

Unusual isomorphism in crystals of organic solvates with hydrazine and water

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FTIR spectra of compound **3**. 1
TG-MS analysis of compound **3**. 2

FTIR spectra of anhydrous hydrazine (blue curve), hydrazine hydrate (magenta curve), water (yellow curve), and crystalline $\text{Bu}_4\text{N}^+\text{AcO}^- \cdot 0.9(\text{N}_2\text{H}_4) \cdot 0.1(\text{H}_2\text{O})$ (compound **3**, black curve) were recorded in the $400 - 4000 \text{ cm}^{-1}$ region using a Spectrum-65 Perkin Elmer FTIR spectrometer. In the spectra of pure N_2H_4 and $\text{N}_2\text{H}_4 \cdot \text{H}_2\text{O}$ the N-H stretching vibrations at 3190 and 3330 cm^{-1} are observed. As expected, in the spectrum of **3** these bands are much less intensive due to strong hydrogen bonding between hydrazine and acetate anion. Both stretching and scissor vibration bands of water are clearly seen in the spectrum of **3** supporting the results of X-ray diffractions studies.

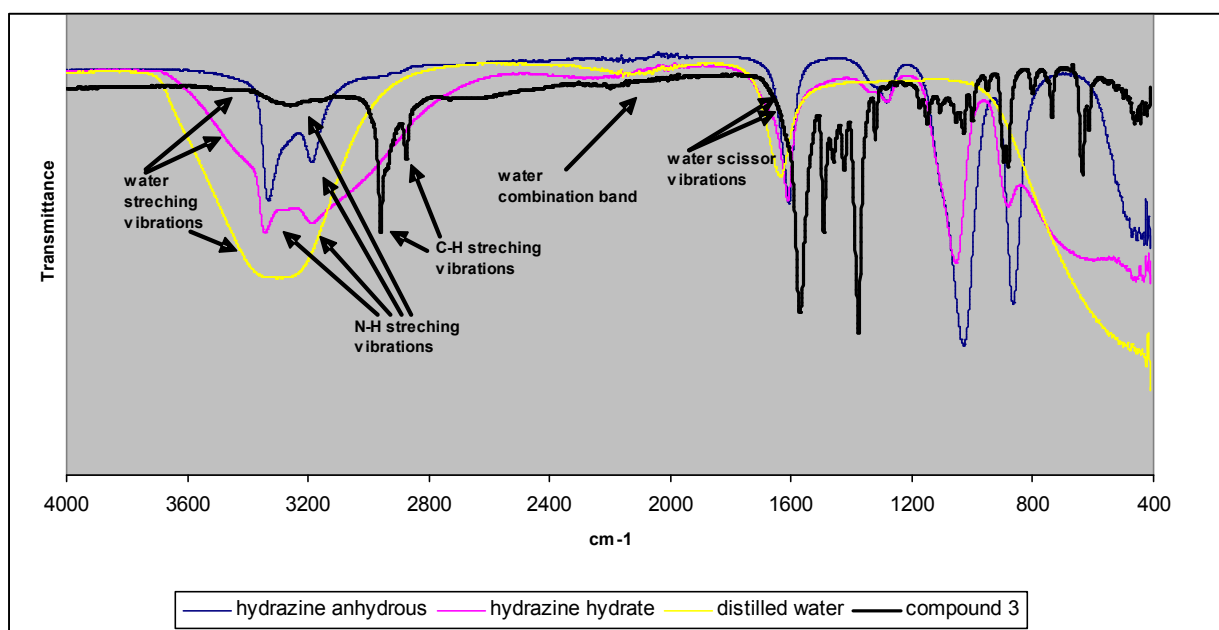


Figure 1. FTIR spectra of of anhydrous hydrazine (blue curve), hydrazine hydrate (magenta curve), water (yellow curve), and crystalline $\text{Bu}_4\text{N}^+\text{AcO}^- \cdot 0.9(\text{N}_2\text{H}_4) \cdot 0.1(\text{H}_2\text{O})$ (compound **3**, black curve).

The TGA measurements were performed on a NETZSCH TG 209F1 instrument in Al₂O₃ crucibles at a heating rate of 2 °C/min in the temperature range 35 - 150°C in argon flow (Ar > 99.998%). Evolved gas mass spectra were collected on a QMS 403C Aëolos mass spectrometer. The ionizing electron energy was 70 eV. A sample of **3** (24.73 mg) was weighed on a SARTORIUS RESEARCH R 160P analytical balance with an accuracy of 1•10⁻² mg.

The sample started to lose its weight from the very beginning of the measurement. Ions with m/z = 18 and 32 were attributed to (H₂O)⁺ and (N₂H₄)⁺, respectively. Hydrazine / water ratio is close to 8 over the entire spectra supporting the composition Bu₄N⁺AcO⁻•0.9(N₂H₄)•0.1(H₂O) found from X-ray experiment.

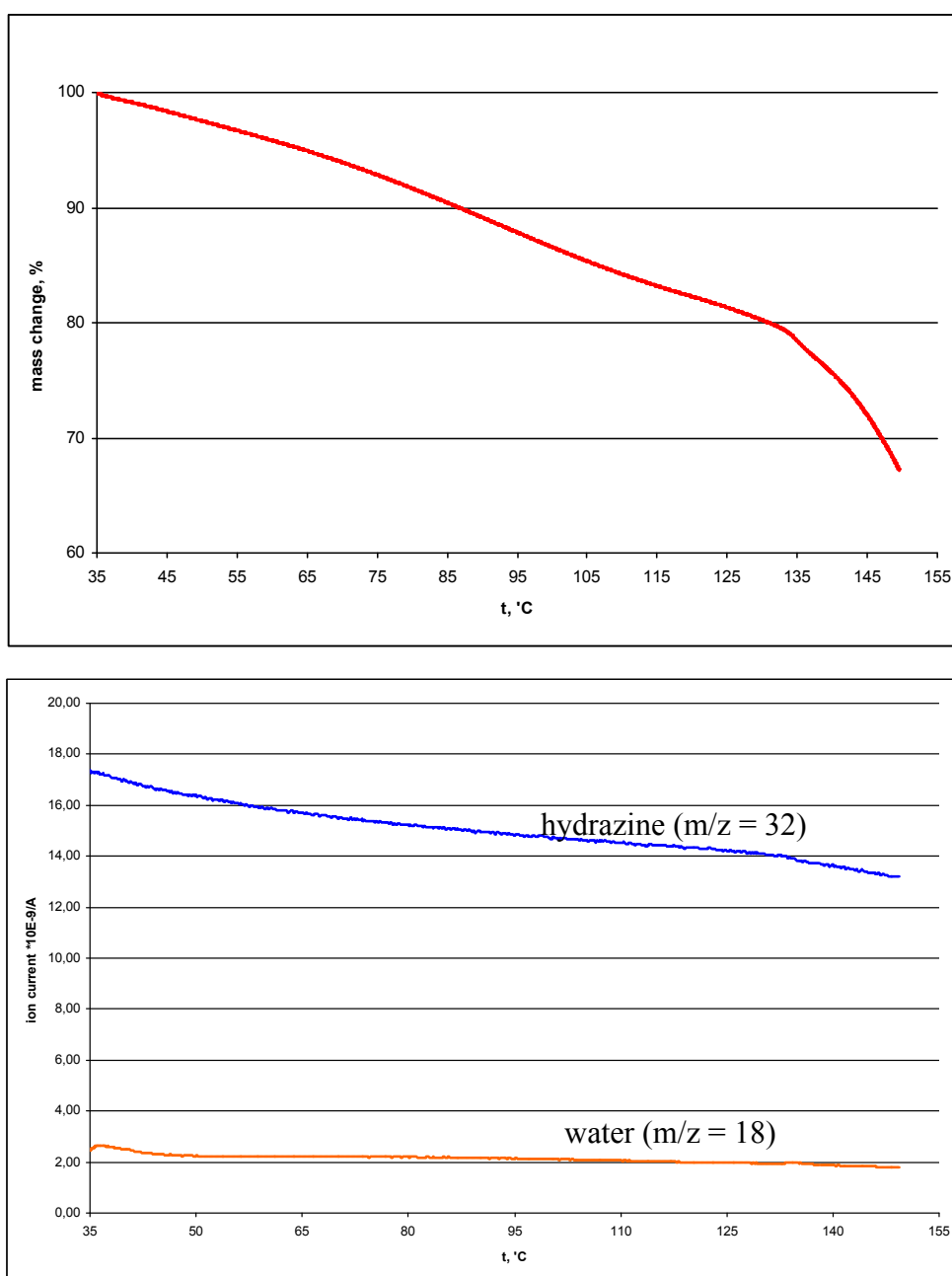


Figure 2. TG-MS analysis of compound **3**. Hydrazine / water ratio is close to 8 over the entire spectra.