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The cubic form of carbon tetrachloride.* By BEN POST, *Polytechnic Inst. of Brooklyn, Brooklyn 1, N. Y., U.S.A.*

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Carbon tetrachloride melts at 250.3 °K.; specific heat data (Hicks *et al.*, 1944) indicates that the compound undergoes a solid phase transformation at 225.3 °K. The entropy of the transition is 4.9 e.u. per mole compared with 2.4 e.u. per mole for the entropy of fusion, indicating that major structural changes take place in the transition. In this note some results of an X-ray diffraction examination of the high temperature solid phase (stable from 225.3 °K. to the melting point) are reported.

Experimental data were obtained at -35 °C. Single crystals were grown using conventional low temperature techniques; an oscillation camera was used with filtered Mo K radiation. Just below the melting point the crystal is optically isotropic; below 225 °K. it transforms to a birefringent phase. In the isotropic phase the crystal diffracts very poorly; exposures of three hours and more per 20° oscillation diagram (operating at 40 kV. and 20 ma., sample diameter 0.3 mm.) were needed to obtain usable diffraction diagrams. Intensities of reflections fell off very rapidly with increasing Bragg angles; all diagrams were marked by extremely heavy non-radial diffuse scattering. Crystals generally grew along [111]. The unit cell is face-centered cubic, with $a_0 = 8.34 \text{ \AA} \pm 0.03 \text{ \AA}$ at -35 °C. There are four molecules per unit cell. The calculated density of the solid at -35 °C. is 1.71 g.cm.⁻³ compared with 1.594 g.cm.⁻³ for the density of the liquid at 20 °C.

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Table 1. *Observed structure factors*

<i>hkl</i>	$ F _{\text{obs.}}$	<i>hkl</i>	$ F _{\text{obs.}}$
111	10.0	400	not obs.
200	9.2	331	1.4
220	3.9	420	not obs.
311	3.2	422	1.7
222	4.3	333	3.0

Values of $|F|_{\text{obs.}}$ are listed in Table 1. It was not possible to obtain satisfactory agreement between $|F|_{\text{obs.}}$ and structure factors calculated on the assumption of anyone of a number of physically possible ordered or partially ordered structures; the assumption of 'freely rotating' molecules (i.e., with chlorine atoms distributed uniformly on the surfaces of spheres, with $r = 1.76 \text{ \AA}$, about each lattice point) led to somewhat better, but still unsatisfactory, agreement. In any event, simple geometrical considerations rule out the possibility of freely rotating carbon tetrachloride molecules in the solid; the centers of adjacent molecules are only 5.9 Å apart. To permit completely free rotation, the distance would have to be increased to about 7.1 Å (the sum of two C-Cl bond lengths plus 2 chlorine van der Waals radii). It does appear likely that some sort of coupled, or geared, 'rotation' exists in the solid; it was felt, however, that the poor diffraction data obtainable did not warrant a more detailed analysis.

Reference

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Note on glutamic acid hydrochloride. By W. C. QUENÉ and F. JELLINEK,* *Laboratorium voor Kristalchemie der Rijksuniversiteit, Utrecht, the Netherlands*

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Some years ago Dawson (1953) reported the crystal structure of what he described as 'DL-glutamic acid hydrochloride'. The space group of the proposed structure is $P2_12_12_1$ and since there is only one molecule in the asymmetric unit, all molecules within a crystallite must have the same configuration, either D or L. Therefore, it seems that either (i) the space group given by Dawson (1953) is incorrect, or (ii) DL-glutamic acid hydrochloride is not a true racemate, but rather a conglomerate of D- and L-crystals, or (iii) Dawson (1953) made a mistake in that his starting material was not the DL-glutamic acid, but one of the active forms.

In order to decide which of the above possibilities is the correct one we prepared crystals of the hydrochlorides of both DL- and L-glutamic acid in the same way as described by Dawson (1953). Rotation and Weissenberg diagrams of the two kinds of crystals were

identical; our unit-cell dimensions ($a = 5.14$, $b = 11.73$, $c = 13.26 \text{ \AA}$), absences and reflexion intensities are in agreement with Dawson's data. It appears, therefore, that DL-glutamic acid hydrochloride is one of the not-too-numerous compounds which crystallize as a mixture of D- and L-crystals, rather than as a racemate.

After our investigation had been completed a paper by Kaplanova (1917) came to our attention, in which the author points out the close macroscopic similarity of L- and DL-glutamic acid hydrochloride crystals; the same also applies to the hydrobromides, but not to the hydroiodides.

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References

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