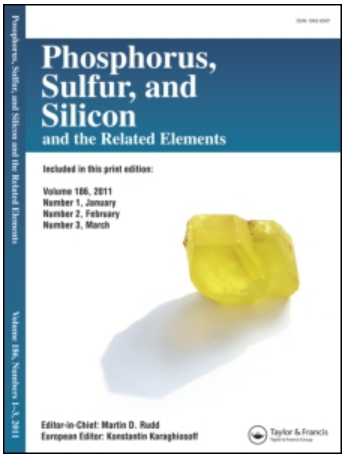

Phosphorus, Sulfur, and Silicon and the Related Elements
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713618290

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To cite this Article Yoo, Bok Ryul , Kim, Jeong Hyun , Lee, Myong Euy and Jung, Il Nam(2001) 'Synthesis and Characterization of Stable (Triphenylmethyl)Silanetriol', Phosphorus, Sulfur, and Silicon and the Related Elements, 169: 1, 227 — 230
To link to this Article: DOI: 10.1080/10426500108546630
URL: http://dx.doi.org/10.1080/10426500108546630

Phosphorus, Sulfur and Silicon, 2001, Vol. 169, pp. 227-230
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Synthesis and Characterization of Stable (Triphenylmethyl)Silanetriol

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The (triphenylmethyl)silanetriol (**1**) was obtained in good yield by the hydrolysis of (triphenylmethyl)trichlorosilane, prepared by the Friedel-Crafts alkylation of benzene with (trichloromethyl)trichlorosilane in the presence of aluminum chloride, with ice. The single crystal of [**1**·THF] for X-ray diffraction was grown as colorless in a saturated THF solution. The solid structure of [**1**·THF] was disclosed to be two-dimensional array in head to head hydrogen-bonding fashion.

Keywords: Silanetriol; (Triphenylmethyl)silanetriol; Siloxane

INTRODUCTION

Much attention has been paid to synthetic approaches to silanetriols [1] that can be useful precursors to silsesquioxanes, ladder polymers, and metallasiloxanes [2]. Stable silanetriol was first isolated and characterized by X-ray diffraction study in 1982 [1a]. Then, several stable silanetriols have been isolated and tried to react with MXn (M = main group metals, transition metals) metal halides. Such silanetriols can be used as models for studies on the building block of silica [1,3a], the surface between the inorganic world and biological world, and heterogeneous silica-supported transition metal catalysts [3b].

RESULTS AND DISCUSSION

The stable (triphenylmethyl)silanetriol (**1**) was obtained in good yield by the hydrolysis of (triphenylmethyl)trichlorosilane with ice (eq 1).

$$Ph_3CSiCl_3 + H_2O\,(ice) \longrightarrow \underset{\mathbf{1}}{Ph_3CSi(OH)_3} \quad (1)$$

The single crystal of [**1**•THF] for X-ray diffraction study was grown as colorless in a saturated THF solution. Crystal data for [**1**•THF] are as follows: $C_{23}H_{26}O_4Si$ (fw =394.54), triclinic, a=8.834(2) Å [α=85.46(2) °], b=9.854(2) Å [β=86.52(1)°], c=12.626(2) Å [γ=69.79(2)°], V= 1027.5(4) Å^3, λ=0.71073 Å, μ=0.140 mm^{-1}, Z=2, $d_{calcd.}$=1.270 g cm^{-3}, $R1$=0.0855, $wR2$=0.1997. The ORTEP plot of compound **1** is depicted in Fig 1, and selected bond lengths and bond angles in the typical rage for Si-C and Si-O units are listed in Table 1. The packing diagram of [**1**•THF] is shown in Fig 2. In Fig 2, the solid structure of [**1**•THF] was disclosed to be two-dimensional array in head to head hydrogen-bonding fashion. The oxygen-oxygen distances of the O-H-O units between silanetriol molecules and between the OH group of **1** and the

oxygen of THF range were 2.90-3.08 and 2.77 Å, respectively.

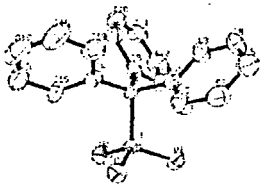

FIGURE 1 STRUCTURE OF 1

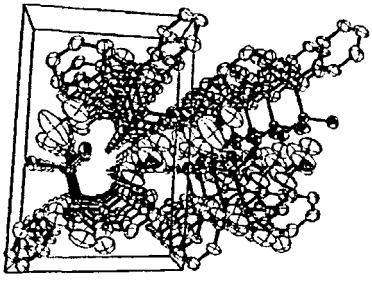

FIGURE 2 PACKING DIAGRAM OF 1•THF

TABLE 1 SELECTED BOND LENGTHS AND ANGLES

Bond Length (Å)		Angle (°)	
Si(1)-O(1)	1.621(6)	O(1)-Si(1)-O(2)	108.7(3)
Si(1)-O(2)	1.631(6)	O(1)-Si(1)-O(3)	109.8(3)
Si(1)-O(3)	1.627(6)	O(2)-Si(1)-O(3)	108.1(4)
Si(1)-C(1)	1.925(8)	C(1)-Si(1)-O(1)	110.2(3)
		C(1)-Si(1)-O(2)	110.3(3)
		C(1)-Si(1)-O(3)	109.8(3)

The reaction of compound **1** with MXn (M = main groups and transition metals), giving metallasilsesquioxanes is in progress.

EXPERIMENTAL SECTION

Aluminum chloride and benzene were purchased from Aldrich Chemical Co. and (trichloromethyl)trichlorosilanes from Gelest, Inc.

and used without purification except for benzene. Benzene was dried by the distillation from sodium wires prior to use. NMR spectra were recorded on a Varian Unity Plus 600 (FT, 600 MHz, ^{1}H) spectrometer in acetone-d_6 solvent. (Triphenylmethyl)trichlorosilane was obtained by the reaction of benzene with (trichloromethyl)trichlorosilane in the presence of aluminum chloride [4]. (Trichloromethyl)trichlorosilane was hydrolyzed with ice in THF to give (triphenylmethyl)silanetriol (**1**, 90%). ^{1}H NMR: δ 5.63 (s, 3H, OH), 7.12-7.27 (m, 15H, phenyl-H).

ACKNOWLEDGMENT

This research was supported financially by the Ministry of Science and Technology.

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