

Discrimination of Chiral Guests by Chiral Channels: Variable Temperature Studies by SXR D and Solid State ¹³C NMR of the Deoxycholic Acid Complexes of Camphorquinone and Endo-3-Bromocamphor

ABSTRACT

3 α ,12 α -Dihydroxy-5 β -cholan-24-oic acid (deoxycholic acid DCA) is able to discriminate between the R- and S-enantiomers of camphorquinone and endo-(1)-3-bromocamphor and select only the S-enantiomers from a racemic mixture. DCA forms novel well ordered 1:1 adducts with (1S)-(1)-camphorquinone and (1S)-endo-(-)-3-bromocamphor, both of which have been characterized by single crystal X-ray diffraction (SXR D). When DCA is cocrystallized with (RS)-camphorquinone and (RS)-endo-3-bromocamphor, 1:1 adducts of the S-enantiomers are produced together with crystals of the free racemic guest. In contrast, in the absence of (1S)-(1)-camphorquinone, DCA forms a 2:1 adduct with (1R)-(2)-camphorquinone. In this 2:1 adduct the guest is disordered at ambient temperature and undergoes a phase change in the region 160–130 K similar to that observed for the ferrocene adduct, but with only partial ordering of the guest. The SXR D structure of the low temperature form and the variable temperature ¹³C CP/MAS NMR are reported. Cocrystallizing DCA with (1R)-endo-(1)-3-bromocamphor gives the free guest and a glassy solid.

Keyword: molecular complexes, chiral discrimination, chiral selection