and after several cycles of least squares refinement the locations of the hydrogens were established by a difference synthesis. The final R value was 0.068 for the observed reflections.

The structure consists of a 31-membered macrolide ring with an oxygen bridge between C(9) and C(13). The absolute configuration as shown in the figure was ascertained (J. A. Findlay and L. Radics, Can. J. Chem. 58, 579-590, 1980) by isolating L(-)-pipecolic acid from hydrolysis products.

03.4-02 CHOLESTERYL ESTERS : CRYSTAL AND LIQUID CRYSTALLINE STRUCTURES. Patricia Sawzik and B. M. Craven, Department of Crystallography, University of Pittsburgh, Pittsburgh, PA 15260 USA.

A series of X-ray crystal structure determinations of cholesteryl n-alkanoate (n = 2, 6, 8-12, 14) and n-alkenoate (n = 16:1$^{\Delta 9}$, 18:1$^{\Delta 9}$) esters has been undertaken, one aim being to seek features which may be relevant to molecular association in the less ordered liquid crystalline phases. The saturated cholesteryl esters with chain length C_6-C_{18} and the unsaturated palmitoleate and oleate have one of three crystal structure types as the most stable form at room temperature. These crystal structure types are designated as monolayers II (ester chain length C_6-C_9, $C^{\Delta 7}_{18:1}$), monolayers I (C_9-C_{12}, $C^{\Delta 9}_{16:1}$) and bilayers (C_{13}-C_{18}) with cholesteryl-cholesteryl, cholesteryl-alkyl, and alkyl-alkyl interactions becoming successively dominant. The X-ray diffraction patterns for the smectic phase of the cholesteryl esters suggest a relationship with the monolayer type I crystal structures. Diffracted orders from the crystal monolayers (ℓ = 2 through 5) are very weak. The strong first order has a d-spacing similar to that of the single sharp intense inner diffraction ring of the smectic phase. X-ray diffraction patterns for the cholesteric and smectic phases are similar but in the cholesteric the inner ring is more diffuse. This may be due to a short range ordering of antiparallel pairs of molecules as found in the bilayer crystal structures.

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03.4-01 PHASE TRANSITION AND 37°C CRYSTAL STRUCTURE OF CHOLESTEROL. Leh-Yeh Hsu and C. E. Nordman, Department of Chemistry, University of Michigan, Ann Arbor, MI 48109, U.S.A.

The unit cell of cholesterol ($C_{27}H_{46}O$) above the 31.6°C phase transition (Petropavlov & Kostin, Kristallografiya (1976) 21, 168) is triclinic, space group P1, with a = 27.565, b = 35.776, c = 10.748, α = 94.45, β = 90.90, γ = 73.87°, at 37°C. There are 16 independent molecules, compared to 8 in the room temperature (RT) cell (Shieh et al., Nature(1977) 267, 287; Acta Cryst., in press). A restrained-group Gauss-Seidel (FGLS) refinement procedure (Hoard & Nordman, Acta Cryst. (1979) A35, 1010) was used to deduce a refinable structure from the RT starting model. A combination of FGLS and anisotropic block-diagonal refinement presently gives R = 0.09 for 18,047 reflections. The bilayer structure of hydrogen-bonded chains of molecules bears an overall resemblance to the RT phase, differing from the latter in that several molecules have turned about their long axes by varying amounts, up to 160°. Side chain conformations also differ in the two phases. Two of the 16 molecules have side chains forming an 80° angle with the steroid long axis, a feature not previously encountered in cholesterol structures. Strong thermal motion is present in all side chains. A remarkable rotational pseudosymmetry relates eight of the sixteen independent molecules to the other eight, giving a pseudo-asymmetric unit of 8 molecules as contrasted with 4 in the RT phase.

03.4-03 CRYSTAL STRUCTURE OF THE 2:1 COMPLEX BETWEEN DEOXYCHOLIC ACID AND d-CAMPHOR. By J.G. Jones, S. Schwarzbaum, and L. Lessinger, Chemistry Dept., Barnard College, New York, USA

Bile is the source of several hydroxylated derivatives of the steroid 5β-cholan-24-oic acid which play important physiological roles in the digestion of fats and in excretion. One bile acid, deoxycholic acid (DCA), forms stoichiometric crystalline complexes with a wide variety of organic compounds. The complex 2:1 DCA:camphor crystallizes in space group $P2_12_12$ with a=27.353, b=13.814, c=7.233 Å, D_m=1.137, D_x=1.139 g/cm^3 for Z=4 of $C_{24}H_{40}O_4 \cdot \frac{1}{2}(C_{10}H_{16}O)$.

The structure was solved by direct methods and refined to R=0.07. It consists of bilayers of DCA molecules, held together by hydrogen bonds between the two halves of the bilayer, and stacked with their hydrophobic surfaces in contact. The shape of the DCA steroid is such that between adjacent bilayers are formed channels, in which the camphor molecules stack. The channels are centered on crystallographic two-fold rotation axes; the roughly spherical camphor molecules are two-fold disordered.

The structure is compared to the several other known crystal structures of DCA with hydrophobic guest molecules. While DCA forms very similar bilayers in all these structures, there are some major and some subtle differences among them. The differences which allow for the formation of DCA complexes with molecules of such widely varying sizes and shapes as camphor, phenanthrene, cyclohexanone, acetic acid, and palmitic acid will be illustrated.