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2,4,6-Tris[2-(trimethylsilyl)ethynyl]-1,3,5-triazene: a novel precursor to C–N two-dimensional structures

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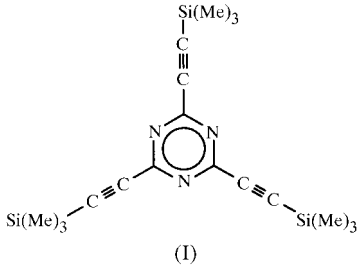
The title structure, $C_{18}H_{27}N_3Si_3$, consists of separate molecules arranged in sheets parallel to the *ab* plane. In each molecule, the central $(CN)_3$ ring, the ethynyl units, and the Si atoms lie in the same plane. Typical C—C and C—N bond distances are 1.197 (5) and 1.42 (4) Å, respectively. The angles inside the $(CN)_3$ ring range from 114 (3) to 125 (3)° and the acetylenic branches are nearly linear.

Comment

There has been considerable ongoing research toward the synthesis of novel forms of carbon and related two-dimensional polymers that contain heteroatoms (N, B, S) in the graphitic structure (Nieman & Whitesides, 1988; Callstrom *et al.*, 1990). We are particularly interested in the preparation of similar low-dimensional hydrogen-free solids that are electrically conductive, stable in ambient atmosphere, and refractory. An example of a target material involves $(CN)_3$ aromatic rings linked by ethynyl units in a sheet-like structure. Efforts to prepare trimethylsilyl-substituted ethynyl precursors of such materials led us to the synthesis of 2,4,6-tris[2-(trimethylsilyl)ethynyl]-1,3,5-triazene, (I).



(I)

The structure consists of individual molecules packed into the unit cell with no unusually short intermolecular contacts. The packing is in sheets parallel to the *ab* plane. Each molecule is planar with the exception of its methyl groups. The maximum deviation from the least-squares plane through the $(CN)_3$ central framework and the acetylenic branches including the Si atoms is 0.1 Å. The molecule is, however, slightly distorted; the ring angles deviate from 120° at N1—C7—N2 [125.5 (3)°] and opposite to this at C1—N3—C13 [114.0 (3)°]. This distortion presumably originates from packing effects in the solid state. The distances in the central ring and the acetylenic branches appear to be normal.

Experimental

The title compound, (I), was prepared by reacting trifluorotriazene with lithium trimethylsilylacetylide in hexane at 195 K (Kouvetakis *et al.*, 1994). Clear colourless crystals were grown by sublimation in a temperature gradient under high vacuum.

Crystal data

$C_{18}H_{27}N_3Si_3$	D_x = 1.029 Mg m^{-3}
M_r = 369.70	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 24 reflections
a = 11.265 (2) Å	θ = 13–14°
b = 19.946 (4) Å	μ = 0.203 mm^{-1}
c = 10.927 (2) Å	T = 167 (2) K
β = 103.65 (3)°	Cleaved fragment, colourless
V = 2385.9 (8) Å^3	0.52 × 0.27 × 0.25 mm
Z = 4	

Data collection

Enraf–Nonius CAD-4 diffractometer	2716 reflections with $I > 2\sigma(I)$
θ–2θ scans	θ_{max} = 24.98°
Absorption correction: ψ scan (North *et al.*, 1968)	$h = 0 \rightarrow 13$
T_{min} = 0.852, T_{max} = 0.998	$k = 0 \rightarrow 23$
4194 measured reflections	$l = -12 \rightarrow 12$
4194 independent reflections	3 standard reflections
	frequency: 60 min
	intensity decay: none

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0559P)^2 + 2.2202P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.060	$(\Delta/\sigma)_{max}$ = 0.007
$wR(F^2)$ = 0.158	$\Delta\rho_{max}$ = 0.36 e Å^{-3}
S = 1.066	$\Delta\rho_{min}$ = −0.36 e Å^{-3}
4194 reflections	Extinction correction: *SHELXL*97
218 parameters	Extinction coefficient: 0.0046 (9)
H-atom parameters constrained	

Data collection, cell refinement and data reduction: *CAD*-4 *Software* (Enraf–Nonius, 1989); program(s) used to solve and refine structure, and prepare material for publication: *SHELXTL* (Sheldrick, 1997).

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