ligands. The Th—Cl and Th—N distances are in the ranges 2.683 (3)–2.692 (3) and 2.800 (7)–2.825 (9) Å, respectively.

Comment

The preparation and characterization of the thorium and uranium complexes $[\text{MCl}_4(\text{Me}_2\text{NCH}_2\text{CH}_2\text{NMe}_2)_2]$ ($M = \text{Th}, \text{U}$) were reported by Edwards, Weydert, Petrie & Andersen (1994). The determination of the molecular structure of the uranium species (Zalkin, Edwards, Zhang & Andersen, 1986) is now compared with that of the thorium analog described herein. The eight-coordinate thorium complex $[\text{ThCl}_4(\text{Me}_2\text{NCH}_2\text{CH}_2\text{NMe}_2)_2]$, (I), is isostructural with its uranium analog and consists of a Th atom surrounded by the four N atoms of two tetramethylethylenediamine

