

Table 3. *X-ray data*

h,k,l	calc. d	obs. d	obs. int.	h,k,l	calc. d	obs. d	obs. int.
110	7.52	7.45	s	119;526	1.603, 1.592	1.596	vw
-	-	6.39	vw	633;722	1.558, 1.557	1.559	vvvw
003;202	4.92, 4.89	4.87	vw	544	1.521	1.522	vvw
300	4.34	4.32	s	811	1.517	1.516	vvvw
113	4.12	4.11	vvw	446	1.495	1.492	vvvw
220	3.76	3.74	mw	428	1.477	1.479	vvvw
104	3.55	3.56	mw	642	1.465	1.464	vvw
303;312	3.26, 3.25	3.26	m	900	1.448	1.450	vvw
401	3.18	3.22	mw	805	1.427	1.429	vvw
223	2.99	2.99	m	820;419	1.422, 1.421	1.423	w
214	2.95	2.96	m	2, 1, 10	1.415	1.418	vvw
410	2.84	2.84	ms	716;814	1.413, 1.410	1.411	vvvw
-	-	2.59	mw	903	1.389	1.389	vw
330	2.51	2.50	mw	339;627	1.373	1.377	vw
413;006;502	2.46	2.46	mw	823;636	1.366	1.369	w
404	2.44	2.45	vvw	618;740	1.353, 1.352	1.352	vw
116;422	2.34	2.35	w	4, 0, 10;912	1.345, 1.344	1.344	vvvw
324;511	2.32, 2.31	2.33	w	645	1.334	1.334	vvvw
333	2.23	2.23	vvw	3, 2, 10;2, 0, 11	1.324, 1.315	1.319	vvvw
600	2.17	2.17	vvw	743;832	1.303	1.305	vvvw
306;431	2.14, 2.12	2.13	vvvw	10, 0, 1	1.298	1.296	vvvw
520	2.09	2.08	w	556;654	1.284, 1.281	1.283	vw
107	2.08	2.07	vw	921	1.279	1.275	vw
226	2.06	2.05	vw	3, 1, 11	1.259	1.258	vvw
514	1.977	1.978	vw	660	1.254	1.255	vvw
523;612	1.922, 1.919	1.922	vw	5, 1, 10;906	1.249, 1.248	1.248	vvvw
416	1.861	1.867	ms	915	1.240	1.242	vvvw
434	1.853	1.856	vvw	10, 1, 0;449;817	1.237, 1.237, 1.236	1.234	w
532	1.805	1.806	vvvw	841;808	1.227, 1.222	1.224	vw
407	1.771	1.763	w	4, 3, 10	1.216	1.217	vvw
443;336	1.757	1.752	vw	663;1, 1, 12	1.215	1.215	vvw
710;327	1.726, 1.724	1.724	vw	728;930	1.206, 1.205	1.206	vw
704	1.663	1.664	vw	10, 1, 3	1.200	1.201	vvw
541;615	1.658, 1.649	1.652	vvvw	-	-	1.195	vw
318;630;009	1.644, 1.642, 1.641	1.641	m	5, 0, 11	1.194	1.194	vw
713	1.629	1.631	vw	719	1.189	1.188	w
606	1.629	1.628	vvw	746;3, 0, 12	1.185, 1.184	1.184	vw

content of 32-35% and a molecular weight of 2273 per rhombohedral unit cell containing four Ca_6WO_9 molecules.

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On the crystal structure of the chloranil-hexamethylbenzene complex.* By NOEL D. JONES and RICHARD E. MARSH, *Gates and Crellin Laboratories of Chemistry, California Institute of Technology, Pasadena, California, U.S.A.*

(Received 8 March 1962)

The crystal structure of the complex between chloranil and hexamethylbenzene— $\text{C}_6\text{Cl}_4\text{O}_2 \cdot \text{C}_6(\text{CH}_3)_6$ —was recently investigated by Harding & Wallwork (HW) (1955). They reported extreme non-planarity for the chloranil and hexamethylbenzene molecules and explained the puckering in terms of steric interactions and polarization bonding between the two molecules. Since then, Donohue & Trueblood (1956) and Nyburg (1961) have expressed surprise at this and other features of the reported structure and have suggested that, in view of the small number of observed reflections (185), the reported non-planarity might not be of significance. We have now shown that the observed intensities are indeed consistent with entirely coplanar structures for the two molecules.

We have carried out a least-squares refinement of the

positional parameters, based on the observed structure factors and atomic coordinates of HW. The quantity minimized was $\sum w(F_o^2 - F_c^2)^2$ and the weights w were taken inversely proportional to the square of the form factor of carbon.† Individual isotropic temperature parameters were adjusted only approximately, with the restriction that chemically equivalent atoms have identical parameters. In order to place the data of HW (their Table 2) on an absolute scale we found it necessary to multiply their observed structure factors by 4.25.

Our final parameters and their estimated standard

† A number of pronounced and persistent discrepancies between calculated and observed structure factors among the weak reflections discouraged us from the usual scheme of assigning weights inversely proportional to the observed structure factors. In addition, the 100 reflection was given zero weight.

* Contribution No. 2745 from the Gates and Crellin Laboratories of Chemistry.

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Table 1. *Refined atomic parameters*

Positional parameters and thermal parameters, B_i , where
 $T_i = \exp(-B_i \sin^2 \theta / \lambda^2)$,
 from the final least-squares analysis

No.	Type	x	σ_x	y	σ_y	z	σ_z	B
1	C	0.071	(5)	-0.140	(6)	0.028	(2)	1.0
2	C	0.056	(7)	-0.024	(5)	0.093	(3)	1.0
3	C	-0.011	(6)	0.124	(5)	0.063	(3)	1.0
4	Cl	-0.024	(2)	0.260	(2)	0.142	(1)	5.5
5	Cl	0.141	(2)	-0.335	(2)	0.071	(1)	5.5
6	O	0.095	(4)	-0.058	(4)	0.181	(2)	3.4
7	C	0.576	(6)	-0.132	(6)	0.057	(3)	1.9
8	C	0.542	(8)	0.007	(5)	0.098	(3)	1.9
9	C	0.479	(6)	0.130	(6)	0.037	(3)	1.9
10	CH ₃	0.446	(6)	0.302	(7)	0.086	(3)	6.0
11	CH ₃	0.654	(6)	-0.265	(8)	0.116	(4)	6.0
12	CH ₃	0.590	(7)	0.034	(8)	0.202	(4)	6.0

deviations are listed in Table 1; the standard deviations are about 0.06 Å for the positions of the light atoms and 0.02 Å for the chlorine atoms. (These values are in approximately the expected ratio whereas, as Donohue & Trueblood point out, the standard deviations given by HW are greater for the chlorine atoms than for the lighter atoms.) The standard deviations in the temperature factors B are about 2.0 for the light atoms and 0.7 for the chlorine atoms. In the last least-squares refinement cycle no coordinate shift was as much as 1/3 of its standard deviation. The final R factor was 0.14 compared with the value 0.27 we obtained with the parameters of HW.

Our results indicate that, within the large experimental errors, both the chloranil and the hexamethylbenzene

molecules are planar. The maximum deviation from the least-squares plane of the chloranil molecule, calculated with weights proportional to the atomic numbers,* is 0.087 Å for two of the carbon atoms; the maximum deviation from the best plane of the hexamethylbenzene molecule is 0.045 Å. The two planes are closely parallel, the calculated dihedral angle being 2.1°. Similarly, the bond distances show no significant deviations from the expected values, the extreme value 1.71 Å calculated for a pair of C-CH₃ bonds being only two standard deviations from the normal distance of 1.54 Å.

Accordingly, we reach the conclusion that the experimental data are compatible with planar molecules having normal dimensions and that it is not necessary to postulate, as HW have done, a zigzag arrangement of polarization bonds to explain the structure.

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* It is clearly more appropriate to include all twelve atoms of the chloranil molecule in the calculation of the best plane than (as HW did) to consider only the six carbon atoms.

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The crystal structure of the chloranil-hexamethylbenzene complex. By S. C. WALLWORK, *Department of Chemistry, University of Nottingham, England* and T. T. HARDING, *Imperial Chemical Industries, Ltd., Billingham Division, Billingham, Co. Durham, England*

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The refinement of the crystal structure of the chloranil-hexamethylbenzene complex by Jones & Marsh (1962) confirms the suggestion already made (Wallwork, 1961) that the molecular distortions originally reported (Harding & Wallwork, 1955) are probably not significant. They were mistakenly interpreted as being significant in the original work because the standard deviations of atomic positions were incorrectly calculated. In spite of the improvements in R and molecular planarity brought about by the recent refinement it is clear that the present X-ray data are not sufficient to establish accurate atomic

positions. New and more extensive data are now being obtained at about -100 °C. and these will be used in a further refinement of the structure.

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Some observations on growing crystals of argon. By L. H. BOLZ, H. P. BROIDA and H. S. PEISER, *National Bureau of Standards, Washington D.C., U.S.A.*

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There is considerable interest in the measurement of the properties of single crystals of the rare gases [Kyoto

Conference on Magnetism and Crystallography (1961), Cook (1961), Jones (1958), and Dobbs & Jones (1957)].