Proc. Indian Acad. Sci. (Chem. Sci.), Vol. 102, No. 1, February 1990, pp. 39–43.

Crystal and molecular structure of 3-benzoylpropionic acid

S SELLADURAI, M SENTHIL KUMAR and K SUBRAMANIAN*
Department of Physics, Anna University, Madras 600 025, India

MS received 5 June 1989; revised 31 July 1989

Abstract. $C_{10}H_{10}O_3$, monoclinic, $P2_1/c$, $a = 15{\cdot}071(10)$, $b = 5{\cdot}435(9)$, $c = 16{\cdot}058(10)$ Å, $\beta = 129{\cdot}57\ (10)^\circ$, $V = 1013{\cdot}7$ Å^3, $Z = 4$, $Dm = 1{\cdot}17$ gm/cm^3, $Dc = 1{\cdot}16$ gm/cm^3, λ (CuK_α) = 1·5418Å, $\mu = 6{\cdot}8\ \mathrm{cm}^{-1}$, $Mr = 178{\cdot}2$. The structure was refined to $R = 0{\cdot}10$ for 637 observed reflections. The intensity data were estimated visually by the equi-inclination multiple film Weissenberg technique with b as the rotation axis using Ni-filtered CuK_α radiation. The interplanar angle between the carboxyl group and the benzene ring is 81(1)°. The molecules are linked in dimers around a centre of symmetry by O–H···O hydrogen bonds (2·76Å).

Keywords. Benzoylpropionic acid; Weissenberg technique; crystal structure; molecular structure.

1. Introduction

3-Benzoyl propionic acid was supplied by Prof. S Kannan, Department of Chemistry, Indira Gandhi National Open University, New Delhi. Propionic acid is an esterifying agent in the production of cellulose propionate and other propionates, which are used as mould inhibitors and preservatives. They are also used to manufacture ester solvents, fruit flavours and perfume bases (Windholz 1976). For this reason the crystal structure of 3-benzoyl propionate is of considerable interest to us in our continuing investigations on X-ray crystal structure analyses of biologically important compounds.

2. Experimental

Small needle-shaped crystals were obtained from methanol solvent by the slow evaporation method. A specimen with dimensions 0·30 × 0·40 × 0·45 mm^3 was selected. The density of the crystal was measured by using the flotation method (mixture of hexane and CCl_4 solution). Accurate values of cell parameters were obtained by using the CELN program (Shiono 1964). Intensity data were collected by the multiple film equi-inclination Weissenberg technique and estimated visually. The total number of 828 reflections recorded, in layers from 0 to 5 along the b axis, is brought to a common scale using the cross-layer data 0 and 1 along the c axis. MERGE, a part of the SHELX-76 (Sheldrick 1976) program was used for this purpose. 637 reflections were considered observed and used for refinement. The intensities were corrected for Lorentz and polarisation factors and the absorption correction was not applied.

---

* For correspondence

Table 1. Final positional parameters and equivalent isotropic thermal parameters of the non-hydrogen atoms with e.s.d.'s in parentheses.

$B_{eq} = 8/3\pi^2 \sum_i \sum_j u_{ij} a_i^* a_j^* a_i a_j$

Atom	X/a	Y/b	Z/c	B_{eq}(Å^2)
C(1)	0·810(1)	−0·150(2)	0·481(1)	4·6(5)
C(2)	0·758(1)	−0·137(2)	0·528(1)	5·3(7)
C(3)	0·788(1)	−0·289(3)	0·613(1)	7·2(8)
C(4)	0·881(2)	−0·461(3)	0·656(1)	8·5(9)
C(5)	0·936(1)	−0·477(3)	0·611(1)	7·3(8)
C(6)	0·903(1)	−0·319(3)	0·524(1)	6·6(7)
C(7)	0·774(1)	0·014(2)	0·386(1)	5·1(7)
O(7)	0·816(1)	−0·006(2)	0·339(1)	7·4(5)
C(8)	0·675(1)	0·212(2)	0·342(1)	5·9(7)
C(9)	0·644(1)	0·351(2)	0·239(1)	7·7(8)
C(10)	0·584(1)	0·190(3)	0·138(1)	6·2(7)
O(11)	0·590(1)	0·279(2)	0·063(1)	7·7(6)
O(12)	0·529(1)	−0·005(2)	0·124(1)	7·9(5)

The structure was solved by direct methods using SHELXS86 (Sheldrick 1986). Full-matrix least-squares refinement using SHELX-76 (Sheldrick 1976) with isotropic thermal parameters for the non-hydrogen atoms gave an R value of 0·16. All the hydrogen atoms were geometrically fixed by stereochemical considerations and assigned the isothermal parameters of the non-hydrogen atoms to which they are covalently bonded. They were included in the structure factor calculation but were not refined. All the non-hydrogen atoms were anisotropically refined and the R value is converged to 0·10. Unit weights were used. A final difference Fourier map was featureless with $\Delta\rho$ within $\pm 0{\cdot}13\,\mathrm{e\AA^{-3}}$. Ratio of final shift to e.s.d. is $< 0{\cdot}1$. The atomic scattering factors used for all the atoms were as provided in program SHELX-76. Computer program PARST (Nardelli 1983) was used for geometrical calculations. The final positional and thermal parameters are listed in table 1.

3. Discussion

The numbering of atoms and bond lengths are given in figure 1a. The bond angles are given in figure 1b. The average bond length in the benzene ring is 1·43 (3) Å. The equations of the least-squares planes through C(1) to C(8) and through O(11), O(12), C(10) and C(9) are given in table 2. The angle between these two planes is 81(1)°. This is comparable to the angle between the ring and the carboxyl group planes in other related structures such as β-methyl-*cis*-cinnamic acid (83°) and β-chloro-*cis*-cinnamic acid (65·4°) (Fillipakis *et al* 1972) but differs significantly from that in 3-coumaric acid (6·5°) (Raghunathan and Pattabhi 1981), 2-coumaric acid (4·8°) (Raghunathan and Pattabhi 1979), and *p*-coumaric acid (5°) (Utsumi *et al* 1970). The bond distances and angles agree with related structures (Ammon and Sundaralingam 1966; Koch and Germain 1972). C(1) is *trans* to C(9), with torsion angles C(1)–C(7)–C(8)–C(9) = −176·2(3)°. The torsion angles about the carboxyl group are given in table 3. The packing of the molecules in the unit cell viewed down the b axis is shown in

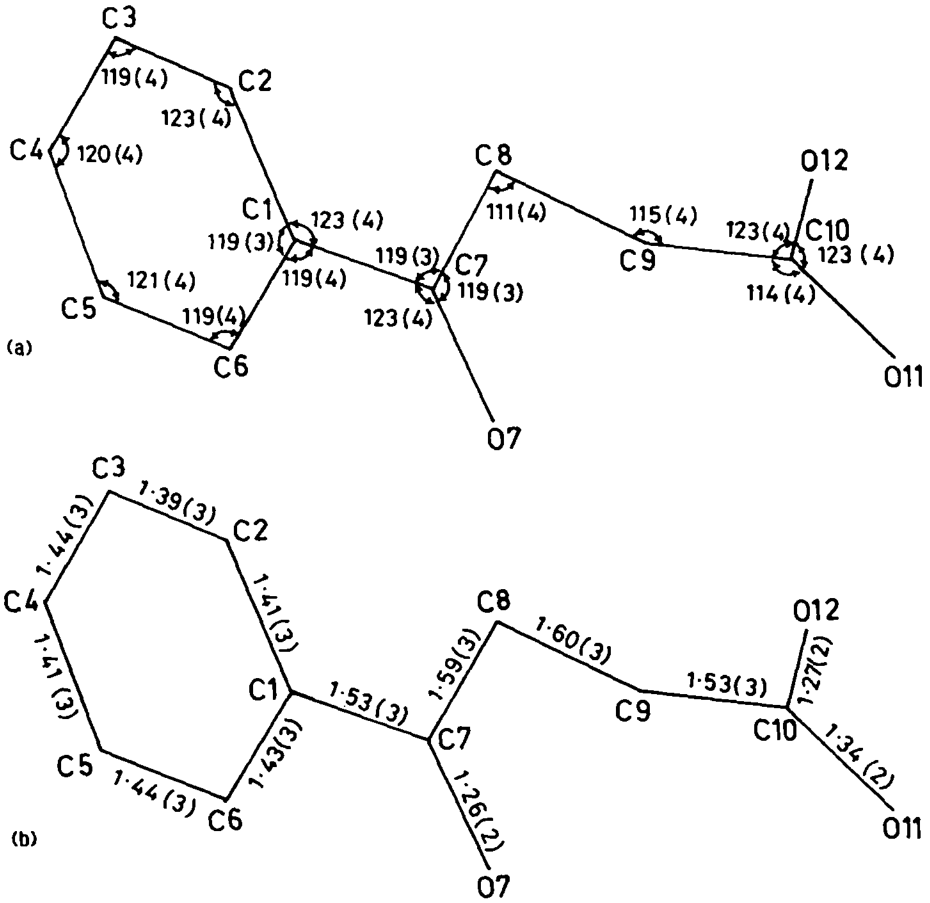

Figure 1. The molecular diagram numbering of atoms and bond lengths (**a**), bond angles (**b**).

Table 2. Equations of least-squares planes and atomic deviations (Å).

(i) Benzene ring

Plane through C(1), C(2), C(3), C(4), C(5), C(6), C(7), O(7), C(8).

$-0{\cdot}3204X-0{\cdot}6893Y-0{\cdot}6498Z+5{\cdot}6655=0.$

C(1)	0·01(4)	C(6)	−0·01(4)
C(2)	−0·01(4)	C(6)	−0·01(4)
C(3)	0·01(5)	C(7)*	0·03(4)
C(4)	−0·00(5)	O(7)*	0·12(4)
C(5)	0·01(4)	C(8)*	−0·03(4)

(ii) Carboxyl group

Plane through O(11), O(12), C(10), C(9)

$-0{\cdot}7393X+0{\cdot}5245Y-0{\cdot}4223Z+5{\cdot}6324=0.$

O(11)	0·01(4)
O(12)	0·01(4)
C(10)	−0·02(5)
C(9)	0·01(5)

* Atoms not included in plane calculation

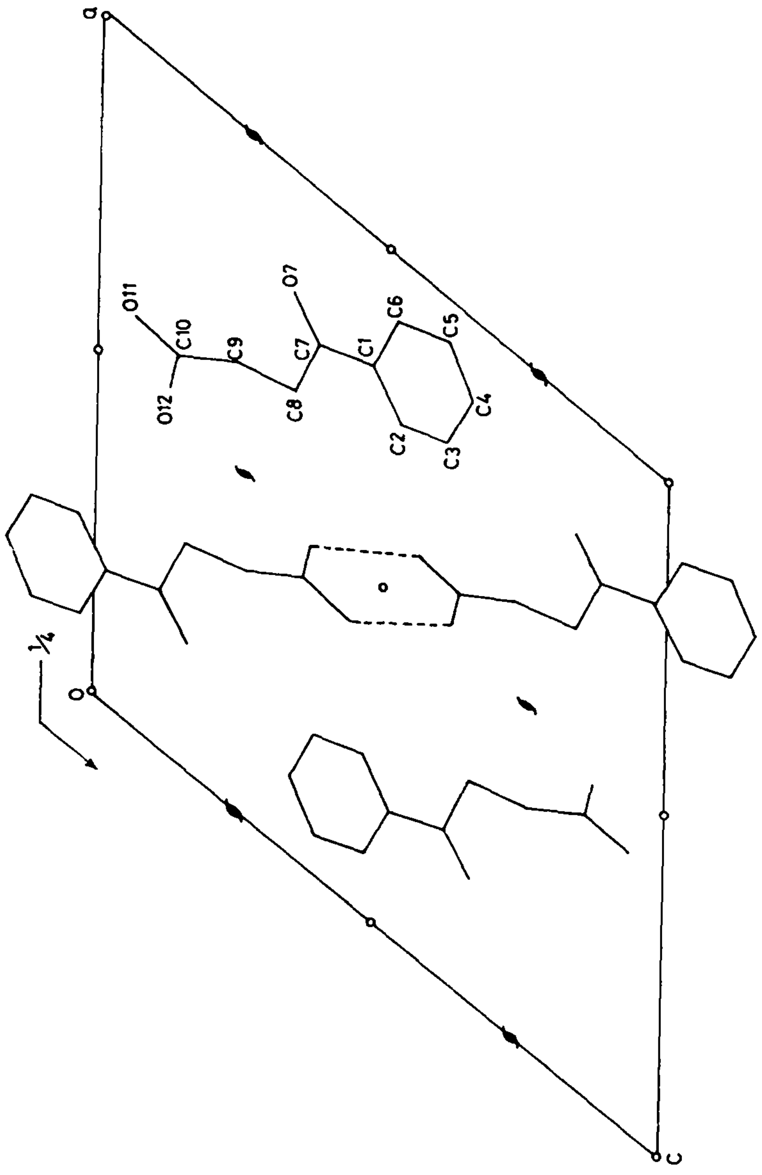

Figure 2. Packing of the molecules in the unit cell viewed down the *b* axis.

Table 3. Torsion angles (degrees) about the carboxyl group.

C(1) - C(7) - C(8) - C(9)	− 176·2(4)
O(7) - C(7) - C(8) - C(9)	2·0(6)
C(6) - C(1) - C(7) - O(7)	5·2(8)
C(6) - C(1) - C(7) - C(8)	− 176·6(4)
C(2) - C(1) - C(7) - O(7)	− 176·2(4)
C(2) - C(1) - C(7) - C(8)	2·0(6)
C(7) - C(1) - C(6) - C(5)	− 178·6(4)
C(7) - C(1) - C(2) - C(3)	178·3(4)
C(7) - C(8) - C(9) - C(10)	68·7(5)
C(8) - C(9) - C(10) - O(11)	− 160·7(4)
C(8) - C(9) - C(10) - O(12)	22·4(6)

figure 2. The molecules are linked in dimers around a centre of symmetry by O–H ··· O hydrogen bonds (2·76Å). The positions of the hydrogen atoms do not appear in the difference Fourier map, and hence the hydrogen bond angle is not reported.

Acknowledgement

One of the authors (SS) thanks the University Grants Commission, for a research fellowship.

References

Ammon H L and Sundaralingam M 1966 *J. Am. Chem. Soc.* **88** 21, 4794

Fillippakis S E, Leiserowitz L, Rabinovich D and Schmidt G M J 1972 *J. Chem. Soc., Perkin* 2, 1750

Koch M H K and Germain G 1972 *Acta Cryst.* **B28** 121

Nardelli M 1983 *J. Comput. Chem.* **7** 95

Raghunathan S and Pattabhi V 1979 *Acta Cryst.* **B34** 214

Raghunathan S and Pattabhi V 1981 *Acta Cryst.* **B37** 1299

Sheldrick G M 1976 SHELX-76. Program for crystal structure determination, University of Cambridge, England

Sheldrick G M 1986 SHELXS86. Program for crystal structure structure determination, University of Gottingen, FRG

Shiono R 1964 CELN, Program for least-squares refinement of cell constants, University of Pittsburg

Utsumi H, Fujii K, Irie H, Furusaki A and Nitta I 1970 *Nippon Kagaku Zasshi* **91** 439

Windholz M 1976 The Merck Index, Merck & Co., Rahway, NJ, USA, p. 1014