Crystal structure of 1-trimethylsilyloxy-4,4-dimethylcyclohexane-*r*-1,*t*-3-dicarbonitrile, (CH₃)₂(CN)₂[OSi(CH₃)₃]C₆H₇

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Table 1. Parameters used for the X-ray data collection

Crystal:	colorless prism, size 0.5 x 0.8 x 0.3 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	1.40 cm ⁻¹
Diffractometer:	Siemens P3
Scan mode:	Wyckoff
Temperature:	293 K
$2\theta_{\max}$:	55°
$N(hkl)_{\text{unique}}$:	3520
Criterion for F_o :	$F_o > 3 \sigma(F_o)$
$N(\text{param})_{\text{refined}}$:	155
Program:	SHELXTL-plus

Table 2. Final atomic coordinates and displacement parameters (in Å²)

Atom	Site	x	y	z	U_{iso}
H(2A)	2i	0.4035(2)	0.5993(2)	0.7301(3)	0.08
H(2B)	2i	0.3456(2)	0.6878(2)	0.8537(3)	0.08
H(3)	2i	0.5722(2)	0.8390(2)	0.9403(3)	0.08
H(5A)	2i	0.7075(2)	0.8250(2)	0.5485(3)	0.08
H(5B)	2i	0.6501(2)	0.9127(2)	0.6742(3)	0.08
H(6A)	2i	0.4802(2)	0.6777(2)	0.4494(3)	0.08
H(6B)	2i	0.4674(2)	0.8110(2)	0.4123(3)	0.08
H(12A)	2i	-0.1001(3)	0.5638(3)	0.1659(4)	0.08
H(12B)	2i	-0.0658(3)	0.5264(3)	0.3596(4)	0.08
H(12C)	2i	0.0166(3)	0.4938(3)	0.2133(4)	0.08
H(13A)	2i	0.1233(3)	0.7804(3)	0.0702(4)	0.08
H(13B)	2i	0.2285(3)	0.6981(3)	0.1085(4)	0.08
H(13C)	2i	0.2744(3)	0.8409(3)	0.2106(4)	0.08
H(14A)	2i	-0.0287(3)	0.8477(4)	0.3517(5)	0.08
H(14B)	2i	0.1177(3)	0.9100(4)	0.5009(5)	0.08
H(14C)	2i	-0.0052(3)	0.8025(4)	0.5408(5)	0.08
H(41A)	2i	0.6018(3)	0.5748(3)	0.6390(4)	0.08
H(41B)	2i	0.7187(3)	0.5883(3)	0.8204(4)	0.08
H(41C)	2i	0.7657(3)	0.6354(3)	0.6491(4)	0.08
H(42A)	2i	0.8570(3)	0.8031(3)	1.0097(4)	0.08
H(42B)	2i	0.8218(3)	0.9253(3)	0.9543(4)	0.08
H(42C)	2i	0.9018(3)	0.8540(3)	0.8394(4)	0.08

Source of material: The title compound was prepared by the addition of trimethylsilyl cyanide and 6,6-dimethyl-3-oxo-1-cyclohexanecarbonitrile in dichloromethane solution in the presence of a small amount of trimethylsilyl trifluoromethanesulfonate as catalyst. Distillation of the crude product at 323 K bath temperature / 10^{-2} Torr gave a colorless oil (94% yield) which afforded coarse, colorless crystals, mp 316 K – 317 K on treatment with diethyl ether (see ref. 1).

The structure of the compound reveals the *trans* position of the nitrile groups, which results from a highly diastereoselective axial attack of the cyanide at the carbonyl group of the precursor.

C₁₃H₂₂N₂OSi, triclinic, $P\bar{1}$ (No. 2), $a=9.943(3)$ Å, $b=11.053(5)$ Å, $c=7.566(2)$ Å, $\alpha=97.23(3)^\circ$, $\beta=101.83(2)^\circ$, $\gamma=105.62(3)^\circ$, $V=769.2$ Å³, $Z=2$, $R(F)=0.052$, $R_w(F)=0.055$.

Table 3. Final atomic coordinates and displacement parameters (in Å²)

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Si	2i	0.11522(7)	0.71106(6)	0.35031(9)	0.0485(3)	0.0577(4)	0.0517(4)	0.0133(3)	0.0075(3)	0.0108(3)
O	2i	0.2352(2)	0.6814(1)	0.5120(2)	0.0460(8)	0.0495(8)	0.0545(9)	0.0041(6)	0.0031(7)	0.0125(7)
C(1)	2i	0.3760(2)	0.7536(2)	0.6185(3)	0.048(1)	0.040(1)	0.045(1)	0.0085(8)	0.0055(8)	0.0066(8)

Table 3. (Continued)

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
C(2)	2i	0.4121(2)	0.6864(2)	0.7782(3)	0.050(1)	0.049(1)	0.046(1)	0.0130(9)	0.0127(9)	0.0138(9)
C(3)	2i	0.5671(2)	0.7520(2)	0.8971(3)	0.058(1)	0.043(1)	0.041(1)	0.0147(9)	0.0059(9)	0.0041(8)
C(4)	2i	0.6834(2)	0.7604(2)	0.7860(3)	0.048(1)	0.062(1)	0.052(1)	0.009(1)	0.0055(9)	0.007(1)
C(5)	2i	0.6416(2)	0.8258(2)	0.6250(3)	0.051(1)	0.068(1)	0.056(1)	0.001(1)	0.010(1)	0.016(1)
C(6)	2i	0.4875(2)	0.7627(2)	0.5060(3)	0.051(1)	0.061(1)	0.046(1)	0.006(1)	0.0095(9)	0.014(1)
C(10)	2i	0.3780(3)	0.8853(2)	0.6942(3)	0.073(2)	0.051(1)	0.054(1)	0.021(1)	-0.001(1)	0.005(1)
N(11)	2i	0.3835(3)	0.9870(2)	0.7533(4)	0.132(2)	0.063(1)	0.089(2)	0.044(2)	-0.006(2)	-0.005(1)
C(12)	2i	-0.0255(3)	0.5551(3)	0.2618(4)	0.061(2)	0.087(2)	0.082(2)	-0.002(1)	-0.006(1)	0.024(2)
C(13)	2i	0.1949(3)	0.7641(3)	0.1624(4)	0.076(2)	0.106(2)	0.067(2)	0.012(2)	0.009(1)	0.034(2)
C(14)	2i	0.0406(3)	0.8321(4)	0.4477(5)	0.083(2)	0.137(3)	0.098(2)	0.062(2)	0.002(2)	-0.007(2)
C(30)	2i	0.5962(3)	0.6876(2)	1.0537(3)	0.068(1)	0.060(1)	0.045(1)	0.021(1)	0.009(1)	0.007(1)
N(31)	2i	0.6214(3)	0.6391(2)	1.1745(3)	0.101(2)	0.086(2)	0.056(1)	0.037(1)	0.012(1)	0.023(1)
C(41)	2i	0.6932(3)	0.6273(3)	0.7172(4)	0.076(2)	0.090(2)	0.075(2)	0.041(2)	0.022(1)	0.010(1)
C(42)	2i	0.8298(3)	0.8432(3)	0.9084(4)	0.056(2)	0.106(2)	0.074(2)	0.000(1)	-0.005(1)	0.015(2)

References

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