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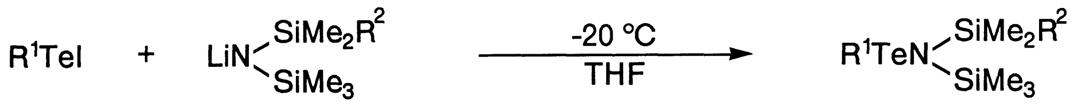
Isolation and Characterization of *N*,*N*-Disilylarenetellurenamides

Toshiaki MURAI,* Koji KIMURA, and Shinzi KATO
Department of Chemistry, Faculty of Engineering,
Gifu University, Yanagido, Gifu 501-11

N,*N*-Disilylarenetellurenamides were prepared by the reaction of benzenetellurenyl iodide with lithium *N*,*N*-disilylamide and characterized for the first time.

Considerable attention has been paid to organotellurium compounds, because of their highly potential availability.[1)] As a result, much attention has been devoted to develop new methods for forming and cleaving tellurium-carbon bond. On the contrary, little is known about the nature of organic compounds involving tellurium-nitrogen bond, probably due to their sensitivity towards oxygen and moisture. Especially, no tellurenamide derivatives ($RTeNR'_2$)[2)] has so far been reported to our best knowledge, although cyclic compounds involving Te(II)-N bond, such as tellurazole[3)] and tellurodiazole,[4)] have appeared in the literatures. Nevertheless, they should be fundamentally interesting class of compounds with respect to their chemical stability and reactivity in a series of chalcogenoamides.[5)] Herein, we describe the first example of arenetellurenamides as a form protected with trialkylsilyl groups and their characterization.



$$R^1TeI + LiN\begin{cases}SiMe_2R^2\\SiMe_3\end{cases} \xrightarrow[THF]{-20\ ^\circ C} R^1TeN\begin{cases}SiMe_2R^2\\SiMe_3\end{cases} \quad (1)$$

1

Lithium *N*,*N*-bis(trimethylsilyl)amide was treated with benzenetellurenyl iodide (PhTeI) in THF at -20 °C (Eq. 1). Within 5 h, the dark brownish color of PhTeI completely disappeared. After filtering a white solid through a glass filter, the solvent was removed in vauco to give a slightly yellow oil. The CI Mass spectrum clearly showed the molecular ionic peak at 367 corresponding to **1a**. In the ^1H NMR spectrum, two signals for phenyl protons and methyl protons on silicons were observed in a ratio of 5 to 18. The ^{13}C NMR spectrum is in good agreement with a structure of **1a** and does not show any peaks due to PhTeI and PhTeTePh. In ^{125}Te NMR spectrum, Te of **1a** is observed at 1209 ppm based on PhTeTePh. The product **1a** is highly sensitive towards air and moisture, and attempts to distill **1a** have not been successful yet. This instability is in marked contrast to that of sulfur[6)] and selenium[7)] analogues of **1a**, which can be distilled.

Table 1. Yields and Physical Properties of 1

Compound	R^1	R^2	Yield/%	Mp/°C	^{125}Te NMR
1a	Ph	Me	84	oil	1209.9
1b	Ph	*t*-Bu	76	-[a)]	-
1c	2-naphthyl	Me	93	63-65	1214.2

a) Decomposed at room temperature.

In order to enhance the stability of tellurenamides, either *t*-butyldimethyl group or 2-naphthyl group was attached to tellurium or nitrogen atom. Both arenetellurenamides **1b** and **1c** were obtained as yellow solids. Their yields and physical properties are shown in Table 1 with the result of **1a.** The product **1c**[8)] was stable towards heating and can be stored at room temperature under argon for at least several days.

As a preliminary result, the formation of addition product of **1a** to dimethylacetylenedicarboxylate was confirmed by ^{1}H NMR and high resolution mass spectra, although the yield was still low, whereas the reaction of **1a** with phenylacetylene resulted in the quantitative recovery of PhTeTePh. Further details of reactions using **1** as well as of synthesis of tellurenamides[9)] will be reported in due course.

References
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8) **1c**: IR (KBr) 3050, 2950, 2850, 2800, 1619, 1583, 1498, 1465, 1255, 1245, 1128, 903, 860, 835, 820, 781, 726, 721, 640, 618, and 467 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ = 0.35(18H, $SiCH_3$), and 7.51-8.00 (m, 7H, Ar); ^{13}C NMR ($CDCl_3$) δ = 4.7 ($SiCH_3$), 121.5, 125.7, 126.2, 127.1, 127.8, 128.0, 128.4, 130.1, 132.6, and 134.3 (Ar); $C_{16}H_{25}NSi_2Te$ from HR-MS m/z 417.05837 (calcd, 417.05867).
9) Similarly to 1, the synthesis of *N*,*N*-dialkyl derivatives has been attempted by using LDA. After the reaction with PhTeI, the light yellow color solution was obtained at -70 °C and CI mass spectrum showed the parent peak corresponding to $PhTeN[CH(CH_3)_2]_2$. However, the product instanly decomposed to PhTePh when the solution was raised to 0 °C.

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