

A bug in enantiomer separation: double salt formation – diastereomeric and double salt structures of 1-cyclohexylethylammonium 2- and 4-chloromandelate

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Crystal structures of diastereomeric salt pairs, as well as double salts are rare to find in the literature [1, 2]. Present work involves the crystallization and structural elucidation of two constitutional isomer double salts along with their related diastereomeric salt pairs. The investigated systems are 1-cyclohexylethylammonium 2-chloromandelate (S-S, R-S, SS-SR) and 1-cyclohexylethylammonium 4-chloromandelate (R-R, S-R, SS-SR). Alongside structural elucidation, the thermal properties of all diastereomers and double salts have been determined and compared. In the crystal of five of the six chiral salts, hydrogen bonded layers are formed with the participation of the ionic groups and the hydroxyl group of the mandelate anion. In one structure, the formation of one-dimensional hydrogen bonded columns is observed. Due to the different position of the chlorine substituent in the two compound families, the halogen interactions are oriented towards the inside of the hydrogen-bonded structures or positioned between the layers and establish a relatively strong connection between them. The two different halogen positions and every possible combination of configurations in the six investigated salts provide a quite detailed landscape of the effect of stereochemistry on the solid-state structure of the salts.

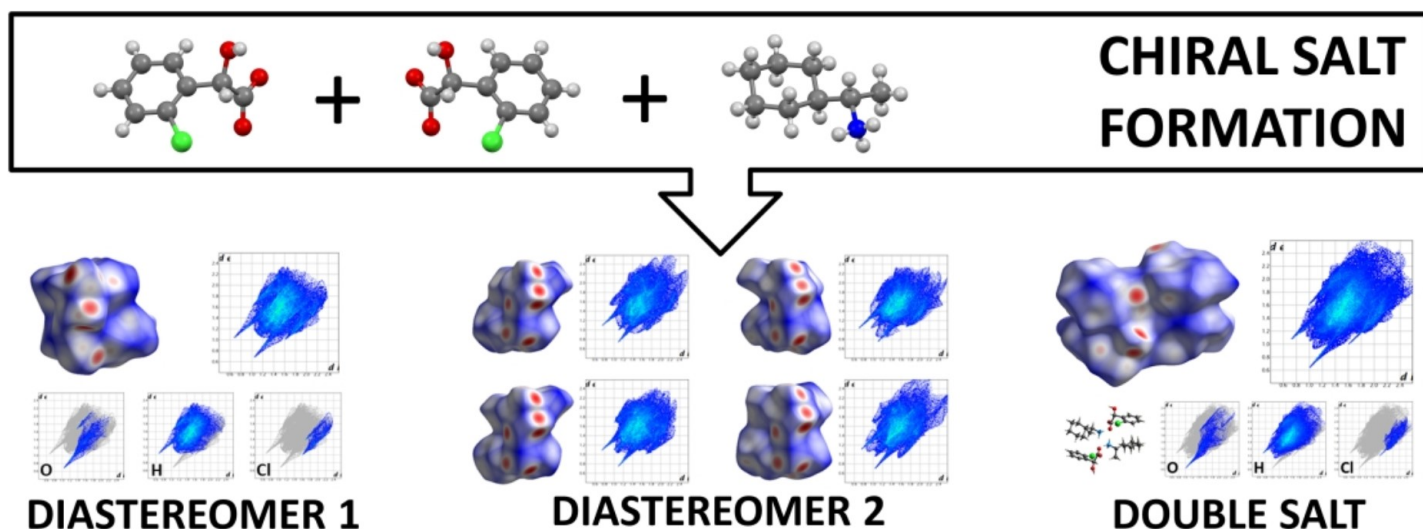


Figure 1. Hirshfeld surfaces and fingerprint plots for the optical isomers.

[1] Langkilde, A., Oddershede, J. & Larsen, S. (2002). *Acta Crystallogr B*. **58**, 1044.

[2] Peeters, O. M., Blaton, N. M. & De Ranter, C. J. (1997) *Acta Cryst C*. **53**, 597.

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