structure factor calculations, with hydrogen contributions included, reduced R to 0·078.

Results

The list of structure factors is given in Table 1. The final coordinates of the atoms are given in Table 2 with the earlier results of Gupta & Sahu (1970) in parentheses. The thermal parameters of the atoms are given in Table 3. The bond lengths and angles are given in Table 4.

Table 4. *Bond lengths and angles*

Bond lengths		e.s.d.
C(1)–C(2)	1·524 (1·543) Å	0·011 (022)° Å
C(2)–C(3)	1·305 (1·320)	0·005 (023)
C(3)–C(4)	1·531 (1·562)	0·010 (022)
C(1)–O(1)	1·230 (1·231)	0·004 (019)
C(1)–O(2)	1·225 (1·246)	0·009 (019)
C(4)–O(3)	1·190 (1·215)	0·010 (012)
C(4)–O(4)	1·294 (1·276)	0·007 (012)
C(5)–C(6)	1·495 (1·495)	0·010 (020)
C(5)–C(5′)	1·333 (1·354)	0·017 (051)
C(6)–O(5)	1·215 (1·218)	0·012 (013)
C(6)–O(6)	1·295 (1·300)	0·009 (014)
C(2)–H(1)	1·05	
C(3)–H(2)	1·03	
C(5)–H(4)	1·11	
O(4)–H(3)	1·00	
O(6)–H(5)	1·05	
Bond angles		**e.s.d.**
C(1)–C(2)–C(3)	123·84 (118·07)°	0·57 (1·33)°
C(2)–C(3)–C(4)	122·67 (115·97)	0·61 (1·15)
O(1)–C(1)–O(2)	124·43 (126·35)	0·80 (1·33)
O(1)–C(1)–C(2)	116·20 (113·33)	0·70 (1·00)
O(2)–C(1)–C(2)	119·20 (119·50)	0·67 (1·17)
O(4)–C(4)–O(3)	125·02 (125·53)	0·80 (1·17)
O(4)–C(4)–C(3)	113·16 (114·70)	0·62 (1·03)
O(3)–C(4)–C(3)	121·73 (116·70)	0·62 (1·03)
C(5)–C(6)–O(5)	119·63 (120·47)	0·63 (1·12)
C(5)–C(6)–O(6)	115·85 (114·52)	0·73 (1·08)
O(5)–C(6)–O(6)	122·85 (124·64)	0·63 (1·10)
C(6)–C(5′)–C(5)	121·75 (123·50)	0·60 (1·20)
O(4)–H(3)–$_b$O(2)	166·83	
O(6)–H(5)–$_b$O(1)	167·95	

Conclusion

The evidence produced earlier by Gupta & Sahu (1970) has been confirmed by this analysis. The molecular dimensions of the two kinds of 'molecules' ($KC_4H_3O_4$ and $C_4H_4O_4$) are significantly different ($>2\sigma$). The dimensions of the molecule in the special position $\frac{1}{2},\frac{1}{2},\frac{1}{2}$ show this to be a molecule of fumaric acid because of the un-ionized carboxyl groups in it, the bond lengths and angles agreeing fairly well with values reported for the fumaric acid molecule by Brown (1966) and Bednowitz & Post (1966), Table 5. Moreover, the positions of the hydrogen atoms and their distances from the nearest oxygen atoms show quite conclusively which of them are of the type O(H) and which carboxyl groups are ionized or unionized. The correct chemical composition in this crystal is therefore $2KC_4H_3O_4.C_4H_4O_4$.

Table 5. *Comparison of molecular dimensions of the acid molecule in the special position*

	Brown (1966)	Bednowitz & Post (1966)	This work
C–C	1·46 Å	1·49 Å	1·49 Å
C=C	1·36	1·32	1·33
C–O···H	1·29	1·29	1·29
C=O	1·23	1·23	1·22
∠O–C–C	116·7°	116·0°	115·9°
∠O=C–C	119·0	119·5	119·6
∠O=C–O	124·3	124·4	122·9
∠C–C=C	122·8	122·5	121·8

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A refinement of the crystal struture of durene.

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(Received 24 *April* 1972*)*

The crystal structure of durene has been refined by means of three-dimensional Cu $K\alpha$ counter data.

Introduction

The crystal structure of durene (1,2,4,5-tetramethylbenzene) was determined by Robertson (1933) from projections. In the light of a crystal structure determination of 1,2,4,5-tetra-t-butylbenzene (van Bruynsvoort, Eilermann, van der Meer & Stam, 1968) it was of interest for reasons of comparison to have more accurate data for durene than were provided by the old determination.

Experimental

A redetermination of the cell constants gave: $a=11{\cdot}59$ (1), $b=5{\cdot}74$ (1), $c=7{\cdot}04$ (1) Å, $\beta=112{\cdot}8$ (1)°, in good agreement

Table 1. *Final positional and thermal parameters*

The fractional positional parameters of carbon have been multiplied by 10^4, those of hydrogen by 10^3. The U_{ij} (Å^2) have been multiplied by 10^4. Estimated standard deviations are given in parentheses.

	x	y	z	U_{11}	U_{22}	U_{33}	$2U_{12}$	$2U_{23}$	$2U_{31}$
C(1)	917 (3)	1550 (5)	1256 (6)	46 (2)	45 (2)	76 (2)	8 (3)	−13 (3)	50 (3)
C(2)	529 (3)	1659 (6)	−886 (6)	53 (2)	43 (2)	78 (2)	16 (3)	10 (3)	70 (3)
C(3)	−382 (3)	107 (6)	−2093 (5)	57 (2)	55 (2)	63 (2)	21 (3)	4 (3)	51 (3)
C(4)	1903 (4)	3176 (8)	2678 (8)	65 (2)	73 (3)	106 (3)	−23 (4)	−45 (5)	55 (4)
C(5)	1073 (5)	3422 (7)	−1896 (8)	90 (3)	62 (3)	117 (4)	1 (4)	29 (5)	126 (5)

Table 1 (*cont.*)

	x	y	z	B
H(1)	−68 (4)	19 (8)	−366 (7)	3 (1)
H(2)	212 (6)	294 (12)	422 (9)	8 (1)
H(3)	271 (5)	314 (10)	239 (8)	6 (1)
H(4)	162 (6)	473 (12)	215 (10)	8 (2)
H(5)	66 (5)	318 (8)	−337 (7)	5 (1)
H(6)	200 (5)	318 (9)	−149 (8)	6 (1)
H(7)	84 (6)	498 (11)	−154 (11)	7 (2)

with the values reported by Robertson ($a = 11{\cdot}57$, $b = 5{\cdot}77$, $c = 7{\cdot}03$ Å, $\beta = 113{\cdot}3°$). The space group is $P2_1/a$ with $Z = 2$.

654 independent reflexions were collected on a Nonius 3-circle diffractometer by means of the θ–2θ scan technique, using Ni-filtered Cu $K\alpha$ radiation and a pulse height analyser. The crystal dimensions were about 0·5 mm and no absorption correction was applied.

Starting with the coordinates reported by Robertson (1933), an anisotropic block-diagonal least-squares refinement was carried out using weights according to Cruickshank (1961). The hydrogen atoms were located from a difference map and included in the refinement with isotropic thermal parameters. In the last cycles 7 strong reflexions which apparently suffered from extinction were allotted zero weight and the same was done with about 20 very weak reflexions which acquired an unduly large weight. The final R value for the observed reflexions, omitting those affected by extinction, was 6·1 %. The final parameters are listed in Table 1.

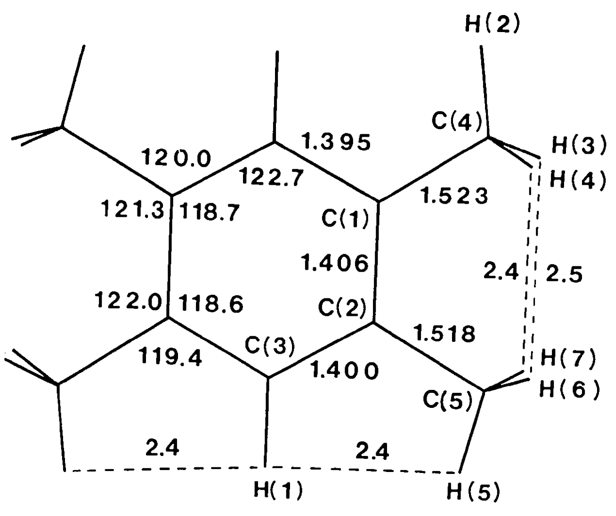

Fig. 1. Bond distances and angles and some intramolecular H–H distances.

Results

The bond lengths and angles are given in Table 2 and Fig. 1. The bond lengths have been corrected for librational motion of the molecule.

Table 2. *Bond distances and angles*

The C–C distances have been corrected for thermal motion; uncorrected values between brackets. Calculated standard deviations: C–C 0·006 Å, C–H 0·07 Å, C–C–C 0·4°.

C(1)–C(2)	1·406 (1·394) Å		
C(2)–C(3)	1·400 (1·391)		
C(1)–C(3′)	1·395 (1·386)	C(2)C(1)C(3′)	118·6°
C(1)–C(4)	1·523 (1·513)	C(2)C(1)C(4)	122·0
C(2)–C(5)	1·518 (1·508)	C(3′)C(1)C(4)	119·4
C(3)–H(1)	1·02	C(1)C(2)C(3)	118·7
C(4)–H(2)	1·03	C(1)C(2)C(5)	121·3
C(4)–H(3)	1·03	C(3)C(2)C(5)	120·0
C(4)–H(4)	0·97	C(2)C(3)C(1′)	122·7
C(5)–H(5)	0·97		
C(5)–H(6)	1·01		
C(5)–H(7)	0·99		

The bond distances in the ring do not differ significantly [σ(C–C) = 0·006 Å, σ(C–H) = 0·07 Å] and the mean C–C distance of 1·400 Å is in excellent agreement with the corresponding value for benzene (1·397 ± 0·001 Å, Langseth & Stoicheff, 1956). The C–CH_3 distances are 1·52 Å (toluene 1·51 ± 0·02 Å, Keidel & Bauer, 1956).

The deviations of the bond angles from the ideal value of 120° are for the greater part significant [σ(C–C–C) = 0·4°] and can be seen as a consequence of the steric interaction between the neighbouring CH_3 groups. In Fig. 1 the relevant intramolecular H···H distances have been indicated. There are two distances of 2·4 and 2·5 Å between hydrogen atoms of adjacent CH_3 groups which may be considered as real contacts. These cause the angles C(4)C(1)C(2) and C(5)C(2)C(1) to increase by about 2°. At the same time there are two short distances of 2·3 and 2·4 Å between H(2) and H(1′) and between H(5) and H(1) respectively, which prevent the angles C(4)C(1)C(3′) and C(5)C(2)C(3) from decreasing. Therefore, compensation for the angular increase of the first-mentioned two angles has to come from the angles C(2)C(1)C(3′) and C(1)C(2)C(3). Since the ring stays planar the angle C(1′)C(3)C(2) has to increase accordingly.

The carbon skeleton is planar within 0·001 Å. The methyl groups are symmetrical with respect to the benzene ring such that H(2) and H(5) are coplanar with it (Fig. 1).

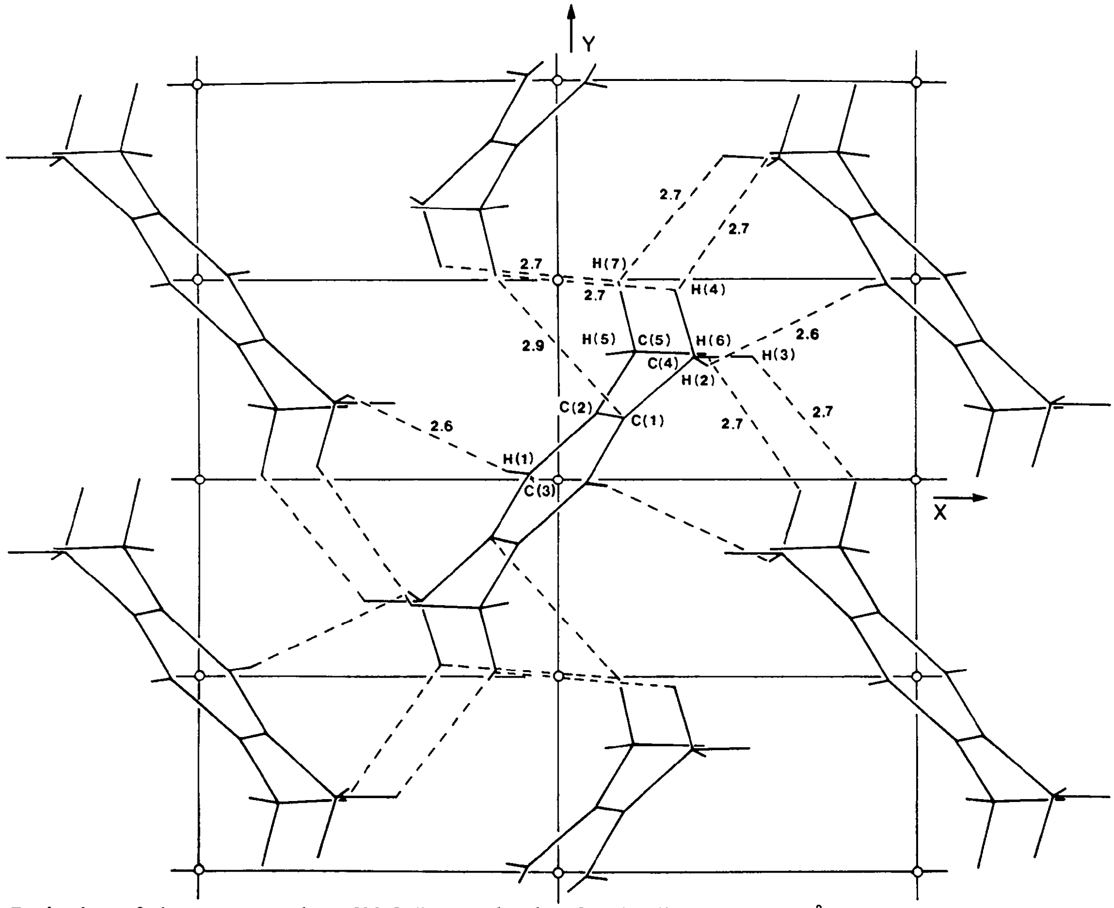

Fig. 2. Projection of the structure along [001]. Intermolecular C···H distances ≤3·0 Å and H···H distances ≤2·7 Å have been indicated.

In Table 3 the axes of the thermal ellipsoids are given relative to **a**, **b** and **c***. A rigid body analysis according to Cruickshank (1956) gave good agreement between observed and calculated thermal parameters (Table 4). The axis of largest libration is in the plane of the benzene ring and perpendicular to the line C(3)–C(3′), both within 1°. The next largest libration axis makes an angle of about 14° with the ring normal. The corrections to the bond lengths range from 0·009 to 0·012 Å.

Table 3. *Principal axes, U_i (Å^2) and their direction cosines l_i of the thermal ellipsoids, relative to* **a**, **b** *and* **c*** *respectively*

	U_i	l_1	l_2	l_3
C(1)	0·0782	−0·1907	−0·2321	+0·9538
	0·0493	−0·6962	−0·6531	−0·2981
	0·0397	−0·6921	+0·7209	+0·0370
C(2)	0·0800	+0·1663	+0·1578	+0·9734
	0·0485	+0·7222	+0·6526	−0·2292
	0·0370	+0·6714	−0·7411	+0·0054
C(3)	0·0443	+0·7008	−0·7123	+0·0396
	0·0671	+0·6125	+0·6291	+0·4786
	0·0617	+0·3658	+0·3112	−0·8771
C(4)	0·1195	−0·2297	−0·4056	+0·8847
	0·0736	+0·7837	−0·6161	−0·0790
	0·0547	+0·5771	+0·6752	+0·4594
C(5)	0·1259	+0·2952	+0·1871	+0·9369
	0·0710	+0·7621	−0·6375	−0·1128
	0·0506	−0·5762	−0·7474	+0·3308

Table 4. *Principal axes of librational and translational tensors relative to* **a**, **b**, *and* **c***

Libration			
Eigenvalue	Direction cosines		
33·7 (°)^2	0·632	0·770	0·086
24·4	0·769	−0·610	−0·190
11·5	0·094	−0·186	0·980
Translation			
Eigenvalue	Direction cosines		
0·0654 Å^2	0·013	0·055	0·998
0·0484	0·695	0·718	−0·049
0·0267	0·719	−0·694	0·029

$$\left(\frac{\sum \Delta U_i^2}{m-n}\right)^{1/2} = 0·0015 \text{ Å}^2$$

The packing is shown in Fig. 2 with the shorter intermolecular distances indicated.

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