



1-[(N-Methyl-N-tritylamino)methyl]silatrane: Synthesis and structure



N.F. Lazareva*, I.V. Sterkhova, I.M. Lazarev, V.I. Smirnov

A. E. Favorsky Irkutsk Institute of Chemistry, Siberian Division of the Russian Academy of Science, 1, Favorsky Street, 664033 Irkutsk, Russian Federation

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ABSTRACT

The reaction of 1-(methylaminomethyl)silatrane with Ph_3CCl in the presence of base leads to the formation of 1-[(N-methyl-N-tritylamino)methyl]silatrane **1**. Recrystallization of this silatrane from benzene/heptane (10:1) led to the formation of the solvate complex $\text{Ph}_3\text{C}(\text{Me})\text{NCH}_2\text{Si}(\text{OCH}_2\text{CH}_2)_3\text{N}\cdot\text{C}_6\text{H}_6$, the molecular structure of which was determined by single crystal X-ray analysis.

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1. Introduction

The triphenylmethyl group Ph_3C , known as trityl (Trt), is a sterically bulky protecting group which is widely used for the synthesis of peptides, nucleotides and carbohydrates [1]. Among the well-studied derivatives of triphenylmethane, Ar_3CX compounds have attracted considerable interest, especially triphenylmethylamine along with many other related substances of the type $\text{Ar}_3\text{CNRR}'$. Triphenylmethylamine, as an ammonia equivalent, has been successfully applied in the synthesis of amines, amides and other novel compounds [2–10]. Triaryl functionalized N-phenyldiethanolamine derivatives have been used as building blocks to obtain molecular glasses with non-linear optical activity [11]. Crystal engineering research of $\text{N,N}'$ -ditritylureas, $\text{N,N}'$ -ditrityl amino amide and related compounds has shown that the trityl group's large size and its ability to promote crystallinity make it a valuable component of inclusion crystals [12–14]. An X-ray crystallographic study of salts between N-trityl amino acids and tert-butylamine showed that the steric bulk of the trityl group was essential for making a new host–guest hydrogen bonded network [15].

Introduction of silyl functionalities into organic amines along with the formation of the Si–C–N geminal fragment is of great interest in chemistry. The silyl group, in an α -position to a nitrogen atom, significantly decreases the oxidation potential of amines and increases their basicity as compared with their carbon analogs [16–19]. Such changes are responsible for the drastic difference

between the reactivity of α -silylamines and isostructural organic amines. The α -silylamines $\text{RR}'\text{NCH}_2\text{SiX}_3$ have unusual physical and chemical properties and widely apply as synthons in synthetic organic chemistry [20–22]. We have found that the α -silatranyl amines $\text{RR}'\text{NCH}_2\text{Si}(\text{OCH}_2\text{CH}_2)_3\text{N}$ exhibit enhanced reactivity with respect to polychloromethanes $\text{CH}_n\text{Cl}_{4-n}$ [23–26]. They react with alcohols and phenols in inert solvents at room temperature with cleavage of the Si–C bond [27] and reduce the salts of some metals [28]. It is not inconceivable that triphenylmethylamines, $\text{Ar}_3\text{CN}(\text{R})\text{CH}_2\text{SiX}_3\text{w}$, will show unusual properties, however at the moment such amines remain unknown. As part of our research program directed towards the studies of α -silylamines, we have synthesized 1-[(N-methyl-N-tritylamino)methyl]silatrane $\text{Ph}_3\text{CN}(\text{Me})\text{CH}_2\text{Si}(\text{OCH}_2\text{CH}_2)_3\text{N}$ (**1**) and below describe these results.

2. Experimental

2.1. General

All reactions were performed in flame dried glassware under an atmosphere of dry argon. The used solvents were purified according to standard procedures [29]. As a further precaution, diethyl ether was dried by filtration through a column packed with neutral alumina under a positive pressure of argon. The solvents were stored under argon over 4 Å molecular sieves. Chloroform was purified just before use. The purification of chloroform involves washing with water to remove EtOH, drying with K_2CO_3 , refluxing with P_2O_5 and distilling.

* Corresponding author.

E-mail address: nataly_lazareva@irioch.irk.ru (N.F. Lazareva).

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