Crystal structure of *catena*-octachlorotrisiloxane, $Si_3O_2Cl_8$

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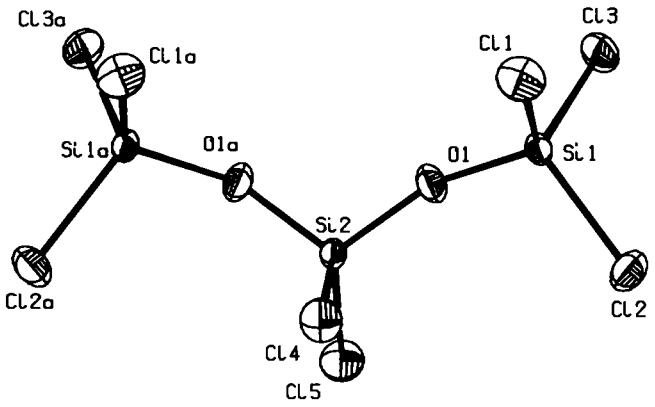

Abstract

$Cl_8O_2Si_3$, orthorhombic, *Pnma* (No. 62), $a = 10.669(1)$ Å, $b = 20.317(3)$ Å, $c = 6.183(1)$ Å, $V = 1340.4$ Å^3, $Z = 4$, $R_{gt}(F) = 0.023$, $wR_{obs}(F^2) = 0.055$, $T = 90$ K.

Source of material

The preparation method for chlorosiloxanes $Si_xO_yCl_z$ is described in the literature [1]. $Si_3O_2Cl_8$ can be isolated and purified by fractional destillation. The compound is highly moisture sensitive.

Discussion

High temperature reaction between siliconchloride ($SiCl_4$) and oxygen leads to SiO_2. At about 1300 K chlorosiloxanes like $Si_3O_2Cl_8$ are formed as intermediates during this reaction.
The molecular structure of these compounds are of particular interest regarding the pathway from molecular $SiCl_4$ to solid SiO_2. $Si_3O_2Cl_8$ shows the expected chain structure with bridging oxygen atoms.

Table 1. Data collection and handling.

Crystal:	colourless needle, size 0.2 x 0.2 x 1.0 mm
Wavelength:	Ag K_α radiation (0.56086 Å)
μ:	9.65 cm^{-1}
Diffractometer, scan mode:	Syntex $P2_1$, ω
$2\theta_{max}$:	52.14°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5622, 2769
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2266
$N(param)_{refined}$:	64
Programs:	SHELXS-86 [2], SHELXL-93 [3], PLATON [4]

References

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2. Sheldrick, G. M.: SHELXS-86. A program for crystal structure determination, University of Göttingen, Germany 1990.
3. Sheldrick, G. M.: SHELXL-93. A program for refining crystal structures, University of Göttingen, Germany1993.
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Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Si(1)	8*d*	0.91472(3)	0.38581(1)	0.11360(4)	0.0195(1)	0.0133(1)	0.0187(1)	–0.00166(9)	–0.00075(9)	–0.00163(8)
Si(2)	4*c*	0.79252(3)	1/4	0.02150(6)	0.0160(2)	0.0120(2)	0.0186(2)	0	0.0000(1)	0
Cl(1)	8*d*	1.07992(3)	0.38149(2)	–0.04216(5)	0.0248(1)	0.0437(2)	0.0335(1)	–0.0037(1)	0.0086(1)	–0.0005(1)
Cl(2)	8*d*	0.79685(3)	0.44556(2)	–0.04713(5)	0.0414(2)	0.0256(1)	0.0314(1)	0.0109(1)	–0.0096(1)	0.0010(1)
Cl(3)	8*d*	0.94030(3)	0.41963(2)	0.41488(4)	0.0367(1)	0.0267(1)	0.0221(1)	–0.0011(1)	–0.0049(1)	–0.00781(9)
Cl(4)	4*c*	0.81927(4)	1/4	–0.30012(6)	0.0388(2)	0.0321(2)	0.0189(2)	0	0.0026(1)	0
Cl(5)	4*c*	0.60814(4)	1/4	0.08485(8)	0.0170(1)	0.0348(2)	0.0437(2)	0	0.0054(1)	0
O(1)	8*d*	0.85557(8)	0.31359(4)	0.1295(1)	0.0306(4)	0.0160(3)	0.0272(3)	–0.0063(3)	–0.0028(3)	–0.0011(3)

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