Thermal analysis of crown ether complexes

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Stability properties of crown ether complexes have been examined by thermoanalytical methods. Simultaneous TGA-DTA (Thermogravimetric Analysis – Differential Thermal Analysis), DSC (Differential Scanning Calorimetry) and hot stage microscopy (HSM) meaurements have been completed.

The examined complexes contain benzylammonium- [BA], (*R*)-(+)-á-phenylethylammonium- [(*R*)-PEA] and (*R*)-(+)-, and (*S*)-(-)-á-(1-naphthyl)ethylammonium perchlorate [(*R*)-NEA and (*S*)-NEA] salts as guests. In the cases of the BA and (*R*)-PEA an achiral pyridono-18-crown-6 ligand, and in the case of the (*R*)-NEA and (*S*)-NEA a chiral (*R,R*)-dimethylphenazino-18-crown-6 ligand [(*R,R*)-DMP18C6] were used as host molecules to obtain four different crown ether complexes.

In all cases, the melting points of the complexes were much higher than those of both the host and the guest compounds, which prove the high thermal stability of the complexes. The decomposition of the complexes begins immediately after their melting is completed, while the salts and the crown ether ligands are thermally stable by 50 to 100°C above their melting points. During the decomposition of BA and (*R*)-PEA and all the four complexes a strongly exothermic process can be observed which is due to oxidative reactions of the perchlorate anion.

BA was observed to exhibit a reversible phase transition upon heating.

The heterochiral complex consisting of (*S*)-NEA and (*R,R*)-DMP18C6 shows an interesting thermal behaviour. It has a solid-solid phase transition followed by two melting points. HSM observations identified two different, simultaneously existing crystal modifications.

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