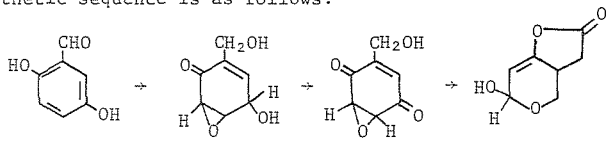
03.3-16 THE CRYSTAL STRUCTURES OF ANTIMICROBIAL PRODUCTS; HEXADECYLTRIMETHYL AMMONIUM BROMIDE AND IODIDE. By J.M. Grochowski, Jagiellonian University, Cracow, Poland and Georgetown University, Washington, DC 20057 USA

Hexadecyltrimethyl ammonium bromide (Cetrimide -The US Pharmacopea, XX Revision, 1980) is widely used in medicine and dairy industry as a fungicide and antibacterial agent. Preliminary structural investigations (Grochowski, Dissertation, 1975) indicated that crystals of both compounds are monoclinic, belonging to the symmetry class 2/m; however, their structures can be described in the centrosymmetric $P2_1/m$ group only within low accuracy reflected in a large range of CH_2-CH_2 bond lengths for the hexadecyl radical and large U_{22} temperature factors. Such apparent shortening of the CH_2-CH_2 bond lengths and enlargement of the carbon atom temperature factors was reported for several structures of a similar type (Bandoli, Clemente, Nicolini, J.Cryst.Mol. Struct. (1978) 8, 279). Further refinement of the Cetrimide structure was conducted in an enlarged unit cell consistent with very weak reflections observed on overexposed Weissenberg photographs and then measured on a diffractometer. In the proposed superstructure model in $P2_1/c$ space group, the zigzag hydrocarbon chains pack "head to tail" and are alternately twisted from the old mirror position. The direction of the long chain axis is parallel to $[\bar{4}01]$. The chain packing corresponds to two interpenetrating idealized orthorhombic lattices with a mutual shift of $\frac{1}{2}a_s + \frac{1}{2}b_s$, where; $a_s = 5.07(7)$, $b_s = b = 7.26(1)$, $c_s = 2.54(1)$Å, $a_s \parallel [1,0,22]$, $c_s \parallel [\bar{4}01]$. The bromide anion is close to the "head" methyl groups and binds four $(CH_3)_3NC_{16}H_{33}$ cations. The final refinement, including the hydrogen atoms of the $-CH_3$ groups revealed by difference Fourier synthesis and hydrogen atoms of $-CH_2-$ groups calculated from sp^3 geometry, resulted in R = 5.9%. The average CH_2-CH_2 bond length is 1.519(4)Å and the average C-C-C angle is 113(1)°. The structure of hexadecyltrimethyl ammonium iodide is basically isomorphic with Cetrimide. On overexposed Weissenberg photographs only one (0,1,23) superstructural reflection suggesting a doubled c identity period was observed. The structure was refined in $P2_1$ unit cell. Neglecting the doubled c parameter in the structure of Cetrimide, the hydrocarbon chain packing is virtually identical. However due to the larger I^- radius the subcell of long chains has dimensions $a_s = 5.13(2)$, $b_s = b = 7.55(1)$, $c_s = 2.54(3)$Å. The temperature factors for carbon atoms are larger too. The final 4.4% discrepancy factor is smaller than for Cetrimide but the CH_2-CH_2 bond length range is larger than for Cetrimide. The average CH_2-CH_2 bond length equals to 1.492(9)Å and the average C-C-C angle is 116(1)°.

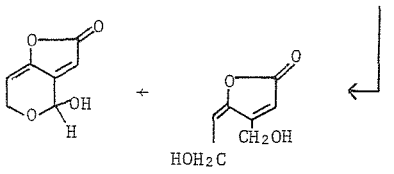
03.3-17 STRUCTURE AND CONFORMATION OF SOME POLYKETIDES FROM THE PATULIN PATHWAY. K. Ann Kerr and P.W. Codding, Departments of Chemistry and Physics, University of Calgary, Calgary, Alberta, Canada, T2N 1N4.

Patulin is a powerful antibiotic, active against both gram-positive and gram-negative bacteria but not used clinically because of its extreme toxicity and carcinogenicity. Like the antibiotics grisofulvene and tetracycline, it is a polyketide, a secondary metabolite synthesized from acetyl coenzyme A. Details of the biosynthetic pathway of patulin are important because portions of that pathway are shared by other polyketide synthesized by non-patulin-producing microbes.

In a series of papers (Biochem. J. (1979) 445-453, and references cited therein) Gaucher and coworkers have isolated and identified a number of compounds in the post-gentisaldehyde portion of the patulin pathway. The synthetic sequence is as follows:



gentisaldehyde isoepoxydon phyllostine neopatulin



patulin ascladiol

The structure of patulin has been reported (Acta. Cryst. (1977) B33, 928-931). We have obtained crystals of isoepoxydon, phyllostine, and neopatulin. In addition, we have solved the structure of the closely related patulin lactone. Structural parameters will be presented for the four compounds. NMR data will be correlated with conformational features.

Crystal Data. Patulin Lactone, $C_7H_4O_4$; FW = 152.1; a = 6.222(2); b = 5.368(2); c = 17.726(3); β = 99.52(2); ρ = 1.73; $P2_1/c$, Z = 4.