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Crystal data of acetyl choline chloride.* By K. W. ALLEN,† *The Crystallography Laboratory, The University of Pittsburgh, Pittsburgh 13, Pennsylvania, U.S.A.*

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While the crystal structures of acetyl choline bromide and iodide (Sørum, 1956, 1959) have both been investigated, that of the chloride has not. This is almost certainly due to the difficulty of preparing satisfactory crystals. Acetyl choline chloride ((CH₃)₃N.C₂H₄OOCCH₃.Cl) is very hygroscopic and extremely soluble, giving a solution of high viscosity. At 25 °C. a saturated solution contains approximately 0.962 g.ml.⁻¹, has a density of 1.19 g.ml.⁻¹ and has a viscosity rather greater than that of glycerine.

When a solution nearly saturated at room temperature is cooled to approximately -10 °C. the anhydrous salt crystallizes slowly as clusters of needles. There was no evidence of hydrate formation, which was the original cause of the investigation. Single crystals, mounted and sealed in glass capillaries were examined about the needle axis [α] by oscillation and Weissenberg methods using filtered copper radiation and by the precession method using molybdenum radiation. Provided that the sealing was satisfactory these mounted crystals remained unchanged for a period of several weeks.

The crystal data obtained were as follows:

Acetyl choline chloride. (CH₃)₃N.C₂H₄.OOCCH₃.Cl.
Orthorhombic. Formula weight 181.5.
 $a = 6.28 \text{ \AA}$, Unit cell volume 952 Å³.
 $b = 9.93 \text{ \AA}$,
 $c = 15.26 \text{ \AA}$.
Space group $P2_12_12_1$.

Systematic extinctions observed:

$$h00 \text{ for } h = 2n, \quad 0k0 \text{ for } k = 2n, \quad 00l \text{ for } l = 2n.$$

Density: observed 1.202 g.ml.⁻¹; calculated 1.27 g.ml.⁻¹.
Molecules per unit cell 4.

The density of the crystals was estimated by floatation in benzene/chloroform mixtures. It is recognized that the value is not very accurate and is almost certainly lower than the true value because of the difficulty of freeing the crystals completely from the viscous mother liquor. However it is sufficient to confirm the number of molecules per unit cell.

There are no apparent similarities between this structure and that of the other acetyl choline halides. No further work on this structure is contemplated.

References

- SØRUM, H. (1956). *Acta Chem. Scand.* **10**, 1669.
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Refinement of the crystal structure of trimethylamine oxide hydrochloride, (CH₃)₃NO.HCl.

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During the course of the determination of the structure of trimethylamine oxide (Caron & Donohue, 1962), we felt that it would be desirable to carry out additional refinement of the structure of trimethylamine oxide hydrochloride (Rérat, 1960) by making full allowance for thermal anisotropy and by including the effect of the hydrogen atoms. This refinement was carried out on Rérat's observed structure factors by both least-squares and Fourier methods. The complete details of the results of these calculations (Caron, 1962) will not be presented here.

The full matrix was used in the least-squares treatment. The weighting function used was that of Hughes (1941), with $4F_{\min}$ assigned a value of 2.4. The unobserved F 's, listed by Rérat as zero, and F_{002} , which apparently suffers from extinction, were not included in the calculations. The F_c were calculated with the form factor curves of Berghuis *et al.* (1956) for C, N, O, and Cl⁻, and the curve of McWeeny (1951) for H.

The refinement was started at Rérat's final parameter values, omitting the hydrogen atoms. Thus, the published F_c and R value (of 24.4%) were duplicated. Convergence was reached after the computation of a few cycles, during which R dropped to 15.6%. The coordinates of the hydrogen atoms were then estimated by assuming C-H bond lengths of 1.0 Å, H-C-H angles of 110°,

staggered conformations of the methyl groups, and the presence of a proton on the oxygen atom along the direction of the shortest O...Cl distance (2.95 Å). These coordinates, together with isotropic B values of 6.0 Å², were introduced in the computations as variables, and the refinement was resumed. The ultimate R value, including the hydrogen contributions, was 14.0%. The final values of the various parameters are shown in Tables 1, 2, and 3.

Table 1. *Coordinates for the heavy atoms**

		All values have been multiplied by 10 ⁴				
		Least squares		Fourier	Final	(σ)
Atom	Rérat					
Cl	x	3967	3965	3967	3966	(2)
	y	6394	6386	6391	6388	(6)
O	x	2237	2235	2228	2232	(8)
	y	3441	3422	3422	3422	(9)
N	x	1439	1436	1437	1437	(4)
	y	5015	5015	5019	5017	(12)
C ₁	x	619	630	633	631	(6)
	y	3287	3305	3301	3303	(14)
C ₂	x	1442	1452	1445	1449	(8)
	y	6575	6532	6557	6544	(27)
	z	9136	9118	9111	9115	(10)

* For the molecule lying on the mirror plane at $z = \frac{1}{2}$.