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Received October 20, 1976.

The Crystal Structure of Succinylcholine Chloride Dihydrate

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The crystal structures of three different succinylcholine salts have earlier been determined.[1–4] Succinylcholine chloride is the only choline ester salt known to crystallize as a hydrate. It was therefore found worth-while to examine the crystal structure as part of solid state studies of choline ester salts, in which hydrogen bonding of the choline ester ion cannot *a priori* be excluded.

Bond lengths and angles calculated from the final parameters (Table 1) are shown in Fig. 1 and are in general agreement with accepted values. The conformations of the succinylcholine ions in the present crystal structure (the torsion angles are given in the legend to Fig. 1) and in the crystals of succinylcholine perchlorate[3] are approximately the same. A stereo view of the crystal packing is given in Fig. 2. The water oxygen atom is donor for two hydrogen bonds $O11-H111\cdots Cl^-_{-x,-y,1-z}$ and $O11-H112\cdots Cl^-$. The $O\cdots Cl^-$ distances are 3.300(4) and 3.255(4) Å, respectively, and the $O-H\cdots Cl^-$ angles are 170(3) and 172°(5), respectively. No hydrogen bonding involves the ester oxygen atoms O3 and O4. The ability of choline ester ions to be acceptors for hydrogen bonds seems to be low, as the same situation now has been found in several choline ester salts: Acetylcholine β-resorcylate,[5] acetylcholine (+)-bitartrate[6] (both of which have two formula units in the asymmetric unit), γ-aminobutyric acid choline ester diiodide and (±)-tartrate,[7] lactoylcholine iodide,[8] and the present structure.

Experimental. Succinylcholine chloride dihydrate appears as a white microcrystalline material. Several attempts to grow single crystals from a variety of solvents using different techniques have been unsuccessful. Finally a few transparent crystals were found among the microcrystalline material obtained by slow evaporation of an aqueous solution.

Crystal data. Succinylcholine chloride dihydrate, $C_{14}H_{34}N_2O_6Cl_2$, $M=397.34$, m.p. 190–191 °C. Space group $P\bar{1}$, $a=8.941(4)$, $b=$

Table 1. Final positional and thermal parameters. The estimated standard deviations, referring to the last figure, are given in parentheses. Thermal parameters are $\times 10^3$. The temperature factor is defined by:

$$\exp\left[-2\pi^2(U_{11}h^2a^{*2}+\cdots+2U_{12}hka^*b^*+\cdots)\right]$$

ATOM	X/A	Y/B	Z/C	U11	U22	U33	U12	U13	U23
C 1	.0895(4)	.5084(5)	.5768(3)	2.4(1)	4.5(2)	2.4(1)	0.6(1)	1.0(1)	1.1(1)
C 2	.2318(4)	.4385(4)	.5416(3)	2.5(1)	3.8(2)	2.6(2)	0.5(1)	0.8(1)	1.3(1)
O 3	.2157(3)	.3876(4)	.4108(3)	3.3(1)	6.3(1)	3.0(1)	1.2(1)	1.5(1)	1.5(1)
O 4	.3865(3)	.4383(4)	.6777(3)	2.4(1)	6.0(1)	2.9(1)	1.1(1)	1.0(1)	1.8(1)
C 5	.5245(4)	.3509(6)	.6498(4)	3.2(2)	7.1(2)	3.8(2)	1.4(2)	1.9(1)	2.6(2)
C 6	.6963(4)	.3760(5)	.8088(4)	2.6(2)	5.2(2)	4.0(2)	0.5(1)	1.5(1)	1.9(1)
N 7	.7170(3)	.2245(4)	.8963(3)	2.3(1)	3.5(1)	2.5(1)	0.6(1)	1.1(1)	0.8(1)
C 8	.5963(4)	.2570(6)	.9590(4)	3.1(2)	6.0(2)	4.1(2)	1.3(1)	2.2(1)	2.2(2)
C 9	.6908(5)	.0041(6)	.7949(5)	4.0(2)	4.2(2)	3.8(2)	0.5(1)	1.2(2)	0.0(1)
C10	.9047(4)	.2669(5)	1.0390(4)	2.4(1)	4.7(2)	3.3(2)	0.3(1)	1.0(1)	0.7(1)
O11	-.2375(4)	.0587(5)	.4630(4)	4.0(2)	9.6(2)	4.8(2)	-0.7(1)	1.9(1)	0.3(2)
CL-	.1331(1)	.1470(1)	.8119(1)	4.35(5)	5.12(5)	3.72(5)	-0.04(3)	2.03(4)	1.20(3)

ATOM	X/A	Y/B	Z/C	ATOM	X/A	Y/B	Z/C
H 11	.121(5)	.647(6)	.643(5)	H 91	.571(5)	-.026(5)	.717(5)
H 12	.085(5)	.427(5)	.638(5)	H 92	.767(5)	-.009(6)	.757(5)
H 51	.492(5)	.188(6)	.587(4)	H 93	.714(5)	-.082(6)	.860(5)
H 52	.548(5)	.439(5)	.600(5)	H101	.977(5)	.242(5)	.994(4)
H 61	.792(5)	.359(5)	.787(4)	H102	.916(5)	.414(6)	1.102(5)
H 62	.711(5)	.525(6)	.887(4)	H103	.914(5)	.176(6)	1.097(5)
H 81	.623(5)	.405(6)	1.030(5)	H111	-.200(5)	.015(6)	.407(5)
H 82	.472(5)	.224(5)	.864(5)	H112	-.149(6)	.070(6)	.538(5)
H 83	.614(5)	.170(6)	1.020(5)				

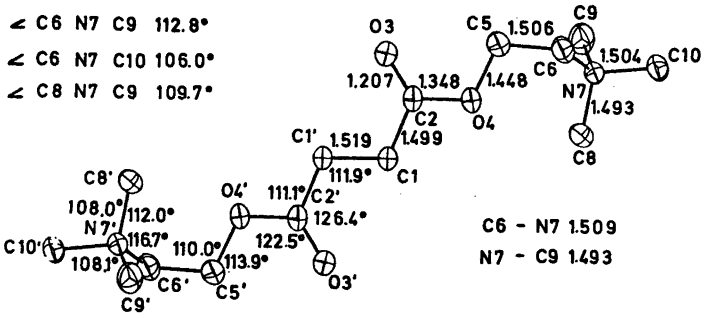

Fig. 1. The dimensions of the succinylcholine ion. The torsion angles are C1′ C1 C2 O4∓175.0°; C1 C2 O4 C5±173.6°; C2 O4 C5 C6±174.6°; O4 C5 C6 N7±78.8°; C5 C6 N7 C8∓68.8°. The estimated standard deviations on bond lengths and angles are about 0.005 Å and 0.3°, respectively. The drawings were produced by ORTEP.[12]

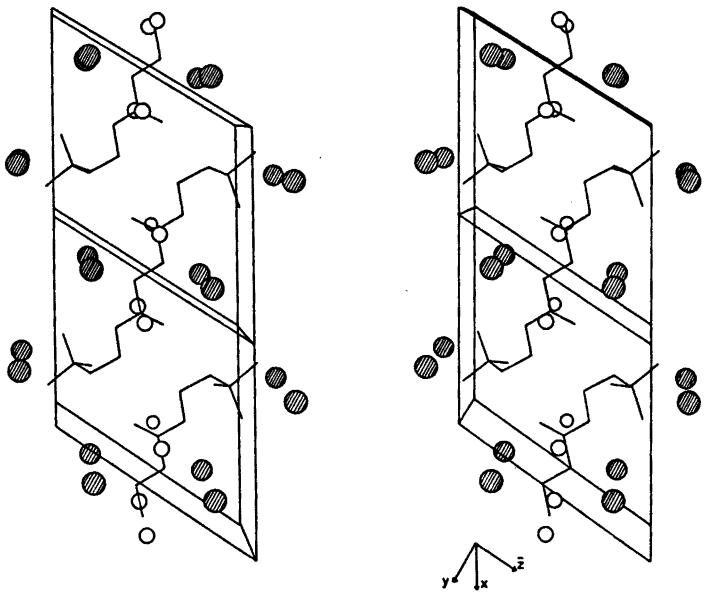

Fig. 2. A stereo view of the packing of succinylcholine chloride dihydrate. The chloride ions are shaded.

6.825(3), $c = 10.260(5)$ Å, $\alpha = 108.13(3)$, $\beta = 122.00(3)$, $\gamma = 84.91(3)°$. $V = 502.3$ Å^3. $D_m = 1.31$ g cm^{-3}, $Z = 1$, $D_c = 1.31$ g cm^{-3}. Linear absorption coefficient for X-rays [λ(Mo$K\alpha$ = 0.7107 Å)], $\mu = 3.5$ cm^{-1}. $F(000) = 214$. The unit-cell parameters were refined by least-squares techniques from the θ angles measured for 45 reflections on a NONIUS three-circle automatic diffractometer. The density was measured by flotation. The melting point was determined on a Leitz hot stage microscope.

Intensity data were collected on the diffractometer from a slightly imperfect single crystal of irregular shape (*ca.* 0.25 × 0.30 × 0.45 mm) using Mo$K\alpha$ radiation and omega scan. Out of the 1578 independent reflections in the range $2.5 \leq \theta \leq 25.0°$, 1382 had $I_{net} \geq 3.0\ \sigma(I)$, where σ is the standard deviation from counting statistics. No absorption corrections have been made.

The structure was solved by the heavy atom method and refined by full matrix least-squares techniques to a final R value of 0.055, using the X-RAY-system.[9] The final cycles of refinement included positional parameters for all atoms and anisotropic thermal parameters for

all non-hydrogen atoms while a fixed common thermal parameter ($B=3.5$) was assigned to all hydrogen atoms. The quantity minimized was $\sum w(|F_o|-|F_c|)^2$ where $w=1/\{1+[(F_o-B)/A]^2\}$, $A=8.0$ and $B=6.0$. The X-ray atomic scattering factors used for hydrogen were those of Stewart, Davidson and Simpson [10] and for all other atoms those listed in International Tables for X-Ray Crystallography.[11] All atoms but Cl^- were treated as uncharged. The final list of structure factors is available from the author on request.

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Received September 23, 1976.