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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: JZ1111). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- Beno, M. A., Hope, H., Olmstead, M. M. & Power, P. P. (1985). *Organometallics*, **4**, 2117–2121.
- Enraf-Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf-Nonius, Delft, The Netherlands.
- Fair, C. K. (1990). *MolEN. An Interactive Intelligent System for Crystal Structure Analysis*. Enraf-Nonius, Delft, The Netherlands.
- Hoffmann, D., Bauer, W., Hampel, F., van Eikema Hommes, N. J., Ragué Schleyer, P. v., Otto, P., Pieper, U., Stalke, D., Wright, D. S. & Snaith, R. (1994). *J. Am. Chem. Soc.* **116**, 528–536.
- Johnson, C. K. (1976). *ORTEP II*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- Patterson, S. P., Karle, I. L. & Stucky, G. D. (1970). *J. Am. Chem. Soc.* **92**, 1150–1157.
- Schade, C., Ragué Schleyer, P. v., Dietrich, H. & Mahdi, W. (1986). *J. Am. Chem. Soc.* **108**, 2484–2485.
- Sheldrick, G. M. (1985). *SHELXS86. Crystallographic Computing 3*, edited by G. M. Sheldrick, C. Krüger & R. Goddard, pp. 175–189. Oxford University Press.
- Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.
- Syguła, A. & Rabideau, P. W. (1992). *J. Am. Chem. Soc.* **114**, 821–824.
- Waldkircher, M. (1994). Unpublished observations.
- Zarges, W., Marsch, M., Harms, K. & Boche, G. (1989). *Chem. Ber.* **122**, 2303–2309.

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(Acridine)trimethylgallium–Acridine (2/1)

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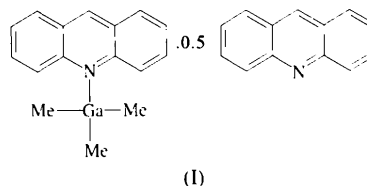
(Received 10 January 1995; accepted 23 October 1995)

Abstract

The reaction of excess trimethylgallium with acridine in diethyl ether gave the title layer inclusion compound, [Ga(CH₃)₃(C₁₃H₉N)]_{0.5}C₁₃H₉N. The Ga—N bond length is 2.203 (3) Å.

Comment

Metal-organic chemical vapour deposition (MOCVD) has become a powerful method for the preparation of thin films of electronic materials. A major effort has been concentrated on exploring the toxicity, stability and volatility of precursors for MOCVD (Bradley, Dawes, Hursthouse, Smith & Thornton-Pett, 1990; Bradley, Frigo, Harding, Hursthouse & Motevalli, 1992). The title compound, (I), was obtained and studied by X-ray analysis, revealing that the (acridine)trimethylgallium and free acridine molecules lie in layers.



Analysis of (I) (Fig. 1) shows that the asymmetric unit has one molecule of (acridine)trimethylgallium in a general position and an uncoordinated acridine molecule lying about an inversion centre (which requires that it be disordered). The Ga atom has distorted tetrahedral coordination, with N—Ga—C angles in the range 98.9(1)–108.3(1)° and C—Ga—C angles in the range 107.5(1)–117.3(1)°. The Ga—N distance of 2.203 (3) Å is slightly longer than previously reported

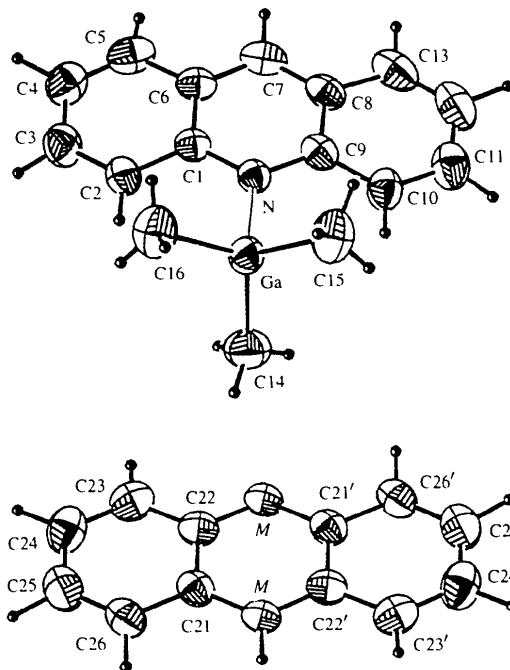


Fig. 1. The molecular structure of the title compound showing 50% probability displacement ellipsoids.

Ga—N distances (Bradford, Bradley, Hursthouse & Motevalli, 1992). We ascribe this to the steric hindrance of the acridine moiety. The plane composed of atoms C1, C6—C9 and N makes angles of 4.9 (4) and 4.0 (5)° with the C1—C6 and C8—C13 rings, respectively.

The layer structure of the complex can be seen clearly in the packing diagram (Fig. 2). Free acridine molecules (disordered on inversion centres) are sandwiched between the acridine moieties of the (acridine)trimethylgallium molecules; the shortest interplanar separation is 3.926 (3) Å and the two planes (coordinated and free acridine) are nearly parallel, with a dihedral angle of 1 (1)°.

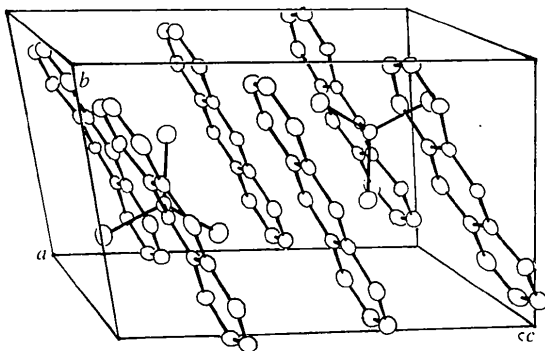


Fig. 2. The molecular packing in the unit cell.

Experimental

The title product was prepared by reaction of acridine and Me₃Ga (molar ratio 1:1.2) in Et₂O for 4 h at room temperature. Removal of the solvent and recrystallization from cyclohexane afforded crystals suitable for analysis. All operations were carried out under an atmosphere of specially dried oxygen-free nitrogen using Schlenk tube techniques and a dry box. Solvents were carefully dried by distillation from sodium diphenyl ketone under nitrogen before use.

Crystal data

[Ga(CH₃)₃(C₁₃H₉N)]_n·
0.5C₁₃H₉N

M_r = 383.66

Triclinic

*P*1

a = 9.474 (1) Å

b = 9.529 (2) Å

c = 12.160 (2) Å

α = 99.98 (1)°

β = 97.16 (1)°

γ = 115.92 (1)°

V = 947.2 Å³

Z = 2

D_r = 1.343 Mg m⁻³

D_m not measured

Mo *K*α radiation

λ = 0.71073 Å

Cell parameters from 25
reflections

θ = 14–16°

μ = 1.45 mm⁻¹

T = 296 K

Block

0.25 × 0.25 × 0.15 mm

Red

Data collection

Enraf–Nonius CAD-4
diffractometer

ω/2θ scans

Absorption correction:

refined from Δ*F*

(*DIFABS*; Walker &

Stuart, 1983)

T_{min} = 0.80, *T_{max}* = 1.00

3707 measured reflections

3527 independent reflections

2758 observed reflections

[*I* > 3σ(*I*)]

R_{int} = 0.02

θ_{max} = 26°

h = -11 → 11

k = -11 → 11

l = 0 → 14

2 standard reflections

frequency: 60 min

intensity decay: 8.6%

Refinement

Refinement on *F*

R = 0.0385

wR = 0.0393

S = 1.534

2758 reflections

226 parameters

Only coordinates of H atoms
refined

w = 1/[σ²(*F_o*) + (0.01*F_o*)²
+ 1.0]

(Δ/σ)_{max} < 0.001

Δρ_{max} = 0.262 e Å⁻³

Δρ_{min} = -0.445 e Å⁻³

Extinction correction: none

Atomic scattering factors

from *International Tables*

for *X-ray Crystallography*

(1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

$$B_{\text{eq}} = (4/3)\sum_i \sum_j \beta_{ij} \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B_{eq}</i>
Ga	0.38354 (4)	0.39712 (4)	0.21437 (3)	3.766 (8)
N	0.1584 (3)	0.4147 (3)	0.1828 (2)	3.23 (6)
C1	0.0204 (3)	0.3026 (3)	0.2034 (2)	3.32 (7)
C2	0.0073 (4)	0.1523 (4)	0.2164 (3)	4.19 (8)
C3	-0.1299 (4)	0.0400 (4)	0.2372 (3)	5.0 (1)
C4	-0.2618 (4)	0.0672 (4)	0.2475 (3)	5.2 (1)
C5	-0.2555 (4)	0.2056 (4)	0.2332 (3)	5.0 (1)
C6	-0.1161 (3)	0.3285 (4)	0.2093 (3)	3.76 (8)
C7	-0.1086 (3)	0.4690 (4)	0.1880 (3)	4.45 (8)
C8	0.0242 (4)	0.5779 (3)	0.1574 (3)	3.89 (8)
C9	0.1579 (3)	0.5462 (3)	0.1544 (3)	3.55 (7)
C10	0.2905 (4)	0.6552 (4)	0.1175 (3)	4.45 (9)
C11	0.2902 (4)	0.7860 (4)	0.0880 (3)	5.3 (1)
C12	0.1601 (5)	0.8187 (4)	0.0931 (3)	5.7 (1)
C13	0.0315 (4)	0.7180 (4)	0.1257 (3)	5.4 (1)
C14	0.3688 (4)	0.2844 (4)	0.0576 (3)	5.9 (1)
C15	0.5613 (4)	0.6191 (5)	0.2756 (3)	5.9 (1)
C16	0.3768 (4)	0.2820 (4)	0.3351 (3)	6.0 (1)
C21	1.0012 (3)	0.3621 (3)	0.5207 (3)	3.69 (8)
C22	0.8628 (3)	0.3860 (4)	0.5238 (3)	3.80 (8)
C23	0.7262 (4)	0.2640 (4)	0.5487 (3)	4.76 (9)
C24	0.7274 (4)	0.1300 (4)	0.5697 (3)	5.3 (1)
C25	0.8647 (4)	0.1077 (4)	0.5667 (3)	5.3 (1)
C26	0.9972 (4)	0.2193 (4)	0.5427 (3)	4.57 (9)
<i>M</i> †	1.1380 (3)	0.4793 (3)	0.4977 (3)	3.51 (7)

† *M* = ½(*C* + *N*).

Table 2. Selected geometric parameters (Å, °)

Ga—N	2.203 (3)	N—C9	1.358 (4)
Ga—C14	1.975 (3)	C21— <i>M</i>	1.392 (4)
N—C1	1.366 (4)		
Ga—N—C1	120.8 (2)	N—Ga—C15	107.1 (1)
Ga—N—C9	120.6 (2)	N—Ga—C16	108.3 (1)
N—Ga—C14	98.9 (1)		

H atoms were located on a difference map and refined with isotropic displacement parameters.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1985a). Cell refinement: *CAD-4 Software*. Data reduction: *SDP*

(Enraf–Nonius, 1985b). Program(s) used to solve structure: *MULTAN11/82* (Main *et al.*, 1982). Program(s) used to refine structure: *SDP*. Molecular graphics: *ORTEP* (Johnson, 1965). Software used to prepare material for publication: *SDP OMEGA*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and least-squares-planes data have been deposited with the IUCr (Reference: FG1063). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- Bradford, A. M., Bradley, D. C., Hursthouse, M. B. & Motevalli, M. (1992). *Organometallics*, **11**, 1111–1115.
- Bradley, D. C., Dawes, H. M., Hursthouse, M. B., Smith L. M. & Thornton-Pett, M. (1990). *Polyhedron*, **9**, 343–351.
- Bradley, D. C., Frigo, D. M., Harding, I. S., Hursthouse, M. B. & Motevalli, M. (1992). *J. Chem. Soc. Chem. Commun.* pp. 577–578.
- Enraf–Nonius (1985a). *CAD-4 Software*. Fortran Version 4.0. Enraf–Nonius, Delft, The Netherlands.
- Enraf–Nonius (1985b). *Structure Determination Package. SDP/VAX User's Guide*. Version 3.0. Enraf–Nonius, Delft, The Netherlands.
- Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
- Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1982). *MULTAN11/82. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Universities of York, England, and Louvain, Belgium.
- Walker, N. & Stuart, D. (1983). *Acta Cryst.* **A39**, 158–166.

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Diammine(dimethylmalonato)platinum(II) Monohydrate

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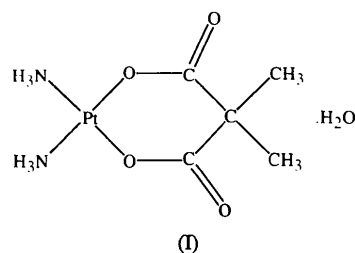
Abstract

The complex molecule of the title compound, [Pt(C₅H₆-O₄)(NH₃)₂].H₂O, assumes a boat conformation that is more planar than expected from an optimization of bond

lengths and angles. Likely origins of this extra flattening are the repulsion between the Pt centre and the methyl group in the axial position, and hydrogen bonding with the water of crystallization.

Comment

Fig. 1 shows an *ORTEP*II (Johnson, 1976) drawing of the complex molecule, and Fig. 2 shows the crystal packing. The complex of the title compound, (I), possesses a mirror plane which contains the Pt, C(2), C(3) and C(4) atoms, and is perpendicular to the Pt coordination plane. The Pt atom exhibits usual square-planar coordination. The Pt—N and Pt—O bond distances are as expected for the *cis*-PtN₂O₂ chromophore (Rochon, Melanson, Macquet, Bellanger-Gariepy & Beauchamp, 1985, and references therein). There is one water molecule present in the lattice (Fig. 2), which is connected by hydrogen bonding to both ammine ligands of one molecule and to one carbonyl O atom [O(2)] of each of two other molecules. The compound is isostructural with the recently determined cyclopropylmalonato derivative (Dong *et al.*, 1990).



The molecule adopts a boat conformation, with the Pt and C(2) atoms at the bow and the stern, respectively, similar to that found in other malonato complexes of platinum(II) (Rochon, Melanson, Macquet, Bellanger-Gariepy & Beauchamp, 1985; Dong *et al.*, 1990). As in these complexes, the boat is flattened at the Pt end, with O—Pt—O—C torsion angles of $\pm 41^\circ$ (instead of $\pm 60^\circ$ if all the bond angles were tetrahedral) (Table 4). The C2 end of the boat is flattened as well, with C—C—C—O torsion angles of $\pm 44^\circ$ (Table 4). In order to find out whether this flattening is simply due to the strain introduced to the six-membered ring by the two perpendicular Pt—O bonds of 2.00(6) Å, we have constructed energy-minimized models using the program *AMBER* (Weiner & Kollman, 1981). The energy function contained only bond and bond angle terms. The energy minimization resulted in a completely relaxed boat structure, with the bond lengths and bond angles having exactly the required values. The torsion angles of the relaxed model are given in Table 4. Their comparison with the experimental values shows that the boat is flattened at both ends more than expected for