Crystal structure of decachloro-bicyclo[5.5.1]heptaoxatridecasilane, $Si_6O_7Cl_{10}$

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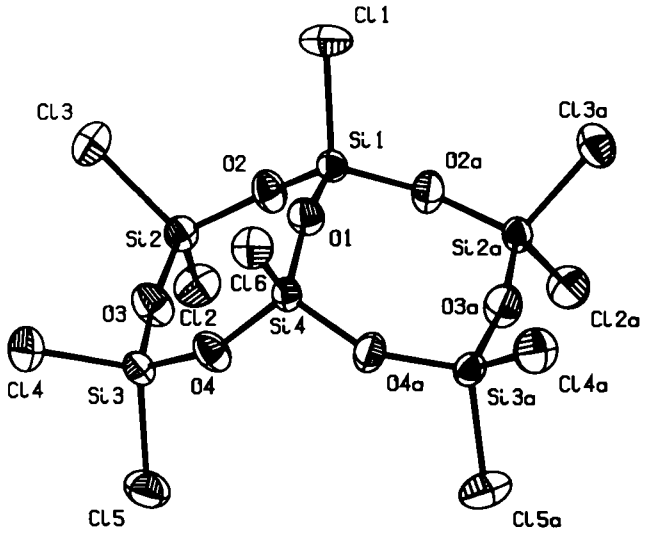

Abstract

$Cl_{10}O_7Si_6$, monoclinic, $P12_1/m1$ (No. 11), $a = 6.094(1)$ Å, $b = 20.676(4)$ Å, $c = 8.295(2)$ Å, $\beta = 99.47(3)°$, $V = 1030.9$ Å^3, $Z = 2$, $R_{gt(F)} = 0.039$, $wR_{obs(F^2)} = 0.114$, $T = 203$ K.

Source of material

The general preparation method is described in [1]. Solid $Si_6O_7Cl_{10}$ could be isolated in small amounts by sublimation of one destillate fraction.

Discussion

$Si_6O_7Cl_{10}$ is a structural link between the eight-membered ring compound $Si_4O_4Cl_8$ [2] and the cubane like silsesquioxane $Si_8O_{12}Cl_8$ [3]. $Si_6O_7Cl_{10}$ marks one step in the reaction sequence between $SiCl_4$ and O_2 leading to amorphous SiO_2. This isomer of $Si_6O_7Cl_{10}$ is a bicyclic compound consisting of two edge connected eight-membered rings.

Table 1. Data collection and handling.

Crystal:	colourless, irregular shape, size 0.2 × 0.2 × 0.2 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	17.20 cm^{-1}
Diffractometer, scan mode:	Stoe IPDS, 100 exposures, $\Delta\varphi = 0.8°$
$2\theta_{max}$:	48°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	3659, 1341
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1289
$N(param)_{refined}$:	112
Programs:	SHELXS-86 [4], SHELXL-93 [5], PLATON [6]

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)	2e	–0.2219(2)	1/4	0.3737(1)	0.0331(6)	0.0221(5)	0.0215(6)	0	0.0054(5)	0
Si(2)	4f	–0.0008(1)	0.38307(3)	0.4467(1)	0.0327(5)	0.0249(5)	0.0274(5)	–0.0032(2)	0.0090(4)	0.0013(2)
Si(3)	4f	–0.1247(1)	0.38101(3)	0.80369(9)	0.0332(5)	0.0251(5)	0.0252(5)	–0.0040(2)	0.0070(3)	–0.0043(2)
Si(4)	2e	–0.3613(2)	1/4	0.7220(1)	0.0313(6)	0.0214(5)	0.0243(6)	0	0.0086(5)	0
O(1)	2e	–0.3363(5)	1/4	0.5335(3)	0.044(2)	0.034(1)	0.026(2)	0	0.013(1)	0
O(2)	4f	–0.0678(4)	0.31279(8)	0.3742(3)	0.047(1)	0.026(1)	0.039(1)	–0.0074(8)	0.015(1)	–0.0026(8)
O(3)	4f	–0.0501(4)	0.38869(9)	0.6292(3)	0.057(2)	0.042(1)	0.033(1)	–0.0120(8)	0.020(1)	–0.0043(8)
O(4)	4f	–0.2468(4)	0.31289(8)	0.8115(3)	0.054(2)	0.029(1)	0.032(1)	–0.0132(8)	0.012(1)	–0.0044(7)
Cl(1)	2e	–0.4572(2)	1/4	0.1743(2)	0.0558(8)	0.0728(8)	0.0298(7)	0	–0.0095(5)	0
Cl(2)	4f	0.3241(1)	0.39722(4)	0.4443(1)	0.0308(5)	0.0501(5)	0.0506(6)	–0.0030(3)	0.0077(4)	0.0055(3)
Cl(3)	4f	–0.1772(1)	0.45067(3)	0.3077(1)	0.0403(5)	0.0371(5)	0.0521(6)	0.0057(3)	0.0073(4)	0.0110(3)
Cl(4)	4f	–0.3320(1)	0.45268(3)	0.8382(1)	0.0510(6)	0.0401(5)	0.0535(6)	0.0124(3)	0.0073(4)	–0.0071(3)
Cl(5)	4f	0.1435(1)	0.38397(4)	0.9801(1)	0.0401(5)	0.0621(6)	0.0456(6)	0.0024(3)	–0.0051(4)	–0.0161(3)
Cl(6)	2e	–0.6872(2)	1/4	0.7383(1)	0.0319(6)	0.0467(6)	0.0425(7)	0	0.0122(5)	0

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