Note

MASS SPECTRAL AND THERMAL DECOMPOSITION STUDIES OF PIPERZINIUM DIPERCHLORATE

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Extensive studies have been made on thermal decomposition studies of perchlorates, in particular ammonium perchlorate, due to their importance as oxidizers in explosives, pyrotechnics and rocket propellants [1]. We have been interested in the thermal stabilities of amine-onium perchlorates such as morpholinium [2], ethylenediammonium [3], cetyl trimethylammonium [4] and diphenylammonium perchlorates [5]. Reported in this note are the results of thermogravimetric, differential thermal analysis and mass spectral studies of hydrated piperzinium diperchlorate.

EXPERIMENTAL

Piperzinium (diethylenediammonium) diperchlorate dihydrate $[H_2N(C_2H_4)_2NH_2](ClO_4)_2 \cdot 2H_2O$ was prepared by adding dropwise 40% perchloric acid to an aqueous solution of piperzine, kept on a magnetic stirrer till the whole solution was just acidic to litmus. The resultant solution was heated on a water bath for 15 min and cooled. The separated crystalline product was filtered through suction, washed with a small amount of acetone and dried. Analysis: C, 15.2; H, 4.4; N, 8.9%. Calculated for $C_4H_{16}N_2O_6Cl$: C, 14.87; H, 4.99; N, 8.67%.

The X-ray powder diffraction patterns obtained with a Philips diffractometer using $Cu K_{\alpha}$ radiation. The IR spectra were recorded on a Perkin-Elmer 257 spectrophotometer using KBr pellet technique. Simultaneous TG and DTA runs were made in an argon atmosphere using a Mettler thermal analyzer at a continuous heating rate of 2°C min⁻¹. Mass spectral analyses were carried out using a Varian mass spectrometer and quartz crucibles with the filament operating at 70 eV and 300 μ A.

RESULTS AND DISCUSSION

Piperzinium diperchlorate dihydrate crystals are colourless, non-hygroscopic and stable in air. The X-ray powder patterns gave the following d_{hkl}

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16 0 4 17 OH 33 18 OH2 98 26 NC 18 27 NCH 9 28 CO, NCH2 61 29 HNCH3 23 30 NO, H2NCH2 23 32 O2 6 35 CI 23 36 HCI 62 39 NCCH 3 40 NCCH2 3 41 HNCH3 9 42 N(CH2)2 18 43 HN(CH3)2 8 44 CO2, N2O 46 51 CIO 38 52 HCO 8 53 NCC2H3, 3 54 NC(CH2)2 3 55 HNC(CH2)2 3 56 HNC(H2)2N 15 57 HN(CH2)2N(CH2 27 80 N(CH2)2N(CH2 27 80 N(CH2)2N(CH2 27 81 HN(CH2)	m/e	m ⁺	Intensity (%)	
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56 HNCH(CH2)2 23 57 HN(CH2)2N 15 67 ClO2 100 68 HClO2 3 69 HN(CH2)2NCH 27 80 N(CH)2N(CH)2 24 81 HN(CH2)2N(CH2 25 83 H2N(CH2)2N(CH2)2 27 86 HN(CH2)2N(CH2)2 27 86 HN(CH2)2N(CH2)2 27 86 HN(CH2)2N(CH2)2 27 93 N(CH2)2N(CH2)2 27 86 HN(CH2)2N(CH2)2 12 93 N(CH2)2N(CH2)2 12 93 N(CH3NNC 3 100 HO(CD4 42 107 HN(CH3NNCH3 4 110 HN(CH3NNCH3 4 114 HN(CH3NNCH3 13 119 N(CH4NNCH2 7 110 HN(CH3NNCC3 13 121 N(CH3NNNCC42 6 122 HN(CH3NNH(CH)2 25 133 (CH4NN(CH3CH2)2 5 134 (CH4NN(CH3N	55	$HNC(CH_2)_2$	3	
57 HN(CH ₂) ₂ N 15 67 ClO ₂ 100 68 HClO ₂ 3 69 HN(CH ₂) ₂ NCH 27 80 N(CH) ₂ N(CH) ₂ 24 81 HN(CH ₂) ₂ NCH 25 83 H ₂ N(CH) ₂ N(CH) ₂ 83 85 HN(CH ₂) ₂ NH(CH ₂) ₂ 27 86 HN(CH ₂) ₂ NH(CH ₂) ₂ 12 93 N(CH) ₂ N(CH) ₂ ·N 6 100 HClO ₄ 42 107 HN(CH) ₄ N·NCH 19 108 HN(CH) ₄ N·NCH ₃ 4 114 HN(CH) ₄ N·NCH ₃ 13 119 N(CH) ₄ N·NCH ₃ 13 120 N(CH) ₄ N·NC(H) ₂ 25 133 (CH) ₄ N·N(CH) ₂ 6 121 N(CH) ₄ N·N(CH) ₂ 6 122 HN(CH) ₄ N·N(CH) ₂ 5 133 (CH(₄ N·N(CH) ₂) ₄ 3 164 H ₂ N(CH) ₄ N·N(CH) ₂ / ₄ 3 164 H ₂ N(CH) ₄ N·N(CH) ₂ / ₄ NH 2	56	HNCH(CH ₂) ₂	23	
67 ClO_2 100 68 $HClO_2$ 3 69 $HN(CH_2)_2NCH$ 27 80 $N(CH)_2N(CH)_2$ 24 81 $HN(CH_2)_2N(CH_2)_2$ 25 83 $H_2N(CH)_2N(CH_2)_2$ 27 86 $HN(CH_2)_2N(CH_2)_2$ 27 86 $HN(CH_2)_2N(CH_2)_2$ 12 93 $N(CH)_2N(CH)_2 \cdot N$ 6 100 HCO_4 42 107 $HN(CH)_4N \cdot NCH$ 19 108 $HN(CH)_4N \cdot NCH_2$ 7 110 $HN(CH)_4N \cdot NCH_3$ 13 119 $N(CH)_4N \cdot NCH_3$ 13 120 $N(CH)_4N \cdot NCH_2$ 10 121 $N(CH)_4N \cdot N(CH)_2$ 10 121 $N(CH)_4N \cdot NH(CH)_2$ 25 133 $(CH)_4N \cdot N(CH)_3CH_2$ 6 134 $(CH(_4N \cdot N(CH)_2(CH_2)_2)_2$ 5 136 $(CH)_4N \cdot N(CH)_2(MH_2)_4$ 3 164 $H_2N(CH)_4N \cdot N(CH)_4NH_2$ 4 170 $HN(CH)_2(A \cdot N(CH)_2(A + A))_4NH_2$ 4	57	HN(CH ₂),N	15	
68 HClO ₂ 3 69 HN(CH ₂) ₂ NCH 27 80 N(CH) ₂ N(CH) ₂ 24 81 HN(CH) ₂ N(CH) ₂ 25 83 H ₂ N(CH) ₂ N(CH ₂) ₂ 27 86 HN(CH ₂) ₂ N(CH ₂) ₂ 27 86 HN(CH ₂) ₂ N(CH ₂) ₂ 12 93 N(CH) ₂ N(CH) ₂ ·N 6 100 HClO ₄ 42 107 HN(CH) ₄ N·NCH 19 108 HN(CH) ₄ N·NCH ₂ 7 110 HN(CH) ₄ N·NCH ₃ 4 114 HN(CH) ₂ /N·NCH ₃ 13 119 N(CH) ₄ N·NCH ₃ 13 120 N(CH) ₄ N·NCH ₂ 6 121 N(CH) ₄ N·N(CH) ₂ 6 122 HN(CH) ₄ N·N(CH) ₂ 5 133 (CH) ₄ N·N(CH) ₂ (CH ₂) ₂ 5 134 (CH(₄ N·N(CH) ₂ (CH ₂) ₄ 3 164 H ₂ N(CH) ₄ N·N(CH) ₄ NH ₂ 4 170 HN(CH ₂) ₄ N·N(CH ₂) ₄ NH 2	67	ClO ₂	100	
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	68	HCIO	3	
80 N(CH) ₂ N(CH) ₂ 24 81 HN(CH) ₂ N(CH) ₂ 25 83 H ₂ N(CH) ₂ NH(CH) ₂ 83 85 HN(CH ₂) ₂ N(CH ₂) ₂ 27 86 HN(CH ₂) ₂ NH(CH ₂) ₂ 12 93 N(CH) ₂ N(CH) ₂ ·N 6 100 HClO ₄ 42 107 HN(CH) ₄ NNC 3 108 HN(CH) ₄ N·NCH 19 109 HN(CH) ₄ N·NCH ₃ 4 110 HN(CH) ₄ N·NCH ₃ 13 110 HN(CH) ₄ N·NCH ₃ 13 120 N(CH) ₄ N·N·C ₂ H 13 120 N(CH) ₄ N·N(CH) ₂ 6 121 N(CH) ₄ N·N(CH) ₂ 6 122 HN(CH) ₄ N·N(CH) ₂ 5 133 (CH) ₄ N·N(CH) ₂ (CH ₂) ₂ 5 134 (CH(₄ N·N(CH) ₂) ₄ 3 164 H ₂ N(CH) ₄ N·N(CH ₂) ₄ 3 164 H ₂ N(CH) ₄ N·N(CH ₂) ₄ NH 2	69	HN(CH ₂),NCH	27	
81 HN(CH) ₂ N(CH) ₂ 25 83 H ₂ N(CH) ₂ NH(CH) ₂ 83 85 HN(CH ₂) ₂ N(CH ₂) ₂ 27 86 HN(CH ₂) ₂ NH(CH ₂) ₂ 12 93 N(CH) ₂ N(CH) ₂ ·N 6 100 HClO ₄ 42 107 HN(CH) ₄ NNC 3 108 HN(CH) ₄ N·NCH ₂ 7 110 HN(CH) ₄ N·NCH ₃ 4 114 HN(CH) ₂ N·NCH ₃ 13 120 N(CH) ₄ N·NC ₂ H 13 120 N(CH) ₄ N·N·C ₂ H 13 120 N(CH) ₄ N·N(CH) ₂ 6 121 N(CH) ₄ N·N(CH) ₂ 6 122 HN(CH) ₄ N·N(CH) ₂ CH ₂) ₂ 5 133 (CH) ₄ N·N(CH) ₂ (CH ₂) ₂ 5 134 (CH(₄ N·N(CH) ₂ (CH ₂) ₂ 5 136 (CH) ₄ N·N(CH) ₂ (M) ₄ NH ₂ 4 170 HN(CH) ₂ N·N(CH) ₂ MNH 2	80	$N(CH)_2 N(CH)_2$	24	
83 $H_2N(CH)_2NH(CH)_2$ 83 85 $HN(CH_2)_2N(CH_2)_2$ 27 86 $HN(CH_2)_2NH(CH_2)_2$ 12 93 $N(CH)_2N(CH)_2 \cdot N$ 6 100 $HClo_4$ 42 107 $HN(CH)_4NNCC$ 3 108 $HN(CH)_4N \cdot NCH$ 19 109 $HN(CH)_4N \cdot NCH_2$ 7 110 $HN(CH)_4N \cdot NCH_3$ 4 114 $HN(CH)_2 \cdot N \cdot C_2 H$ 13 120 $N(CH)_4N \cdot N \cdot C_2 H$ 13 120 $N(CH)_4N \cdot N \cdot (CH)_2$ 6 121 $N(CH)_4N \cdot NH(CH)_2$ 25 133 $(CH)_4N \cdot N(CH)_3CH_2$ 6 134 $(CH(_4N \cdot N(CH)_2(CH_2)_2)_2$ 5 136 $(CH)_4N \cdot N(CH)_4N \cdot N(CH)_4NH_2$ 4 170 $HN(CH)_2 A \cdot N(CH)_2 ANH$ 2	81	HN(CH) ₂ N(CH) ₃	25	
85 $HN(CH_2)_2N(CH_2)_2$ 27 86 $HN(CH_2)_2NH(CH_2)_2$ 12 93 $N(CH)_2N(CH)_2 \cdot N$ 6 100 $HCIO_4$ 42 107 $HN(CH)_4NNC$ 3 108 $HN(CH)_4N \cdot NCH$ 19 109 $HN(CH)_4N \cdot NCH_2$ 7 110 $HN(CH)_4N \cdot NCH_3$ 4 114 $HN(CH)_4N \cdot NCH_3$ 13 119 $N(CH)_4N \cdot N \cdot C_2H$ 13 120 $N(CH)_4N \cdot N \cdot (CH)_2$ 6 121 $N(CH)_4N \cdot NH(CH)_2$ 6 122 $HN(CH)_4N \cdot NH(CH)_2$ 5 133 $(CH)_4N \cdot N(CH)_3CH_2$ 6 134 $(CH(_4N \cdot N(CH)_2(CH_2))_2$ 5 136 $(CH)_4N \cdot N(CH)_2(A)_4$ 3 164 $H_2N(CH)_4N \cdot N(CH)_2(A)_4$ 4 170 $HN(CH)_2(A) \cdot N(CH)_2(A)_4$ 2	83	H,N(CH),NH(CH),	83	
86 HN(CH ₂) ₂ NH(CH ₂) ₂ 12 93 N(CH) ₂ N(CH) ₂ ·N 6 100 HClO ₄ 42 107 HN(CH) ₄ NNC 3 108 HN(CH) ₄ N·NCH 19 109 HN(CH) ₄ N·NCH ₂ 7 110 HN(CH) ₄ N·NCH ₃ 4 114 HN(CH) ₄ N·NCH ₃ 13 119 N(CH) ₄ N·N·C ₂ H 13 120 N(CH) ₄ N·N·(CH) ₂ 6 121 N(CH) ₄ N·NH(CH) ₂ 6 122 HN(CH) ₄ N·N(CH) ₃ CH ₂ 6 133 (CH) ₄ N·N(CH) ₂ (CH ₂) ₂ 5 136 (CH) ₄ N·N(CH) ₂ (CH ₂) ₄ 3 164 H ₂ N(CH) ₄ N·N(CH) ₄ NH ₂ 4 170 HN(CH ₂) ₄ N·N(CH ₂) ₄ NH 2	85	$HN(CH_2)_2N(CH_2)_2$	27	
93 $N(CH)_2 N(CH)_2 \cdot N$ 6100 $HCIO_4$ 42107 $HN(CH)_4 NNC$ 3108 $HN(CH)_4 N \cdot NCH$ 19109 $HN(CH)_4 N \cdot NCH_2$ 7110 $HN(CH)_4 N \cdot NCH_3$ 4114 $HN(CH)_4 N \cdot NCH_3$ 13119 $N(CH)_4 N \cdot N \cdot C_2 H$ 13120 $N(CH)_4 N \cdot N \cdot (CH)_2$ 6121 $N(CH)_4 N \cdot NH(CH)_2$ 25133 $(CH)_4 N \cdot N(CH)_3 CH_2$ 6134 $(CH(_4 N \cdot N(CH)_2(CH_2)_2)_2$ 5136 $(CH)_4 N \cdot N(CH)_2 NH_2$ 4170 $HN(CH)_2 N \cdot N(CH)_2 NH$ 2	86	HN(CH ₂) ₂ NH(CH ₂) ₂	12	
100 HClO ₄ 42 107 HN(CH) ₄ NNC 3 108 HN(CH) ₄ N·NCH 19 109 HN(CH) ₄ N·NCH ₂ 7 110 HN(CH) ₄ N·NCH ₃ 4 114 HN(CH) ₄ N·NCH ₃ 13 119 N(CH) ₄ N·N·C ₂ H 13 120 N(CH) ₄ N·N·(CH) ₂ 6 121 N(CH) ₄ N·NH(CH) ₂ 6 122 HN(CH) ₄ N·NH(CH) ₂ 6 133 (CH) ₄ N·N(CH) ₃ CH ₂ 6 134 (CH(₄ N·N(CH) ₂ (CH ₂) ₂) 5 136 (CH) ₄ N·N(CH) ₂ (M) ₄ NH ₂ 4 170 HN(CH ₂) ₄ N·N(CH ₂) ₄ NH 2	93	N(CH) ₂ N(CH) ₂ ·N	6	
107HN(CH) ₄ NNC3 108 HN(CH) ₄ N·NCH19 109 HN(CH) ₄ N·NCH ₂ 7 110 HN(CH) ₄ N·NCH ₃ 4 114 HN(CH) ₂) ₄ N·NCH ₃ 13 119 N(CH) ₄ N·N·C ₂ H13 120 N(CH) ₄ N·N·(CH) ₂ 10 121 N(CH) ₄ N·NH(CH) ₂ 6 122 HN(CH) ₄ N·NH(CH) ₂ 6 133 (CH) ₄ N·N(CH) ₃ CH ₂ 6 134 (CH(₄ N·N(CH) ₂ (CH ₂) ₂)5 136 (CH) ₄ N·N(CH) ₂ (H) ₄ NH ₂ 4 170 HN(CH ₂) ₄ N·N(CH ₂) ₄ NH2	100	HClO ₄	42	
108 $HN(CH)_4N \cdot NCH$ 19109 $HN(CH)_4N \cdot NCH_2$ 7110 $HN(CH)_4N \cdot NCH_3$ 4114 $HN(CH)_2AN \cdot NCH_3$ 13119 $N(CH)_4N \cdot N \cdot C_2H$ 13120 $N(CH)_4N \cdot N \cdot (CH)_2$ 10121 $N(CH)_4N \cdot NH(CH)_2$ 6122 $HN(CH)_4N \cdot NH(CH)_2$ 6133 $(CH)_4N \cdot N(CH)_3CH_2$ 6134 $(CH(_4N \cdot N(CH)_2(CH_2)_2)_2$ 5136 $(CH)_4N \cdot N(CH)_4NH_2$ 4170 $HN(CH)_2AN \cdot N(CH)_2ANH_2$ 2	107	HN(CH) ₄ NNC	3	
109 $HN(CH)_4N \cdot NCH_2$ 7 110 $HN(CH)_4N \cdot NCH_3$ 4 114 $HN(CH)_2_4N \cdot NCH_3$ 13 119 $N(CH)_4N \cdot N \cdot C_2H$ 13 120 $N(CH)_4N \cdot N \cdot (CH)_2$ 10 121 $N(CH)_4N \cdot NH(CH)_2$ 6 122 $HN(CH)_4N \cdot NH(CH)_2$ 25 133 $(CH)_4N \cdot N(CH)_3CH_2$ 6 134 $(CH(_4N \cdot N(CH)_2(CH_2)_2)_2$ 5 136 $(CH)_4N \cdot N(CH)_4NH_2$ 4 170 $HN(CH)_2(_AN \cdot N(CH)_2)_4NH_2$ 2	108	HN(CH)₄N·NCH	19	
110HN(CH)_4N · NCH_34114HN(CH)_2_4N · NCH_313119N(CH)_4N · N · C_2H13120N(CH)_4N · N · (CH)_210121N(CH)_4N NH(CH)_26122HN(CH)_4N · NH(CH)_225133(CH)_4N · N(CH)_3CH_26134(CH(_4N · N(CH)_2(CH_2)_2)5136(CH)_4N · N(CH)_4NH_24170HN(CH)_2_4N · N(CH)_2_4NH2	109	HN(CH) ₄ N·NCH ₂	7	
114 $HN(CH_2)_4N \cdot NCH_3$ 13119 $N(CH)_4N \cdot N \cdot C_2H$ 13120 $N(CH)_4N \cdot N \cdot (CH)_2$ 10121 $N(CH)_4NNH(CH)_2$ 6122 $HN(CH)_4N \cdot NH(CH)_2$ 25133 $(CH)_4N \cdot N(CH)_3CH_2$ 6134 $(CH(_4N \cdot N(CH)_2(CH_2)_2)_2$ 5136 $(CH)_4N \cdot N(CH)_4NH_2$ 4170 $HN(CH)_2(AN \cdot N(CH_2)_4NH_2$ 2	110	HN(CH) ₄ N·NCH ₃	4	
119 $N(CH)_4 N \cdot N \cdot C_2 H$ 13120 $N(CH)_4 N \cdot N \cdot (CH)_2$ 10121 $N(CH)_4 N N H(CH)_2$ 6122 $HN(CH)_4 N \cdot NH(CH)_2$ 25133 $(CH)_4 N \cdot N(CH)_3 CH_2$ 6134 $(CH(_4 N \cdot N(CH)_2(CH_2)_2)_2$ 5136 $(CH)_4 N \cdot N(CH)_2 (H_2)_4$ 3164 $H_2 N(CH)_4 N \cdot N(CH)_2 N H_2$ 4170 $HN(CH_2)_4 N \cdot N(CH_2)_4 N H_2$ 2	114	$HN(CH_2)_4N \cdot NCH_3$	13	
120 $N(CH)_4 N \cdot N \cdot (CH)_2$ 10 121 $N(CH)_4 N N H(CH)_2$ 6 122 $HN(CH)_4 N \cdot NH(CH)_2$ 25 133 $(CH)_4 N \cdot N(CH)_3 CH_2$ 6 134 $(CH)_4 N \cdot N(CH)_2 (CH_2)_2$ 5 136 $(CH)_4 N \cdot N(CH)_4 N H_2$ 3 164 $H_2 N(CH)_4 N \cdot N(CH)_2 N H_2$ 4 170 $HN(CH_2)_4 N \cdot N(CH_2)_4 N H_2$ 2	119	$N(CH)_4 N \cdot N \cdot C_2 H$	13	
121 N(CH) ₄ NNH(CH) ₂ 6 122 HN(CH) ₄ N · NH(CH) ₂ 25 133 (CH) ₄ N · N(CH) ₃ CH ₂ 6 134 (CH(₄ N · N(CH) ₂ (CH ₂) ₂) 5 136 (CH) ₄ N · N(CH) ₄ NH ₂ 3 164 H ₂ N(CH) ₄ N · N(CH) ₂) ₄ NH ₂ 4 170 HN(CH ₂) ₄ N · N(CH ₂) ₄ NH 2	120	$N(CH)_4 N \cdot N \cdot (CH)_2$	10	
122 $HN(CH)_4 N \cdot NH(CH)_2$ 25 133 $(CH)_4 N \cdot N(CH)_3 CH_2$ 6 134 $(CH(_4 N \cdot N(CH)_2(CH_2)_2)_2$ 5 136 $(CH)_4 N \cdot N(CH)_2 H_4$ 3 164 $H_2 N(CH)_4 N \cdot N(CH)_2 H_4$ 4 170 $HN(CH_2)_4 N \cdot N(CH_2)_4 NH$ 2	121	N(CH) ₄ NNH(CH) ₂	6	
133 $(CH)_4 N \cdot N(CH)_3 CH_2$ 6 134 $(CH)_4 N \cdot N(CH)_2 (CH_2)_2$ 5 136 $(CH)_4 N \cdot N(CH)_2 H_4$ 3 164 $H_2 N(CH)_4 N \cdot N(CH)_4 N H_2$ 4 170 $HN(CH_2)_4 N \cdot N(CH_2)_4 N H$ 2	122	$HN(CH)_4N \cdot NH(CH)_2$	25	
134 $(CH(_4N \cdot N(CH)_2(CH_2)_2)_2$ 5 136 $(CH)_4N \cdot N(CH_2)_4$ 3 164 $H_2N(CH)_4N \cdot N(CH)_4NH_2$ 4 170 $HN(CH_2)_4N \cdot N(CH_2)_4NH$ 2	133	$(CH)_4 N \cdot N(CH)_3 CH_2$	6	
136 $(CH)_4 N \cdot N(CH_2)_4$ 3164 $H_2 N(CH)_4 N \cdot N(CH)_4 N H_2$ 4170 $HN(CH_2)_4 N \cdot N(CH_2)_4 N H$ 2	134	$(CH(_4N \cdot N(CH)_2(CH_2)_2))$	5	
164 $H_2N(CH)_4N\cdot N(CH)_4NH_2$ 4170 $HN(CH_2)_4N\cdot N(CH_2)_4NH$ 2	136	$(CH)_4 N \cdot N(CH_2)_4$	3	
170 $HN(CH_2)_4N \cdot N(CH_2)_4NH$ 2	164	$H_2N(CH)_4N\cdot N(CH)_4NH_2$	4	
	170	$HN(CH_2)_4N \cdot N(CH_2)_4NH$	2	

TABLE 1

Mass spectral fragments of piperzinium diperchlorate dihydrate

(Å) values: 6.91w, 6.15m, 4.98m, 4.77s, 4.13m, 3.74s, 3.65s, 3.37m, 3.21s, 3.06w, 3.01m, 2.67m, 2.56m, 2.25w, 1.78w. The IR spectrum of the compound exhibited characteristic absorptions (cm⁻¹) at 1100s, 970m, and 620s due to Cl–O stretching vibrations of ClO₄ with T_d symmetry [6]. The broad absorption band at 3480 and a medium intensity band at 1630 cm⁻¹ are the characteristics of lattice water. The absorption frequencies at 3180s and 1595s are attributed to the $-NH_2$ groups of the piperzinium cation.

The TG and DTA curves of piperzinium diperchlorate are reproduced in Fig. 1. It is quite evident that the compound loses weight in two stages. The first stage, registering a loss of 9.5%, occurs in the temperature range $55-170^{\circ}$ C. The weight loss observed is attributed to the dehydration process as it corresponds to the calculated weight loss of 11% for the removal of two molecules of water from the parent compound. This inference is supported by the appearance of an endothermic peak around 75° C in the DTA curve. The anhydrous material is found to be stable up to 240° C and does not seem to rehydrate on exposure to cooled atmosphere. In the second stage, between 240 and 280° C, the compound decomposes with a mild explosion and the reaction is found to be highly exothermic. No residue was left behind at 280° C, thereby all the organic moiety was oxidized into gaseous products. The DTA plot exhibits a sharp exothermic peak at 270° C.

The mass spectral results of the salt obtained at 280° C along with the probable assignment of the peaks are given in Table 1. There is no molecular peak of the salt (m/e 187), which supports the theory that -onium type salts cannot be volatilized as such but they undergo decomposition into neutral particles which subsequently ionize and vaporize [7]. Thus, the major fragmentation routes are perchloric acid and its decomposition products and, piperzine and its decomposition products. In addition, there are peaks attributed to CO, NO, CO₂ and N₂O resulting from the oxidation of the



Fig. 1. TG and DTA traces of piperzinium diperchlorate dihydrate in argon.

organic moiety by the perchlorate. Further, there are several peaks in the spectrum with mass number higher than 100. These peaks are attributed to the decomposition products of



 $(m/e \ 170)$ which result from the dimerization of the piperzine radical obtained under the experimental conditions.

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