Crystal structure of catena-octachlorotrisiloxane, $\text{Si}_3\text{O}_2\text{Cl}_8$

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Table 1. Data collection and handling.

Crystal:	colourless needle, size 0.2 × 0.2 × 1.0 mm
Wavelength:	Ag $K\alpha$ radiation (0.56086 Å)
μ :	9.65 cm ⁻¹
Diffractometer, scan mode:	Syntex P2 ₁ , ω
$2\theta_{\text{max}}$:	52.14°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	5622, 2769
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2266
$N(\text{param})_{\text{refined}}$:	64
Programs:	SHELXS-86 [2], SHELXL-93 [3], PLATON [4]

Abstract

$\text{Cl}_8\text{O}_2\text{Si}_3$, orthorhombic, $Pnma$ (No. 62), $a = 10.669(1)$ Å, $b = 20.317(3)$ Å, $c = 6.183(1)$ Å, $V = 1340.4$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.023$, $wR_{\text{obs}}(F^2) = 0.055$, $T = 90$ K.

Source of material

The preparation method for chlorosiloxanes $\text{Si}_x\text{O}_y\text{Cl}_z$ is described in the literature [1]. $\text{Si}_3\text{O}_2\text{Cl}_8$ can be isolated and purified by fractional distillation. 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 $\text{Si}_3\text{O}_2\text{Cl}_8$ 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 . $\text{Si}_3\text{O}_2\text{Cl}_8$ shows the expected chain structure with bridging oxygen atoms.

References

1. Quellhorst, H.; Wilkening, A.; Binnewies, M.: Trennung und massenspektrometrische Charakterisierung von Perchlorsiloxanen. *Z. Anorg. Allg. Chem.* **623** (1997) 1871-1874.
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, Germany 1993.
4. Spek, A. L.: PLATON, an integrated tool for the analysis of the results of a single crystal structure determination. *Acta Crystallogr. Suppl.* **A46** (1990) C-34.

Table 2. 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(1)	8d	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)	4c	0.79252(3)	1/4	0.02150(6)	0.0160(2)	0.0120(2)	0.0186(2)	0	0.0000(1)	0
Cl(1)	8d	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)	8d	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)	8d	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)	4c	0.81927(4)	1/4	-0.30012(6)	0.0388(2)	0.0321(2)	0.0189(2)	0	0.0026(1)	0
Cl(5)	4c	0.60814(4)	1/4	0.08485(8)	0.0170(1)	0.0348(2)	0.0437(2)	0	0.0054(1)	0
O(1)	8d	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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