

MS30-04 | OPTICAL RESOLUTION OF 2- AND 4-CHLOROMANDELIC ACIDS WITH CYCLOHEXYLETHYLAMINE RESOLVING AGENT—CRYSTAL STRUCTURES OF THE DIASTEREOMERS AND THE DOUBLE SALT

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Racemic mandelic acid can efficiently be resolved with cyclohexylethylamine. From this starting point, cyclohexylethylamine can be considered as an eligible resolving agent for 2- and 4-chloromandelic acids. Both target compounds are important pharmaceutical and agrochemical intermediates and are utilized in enantiopure form. In spite of this, the resolution experiments showed that neither of the chloromandelic acids can be resolved with cyclohexylethylamine. In both cases, the double salt (containing the racemic chloromandelic acid and the resolving agent in optically pure form) will crystallize. The attainable enantiomeric excess is practically zero. In the cases of the optical resolutions of 2- and 4-chloromandelic acids with cyclohexylethylamine resolving agent, the crystal structures of both diastereomers and the double salt have been determined by single crystal X-ray diffraction and compared. The structural background of the formation of the double salt has been explored. The physico-chemical properties of the diastereomers and the double salt have been correlated with the structures.

The crystal structure of the less soluble diastereomer had in both cases high symmetry and crystal density. The more soluble diastereomer had in both cases the least dense structure, more than one ion pair in the asymmetric unit and eventually disordered molecular fragments. The shortest hydrogen bond interactions were formed in the structures of the double salts corresponding to a lower solubility and higher melting enthalpy as compared to the diastereomers.