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Ch. Hartinger; A. A. Nazarov; T. H. Brehmer; G. Giester; M. Galanski; B. K. Keppler

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Synthesis and New Crystal Structure of 1,1,3,3-Tetramethyl-1,3-Disila-2-Oxa[3] Ferrocenophane

CH. HARTINGER^a, A. A. NAZAROV^a, T. H. BREHMER^b,
G. GIESTER^b, M. GALANSKI^a and B. K. KEPPLER^{a*}

^a*Institute of Inorganic Chemistry, University of Vienna, Waehringerstr. 42,
A-1090 Vienna, Austria and* ^b*Institute of Mineralogy and Crystallography,
University of Vienna, Althanstr. 14, A-1090 Vienna, Austria*

We prepared 1,1,3,3-tetramethyl-1,3-disila-2-oxa[3]ferrocenophane $\text{Fe}(\eta^5\text{-C}_5\text{H}_5)_2\text{-(SiCH}_3)_2\text{O}$ **1** via the reaction of 1,1'-dilithioferrocene TMEDA with dichlorodimethylsilane followed by hydrolysis. Crystals **1A** obtained from sublimation crystallize in the space group *P*-1 in analogy to the crystal structure recently published [1]. A new crystal structure **1B** of compound **1** could be obtained by slow evaporation of the solvent (CDCl_3) in the NMR tube. **1B** crystallizes in the orthorhombic space group *P*2₁2₁2₁.

Whereas so far there have only been reports on a mixture of 3 conformers, we have succeeded in isolating one single conformer.

Keywords: ferrocenophane; silane

INTRODUCTION

Many different ferrocenophanes have been synthesized in the last years. Most of them have been from the group of the all-carbon-bridged ferrocenophanes.

Today increasing attention is paid to heteroatom-bridged ferrocenophanes [2]. The synthesis of 1,1,3,3-tetramethyl-1,3-disila-2-oxa[3]ferrocenophane **1** has also been reported but we have developed

* Corresponding author.

a method of synthesizing **1** in rather short time and with very good yields. Whereas so far there have only been reports on a mixture of 3 conformers [1]. We have succeeded in isolating one single conformers.

Ferrocenophanes are used as precursors to polymers. A few experiments have been done to polymerise ferrocenophanes by temperature induced, by transition metal catalysed or by anionic ring opening polymerisation [3]. The polymers are expected to be a new class of materials with novel electrical, optical, magnetic or preceramic properties [4].

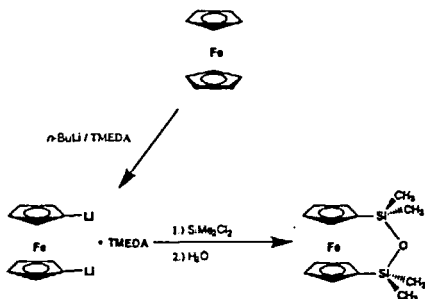


FIGURE 1: Synthesis of 1,1,3,3-tetramethyl-1,3-disila-2-oxa[3]ferrocenophane

SYNTHESIS OF 1,1,3,3-TETRAMETHYL-1,3-DISILA-2-OXA-[3]FERROCENOPHANE

To a solution of 2.0 ml dichlorodimethylsilane (1.88 g, 15 mmol) in 10 ml of dry *n*-hexane a suspension of 1.97 g of 1,1'-dilithioferrocene· tetramethylethylenediamine (6.2 mmol) in 40 ml of dry *n*-hexane was added under argon-atmosphere. This reaction mixture was allowed to stir for 72 hours, then it was filtered and poured on a mixture of crushed ice and water. The phases were separated and the organic phase was dried over Na_2SO_4 . The solvent was removed, purification was done by sublimation at 80°C and 4 mbar and yielded 1.98 g (58.1 %) of orange crystals.

CHARACTERISATION

The characterisation was done by ^1H - and ^{13}C -NMR-measurement, elemental analysis and X-ray diffraction.

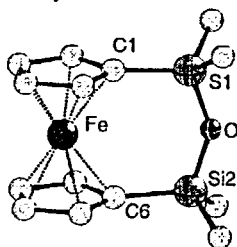


FIGURE 2: Molecular geometry of 1,1,3,3-tetramethyl-1,3-disila-2-oxa[3]ferrocenophane **1** in crystal structure **1B**

DISCUSSION

From crystal structure analysis we know that Structures **1A** and **1B** possess a ferrocene unit with each cyclo-pentadienyl ring linked by a Si-O-Si bridge (Figure 2). The molecular geometry of compound **1** is nearly the same in both structures. In structure **1A**, three structurally distinct molecules are found which differ slightly in the conformation, whereas in structure **1B** only one conformer of compound **1** exists.

Identification code		1A		1B
Crystal system		triclinic	triclinic	orthorhombic
Space group		P-1	P-1	P2 ₁ 2 ₁ 2 ₁
Unit cell	a [Å]	8.5893(14)	8.533(1)	8.498(2)
	b [Å]	15.703(2)	15.610(3)	11.389(2)
	c [Å]	19.089(4)	18.774(5)	15.448(3)
	α [°]	69.684(13)	70.68(2)	90
	β [°]	77.860(15)	77.94(2)	90
	γ [°]	74.889(12)	75.15(1)	90
crystallization solvent		n-hexane	from the crude oil	CDCl ₃
Temperature		110 K	110 K	110 K

TABLE 1: The data of the structures **1A** and **1B**

Acknowledgements

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