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The Crystal and Molecular Structure of 3-Methoxypentafluorotropone at −50°C

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3-Methoxypentafluorotropone is monoclinic, space group $P2_1/c$, with $a = 8.61$ (1), $b = 16.32$ (1), $c = 6.07$ (1) Å, $\beta = 100.80$ (5)°, $Z = 4$. The structure was determined by direct methods from X-ray counter data measured at −50°C and refined by least-squares calculations to R 6.06% for 568 observed amplitudes. Estimated standard deviations average 0.01 Å for bond lengths and 0.8° for bond angles. The atoms of the ring deviate slightly from coplanarity such that the ring has a shallow boat-like conformation. The endocyclic bonds show marked alternation in length and it is concluded that the π-electron system is only partially delocalized.

Introduction

Hexafluorotropone (Dodsworth, Jenkins, Stephens & Tatlow, 1972) reacts readily with aqueous NaOH to give a mixture of hydroxypentafluorotropones. The major isomer on treatment with diazomethane formed the corresponding methoxypentafluorotropone. Mass spectrum, ^{19}F and ^{1}H NMR measurements indicated that this was most probably the 3-methoxy isomer (Allen, Jenkins, Stephens & Tatlow, 1974). The present analysis has been undertaken in order to confirm this structural assignment and, in addition, to provide further geometric data relevant to the understanding of the nature of the bonding in the cycloheptatrienone system. Recent structural work on tropones includes X-ray analyses of 2-chlorotropone (Watkin & Hamor, 1971), 3-azidotropone (Cruickshank, Filippini & Mills, 1972), tropone (Barrow, Mills & Filippini, 1973) and hexafluorotropone (Guy, Hamor & Jenkins, 1975).

Experimental

The crystals used in the analysis were grown by sublimation. Initial cell dimensions were obtained from oscillation and Weissenberg photographs. Inspection of the photographs indicated a rapid fall-off in reflexion intensities with increasing angle, and it was therefore decided to collect the intensities at low temperature.

For this purpose a low-temperature device built by Dr A. J. Edwards of this Department was used in conjunction with a Stoe two-circle computer-controlled diffractometer, and unit-cell dimensions and intensities were measured with graphite-monochromated Mo $K\alpha$ radiation at −50°C, the lowest temperature accessible with the apparatus. Crystal data are given in Table 1. The crystal, $0.2 \times 0.2 \times 0.5$ mm, was mounted inside a Pantak capillary tube and set about the direction of elongation (c). 1756 reflexions were scanned within the range $0.1 < \sin \theta/\lambda < 0.59$, of which 568 having $I > 2.5\sigma(I)$ were considered to be observed. The ω scan technique was used; for the zero layer 140 counts of 1s at intervals of 0.01° were taken for each reflexion, backgrounds being measured for 30s at each end of the scan; for layers 1 and 2 the range was increased to 150 counts at the same intervals. For reflexions on layers 3–6 a variable scan technique was used in order to increase the scan range at low values of the azimuth angle and high values of the equi-inclination angle. Intensity measurements were standardized by applying interlayer scale factors obtained by measuring three of the stronger reflexions from each layer at the end of data collection and comparing with the values found during the systematic data collection process. In the conversion of intensities to structure amplitudes, the polarization factor for monochromated radiation was used, but absorption corrections were not applied.

Table 1. *Crystal data*

Formula: $C_8H_3F_5O_2$, M.W. 226·1
Crystal system   Monoclinic
Systematic absences $h0l$ when l is odd
                    $0k0$ when k is odd
Space group $P2_1/c$ (C_{2h}^5)

a	8·68*	8·61 (1) Å
b	16·41*	16·32 (1)
c	6·11*	6·07 (1)
β	101·97*	100·80 (5)°

Unit-cell volume   837·8 Å³
Z 4
Calculated density   1·792 g cm⁻³
μ(Mo $K\alpha$, $\lambda = 0·71069$ Å)   2·2   cm⁻¹

* Room-temperature measurements.

Structure determination

The structure was solved by direct methods (Germain & Woolfson, 1968) and tangent refinement of phases with the automatic phasing program *TANFIZ* (Kennard, Isaacs, Motherwell, Coppola, Wampler, Larson & Watson, 1971). Phases (0° or 180°) were assigned to 147 of the 151 reflexions with $E > 1·40$, and from the E map calculated with these phases, the seven-membered ring and the seven atoms bonded directly to the ring atoms were located. At this stage R was 47·5%. After two cycles of isotropic, full-matrix least-squares refinement with all the atoms attached to the ring treated as F atoms, a difference synthesis gave the position of the methyl C atom. Three more cycles of isotropic refinement reduced R to 17·5%. Consideration of the bond lengths clearly indicated the identity of the carbonyl O atom. The atoms were then allowed to vibrate anisotropically. After further refinement another difference synthesis was computed from which the three H atoms were located. These were included in the calculations but not refined. Refinement was continued until all calculated shifts were $<0·1\sigma$ and

R 6·06% for the 568 observed amplitudes.* Final atomic parameters and estimated standard deviations are listed in Table 2.

The weighting scheme was $w = 1/[\sigma^2(F) + 0·00015F^2]$ where $\sigma(F)$ is the standard deviation in the observed $|F(hkl)|$ values derived from counting statistics.

Computations were carried out on the Birmingham University 1906A computer and also on the IBM 360/44 of the Department of Physics (phase determination and rigid-body thermal motion analysis). Final least-squares and molecular-geometry calculations were performed with the *SHELX* system of programs (Sheldrick, 1975).

Results and discussion

Bond lengths and angles are shown in Fig. 1. Estimated standard deviations average 0·01 Å for lengths and 0·8° for angles. The results of analysing the thermal parameters (U_{ij}) of all the atoms of the molecule except those of the methyl group in terms of rigid-body motion (Cruickshank, 1956; Schomaker & Trueblood, 1968) are given in Table 3. Bond lengths corrected for this effect are uniformly 0·008–0·010 Å greater than the uncorrected values. The r.m.s. deviation between observed and calculated U_{ij} values is 0·007 Å², somewhat greater than the estimated standard deviations in the observed thermal parameters.

C–C bond lengths alternate in length in the sense predicted by the classical valence bond structure (I). As in other tropones, the formal single bonds fall into two categories, C(1)–C(2) and C(1)–C(7), each rather longer than C(3)–C(4) and C(5)–C(6). The mean lengths are 1·439 (uncorrected), 1·449 Å (corrected) for the

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Table 2. *Fractional atomic coordinates* ($\times 10^4$) *and anisotropic thermal parameters* ($Å^2 \times 10^4$)

	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	7852 (11)	1662 (5)	−3057 (16)	762 (71)	328 (51)	561 (61)	−48 (47)	96 (53)	−95 (44)
C(2)	9179 (10)	1362 (5)	−1447 (16)	522 (54)	486 (57)	719 (65)	−32 (49)	303 (50)	−50 (50)
C(3)	9312 (10)	964 (5)	522 (15)	543 (58)	324 (52)	627 (62)	28 (43)	144 (52)	−120 (45)
C(4)	8057 (12)	728 (5)	1549 (12)	828 (76)	299 (49)	383 (55)	108 (48)	48 (50)	73 (40)
C(5)	6489 (12)	840 (5)	903 (15)	711 (74)	496 (57)	666 (69)	−165 (54)	318 (58)	−83 (49)
C(6)	5684 (10)	1250 (5)	−1019 (16)	367 (52)	559 (64)	734 (72)	−83 (49)	58 (49)	−107 (51)
C(7)	6262 (10)	1589 (5)	−2680 (15)	402 (56)	503 (58)	612 (62)	−61 (45)	−93 (46)	−43 (46)
O(1)	8100 (7)	1996 (4)	−4783 (10)	1014 (51)	600 (41)	557 (41)	44 (37)	169 (36)	94 (34)
F(2)	10561 (5)	1517 (3)	−2091 (8)	670 (37)	787 (41)	934 (40)	−24 (30)	299 (31)	−83 (29)
O(3)	10774 (7)	718 (4)	1613 (11)	635 (42)	557 (41)	1031 (49)	155 (36)	−200 (35)	−124 (37)
F(4)	8491 (7)	294 (3)	3458 (8)	1234 (50)	508 (33)	684 (36)	85 (32)	71 (32)	154 (28)
F(5)	5538 (6)	539 (3)	2249 (9)	952 (43)	758 (42)	978 (40)	−236 (34)	450 (34)	−40 (31)
F(6)	4117 (6)	1278 (3)	−1158 (10)	507 (34)	898 (45)	1300 (53)	−77 (33)	63 (29)	−113 (35)
F(7)	5202 (6)	1949 (3)	−4290 (9)	732 (36)	782 (40)	964 (42)	46 (32)	−307 (30)	82 (31)
C(8)	11717 (10)	1343 (6)	2890 (15)	596 (57)	846 (76)	691 (64)	101 (59)	−61 (47)	−60 (55)
H¹[C(8)]	12602	1066	3665						
H²[C(8)]	11571	1861	2281						
H³[C(8)]	11156	1458	4373						

Table 3. *Results of rigid-body motion analysis*

The eigenvectors of the libration tensor **L** and translation tensor **T** are referred, in terms of the corresponding direction cosines, to the orthogonal molecular system defined by the eigenvectors of the molecular tensor of inertia **I**. The calculation is referred to the origin which symmetrizes the screw tensor and reduces the trace of **T**. The components of the origin shift from the molecular centroid ($x = -0.7534$, $y = 0.1175$, $z = -0.0657$) are 0.51, 0.09, 0.25 Å.

	Eigenvalues	Eigenvectors		
I	648 (at. wt. Å²)	0.4543	−0.4391	0.7751
	654	0.8908	0.2321	−0.3907
	1302	−0.0083	0.8679	0.4966
L	89 × 10⁻⁴ rad²	−0.2078	0.2305	0.9506
	58	0.9387	−0.2264	0.2601
	47	0.2752	0.9464	−0.1693
T	535 × 10⁻⁴ Å²	0.5628	−0.8046	0.1893
	437	0.8018	0.5871	0.1119
	267	−0.2011	0.0888	0.9755

r.m.s. discrepancy, $\langle \Delta U^2 \rangle^{1/2} = 0.0071$ Å².

former, and 1.405 (uncorrected), 1.415 Å (corrected) for the latter. The formal double bonds average 1.339 (uncorrected), 1.349 Å (corrected). Standard values for the length of a C–C double bond and $C(sp^2)$–$C(sp^2)$ single bond have been quoted as 1.335 Å (Sutton, 1965) and 1.48–1.50 Å (Cruickshank, 1962; Hoard, 1975) respectively. Bond angles within the ring, however, average 128.6° and the hybridization ratios (Coulson, 1948) correspond to $sp^{1.6}$ rather than sp^2-hybridization for these bonds. The corresponding decrease in the covalent radius calculated by the formula given by Coulson (1948) is 0.008 Å, so that the standard lengths quoted above should be decreased by ca 0.016 Å for comparison purposes in this case.

The measured double bonds are therefore slightly longer than a pure double bond and the single bonds are shorter than a pure single bond, indicating some degree of π-electron delocalization as depicted by (II). Consistent with this picture, the C–O(carbonyl) bond is slightly longer than the accepted value (1.215 Å) (Sutton, 1965).*

The actual pattern of bond-angle variation within the ring is similar to that found in other tropones, with the smallest angle at the carbonyl C(1), and the largest

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* Angles at the carbonyl C atom are all close to 120°, so the standard bond-length values for sp^2-hybridization apply.

Table 4. *Mean-plane calculations*

(*a*) Deviations (Å) of atoms from least-squares planes. Distances marked with asterisks refer to atoms defining the plane. In the equations of the planes, x, y and z are fractional coordinates relative to the cell axes.

	(i)	(ii)	(iii)	(iv)	(v)
C(1)	−0.020*	0.000*	−0.022		
C(2)	0.006*	0.000*	0.001*		
C(3)	0.013*		−0.001*	−0.002*	0.000*
C(4)	−0.006*		−0.028	0.004*	
C(5)	−0.015*		−0.038	−0.004*	
C(6)	0.017*		0.001*	0.002*	
C(7)	0.007*	0.000*	−0.001*		
O(1)	−0.053	0.014			
F(2)	0.021	0.047	0.022		
O(3)	−0.036		−0.051	−0.043	0.000*
F(4)	−0.072			−0.032	
F(5)	−0.039			0.001	
F(6)	0.036		0.017	0.029	
F(7)	0.062	0.086	0.059		
C(8)	1.214				0.000*

Equations of planes

(i)	$0.129x - 14.245y - 2.925z = -1.522$
(ii)	$0.021x - 14.521y - 2.724z = -1.585$
(iii)	$0.104x - 14.291y - 2.891z = -1.536$
(iv)	$0.196x - 14.061y - 3.049z = -1.530$
(v)	$4.699x + 3.947y - 5.404z = -0.225$

(*b*) Interplanar angles (°)

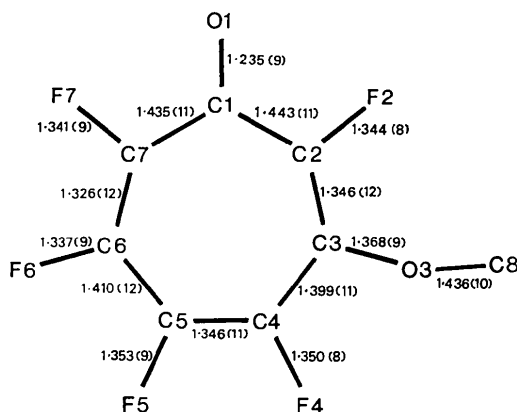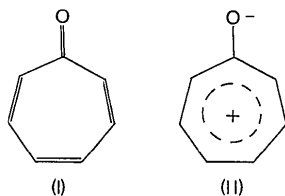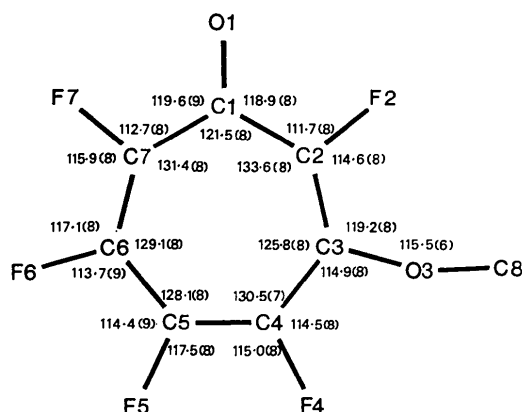| (i)–(v)	79.1	(iii)–(iv)	1.7
(ii)–(iii)	1.8		

(I)   (II)

Fig. 1. Bond lengths (Å) and angles (°) with estimated standard deviations in parentheses. Bond lengths corrected for the effect of thermal libration are 0.008–0.010 Å greater than the uncorrected values.

at the adjacent C(2) and C(7). Some distortion is introduced by the 3-methoxy substituent, the angle at C(3) being smaller than the angles at C(4), C(5) and C(6). In hexafluorotropone (Guy, Hamor & Jenkins, 1975) these four angles are all within the range 128·3–129·3°.

The results of mean-plane calculations are given in Table 4. The seven-membered ring is very nearly planar. Nevertheless the small (maximum 0·02 Å) deviations from planarity are statistically significant ($\chi^2 = 18\cdot1$ for four degrees of freedom). The shape of the ring can best be described in terms of three planes defined by C(1), C(2), C(7); C(2), C(3), C(6), C(7); and C(3), C(4), C(5), C(6). These atomic groupings are accurately planar and form a shallow boat-like conformation with interplanar angles of 1·8 and 1·7°. Tropone itself (Barrow, Mills & Filippini, 1973) and also the tropone rings of various benzotropones (Ibata, Shimanouchi, Sasada & Hata, 1975) adopt similar conformations, but generally the interplanar angles are larger. On the other hand, the ring systems of 3-azidotropone (Cruickshank, Filippini & Mills, 1972) and hexafluorotropone are planar to well within the limits of experimental accuracy.

Of considerable interest is the change, if any, in the degree of π-electron delocalization caused by the substitution of H by highly electronegative F atoms. X-ray analyses of 2-chlorotropone (Watkin & Hamor, 1971), 3-azidotropone (Cruickshank et al., 1972) and tropone (Barrow et al., 1973), dipole moment and NMR measurements (Bertelli & Andrews, 1969; Bertelli, Andrews & Crews, 1969), resonance energy data (Lewis & Peters, 1975) and theoretical calculations (Dewar & Trinajstic, 1970) all indicate that the tropone system possesses only limited aromatic character. In hexafluorotropone the pattern of bond-length alternation in the carbocyclic ring is similar to that in tropone (Guy et al., 1975).

Julg & Francois (1967) have proposed a quantitative measure of π-electron delocalization based on meas-ured bond lengths, in terms of an 'aromaticity constant', A, defined as

$$A = 1 - K \sum_{i=1}^{n} (l_i - \bar{l})^2 / n\bar{l}^2,$$

where $\bar{l}$ is the mean of the n bond lengths in the ring. The constant K was assigned the value 225, so as to result in an aromaticity of zero for a system of bonds of length alternately 1·33 and 1·52 Å. Application of this formula to a number of tropones for which bond-length measurements are available leads to the values listed in Table 5. In these calculations the constant K was taken to be 377, so as to give an aromaticity of zero for bonds of length 1·335 and 1·48 Å, the probable values for pure double and pure single bond lengths in a system of alternate double and single bonds. The shortening of the C–C bonds in tropones due to hybridization effects would not affect the results, as long as all bonds are affected equally. This is not quite the case since there is some variation in bond angle, but any errors due to this would be small compared with the uncertainties in the measured bond lengths. Corrections to bond lengths for the effects of rigid-body thermal motion are very nearly the same for all the bonds in any one structure, and it does not, therefore, matter whether the corrected or uncorrected bond lengths are used.*

All the calculated values of the aromaticity constant are within the range 0·53–0·65 and there is no obvious trend which could be attributed to F substitution. Also listed in Table 5 are the aromaticity constants for the five-bond system C(2)$\cdots$C(7) [i.e. excluding bonds C(1)–C(2) and C(1)–C(7)]. Here there is some, but by no means conclusive, evidence that over these five bonds there is a slightly greater degree of electron delocalization in the fluorinated tropones.

The C–O(carbonyl) lengths (Table 5) appear to be slightly shorter in the fluorinated tropones. This would imply that this bond is involved to a lesser extent in the electron delocalization, possibly due to the F substituents competing with the carbonyl O atom for the negative charge [cf. (II)].

The C(3)–O(3) length is 1·368 Å. The C(3), O(3), C(8) plane is almost perpendicular to the mean plane of the ring (interplanar angle 79°), so that one might have expected that O(3) could not be involved in the electron delocalization of the tropone system to any appreciable degree. The evidence regarding the length of a C(sp^2)–O pure single bond is not, however, at all clear. Structure analyses of a number of molecules containing C(aromatic)–O–Me groupings have shown that the methyl group generally lies close to the aromatic ring plane with C(aromatic)–O lengths 1·36–1·39, mean ca 1·375 Å, and bond angles at the O atom of 116–119°. On the

Table 5. Comparative data for tropones

	(1)	(2) a	(2) b	Mean	(3)	(4)
Aromaticity constant*	0·53	0·65	0·58	0·61	0·55	0·63
Aromaticity constant* excluding bonds C(1)–C(2) and C(1)–C(7)	0·62	0·79	0·61	0·70	0·77	0·78
C–O(carbonyl) length (Å)	1·234	1·259	1·257	1·258	1·226	1·235
Mean C–C length (Å)	1·395	1·403	1·398	1·401	1·382	1·386

* See text. (1) 3-Azidotropone (Cruickshank et al., 1972). (2) Tropone, two independent molecules in the unit cell (Barrow et al., 1973). (3) Hexafluorotropone (Guy et al., 1975). (4) 3-Methoxypentafluorotropone (present work).

* In Table 5 and in the following discussion the uncorrected values for bond lengths are used since the published lengths for tropone, 3-azidotropone and other pertinent molecules have not been corrected for the effect of thermal motion.

basis of similar results obtained for β-methylphenyl-choline ether bromide, Hamodrakas, Geddes & Sheldrick (1974) have concluded that the C(aromatic)–O bond possesses partial double-bond character. In reserpine (Karle & Karle, 1968) and mescaline hydrobromide (Ernst & Cagle, 1973), which each contain three adjacent methoxy groups substituted into a phenyl ring, the two outer groups are approximately coplanar with the ring while the middle group is nearly perpendicular to the plane of the ring. The middle C(aromatic)–O bonds are not, however, longer than the outer ones; in fact the mean of the four outer lengths is 1·367 Å, rather longer than the mean of the two middle ones (1·354 Å). The bond angle at the O atom is, however, smaller in the perpendicularly oriented groups. Longer C(aromatic)–O lengths of 1·40–1·41 Å have been found in a number of structures containing C(aromatic)–O–S and C(aromatic)–O–P groupings steeply inclined to the plane of the phenyl ring (Fries & Sundaralingam, 1971; Domenicano, Vaciago & Coulson, 1975a; Grand & Robert, 1975). Also, if it is assumed that the radius of a C atom decreases by ca 0·02–0·03 Å in changing its hybridization state from sp^3 to sp^2 (Cruickshank, 1962; Coulson, 1948) then the length of the C(sp^2)–O single bond would be 0·02–0·03 Å shorter than the C(sp^3)–O single bond length of 1·43 Å quoted by Sutton (1965), i.e. 1·40–1·41 Å.*

It would appear, therefore, that the C(3)–O(3) bond in the present structure is involved in the π-electron delocalization, at least to a small extent. The relatively

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* Consideration of the hybridization effects associated with the distortion of bond angles at carbon from the trigonal value discussed earlier would lead to a slightly greater length for the C(tropone)–O pure single bond.
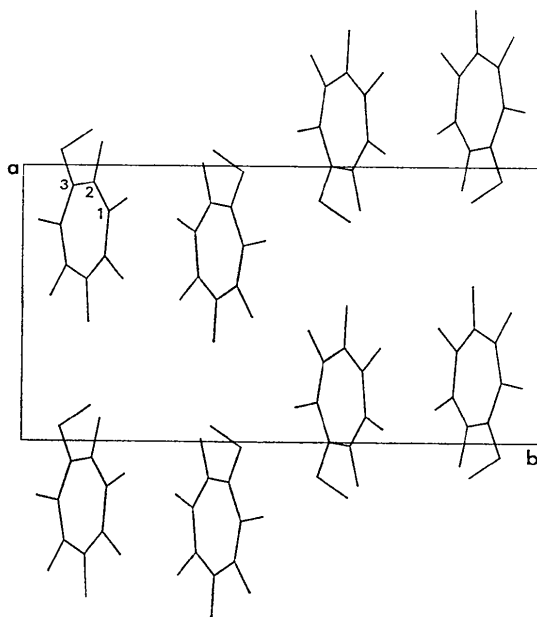

Fig. 2. The crystal structure projected along c*.

small endocyclic angle at C(3) would then be accounted for by a combination of the effects due to the partial double-bond character of the C(3)–O(3) bond (Bent, 1961), and the more electronegative F substituents at the adjacent C atoms (Domenicano, Vaciago & Coulson, 1975b).

The C–F bonds have lengths 1·337–1·353 (uncorrected), 1·347–1·363 Å (corrected). The mean length is 1·345 (uncorrected), 1·355 Å (corrected); ca 0·01 Å longer than in hexafluorotropone (Guy et al., 1975).

In discussing the structure of hexafluorotropone it had been noted that there was an overall shortening of the C–C bonds relative to hydrocarbon systems. A similar shortening of the C–C double bond in fluorinated ethylenes has been discussed by Laurie (1961) and Laurie & Pence (1963), and Meresse, Courseille & Chanh (1974) have observed bond shortenings in the crystal structure of tetrafluoro-p-benzoquinone. Bent (1961), Carter, McPhail & Sim (1966) and Domenicano, Vaciago & Coulson (1975b) have explained such bond shortenings in terms of changes in the hybridization state of the C atoms induced by highly electronegative substituents. Ab initio calculations on fluoroethylenes (Pappas, 1974) also predict bond shortening. In 3-methoxypentafluorotropone the mean C–C length is 1·386 Å, only very slightly greater than the mean length in hexafluorotropone but less than the means for tropone and 3-azidotropone (Table 5).

Table 6. *The shorter intermolecular contacts*

$H^3[C(8)]\cdots F(2^i)$	2·30		$F(5)\cdots F(7^i)$	3·17
$H^1[C(8)]\cdots F(7^{ii})$	2·76		$F(1)\cdots C(6^{iii})$	3·17
$F(2)\cdots H^2[C(8)^{iii}]$	2·83		$O(3)\cdots O(3^v)$	3·18
$H^3[C(8)]\cdots O(1^i)$	2·91		$F(7)\cdots F(6^{iii})$	3·18
$O(1)\cdots C(1^{iii})$	2·94		$F(4)\cdots C(1^i)$	3·19
$F(5)\cdots H^1[C(8)^{iv}]$	2·95		$F(4)\cdots F(2^v)$	3·22
$F(4)\cdots O(1^i)$	3·02		$F(5)\cdots F(5^{vii})$	3·23
$O(3)\cdots C(3^v)$	3·03		$F(5)\cdots C(5^{vii})$	3·24
$F(6)\cdots F(2^{iv})$	3·03		$C(5)\cdots O(1^i)$	3·31
$C(4)\cdots O(1^i)$	3·04		$C(8)\cdots F(7^{ii})$	3·32
$F(4)\cdots F(4^{vi})$	3·06		$O(3)\cdots C(4^v)$	3·32
$F(6)\cdots F(5^{vii})$	3·06		$F(7)\cdots C(7^{iii})$	3·39
$O(1)\cdots C(2^{iii})$	3·07		$O(3)\cdots C(2^v)$	3·40
$O(1)\cdots C(7^{iii})$	3·14		$C(3)\cdots C(3^v)$	3·46
$F(5)\cdots C(6^{vii})$	3·15			

Symmetry code

(i)	$x,$	$y, z+1$		(v)	$2-x, \bar{y},$	$\bar{z}$
(ii)	$x+1,$	$y, z+1$		(vi)	$2-x, \bar{y}, 1-z$	
(iii)	$x, \frac{1}{2}-y, \frac{1}{2}+z$			(vii)	$1-x, \bar{y},$	$\bar{z}$
(iv)	$x-1,$	$y,$	z			

Intermolecular contact distances are listed in Table 6 and the packing arrangement is illustrated in Fig. 2. There are two short intermolecular contact distances; 2·94 Å between carbonyl O(1) of the reference molecule and C(1) of the molecule related to it by the c glide plane, and ca 2·3 Å between F(2) and methyl $H^3[C(8)]$ of molecules separated by the c unit-cell translation. None of the other intermolecular distances is substantially shorter than the sum of the van der Waals radii of the atoms concerned.

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

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Crystals of 3,5-di-t-butylbenzoic acid are monoclinic, space group $C2/c$ with $a = 36\cdot723$ (7), $b = 9\cdot500$ (1), $c = 17\cdot963$ (9) Å, $\beta = 111\cdot01°$ (1) and $Z = 16$. The structure was solved by direct methods and refined by full-matrix least-squares calculations to a final R of $0\cdot071$ 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 axes 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