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THE THERMAL STABILITY OF SOME NEW CYCLIC TELLURIUM COMPOUNDS

GEORGIUS A. ADAM, ALI Z. AL-RUBAIE and HATIF A. AL-SHYIRAYDA
Department of Chemistry, College of Science, University of Basrah, Basrah (Iraq)
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ABSTRACT

The thermal stability of some new cyclic tellurium compounds, i.e., C_4H_8TeRX: R,X = I, I (**1**); CH_3, I (**2**); C_2H_5, Br (**3**); C_3H_5, Cl (**4**); $PhCOCH_2$, Br (**5**); 4-$BrPhCOCH_2$, Br (**6**); Ph, BPh_4(**7**); and $C_5H_{10}TeRX$: R, X = I, I (**8**); CH_3, I (**9**), were studied by thermogravimetric analysis. Compounds **2** and **9** vaporised at 245 and 165°C, respectively. The other compounds decompose in three stages, losing RX in the first stage, then eliminating tellurium to form cyclobutane.

INTRODUCTION

During an earlier research programme a new series of cyclic organotellurium compounds were synthesised and characterised [1] by various techniques, i.e., conductivity measurements, mass spectrometry and ^1H, ^{13}C and ^{125}Te NMR.

In the present work we decided to study the thermal stability behaviour of these new compounds in the hope of using them as stabilisers for some synthetic polymers.

EXPERIMENTAL

Materials

The cyclic organotellurium compounds used in this study are:
(**1**) 1,1-diiodo-1-telluracyclopentane
(**2**) 1-methyl-1-iodo-1-telluracyclopentane
(**3**) 1-ethyl-1-bromo-1-telluracyclopentane
(**4**) 1-allyl-1-chloro-1-telluracyclopentane
(**5**) 1-phenacyl-1-bromo-1-telluracyclopentane
(**6**) 1-(4-bromophenacyl)-1-bromo-1-telluracyclopentane

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(7) 1-phenyl-1-telluracyclopentane tetraphenylborate
(8) 2-methyl-1,1-diiodo-1-telluracyclopentane
(9) 1,2-dimethyl-1-iodo-1-telluracyclopentane.

Compounds **1** and **2** were synthesised and characterised according to refs. 2 and 3 while the other compounds were synthesised and characterised by the method of Al-Rubaie et al. [1,2].

Thermogravimetric analysis

TG was carried out using a MOM derivatograph thermoanalyser, which measures TG, DTG, DTA and temperature. The instrument was pre-calibrated with standard materials. All the measurements were carried out in the presence of air and against standard α-Al_2O_3. The analyses were performed by heating the sample at a steady heating rate 10° min^{-1} until the sample was substantially decomposed. Typical thermograms are shown in Figs. 1–3.

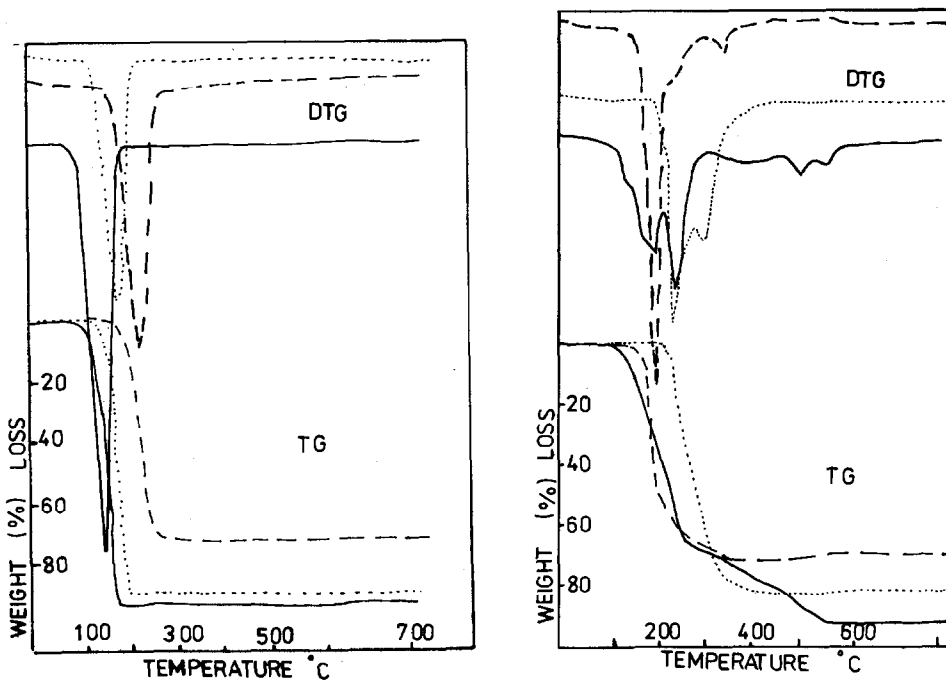


Fig. 1. Thermogravimetric curves (DTG and TG) for: (· · · · · ·) sample **9**; (- - - - - -) sample **1**; (———) sample **2**.

Fig. 2. Thermogravimetric curves (DTG and TG) for: (· · · · · ·) sample **7**; (———) sample **6**; (- - - - - -) sample **8**.

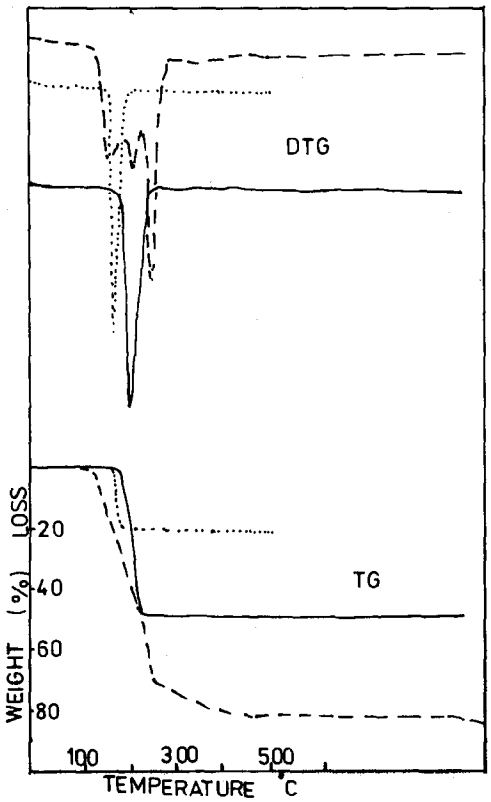


Fig. 3. Thermogravimetric curves (TG and DTG) for: (·····) sample **4**; (———) sample **3**; (- - - - - -) sample **5**.

RESULTS AND DISCUSSION

(i) The thermogravimetric curves for compounds **2** and **9** (Fig. 1) show that they evaporate at 245 and 165°C, respectively. On the other hand, their thermograms indicate that they are bonded to one molecule of water, i.e., $C_4H_8TeCH_3I \cdot H_2O$ and $C_5H_{10}TeCH_3I \cdot H_2O$. A similar basis has been found with other tellurium and selenium compounds by X-ray crystallography (crystallisation solvents) [4].

(ii) Concerning the other organocyclic tellurium compounds, the thermal decomposition takes place in three stages; first, losing alkyl halide (RX) or I_2 which is well established with other telluronium salts [3a,5,6]; second, forming cyclobutane and tellurium metal at about 200–235°C; and third, evaporation of tellurium metal at high temperature, ca. 1000°C. The first stage of decomposition has been observed with other telluronium salts [5,6], e.g.

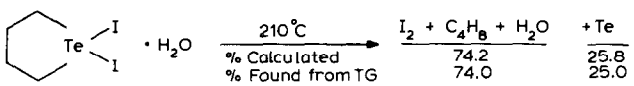
$$R_3TeX \xrightarrow{\Phi} R_2Te + RX \text{ (X is halide or pseudohalide)}$$

The same pattern of decomposition has also been suggested for these new cyclic telluronium salts according to mass spectrometric analysis [1,7,8].

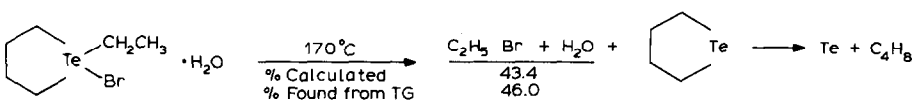
Concerning the second stage for the decomposition of cyclic tellurium compounds, it has been reported that thermal elimination of tellurium metal takes place from 3,4-benzo-1-telluracyclopentane at 500°C and 0.4–0.5 mm Hg of helium gas, a procedure which has been used as a synthetic method for benzocyclobutane [9].

According to the thermogravimetric analysis calculations the following schemes are the expected routes for the thermal decomposition of these compounds.

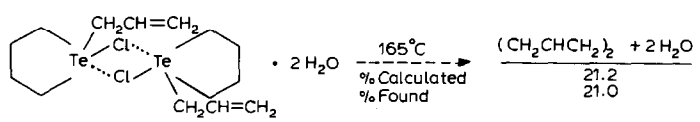
Compound 1

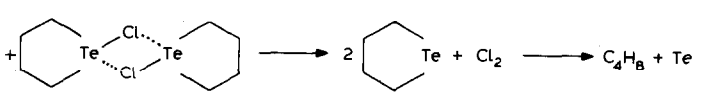


Compound 3



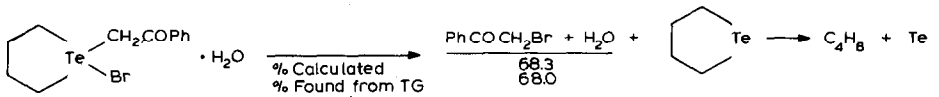
Compound 4





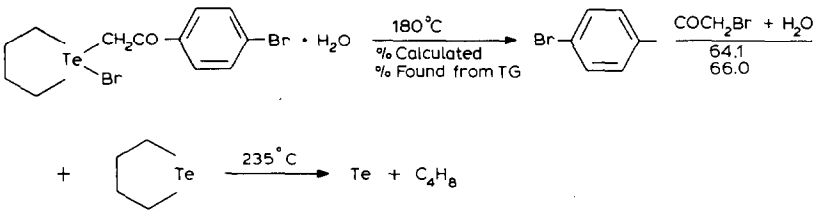
The mass spectra clearly indicated that this compound exists as a dimer [1] which is confirmed by our thermogravimetric analysis (TG) results.

Compound 5 (see Fig. 3)

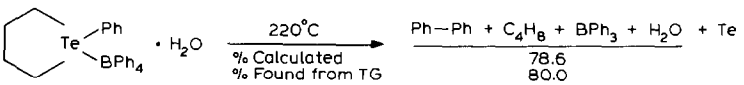


Three decomposition peaks were observed on the DTG curve at 150, 210 and 260°C for the above three components leaving tellurium metal which finally evaporates at about 1000°C.

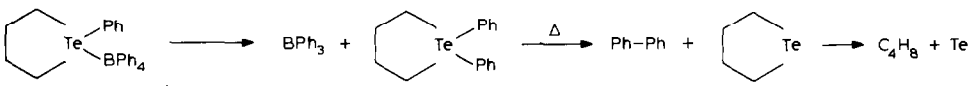
Compound 6



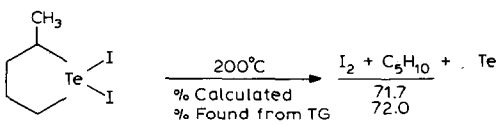
Compound 7



Two main decomposition peaks were observed on the DTG curve at 220 and 270°C for these components leaving tellurium metal. This compound merits special mention. The compound $C_4H_8TePh_2$ is presumably formed as an intermediate via further phenylation during the thermolysis of the compound (**7**). This review is supported by the mass spectra of analogous compounds [2,7]. The expected route for the thermolysis of this compound may be represented by the following scheme.



Compound 8



The thermogravimetric analysis calculations indicate that compounds **1**, **3**, **5**, **6** and **7** are bonded to one molecule of water, similarly to compounds **2** and **9**. Such behaviour has been reported to exist with other tellurium and selenium compounds [5,6].

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