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(*tert*-Butyl isocyanide-κC)trichloridogallium(III)

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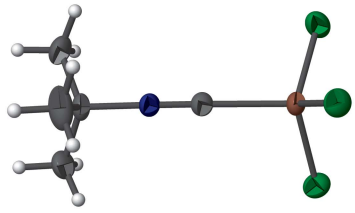
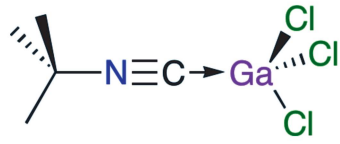
The crystal structure of (*tert*-butyl isocyanide-κC)trichloridogallium(III), $[GaCl_3(C_5H_9N)]$, features the first reported isocyanide–gallium trihalide complex. The Ga—C—N—C fragment is essentially linear. The methyl fragments of the *tert*-butyl group are eclipsed with the chloride ligands on the Ga atom. The molecule does not, however, exhibit threefold crystallographic symmetry, as it crystallizes within the $P2_1/c$ space group.

3D view



Chemical scheme



Structure description

The Ga—C—N—C fragment deviates only slightly from linearity with N1—C1—Ga1 and C1—N1—C2 angles of 179.26 (16) and 179.35 (18)°, respectively (Fig. 1). The angle between the Cl1—Ga1—C1 and N1—C2—C3 planes of 1.5 (2)° indicates a nearly perfect eclipsed conformation between the $-C(CH_3)_3$ and $-GaCl_3$ groups. In the crystal, there are no notable interactions between neighbouring molecules (Fig. 2).

The synthesis of trialkylgallium–isocyanide complexes was reported by Kingsley *et al.* (2012). For adducts of isocyanides with other main group elements, see: Bertani *et al.* (2001); Casanova *et al.* (1965); Fisher *et al.* (1994); Green *et al.* (1987); Meller & Batka (1969, 1970); Uhl *et al.* (1998). For an extensive theoretical study on main group element–isocyanide adducts, see: Timoshkin & Schaefer (2003).

Synthesis and crystallization

The title compound was obtained serendipitously from an attempted trapping experiment, involving the reaction of *tert*-butylisocyanide and tetramesityldisilene. $GaCl_3$ was added to act as a Lewis acid. X-ray quality single crystals were obtained from a solution of diethyl ether cooled to 253 K.

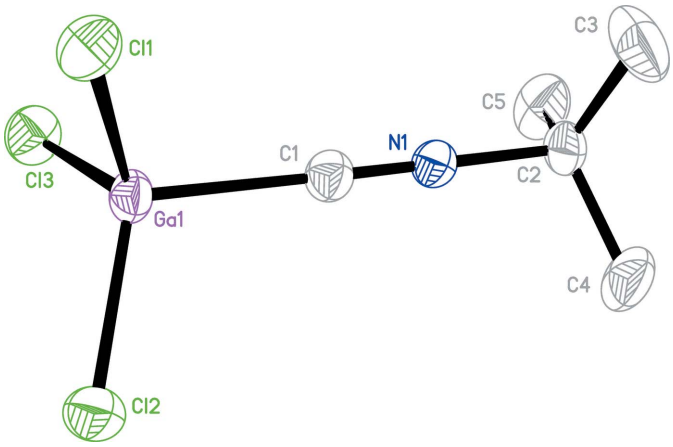


Figure 1
The molecular structure of the title compound, showing the atom-numbering scheme and with displacement ellipsoids drawn at the 50% probability level. H atoms have been omitted for clarity.

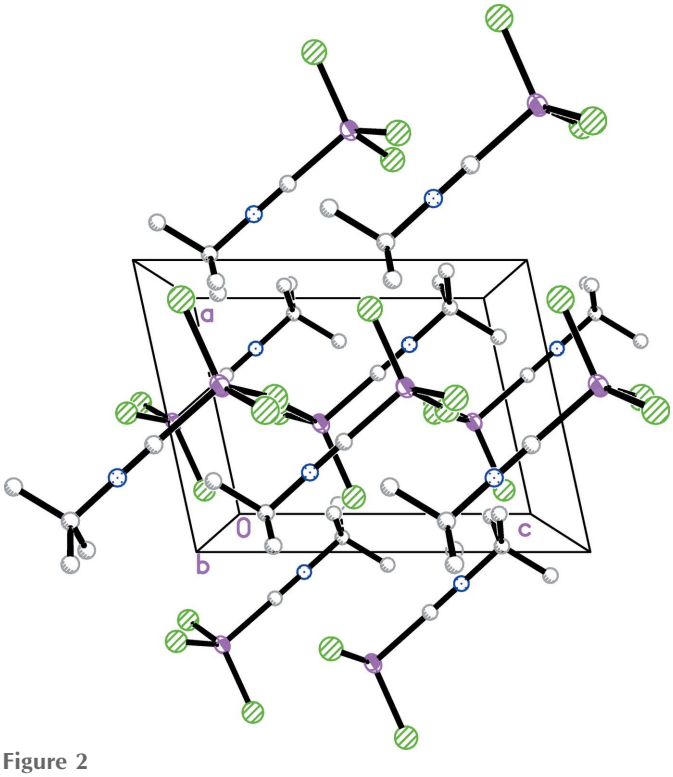


Figure 2
Crystal packing of the title compound viewed along the *b* axis. Hydrogen atoms are omitted for clarity.

Refinement

Crystal data, data collection and refinement details are shown in Table 1.

Table 1
Experimental details.

Crystal data	
Chemical formula	$[GaCl_3(C_5H_9N)]$
M_r	259.20
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	110
a, b, c (Å)	6.5170 (12), 19.393 (3), 8.5991 (16)
β (°)	102.287 (5)
V (Å^3)	1061.9 (3)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	10.00
Crystal size (mm)	0.23 × 0.17 × 0.11
Data collection	
Diffractometer	Bruker–Nonius KappaCCD APEXII
Absorption correction	Multi-scan (*SADABS*; Bruker, 2013)
T_{min}, T_{max}	0.557, 0.753
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	10964, 1876, 1791
R_{int}	0.024
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.596
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.021, 0.055, 1.11
No. of reflections	1876
No. of parameters	127
H-atom treatment	All H-atom parameters refined
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.33, −0.31

Computer programs: *APEX2* (Bruker, 2013), *SAINT* (Bruker, 2013), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*), *XP* (Sheldrick, 2008), *cif2tables.py* (Boyle, 2008).

Acknowledgements

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full crystallographic data

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Crystal data

$[GaCl_3(C_5H_9N)]$
M_r = 259.20
Monoclinic, $P2_1/c$
a = 6.5170 (12) Å
b = 19.393 (3) Å
c = 8.5991 (16) Å
β = 102.287 (5)°
V = 1061.9 (3) Å^3
Z = 4
$F(000)$ = 512
D_x = 1.621 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 6667 reflections
θ = 4.6–66.7°
μ = 10.00 mm^{-1}
T = 110 K
Needle, orange
0.23 × 0.17 × 0.11 mm

Data collection

Bruker–Nonius KappaCCD APEXII diffractometer
Radiation source: sealed tube
phi and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2013)
T_{min} = 0.557, T_{max} = 0.753
10964 measured reflections
1876 independent reflections
1791 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 66.7°, θ_{min} = 4.6°
h = −7→7
k = −22→22
l = −10→10

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.021
$wR(F^2)$ = 0.055
S = 1.11
1876 reflections
127 parameters
0 restraints
Primary atom site location: dual
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.031P)^2 + 0.275P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Ga1	0.57250 (3)	0.61614 (2)	0.63792 (2)	0.02511 (9)
Cl1	0.53074 (7)	0.70861 (2)	0.76750 (5)	0.03418 (12)
Cl2	0.87880 (7)	0.60850 (3)	0.58522 (6)	0.03958 (13)
Cl3	0.48498 (7)	0.52514 (2)	0.75065 (5)	0.03701 (12)
C1	0.3619 (3)	0.62469 (9)	0.4270 (2)	0.0275 (4)
N1	0.2429 (2)	0.62920 (7)	0.31030 (16)	0.0247 (3)
C2	0.0869 (3)	0.63437 (10)	0.1590 (2)	0.0295 (4)
C3	−0.0287 (4)	0.70142 (13)	0.1670 (3)	0.0529 (6)
H3A	−0.133 (5)	0.7048 (15)	0.071 (4)	0.070 (8)*
H3B	−0.102 (5)	0.6985 (18)	0.241 (4)	0.082 (11)*
H3C	0.064 (4)	0.7425 (16)	0.171 (4)	0.065 (8)*
C4	0.2098 (4)	0.63342 (14)	0.0282 (2)	0.0452 (5)
H4A	0.114 (4)	0.6379 (13)	−0.068 (3)	0.047 (6)*
H4B	0.314 (4)	0.6705 (13)	0.038 (3)	0.048 (7)*
H4C	0.286 (5)	0.5912 (16)	0.029 (3)	0.058 (8)*
C5	−0.0554 (4)	0.57180 (13)	0.1521 (3)	0.0450 (5)
H5A	−0.160 (4)	0.5739 (13)	0.054 (3)	0.048 (6)*
H5B	0.017 (5)	0.5279 (16)	0.152 (3)	0.060 (8)*
H5C	−0.124 (5)	0.5744 (15)	0.238 (4)	0.062 (8)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ga1	0.02527 (14)	0.02897 (14)	0.01932 (13)	−0.00094 (7)	0.00074 (9)	−0.00039 (7)
Cl1	0.0395 (2)	0.0326 (2)	0.0306 (2)	−0.00260 (17)	0.00762 (18)	−0.00620 (16)
Cl2	0.0283 (2)	0.0564 (3)	0.0345 (2)	−0.00090 (18)	0.00752 (18)	−0.00601 (19)
Cl3	0.0428 (3)	0.0308 (2)	0.0380 (2)	−0.00061 (17)	0.00984 (19)	0.00590 (17)
C1	0.0281 (9)	0.0294 (8)	0.0244 (9)	−0.0006 (6)	0.0044 (7)	−0.0010 (6)
N1	0.0253 (7)	0.0278 (7)	0.0207 (7)	0.0002 (5)	0.0040 (6)	−0.0019 (5)
C2	0.0278 (9)	0.0374 (9)	0.0196 (8)	0.0015 (7)	−0.0028 (7)	−0.0012 (7)
C3	0.0491 (13)	0.0528 (14)	0.0459 (13)	0.0199 (11)	−0.0142 (11)	−0.0072 (11)
C4	0.0473 (12)	0.0664 (15)	0.0206 (9)	−0.0050 (11)	0.0045 (9)	0.0018 (9)
C5	0.0424 (11)	0.0575 (14)	0.0308 (10)	−0.0171 (10)	−0.0016 (9)	−0.0067 (9)

Geometric parameters (Å, º)

Ga1—C1	2.0351 (18)	C3—H3A	0.95 (3)
Ga1—Cl2	2.1441 (6)	C3—H3B	0.88 (4)
Ga1—Cl3	2.1481 (5)	C3—H3C	1.00 (3)
Ga1—Cl1	2.1589 (5)	C4—H4A	0.92 (3)
C1—N1	1.133 (2)	C4—H4B	0.98 (3)
N1—C2	1.474 (2)	C4—H4C	0.96 (3)
C2—C3	1.512 (3)	C5—H5A	0.96 (3)
C2—C4	1.513 (3)	C5—H5B	0.97 (3)
C2—C5	1.521 (3)	C5—H5C	0.94 (3)

C1—Ga1—Cl2	107.40 (5)	H3A—C3—H3B	103 (3)
C1—Ga1—Cl3	106.00 (5)	C2—C3—H3C	112.6 (17)
Cl2—Ga1—Cl3	112.78 (2)	H3A—C3—H3C	107 (2)
C1—Ga1—Cl1	104.90 (5)	H3B—C3—H3C	117 (3)
Cl2—Ga1—Cl1	113.07 (2)	C2—C4—H4A	107.3 (16)
Cl3—Ga1—Cl1	112.01 (2)	C2—C4—H4B	113.1 (14)
N1—C1—Ga1	179.26 (16)	H4A—C4—H4B	109 (2)
C1—N1—C2	179.35 (18)	C2—C4—H4C	111.2 (17)
N1—C2—C3	105.96 (15)	H4A—C4—H4C	110 (2)
N1—C2—C4	106.28 (15)	H4B—C4—H4C	106 (2)
C3—C2—C4	113.1 (2)	C2—C5—H5A	108.1 (15)
N1—C2—C5	106.27 (15)	C2—C5—H5B	114.0 (17)
C3—C2—C5	112.49 (19)	H5A—C5—H5B	107 (2)
C4—C2—C5	112.12 (18)	C2—C5—H5C	108.3 (18)
C2—C3—H3A	106.9 (18)	H5A—C5—H5C	109 (2)
C2—C3—H3B	109 (2)	H5B—C5—H5C	111 (2)