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THE PREPARATION OF 1,2- Te_2E_5 ($\text{E} = \text{S}, \text{Se}$) FROM TELLURIUM CHLORIDE Te_2Cl_2

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A novel tellurium chloride Te_2Cl_2 has been prepared. Its application for the preparation of 1,2- Te_2Se_3 and 1,2- Te_2S_3 is described.

Keywords: tellurium chloride, tellurium chalcogen rings

INTRODUCTION

Several heterocyclic seven-membered selenium sulfides $\text{Se}_n\text{S}_{7-n}$, are known.^[1-7] The information on analogous $\text{Te}_n\text{E}_{7-n}$ -rings, however, is sparse due to the instability of these compounds as well as lack of suitable tellurium-containing reagents.^[1,8]

In the solid state the $\text{Se}_n\text{S}_{7-n}$ -rings are found in the chair-conformation with a fragment of four approximately coplanar atoms. In solvent they undergo facile pseudorotation similar to that deduced

for cycloheptasulfur S_7 .^[1,6,7] It is probable that the tellurium-containing Te_nE_{7-n} -rings are also fluxional.

EXPERIMENTAL

Te_2Cl_2 was prepared by the reaction between equimolar amounts of Li_2Te and $TeCl_4$ in toluene. Upon filtration and removal of the solvent the brownish yellow liquid Te_2Cl_2 was obtained.

1,2- Te_2E_5 ($E = S, Se$) were prepared by treating $[Ti(C_5H_5)_2E_5]$ ($E = S$ or Se)^[10] with Te_2Cl_2 in CS_2 as described previously for 1,2- S_2Se_5 .^[2]

RESULTS AND DISCUSSION

Te_2Cl_2 gives one ^{125}Te NMR resonance at 1297.3 ppm, which indicates that the product is rather $ClTeTeCl$ than Cl_2TeTe .

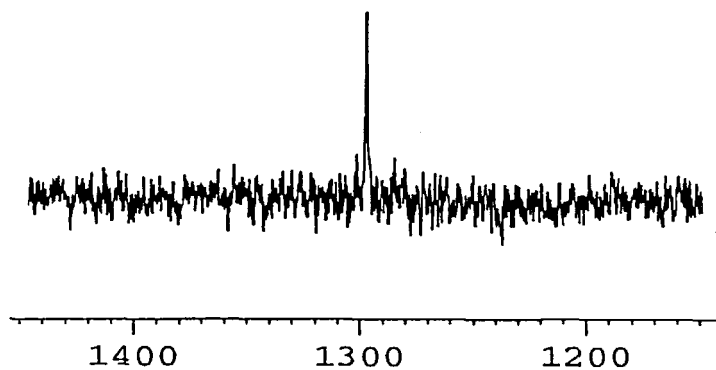


FIGURE 2 The ^{125}Te NMR spectrum of Te_2Cl_2 .

The mass spectrum of Te₂Cl₂ exhibited the following fragments: m/z 328 (Te₂Cl₂⁺), 291 (Te₂Cl⁺), 270 (TeCl₄⁺), 256 (Te₂⁺), 244 (?), 223 (?), 200 (TeCl₂⁺), 165 (TeCl⁺), 128 (Te⁺).

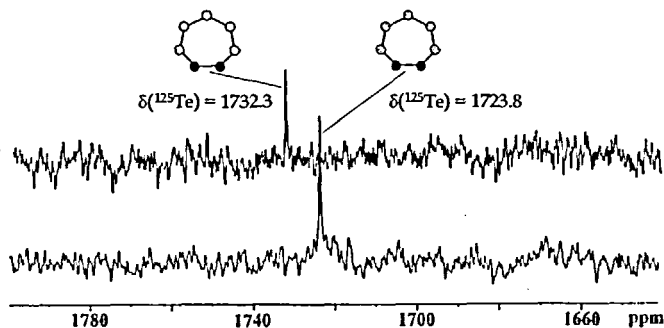


FIGURE 3 The ¹²⁵Te NMR spectra of 1,2-Te₂S₃ and 1,2-Te₂Se₃.

The ¹²⁵Te NMR resonances observed at 1732.3 and 1723.8 ppm are assigned to 1,2-Te₂S₃ and 1,2-Te₂Se₃, respectively. The ⁷⁷Se chemical shifts of 1,2-Te₂Se₃ are 1040.4, 1019.5 and 982.3 ppm (intensity ratio 2:1:2). These signals are consistent with the ⁷⁷Se resonances of 1,2-S₂Se₅.^[3,6]

1,2-Te₂S₃ decomposes through a tellurium-atom transfer forming TeS₃ and 1,2,5-Te₃S₃ as main decomposition products. They are identified from the ¹²⁵Te NMR spectrum by application of the well-known relationship between the ⁷⁷Se and ¹²⁵Te chemical shifts^[10] and taking into account the trends known for selenium sulfides.^[11] The decomposition of 1,2-Te₂Se₃ seems to involve both the tellurium- and selenium-atom transfer. The definite assignment of the complicated

^{125}Te and ^{77}Se NMR spectra is currently in progress utilizing tellurium enriched in the ^{125}Te -isotope (enrichment 94 %).

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