650. *The Crystal Structure of Di-*m-*xylylene.*

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The atomic arrangement in crystals of di-*m*-xylylene has been worked out completely, using *X*-ray diffraction data and three-dimensional Fourier syntheses. The molecules are centrosymmetric and the two halves form a stepped system. The benzene rings are boat-shaped to achieve adequate separation between the central carbon atoms which are 2·69 Å apart. The mean aromatic carbon to carbon bond-length is 1·386 Å, and the aliphatic 1·543 Å.

DURING the determination of the structure of di-*p*-xylylene (Brown, *J.*, 1953, 3265, 3270) attention was directed to the isomeric compounds di-*o*-xylylene and di-*m*-xylylene. Di-*o*-xylylene had been prepared by Baker, Banks, Lyon, and Mann (*J.*, 1945, 27), but the *X*-ray work by Davidson and Perutz proceeded no further than the determination of the unit-cell dimensions and space-group. Di-*m*-xylylene had been prepared originally by Pellegrin (*Rec. Trav. chim.*, 1899, **18**, 458), and this work has been confirmed more recently by Baker, McOmie, and Norman (*Chem. and Ind.*, 1950, 77 ; *J.*, 1951, 1114). It was suggested, in discussion with Professor Baker, that an *X*-ray investigation of the structure of di-*m*-xylylene would throw light on the stereochemistry of the molecule, and this work has been carried out on samples of his preparations.

The benzene ring has long been regarded as a regular, plane hexagon of carbon atoms. There is evidence that in a number of fused benzene rings, the regularity is destroyed, *e.g.*, in coronene (Robertson and White, *J.*, 1945, 607), but in all simple aromatic compounds no great divergence from regularity is expected. It was pointed out, however, by Cook (*Ann. Reports*, 1942, **39**, 173) that this would lead to abnormally close approaches between atoms in certain cases, particularly 3 : 4-5 : 6-dibenzophenanthrene, and the crystal-structure determination of one form of this compound (McIntosh, Robertson, and Vand,

Nature, 1952, **169**, 322) confirms that there is considerable distortion in the molecule to avoid clashing between neighbouring non-bonded carbon atoms. A similar phenomenon would be expected in the case of di-*m*-xylylene where, if all the bond lengths and inter-bond angles possessed standard values, the central non-bonded carbon atoms would be approximately 1·54 Å apart. In order to find out the true distance between these atoms, and how the remainder of the molecule would be distorted, the crystal-structure determination was commenced.

The unit-cell dimensions are $a = 12·22$, $b = 8·28$, and $c = 5·82$ Å, and the angle $\beta = 93° 18'$. On the assumptions of two molecules per unit cell and a molecular weight (for $C_{16}H_{16}$) of 208·1, the calculated specific gravity should be 1·176; that observed was 1·178. Absent reflexions were $\{0k0\}$ for k odd, and $\{h0l\}$ for h odd, so the space-group was fixed unambiguously as $P2_1/a$ (C_{2h}^5). As this space-group has a four-fold general position, it

FIG. 1.   *Electron-density map of di-*m-*xylylene obtained by projection along* c *axis.*
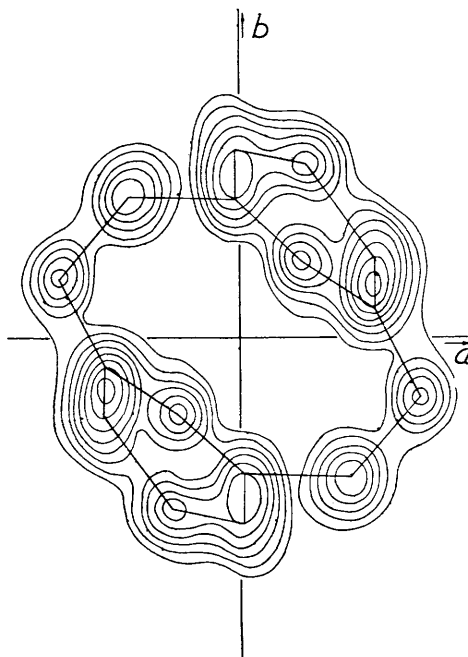
followed that the molecules of di-*m*-xylylene possessed centres of symmetry and that the centres of molecules would coincide with centres of symmetry in the unit cell.

Despite the close similarity between the cell dimensions of di-*m*-xylylene and of di-benzyl (Jeffrey, *Proc. Roy. Soc.*, 1947, *A*, **188**, 222) as well as a resemblance between the general shapes of the molecules, there is no correspondence between their arrangement in the unit cell, and attempts to solve the structure by analogy were unsuccessful. Indeed, all the usual methods of finding a suitable trial structure, such as Patterson syntheses, molecular structure factors, and inequalities were unsuccessful. The approach which eventually led to a solution was a graphical method in which the planes giving strongest reflexions were represented by lines in a scale diagram and various orientations of a molecular model were tried in order to place individual atoms in phase with respect to these strong planes. Taken in conjunction with the fact that a group of strong planes lying almost parallel to each other, *i.e.*, (43Ī), (43Ī), (42Ī), (32Ī), (22Ī), and (44Ī), appeared to coincide roughly with a prominent plane in the molecule, this method gave a molecular arrangement where a moderate number of calculated structure amplitudes agreed with the experimental values.

From this point, refinement of the atomic co-ordinates proceeded in the usual way. Two-dimensional Fourier syntheses were used for the b and c axis projections, neither of which was entirely free from overlapping atoms. The electron-density map obtained by

plotting the best c projection is shown in Fig. 1. Following these, three-dimensional Fourier syntheses were used, first with a limited number of terms, and then more being gradually introduced as their phases were determined until all the 1200 were included. The progress of the refinement was followed by calculating the residual

$$R = \frac{\Sigma|F(\text{exp.})| - |F(\text{calc.})|}{\Sigma F(\text{exp.})}$$

which, for the last three sets of structure amplitudes had values of 0·26, 0·208, and 0·163. The last synthesis resulted in no changes of phase of the calculated structure amplitudes, and the refinement was then considered to be complete. The resulting atomic co-ordinates are listed in Table 1.

TABLE 1. *Atomic co-ordinates.*

	x	y	z		x	y	z
C_1	0·882	0·231	0·817	C_5	0·198	0·904	0·111
C_2	0·988	0·232	0·978	C_6	0·999	0·319	0·179
C_3	0·068	0·119	0·942	C_7	0·081	0·285	0·351
C_4	0·142	0·067	0·119	C_8	0·149	0·156	0·322

Description of the Structure.—The crystal structure of di-*m*-xylylene comprises discrete molecules, each making van der Waals contacts with eight others. There are 40 inter-atomic distances of this type of less than 4 Å between molecules, and these are shown in Table 2.

TABLE 2. *Intermolecular ("van der Waals") distances (Å).*

With molecule along a :		With molecule along c :	
C_3–C_5' (four)	3·74	C_7–C_1 (four)	3·77
C_4–C_5' (four)	3·71	C_7–C_2 (four)	3·96
C_4–C_1 (four)	3·88	C_7–C_3 (four)	3·72
With molecule along b :		C_8–C_3 (four)	3·81
C_2–C_2' (two)	3·64		
C_6–C_6' (two)	3·65		
C_2–C_6' (four)	3·70		
C_1–C_6' (four)	4·00		

Each molecule of di-*m*-xylylene has a centre of symmetry, and consists of two benzene rings joined by ·CH_2·CH_2· linkages in the *meta*-positions. This has the effect of producing a 10-membered ring system flanked by two 6-membered benzene rings. The benzene rings are located step-wise in order to permit adequate clearance between the central carbon atoms (C_3 and C_3'; see Fig. 2) and their attached hydrogen atoms. Even then, were strict planarity and regularity of the hexagonal ring systems and substituents to be maintained, the C_3–C_3' distance would be almost the same as C_1–$C_5' = 1\cdot56$ Å. This is impossibly close for non-bonded carbon atoms; and the situation is relieved by a distortion of the rings. Carbon atom 3 is displaced out of the mean plane of $C_2C_4C_6C_8$ by 0·143 Å, and the substituent methylene carbon atoms 1 and 5 by approximately 0·4 Å, so that the substituting bonds make angles of approx. 15° with the mean plane of the ring (Fig. 3). A secondary effect is that the carbon atom 7 at the far end of the benzene ring is also displaced out of the plane by 0·042 Å. The result of this distortion is to increase the distance between C_3 and C_3' to 2·689 Å. The bond lengths and inter-bond angles are listed in Table 3, and are shown diagrammatically in Fig. 2.

TABLE 3. *Bond lengths (Å) and inter-bond angles.*

C_1–$C_5' = 1\cdot559$	C_4–$C_5 = 1\cdot515$	$C_2\widehat{C}_1C_5' = 110° 48'$	$C_4\widehat{C}_5C_1' = 109° 39'$
C_1–$C_2 = 1\cdot554$		Mean tetrahedral angle $= 110° 18'$	
Mean aliphatic C–C $= 1\cdot543 \pm 0\cdot013$ Å			
C_2–$C_3 = 1\cdot378$	C_6–$C_7 = 1\cdot404$		
C_3–$C_4 = 1\cdot399$	C_7–$C_8 = 1\cdot369$	$C_3\widehat{C}_2C_6 = 116° 42'$	$C_3\widehat{C}_4C_5 = 121° 45'$
C_2–$C_6 = 1\cdot374$	C_8–$C_4 = 1\cdot391$	$C_1\widehat{C}_2C_6 = 123° 36'$	$C_8\widehat{C}_4C_5 = 119° 32'$
Mean aromatic C–C $= 1\cdot386 \pm 0\cdot008$ Å		$C_1\widehat{C}_2C_3 = 118° 38'$	$C_5\widehat{C}_6C_7 = 122° 14'$
		$C_2\widehat{C}_3C_4 = 122° 16'$	$C_6\widehat{C}_7C_8 = 118° 48'$
		$C_3\widehat{C}_4C_8 = 117° 55'$	$C_7\widehat{C}_8C_4 = 120° 40'$
		Mean hexagonal angle $= 120° 13' \pm 1° 16'$	

The equation of the mean plane through $C_2C_4C_6C_8$ was calculated, by the method of least squares, to be $x + 0.952y - 0.624z = 1.782$. The distances (in Å) of the various carbon atoms from this plane are :

C_1, -0.448	C_3, $+0.143$	C_5, -0.368	C_7, $+0.042$
C_2, -0.009	C_4, $+0.006$	C_6, $+0.007$	C_8, -0.005

The angle which the C_1–C_2 bond makes with this mean plane is 16° 45', and that made by the C_4–C_5 bond is 14° 4'.

No account has been taken of the hydrogen atoms at any stage of this work, nor have any atomic co-ordinates been calculated for them.

Although hydrocarbons are possibly one of the least interesting and least reactive classes of organic compounds, more attention appears to have been paid to them by X-ray analysts than to any other. Many of them have been investigated to a preliminary stage by X-ray
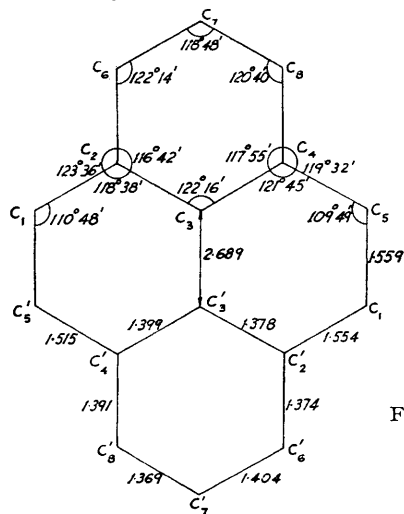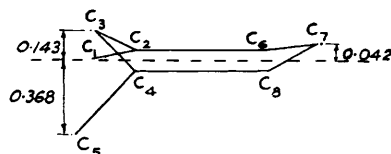

FIG. 3. *Showing the distortion of the benzene ring in di-*m-*xylylene from planarity.*

FIG. 2. *Diagram of molecule of di-*m-*xylylene showing bond lengths (in Å) and inter-bond angles.*

diffraction, and the crystal structures of about 20 of them have been determined. Of these, at least 7 have been refined by three-dimensional Fourier methods, and it can be expected that the resulting bond lengths are fairly accurate. Table 4 lists the C–C bond lengths obtained in these analyses. It shows that the values obtained for these bond

TABLE 4. *Mean carbon-to-carbon bond lengths* (Å) *in aromatic hydrocarbons.*

	Aliphatic	Aromatic
Naphthalene [1]	—	1.391 ± 0.015
Anthracene [2]	—	1.397 ± 0.016
Dibenzyl [3]	1.49	1.373 ± 0.008
1 : 2 : 3 : 4-Tetraphenyl*cyclo*butane [4]	1.537	1.388 ± 0.009
Bisdiphenylene-ethylene [5]	1.48	1.40
Di-*p*-xylylene [6]	1.544	1.395
Di-*m*-xylylene	1.543 ± 0.013	1.386 ± 0.008

(The C–C value in diamond is 1.5445 Å.)
[1] Abrahams, Robertson, and White, *Acta Cryst.*, 1949, **2**, 238. [2] Sinclair, Robertson, and Mathieson, *ibid.*, 1950, **3**, 251. [3] Jeffrey, *loc. cit.* [4] Dunitz, *Acta Cryst.*, 1949, **2**, 1. [5] Fenimore, *ibid.*, 1948, **1**, 295. [6] Brown, *J.*, 1953, 3265.

lengths in di-*m*-xylylene agree well with those found in other accurate analyses. It is interesting that the benzene ring, normally hexagonal, can be distorted to such an extent without interfering appreciably with the interatomic distances.

EXPERIMENTAL

The sample of di-*m*-xylylene was prepared by Professor W. Baker and Miss J. M. Norman by the action of sodium on *m*-xylylene dibromide. Good crystals were readily obtained by evaporation of an alcoholic solution; they were prismatic and roughly equidimensional in shape.

TABLE 5. *Experimental and calculated structure amplitudes.*

hkl	F (exp.)	F (calc.)	hkl	F (exp.)	F (calc.)	hkl	F (exp.)	F (calc.)	hkl	F (exp.)	F (calc.)
000	—	+224	006	4	+ 4	712	6	− 4	016	3	+ 2
200	40	+ 40	206	4	+ 5	10,12	2	+ 1	116	2	+ 2
400	17	− 16	406	2	− 2	11,12	2	− 1	516	1	− 1
600	13	+ 13	606	2	+ 2	13,12	2	− 1	616	1	+ 1
800	4	+ 7	806	3	+ 4	14,12	1	+ 2	716	2	+ 2
10,00	2	+ 4	607̄	1	+ 1	13,13̄	1	+ 1	617̄	1	+ 2
12,00	2	+ 3	207̄	1	− 1	11,13̄	3	+ 3	317̄	2	− 1
14,00	5	+ 6	007	1	− 1	10,13̄	2	+ 1	217̄	1	0
14,01̄	2	+ 2	207	1	− 1	913̄	2	+ 1	017	1	0
10,01̄	5	+ 6	110	26	+29	813̄	6	+ 6	117	1	+ 1
801̄	5	+ 5	210	18	−19	713̄	3	+ 4	217	1	+ 2
601̄	3	− 1	310	37	−37	613̄	2	+ 2	417	1	+ 1
401̄	2	+ 2	410	14	−14	513̄	6	− 5	020	48	−48
201̄	76	+ 76	510	2	− 1	413̄	9	−10	120	7	− 8
001	64	+ 64	610	3	+ 3	313̄	1	+ 1	220	42	−38
201	37	− 37	810	1	− 1	213̄	20	−22	320	27	−21
401	29	+ 23	910	5	− 4	113̄	9	+ 9	420	3	− 5
601	23	+ 24	10,10	10	+12	013	8	+ 8	520	11	+ 9
801	1	+ 3	11,10	3	− 3	113	1	− 3	620	2	− 2
14,01	3	+ 3	12,10	10	+ 8	213	11	−14	720	2	− 1
14,02̄	1	− 3	13,10	1	0	313	11	+12	820	12	− 9
12,02̄	3	+ 4	14,10	1	− 2	413	3	+ 4	920	10	− 8
10,02̄	8	+ 8	15,10	3	+ 2	513	7	+ 7	10,20	6	− 6
802̄	2	− 4	15,11̄	4	+ 3	615	7	+ 7	11,20	9	+ 7
602̄	7	− 6	14,11̄	3	+ 4	813	1	+ 1	12,20	2	− 3
402̄	9	− 8	12,11̄	9	+ 8	913	2	− 1	13,20	2	+ 2
202̄	5	+ 11	10,11̄	1	+ 1	10,13	3	+ 3	15,20	2	− 2
002	6	− 4	911̄	1	+ 2	11,13	2	− 2	15,21̄	2	− 2
202	6	+ 4	811̄	3	+ 2	12,13	2	+ 2	14,21̄	1	0
402	34	+ 34	711̄	3	− 3	13,13	2	+ 2	13,21̄	4	+ 2
602	5	+ 1	611̄	13	+10	12,14̄	2	− 2	12,21̄	2	− 1
802	7	− 6	511̄	16	−16	11,14̄	3	+ 2	11,21̄	1	− 1
10,02	4	− 6	311̄	15	−15	10,14̄	2	− 2	10,21̄	6	− 4
12,02	2	− 2	211̄	27	+29	914̄	2	+ 2	921̄	5	− 4
14,02	3	+ 3	111̄	44	+44	814̄	3	+ 2	821̄	5	− 5
14,03̄	1	+ 1	011	15	−19	714̄	1	− 1	721̄	17	+13
12,03̄	4	+ 5	111	2	− 2	614̄	4	− 4	621̄	3	− 3
803̄	3	− 4	211	4	+ 1	514̄	6	− 7	521̄	12	−14
603̄	4	− 3	311	6	− 9	414̄	9	− 8	421̄	29	−28
403̄	8	− 10	411	11	− 6	314̄	4	+ 4	321̄	39	−38
203̄	10	+ 10	511	15	+14	214̄	4	− 3	221̄	34	−36
003	1	+ 2	611	6	− 6	114̄	1	− 1	121̄	13	−11
203	2	+ 3	711	9	− 8	114	3	− 3	021	3	− 2
403	10	+ 9	811	1	− 1	214	8	+11	121	21	+16
603	4	− 6	911	7	− 5	314	9	+ 9	221	5	− 1
803	4	− 5	10,11	6	+ 4	414	14	+16	321	4	− 1
10,03	3	− 4	11,11	2	− 1	514	3	+ 2	421	5	− 5
12,03	2	+ 2	12,11	1	− 1	714	3	− 2	521	3	0
12,04̄	2	+ 1	13,11	2	+ 2	10,14	2	+ 2	621	14	−13
10,04̄	2	+ 1	14,11	2	− 2	12,15̄	2	− 3	721	7	− 6
804̄	4	+ 5	15,11	2	+ 1	11,15̄	2	+ 2	821	13	−11
404̄	4	+ 2	15,12̄	2	+ 2	10,15̄	2	+ 2	921	2	+ 4
204̄	7	+ 7	14,12̄	2	+ 2	815̄	3	+ 2	10,21	4	− 4
004	8	− 11	12,12̄	2	+ 1	715̄	2	− 2	11,21	11	+ 9
204	3	+ 5	11,12̄	4	+ 4	615̄	3	− 3	14,21	1	+ 1
404	12	+ 12	912̄	1	− 1	315̄	1	+ 2	15,21	1	+ 1
804	2	− 1	812̄	11	+10	215̄	1	− 1	15,22̄	1	− 1
12,05̄	4	+ 4	712̄	3	− 3	115̄	6	− 7	13,22̄	2	+ 1
10,05̄	5	+ 5	612̄	13	+13	015	9	+ 8	12,22̄	3	− 3
805̄	4	+ 5	512̄	4	− 4	215	9	+ 9	11,22̄	1	− 1
605̄	1	+ 1	412̄	9	+11	315	6	+ 5	10,22̄	3	− 3
405̄	3	+ 3	312̄	2	+ 4	415	2	+ 2	922̄	3	+ 3
205̄	3	− 2	212̄	2	0	515	2	− 2	822̄	1	+ 1
005	1	− 1	112̄	19	+23	615	2	− 2	722̄	7	+ 4
205	13	+ 15	012	30	+32	815	2	+ 2	622̄	4	− 3
405	6	+ 6	112	1	− 4	915	2	+ 1	522̄	19	−18
605	2	− 2	212	5	− 3	10,16̄	2	+ 3	422̄	16	−15
805	2	+ 2	312	11	+11	416̄	2	+ 2	322̄	9	− 8
10,06̄	1	+ 2	412	17	−18	316̄	3	− 2	222̄	5	+ 7
806̄	2	+ 2	512	10	+ 8	216̄	2	− 2	122̄	11	−13
606̄	2	+ 2	612	3	− 5	116̄	4	− 4	022	8	+ 9

TABLE 5. (*Continued.*)

hkl	F (exp.)	F (calc.)	hkl	F (exp.)	F (calc.)	hkl	F (exp.)	F (calc.)	hkl	F (exp.)	F (calc.)
122	15	+15	127	1	+ 2	12,3̄3̄	1	− 1	740	4	+ 4
222	14	−14	227	1	+ 2	11,3̄3̄	2	− 2	840	12	+ 8
3̄22	4	− 8	327	2	+ 2	83̄3̄	5	+ 5	940	2	− 1
422	10	−10	427	1	− 2	73̄3̄	8	− 8	10,40	4	+ 4
522	3	+ 3	130	2	− 2	63̄3̄	2	− 3	11,40	2	+ 1
622	5	− 5	230	22	−18	53̄3̄	6	− 6	14,40	1	− 2
822	1	0	330	17	+17	43̄3̄	1	+ 3	14,4̄1̄	1	− 1
922	2	+ 3	430	12	− 9	33̄3̄	3	+ 3	13,4̄1̄	2	+ 2
10,22	2	+ 2	530	2	+ 4	23̄3̄	6	+ 9	12,4̄1̄	1	+ 1
11,22	4	+ 3	630	12	+12	13̄3̄	6	− 5	11,4̄1̄	3	+ 2
12,22	1	+ 1	830	8	− 8	033	2	+ 5	10,4̄1̄	3	+ 1
14,2̄3̄	3	− 3	930	2	+ 4	133	11	−11	94̄1̄	3	− 2
13,2̄3̄	1	+ 1	10,30	4	− 5	233	5	+ 5	84̄1̄	6	+ 5
12,2̄3̄	3	− 3	11,30	2	+ 3	333	5	− 8	74̄1̄	6	+ 4
11,2̄3̄	1	− 1	12,30	3	+ 2	533	4	− 6	64̄1̄	7	+ 6
10,2̄3̄	3	+ 3	13,30	1	± 1	633	8	−11	54̄1̄	4	+ 4
92̄3̄	4	+ 2	14,30	1	0	733	4	− 7	44̄1̄	15	+15
82̄3̄	4	+ 4	15,3̄1̄	1	− 1	833	6	− 6	34̄1̄	2	+ 3
72̄3̄	7	+ 6	14,3̄1̄	2	− 2	10,33	2	+ 2	24̄1̄	9	+ 8
62̄3̄	4	− 4	13,3̄1̄	1	+ 1	11,33	2	+ 2	14̄1̄	10	+11
42̄3̄	5	− 6	12,3̄1̄	1	− 1	12,33	1	− 1	041	4	− 4
22̄3̄	1	+ 3	11,3̄1̄	3	− 2	13,3̄4̄	2	− 1	141	17	−17
12̄3̄	8	−12	10,3̄1̄	5	− 4	12,3̄4̄	2	− 2	241	1	− 1
02̄3	11	− 8	93̄1̄	3	− 3	11,3̄4̄	1	0	341	12	−12
22̄3	7	− 6	83̄1̄	4	+ 6	10,3̄4̄	1	+ 1	441	10	− 9
42̄3	4	+ 4	73̄1̄	8	+ 7	93̄4̄	2	− 3	541	7	+ 8
52̄3	4	+ 4	63̄1̄	11	+ 9	83̄4̄	6	+ 5	641	12	+10
72̄3	8	− 8	53̄1̄	11	+13	73̄4̄	2	− 2	741	5	− 4
92̄3	2	− 1	43̄1̄	31	−29	53̄4̄	5	+ 5	841	14	+12
11,2̄3	3	+ 2	33̄1̄	3	− 2	43̄4̄	3	+ 4	941	10	− 9
11,2̄4̄	1	− 2	23̄1̄	17	−20	33̄4̄	2	+ 1	11,41	2	− 1
10,2̄4̄	1	+ 2	13̄1̄	11	−13	13̄4̄	3	− 4	13,41	1	− 1
92̄4̄	11	+ 6	031	2	− 1	034	5	+ 5	14,41	1	− 1
82̄4̄	4	− 3	131	1	+ 1	134	3	− 3	13,4̄2̄	2	+ 2
72̄4̄	9	+ 7	231	15	−14	334	4	− 5	11,4̄2̄	2	− 2
62̄4̄	10	− 8	331	7	− 7	434	5	− 5	10,4̄2̄	2	+ 2
42̄4̄	6	− 4	431	5	− 4	534	4	− 3	84̄2̄	8	+ 7
02̄4	3	− 3	531	4	− 5	634	8	− 9	74̄2̄	13	+10
32̄4	4	+ 4	631	1	− 1	10,3̄5̄	1	+ 1	64̄2̄	7	+ 4
42̄4	2	− 1	731	9	+ 9	83̄5̄	2	+ 1	54̄2̄	4	+ 3
52̄4	6	− 5	831	12	−11	73̄5̄	1	+ 1	44̄2̄	10	+10
62̄4	3	− 2	931	4	+ 4	53̄5̄	4	+ 1	24̄2̄	6	+ 6
72̄4	9	− 8	10,31	2	+ 2	43̄5̄	4	+ 4	14̄2̄	5	+ 7
92̄4	3	+ 2	12,31	4	+ 4	33̄5̄	2	+ 1	042	2	− 2
10,2̄5̄	3	− 3	14,31	1	− 1	13̄5̄	2	+ 3	142	6	− 8
92̄5̄	6	+ 6	13,3̄2̄	2	− 3	035	2	0	242	2	− 2
82̄5̄	6	− 5	11,3̄2̄	6	− 5	23̄5̄	2	− 2	342	2	+ 2
32̄5̄	4	+ 5	10,3̄2̄	2	+ 1	335	2	0	442	2	+ 3
12̄5̄	1	0	93̄2̄	2	+ 2	435	3	− 2	542	1	− 1
02̄5	6	− 7	83̄2̄	7	+ 6	535	2	+ 2	642	9	+11
12̄5	4	+ 3	73̄2̄	4	+ 4	635	2	− 2	742	14	−14
22̄5	4	− 5	63̄2̄	6	− 8	935	1	− 1	842	2	+ 1
42̄5	3	− 2	53̄2̄	12	−10	10,35	3	− 3	942	3	− 3
52̄5	4	− 4	43̄2̄	21	−21	73̄6̄	1	− 1	11,42	4	+ 3
10,2̄6̄	2	− 2	33̄2̄	11	−13	63̄6̄	2	+ 2	12,42	3	+ 3
82̄6̄	2	− 2	23̄2̄	4	+ 6	53̄6̄	1	+ 1	13,42	2	− 2
72̄6̄	1	− 2	13̄2̄	5	− 5	43̄6̄	4	+ 4	543	8	−10
52̄6̄	4	+ 4	032	7	− 7	33̄6̄	3	+ 3	443	14	+13
32̄6̄	1	+ 1	132	13	−11	13̄6̄	2	+ 2	24̄3̄	6	+ 4
22̄6̄	5	− 5	232	6	− 6	036	2	0	14̄3̄	4	+ 4
12̄6̄	5	− 4	332	11	−13	336	2	+ 2	243	2	+ 2
02̄6	5	− 5	432	4	+ 4	436	1	0	343	2	+ 2
12̄6	2	+ 2	532	1	+ 1	736	2	− 2	543	6	− 4
32̄6	2	+ 2	632	4	− 4	437̄	1	+ 1	54̄4̄	3	− 3
42̄6	1	+ 1	732	1	0	337	2	− 1	44̄4̄	8	+ 7
82̄6	1	− 2	832	12	− 8	137	1	+ 1	34̄4̄	4	+ 4
52̄7̄	2	+ 2	932	3	− 5	040	7	− 3	044	2	+ 3
42̄7̄	1	− 2	10,32	2	+ 3	140	4	− 3	144̄	3	+ 5
32̄7̄	1	− 2	12,32	1	0	240	12	+11	444	2	− 2
22̄7̄	2	− 3	14,32	2	− 2	340	19	−16	54̄5̄	2	+ 2
12̄7̄	2	− 2	13,3̄3̄	4	− 4	540	2	− 1	345̄	1	− 1

TABLE 5. *(Continued.)*

hkl	F (exp.)	F (calc.)	hkl	F (exp.)	F (calc.)	hkl	F (exp.)	F (calc.)	hkl	F (exp.)	F (calc.)
245̄	2	+ 1	15̄4	3	+ 5	12,62	2	− 2	573	3	− 4
145̄	2	− 4	054	2	− 2	463̄	5	− 5	574̄	1	− 1
045	5	+ 6	154	4	+ 5	263̄	8	− 8	474̄	2	− 2
446̄	1	+ 1	354	2	− 1	163̄	5	+ 5	374̄	1	− 1
246̄	5	+ 4	454	2	+ 2	263	3	− 3	174̄	1	+ 1
146̄	2	+ 1	554	2	+ 1	363	5	+ 4	074	2	− 1
046̄	2	+ 3	55̄5	1	+ 2	463	14	+10	174	2	− 2
446	1	− 1	155	3	+ 3	563	3	+ 2	374	2	− 4
546	1	− 2	355	4	− 5	564̄	2	− 2	574	2	+ 2
150	11	−12	555	2	− 2	464̄	3	− 2	575̄	1	− 1
250	5	+ 7	55̄6	1	0	164̄	3	+ 2	375̄	2	− 2
350	1	+ 1	45̄6	1	− 1	064	2	+ 2	275̄	1	+ 1
450	3	+ 1	35̄6	1	− 1	264	5	− 5	375	1	+ 1
550	2	− 2	25̄6	1	+ 1	464	4	− 2	475	2	− 2
650	2	0	15̄6	1	+ 2	565̄	2	− 3	180	3	− 5
750	8	+ 6	356	1	− 2	375̄	1	+ 1	380	1	− 2
950	3	+ 2	060	10	− 9	265̄	1	0	580	1	+ 1
10,50	1	+ 2	160	2	+ 3	165	3	+ 4	680	2	− 3
11,50	2	− 2	360	10	+ 9	265	2	− 2	880	2	− 2
12,50	1	+ 1	460	3	+ 3	465	1	+ 1	10,8̄1	1	+ 1
13,50	1	− 2	560	6	+ 4	170	2	0	98̄1	1	0
12,5̄1	2	+ 2	660	2	+ 2	270	3	− 3	88̄1	3	− 2
11,5̄1	1	+ 1	10,60	2	− 2	370	1	+ 2	78̄1	3	− 2
10,5̄1	1	− 1	11,60	1	− 1	470	4	+ 5	68̄1	3	− 2
95̄1	6	+ 4	12,60	1	+ 2	570	2	− 3	58̄1	1	− 2
85̄1	6	− 6	12,6̄1	1	− 1	770	5	− 4	28̄1	2	+ 1
75̄1	1	+ 1	11,6̄1	1	− 1	870	1	− 1	18̄1	2	+ 3
55̄1	2	− 4	10,6̄1	3	− 1	970	3	− 3	081	2	+ 2
45̄1	7	+ 7	96̄1	1	− 1	10,70	3	− 3	181	3	− 4
35̄1	7	+ 6	86̄1	1	+ 1	11,7̄1	2	− 2	281	3	+ 3
25̄1	3	+ 1	76̄1	2	− 1	10,7̄1	1	− 1	381	5	+ 5
15̄1	8	− 7	66̄1	1	− 2	97̄1	3	− 2	481	1	+ 1
051	8	+ 7	56̄1	5	+ 5	77̄1	3	− 2	581	1	+ 1
251	2	+ 2	46̄1	3	+ 2	67̄1	2	+ 2	681	2	− 2
351	2	+ 3	36̄1	2	+ 1	47̄1	4	+ 3	882̄	1	− 1
451	10	+ 6	26̄1	3	− 2	37̄1	2	+ 3	782̄	3	− 3
651	7	+ 8	16̄1	8	− 7	27̄1	3	+ 2	482̄	3	+ 3
751	6	+ 3	061	12	−10	17̄1	6	− 6	382̄	1	+ 1
10,51	2	+ 1	161	3	+ 3	071	10	− 9	282̄	2	+ 2
12,51	2	+ 2	261	2	+ 4	171	4	− 4	182̄	1	+ 1
13,5̄2	1	+ 2	361	5	+ 4	271	3	− 2	082	4	+ 3
10,5̄2	5	− 5	461	2	+ 2	371	2	+ 2	182	2	+ 2
95̄2	1	+ 1	561	3	+ 2	471	4	+ 3	282	4	+ 4
85̄2	2	− 1	661	4	− 3	571	3	− 2	382	2	+ 2
75̄2	3	0	761	1	+ 2	671	2	− 3	482	2	+ 2
65̄2	8	+ 7	861	3	− 3	771	4	− 3	583	2	− 3
55̄2	9	+ 9	961	3	+ 2	871	2	− 2	882	1	+ 1
35̄2	12	+14	10,61	2	+ 1	10,71	3	− 3	982	1	− 1
25̄2	6	− 7	11,61	1	+ 1	11,71	2	+ 2	583̄	1	+ 1
15̄2	1	− 1	12,61	2	+ 2	97̄2	1	− 1	483̄	2	+ 1
152	3	+ 3	12,6̄2	1	− 1	87̄2	1	− 1	083	3	+ 3
252	3	+ 2	10,6̄2	3	− 3	77̄2	1	− 1	183	2	− 2
452	9	+ 9	96̄2	3	− 3	472̄	3	+ 3	283	3	+ 5
552	1	− 1	86̄2	3	− 3	372̄	5	− 5	383	2	− 2
652	2	+ 2	76̄2	2	− 2	275̄	4	+ 4	483	3	+ 3
752	5	+ 5	66̄2	2	− 2	172̄	7	− 8	484̄	1	− 1
852	2	+ 1	56̄2	1	0	072	7	− 7	384̄	1	+ 1
952	2	+ 3	36̄2	3	− 3	172	4	+ 5	184̄	3	+ 4
10,52	6	+ 4	26̄2	10	−11	372	2	+ 4	084	1	+ 2
11,52	3	+ 3	16̄2	2	+ 2	472	2	− 1	284	2	+ 2
55̄3	9	+11	062	7	− 6	572	5	− 4	484	3	+ 1
45̄3	5	− 6	162	5	− 5	672	1	− 2	190	4	+ 2
35̄3	4	+ 4	262	2	+ 4	772	1	− 2	290	1	0
25̄3	2	+ 2	362	4	− 4	872	1	− 1	390	4	+ 3
15̄3	2	− 2	462	9	− 8	972	1	0	490	1	+ 1
053	6	− 7	562	5	+ 5	10,72	4	− 4	590	2	+ 3
153	3	+ 4	662	6	− 6	573̄	2	− 2	690	1	− 2
453	2	+ 2	762	5	+ 5	373̄	4	− 5	79̄1	1	− 1
553	2	+ 4	862	1	+ 1	073	3	− 3	69̄1	1	− 1
55̄4	3	+ 3	962	4	+ 3	173	4	+ 5	59̄1	2	+ 1
25̄4	4	+ 4	10,62	2	+ 2	373	4	− 4	39̄1	1	0

TABLE 5.   (*Continued.*)

hkl	F (exp.)	F (calc.)	hkl	F (exp.)	F (calc.)	hkl	F (exp.)	F (calc.)	hkl	F (exp.)	F (calc.)
29$\bar{1}$	2	− 2	69$\bar{2}$	1	− 1	592	1	+ 1	2,10,$\bar{1}$	2	− 2
19$\bar{1}$	4	+ 3	39$\bar{2}$	1	+ 1	69$\underline{2}$	1	+ 1	1,10,$\bar{1}$	1	− 2
091	2	+ 2	29$\bar{2}$	2	− 2	59$\bar{3}$	1	+ 1	1,10,1	1	+ 2
191	6	+ 5	19$\bar{2}$	2	+ 3	393	3	+ 4	3,10,1	2	− 3
291	4	+ 3	092	2	+ 2	1,10,0	1	+ 2	5,10,1	3	− 4
391	4	+ 3	192	1	0	3,10,0	1	+ 2	4,10,$\bar{2}$	3	− 3
591	1	+ 1	292	2	+ 1	5,10,0	2	− 2	2,10,$\bar{2}$	1	− 1
691	2	− 2	392	1	0	5,10,$\bar{1}$	1	+ 1	1,10,$\bar{2}$	1	− 1
891	2	+ 3	492	1	− 1	3,10,$\bar{1}$	1	+ 2	3,10,2	3	− 3
79$\bar{2}$	1	− 1									

X-Ray rotation photographs were taken with the crystals mounted successively about the three principal axes and [101] to determine the cell dimensions, which were obtained by measurement of the layer lines: $a = 12\cdot22$, $b = 8\cdot28$, $c = 5\cdot82$ Å, and $\beta = 93° 18'$. The density, $1\cdot178$, determined by flotation in sodium iodide solution, agreed well with that calculated ($1\cdot176$) for two molecules of $C_{16}H_{16}$ per unit cell. Intensity data were obtained by means of Weissenberg moving-film photographs about *a*, *b*, and *c*, for the zero and several layers by the equi-inclination method up to a limit of $\mu = 20°$. Multiple films were used in the usual ways, and the intensities were estimated visually by comparison with a calibrated film strip. Some 1200 different planes gave reflexions which were observed; the structure amplitudes for each were calculated from the intensities by applying the usual corrections, and they were placed on the absolute scale by Wilson's statistical method. A few reflexions were lost by the geometrical limitations of the Weissenberg camera, but on account of the high temperature factor ($B = 5\cdot0 \times 10^{-16}$ Å²) the numerical values of these F's would be expected to be small, and no attempt was made to correct for their absence.

All the Fourier syntheses were computed manually by means of Beevers–Lipson strips, the *x* and *y* values being summed at intervals of 1/120ths of the cell edge, and *z* at 1/60ths. The structure amplitudes were also computed manually, and the final set were used to construct an experimental atomic scattering curve having the following values for f_c :

$(\sin\theta)/\lambda$	0	0·1	0·2	0·3	0·4	0·5	0·6
f_c	6·00	4·72	2·95	1·62	0·85	0·48	0·32

No account was taken of the hydrogen atoms. The agreement between the calculated and the experimental values of the structure-amplitudes is shown in Table 5. About 300 planes with unobserved intensities have been omitted from this Table; all of these gave very small values for F(calc.). The discrepancy as given by the usual residual *R* is 0·123 for the {*h*0*l*}, and 0·163 for all the {*hkl*} terms.

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