Structure of a 2 : 1 Complex between Deoxycholic Acid and Ferrocene

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The structure of a 2 : 1 complex between deoxycholic acid and ferrocene has been determined by *X*-ray crystallography; this is the first example of an organometallic compound tightly included in the lattice channel of deoxycholic acid.

Deoxycholic acid (DCA), a typical bile acid, has been extensively used as a host molecule in host–guest compounds of the lattice inclusion type.[1] A variety of guest molecules have been accommodated within the channel of DCA, but until now they have been limited to organic molecules. Organometallic compounds have recently received attention as guest molecules in inclusion compounds of thiourea,[2] cryptands,[3] and cyclodextrins.[4—6] We report here the first example of a lattice-type inclusion complex between DCA and an organometallic compound, where a ferrocene molecule is tightly accommodated in the DCA channel. The crystal structure reported in this communication has a fixed orientation of ferrocene for the host molecule, so is in contrast with that of the complex between thiourea and ferrocene.[2]

The inclusion complex was crystallized from a methanol solution containing a 2 : 1 molar ratio of DCA and ferrocene.†

The crystal structure as viewed along the crystallographic *c* axis is shown in Figure 1. The arrangement of the DCA channel, including the helical hydrogen-bond system, is very

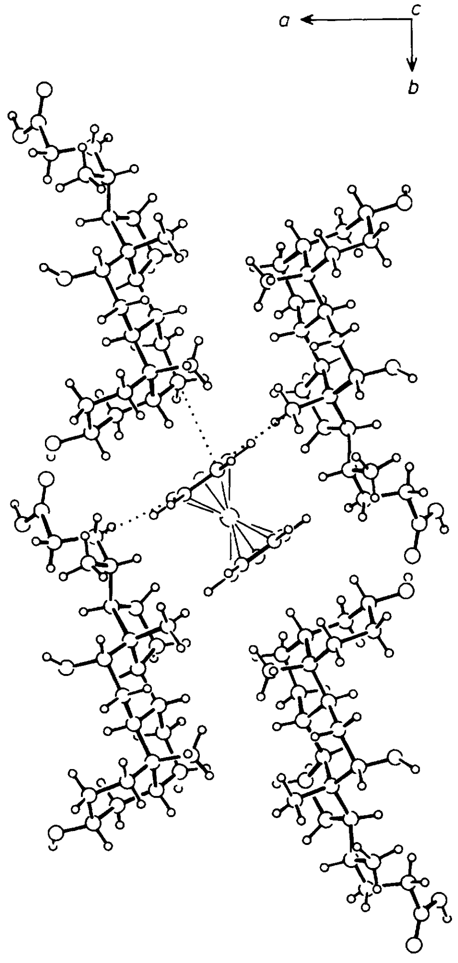

Figure 1. The crystal structure of the DCA–ferrocene complex as viewed along the crystallographic *c* axis. The short contacts between the host and guest molecules (less than 3.8 Å between non-hydrogen atoms) are indicated by broken lines.

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† *Crystal Data:* $C_{24}H_{40}O_4\cdot1/2C_{10}H_{10}Fe$, $M = 485.60$, orthorhombic, space group $P2_12_12$, $a = 27.288(10)$, $b = 13.679(3)$, $c = 7.072(3)$ Å, $Z = 4$, $D_c = 1.222$ g cm^{-3}. Intensity data were collected by the θ—2θ scan technique with $\sin\theta/\lambda$ up to 0.55 on a Rigaku automated four-circle diffractometer using graphite-monochromatized Mo-K_α radiation. Since the crystal is isomorphous with that of the complex between DCA and camphor,[7] these co-ordinates of DCA were used for the starting model of the present complex. After several cycles of isotropic refinement, the electron densities of ferrocene were easily found on the difference Fourier map. The position of the ferrocene molecule was unambiguously refined with the rigid-body of the C_5H_5 group. On the final difference Fourier map, in particular in the region of the DCA channel, there were no significant remaining peaks. These facts indicate that the orientation of the ferrocene molecule is fixed in the DCA channel. The final R factor is 0.101 for 1422 $[|F_o|>3\sigma(|F_o|)]$ reflexions. Computations were carried out on an ACOS 850 computer at the Crystallographic Research Centre, Institute for Protein Research, Osaka University. Atomic co-ordinates, bond lengths and angles, and thermal parameters have been deposited at the Cambridge Crystallographic Data Centre. See Notice to Authors, Issue No. 1.

similar to that found in the crystal structure of the DCA–camphor complex.[7] The iron atom is on a two-fold axis so the ferrocene molecule adopts the eclipsed (D_{5h}) form. It is located in the DCA channel in such a way that the plane of the C_5H_5 group is parallel to the *c* axis and inclined at 36.7° to the *ac* plane. The distances between the Fe atom and C atoms in the C_5H_5 group lie in the range of 2.01—2.05 Å. There are three non-hydrogen contacts between the ferrocene molecule and the DCA channel which are less than 3.8 Å; these are the C(6), C(18), and C(22) atoms‡ at 3.61, 3.75, and 3.70 Å, respectively, which are indicated in Figure 1.

This complex is the first example of a crystal structure of an organometallic compound accommodated in the channel of deoxycholic acid as a guest molecule. It has been reported previously that ferrocene molecules are included in channel lattices formed by thiourea[2] and in the cavities of cyclodextrins.[4] In these cases, however, the orientation of the ferrocene could not be uniquely determined by *X*-ray crystallography mainly owing to the disorder of the cyclopentadienyl ring. The present *X*-ray structure is the first case in which the orientation of ferrocene has been established in the channel of a lattice inclusion compound.

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‡ The atomic numbering system of DCA is in accord with the convention for steroid molecules (ref. 1).