While ethanesulphonate salts of antifolate drugs crystallize well and have been extensively studied, carboxylate salts have not. Trimethoprim acetate (TA; R.C. Haltiwanger Jr., MSc Thesis, University of Virginia, 1971) is presented for comparison. Introduction of a polar group into the 5-substituent will perturb the binding to DHFR; structure (5) was examined for changes in packing relative to (1)-(4).

	a	b	c(Å)	α	β	γ°	Z	S.Gp.
1	11.103	8.398	14.652	90	100.30	90	4	$P2_1/c$
2	26.247	10.254	14.562	90	120.69	90	8	$C2/c$
3	11.723	13.186	13.899	79.35	66.29	86.34	4	$P\bar{1}$
4	9.101	9.539	14.979	84.05	74.81	74.45	2	$P\bar{1}$
5	18.213	12.385	19.179	90	116.41	90	8	C2/c

	A	B	X	C2-N2	C4-N4(Å)	τ	ω*(°)
1**			3.095	1.320	1.315	63	
2	2.670	2.773	3.054	1.323	1.322	76	38
3a	2.708	2.860	2.980	1.325	1.340	70	12
3b	2.668	2.847	2.964	1.337	1.331	76	7
4	2.72	2.75	3.06	1.38	1.37	79	27
TA	2.60	2.78	3.04	1.33	1.34		11
5**			3.030	1.324	1.325	57	

*Angle between C'COO and pyrimidine ring planes.
**Chloride salt.

The interaction of protonated ring and carboxylate ion is uniformly strong. It does not impose coplanarity, but the consistency in distance should serve as a useful anchor point for model-building.

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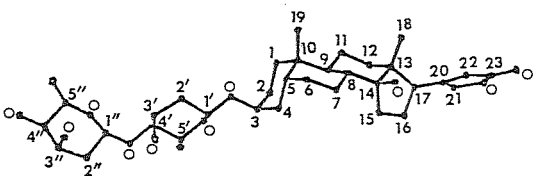
03.3–21 STRUCTURE OF DIGITOXIGENIN BISDIGITOXOSIDE, $C_{35}H_{54}O_{10}$. Kuantee Go and Gopinath Kartha, Biophysics Department, Roswell Park Memorial Institute, Buffalo, New York 14263, U.S.A.

Digitoxigenin bisdigitoxoside recrystallized from ethyl acetate and hexane is orthorhombic, $P2_12_12_1$, a=11.419(2), b=14.310(2), c=23.959(3)Å, V=3915 Å^3, Z=4.

The structure was solved by multisolution methods and refined by block diagonal least-squares to an R index of 10.4%. An ORTEP sketch of the molecule is shown below. The D-ring has a 13α,14β-half-chair conformation. The torsion angle C(13)-C(17)-C(20)-C(22) is -116°, C(21)...O(14) distance is 2.943Å. Unlike digoxin and digoxigenin bisdigitoxoside, there is no intramolecular H-bond between the OH at C(3') and the ring oxygen of the adjacent sugar; this distance is 3.982Å (longer than 3.269Å in gitoxin). There is a disordered solvent, presumably a molecule of ethyl acetate; this solvent molecule along with the OH of the cardiac steroid and those of the sugars formed H-bonds in stabilizing the structure.



03.3–22 THE CRYSTAL AND MOLECULAR STRUCTURE OF CINCHONINIUM TETRACHLOROCUPRATE 1.5-HYDRATE, $(C_{19}H_{24}N_2O)^{2+}[CuCl_4]^{2-}.1.5H_2O$. By B. J. Oleksyn and S. A. Hodorowicz, Faculty of Chemistry, Jagiellonian University, Krakow, Poland.

Cinchona alkaloids - metal ions interactions are important as factors which could modify processes undergoing in living organisms. In reaction of MCl_2, where M=Zn, Co, Cd, Hg and Cu, with cinchoninium chloride, $CinCl_2$, tetrachloro-salts of general formula: $(Cin)^{2+}[MCl_4]^{2-}.nH_2O$ were obtained (Dyrek, Polish J. Chem.(1976) 50, 2027). Preliminary crystallographic investigation showed that only the Cu^{2+} compound is not isomorphous with the others (Chojnacki, Oleksyn, Hodorowicz, Polish J. Chem. (1975) 49, 429; Oleksyn, Stadnicka , Hodorowicz, ibid. (1976) 50, 1645). To explain this we have undertaken the crystal structure determination, which was carried out for 4426 independent reflections (3335 with $|F_o| > 3\sigma(F_o)$) measured on a CAD-4 diffractometer. The positions of Cu^{2+} ions were found with Patterson method, while those of other atoms were obtained from Fourier and difference Fourier syntheses. The current R value after anisotropic refinement of non-hydrogen atoms (513 parameters) with H atoms in fixed positions, is 0.077.

The main difference between this structure and the group of isomorphous structures of $(Cin)^{2+}[MCl_4]^{2-}.nH_2O$, where M≠Cu and n=2, are the packing conditions resulting from the fact that the asymmetric unit consists of 2 salt and 3 water molecules. The N and O atoms of Cin^{2+}, Cl^- ions, and H_2O molecules form a complicated net of hydrogen bonds (10 bond kinds of length 2.72 - 3.31Å). The geometry of $[CuCl_4]^{2-}$ tetrahedrons and Cin^{2+} cations is comparable to that described for Cd salt (Oleksyn, Stadnicka, Hodorowicz, Acta Cryst. (1978) B34, 811).

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