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Thermochimica Acta 338 (1999) 113–123

thermochimica acta

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# Preparation and thermal behaviour of some hydrazinium dicarboxylates

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Received 10 March 1999; accepted 24 June 1999

**Abstract**

Hydrazinium hydrogendicarboxylates of pimelic, suberic, azelaic, sebacic, tartaric, $\alpha$-ketoglutaric and iminodiacetic acids and dihydrazinium dicarboxylates of malic, aspartic and glutamic acids have been prepared by accomplishing neutralisation of aqueous hydrazine hydrate with the respective acids. Formation of these hydrazinium salts has been confirmed by analytical, IR spectral and thermal studies. All these salts are anhydrous except hydrazinium hydrogenpimelate and iminodiacetate which are monohydrated. IR spectra of the salts register N–N stretching frequencies of the hydrazinium ion in the region 955–1000 $\mathrm{cm}^{-1}$. The thermal decomposition behaviour of these hydrazine derivatives have been studied by simultaneous TG and DTA. Among the salts prepared, the higher dicarboxylates decompose through their respective acid intermediates. Almost, all of the intermediates decompose exothermically with the complete elimination of the sample to gaseous products. © 1999 Elsevier Science B.V. All rights reserved.

*Keywords:* Hydrazinium salts; Dicarboxylic acids; IR spectra; Thermal decomposition

## 1. Introduction

Hydrazine hydrate, a diacidic base forms $N_2H_5^+$ [hydrazinium (1+)] and $N_2H_6^{2+}$ [hydrazinium (2+)] ionic salts with a wide variety of mineral and carboxylic acids. Among them, $N_2H_5^+$ salts are the best known, whereas $N_2H_6^{2+}$ salts are formed only with strong mineral acids [1]. A variety of chemical behaviours, various thermal decomposition paths and therefore their potential use make this region of chemistry still interesting and expanding. These hydrazinium salts have numerous applications, such as a source of anhydrous hydrazine, additives in propellants, drugs to treat cancer and Hodgkin's disease, explosives [1] and also as ligands to prepare metal hydrazinium/hydrazine complexes [2–5]. A few of these salts are also used as flame retardants [6,7] and proton conductors [8].

In contrast to the chemistry of many hydrazinium derivatives, detailed studies into the thermal chemistry of hydrazinium carboxylates have only recently come to fruition [9–15]. Among the reported hydrazinium carboxylates, there appears to be only limited work done on hydrazinium aliphatic-dicarboxylates [1,10, 14–16]. Hence, the aim of the present study is to shed new light on the thermal properties and IR spectra of the hydrazinium salts of aliphatic diprotic acids.

## 2. Experimental

All the hydrazinium derivatives have been prepared by neutralising respective dicarboxylic acids with

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PII: S0040-6031(99)00202-6

hydrazine hydrate (99–100%) in aqueous medium with appropriate molar ratios. All the acids were used as purchased without further purification. The easily available (DL)-malic, D-tartaric, L-aspartic and L-glumatic acids have been used for the preparation of respective salts.

### 2.1. Preparation of hydrazinium hydrogendicarboxylates of higher homologs

Hydrazine hydrate (1 ml, 0.02 mol) and respective dicarboxylic acids (for e.g., pimelic acid, 1.6 g, 0.01 mol) were mixed in 50 ml of water and heated over water bath to get clear solutions. The resulting solutions were kept for crystallisation in a vacuum desiccator over calcium chloride. The colourless derivatives obtained were separated, washed with absolute alcohol and dried in vacuo. Hydrazinium hydrogensebacate separated from the solution after 5 days, whereas other salts separated between 4 and 8 weeks. All the salts are powders except hydrazinium hydrogensuberate which is crystalline.

### 2.2. Preparation of hydrazinum hydroxydicarboxylates

Dihydrazinium malate and hydrazinium hydrogentartrate have been prepared by malic acid (1.34 g, 0.01 mol) or tartaric acid (1.5 g, 0.01 mol) and hydrazine hydrate (1 ml, 0.02 mol and 0.5 ml, 0.01 mol, respectively) in 50 ml of water. The resulting solutions were kept as such for crystallisation in a vacuum desiccator. The hydrazinium hydrogentartrate separated after 24 h, whereas dihydrazinium malate separated only after 7 months. It is worth mentioning that the other ratios (1 : 2 and 2 : 1) of acid and base also yielded the same, hydrazinium hydrogentartrate. Both the salts are highly crystalline in nature.

### 2.3. Preparation of dihydrazinium amino dicarboxylates

Both 1 : 1 and 2 : 1 molar mixtures of hydrazine hydrate and aspartic or glumatic acids yielded one and the same dihydrazinium salts. The white shiny crystals obtained after 30 days were isolated and dried as above.

### 2.4. Preparation of hydrazinium hydrogen α-ketoglutaric and iminodiacetic acids

Monohydrazinium salts of the keto and imino acids have been prepared by mixing hydrazine hydrate and the respective acids in 2 : 1 and 1 : 1 molar ratio (1 ml, 0.01 mol; 1.46 g, 0.01 mol and 0.5 ml, 0.01 mol; 1.33 g, 0.01 mol, respectively). The hydrazinium α-keto glutarate separated after a week is highly crystalline. The 1 : 1 and 2 : 1 molar ratios of base and imino acid also yielded the monohydrazinium salt.

All the salts are white solids and highly soluble in cold water except hydrazinium hydrogensebacate which is completely soluble only in hot water. Except the salts of sebacic and tartaric acids, others are

Table 1
Analytical data

| Compound | mp/°C | Hydrazine % | | Carbon % | | Hydrogen % | | Nitrogen % | |
|---|---|---|---|---|---|---|---|---|---|
| | | Found | Calculated | Found | Calculated | Found | Calculated | Found | Calculated |
| $HOOC\text{-}(CH_2)_5\text{-}COON_2H_5\cdot H_2O$ | 79.80 | 14.70 | 15.24 | 39.80 | 40.00 | 8.20 | 8.57 | 13.15 | 13.33 |
| $HOOC\text{-}(CH_2)_6\text{-}COON_2H_5$ | 106.00 | 15.80 | 15.53 | 46.15 | 46.60 | 8.42 | 8.74 | 13.48 | 13.59 |
| $HOOC\text{-}(CH_2)_7\text{-}COON_2H_5$ | 63.80 | 14.30 | 14.55 | 48.78 | 49.09 | 8.72 | 9.09 | 12.86 | 12.73 |
| $HOOC\text{-}(CH_2)_8\text{-}COON_2H_5$ | 106.80 | 13.60 | 13.68 | 51.45 | 51.28 | 9.66 | 9.40 | 11.76 | 11.97 |
| $H_5N_2OOCCH(OH)CH_2COON_2H_5$ | 183.60 | 31.80 | 32.32 | 24.12 | 24.24 | 6.85 | 7.07 | 28.53 | 28.28 |
| $HOOCCH(OH)CH(OH)COON_2H_5$ | 180.20 | 18.20 | 17.58 | 26.75 | 26.37 | 5.23 | 5.49 | 15.22 | 15.38 |
| $H_5N_2OOCCH(NH_2)CH_2COON_2H_5$ | 142.00 | 31.90 | 32.49 | 24.18 | 24.37 | 7.93 | 7.61 | 35.15 | 35.53 |
| $H_5N_2OOCCH(NH_2)(CH_2)_2COON_2H_5$ | 159.50 | 29.70 | 30.33 | 28.96 | 28.44 | 7.75 | 8.06 | 33.45 | 33.18 |
| $HOOC(CH_2)_2COCOON_2H_5$ | 175.20 | 18.30 | 17.98 | 33.44 | 33.71 | 5.26 | 5.62 | 15.42 | 15.73 |
| $HOOCCH_2NHCH_2COON_2H_5\cdot H_2O$ | 161.50 | 17.90 | 17.49 | 26.63 | 26.23 | 7.26 | 7.10 | 22.56 | 22.95 |

slightly hygroscopic and are stored in tightly closed containers by keeping them in the desiccator over calcium chloride.

The hydrazine content in all the salts was determined volumetrically using 0.025 M potassium iodate solution under Andrews' conditions [17]. Melting points were determined on Mettler FP 5 instrument and are uncorrected. Infrared spectra were recorded using KBr discs (4000–400 cm$^{-1}$) in a Shimadzu FTIR-8201 PC spectrophotometer. The simultaneous TG–DTA studies were carried out using a STA 1500 thermal analyser and the curves were obtained in air using platinum cups as sample holders with 5–10 mg of the samples at the heating rate of 10°C min$^{-1}$.

## 3. Results and discussion

The analytical data of the salts were consistent with the proposed formulae for them (Table 1). Hydrazinium hydrogenpimelate and iminodiacetate are monohydrated whereas the other salts are anhydrous. This is borne out by analytical, IR spectral and thermal studies.

Table 2
IR spectral data (cm$^{-1}$)

| Compound | $\nu_{(OH)}$of acid [a] | $\nu_{(NH)}$ | $\nu_{(C=O)}$of COOH & keto groups | $\nu_{asy(OCO)}$ | $\nu_{sy(OCO)}$ | $\nu_{(N-N)}$ |
|---|---|---|---|---|---|---|
| Pimelic acid | 3040–2557 (b) | – | 1697 | – | – | – |
| Pimelate | 3230 | 3040<br>2941<br>2887 | 1720 | 1590 | 1406 | 964 |
| Suberic acid | 3040–2532 (b) | – | 1697 | – | – | – |
| Suberate | 3263 | 3040<br>2926<br>2849 | 1720 | 1640 | 1400 | 964 |
| Azelaic acid | 3040–2630 (b) | – | 1700 | – | – | – |
| Azelate | 3250 | 3020<br>2960<br>2910 | 1719 | 1610 | 1406 | 970 |
| Sebacic acid | 3040–2500 (b) | – | 1697 | – | – | – |
| Sebacate | 3244 | 3020<br>2914<br>2949 | 1719 | 1640 | 1402 | 970 |
| Malic acid | 3450–3000 (b) | – | 1750–1684 (b) | – | – | – |
| Malate | – | 3400<br>3315<br>3059 | – | 1610 | 1450 | 961 |
| Tartaric acid | 3500–2800 (b) | – | 1730 | – | – | – |
| Tartrate | 3440–3280 (b) | 3040–2974 (b) | 1719 | 1580 | 1408 | 962 |
| Aspartic acid | 3440–3320 (b) | 3040–2928 (b) | 1686 | – | – | – |
| Aspartate | – | 3200–3100 (b) | – | 1630 | 1398 | 1000 |
| Glutamic acid | 3180–3020 (b) | 2960–2880 (b) | 1645 | – | – | – |
| Glutamate | – | 3200–3050 (b) | – | 1630 | 1399 | 1000 |
| α-ketoglutaric acid | 3200–2800 | –<br>– | 1720<br>1560 | – | – | – |
| α-ketoglutarate | 3360 | 3200<br>3088 | 1670 | 1624 | 1389 | 990 |
| Iminodiacetic acid | 3099 | 3000<br>2972 | 1720 | – | – | – |
| Iminodiacetate | – | 3065 | – | 1622 | 1398 | 955 |

[a] b, broad.

Table 3
Thermal decomposition data

| Compound | DTA peak Temp./°C[a] | Thermogravimetry (TG) | | | Phenomenon |
|---|---|---|---|---|---|
| | | Temp. range/°C | Mass loss (%) | | |
| | | | Found | Calculated | |
| $HOOC\text{-}(CH_2)_5\text{-}COON_2H_5 \cdot H_2O$ | 75 (+) | – | – | – | Melting |
| | 152 (−) | 100–220 | 20.22 | 23.81 | Dehydration followed by dehydrazination |
| | 300 (−)<br>392 (−)<br>500 (−) | 220–520 | 100.00 | 100.00 | Complete decomposition |
| $HOOC\text{-}(CH_2)_6\text{-}COON_2H_5$ | 99 (+) | – | – | – | Melting |
| | 151 (−) | 127–156 | 15.00 | 15.53 | Dehydrazination |
| | 302 (−)<br>475 (−) | 156–540 | 100.00 | 100.00 | Complete decomposition |
| $HOOC\text{-}(CH_2)_7\text{-}COON_2H_5$ | 79 (+) | – | – | – | Melting |
| | 130 (−) | 90–155 | 16.00 | 14.55 | Dehydrazination |
| | 290 (−)<br>326 (−)<br>398 (−)<br>431 (−)<br>463 (−)<br>510 (−) | 155–550 | 100.00 | 100.00 | Complete decomposition |
| $HOOC\text{-}(CH_2)_8\text{-}COON_2H_5$ | 100 (+) | – | – | – | Melting |
| | 137 (−) | 120–147 | 13.00 | 13.68 | Dehydrazination |
| | 284 (−)<br>500 (−) | 147–520 | 100.00 | 100.00 | Complete decomposition |
| $H_5N_2OOCCH(OH)CH_2COON_2H_5$ | 154 (+) | 100–160 | 32.00 | 32.32 | Melting with dehydrazination |
| | 247 (−)<br>284 (−) | 160–290 | 90.00 | 100.00 | Decomposition |
| | 400 (−) | 290–400 | – | – | Tar decomposition |
| $HOOCCH(OH)CH(OH)COON_2H_5$ | 181 (+) | 172–190 | 18.40 | 17.58 | Dehydrazination |
| | 291 (−)<br>300 (−)<br>526 (−) | 190–600 | 99.00 | 100.00 | Complete decomposition |
| $H_5N_2OOCCH(NH_2)CH_2COON_2H_5$ | 150 (+) | 125–154 | 30.00 | 32.49 | Melting with dehydrazination |
| | 268 (−) | 154–280 | 100.00 | 100.00 | Complete decomposition |
| $H_5N_2OOCCH(NH_2)(CH_2)_2COON_2H_5$ | 149 (+) | 147–153 | 58.00 | 58.10 | Melting with dehydrazination followed by the formation of glycine |
| | 275 (−) | 153–280 | 100.00 | 100.00 | Complete decomposition |
| $HOOC(CH_2)_2COCOON_2H_5$ | 185 (+) | 179–194 | 34.00 | 33.71 | Melting with dehydrazination followed by the formation of succinic acid |
| | 502 (−) | 194–600 | 92.00 | 100.00 | Complete decomposition |
| $HOOCCH_2NHCH_2COON_2H_5 \cdot H_2O$ | 42 (+) | 30–45 | 10.00 | 09.84 | Dehydration |
| | 167 (+) | – | – | – | Melting |
| | 190 (+) | 167–200 | 42.00 | 45.36 | Dehydration followed by the formation of succinic anhydride |
| | 558 (−) | 200–600 | 100.00 | 100.00 | Complete decomposition |

[a] (+), endotherm; (−), exotherm; d, doublet.

It is note worthy that after succinic acid, the higher homologs, in the dicarboxylic acid series, form only monohydrazinium salts. The presence of more methylene groups (+I effect) makes the −I effect of the carboxylate group negligible on the O–H bond [14]. Hence, dihydrazinium salts are not formed with higher dicarboxylic acids.

In order to know the substituent effect of –OH, –$NH_2$, –C=O– and –NH– on the salt forming ability of dicarboxylic acids with hydrazine, the easily available substituted acids have been chosen. Malic, aspartic and glutamic acids gave only dihydrazinium salts, whereas monohydrazinium derivatives are obtained with α-ketoglutaric and iminodiacetic acids. It is difficult to generalise the reason for the formation of either mono- or dihydrazinium salts.

Due to the distinct $pK_a$ values of malic acid ($pK_{a1} = 3.46$ and $pK_{a2} = 5.10$), it is expected to form both mono as well as dihydrazinium salts, but it gave only dihydrazinium salt after 7 months. The aspartic and glutamic acids are expected to give only monohydrazinium salts, due to the presence of inner salt (zwitter ion) in the acids. In contrary, amino dicarboxylic acids yielded only dihydrazinium salts above their isoelectric points. Our attempt to prepare monohydrazinium salts was unsuccessful. It is interesting to mention here that succinic acid forms both mono and dihydrazinium derivatives and glutaric acid forms only monohydrazinium salt [14]. But, the hydroxy and/or amino substituted succinic and glutaric acids behave indifferently on the salt forming ability.

Irrespective of the acid–base ratio, D-tartaric acid forms only monohydrazinium salt. Its composition is in accordance with hydrazinium L-tartrate [16] which is stabilised by numerous hydrogen bonds.

In the case of α-ketoglutaric acid, the electron withdrawing (−I) effect of the keto group makes the adjacent carboxyl group more acidic favouring the formation of monohydrazinium salt. As expected [11], due to the formation of inner salt (zwitter ion), iminodiacetic acid forms only monohydrazinium derivative.

## 4. Infrared spectra

The important IR bands of the acids and their salts with assignments are listed in Table 2. The IR spectra of the hydrazinium hydrogenpimelate and iminodiacetate monohydrate show a band at 3400 and 3440 $cm^{-1}$, respectively, due to the O–H stretching of the water molecule. The carbonyl stretching frequencies of free carboxyl groups in the hydrazinium hydrogendicarboxylates register their absorption frequencies in the region 1670–1720 $cm^{-1}$. The bands in the region 1375–1450 and 1580–1640 $cm^{-1}$ for these

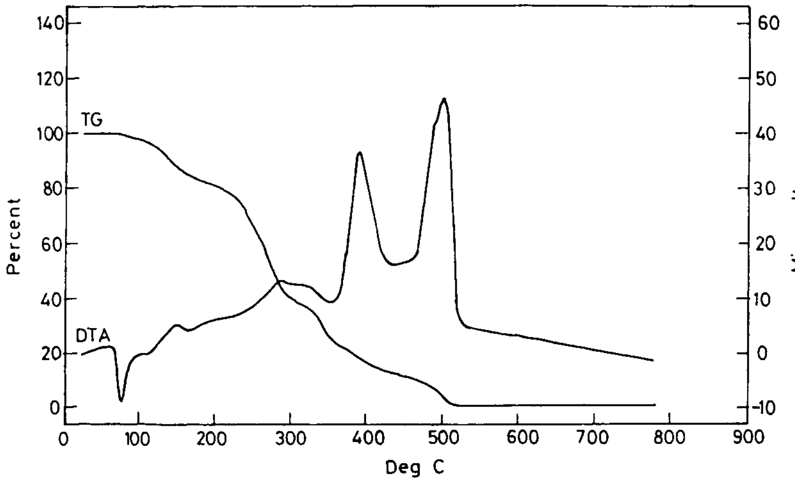

Fig. 1. Simultaneous TG–DTA of HOOC-$(CH_2)_5$-$COON_2H_5 \cdot H_2O$.

salts are assigned for symmetric and asymmetric stretching frequencies of the carboxylate ions, respectively. The N–N stretching frequencies in the range 955–1000 $cm^{-1}$are indicative of the presence of $N_2H_5^+$ ion in the salts [1].

## 5. Thermal decomposition studies

The thermal decomposition modes of all the salts are given in Table 3. The observed mass losses from TG coincide well with the theoretical mass losses. Most of the salts start decomposition with or after melting.

### *5.1. Hydrazinium hydrogenpimelate monohydrate and hydrazinium hydrogensuberate, azelate and sebacate*

The simultaneous TG–DTA curves of the salts are shown in Figs. 1–4, respectively. All of them show distinct melting before decomposition. In all cases

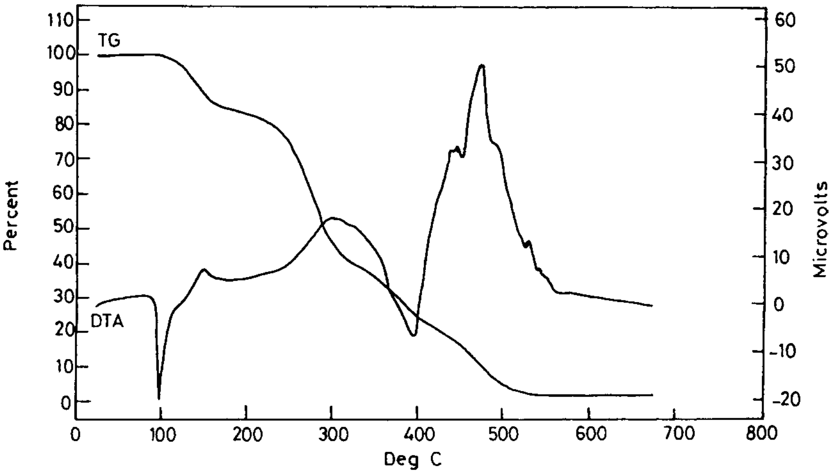

Fig. 2. Simultaneous TG–DTA of HOOC-$(CH_2)_6$-$COON_2H_5$.

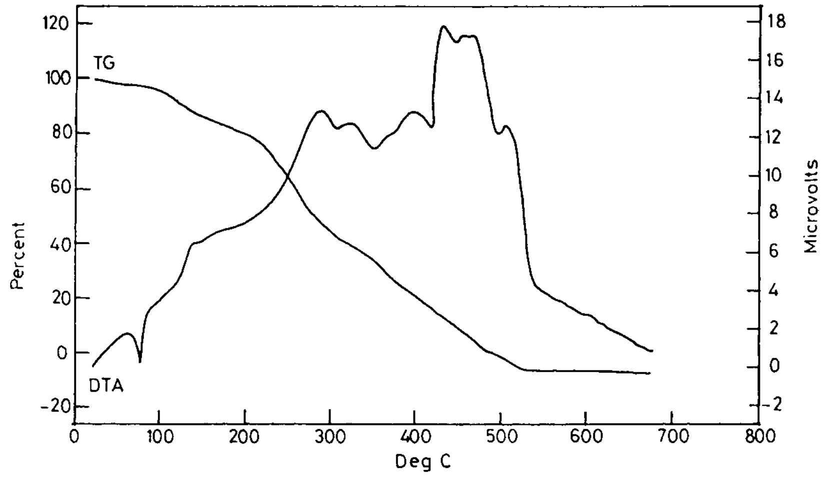

Fig. 3. Simultaneous TG–DTA of HOOC-$(CH_2)_7$-$COON_2H_5$.

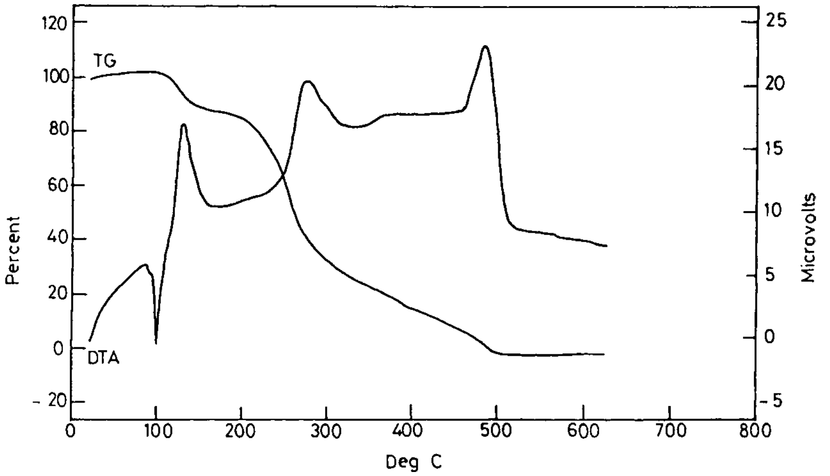

Fig. 4. Simultaneous TG–DTA of HOOC-$(CH_2)_8$-$COON_2H_5$.

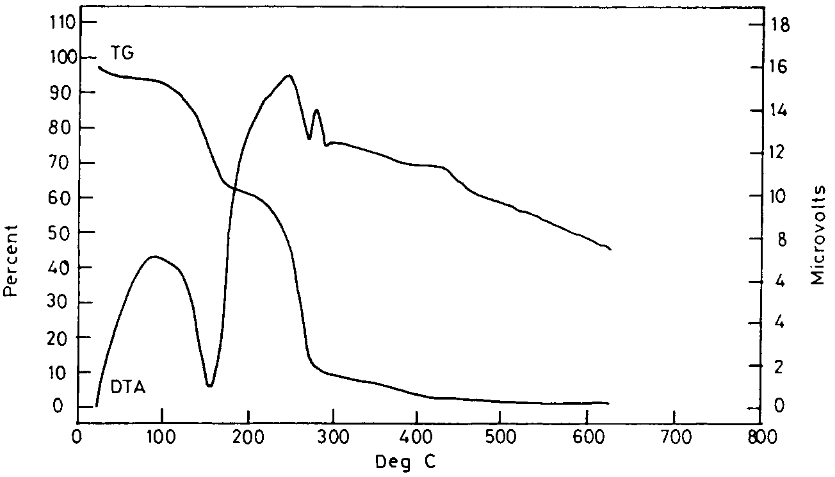

Fig. 5. Simultaneous TG–DTA of $H_5N_2OOCCH(OH)CH_2$ $COON_2H_5$.

the first exotherm corresponds to dehydrazination (Table 3) of the salts (in the case of pimelic acid, dehydration takes place prior to dehydrazination). The successive exotherms obtained as complex multiplets, are assigned for the complete decomposition of the respective acids into gaseous products. The decomposition temperatures of the acids obtained during decomposition of the salts are in good agreement with the literature decomposition temperatures [18].

### *5.2. Dihydrazinium malate and hydrazinium hydrogentartrate*

Both the salts decompose with melting as observed from TG mass losses (Figs. 5 and 6). The first endotherms in the DTA curves correspond to the mass losses in TG curves for the formation of respective acids with melting and dehydrazination. Then the acids decompose completely with exothermic trans-

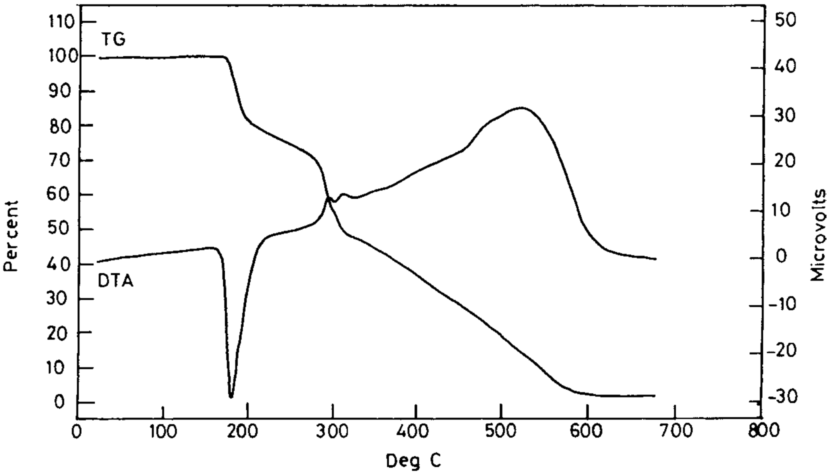

Fig. 6. Simultaneous TG–DTA of HOOCCH(OH)CH(OH) $COON_2H_5$.

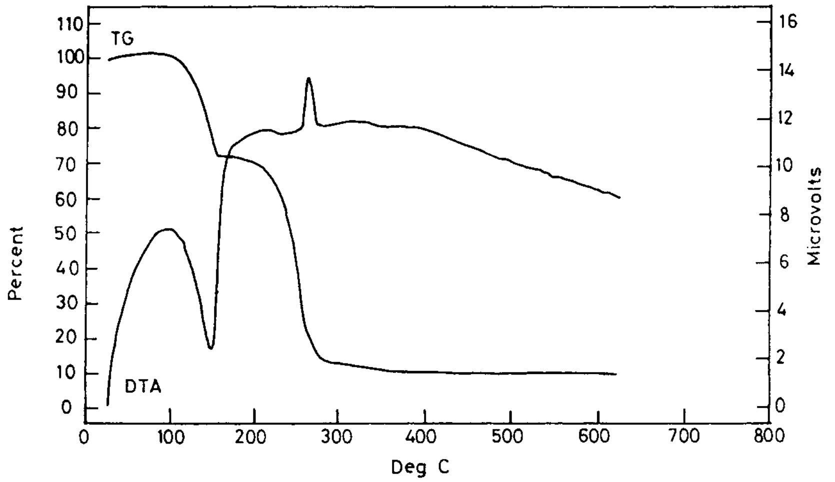

Fig. 7. Simultaneous TG–DTA of $H_5N_2OOCCH(H_2N)CH_2$ $COON_2H_5$.

formation. The second exotherms for the salts appear as a doublet. In the dihydrazinium malate, there is a break in the TG curve at 285°C with a mass loss of 90%. Then, the 10% mass decomposes exothermically at 400°C . This may be due to the decomposition of tar [14] which remains in the cup as shown by slow decomposition in the TG curves.

### 5.3. Dihydrazinium aspartate and glutamate

Similar modes of decomposition has been observed for these two salts but with different intermediates (Figs. 7 and 8). Both undergo melting with dehydrazination to give intermediates at 150 and 149°C, respectively. Dihydrazinium aspartate decomposes through aspartic acid intermediate, whereas glutamate through glycine intermediate. The thermal decomposition of glycine observed at 235°C is in good agreement with the literature decomposition temperature of 233°C [18]. Then, the free amino acids decompose exothermically into gaseous products within 280°C. Among the prepared salts, only these two salts decompose at low temperatures.

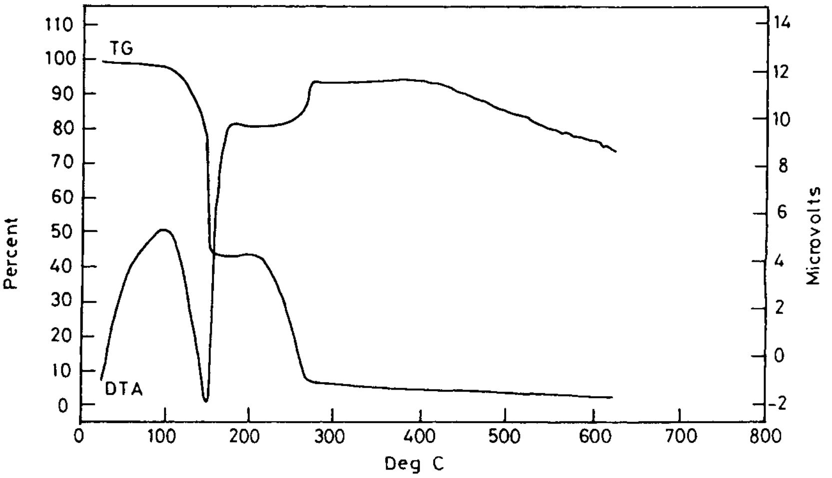

Fig. 8. Simultaneous TG–DTA of $H_5N_2OOCCH(H_2N)(CH_2)_2COON_2H_5$.

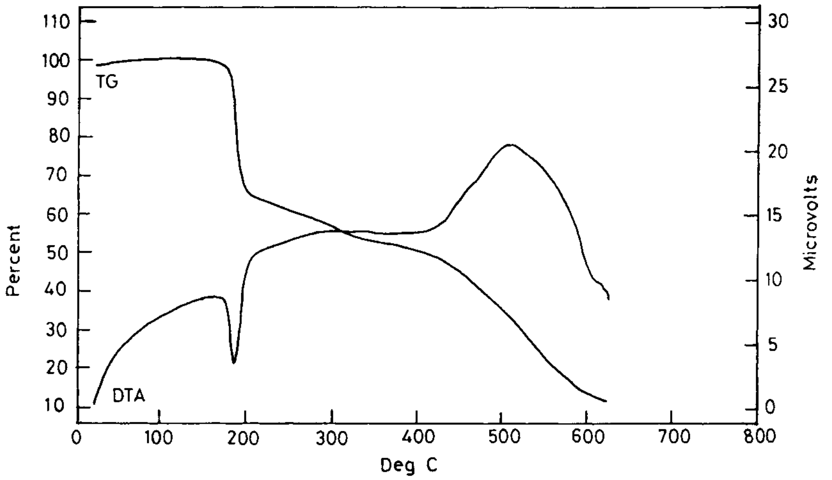

Fig. 9. Simultaneous TG–DTA of $HOOC(CH_2)_2COCOON_2H_5$.

### 5.4. Hydrazinium hydrogen α-ketoglutarate

The simultaneous TG and DTA traces of this salt are given in Fig. 9. It undergoes melting with decomposition. The TG shows a two step decomposition in accordance with DTA showing a sharp endotherm followed by a broad exotherm. The endotherm at 185°C is assigned to melting with decomposition to form succinic acid as an intermediate which further decomposes exothermically at 502°C to gaseous products.

### 5.5. Hydrazinium hydrogeniminodiacetate monohydrate

The dehydration of this salt takes place at 42°C (Fig. 10). The anhydrous salt melts at 167°C and the melt then decomposes in the temperature range of 167–200°C (TG) with an endothermic peak in DTA at 190°C. The observed mass loss corresponds to the formation of succinic anhydride which further decomposes continuously upto 600°C without leaving any residue.

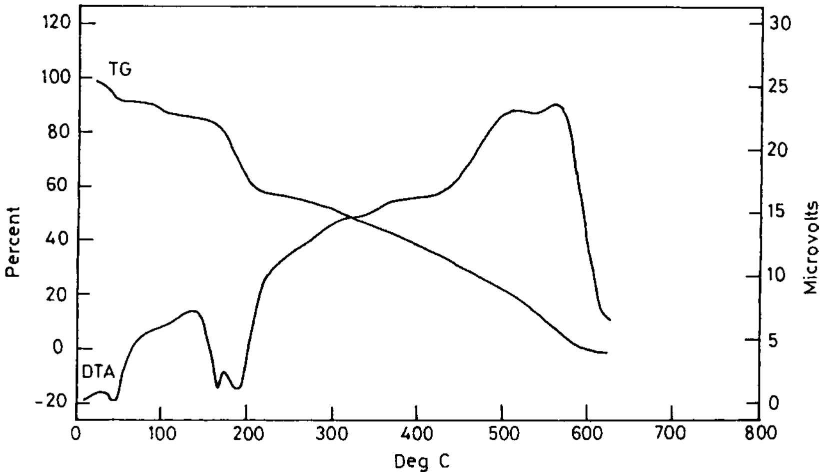

Fig. 10. Simultaneous TG–DTA of $HOOCCH_2NHCH_2COON_2H_5 \cdot H_2O$.

## 6. Conclusions

The higher homologs of dicarboxylic acids, tartaric, α-ketoglutaric and iminodiacetic acids form only monohydrazinium salts, whereas malic, aspartic and glutamic acids (all monosubstituted) mainly form dihydrazinium salts. No acid forms both mono and dihydrazinium derivatives.

The dehydrazination is exothermic in higher dicarboxylates, whereas this is endothermic (along with melting) in the other salts reported. The intermediates of the prepared hydrazinium dicarboxylates during decomposition undergo exothermic decomposition in contrast to the already reported hydrazinium dicarboxylates, which show complete endothermic decomposition [14], with the exception of oxalates [1].

The dihydrazinium salts of amino acids decompose at a lower temperature (280°C) than that of the other salts, which show complete decomposition upto 600°C.

## Acknowledgements

S.Y. thanks the Council of Scientific and Industrial Research, New Delhi, India, for the award of a Senior Research Fellowship.

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