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Thermal behaviour of some new organotellurium compounds containing an *ortho*-acetamido group

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Abstract

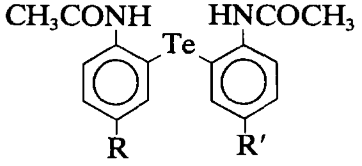
The thermal stability of some new organotellurium compounds containing an acetamido group in the *ortho*-position to the tellurium atom (i.e. Ar_2Te, ArAr'Te and Ar_2Te_2, where Ar' is 2-(CH_3CONH)-5-R-C_6H_3, R is CH_3, Br, NO_2; Ar' is 2-(CH_3CONH)-5-R'-C_6H_3, and R' is Br, NO_2) have been studied by thermogravimetric analysis. In general, these compounds extruded tellurium in addition to one or two acetamido groups in one or two steps.

INTRODUCTION

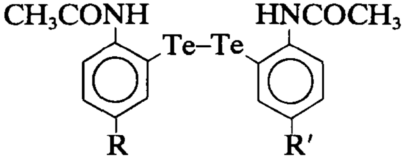
It is not common to find qualitative and quantitative thermochemical studies about organotellurium compounds in the solid state [1]. Our interest in the study of the thermal stability of organotellurium compounds [2, 3] aims to relate their composition, structure and material processing with those properties suitable for various technological applications.

During an earlier research programme, a new series of 2-acetamidoaryltellurium compounds was synthesised and characterised [4, 5] by various techniques, i.e. elemental analyses, together with IR, ^1H and ^{13}C NMR and mass spectroscopy.

Here we report on the thermal stability of new diaryl telluride (**1–4**)



1: R = R' = NO_2
2: R = R' = CH_3
3: R = CH_3, R' = Br
4: R = CH_3, R' = NO_2



5: R = R' = CH_3
6: R = R' = Br
7: R = R' = NO_2

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and diaryl ditelluride (**5–7**) compounds in the hope of using the compounds as antioxidants for some synthetic polymers [6], and we shall discuss the main factors influencing their thermal stability.

EXPERIMENTAL

The following compounds were used in this study: bis(2-acetamido-5-nitrophenyl) telluride (**1**), bis(2-acetamido-5-methylphenyl) telluride (**2**), 2-acetamido-5-methylphenyl 2′-acetamido-5′-bromophenyl telluride (**3**), 2-acetamido-5-methylphenyl 2′-acetamido-5′-nitrophenyl telluride (**4**), bis(2-acetamido-5-methylphenyl) ditelluride (**5**) bis(2-acetamido-5-bromophenyl) ditelluride (**6**), and bis(2-acetamido-5-nitrophenyl) ditelluride (**7**).

Compounds **1**, **2**, **3**, **4**, **6** and **7** were synthesised and characterised in our laboratory [4, 5]; compound **5** was synthesised and characterised according to the method of Junk and Irgolic [7].

Thermogravimetric analysis

The thermal analysis was carried out using a Du Pont thermoanalyser, model 1090, which records TG and DTG curves and measures temperature. The instrument was precalibrated with standard materials. All measurements were carried out in nitrogen atmosphere and against

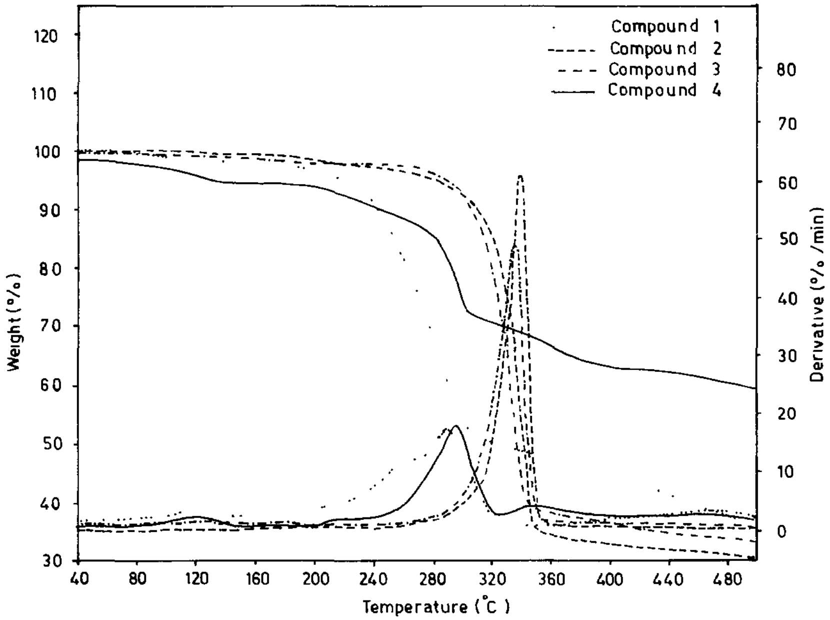


Fig. 1. Thermogravimetric curves (DTG and TG) for compounds **1**, **2**, **3** and **4**.

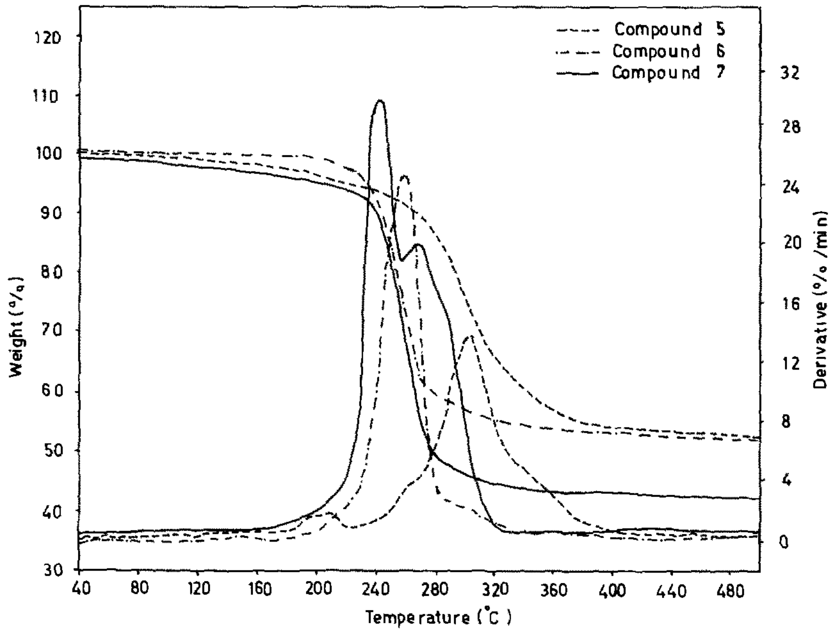


Fig. 2. Thermogravimetric curves (DTG and TG) for compounds **5**, **6** and **7**.

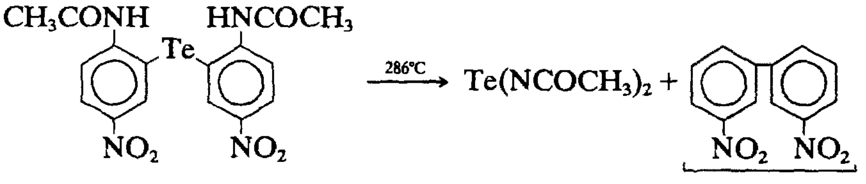
standard α-Al_2O_3. The analyses were performed by heating the sample at a steady rate of 20°C min^{-1} between ambient temperature and 500°C.

Typical thermograms are shown in Figs. 1 and 2.

RESULTS AND DISCUSSION

Compound 1

The thermogram (Fig. 1) shows a maximum loss at 286°C, which corresponds to the loss of biaryl (i.e. (m-$NO_2C_6H_4$–). This may be attributed to the elimination of a tellurium atom together with two acetamido groups, forming $Te(NCOCH_3)_2$ as a residual product. The proposed compound $Te(NCOCH_3)_2$ is not unique, because it is well known that tellurium is capable of forming stable compounds of type $(RN)_2Te$, where R = $COCH_3$ or $PhSO_2$ [8, 9]



Calculated, 50.25%
Found from TG, 49.34%

Such aryl coupling reactions of organotellurium compounds, as is well known, may be promoted thermally [10, 11] or photochemically [12]. Thus we believe that tellurium is thermally extruded from compound **1**.

Compound **2**

The thermogram (Fig. 1) shows a maximum loss at 338°C, which corresponds to removal of Ar_2 (i.e. (m-$CH_3C_6H_4$–$)_2$); mass loss observed 65.09%, compared with the calculated 65.28%. The remaining product is probably tellurium dioxide. The formation of tellurium dioxide could be explained as a result of the presence of two molecules of water, as indicated from the thermogram, which may hydrolyse the proposed intermediate $Te(NCOCH_3)_2$ to TeO_2 and the amide. Compounds of types $Te(NCOR)_2$ are known to give tellurium dioxide and amide on hydrolysis [8, 9].

Compound **3**

A rather pronounced loss at 336°C is observed; see Fig. 1. This effect seems to correspond to the removal of $C_{15}H_4NOBr$ (mass loss observed 59.56%, compared with the calculated 60.01%). The loss of $C_{15}H_4NOBr$ can be associated with the formation of $TeO(NCOCH_3)$ owing to the presence of one molecule of water, which cause a partial hydrolysis to the proposed residual product (i.e. $Te(NCOCH_3)_2$), as is shown for compound **2**. Furthermore, the thermogram indicates that this compound is bonded to one molecule of water (crystallisation solvent) [13].

Compound **4**

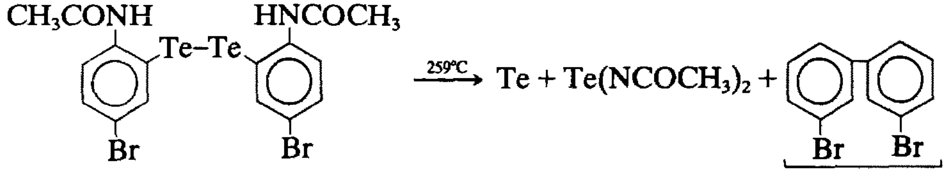
The thermogram in Fig. 1 shows a large mass loss of 26.88%, occurring at 296°C. This may be due to the loss of the two acetamido groups together with one molecule of water, calculated mass loss 27.94%.

Compound **5**

One decomposition peak was observed on the DTG curve at 303°C (Fig. 2). The mass loss reaches a value of 40.24% (calculated 40.47%), which points to the formation of Te plus $TeO(NCOCH_3)$ as residual products. At 208°C there is a small loss probably associated with the loss of H_2O, which cause a partial hydrolysis to the proposed intermediate $Te(NCOCH_3)_2$.

Compound **6**

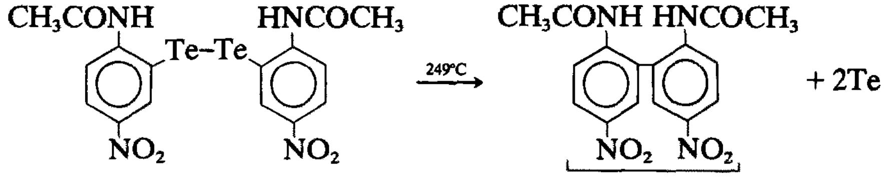
A pronounced and massive loss occurs at 259°C, amounting to 45.35%, which can be explained by the loss of $(m\text{-BrC}_6\text{H}_4\text{–})_2$ and formation of Te plus $\text{Te(NCOCH}_3)_2$ as residual products



Calculated, 45.79%
Found from TG, 45.35%

Compound **7**

On heating compound **7**, two main overlapping peaks are observed on the TG curve (Fig. 2). These could be attributed to the thermal extrusion of two tellurium atoms from compound **7** leading to loss of biaryl



Calculated, 58.39%
Found from TG, 58.84%

From the above thermogravimetric analysis it may be concluded that the degradation of compounds **1**–**7** is a complex process involving two competing reactions, extrusion of tellurium and/or the acetamido group. Generally, the compounds readily extruded tellurium thermally, and this may react with acetamido groups to form the residual compound of type $\text{Te(NCOCH}_3)_2$. In the presence of one molecule of water, $\text{TeO(NCOCH}_3)$ could be formed, and, if two molecules of water are present, TeO_2 may be formed.

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