of 0·05 × 0·05 mm. The (0*kl*) reflexions were recorded as the equatorial layer line of a moving-film photograph. The strong and weak reflexions were correlated by the multiple-film technique, and were estimated visually. Structure amplitudes, derived by the usual formulae for a mosaic crystal, are listed in Table 2.

In the Fourier synthesis the axial subdivisions were $\frac{1}{60}b = 0{\cdot}242$ A. and $\frac{1}{60}c = 0{\cdot}270$ A. The co-ordinates estimated for all the atoms were used in a recalculation of the structure factors, and the results are given in Table 2. The average discrepancy, referred to the measured structure factors, is 14 %. A systematic survey for all the reflexions possible with copper radiation was made, but to save space in the table only the structure factors actually measured are listed. These amount to only about 40 % of the possible number, owing to the small size of the crystal.

In the calculation of the structure factors the atomic scattering curves given in the *International Tables for the Determination of Crystal Structures* were employed, and were corrected for temperature according to the Debye-Waller formula, the constant B being given the value 2×10^{-16} A.^{2} The absolute scale was obtained by correlation with these calculated values.

In conclusion, one of us (W. P. B.) wishes to express his thanks to Messrs Imperial Chemical Industries Ltd., for a grant which enabled him to take part in this work.

References

Bunn, C. W. & Garner, E. V. (1947). *Proc. Roy. Soc.* A, **189**, 39.

Harker, D. (1936). *J. Chem. Phys.* **4**, 381.

MacGillavry, C. H. (1941). *Rec. Trav. Chim. Pays-Bas*, **60**, 605.

Morrison, J. D. & Robertson, J. M. (1949). *J. Chem. Soc.*

Müller, A. (1928). *Proc. Roy. Soc.* A, **120**, 437.

Pauling, L. (1939). *The Nature of the Chemical Bond.* Cornell: University Press.

Robertson, J. M. (1943). *Nature, Lond.*, **152**, 411.

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The Crystal Structure of Diethyl Terephthalate

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The crystal structure of diethyl terephthalate has been determined. The crystals are monoclinic, with cell dimensions $a = 9{\cdot}12$ A., $b = 15{\cdot}39$ A., $c = 4{\cdot}21$ A., $\beta = 93{\cdot}4°$. The space group is $P2_1/n$ with two centrosymmetrical molecules in the unit cell. The atomic co-ordinates were obtained from packing considerations and were refined by c-axis Fourier projections and line syntheses. The molecule is planar, with the exception of the terminal CH_3 groups, and the only intermolecular forces are of the van der Waals type, the shortest distance between adjacent molecules being 3·4 A.

Introduction

The structure of diethyl terephthalate is of particular interest since no previous detailed X-ray work on aromatic esters has been reported. It was hoped to obtain information concerning the molecular configuration, including the planarity of the molecule, interatomic distances and interbond angles, and also concerning the nature of the intermolecular contacts and binding forces in this type of compound.

Unit-cell and space-group measurements were made on some related esters (Table 1), and diethyl terephthalate was selected as the most suitable for a complete structure determination.

Experimental

Diethyl terephthalate crystallizes from most organic solvents in the form of c-axis needles with predominant {110} faces, terminated by the two {101} dome faces. There is also a good (101) cleavage. Pseudo-orthorhombic contact twins occur frequently by 180° rotation about the c axis, but these could be identified by their anomalous extinction effects between crossed nicols.

The unit-cell dimensions were obtained with an accuracy of ±0·015 A. from symmetrical oscillation photographs taken on a camera with calibrated knife-edges. Measurements were made from zero-layer-line reflexions for which θ approached 90°, and Cu $K\alpha_1$ and

Table 1

Compound	Formula	Space group	a (A.)	b (A.)	c (A.)	β (°)	No. of molecules
Diethyl terephthalate	$C_6H_4(COO.C_2H_5)_2$	$P2_1/n$	9·12	15·39	4·21	93·4	2
Dimethyl terephthalate	$C_6H_4(COO.CH_3)_2$	$Pbca$	22	7·1	5·7	—	4
Diethylol terephthalate	$C_6H_4(COO.C_2H_4OH)_2$	$P2_1/a$	25·9	5·48	8·58	99	4
Ethylene dibenzoate	$C_2H_4(OOC.C_6H_5)_2$	$Pbca$	21·9	16·36	7·60	—	8

Cu $K\alpha_2$ were resolved. The density determined at 18° C. by flotation in sodium iodide solution was 1·243 g.cm.$^{-3}$, indicating two molecules of $C_6H_4(COO.C_2H_5)_2$ in the unit cell. (The theoretical density = 1·248 g.cm.$^{-3}$.)

Intensity data using Cu $K\alpha$ radiation were recorded on Weissenberg photographs for the zero to third *a*-axis layers, zero to fifth *b*-axis layers, and zero and first *c*-axis layers, and were estimated visually by the multiple-film technique (Robertson, 1943) with slight modification. Several very strong reflexions were re-estimated more accurately from 5° oscillation photographs, on which they were compared with reflexions of reliable intensity and appropriately longer exposure. The experimental structure amplitudes of 665 planes were obtained after application of the necessary correction factors to the observed intensities. Few reflexions were recorded with $\sin\theta > 0{\cdot}8$, and the series terminated naturally.

Preliminary structure

In the space group $P2_1/n$ there are four general positions: x, y, z; $\bar{x}, \bar{y}, \bar{z}$; $\frac{1}{2}+x, \frac{1}{2}-y, \frac{1}{2}+z$; $\frac{1}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$. Since the structure has only two $C_6H_4(COO.C_2H_5)_2$ molecules in the unit cell, these must have the centrosymmetrical *trans*-configuration, and the centres were taken at 0, 0, 0 and $\frac{1}{2}, \frac{1}{2}, \frac{1}{2}$. There were then twenty-four unrelated atomic parameters to be determined.

Considering the short *c*-axis length of 4·21 A., it seemed unlikely that the plane of the molecule would be much inclined to (001), and if the approximate orientation of the molecules was determined, the *x* and *y* coordinates could be readily refined by Fourier projections parallel to this axis.

In the preliminary calculations the molecule was assumed planar with bond lengths

C–C (aromatic) = 1·40 A.,
C (aromatic)–C (aliphatic) = 1·50 A.,
CH_2–CH_3 = 1·55 A.,
C=O = 1·25 A.,
and C–O = 1·45 A.,

and with the normal interbond angles.

From a wire model constructed to allow free rotation of the skeleton molecules about their centres it was found that only two orientations were possible from packing considerations, and this was so for any moderate variation from the assumed configuration. In one of these the length of the molecule was in the direction of the *b* axis, in the other it was approximately parallel to [21$\bar{2}$]. The latter arrangement allowed more satisfactory clearance between adjacent molecules, and was favoured by a preliminary optical examination of the crystals, which showed the direction of highest refractive index γ to be nearly parallel to the [10$\bar{1}$] axis. Series of (*hk*0) structure amplitudes were calculated for systematic variations about these two positions, and comparison with the observed values showed that the orientation with the length of the molecule in the [21$\bar{2}$] direction was fundamentally correct.

After further adjustment the signs of about 50 (*hk*0) reflexions were determined, and refinement of the *x* and *y* co-ordinates was continued by successive Fourier approximations.

Fourier refinement

The structure was very suitable for projection on (001). There was no overlap of molecules on this plane, and an initial Fourier synthesis of forty-four terms showed resolution of all the atoms. The refinement was complete after the fourth synthesis, which included 108 of the 110 (*hk*0) reflexions observed. The signs of the two remaining terms were unreliable, but their amplitudes were small. The *x* and *y* co-ordinates were measured from the peaks in the final electron-density projection map, which is shown in Fig. 1.

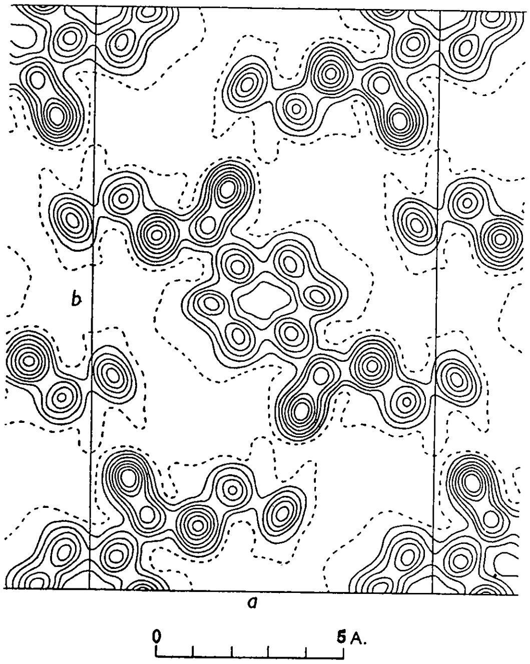

Fig. 1. Electron-density map projected on (001). Contours at intervals of 1 electron per A.^{2}, the unit contour being dotted.

It remained to determine the *z* co-ordinates. The three strongest reflexions observed were (101), (031) and (021); and these gave an indication of the tilt of the molecule from the (001) plane. The *z* co-ordinates were calculated for the molecule in each plane, and experimental and calculated structure amplitudes compared for intermediate values. In this way moderate agreement was obtained for *F*(*hkl*)'s.

Consideration of the structure indicated that projections parallel to the *a* or *b* axis would show very little resolution of the atoms, and would be of no use in refining the *z* co-ordinates. At this stage the signs of thirty-six large (*hkl*) terms could be fixed, and from these a three-dimensional Fourier synthesis was computed along lines parallel to the *c* axis through each of the peaks in the (001) projection. Structure amplitudes were recalculated using the revised z co-ordinates, and showed a marked improvement in agreement. The synthesis was repeated to include more terms, and continued to refine rapidly. The final z co-ordinates were measured from the fourth-line syntheses which included 649 terms, the remaining sixteen being of uncertain sign but too small to have an appreciable effect.

The interatomic distances were calculated, and were in reasonable agreement with the generally accepted values with the exception of the C_4–O_1 and C_5–O_1 distances, which were respectively shorter and longer than comparable C–O distances found in similar types of compound. To check the position of O_1, a section of the three-dimensional Fourier synthesis was plotted at $z=0{\cdot}675$ to include the C_4 and O_1 atoms (Fig. 2). The fractional x co-ordinate of O_1 in the section differed from that in projection by 0·006 (the mean value was finally taken); the remaining x and y co-ordinates were consistent with the projection to within 0·002.

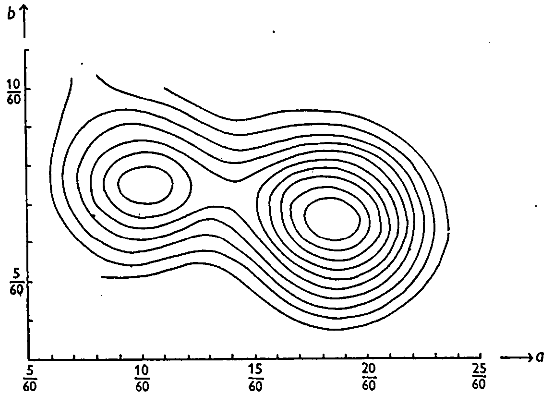

Fig. 2. Part of the section of the three-dimensional Fourier synthesis at $z=0{\cdot}675$, showing C_4 and O_1.

To bring the C_4–O_1 and C_5–O_1 to equal distances of about 1·45 A. a change in the x co-ordinate of O_1 of the order of 0·015 is required. The agreement between the parameters from the projection and from the three-dimensional section suggests that the O_1 position is not in error by this amount. Moreover, the final Fourier syntheses have been computed for almost completely terminated series, which should eliminate possible displacement of the peaks on this account. Neither should any considerable error have been introduced in the measurement of the x and y co-ordinates from the peaks in the (001) projection, since each atom was clearly resolved and had moderately regular electron-density contours from which the centre could be estimated. Many of the experimental structure ampitudes have been estimated from a-, b- and c-axis photographs, and comparison of the three independent observations indicated an agreement to within about 15 % for amplitudes greater than 5, and a decrease to within about 40 % for an amplitude of 2, which was the smallest recorded. That the accuracy of the parameters was limited by the accuracy of the observed structure amplitudes was illustrated by the fact that when the ($hk0$) reflexions were recalculated with the x co-ordinate of $O_1=0{\cdot}330$ (corresponding to C_4–O_1 and C_5–$O_1\approx 1{\cdot}45$ A.) instead of the value 0·315 obtained from Fourier refinement, the agreement with the observed $F(hk0)$'s was not very appreciably impaired. However, the uncertainty in the scattering factors to be used must also be borne in mind when the accuracy is estimated from structure-amplitude agreement (see next section).

The atomic co-ordinates finally adopted from measurement of the peaks in the Fourier syntheses are recorded in Table 2.

Table 2. *Fractional atomic co-ordinates*

Atom	x	y	z
C_1	0·153	−0·008	0·990
C_2	−0·070	0·067	0·849
C_3	0·083	0·062	0·841
C_4	0·172	0·125	0·678
C_5	0·408	0·175	0·511
C_6	0·562	0·133	0·517
O_1	0·315	0·110	0·672
O_2	0·112	0·192	0·539

Structure-amplitude agreement

The observed and calculated structure amplitudes are listed in Tables 3 and 4 for the zero and first and second c-axis layers, the relative observed values having been adjusted to the absolute scale by applying the factor $\Sigma\,|F_{\text{calc.}}|\div\Sigma\,|F_{\text{obs.}}|$. In the preliminary structure-amplitude calculations the Hartree scattering factor for carbon modified by a temperature factor

$$\exp\left[-B\{(\sin\theta)/\lambda\}^2\right],$$

where $B=5\times10^{-16}$ A.^{2}, was found to be satisfactory. The large temperature factor required was to be expected from the low melting-point of 44° C. and the weak intermolecular forces in the crystal. When the atomic co-ordinates had been determined more accurately, a more appropriate experimental scattering curve was used in the structure-amplitude calculations:

$\sin\theta$ ($\lambda=1{\cdot}542$ A.)	0	0·1	0·2	0·3	0·4
f_c	6·0	5·7	4·8	3·5	2·2
$\sin\theta$ ($\lambda=1{\cdot}542$ A.)	0·5	0·6	0·7	0·8	0·9
f_c	1·5	1·0	0·6	0·4	0·3

The oxygen contribution was approximated to four-thirds the carbon contribution, and the hydrogen contribution was neglected. The simplest way to allow for the hydrogen atoms would be to take the scattering factors as 7, 8 and 9, for CH, CH_2 and CH_3 respectively. This was tested for the ($hk0$) reflexions, and the results are given in column (ii) of Table 3. Comparison with the figures in column (i) (hydrogen atoms omitted) shows a change in sign of only three small terms included in the final (001) projection, and since no improvement in the agreement between calculated and observed structure amplitudes was effected, the $F(hkl)$'s were computed omitting the hydrogen contributions. In the c-axis line syntheses the areas under the C_1, C_2, C_3 and C_4 peaks were approximately equal, the area under C_5 was some-

Table 3. *Comparison of observed and calculated $F(hk0)$'s*

(i) Hydrogen contributions omitted. (ii) Hydrogen included in the carbon contributions.

Plane	$F_{obs.}$	$F_{calc.}$ (i)	$F_{calc.}$ (ii)
000		208	236
020	19	22	22
040	53	−47	−57
060	13	11	17
080	4	−3	−1
0,10,0	11	8	7
0,12,0	3	−3	−3
0,14,0	3	1	2
0,16,0	10	9	11
110	31	33	38
120	56	−58	−56
130	36	35	51
140	23	18	21
150	12	15	16
160	13	10	8
170	25	−25	−31
180	12	9	9
190	9	−6	−6
1,10,0	<2	1	3
1,11,0	4	2	3
1,12,0	7	−6	−7
1,13,0	<2	2	3
1,14,0	9	6	6
1,15,0	<2	7	7
1,16,0	4	4	4
200	16	15	26
210	16	−16	−14
220	6	−5	−7
230	11	9	7
240	2	2	−4
250	11	9	12
260	3	1	2
270	9	−3	1
280	23	−18	−18
290	4	−2	−5
2,10,0	14	−9	−10
2,11,0	<2	−2	−3
2,12,0	5	1	0
2,13,0	12	9	11
2,14,0	5	3	3
2,15,0	<2	2	2
2,16,0	<2	1	2
310	16	−14	−18
320	2	−1	4
330	2	0	0
340	29	29	30
350	19	−22	−22
360	26	−24	−28
370	15	−13	−22
380	7	−3	−3
390	5	−3	−5
3,10,0	14	11	14
3,11,0	2	1	1
3,12,0	<2	−4	−6
3,13,0	<2	−1	−1
3,14,0	<2	1	0
3,15,0	6	−5	−6
3,16,0	<2	2	3
400	40	−45	−50
410	13	−7	−8
420	8	1	0
430	14	−10	−12
440	6	−2	−2
450	<2	−1	2
460	18	−11	−13
470	5	6	9
480	12	6	6
490	<2	−2	−5
4,10,0	5	−5	−5
4,11,0	8	−6	−8
510	20	−19	−18
520	22	22	24
530	<2	3	6
540	5	2	3
550	12	9	10
560	14	−6	−8
570	16	9	11
580	7	−4	−4
590	<2	−3	−3
5,10,0	<2	3	4
5,11,0	<2	0	1
600	8	−6	−10
610	7	4	2
620	4	4	5
630	5	0	−1
640	3	0	2
650	<2	3	5
660	2	1	0
670	6	1	1
680	20	15	16
690	10	−7	−8
6,10,0	8	6	7
6,11,0	<2	−3	−4
710	5	2	4
720	<2	0	2
730	5	−5	−7
740	9	−6	−7
750	<2	5	5
760	11	10	9
770	12	10	11
780	<2	3	3
790	<2	4	5
7,10,0	5	−5	−5
7,11,0	<2	1	1
800	8	−8	−10
810	3	0	−1
820	5	−4	−4
830	3	0	0
840	9	7	9
850	5	4	4

what lower, and that under the C_6 peak was very appreciably lower. Thus it was evident that the thermal vibrations of the different carbon atoms were not of equal magnitudes, and particularly in the case of C_5 and C_6 they were very asymmetric. It was not possible to estimate these differences quantitatively for each atom, since all the atoms contribute to the majority of the reflexions. For the same reason no separate temperature factor could be estimated for the oxygen atoms.

The factor $\Sigma(|F_{obs.}| - |F_{calc.}|) \div \Sigma |F_{obs.}|$ has a value 0·22 for the $(hk0)$ reflexions, and 0·28 for the complete list of 665 (hkl) reflexions.

Description of the structure

The molecule is planar within the limits of experimental error with the exception of the terminal CH_2–CH_3 bond, which is inclined at 9° to this plane. The perpendicular distances of the atoms from the average plane of the benzene ring are C_1 0·01 A., C_2 0·02 A., C_3 0·00 A., C_4 0·01 A., C_5 0·04 A., C_6 0·28 A., O_1 0·07 A. and O_2 0·03 A.

The bond lengths and interbond angles are shown in Fig. 3. They are probably reliable only to within ±0·05 A. and ±5° respectively, although the C–C distances in the benzene ring are in much better agreement than this, and the average distance is the standard value of 1·39 A. The CH_2–CH_3 bond also has the normal length of 1·55 A., and the C (aromatic)–C (aliphatic) distance of 1·48 A. agrees well with those observed in other molecules, e.g. 1·50 A. in dibenzyl (Jeffrey, 1947). The C=O distance of 1·28 A. may be compared with 1·213 ± 0·026 A. found in the electron-diffraction study of formic acid (Schomaker & O'Gorman, 1947) and 1·33 ± 0·03 A. in pentaerythritol tetracetate (Goodwin

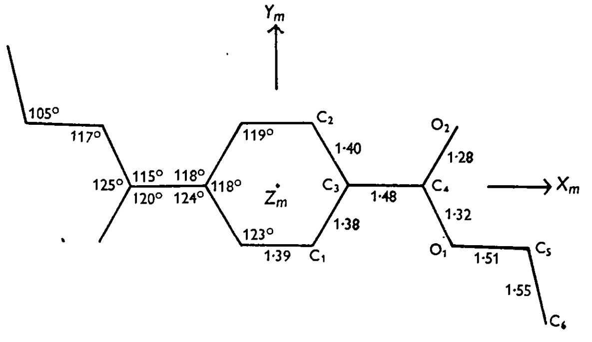

Fig. 3. Molecular dimensions.

Table 4. *Comparison of observed and calculated F(hkl)'s*

(Hydrogen contributions omitted)

Plane	$F_{\text{obs.}}$	$F_{\text{calc.}}$
011	7	−7
021	56	67
031	74	83
041	22	25
051	3	0
061	10	−7
071	8	−8
081	10	−8
091	<2	−1
0,10,1	4	0
0,11,1	3	−1
0,12,1	3	−1
0,13,1	11	10
0,14,1	8	−5
0,15,1	3	1
0,16,1	<2	−1
0,17,1	<2	0
0,18,1	3	2
101	60	86
111	40	44
121	24	27
131	23	20
141	28	−35
151	6	9
161	9	−3
171	14	14
181	14	13
191	11	−9
1,10,1	<2	−1
1,11,1	<2	0
1,12,1	<2	−1
1,13,1	<2	−1
1,14,1	<2	0
1,15,1	<2	−2
1,16,1	4	3
1,17,1	7	3
1,18,1	3	3
211	<2	0
221	6	6
231	5	6
241	4	0
251	7	−7
261	23	14
271	11	−7
281	6	3
291	6	−3
2,10,1	7	−1
2,11,1	7	−5
2,12,1	<2	−3
2,13,1	<2	0
2,14,1	<2	−3
2,15,1	<2	2
2,16,1	9	6
301	6	−3
311	15	14
321	17	−18
331	6	−7
341	<2	−5
351	<2	5
361	<2	1
371	11	12
381	14	−10
391	14	−15
3,10,1	7	−5
3,11,1	<2	0
3,12,1	8	−5
3,13,1	9	8
3,14,1	<2	0
411	4	2
421	<2	−2
431	22	−22
441	<2	1
451	6	3
461	8	−8
471	8	−6
481	7	−4
491	3	−1
4,10,1	<2	3
4,11,1	5	4
4,12,1	<2	−1
501	12	−16
511	16	−14
521	7	−6
531	15	−14
541	7	11
551	<2	−1
561	<2	2
571	<2	2
581	<2	−1
591	<2	1
5,10,1	7	5
5,11,1	5	−4
5,12,1	<2	0
611	10	−2
621	<2	3
631	<2	−3
641	4	−1
651	16	11
661	13	−13
671	8	7
681	<2	1
691	<2	0
6,10,1	<2	6
6,11,1	<2	2
6,12,1	<2	0
701	<2	−3
711	3	−5
721	<2	2
731	3	1
741	<2	1
751	3	−2
761	3	0
771	10	−7
781	<2	8
791	<2	4
7,10,1	<2	4
7,11,1	6	3
811	<2	0
821	<2	−4
831	<2	−2
841	<2	−4
851	<2	−4
861	<2	2
871	<2	7
881	<2	4
891	5	4
8,10,1	<2	−1
$10\bar{1}$	7	5
$11\bar{1}$	17	18
$12\bar{1}$	23	23
$13\bar{1}$	15	−14
$14\bar{1}$	12	12
$15\bar{1}$	18	−22
$16\bar{1}$	2	6
$17\bar{1}$	<2	−1
$18\bar{1}$	8	−4
$19\bar{1}$	4	−2
$1,10,\bar{1}$	14	−11
$1,11,\bar{1}$	14	11
$1,12,\bar{1}$	12	7
$1,13,\bar{1}$	12	9
$1,14,\bar{1}$	7	5
$1,15,\bar{1}$	<2	−1
$1,16,\bar{1}$	<2	0
$1,17,\bar{1}$	<2	−1
$1,18,\bar{1}$	<2	1
$21\bar{1}$	5	−3
$22\bar{1}$	20	22
$23\bar{1}$	12	12
$24\bar{1}$	12	−14
$25\bar{1}$	24	−21
$26\bar{1}$	14	−14
$27\bar{1}$	14	−14
$28\bar{1}$	3	−3
$29\bar{1}$	6	1
$2,10,\bar{1}$	14	13
$2,11,\bar{1}$	2	2
$2,12,\bar{1}$	11	6
$2,13,\bar{1}$	<2	0
$2,14,\bar{1}$	3	−3
$2,15,\bar{1}$	3	−3
$2,16,\bar{1}$	3	3
$30\bar{1}$	3	1
$31\bar{1}$	16	15
$32\bar{1}$	12	−16
$33\bar{1}$	23	−23
$34\bar{1}$	22	−18
$35\bar{1}$	11	−7
$36\bar{1}$	8	−6
$37\bar{1}$	22	20
$38\bar{1}$	<2	−2
$39\bar{1}$	7	−7
$3,10,\bar{1}$	3	−4
$3,11,\bar{1}$	<2	−1
$3,12,\bar{1}$	3	−4
$3,13,\bar{1}$	<2	1
$3,14,\bar{1}$	<2	1
$41\bar{1}$	10	−5
$42\bar{1}$	20	18
$43\bar{1}$	20	−23
$44\bar{1}$	5	5
$45\bar{1}$	14	10
$46\bar{1}$	6	−6
$47\bar{1}$	6	−2
$48\bar{1}$	10	−6
$49\bar{1}$	10	−6
$4,10,\bar{1}$	<2	2
$4,11,\bar{1}$	<2	1
$4,12,\bar{1}$	<2	−1
$50\bar{1}$	10	8
$51\bar{1}$	<2	−3
$52\bar{1}$	6	−3
$53\bar{1}$	10	4
$54\bar{1}$	10	−4
$55\bar{1}$	10	11
$56\bar{1}$	<2	−2
$57\bar{1}$	5	2
$58\bar{1}$	8	4
$59\bar{1}$	11	−9
$5,10,\bar{1}$	11	8
$5,11,\bar{1}$	5	−5
$5,12,\bar{1}$	<2	−3
$61\bar{1}$	8	9
$62\bar{1}$	<2	−2
$63\bar{1}$	7	−5
$64\bar{1}$	8	7
$65\bar{1}$	8	10
$66\bar{1}$	7	5
$67\bar{1}$	2	4
$68\bar{1}$	<2	4
$69\bar{1}$	5	1
$6,10,\bar{1}$	10	−5
$6,11,\bar{1}$	<2	5
$6,12,\bar{1}$	8	−7
$70\bar{1}$	11	−11
$71\bar{1}$	10	−4
$72\bar{1}$	<2	2
$73\bar{1}$	7	9
$74\bar{1}$	13	12
$75\bar{1}$	12	8
$76\bar{1}$	7	3
$77\bar{1}$	<2	−4
$78\bar{1}$	<2	−2
$79\bar{1}$	<2	1
$7,10,\bar{1}$	<2	1
$7,11,\bar{1}$	<2	−2
$81\bar{1}$	<2	1
$82\bar{1}$	11	8
$83\bar{1}$	3	0
$84\bar{1}$	7	5
$85\bar{1}$	<2	−3
$86\bar{1}$	<2	−2
$87\bar{1}$	<2	2
$88\bar{1}$	<2	1
$89\bar{1}$	<2	3
$8,10,\bar{1}$	<2	0
002	18	21
012	14	−15
022	8	−12
032	7	3
042	14	10
052	18	21
062	20	24
072	10	8
082	6	−2
092	6	−3
0,10,2	5	5
0,11,2	12	−11
0,12,2	7	3
0,13,2	<2	−3
0,14,2	<2	0
112	16	−18
122	16	18
132	21	23
142	27	30
152	18	18
162	7	−9
172	4	−4
182	5	−3
192	4	1
1,10,2	5	4
1,11,2	6	4
1,12,2	7	−4
1,13,2	<2	2
1,14,2	<2	0
202	11	9
212	7	2
222	<2	−1
232	20	15
242	14	−12
252	8	7
262	<2	−1
272	5	0
282	6	4
292	5	3
2,10,2	<2	1
2,11,2	<2	−3
2,12,2	<2	−1
312	11	−8
322	8	−3

Table 4 (*contd.*)

Plane	$F_{obs.}$	$F_{calc.}$	Plane	$F_{obs.}$	$F_{calc.}$	Plane	$F_{obs.}$	$F_{calc.}$	Plane	$F_{obs.}$	$F_{calc.}$
332	< 2	0	712	< 2	2	$21\bar{2}$	16	−16	$43\bar{2}$	7	2
342	10	8	722	< 2	2	$22\bar{2}$	16	−16	$44\bar{2}$	12	− 8
352	11	− 8	732	5	0	$23\bar{2}$	17	−15	$45\bar{2}$	8	7
362	< 2	− 5	742	11	− 8	$24\bar{2}$	17	−15			
372	< 2	1	752	8	7	$25\bar{2}$	< 2	− 2	$51\bar{2}$	3	2
382	< 2	4				$26\bar{2}$	8	6	$52\bar{2}$	< 2	1
392	5	− 2	802	< 2	2	$27\bar{2}$	11	8	$53\bar{2}$	6	5
3,10,2	6	6	812	< 2	0	$28\bar{2}$	4	3	$54\bar{2}$	4	3
3,11,2	7	− 5	822	< 2	0	$29\bar{2}$	6	5	$55\bar{2}$	4	− 1
3,12,2	8	− 6	832	< 2	0	$2,10,\bar{2}$	< 2	0			
			842	< 2	1	$2,11,\bar{2}$	3	− 3	$60\bar{2}$	7	− 8
402	17	−15	852	< 2	− 3	$2,12,\bar{2}$	5	− 4	$61\bar{2}$	< 2	5
412	5	2							$62\bar{2}$	6	10
422	< 2	2	$11\bar{2}$	11	5	$31\bar{2}$	15	−14	$63\bar{2}$	5	6
432	4	− 4	$12\bar{2}$	3	− 4	$32\bar{2}$	9	− 7	$64\bar{2}$	10	9
442	< 2	− 3	$13\bar{2}$	11	− 2	$33\bar{2}$	8	− 5	$65\bar{2}$	6	4
452	5	− 2	$14\bar{2}$	6	− 4	$34\bar{2}$	17	15			
			$15\bar{2}$	2	3	$35\bar{2}$	2	0	$71\bar{2}$	8	10
512	6	7	$16\bar{2}$	17	−16	$36\bar{2}$	7	−12	$72\bar{2}$	13	12
522	< 2	− 1	$17\bar{2}$	< 2	− 1	$37\bar{2}$	7	− 4	$73\bar{2}$	4	− 1
532	6	− 5	$18\bar{2}$	< 2	1	$38\bar{2}$	5	− 2	$74\bar{2}$	< 2	− 1
542	8	− 7	$19\bar{2}$	13	11	$39\bar{2}$	6	− 6	$75\bar{2}$	< 2	− 3
552	6	− 6	$1,10,\bar{2}$	13	12	$3,10,\bar{2}$	5	6			
			$1,11,\bar{2}$	6	6	$3,11,\bar{2}$	< 2	− 1	$80\bar{2}$	< 2	4
602	< 2	− 5	$1,12,\bar{2}$	6	4	$3,12,\bar{2}$	< 2	− 3	$81\bar{2}$	< 2	− 1
612	7	− 4	$1,13,\bar{2}$	< 2	− 4				$82\bar{2}$	< 2	1
622	5	5	$1,14,\bar{2}$	< 2	0	$40\bar{2}$	6	− 7	$83\bar{2}$	< 2	− 1
632	12	−12				$41\bar{2}$	7	− 7	$84\bar{2}$	< 2	0
642	6	7	$20\bar{2}$	13	15	$42\bar{2}$	5	5	$85\bar{2}$	6	4
652	< 2	− 2									

To economize in space the (*hk*3), (*hk*4), and (*hk*5) structure amplitudes are not recorded. The factor $\Sigma(|F_{obs.}| - |F_{calc.}|) \div \Sigma|F_{obs.}|$ is 0·27 for these planes.

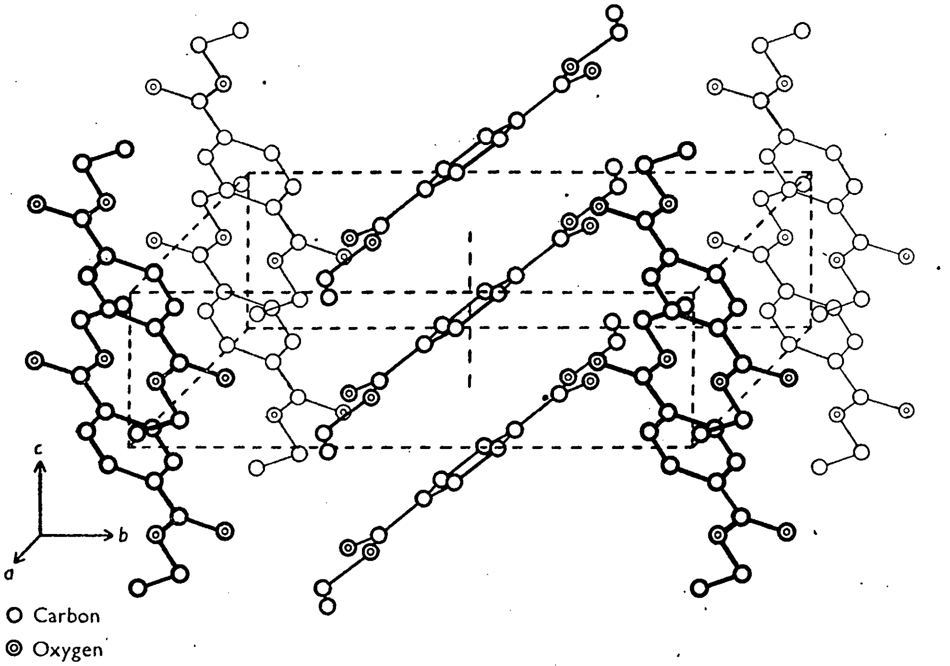

Fig. 4. Arrangement of the molecules in the unit cell.

& Hardy, 1938). Of the C–O distances which have been recorded the majority are within the range 1·43 ± 0·03 A. The distance of 1·51 A. found in diethyl terephthalate is considerably longer than this, being equal to the sum of the carbon radius as in diamond and the oxygen radius as in hydrogen peroxide, with no correction for electronegativity difference. The other C–O distance is 1·32 A., but since the accuracy of the bond lengths is not particularly high, it is not profitable to discuss the difference in the two C–O measurements until the structures of further compounds of this type have been studied.

The orientation of the molecule in the unit cell is

	X_m	Y_m	Z_m
a	55°	147°	87°
b	48°	61°	57°
c^*	118°	104°	33°

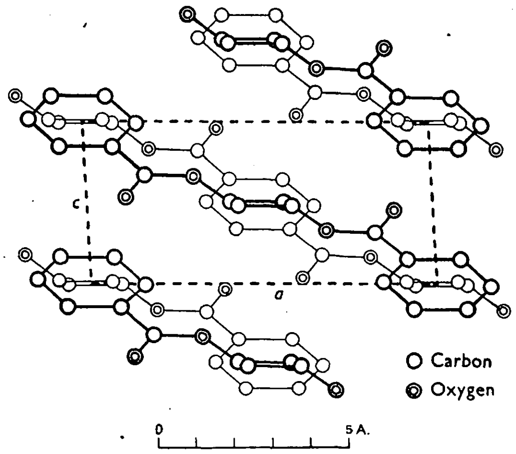

Fig. 5. Diagrammatic projection of the structure on (010).

where X_m, Y_m, Z_m are three orthogonal axes of the molecule defined by the plane and perpendicular of the benzene ring (Fig. 3). The arrangement is illustrated in Fig. 4. The reason for the (101) cleavage, the large (101) structure amplitude, and the direction of very high refractive index in this plane is shown more clearly in Fig. 5, by the diagrammatic projection of the structure on (010).

The shortest intermolecular contacts are between the CH_2 and CH_3 groups of one molecule (e.g. with centre at 0, 0, 0) and the ketonic oxygen atoms of the two adjacent space-group-related molecules (centres at $\frac{1}{2}$, $\frac{1}{2}$, $\frac{1}{2}$ and $\frac{1}{2}$, $\frac{1}{2}$, $\frac{\bar{1}}{2}$). These distances are 3·47 and 3·48 A. for C_5–O_2 and 3·41 and 3·49 A. for C_6–O_2. Other significant intermolecular distances are 3·77 and 3·71 A. respectively between the C_6 atom of one molecule (centre, say, 0, 0, 0), and the C_1 and C_2 atoms of the adjacent molecule (centre 1, 0, 0) and 3·97, 4·00 and 4·25 A. respectively between the same C_6 atom and atoms C_1, O_1 and C_6 of the adjacent molecule (centre 1, 0, $\bar{1}$). Since the perpendicular distance between parallel molecules with centres along the c axis is 3·60 A., there can be no distances shorter than this between these molecules. Hence there are only van der Waals forces between molecules.

The structure is one of layers of molecules at intervals of $\frac{1}{2}b$, with the ketonic oxygen to ethyl group contacts between the layers. This accounts for the observed twinning, since, although the relative tilts of molecules in adjacent layers are different in a twinned crystal, these interlayer contacts are still the same.

In conclusion, I wish to thank Dr A. F. Wells and Dr C. J. Brown for their advice throughout the work.

References

Goodwin, T. H. & Hardy, R. (1938). *Proc. Roy. Soc.* A, **164**, 369.
Jeffrey, G. A. (1947). *Proc. Roy. Soc.* A, **188**, 222.
Robertson, J. M. (1943). *J. Sci. Instrum.* **20**, 169.
Schomaker, V. & O'Gorman, J. M. (1947). *J. Amer. Chem. Soc.* **69**, 2638.

Short Communications

Contributions intended for publication under this heading should be expressly so marked; they should not exceed about 500 *words; they should be forwarded in the usual way to the appropriate Co-editor; they will be published as speedily as possible; and proofs will not generally be submitted to authors. Publication will be quicker if the contributions are without illustrations.*

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The structures of uranium metal.*

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Heat-capacity studies first revealed that uranium metal exists in three modifications. X-ray examination has established that the *alpha* phase, stable to 640° C., is the orthorhombic form with the structure reported by Jacobs & Warren (1937). The *beta* phase, stable between 640 and 760° C., is also a phase of low symmetry, but its structure has not yet been determined. *Gamma*-uranium, stable from 760° C. to the melting-point, is body-centered cubic, the structure reported for uranium at room temperature by McLennan & McKay (1930).

X-ray diagrams of *beta*- and *gamma*-uranium at high temperatures, and also at room temperature, have been obtained. The latter can best be obtained by quenching uranium samples containing chromium or molybdenum in solid solution. In our experience, it is impossible to quench pure *beta*- or *gamma*-uranium to room temperature.

Since uranium at high temperature is a good 'getter', elaborate precautions are necessary to obtain satisfactory X-ray data for the *beta*- and *gamma*-ranges. We have accomplished this by sealing uranium wire in evacuated quartz capillaries. Uranium reacts with quartz, so it was necessary to use columbium spacers between quartz and uranium; the vacuum in the quartz capillary was im-

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