metal-organic compounds

(Acetonitrile-κN)trichloridogallium(III)

Markus Schaffrath,[a] Axel Schulz[a]* and Alexander Villinger[b]

[a]Universität Rostock, Institut für Chemie, Abteilung für Anorganische Chemie, Albert-Einstein-Strasse 3a, 18059 Rostock, Germany, and Leibniz-Institut für Katalyse e.V. an der Universität Rostock, Albert-Einstein-Strasse 29a, 18059 Rostock, Germany, and [b]Universität Rostock, Institut für Chemie, Abteilung für Anorganische Chemie, Albert-Einstein-Strasse 3a, 18059 Rostock, Germany
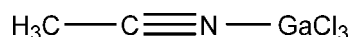Correspondence e-mail: axel.schulz@uni-rostock.de

In the title complex, [GaCl$_3$(CH$_3$CN)], the Ga^{III} atom shows a slightly distorted tetrahedral coordination, with three Cl atoms and one N atom of the acetonitrile as ligands. The Ga atom, one Cl atom, the N atom, both C atoms and one H atom of the acetonitrile molecule are situated on a mirror plane. The crystal packing is stabilized by several weak intermolecular C—H···Cl hydrogen-bonding contacts.

Related literature

For related literature, see: Herler *et al.* (2005).

$$H_3C\!-\!\!\!-\!\!\!-C\!\equiv\!N\!-\!\!\!-\!\!\!-GaCl_3$$

Experimental

Crystal data

[GaCl$_3$(C$_2$H$_3$N)]
M_r = 217.12
Orthorhombic, $Pnma$
a = 9.1760 (18) Å
b = 7.5100 (15) Å
c = 10.857 (2) Å

V = 748.2 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 4.64 mm^{-1}
T = 173 (2) K
$0.73 \times 0.12 \times 0.09$ mm

Data collection

Bruker–Nonius Apex X8 CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
T_{min} = 0.130, T_{max} = 0.660

11546 measured reflections
1011 independent reflections
878 reflections with $I > 2\sigma(I)$
R_{int} = 0.037

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.017
$wR(F^2)$ = 0.042
S = 1.04
1011 reflections

48 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ga1—N1	1.9676 (18)	Ga1—Cl2	2.1432 (5)
Ga1—Cl1	2.1379 (7)		
N1—Ga1—Cl1	104.68 (6)	Cl1—Ga1—Cl2	113.371 (17)
N1—Ga1—Cl2	105.14 (3)	Cl2—Ga1—Cl2^i	113.93 (3)

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

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C2—H1···Cl1^{ii}	0.88 (5)	3.01 (5)	3.786 (3)	147 (5)
C2—H1···Cl2^{iii}	0.88 (5)	3.34 (5)	3.818 (3)	117 (3)
C2—H2···Cl1^{iv}	0.87 (3)	3.44 (3)	3.9172 (10)	117 (2)
C2—H2···Cl2^v	0.87 (3)	2.90 (3)	3.615 (2)	141 (2)
C2—H2···Cl2^{vi}	0.87 (3)	3.23 (3)	3.733 (3)	119 (2)

Symmetry codes: (ii) $x + 1, y, z$; (iii) $x + \frac{1}{2}, y, -z + \frac{1}{2}$; (iv) $-x + 1, y - \frac{1}{2}, -z + 1$; (v) $-x + \frac{3}{2}, -y, z + \frac{1}{2}$; (vi) $-x + 1, -y, -z + 1$.

Data collection: *APEX2* (Bruker, 2003); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2003); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 2005); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WM2142).

References

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Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
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supplementary materials

(Acetonitrile-*κN*)trichloridogallium(III)

M. Schaffrath, A. Schulz and A. Villinger

Comment

In the title adduct, the coordination of the Ga atom by the three Cl atoms and the N atom of the acetonitrile ligand can be considered as slightly distorted tetrahedral, with Cl—Ga—N bond angles in the range 104.68 (6)–105.14 (3) ° and Cl—Ga—Cl bond angles in the range 113.371 (17)–113.93 (3) ° (Fig. 1).

The C_s-symmetric molecule exhibits an almost linear Ga—N1—C1—C2 unit (C1—N1—Ga1 = 179.22 (19) °, N1—C1—C2 = 179.9 (3) °), and the H atoms of the methyl group adopt a staggered conformation relative to the Cl atoms.

In the crystal structure, the $GaCl_3 \cdot CH_3CN$ adducts are linked by very weak intermolecular C—H···Cl interactions (Fig. 2, Table).

Experimental

The title compound was unintentionally obtained as a by-product of an attempted reaction of 4-bis(trimethylsilyl)-amino-1,2,4,3,5-triazadiphosphole $GaCl_3$ adduct (Herler *et al.*, 2005) with $Cr(CH_3CN)_3(CO)_3$ in CH_2Cl_2. Recrystallization from a saturated CH_2Cl_2/Et_2O solution at ambient temperature gave colourless needle-like crystals of $GaCl_3 \cdot CH_3CN$.

Refinement

All Hydrogen atoms were found from difference Fourier maps and were refined freely.
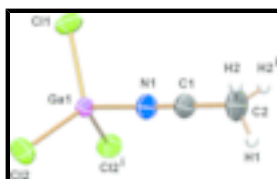
Figures

Fig. 1. Molecular structure of (I), showing the atomic numbering scheme and displacement ellipsoids drawn at the 50% probability level. [Symmetry code: (i) x, $-y + 1/2$, z.]
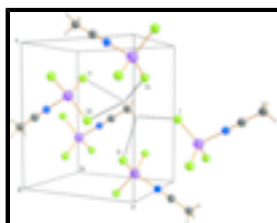

Fig. 2. Part of the packing diagram of (I). Unique C—H···Cl interactions represented by dashed lines are shown for one molecule of (I). Colour code as in Fig. 1. [Symmetry codes: (i) $x + 1$, y, z; (ii) $x + 1/2$, y, $-z + 1/2$; (iii) $-x + 1$, $y - 1/2$, $-z + 1$; (iv) $-x + 3/2$, $-y$, $z + 1/2$; (v) $-x + 1$, $-y$, $-z + 1$.]

supplementary materials

(Acetonitrile-κ*N*)trichloridogallium(III)

Crystal data

$[GaCl_3(C_2H_3N)]$	$F_{000} = 416$
$M_r = 217.12$	$D_x = 1.928$ Mg m^{-3}
Orthorhombic, *Pnma*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -P 2ac 2n	Cell parameters from 4237 reflections
$a = 9.1760$ (18) Å	θ = 5.8–56.4º
$b = 7.5100$ (15) Å	μ = 4.64 mm^{-1}
$c = 10.857$ (2) Å	$T = 173$ (2) K
$V = 748.2$ (3) Å^3	Needle, colourless
$Z = 4$	0.73 × 0.12 × 0.09 mm

Data collection

Bruker–Nonius Apex X8 CCD diffractometer	1011 independent reflections
Radiation source: sealed tube	878 reflections with $I > 2σ(I)$
Monochromator: graphite	$R_{int} = 0.037$
$T = 173(2)$ K	$θ_{max} = 28.5º$
ω scans	$θ_{min} = 2.9º$
Absorption correction: multi-scan (SADABS; Sheldrick, 2004)	$h = -12→12$
$T_{min} = 0.130$, $T_{max} = 0.660$	$k = -10→9$
11546 measured reflections	$l = -14→14$

Refinement

Refinement on F^2	Hydrogen site location: difference Fourier map
Least-squares matrix: full	All H-atom parameters refined
$R[F^2 > 2σ(F^2)] = 0.017$	$w = 1/[σ^2(F_o^2) + (0.0212P)^2 + 0.2025P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.042$	$(Δ/σ)_{max} < 0.001$
$S = 1.04$	$Δρ_{max} = 0.28$ e Å^{-3}
1011 reflections	$Δρ_{min} = -0.38$ e Å^{-3}
48 parameters	Extinction correction: SHELXL, $Fc^* = kFc[1+0.001xFc^2λ^3/\sin(2θ)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0018 (7)
Secondary atom site location: difference Fourier map	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($Å^2$)

	x	y	z	$U_{iso}*/U_{eq}$
Ga1	0.41273 (2)	0.2500	0.356223 (19)	0.02617 (9)
Cl1	0.25406 (7)	0.2500	0.50041 (6)	0.04725 (17)
Cl2	0.41187 (5)	0.01076 (6)	0.24861 (4)	0.04570 (13)
N1	0.60162 (19)	0.2500	0.44199 (18)	0.0359 (4)
C1	0.7096 (2)	0.2500	0.4926 (2)	0.0331 (5)
C2	0.8469 (3)	0.2500	0.5568 (3)	0.0437 (6)
H1	0.926 (5)	0.2500	0.511 (6)	0.13 (2)*
H2	0.861 (3)	0.159 (4)	0.605 (3)	0.106 (10)*

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ga1	0.02312 (12)	0.02984 (13)	0.02554 (13)	0.000	0.00012 (8)	0.000
Cl1	0.0414 (3)	0.0532 (3)	0.0472 (3)	0.000	0.0204 (3)	0.000
Cl2	0.0555 (3)	0.0393 (2)	0.0424 (2)	−0.00081 (19)	0.00219 (17)	−0.01284 (17)
N1	0.0290 (9)	0.0415 (10)	0.0372 (10)	0.000	−0.0038 (8)	0.000
C1	0.0296 (10)	0.0345 (11)	0.0352 (11)	0.000	−0.0010 (8)	0.000
C2	0.0288 (11)	0.0513 (15)	0.0512 (16)	0.000	−0.0133 (11)	0.000

Geometric parameters (Å, °)

Ga1—N1	1.9676 (18)	N1—C1	1.133 (3)
Ga1—Cl1	2.1379 (7)	C1—C2	1.440 (3)
Ga1—Cl2	2.1432 (5)	C2—H1	0.88 (5)
Ga1—Cl2^i	2.1432 (5)	C2—H2	0.87 (3)
N1—Ga1—Cl1	104.68 (6)	C1—N1—Ga1	179.22 (19)
N1—Ga1—Cl2	105.14 (3)	N1—C1—C2	179.9 (3)
Cl1—Ga1—Cl2	113.371 (17)	C1—C2—H1	116 (4)
N1—Ga1—Cl2^i	105.14 (3)	C1—C2—H2	115 (2)
Cl1—Ga1—Cl2^i	113.371 (17)	H1—C2—H2	103 (3)
Cl2—Ga1—Cl2^i	113.93 (3)		

Cl1—Ga1—N1—C1	0.00 (3)	Cl2^{i}—Ga1—N1—C1	119.72 (3)
Cl2—Ga1—N1—C1	−119.72 (4)	Ga1—N1—C1—C2	180 (100)

Symmetry codes: (i) x, $-y+1/2$, z.

Hydrogen-bond geometry (Å, °)

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C2—H1⋯Cl1^{ii}	0.88 (5)	3.01 (5)	3.786 (3)	147 (5)
C2—H1⋯Cl2^{iii}	0.88 (5)	3.34 (5)	3.818 (3)	117 (3)
C2—H2⋯Cl1^{iv}	0.87 (3)	3.44 (3)	3.9172 (10)	117 (2)
C2—H2⋯Cl2^{v}	0.87 (3)	2.90 (3)	3.615 (2)	141 (2)
C2—H2⋯Cl2^{vi}	0.87 (3)	3.23 (3)	3.733 (3)	119 (2)

Symmetry codes: (ii) $x+1$, y, z; (iii) $x+1/2$, y, $-z+1/2$; (iv) $-x+1$, $y-1/2$, $-z+1$; (v) $-x+3/2$, $-y$, $z+1/2$; (vi) $-x+1$, $-y$, $-z+1$.
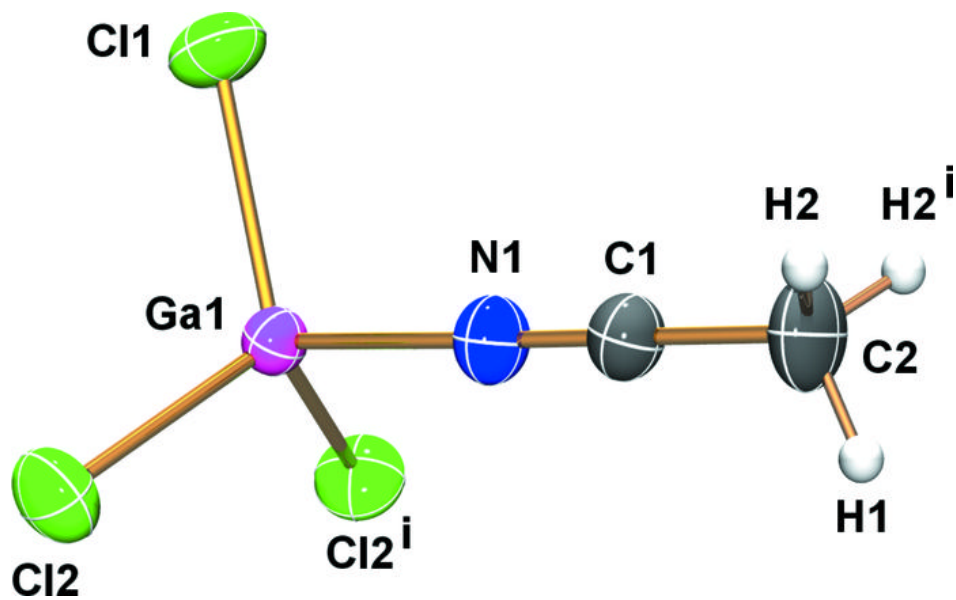
Fig. 1

Fig. 2