Crystal Structure of the Aziridinylgallane Trimer †

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Crystals of the title compound are orthorhombic, a = 18·364(5), b = 10·823(3), c = 7·114(2) Å, Z = 4 (trimeric units), space group *Pnma*. The structure was determined from diffractometer data by Patterson and Fourier syntheses, and refined by full-matrix least-squares methods to R 0·077 for 252 observed reflexions. The six-membered $(Ga–N)_3$ ring is in the chair conformation, torsion angles 59—61°, and lies on a crystallographic mirror plane. The mean dimensions are Ga–N 1·97, N–C 1·55, C–C 1·55 Å; N–Ga–N 100, Ga–N–Ga 121, Ga–N–C 116°, angles in three-membered rings *ca.* 60°.

THE structure of the aziridinylgallane trimer, $[(CH_2)_2NGaH_2]_3$, has been determined as part of a study of cyclogallazanes.[1,2]

EXPERIMENTAL

The reaction of aziridine with an equimolar amount of trimethylamine–gallane at room temperature yields the (*N*-ethylene)cyclogallata-azoniane, $[(CH_2)_2NGaH_2]_3$. Sublimation of the compound in sealed capillaries under low pressures of nitrogen gave colourless crystals suitable for an *X*-ray crystallographic study.

Crystal Data.—$C_6H_{18}Ga_3N_3$, M = 341·4, orthorhombic, a = 18·364(5), b = 10·823(3), c = 7·114(2) Å, U = 1413·9 Å^3, D_m not measured because of instability of the crystals, Z = 4, D_c = 1·60, $F(000)$ = 672. Mo-K_α radiation, λ = 0·7107 Å; μ(Mo-K_α) = 59 cm^{-1}. Space group *Pnma* (D_{2h}^{16}), from absent spectra and structure analysis.

Intensity data were measured on a Datex-automated General Electric XRD 6 diffractometer by our usual methods.[3] Of 732 reflexions, only 252 were considered observed, having $I > 3\sigma(I)$; no absorption correction was made.

The structure was determined from the Patterson map, which could be interpreted in terms of space group *Pnma*, and electron-density syntheses. Refinement was by full-matrix least-squares methods, with the scattering factors of ref. 4 and anisotropic thermal parameters for gallium and isotropic parameters for nitrogen and carbon. The function minimized was $\Sigma w(F_o - F_c)^2$, with $\sqrt{w} = 1$ when $|F_o| \leqslant 100$, $\sqrt{w} = 100/|F_o|$ when $|F_o| > 100$, $\sqrt{w} = 0·1$ for unobserved reflexions (which were given their measured $|F_o|$ values). The final R was 0·077 for the 252 observed reflexions. Measured and calculated structure factors are listed in Supplementary Publication No. SUP 20403 (7 pp., 1 microfiche).‡

TABLE 1

Final positional (fractional $\times 10^4$) and thermal parameters (components of the vibration tensors, U in Å^2 $\times$ 10^2), with standard deviations in parentheses

	x	y	z	U_{iso}
Ga(1)	0851(1)	0931(3)	1828(5)	—
Ga(2)	2345(2)	2500	3376(9)	—
N(1)	0374(12)	2500	2209(36)	4·8(8)
N(2)	1659(9)	1092(18)	3665(29)	7·4(6)
C(1)	1951(15)	−0098(27)	4412(39)	9·6(10)
C(2)	1499(15)	0735(28)	5656(42)	9·8(10)
C(3)	−0222(18)	2500	3711(51)	6·8(11)
C(4)	−0500(19)	2500	1585(62)	8·9(11)

	U_{11}	U_{22}	U_{33}
Ga(1)	5·81(17)	7·42(18)	10·11(24)
Ga(2)	4·73(26)	10·44(36)	10·71(45)

	U_{12}	U_{13}	U_{23}
Ga(1)	−0·24(16)	0·58(29)	−1·39(29)
Ga(2)	0	1·54(36)	0

Final positional parameters are given in Table 1. Atoms are in positions 4(*c*) and 8(*d*), the molecule possessing a crystallographic mirror plane which contains one gallium atom and one aziridine ring. Bond lengths and valency angles are in Table 2.

† Preliminary publication: W. Harrison, A. Storr, and J. Trotter, *Chem. Comm.*, 1971, 1101.

‡ For details see Notice to Authors No. 7 in *J. Chem. Soc.* (*A*), 1970, Issue No. 20 (items less than 10 pp. are sent as full size copies).

[1] A. Storr, *J. Chem. Soc.* (*A*), 1968, 2605.
[2] A. Storr and A. D. Penland, *J. Chem. Soc.* (*A*), 1971, 1237.
[3] *E.g.*, W. Harrison and J. Trotter, *J. Chem. Soc.* (*A*), 1971, 1607.
[4] 'International Tables for *X*-Ray Crystallography,' vol. III, Kynoch Press, Birmingham, 1962.

TABLE 2

Bond lengths (Å), and valency and torsion angles (deg.), with standard deviations in parentheses

(*d*) Bond distances

Ga(1)–N(1)	1·930(12)	N(2)–C(1)	1·494(31)
Ga(1)–N(2)	1·985(19)	N(2)–C(2)	1·498(28)
Ga(2)–N(2)	1·987(19)		
		C(3)–C(4)	1·596(49)
N(1)–C(3)	1·531(38)	C(1)–C(2)	1·513(35)
N(1)–C(4)	1·666(41)		

(*b*) Bond angles

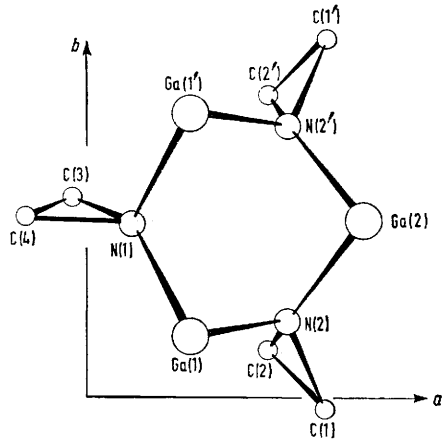
N(1)–Ga(1)–N(2)	99·7(9)	C(3)–N(1)–C(4)	59·7(20)
N(2)–Ga(1)–N(2′)	100·1(11)	C(1)–N(2)–C(2)	60·7(16)
Ga(1)–N(1)–Ga(1′)	123·3(12)	N(1)–C(3)–C(4)	64·3(20)
Ga(1)–N(2)–Ga(2)	118·2(11)	N(1)–C(4)–C(3)	55·9(19)
Ga(1)–N(1)–C(3)	115·0(8)	N(2)–C(1)–C(2)	59·7(16)
Ga(1)–N(1)–C(4)	113·6(8)	N(2)–C(2)–C(1)	59·5(16)
Ga(1)–N(2)–C(1)	115·3(15)		
Ga(1)–N(2)–C(2)	117·0(15)		
Ga(2)–N(2)–C(1)	118·1(15)		
Ga(2)–N(2)–C(2)	114·8(16)		

(*c*) Torsion angles

Ga(1′)–N(1)–Ga(1)–N(2)	−59·6
N(1)–Ga(1)–N(2)–Ga(2)	+58·7
Ga(1)–N(2)–Ga(2)–N(2′)	−61·0

DISCUSSION

The Figure shows the trimeric molecule viewed down *c*. The six-membered $(Ga–N)_3$ ring has a chair conformation, with torsion angles 59—61° (Table 2), and lies on a crystallographic mirror plane. The aziridine rings are perpendicular to the Ga–N–Ga planes. The mean dimensions are: Ga–N 1·97, N–C 1·55, C–C 1·55 Å; N–Ga–N 100, Ga–N–Ga 121, Ga–N–C 116°, angles in three-membered rings 60°. The mean Ga–N bond length (1·97 Å) is in good agreement with that in monomeric trimethylamine–gallane, 1·97 Å,[5] and is also close to the Al–N bond distances in similar trimeric aluminium compounds, $[MeNH,AlMe_2]_3$ 1·95,[6] and $[(CH_2)_2N,AlMe_2]_3$ 1·93 Å.[7] Bond angles in the ring are also in good agreement with those in these related molecules.



The molecular structure viewed along *c* [atom C(3) is displaced for clarity]

Intermolecular distances correspond to van der Waals interactions, the shortest contacts being Ga · · · C 3·96, N · · · C 4·10, and C · · · C 3·96 Å.

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