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Note

THERMAL BEHAVIOUR OF TRIETHANOLAMMONIUM HALIDES

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Triethanolamine is a base of moderate strength and forms the bulky non-spherical triethanolammonium cation on neutratization with acids. The amine-onium salts undergo phase transitions [1] and thermal rearrangement [2] to give new product materials. Part of our research interest is to follow the thermal stability of such salts, including the study of their solid state reactions and the associated crystallographic phase transformations. The results of thermogravimetric and differential thermal analysis of triethanolammonium halides are reported in this communication.

EXPERIMENTAL

The compounds, triethanolammonium chloride, TEC, triethanolammonium bromide, TEB and triethanolammonium iodide, TEI were prepared by neutralizing 1 : 1 aqueous triethanolamine with 50% hydrohalic acids. The resultant solutions were concentrated on a water bath and cooled, thereby the separated crystalline salts were collected on a filter, and washed with acetone and ether. The elemental analyses of the salts gave satisfactory results for the compositions, TEC, $[HN(CH_2CH_2OH)_3]Cl$, TEB, $[HN(CH_2CH_2OH)_3]Br$ and TEI, $[HN(CH_2CH_2OH)_3]I$. The melting points of the salts are TEC, 192°C, TEB, 184°C; and TEI, 168°C.

The infrared spectra of the compounds were recorded on a Perkin Elmer 257 spectrophotometer using the KBr pellet technique. The X-ray powder diffraction patterns were taken on a Philips X-ray unit using Cu K_α radiation. The TG and DTA runs were made in air on a DuPont thermal analyzer using about 5 mg sample at a linear heating rate of 10°C min^{-1}.

RESULTS AND DISCUSSION

The salts are colourless plate-like crystals which are stable in air. The infrared spectra are similar to one another. The characteristic broad band centering at 3340 cm^{-1} is assigned to the stretching frequency of the OH

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TABLE 1

The interplanar spacings (d_{hkl} in Å) of triethanolammonium halides

TEC	TEB	TEI
6.32 s	6.41 s	6.46 s
4.64 s	4.72 s	4.72 m
4.27 s	4.34 s	4.39 s
3.93 m	4.00 m	4.02 w
3.10 m	3.15 s	3.21 w
2.87 m	2.90 s	2.96 m
2.53 w	2.53 m	2.58 w
2.10 w	2.14 w	2.14 w
2.05 m	2.08 s	2.07 s

s = strong, m = medium, w = weak.

group and an intense sharp band at 3140 cm^{-1} is due to ν_{NH} [3] suggesting the presence of the $[HN(CH_2CH_2OH)_3]^+$ cation. The d_{hkl} values calculated from the observed 2θ values in the X-ray diffractograms are given in Table 1. These values suggest that the three salts are isostructural and the

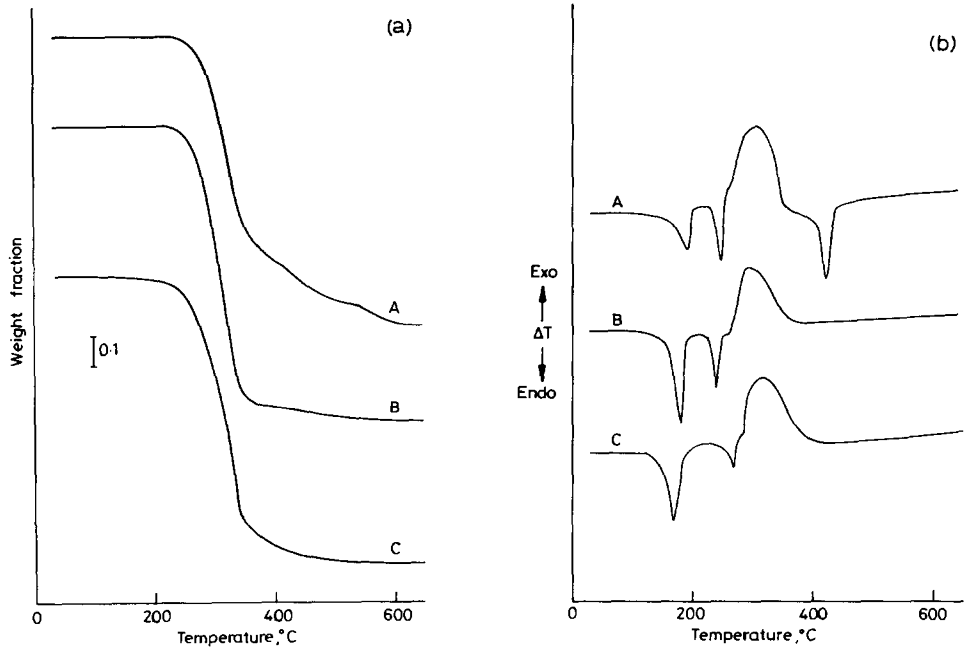

Fig. 1. (a) TG curves of (A) triethanolammonium chloride, (B) triethanolammonium bromide and (C) triethanolammonium iodide. (b) DTA curves of (A) triethanolammonium chloride, (B) triethanolammonium bromide and (C) triethanolammonium iodide.

values are in agreement with those of TEC reported in the literature [4].

The TG and DTA curves of the salts are given in Fig. 1a and b. The salts start decomposing at 215 (TEC), 210(TEB) and 200°C(TEI) which indicates that the thermal stability is of the order chloride > bromide > iodide. TEC loses 65% of its initial weight in the temperature range 215–350°C and thereafter the weight loss is sluggish. TEB and TEI on the other hand, lose about 90% of the weight in the range 200–350°C and in all the cases the residue left behind at 600°C is negligible.

The endothermic effects peaking at 193, 183 and 168°C are ascribed to the melting of TEC, TEB and TEI, respectively. An endotherm around 250°C is immediately followed by a broad exotherm with the maximum around 350°C for all the salts which have been ascribed to the decomposition process. The characteristic exothermicity of the decomposition is probably due to the oxidation of the triethanolammonoum cation in air. An endotherm at 420°C observed for TEC is due to the decomposition of the residual organic moiety after its initial decomposition.

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