References to the preparation and melting point of 1-iodoanthraquinone found in the chemical literature are all based on the results reported by Laubé and consequently require revision.

Experimental

1-Iodoanthraquinone.—Twenty grams of 1-aminoanthraquinone, Eastman (Technical), was dissolved in 80 g. of concentrated sulfuric acid. To this solution 6 g. of sodium nitrite was added a little at a time at room temperature. The mixture was stirred mechanically during the addition of the sodium nitrite and for an additional two-hour period. This solution was then poured slowly into 1 liter of ice water with continual stirring. The mixture was stirred mechanically for one hour. The solution was next filtered and to the filtrate 10 g. of potassium iodide was added. This mixture was allowed to stand at room temperature for three hours and then heated for one hour on a steam-bath. After adding 10 cc. of a saturated sodium bisulfite solution, the mixture was filtered, and the residue was washed with distilled water and dried at 100°. The crude 1-iodoanthraquinone was light chocolate-brown; yield 7.5 g. (25%); m. p. 195.5–197.5°.

The residue from the first filtration mentioned above was put into 1 liter of water and stirred mechanically at room temperature for thirty minutes. The mixture was filtered and the filtrate treated with 10 g. of potassium iodide. By continuing the procedure as described above, an additional yield of crude product was obtained, light brown in color, weighing 19.5 g. (65%), m. p. 195.5–197.5°.

Two recrystallizations of the combined crude product from nitrobenzene produced a yellowish-brown crystalline compound, m. p. 204–205°.[5]

Anal. Calcd. for $C_{14}H_7O_2I$: I, 37.99. Found: I, 38.02.

(5) All melting points are corrected.

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Note on the Molecular Structure of 1,2,4,5-Tetrabromocyclohexane (m. p. 185°)

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In some papers from the Oslo Institute of Physical Chemistry dealing with the structure of cyclohexane and its derivatives[1] investigations carried out on the 1,2,4,5-tetrabromocyclohexane of melting point 185° have been referred to. Although these investigations have not yet been brought as far as we could have wished in the case of the X-ray crystallographic part, we should not like to delay publication much longer, and

(1) A list of previous papers is found in a paper published by J. G. Gudmundsen and O. Hassel, *Z. physik. Chem.* **B40**, 326 (1938); see also O. Hassel and A. M. Sommerfeldt, *ibid.*, **B40**, 391 (1938).

are therefore publishing our results now in a preliminary form.

The substance was obtained from the 1,4-dibromo compound of melting point 111° on eliminating two moles of hydrogen bromide and adding two moles of bromine to the 1,4-cyclohexadiene thus obtained. The crystals were rather well developed, having a size suitable for examination by the simple single crystal methods. They were found to be orthorhombic with axial ratios $1.010_3:1:0.997_7$, and the following faces were observed: $\{110\}$, $\{101\}$, $\{011\}$, and in some cases also $\{001\}$. The optical axes were nearly perpendicular to (110) and ($1\bar{1}0$).

The X-Ray Examination.—Approximate determinations of axial lengths (using the layer line method) gave the values $a = 7.92$ Å., $b = 7.85$ Å., and $c = 7.88$ Å. More accurate values were obtained from Bragg photographs on (001), (110), and (011) combined with the goniometrically determined axial ratios given above. We found $a = 7.98$ Å., $b = 7.90$ Å., $c = 7.89$ Å.

From our X-ray data (chiefly using Weissenberg photographs) we found that no other systematic extinctions can be observed than those of ($h00$) and ($0k0$), h or k being odd. The only space group of the orthorhombic system characterized by the absence of these reflections and showing no other regular extinctions is the Schönflies group $D_2^3-P2_12_12$. This space group contains only twofold and fourfold positions, the former being characterized by the point symmetry C_2 (twofold axis of symmetry) and one degree of freedom, the latter by three degrees of freedom. The density of the crystals being 2.66, the number of molecules in the unit cell must be 2 (calculated 2.01) and the single molecule must have a twofold axis of symmetry. The coördinates of the bromine atoms are of the form xyz, etc., four and four being crystallographically equivalent. It is not possible to evaluate the parameters of the bromine atoms with any accuracy, neglecting the influence of the carbon atoms, but a rough estimate of their positions can be made and this was found useful when trying to interpret the structure with the help of classical conceptions of the configuration of the cyclohexane ring using newer data concerning the interatomic distances. Assuming the carbon–carbon distance to be 1.54 Å. and the carbon–bromine distance to be 1.93 Å., the distribution of the bromine atoms was found to be in fairly

good agreement with positions which may be derived from the highly symmetrical rigid staggered form of the cyclohexane ring. In the model in question the C–Br linkages of two neighboring C-atoms (say 1 and 2) are parallel to the three-fold axis of the cyclohexane ring, but point in opposite directions. The angles between the C–Br linkages of the two remaining carbon atoms attached to bromine (4 and 5) and the axis of the carbon ring are both equal to the tetrahedral angle. That is, two of the C–Br bonds (for C_1 and C_2) project above and below the ring, and the other two (for C_4 and C_5) lie nearly in the median plane of the ring. The twofold axis shown by the molecule as a whole (parallel to the c-axis of the crystal) lies in the plane of the four carbon atoms 1, 2, 4, and 5, running parallel to the lines joining 1 and 5 (2 and 4) and through the middle point of the rectangle 1, 2, 4, 5.

We have tried to get a close agreement with our observed intensities assuming the carbon atoms to occupy the positions of this model and allowing for small changes in the positions of the bromine atoms. A certain difficulty arose as we were somewhat in doubt as to the true intensity of some reflections from (100) and (010), especially the sixth and eighth orders. The values of the bromine parameters to which we have been led are the following ones: $x_1 = -0.018$, $y_1 = 0.282$, $z_1 = -0.106$; $x_2 = 0.218$, $y_2 = 0.018$, $z_2 = 0.442$.
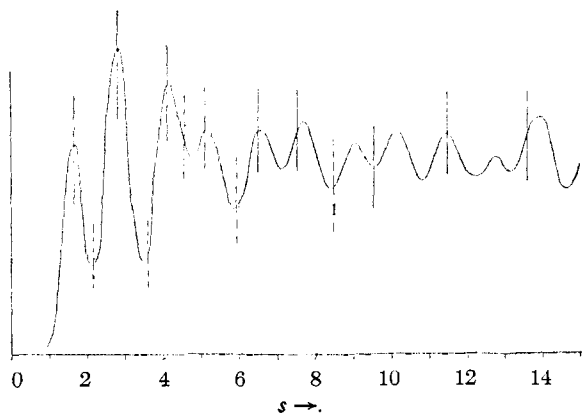

Fig. 1.

The Dipole Moment.—The measurements were carried out some years ago in benzene solution and at room temperature, the method being that described in a series of papers from our institute. The molar polarization P_{A+O}, the sum

of atomic and orientation polarization, is given in Table I for the molar concentrations m employed:

TABLE I

m	0.07536	0.05946	0.04165	0.02525
P_{A+O}	96.5	95.0	98.9	101.4

Extrapolation to infinite dilution gives the value $P_{A+O} = 104.6$, and, neglecting the atomic polarization, $\mu = 2.2_2\ D$.

Electron Diffraction Diagrams of the Vapor.—Some electron diffraction diagrams of the evaporated substance were taken, using electrons of about 35 kv. velocity. The s values of the observed maxima and minima are listed in Table II, s meaning $4\pi \sin \vartheta/\lambda$. We have drawn theoretical curves for all possible models of the 1,2,4,5-tetrabromocyclohexane molecule based on the rigid symmetrical form of the carbon ring, assuming all valency angles to be 109°28', and using the values 1.54 and 1.93 Å. for the C–C and C–Br distances. A comparison with the electron diffraction plates showed clearly that the model used above and fitting the X-ray data is the only one of these models which accounts for the observed maxima and minima, the agreement being

TABLE II

s-VALUES OF OBSERVED MAXIMA AND MINIMA ON ELECTRON DIFFRACTION PLATES

Maxima	Minima
1.66	
	2.18
2.28	
	3.65
4.12	
	4.54
5.10	
	5.95
6.49	
7.53	
	8.52
9.56[a]	
11.51	
13.62	

[a] Some plates indicate here not a single, rather broad maximum, but rather two maxima, but we have nevertheless recorded the mean value $s = 9.56$ in Fig. 1.

surprisingly good. The curve in question is shown in Fig. 1, the observed maxima being indicated by vertical lines, and the minima by dotted lines.

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