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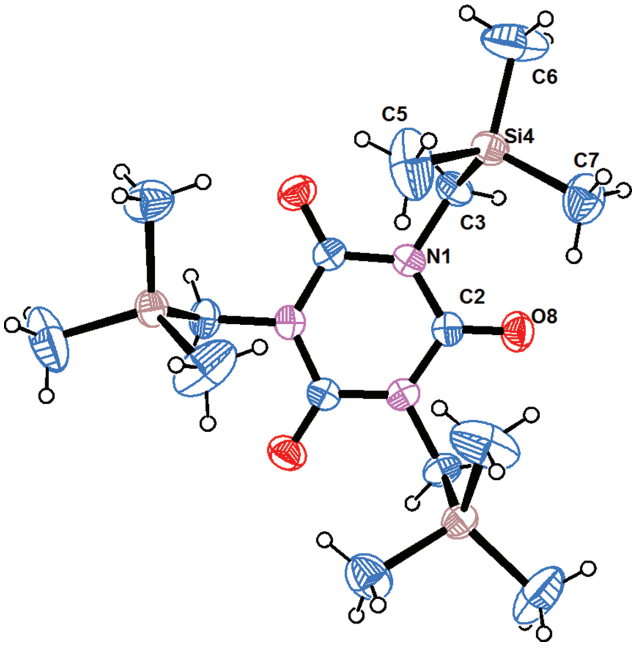
Crystal structure of 1,3,5-tris((trimethylsilyl) methyl)-1,3,5-triazinane-2,4,6-trione, $C_{15}H_{33}N_3O_3Si_3$



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Abstract

$C_{15}H_{33}N_3O_3Si_3$, trigonal, $R\bar{3}$ (no. 148), $a = 17.8622(4)$ Å, $c = 12.5153(3)$ Å, $V = 3458.13(14)$ Å^3, $Z = 6$, $R_{gt}(F) = 0.0689$, $wR_{ref}(F^2) = 0.1876$, $T = 293$ K.

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The molecular structure of the title compound is shown in the figure. Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colourless prism
Size:	0.65 × 0.15 × 0.12 mm
Wavelength:	Cu $K\alpha$ radiation (1.54184 Å)
μ:	20.3 cm^{-1}
Diffractometer, scan mode:	SuperNova, EosS2, ω scans
$2\theta_{max}$, completeness:	144.8°, 98.0%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	4196, 1493, 0.025
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1340
$N(param)_{refined}$:	76
Programs:	CrysAlis^{PRO} [1], SHELX [2], OLEX2 [3, 4]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	x	y	z	U_{iso}*/U_{eq}
N1	0.41074(11)	0.74037(11)	0.30262(13)	0.0496(5)
C2	0.41031(13)	0.66299(14)	0.30298(15)	0.0496(5)
C3	0.49565(15)	0.82104(16)	0.30438(18)	0.0602(6)
H3a	0.48916(15)	0.86650(16)	0.27103(18)	0.0722(7)*
H3b	0.53575(15)	0.81239(16)	0.26111(18)	0.0722(7)*
Si4	0.54506(4)	0.85945(4)	0.44120(5)	0.0592(3)
C5	0.4592(3)	0.8372(4)	0.5377(3)	0.132(2)
H5a	0.428(2)	0.865(3)	0.515(2)	0.199(3)*
H5b	0.4202(17)	0.7760(4)	0.542(3)	0.199(3)*
H5c	0.4842(4)	0.858(3)	0.6067(9)	0.199(3)*
C6	0.6271(4)	0.9754(3)	0.4254(4)	0.143(2)
H6a	0.6623(19)	0.9826(5)	0.364(2)	0.214(3)*
H6b	0.5985(4)	1.0084(5)	0.416(4)	0.214(3)*
H6c	0.663(2)	0.9953(8)	0.4881(17)	0.214(3)*
C7	0.6024(3)	0.8027(3)	0.4865(3)	0.0963(11)
H7a	0.5624(5)	0.7417(3)	0.490(2)	0.1444(16)*
H7b	0.6480(13)	0.8139(17)	0.4373(14)	0.1444(16)*
H7c	0.6264(18)	0.8232(15)	0.5562(11)	0.1444(16)*
O8	0.47660(12)	0.66013(14)	0.30308(16)	0.0719(6)

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Source of materials

The title compound was synthesized by the slow reaction of potassium cyanate (1 mol, 80 g) with (chloromethyl) trimethylsilane (1.3 mol, 150 g) in xylene (400 mL) at 363 K for 5 h. After cooling to room temperature, the reacted

mixture was filtered and organic solvent was gradually removed by evaporation. Colourless prism-shaped single crystals suitable for X-ray diffraction studies were collected from the filtrate.

Experimental details

All H atoms were placed in idealized positions and refined as riding atoms. The U_{iso} values were constrained to be $1.5U_{eq}$ of the carbon atom for methyl H atoms and $1.2U_{eq}$ for the methylene H atoms.

Comment

Organosilane has many uses in organic chemistry [5–7] and polymer chemistry [8], from the most frequently employed protecting groups to radicals, and the rapid developments of functionalized organosilane extend its applications to organometallic chemistry [9], materials science [10], and separation science [11]. Cyanuric acid and its derivatives are industrially useful chemicals, and often used as precursor or a component of bleaches, disinfectants, and herbicides [12, 13]. The combination of cyanuric acid and organosilane has a potential application in the curing agents for epoxy resin.

The title compound crystallizes in the trigonal system with the space group $R\bar{3}$, and has one 3-fold rotation axis (*cf*. the figure). The central ring is nearly planar to which the (trimethylsilyl)methyl side chain are linked to N atoms. All three side chains are on the same side of the ring, forming a flattened chair-like six-membered structure. This three-legged chair also appears in other similar heterocyclic compound such as 1,3,5-triglycidyl-triazinetrione [14], and 1,3,5-trimethoxy-1,3,5,2,4,6-trithiatriazine [15]. The bond lengths and angles of the title compound are within the expected ranges and the four C—Si bond distances are slightly different and fall in the range from 1.833 to 1.891 Å. In this crystal structure, the molecules are loosely packed only by *van der Waals* interactions.

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