

We thank Dr R. Stephens and Professor J. C. Tatlow for suggesting this problem and providing suitable crystals, Professor J. C. Robb for his interest in this work, and Dr J. C. Dewan for assistance with the low-temperature apparatus. We are indebted to the University of Birmingham for funds to purchase the diffractometer and for the award of a Research Studentship (to M.J.H.).

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## The Crystal Structure of 3,5-Di-*t*-butylbenzoic Acid (DTBB)

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(Received 17 February 1976; accepted 30 March 1976)

Crystals of 3,5-di-*t*-butylbenzoic acid are monoclinic, space group  $C2/c$  with  $a = 36.723$  (7),  $b = 9.500$  (1),  $c = 17.963$  (9) Å,  $\beta = 111.01^{\circ}$  (1) and  $Z = 16$ . The structure was solved by direct methods and refined by full-matrix least-squares calculations to a final  $R$  of 0.071 for 2712 observed reflexions. The positions of the hydrogen atoms were located from a difference Fourier synthesis. Bond lengths and bond angles in the two independent molecules in the asymmetric unit are very similar. The structure consists of dimers linked together by hydrogen bonds around a twofold axis in molecule *A* and about centres of symmetry in molecule *B*.

### Introduction

The determination of the crystal structure of the DTBB compound was undertaken as a continuation

of the structural studies of derivatives of benzoic acid, carried out in our Department. In the present paper we pay attention to some interesting features: the interactions between the *t*-butyl and the carboxyl

groups, the influence of these t-butyl groups on the bond lengths and bond angles in the benzene rings, and the conformation of the t-butyl groups.

### Experimental

A purified specimen of DTBB was kindly supplied by Drs M. Colomina and C. Turrion of the Departamento de Termoquímica of our Institute. Single crystals were obtained by slow evaporation from ethanol. The density was measured by flotation in an aqueous potassium iodide solution. Accurate cell constants were obtained by least-squares treatment of 15 high-angle reflexions recorded with Mo  $K\alpha$  radiation. Crystal data are listed in Table 1.

Intensity data were collected with a Philips PW 1100 diffractometer using a graphite monochromator and Mo  $K\alpha$  radiation ( $\lambda=0.7107 \text{ \AA}$ ). 4230 independent reflexions were collected between  $3$  and  $30^\circ$  with the

Table 1. *Crystal data*

Standard deviations, given in parentheses, refer to the least significant digits.

Molecular formula $C_{15}H_{22}O_2$	
Molecular weight 234.339	
Space group $C2/c$	
$a=36.723$ (7) $\text{\AA}$	$D_x=1.06 \text{ g cm}^{-3}$
$b=9.500$ (1)	$Z=16$
$c=17.963$ (9)	$F(000)=2048$
$\beta=111.01$ (1) $^\circ$	$V=5849.987 \text{ \AA}^3$

Table 2. *Coefficients for the weighting scheme*

	$a$	$b$	$\sin \theta/\lambda < 0.20$	$c$	$d$
$ F_o  \leq 0.05$					
$0.05 <  F_o  \leq 1.83$	3.621	-0.318	$0.20 < \sin \theta/\lambda \leq 0.33$	3.724	-7.931
$1.83 <  F_o  \leq 7.25$	2.609	0.024	$0.33 < \sin \theta/\lambda \leq 0.56$	-1.511	7.555
$7.25 <  F_o  \leq 9.63$	3.767	-0.112	$0.56 < \sin \theta/\lambda$	-25.410	50.708
$9.63 <  F_o  \leq 18.90$	2.781	-0.042			
$18.90 <  F_o $	1.423	0.026			

Table 3. *The positional ( $\times 10^4$ ) and thermal ( $\times 10^3$ ) parameters for non-hydrogen atoms*

Standard deviations given in parentheses refer to the least significant digits. Thermal factors are those in the expression  $\exp(-2\pi^2 \sum U_{ij} a_i^* a_j^* h_i h_j)$ .

	$x$	$y$	$z$	$U_{11}$	$U_{22}$	$U_{33}$	$U_{12}$	$U_{13}$	$U_{23}$
C(1)	165 (1)	3280 (4)	775 (2)	43 (3)	38 (2)	39 (2)	-4 (2)	20 (2)	0 (2)
C(2)	529 (1)	3560 (4)	745 (2)	44 (3)	39 (2)	39 (2)	-3 (2)	14 (2)	-0 (2)
C(3)	607 (1)	3297 (4)	53 (2)	47 (3)	37 (2)	45 (2)	2 (2)	24 (2)	2 (2)
C(4)	310 (1)	2703 (4)	-585 (2)	54 (3)	46 (2)	43 (2)	-3 (2)	26 (2)	-6 (2)
C(5)	-54 (1)	2372 (4)	-565 (2)	57 (3)	40 (2)	37 (2)	-2 (2)	18 (2)	-2 (2)
C(6)	-125 (1)	2707 (4)	121 (2)	45 (3)	47 (2)	43 (2)	-7 (2)	17 (2)	1 (2)
C(7)	89 (1)	3540 (4)	1523 (2)	53 (3)	47 (2)	34 (2)	-5 (2)	19 (2)	0 (2)
C(8)	1011 (1)	3620 (4)	27 (2)	45 (3)	63 (3)	53 (2)	2 (2)	27 (2)	2 (2)
C(9)	1023 (1)	3517 (6)	-811 (3)	82 (4)	146 (5)	73 (3)	-13 (3)	46 (3)	-4 (3)
C(10)	1302 (1)	2608 (7)	564 (4)	69 (3)	158 (6)	131 (5)	38 (4)	47 (3)	69 (4)
C(11)	1117 (1)	5157 (6)	290 (3)	87 (4)	99 (4)	129 (4)	-37 (3)	69 (3)	-23 (3)
C(12)	-367 (1)	1652 (4)	-1274 (2)	69 (3)	57 (3)	45 (2)	-17 (2)	15 (2)	-11 (2)
C(13)	-679 (2)	974 (9)	-1046 (3)	122 (5)	226 (8)	88 (4)	-109 (5)	39 (4)	-68 (5)
C(14)	-173 (2)	421 (6)	-1559 (3)	141 (5)	88 (4)	94 (4)	-16 (4)	26 (4)	-37 (3)
C(15)	-525 (2)	2663 (7)	-1937 (4)	200 (7)	96 (4)	89 (4)	-32 (4)	-71 (5)	16 (4)
O(16)	383 (0.7)	3735 (3)	2149 (1)	58 (2)	111 (2)	38 (1)	-20 (2)	19 (1)	-13 (1)
O(17)	-253 (0.7)	3525 (3)	1505 (1)	41 (2)	124 (3)	48 (2)	-5 (2)	23 (1)	-5 (2)
C(18)	2122 (1)	4256 (4)	5090 (2)	38 (5)	41 (2)	40 (2)	-4 (2)	18 (2)	2 (2)
C(19)	1886 (1)	3566 (4)	4408 (2)	46 (2)	45 (2)	36 (2)	2 (2)	18 (2)	5 (2)
C(20)	1719 (1)	2285 (4)	4454 (2)	36 (2)	46 (2)	40 (2)	2 (2)	17 (2)	-1 (2)
C(21)	1802 (1)	1706 (3)	5211 (2)	42 (2)	39 (2)	43 (2)	-4 (2)	17 (2)	1 (2)
C(22)	2036 (1)	2358 (4)	5906 (2)	39 (2)	37 (2)	37 (2)	-1 (2)	11 (2)	4 (2)
C(23)	2197 (1)	3661 (4)	5833 (2)	42 (2)	41 (2)	35 (2)	-4 (2)	11 (2)	1 (2)
C(24)	2286 (1)	5664 (4)	5048 (2)	46 (2)	43 (2)	41 (1)	-4 (2)	18 (2)	9 (2)
C(25)	1440 (1)	1504 (4)	3726 (2)	45 (3)	60 (3)	41 (2)	-6 (2)	11 (2)	-13 (2)
C(26)	1612 (1)	63 (5)	3660 (3)	79 (3)	78 (3)	70 (3)	1 (3)	20 (2)	-35 (2)
C(27)	1046 (1)	1292 (5)	3818 (3)	53 (3)	96 (4)	65 (3)	-13 (2)	13 (2)	-18 (3)
C(28)	1369 (1)	2310 (5)	2953 (2)	85 (3)	98 (4)	42 (2)	-24 (3)	4 (2)	-6 (2)
C(29)	2114 (1)	1749 (4)	6739 (2)	58 (3)	43 (2)	41 (1)	-9 (2)	16 (2)	6 (2)
C(30)	2552 (1)	1636 (5)	7189 (2)	71 (3)	86 (3)	57 (3)	-10 (3)	5 (2)	30 (2)
C(31)	1937 (1)	269 (4)	6697 (4)	116 (3)	52 (3)	62 (3)	-21 (3)	35 (3)	12 (2)
C(32)	1928 (1)	2713 (5)	7187 (2)	112 (4)	70 (3)	49 (3)	0 (3)	41 (3)	6 (2)
O(33)	2454 (1)	6359 (3)	5657 (2)	89 (2)	50 (2)	44 (2)	-24 (2)	17 (1)	1 (1)
O(34)	2244 (1)	6112 (3)	4343 (2)	121 (3)	58 (2)	44 (2)	-35 (2)	24 (2)	8 (1)

$\omega$ - $2\theta$  scan mode. 1518 reflexions were considered as unobserved according to the following criterion:  $I < 2\sigma(I)$  where  $I = C_p - C_b$  and  $\sigma^2(I) = C_p + C_b + (0.005)^2 I^2$ ;  $I$  is the integrated intensity and  $\sigma(I)$  is the estimated standard deviation of each intensity. Lorentz and polarization corrections were applied but no absorption correction was made.

### Structure determination and refinement

The structure was solved by direct methods with the *MULTAN* program (Main, Woolfson & Germain, 1971). 22 non-hydrogen atoms were located from an  $E$  map which was computed with 250 reflexions with  $|E| > 2.1$ . Refinement of this model by full-matrix least-squares calculations led to an  $R$  value of 0.10. The H atoms were then located from a difference map and included in the refinement with fixed isotropic temperature factors assigned to them, equal to those of the corresponding C atoms to which they are bonded. The quantity minimized was  $\sum w(\Delta F)^2$  where  $w = k/\sigma_1^2\sigma_2^2$ ,  $\sigma_1 = a + b|F_o|$  and  $\sigma_2^2 = c + d \sin \theta/\lambda$  with the coefficients given in Table 2. The final unweighted and weighted agreement indices were  $R = 0.076$  and  $R_w = 0.071$ , where  $R_w = (\sum w\Delta^2 / \sum w|F_o|)^{1/2}$ .

The atomic scattering factors used were taken from *International Tables for X-ray Crystallography* (1974), computations were made with programs of the X-RAY 70 system (Stewart, Kundell & Baldwin, 1970) and weighting schemes were computed with the *PESOS* program (Martínez-Ripoll & Cano, 1975).

Table 3 shows atomic parameters and anisotropic temperature coefficients for the non-hydrogen atoms

with their standard deviations. Table 4 give the final coordinates for the H atoms.\*

\* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31766 (18 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 4. *Positional parameters for hydrogen atoms*  
( $\times 10^3$ )

Standard deviations, given in parentheses, refer to the least significant digits.

Molecule A	x	y	z
H(2)	73 (1)	396 (4)	121 (2)
H(4)	35 (1)	250 (4)	-107 (2)
H(6)	-37 (1)	254 (4)	16 (2)
H(91)	95 (2)	255 (7)	-104 (4)
H(92)	83 (2)	421 (7)	-118 (4)
H(93)	128 (2)	374 (7)	-85 (4)
H(101)	122 (2)	158 (7)	338 (4)
H(102)	131 (2)	265 (7)	113 (4)
H(103)	157 (1)	284 (6)	56 (3)
H(111)	139 (1)	537 (5)	27 (2)
H(112)	93 (1)	583 (5)	-10 (3)
H(113)	112 (2)	528 (7)	86 (4)
H(131)	-82 (2)	47 (7)	-155 (4)
H(132)	-90 (2)	150 (7)	-100 (4)
H(133)	-60 (2)	25 (7)	-67 (4)
H(141)	5 (2)	82 (7)	-173 (4)
H(142)	-36 (2)	-4 (7)	-202 (4)
H(143)	-12 (1)	-28 (5)	-115 (3)
H(151)	-66 (2)	352 (7)	-172 (4)
H(152)	-33 (2)	312 (7)	-211 (4)
H(153)	-71 (2)	220 (7)	-231 (4)
H(16)	37 (1)	378 (5)	259 (3)

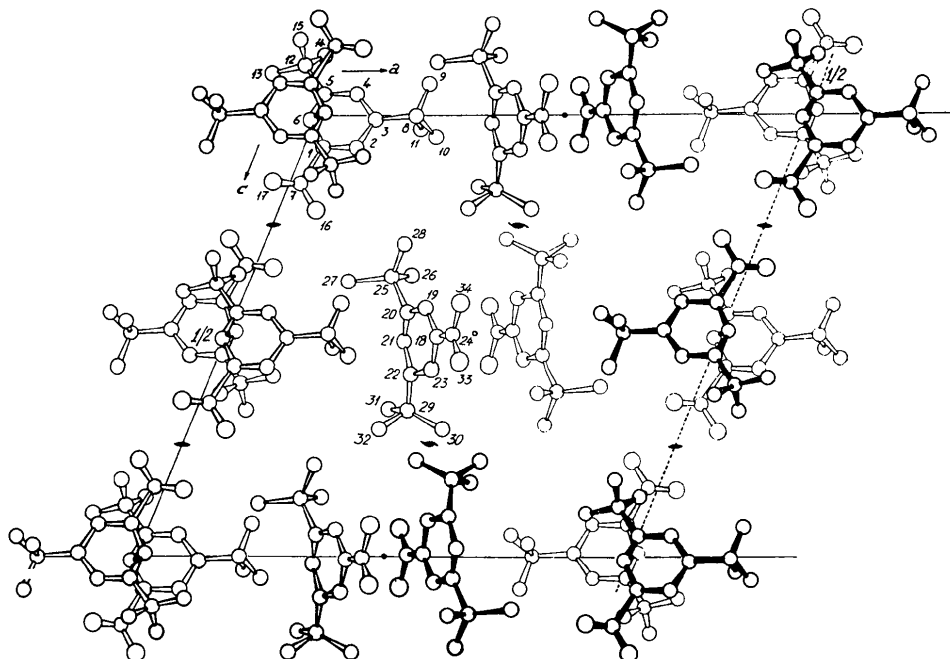


Fig. 1. Projection of the crystal structure along the  $b$  axis and the atom-numbering scheme.

Table 4 (cont.)

Molecule *B*

	<i>x</i>	<i>y</i>	<i>z</i>
H(19)	184 (1)	404 (4)	388 (2)
H(21)	167 (1)	81 (4)	528 (2)
H(23)	239 (2)	405 (7)	631 (4)
H(261)	165 (1)	-58 (5)	418 (3)
H(262)	187 (1)	18 (5)	357 (3)
H(263)	143 (2)	-46 (7)	319 (4)
H(271)	85 (1)	74 (5)	331 (3)
H(272)	94 (1)	218 (5)	386 (3)
H(273)	108 (1)	66 (5)	430 (2)
H(281)	125 (2)	326 (7)	299 (4)
H(282)	116 (1)	187 (5)	252 (3)
H(283)	164 (1)	241 (5)	283 (3)
H(301)	260 (1)	128 (5)	774 (3)
H(302)	268 (1)	95 (5)	695 (3)
H(303)	268 (1)	255 (5)	726 (3)
H(311)	161 (2)	40 (7)	649 (4)
H(312)	200 (1)	-4 (5)	725 (3)
H(313)	209 (1)	-39 (5)	643 (3)
H(321)	202 (1)	368 (5)	725 (3)
H(322)	197 (1)	229 (5)	774 (3)
H(323)	164 (2)	276 (7)	688 (4)
H(34)	233 (1)	697 (5)	433 (3)

## Discussion

A projection of the molecules of DTBB along the *b* axis showing the numbering of the atoms is given in Fig. 1. The two independent molecules are labelled *A* and *B*. Bond distances and angles involving the C atoms are shown in Fig. 2.

From Table 5 and Fig. 2 it is evident that the strain existing in the molecule has not affected the planarity of the benzene ring or the bond lengths, but it is expressed in the bond angles. In each ring, the smaller values of the valence angles (117°) correspond to those that have their vertices attached to the *t*-butyl groups; these values are similar to those found in analogous compounds bearing alkyl substituents (Domenicano, Vaciago & Coulson, 1975; Florencio & Smith, 1972). The distances of type C(3)-C(tb) which average 1.534 Å appear to be slightly stretched. A stretching of 0.029 Å was estimated by comparing the C-C(tb) bond lengths with the value of 1.505 Å (s.d. 0.003 Å) found in isobutylene (Bartell & Bonham, 1960). The C-H distances in the benzene ring average 0.95 Å in molecule *A* and 0.99 Å in molecule *B*.

Table 5. Deviations of atoms from least-squares planes (Å), torsion and dihedral angles (°)

Plane *A* through atoms C(1), C(2), C(3), C(4), C(5), C(6)

$$-0.2038X + 0.9151Y - 0.3479Z = 2.3764$$

C(1)	0.0013	C(7)	-0.0538	C(13)	-0.5488
C(2)	-0.0146	C(8)	0.0019	C(14)	-1.1765
C(3)	0.0110	C(9)	0.2820	C(15)	1.2074
C(4)	0.0054	C(10)	-1.3387	O(16)	-0.3878
C(5)	-0.0184	C(11)	1.1398	O(17)	0.1962
C(6)	0.0153	C(12)	-0.0890		

Plane *B* through atoms C(18), C(19), C(20), C(21), C(22), C(23)

$$0.8835X - 0.4637Y - 0.0677Z = 1.5360$$

C(18)	-0.0003	C(24)	-0.0596	C(30)	1.1187
C(19)	0.0050	C(25)	-0.0699	C(31)	0.0593
C(20)	-0.0065	C(26)	1.1682	C(32)	-1.3791
C(21)	0.0034	C(27)	-1.3182	O(33)	-0.2353
C(22)	0.0013	C(28)	0.01268	O(34)	0.0876
C(23)	-0.0030	C(29)	-0.0452		

Torsion angles

$$C(1)-C(7)=15.576 \quad C(18)-C(24)=8.611$$

$$\text{Angle between plane } A \text{ and plane } B = 54.494.$$

The bond lengths  $C_{\text{arom}}-C_{\text{carb}}$  are 1.488 and 1.480 Å in molecules *A* and *B* which indicate an  $sp^2$  hybridization state. Similar values have been found in several substituted derivatives of benzoic acid such as 3,4,5-trimethylbenzoic acid (Cano, Martínez-Carrera & García-Blanco, 1970), and 3,5-dimethyl-4-nitrobenzoic acid (Apreada, Smith-Verdier & García-Blanco, 1973).

In the carboxyl group O(16)-C(7)-O(17), the values found for the bond length C(7)-O(16) (1.263 Å) and for the bond angle C(1)-C(7)-O(16) (116.8°), show that the H atom is attached to the O(16) atom. In the carboxyl group O(34)-C(24)-O(33) the distance C(24)-O(33) (1.236 Å) and the bond angle C(8)-C(24)-O(34)

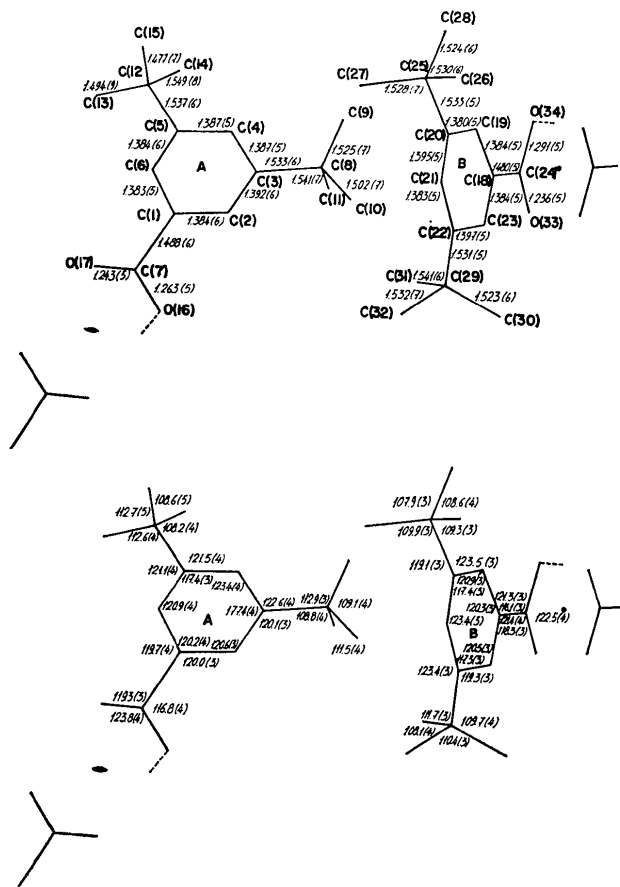


Fig. 2. Bond lengths (Å), valence angles (°), with their estimated standard deviations in parentheses. Angles not shown on the diagram are C(3)-C(8)-C(10)=108.9 (4), C(9)-C(8)-C(11)=105.6 (4), C(5)-C(12)-C(15)=109.8 (4), C(13)-C(12)-C(14)=104.7 (5), C(20)-C(25)-C(28)=112.2 (3), C(26)-C(25)-C(27)=108.9 (4), C(22)-C(29)-C(32)=108.9 (4), C(30)-C(29)-C(31)=110.4 (3)°.

(116.1°) show that the H atom is bonded to the O(34) atom. This is also in agreement with the positions found for these H atoms in the difference synthesis. A feature which is common in most carboxyl derivatives is the dimerization by double hydrogen bonds between two carboxyl groups around the symmetry centres. In the present structure also this dimerization exists but the hydrogen bonds occur about a twofold axis ( $0, y, \frac{1}{2}$ ) in molecule *A*, and around a centre of symmetry ( $\frac{1}{2}, \frac{1}{2}, 0$ ) in molecule *B*. In both cases the two carboxyl groups form the well-known eight-membered ring, but in molecule *A* this ring is distorted. This fact is shown by the separation from the best least-squares plane through the atoms forming the eight-membered ring which vary from 0.086 Å for O(16) to 0.114 Å for O(17), and by the deviation of the H(16) atom which is 0.128 Å. The angle between the planes through the two carboxyl groups in molecule *A* is 160.56°. The distance O(16)···O(17') is 2.636 Å, the bond length C(16)–H(16) is 0.82 Å. The O(16)–H(16)–O(17') bond angle is 165.7° and the C(7)–O(16)–H(16) angle is 123.2°. In molecule *B* the eight-membered ring is practically planar, since the distance between the two planes formed by the C and O atoms of each carboxyl group is 0.069 Å. The distance O(34)···O(33') is 2.647 Å and the C(34)–H(34) bond length is 0.88 Å. The bond angles O(34)–H(34)–O(33') and C(24)–O(34)–H(34) are 174.7 and 115.05° respectively.

The values of torsion angles around the C<sub>arom</sub>–C<sub>carb</sub> are 15.48 and 8.61° in molecules *A* and *B* respectively. It is interesting to note that these twists are greater than that found in 3,4,5-trimethylbenzoic acid (Cano, Martínez-Carrera & García-Blanco, 1970). The small value of the twist in this last acid could be due to the presence of a methyl group in the *para* position. The same feature is present in 2,4,6-trimethylbenzoic acid (Florencio & Smith, 1970) and 2,6-dimethylbenzoic acid (Anca, Martínez-Carrera & García-Blanco, 1967).

In the *t*-butyl groups the C–CH<sub>3</sub> distances vary from 1.447 to 1.549 Å in molecule *A* and from 1.523 to 1.541 Å in molecule *B*. Their mean values, 1.505 and 1.530 Å, respectively, are shorter than the corresponding averages in DTBQ (1.541 Å) (Visser & Vos, 1971*a*) and DTBI (1.540 Å) (Visser & Vos, 1971*b*), but they are very similar to the values found by McKenzie (1975) in the dimer 2,5-di-*t*-butyl-3,6-dineopentylthieno[3,2-*b*]thiophene, and by Bear, Macdonald & Trotter (1973) in 2,4,6-trimethyl-3-pivaloylbenzoic acid.

In molecule *A*, non-bonded contacts among H atoms bonded to C(2), C(4), C(6) and those belonging to the *t*-butyl groups are in agreement with the accepted value for van der Waals distances (2.40 Å). In molecule *B*, some of the corresponding distances among the H atoms attached to the C(19), C(21) and C(23) atoms and those of the *t*-butyl group are appreciably shorter than the van der Waals distances (Table 6).

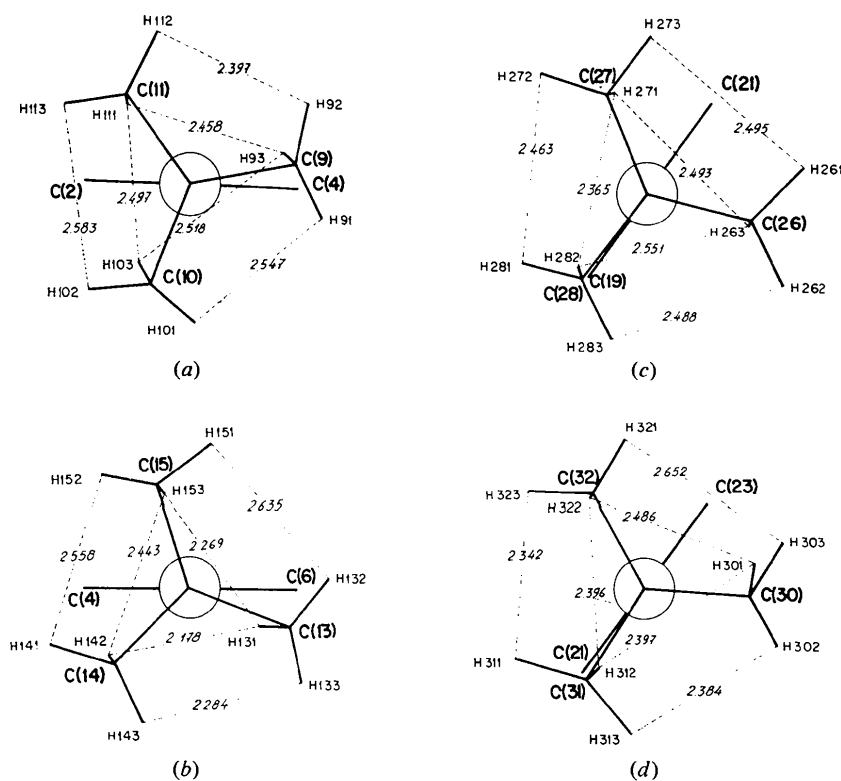


Fig. 3. Projection of *t*-butyl groups. (a) Molecule *A*, along C(3)–C(8). (b) Molecule *A*, along C(5)–C(12). (c) Molecule *B*, along C(20)–C(25). (d) Molecule *B*, along C(22)–C(29).

Table 6. *Some intramolecular non-bonded contacts* (Å)

Standard deviations, given in parentheses, refer to the least significant digits.

Molecule A		Molecule B	
C(3)····C(9)	2·549 (8)	C(20)····C(26)	2·498 (6)
C(3)····C(10)	2·470 (7)	C(20)····C(27)	2·506 (6)
C(3)····C(11)	2·500 (7)	C(20)····C(28)	2·493 (6)
C(5)····C(13)	2·521 (8)	C(22)····C(30)	2·497 (7)
C(5)····C(14)	2·500 (7)	C(22)····C(31)	2·543 (6)
C(5)····C(15)	2·466 (8)	C(22)····C(32)	2·493 (7)
C(2)····C(10)	3·103 (8)	C(19)····C(28)	2·881 (7)
C(2)····C(11)	2·983 (8)	C(21)····C(26)	3·048 (6)
C(4)····C(9)	2·895 (8)	C(21)····C(27)	3·025 (8)
C(4)····C(15)	3·156 (9)	C(21)····C(31)	2·879 (6)
C(4)····C(14)	2·947 (7)	C(23)····C(30)	3·010 (6)
C(6)····C(13)	2·861 (8)	C(23)····C(32)	3·067 (7)
O(16)····C(1)	2·346 (5)	O(33)····C(18)	2·372 (4)
O(16)····C(2)	2·766 (5)	O(33)····C(23)	2·789 (4)
O(16)····O(17)	2·211 (5)	O(33)····O(34)	2·216 (4)
O(17)····C(1)	2·360 (6)	O(34)····C(18)	2·354 (5)
O(17)····C(6)	2·800 (5)	O(34)····C(19)	2·774 (5)
O(16)····H(2)	2·45 (5)	O(33)····H(23)	2·54 (1)
O(17)····H(6)	2·48 (4)	O(34)····H(19)	2·43 (4)
H(2)····H(102)	2·51 (4)	H(19)····H(281)	2·30 (4)
H(2)····H(113)	2·18 (4)	H(19)····H(283)	2·34 (6)
H(4)····H(91)	2·18 (4)	H(21)····H(261)	2·36 (6)
H(4)····H(92)	2·47 (4)	H(21)····H(273)	2·26 (5)
H(4)····H(152)	2·60 (3)	H(21)····H(311)	2·29 (4)
H(6)····H(132)	2·50 (4)	H(21)····H(313)	2·39 (5)
H(6)····H(133)	2·60 (4)	H(23)····H(321)	2·53 (6)
		H(23)····H(303)	2·20 (5)

The conformation of the t-butyl groups is shown in Fig. 3 where they have been projected along C(3)–C(8) and C(5)–C(12) in molecule *A*, and along C(20)–C(25) and C(22)–C(29) in molecule *B*. The three methyl groups of each group are twisted away from the perfectly staggered position. This twisting phenomenon reveals itself in high anisotropic thermal parameters for the atoms concerned (Table 3). The C(tb)–H bond lengths range from 0·88 and 1·10 Å in molecule *A* and from 0·94 to 1·14 Å in molecule *B*.

The packing of the molecules is shown in the projection of Fig. 1. The angle formed by the planes through

the benzene rings of the molecules *A* and *B* is 54·49°. There are no intermolecular contacts shorter than the sums of van der Waals radii.

The authors are grateful to the Centro de Proceso de Datos del Ministerio de Educación y Ciencia, Madrid, Spain, for providing facilities for the use of the 1108 Univac Computer.

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