between 108·4 and 110·1°. In the present molecule, the spread of values is somewhat larger, 106·9 to 114·0°, with the two highest values associated with the two butyl groups, *i.e.* C(1)Si(1)C(11) 113·9 (1) and C(17)-Si(2)C(21) 114·0 (1)°. The nearest intramolecular nonbonded approaches are between substituents on the same Si atom, *e.g.* C(10)···C(11) 3·31, C(5)···C(13) 3·36, C(18)···C(27) 3·36 and C(21)···C(32) 3·40 Å. C atoms of substituents on the different Si atoms are not as closely spaced. The minimum distances are between phenyl groups, C(6)···C(26) at 3·59 and C(12)···C(21) at 3·61 Å. All other C···C distances between moieties on the separate Si atoms are greater than 3·69 Å. The closest approach between different molecules in the cell is C(20)···C(24)(1·5 − x, 0·5 + y, −0·5 − z) at 3·59 Å.

The phenyl groups are distorted from perfect hexagons in the same manner as has been already observed in other Si compounds, *e.g.* in $O[SiPh_3]_2$ and in $[OSiPh_2]_3$ (Tomlins *et al.*, 1985). The interior angle of the four phenyl groups at the C—Si bond has an average value of 116·5 (2)° in the present molecule. The two adjacent interior angles average to 121·7 (2)°, whereas the remaining angles are near 120°.

The *tert*-butyldiphenylsilyl trifluoroacetamide was a gift from Drs Nancy Roth and Anthony Theoharides, Division of Experimental Therapeutics, Walter Reed Army Institute of Research.

The technical assistance of Mr Michael Smyth of Dr Theoharides's mass-spectrometry laboratory is gratefully acknowledged.

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Perhexiline [2-(2,2-Dicyclohexylethyl)piperidine] Maleate

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(*Received* 2 *February* 1985; *accepted* 19 *August* 1985)

Abstract. Perhexiline maleate, $C_{19}H_{36}N^+.C_4H_3O_4^-$, is a vasodilator and calcium-blocking agent. $M_r = 393·6$, $P2_1/a$, $a = 18·354$ (2), $b = 19·659$ (2), $c = 6·247$ (1) Å, $\beta = 90·96$ (1)°, $V = 2253·7$ (4) Å^3, $Z = 4$, $D_x = 1·16$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1·5418$ Å, $\mu = 5·432$ cm^{-1}, $F(000) = 864$, room temperature, final $R = 0·053$ for 3678 observed intensities and 409 parameters. The maleate group is planar with an internal hydrogen bond between the two carboxyl groups. Each of the three six-membered rings of perhexiline maleate is in the chair conformation, but with no apparent overall symmetry to the molecule. The two saturated cyclohexane rings provide a strongly hydrophobic area and the positively charged $-NH_2^+-$ group forms two hydrogen bonds to maleate oxygen atoms 4·49 Å apart. This suggests that similar hydrogen-bond acceptors may occur in the biological perhexiline receptor.

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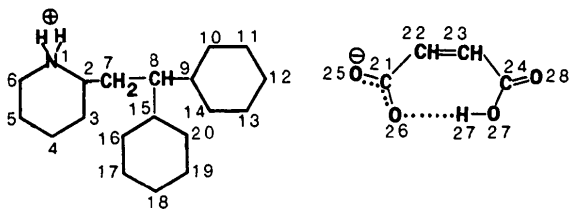
Introduction. Perhexiline maleate (I) is a vasodilator and calcium-blocking agent (*Merck Index*; Windholz, Budavari, Blumetti & Otterbein, 1983). It has been shown to be effective in the treatment of angina pectoris, apparently acting by blocking calcium influx as well as by decreasing myocardial lipid metabolism (White & Lowe, 1982). Although it is reported to be effective even for those patients who do not respond well to treatment with beta blockers, this drug is not in widespread use. This may be due, in part, to the many side effects associated with long-term therapy (Cooper, Evans & Whibley, 1984), including hepatic damage (Forbes, Rake & Taylor, 1980; Pieterse, 1983), peripheral neuropathy (Hauw, Mussini, Boutry, Harpin, Escourolle, Pollet, Albouz & Baumann, 1981) and hypoglycemia (Houdent, Wolf & Corriat, 1977), among others; all of these side effects are reversible when perhexiline administration is ended unless hepatic cirrhosis has developed (Pessayre, Bichara, Feldmann, Degott, Potet & Benhamou, 1979). Since perhexiline is very lipid soluble, renal excretion of the drug can only occur if prior solubilization by hydroxylation occurs; a genetic inability to perform this hydroxylation, presumably owing to lack of sufficient appropriate enzymes, has been reported in persons experiencing serious side effects during therapy (Singlas & Simon, 1981). It has been suggested that the lipophilicity of perhexiline maleate may be a controlling factor in the drug's enhancement of the sensitivity of previously anthracycline-resistant P388 murine leukemia cells to the anthracycline antitumor agents daunorubicin and doxorubicin (Ramu, Fuks, Gatt & Glaubiger, 1984).



(I)

In view of the properties cited above, a crystal structure determination was performed in order to explore further the chemical nature of this biochemically significant compound.

Experimental. Perhexiline maleate recrystallized as needles from ethyl methyl ketone at room temperature. Colorless crystal 0·275 × 0·350 × 0·075 mm was mounted directly onto a glass fiber. Cell parameters obtained by least-squares analysis of diffractometer measurements of 24 centered reflections. Monoclinic space group $P2_1/a$ determined from systematic absences: $0k0$, $k = 2n + 1$, and $h0l$, $h = 2n + 1$. Data collected on a Nicolet $P2_1$ four-circle diffractometer with Cu $K\alpha$ radiation and a highly oriented graphite monochromator. $\theta/2\theta$ scan technique, 4212 unique reflections to 2θ limit of 138·0° (sin $\theta/\lambda = 0{\cdot}61$ Å^{-1}) at variable scan speed of 1·00–29·30° min^{-1}, depending upon intensity. Four check reflections measured every 100 reflections showed no decay in intensity. Scan: background ratio 2·5. 3678 reflections with $I > 2{\cdot}0\sigma(I)$ [$\sigma(I)$ determined from counting statistics] considered observed and included in further calculations. Values of $\sigma(F)$ calculated as $\sigma(F) = (F/2)[\sigma^2(I)/(I)^2 + \delta^2]^{1/2}$, where δ (0·029) is an instrumental uncertainty determined from the variation in the intensity of the check reflections. Max. $h = 22$, max. $k = 23$, max. $l = 7$. Data corrected for Lorentz and polarization factors and put on absolute scale with Wilson plot. One reflection at about $\chi = 90{\cdot}0°$ measured at intervals of 5·0° about diffraction vector ψ and a transmission curve as a function of φ was constructed so that relative absorptions could be applied.

MULTAN (Germain, Main & Woolfson, 1971) employed in direct-method structure solution, using 300 E values greater than 1·8. Solution having highest absolute figure of merit (1·14) and lowest residual (15·6%) gave E map that revealed positions of all non-H atoms. Atomic positions and anisotropic thermal parameters of all non-H atoms refined by full-matrix least-squares computer program, *ICRFMLS* (Carrell, 1975). H atoms from difference map. Inclusion and refinement of H atoms yielded final values $R = 0{\cdot}053$ and $wR = 0{\cdot}066$; av. shift in atomic parameters < 0·22 σ, $w\{|F_o| - |F_c|\}^2$ minimized, $w = 1/\sigma^2(F)$. Atomic scattering factors for non-H atoms from Cromer & Mann (1968), and for H atoms from Stewart, Davidson & Simpson (1965). All computer programs used are part of the *Crystallographic Program Library* written at the Institute for Cancer Research (Carrell & Shieh, undated). Highest peak in final difference map 0·27 e Å^{-3}. The fractional coordinates and averaged isotropic temperature factors of the atoms are given in Table 1.*

Discussion. As shown in Fig. 1, the three six-membered rings of the perhexiline molecule each exist in the chair conformation; these three rings are joined by the $-CH-CH_2-$ group. However, there is no precise symmetry as indicated in this view [approximately down C(8)–H(8)] and by the torsion angles for C(2)–C(7)–C(8)–C(9) and C(2)–C(7)–C(8)–C(15), which are quite different [145·3 (2) and −82·8 (2)° respectively (Table 2)]. The two saturated cyclohexane

* Lists of structure factors, anisotropic thermal parameters, selected torsion angles, distances and angles and the results of least-squares-planes calculations have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42443 (31 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

rings provide a strongly hydrophobic area in this molecule, separate from N(1), which has hydrogen-bonding potential.

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters B_{eq} or B_{iso} (for H atoms), with e.s.d.'s in parentheses*

B_{eq} is defined as $\frac{1}{3}$ the trace of the B_{ij} matrix.

	x	y	z	B_{eq}(Å^2)
N(1)	0·21518 (6)	0·16920 (6)	0·5457 (2)	3·16 (5)
C(2)	0·22297 (8)	0·21345 (7)	0·7414 (2)	3·14 (6)
C(3)	0·25998 (9)	0·17171 (8)	0·9172 (3)	4·03 (7)
C(4)	0·21953 (11)	0·10612 (9)	0·9633 (3)	4·83 (9)
C(5)	0·21178 (12)	0·06420 (8)	0·7600 (3)	5·02 (9)
C(6)	0·17527 (10)	0·10458 (9)	0·5842 (3)	4·33 (8)
C(7)	0·26613 (8)	0·27689 (7)	0·6814 (3)	3·51 (7)
C(8)	0·25419 (8)	0·33667 (7)	0·8356 (2)	3·18 (6)
C(9)	0·32341 (8)	0·38068 (8)	0·8702 (3)	3·45 (7)
C(10)	0·36733 (10)	0·39591 (9)	0·6694 (3)	4·55 (8)
C(11)	0·43445 (11)	0·43966 (11)	0·7223 (4)	5·69 (10)
C(12)	0·48307 (10)	0·40662 (11)	0·8890 (4)	6·07 (11)
C(13)	0·44110 (11)	0·39114 (11)	1·0899 (4)	5·53 (10)
C(14)	0·37321 (10)	0·34854 (9)	1·0408 (3)	4·64 (8)
C(15)	0·18390 (8)	0·37587 (7)	0·7760 (3)	3·41 (7)
C(16)	0·15258 (10)	0·41374 (9)	0·9671 (3)	4·33 (8)
C(17)	0·07903 (10)	0·44678 (11)	0·9136 (3)	5·36 (9)
C(18)	0·08504 (10)	0·49346 (10)	0·7224 (3)	5·37 (9)
C(19)	0·11571 (11)	0·45635 (11)	0·5310 (3)	5·32 (9)
C(20)	0·18906 (10)	0·42450 (10)	0·5864 (3)	4·53 (8)
C(21)	0·07349 (9)	0·25294 (8)	0·3141 (3)	4·11 (7)
C(22)	0·02110 (9)	0·28725 (9)	0·1662 (3)	4·28 (8)
C(23)	−0·04299 (9)	0·31614 (9)	0·2052 (3)	4·44 (8)
C(24)	−0·08495 (9)	0·32131 (9)	0·4030 (3)	4·63 (8)
O(25)	0·13176 (7)	0·23405 (7)	0·2386 (2)	5·64 (7)
O(26)	0·05760 (7)	0·24433 (7)	0·5102 (2)	5·60 (7)
O(27)	−0·05823 (7)	0·29747 (8)	0·5805 (2)	6·33 (8)
O(28)	−0·14523 (7)	0·34856 (7)	0·3920 (3)	6·26 (8)
H(1)	0·260 (1)	0·162 (1)	0·496 (3)	4·7 (4)
H(1′)	0·194 (1)	0·192 (1)	0·443 (3)	4·5 (4)
H(2)	0·172 (1)	0·222 (1)	0·780 (3)	3·5 (4)
H(3)	0·263 (1)	0·200 (1)	1·047 (3)	4·8 (4)
H(3′)	0·309 (1)	0·163 (1)	0·873 (3)	3·9 (4)
H(4)	0·166 (1)	0·116 (1)	1·022 (4)	6·6 (6)
H(4′)	0·245 (1)	0·082 (1)	1·070 (4)	5·9 (5)
H(5)	0·187 (1)	0·026 (1)	0·798 (4)	7·6 (6)
H(5′)	0·264 (1)	0·051 (1)	0·710 (3)	5·8 (5)
H(6)	0·126 (1)	0·118 (1)	0·622 (3)	5·1 (5)
H(6′)	0·176 (1)	0·085 (1)	0·446 (4)	5·6 (5)
H(7)	0·320 (1)	0·260 (1)	0·678 (3)	4·1 (4)
H(7′)	0·250 (1)	0·290 (1)	0·542 (3)	4·2 (4)
H(8)	0·245 (1)	0·315 (1)	0·972 (2)	2·7 (3)
H(9)	0·306 (1)	0·426 (1)	0·932 (3)	4·0 (4)
H(10)	0·388 (1)	0·350 (1)	0·607 (3)	4·3 (4)
H(10′)	0·336 (1)	0·418 (1)	0·554 (3)	4·4 (4)
H(11)	0·461 (1)	0·448 (1)	0·595 (4)	8·2 (7)
H(11′)	0·414 (1)	0·482 (1)	0·766 (4)	6·0 (5)
H(12)	0·500 (1)	0·366 (1)	0·839 (4)	5·9 (5)
H(12′)	0·523 (1)	0·433 (1)	0·931 (3)	5·6 (5)
H(13)	0·471 (1)	0·365 (1)	1·196 (4)	6·9 (6)
H(13′)	0·426 (1)	0·434 (1)	1·151 (3)	5·8 (5)
H(14)	0·394 (1)	0·301 (1)	0·991 (3)	5·3 (5)
H(14′)	0·346 (1)	0·339 (1)	1·175 (3)	5·7 (5)
H(15)	0·142 (1)	0·346 (1)	0·729 (3)	4·5 (4)
H(16)	0·151 (1)	0·380 (1)	1·095 (3)	5·1 (5)
H(16′)	0·193 (1)	0·449 (1)	1·003 (3)	5·7 (5)
H(17)	0·043 (1)	0·406 (1)	0·861 (4)	6·5 (6)
H(17′)	0·059 (1)	0·476 (1)	1·047 (4)	6·9 (6)
H(18)	0·038 (1)	0·518 (1)	0·687 (4)	9·1 (7)
H(18′)	0·125 (1)	0·532 (1)	0·772 (4)	6·8 (6)
H(19)	0·076 (1)	0·418 (1)	0·502 (3)	5·9 (5)
H(19′)	0·121 (1)	0·487 (1)	0·411 (4)	7·3 (6)
H(20)	0·207 (1)	0·398 (1)	0·459 (4)	7·2 (6)
H(20′)	0·222 (1)	0·465 (1)	0·632 (3)	5·8 (5)
H(22)	0·036 (1)	0·286 (1)	0·017 (3)	4·8 (4)
H(23)	−0·065 (1)	0·340 (1)	0·093 (3)	5·5 (5)
H(27)	−0·006 (2)	0·270 (1)	0·558 (5)	9·5 (8)

Data on hydrogen bonding are summarized in Table 3. An intramolecular hydrogen bond, illustrated in Fig. 3(*a*), is found in the mono-ionized maleate ion between the two carboxyl groups in which a hydrogen atom is almost equally shared by the two oxygens. The $-NH_2^+-$ group in perhexiline is completely accessible for hydrogen bonding to two maleate ions. These hydrogen atoms on N(1) are the only ones available for intermolecular bonding, thus allowing the formation of a bridge, or chain, of hydrogen bonds {from a maleate C=O oxygen [O(25)] to N(1) to maleate C=O oxygen [O(28)], and so on}, along the *a* axis (Fig. 2). The chains lie at approximately $y = \frac{1}{4}$ and $y = \frac{3}{4}$ in the unit cell leaving a hydrophobic area at the center of the unit cell and resulting in an apparent cleavage plane parallel to the *ac* plane. The needle axis of the crystal is parallel to the *c* axis.

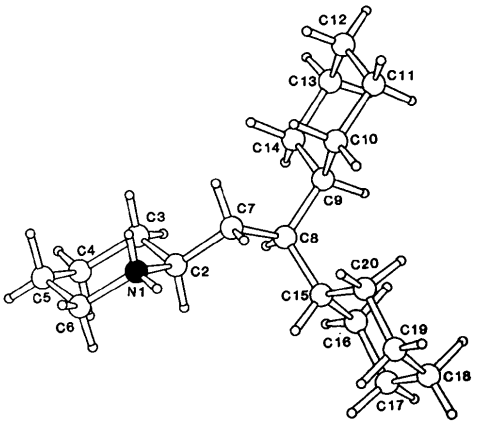


Fig. 1. View of perhexiline showing the chair conformation. In this and all other figures N is black and O is stippled.

Table 2. *Some relevant torsion angles* (°) *and their e.s.d.'s*

N(1)–C(2)–C(7)–C(8)	158·8 (1)
C(2)–C(7)–C(8)–C(9)	145·3 (2)
C(2)–C(7)–C(8)–C(15)	−82·8 (2)
C(21)–C(22)–C(23)–C(24)	−3·1 (4)
O(26)–C(21)–C(22)–C(23)	4·2 (4)
O(27)–C(24)–C(23)–C(22)	3·2 (4)
C(2)–N(1)–O(25)–C(21)	−52·7 (2)
C(2)–N(1)–O(28^{i})–C(24^{i})	−20·9 (3)
C(21)–O(26)–O(27)–C(24)	3·9 (2)

Symmetry code: (i) $\frac{1}{2}+x, \frac{1}{2}-y, z$.

Table 3. *Hydrogen-bond geometry and e.s.d.'s*

X	H	Y	X–H (Å)	$X\cdots Y$ (Å)	H$\cdots Y$ (Å)	X–H$\cdots Y$ (°)
O(27)	H(27)	O(26)	1·11 (3)	2·416 (2)	1·31 (3)	172 (3)
N(1)	H(1′)	O(25)	0·87 (2)	2·748 (2)	1·88 (2)	170 (2)
N(1)	H(1)	O(28^{i})	0·89 (2)	2·773 (2)	1·88 (2)	177 (2)

Symmetry code: (i) $\frac{1}{2}+x, \frac{1}{2}-y, z$.

The results of the least-squares-planes calculations,* as well as the torsion angles (Table 2), show that the maleate ion is almost planar (see Fig. 2). Note that the hydrogen bonds between maleate and perhexiline are slightly out of the plane of the maleate carboxyl group (Tables 2 and 3, and Figs. 2 and 3). These observations suggest that biological receptors for perhexiline will have a group equivalent to two oxygen atoms approximately 4·49 Å apart, as shown in Fig. 3(*b*). It has been suggested (Pessayre *et al.*, 1979) that perhexiline binds to anionic hydrophobic phospholipids.

* See deposition footnote.

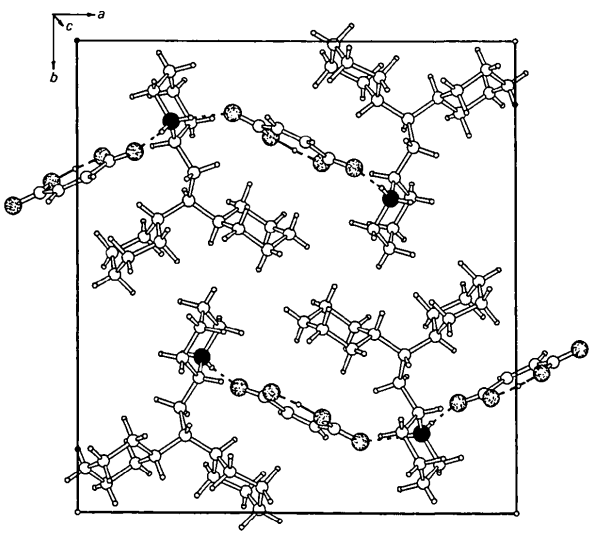


Fig. 2. Packing in the unit cell. The hydrogen-bond chains are indicated by broken lines. Note the planarity of the maleate ion.

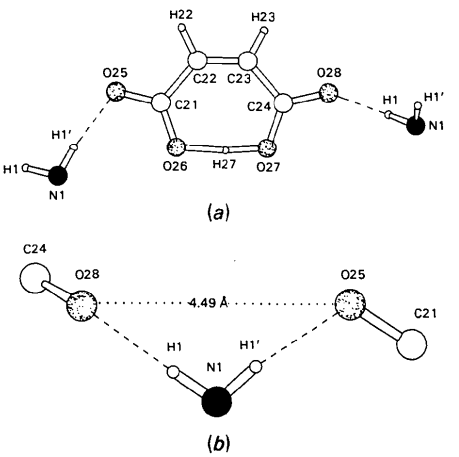
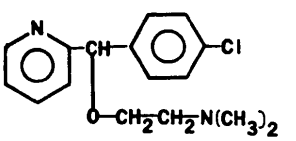
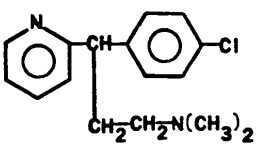


Fig. 3. (*a*) Hydrogen bonding around the maleate ion. Note the asymmetry of the positions of the nitrogen atoms of the perhexiline molecules. (*b*) Hydrogen bonding around N(1). The distance between two maleate oxygen atoms hydrogen bonded to the same N(1) of perhexiline is 4·49 Å.

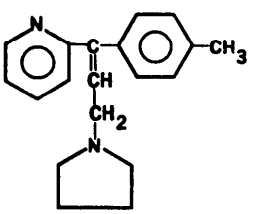
A search of the Cambridge Crystallographic Data File (Allen, Bellard, Brice, Cartwright, Doubleday, Higgs, Hummelink, Hummelink-Peters, Kennard, Motherwell, Rodgers & Watson, 1979) shows no similar saturated systems. However, the maleate of carbinoxamine (II), which is a histamine H1 receptor antagonist, shows considerable similarity in the positions of the two six-carbon-ring systems and the two nitrogen positions. However, carbinoxamine maleate, like other antihistamines such as chloropheniramine (III) and triprolidine (IV) (Bertolasi, Borea, Gilli & Sacerdoti, 1980), has aromatic ring systems instead of the saturated ring systems found in perhexiline. Presumably, this saturation will confer different biological properties on perhexiline. The comparison of perhexiline maleate with carbinoxamine maleate was made using the *DOCK* graphics program (Wood, 1982) by superimposing the first carbon atom of each cyclohexyl group and the carbon atom that connects these groups [C(9), C(15), C(8)] of perhexiline on the corresponding atoms in carbinoxamine [C(4), C(8), C(7)]. The two molecules were superposed as rigid bodies. The two functional nitrogen groups [N(1) of perhexiline maleate and N(17) of carbinoxamine] are then seen to be 1·782 Å apart (Fig. 4). However, the presence of other nitrogen atoms in (II), (III) and (IV), the unsaturation of most of their ring systems and their less-hydrophobic character will obviously affect their biological activity, compared with that of perhexiline.



(II)



(III)



(IV)

This work was supported by grants CA-10925, CA-22780, CA-06927 and RR-05539 from the National Institutes of Health, PCM84-08569 from the National Science Foundation, and by an appropriation from the Commonwealth of Pennsylvania.

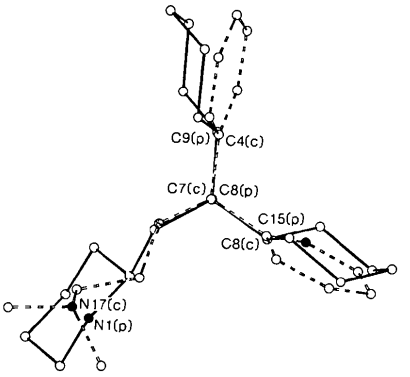


Fig. 4. Comparison of structure of perhexiline with that of an antihistamine containing aromatic rings (carbinoxamine). Atomic labels followed by (*p*) = perhexiline and by (*c*) = carbinoxamine. Perhexiline solid lines, carbinoxamine broken lines. Hydrogen and chlorine atoms have been removed for clarity.

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1-(4-Carboxyphenyl)-2-cyclohexyl-2-methylethanone

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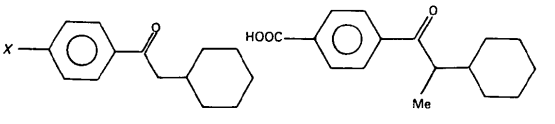
(*Received* 22 *April* 1985; *accepted* 15 *August* 1985)

Abstract. IUPAC name: 4-[(1-cyclohexylethyl)-carbonyl]benzoic acid. $C_{16}H_{20}O_3$, $M_r = 260{\cdot}33$, monoclinic, $P2_1/n$, $a = 6{\cdot}264$ (1), $b = 23{\cdot}977$ (3), $c = 9{\cdot}731$ (1) Å, $\beta = 100{\cdot}072$ (6)°, $V = 1439{\cdot}0$ (3) Å^3, $Z = 4$, $D_x = 1{\cdot}201$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1{\cdot}5418$ Å, $\mu = 6{\cdot}21$ cm^{-1}, $F(000) = 560$, $T = 295$ K, final $R = 0{\cdot}058$ for 1678 observed reflections. The carbonyl-containing side chain is equatorial with respect to the chair-shaped cyclohexane ring. The crystal contains centrosymmetric carboxylic-acid dimers.

Introduction. Irradiation of five derivatives of compound (1) (X=CH_3, Cl, CH_3O, COOH, and CN) in the solid state results in a Norrish type II reaction, *i.e.* γ-H abstraction by the carbonyl O leading to cleavage and cyclization (Ariel, Ramamurthy, Scheffer & Trotter, 1983; Ariel & Trotter, 1985); the geometric requirements for the abstraction of the γ-H have been established. The present crystallographic study was undertaken to determine whether a methyl substituent in the α-position, as in compound (2), changes the molecular conformation and thus changes the solid-state reactivity.



(1) (2)

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