

contribution of the lattice modes will mean that the net nuclear motion is three-dimensional even for a diatomic molecule. Thus the one-dimensional model of equation (9) really considers the effect of the internal vibrational mode of a diatomic molecular crystal.

However, if the X-ray and neutron data were sufficiently accurate that one could estimate the 'half-width', σ_j , of the vibrational motion along the bond as well as the bond length shortening, then equations (13) and (1) could be used to find k_1 and k_2 . These constants are of interest theoretically as they indicate to what extent the electrons near the nuclei are involved in bonding in the molecule.

References

- BADER, R. F. W. & HENNEKER, W. H. (1967). *J. Chem. Phys.* **46**, 3341.
- BENDER, O. F. & DAVIDSON, E. R. (1966). *J. Phys. Chem.* **70**, 2675.
- BINGEL, W. A. (1963). *Z. Naturforsch.* **18A**, 1249.
- CADE, P. E., SALES, K. D. & WAHL, A. C. (1966). *J. Chem. Phys.* **44**, 1973.
- COPPENS, P. (1970). *Thermal Neutron Diffraction*. Edited by B. T. M. WILLIS. London: Oxford Univ. Press.
- COULSON, C. A. & THOMAS, M. W. (1971). *Acta Cryst.* **B27**, 1354.
- CRUICKSHANK, D. W. J. (1956). *Acta Cryst.* **9**, 757.
- HERZBERG, G. (1950). *Molecular Spectra and Molecular Structure*. Vol. I. *Spectra of Diatomic Molecules*. Princeton: Van Nostrand.
- HUO, W. M. (1965). *J. Chem. Phys.* **43**, 624.
- MCLEAN, A. D. & YOSHIMINE, M. (1967). *Tables of Linear Molecule Wave Functions*, Supplement IBM *J. Res. Dev.*
- PACK, R. T. & BYERS, BROWN, W. (1966). *J. Chem. Phys.* **45**, 556.
- THOMAS, M. W. (1971). *Acta Cryst.* **B27**, 1760.

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The Crystal Structure of 3'-Iodobiphenyl-4-carboxylic Acid

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The crystal structure of 3'-iodobiphenyl-4-carboxylic acid $C_{13}H_9O_2I$, has been determined from three-dimensional X-ray diffraction data. The crystals are triclinic, space group $P\bar{1}$, with unit-cell dimensions $a = 8.61$, $b = 15.92$, $c = 4.36$ Å, $\alpha = 92.88$, $\beta = 108.82$, $\gamma = 90.00^\circ$. The structure, which consists of centrosymmetrical hydrogen-bonded dimers, was refined by block-diagonal least-squares refinement with anisotropic thermal parameters to a residual of 6% for the 1471 observed structure factors. The molecular geometry is compared with that of related compounds.

Introduction

Halogen substitution in the 2 and 2' positions of biphenyl has been shown to produce interplanar angles of approximately 50° . As biphenyl in the solid phase is planar it was of interest to study derivatives with halogen in the 3' position. This paper describes the crystal and molecular structure of 3'-iodobiphenyl-4-carboxylic acid.

Experimental

Slow evaporation from toluene produced transparent yellow platelets, very few of which were suitable for a single-crystal X-ray investigation. The observed density at 19°C was measured by the method of flotation using aqueous cadmium n-dodecatungstaborate. Unit-cell dimensions were obtained from Weissenberg and

precession photographs. Crystal data are given in Table 1.

A single crystal of cross section 0.018×0.005 cm perpendicular to the needle axis corresponding to the c axis of the unit cell was selected. Data for the $hk0$, $hk1$, $hk2$ and $hk3$ levels of reciprocal space were collected by the multiple film Weissenberg technique using Mo $K\alpha$ radiation to minimize the effect of absorption. The intensity data for the $0kl$ level were obtained from the same crystal using a precession camera with Mo $K\alpha$ radiation.

Intensities of the spots were measured on a Joyce-Loebl flying-spot densitometer and corrected for Lorentz and polarization factors using the authors' program on an I. C. L. 1905E computer. No correction was applied for absorption.

Determination and refinement of the structure

The $hk0$ projection of the unit cell was solved from the Patterson projection and refined by a combination

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Table 1. Crystal data

C13H9O2I
a=8.61±0.01
α=92.88±0.08
U=564.7 Å³
Z=2
Mo Kα(λ=0.71069 Å)
No absent spots: space group Pī (No. 2)

M.W: 324.1
b=15.92±0.01
β=108.82±0.08
Dm=1.89±0.01 g.cm⁻³
F(000)=312

Triclinic
c=4.36±0.01 Å
γ=90.00±0.08°
Dc=1.90 g.cm⁻³
Cu Kα(λ=1.5418 Å)

Table 2. Observed and calculated structure factors in groups of constant h and l arranged in columns of k, 10|Fo| and 10|Fc|k

An unobserved reflexion has a zero in the Fo column.

Table with multiple columns containing numerical data for structure factors, including observed (Fo) and calculated (Fc) values, and their respective magnitudes.

of electron-density syntheses and block-diagonal least squares to a residual of $R=0.10$ where

$$R = \frac{\sum ||F_o| - |F_c||}{\sum |F_o|}$$

The $0kl$ projection of the sharpened Patterson gave the z coordinates of the iodine atoms.

A structure factor calculation with the 515 strongest three-dimensional reflexions based on the coordinates of the iodine atoms together with a subsequent three-dimensional electron-density synthesis gave provisional z coordinates for the remaining non-hydrogen atoms.

The trial structure was refined using full three-dimensional data by two cycles of three-dimensional Fourier synthesis and structure factor calculations and thereafter by block-diagonal least-squares applied to positional parameters, overall scale factor and individual isotropic thermal parameters to a residual of $R=0.10$. The structure-factor block-diagonal least-squares program was written by the authors for a 1905E computer. After two cycles incorporating anisotropic thermal parameters defined as

$$\exp[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{23}klb^*c^* + 2U_{31}lhc^*a^* + 2U_{12}hka^*b^*)]$$

(Cruickshank, Pilling, Bujosa, Lovell & Truter, 1961), the residual dropped to 0.073. A three-dimensional difference synthesis was calculated; this indicated regions of electron density at the positions expected for the hydrogen atoms attached to the phenyl rings. A region of density between the oxygen atoms involved in the hydrogen bonding indicated that the hydrogen was associated with O(2).

During the subsequent refinement the hydrogen atoms were fixed at distances of 1.08 Å for those attached to the phenyl rings and at 0.97 Å for the hydrogen attached to O(2). The individual scale factors for the batches of data were refined twice. The weighting

$$\text{scheme } w = 1 / \left\{ 2|F_{\min}| + |F_o| + \frac{2|F_o|^2}{|F_{\max}|} \right\} \text{ was employed.}$$

The final residual for the 1471 observed reflexions was $R=0.06$. A final Fourier summation and a difference synthesis were calculated. Slight regions of density were observed around the iodine atoms and between the planes of the two molecules.

The final structure factors are given in Table 2. Those reflexions which were not observed in the region of reciprocal space examined are marked with a zero in the F_o column.

Table 3 gives the heavy-atom coordinates and Table 4 the corresponding thermal parameters. The hydrogen-atom parameters are quoted in Table 5. The numbering of the atoms in the molecules and the arrangement of the molecules in the unit cell viewed along the c axis are shown in Fig. 1. All intermolecular contacts of less than 3.6 Å are given in Table 6 and the bond lengths and angles with some of the intramolecular non-bonded distances in Table 7. The equa-

tions of the ring system C(1)-C(6), C(7)-C(12), and the group C(10), C(13), O(1) and O(2) calculated with respect to the orthogonal triad \mathbf{a}' , \mathbf{b}' , \mathbf{c}' , where \mathbf{b}' coincides with \mathbf{b} , \mathbf{a}' is the projection of \mathbf{a} on a plane perpendicular to \mathbf{b}' and \mathbf{c}' is perpendicular to \mathbf{a}' and \mathbf{b}' , and the deviations of the atoms from the planes, are given in Table 8.

Table 3. *Non-hydrogen-atom positional parameters*

(a) Final coordinates with their estimated standard deviations (in parentheses).

	x	y	z
I	0.7424 (1)	0.5385 (05)	0.9609 (2)
O(1)	0.1909 (11)	-0.0057 (5)	-0.2292 (25)
O(2)	0.0118 (11)	0.0973 (5)	-0.2946 (26)
C(1)	0.6226 (11)	0.2714 (6)	0.8333 (25)
C(2)	0.6229 (11)	0.3578 (6)	0.8079 (27)
C(3)	0.7391 (12)	0.4091 (7)	1.0196 (28)
C(4)	0.8638 (12)	0.3770 (8)	1.2815 (28)
C(5)	0.8599 (14)	0.2891 (8)	1.3021 (30)
C(6)	0.7428 (15)	0.2390 (8)	1.0859 (33)
C(7)	0.4995 (11)	0.2173 (6)	0.5900 (26)
C(8)	0.5363 (13)	0.1372 (6)	0.4958 (31)
C(9)	0.4222 (13)	0.0875 (6)	0.2647 (29)
C(10)	0.2675 (12)	0.1186 (6)	0.1086 (28)
C(11)	0.2291 (13)	0.1978 (6)	0.2042 (30)
C(12)	0.3414 (13)	0.2468 (6)	0.4344 (29)
C(13)	0.1503 (13)	0.0650 (6)	-0.1461 (28)

Table 3 (cont.)

(b) Orthogonal coordinates with respect to the axes:

$$X' = X \sin \gamma + Z (\cos \beta - \cos \alpha \cos \gamma) / \sin \gamma$$

$$Y' = Y + X \cos \gamma + Z \cos \alpha$$

$$Z' = Z [\sin^2 \alpha - \{(\cos \beta - \cos \alpha \cos \gamma) / \sin \alpha\}^2]^{1/2}$$

	X'	Y'	Z'
I	5.044 Å	8.340 Å	3.960 Å
O(1)	1.965	-0.034	-0.945
O(2)	0.515	1.620	-1.214
C(1)	4.192	4.118	3.434
C(2)	4.230	5.500	3.330
C(3)	4.933	6.266	4.202
C(4)	5.640	5.690	5.281
C(5)	5.577	4.286	5.366
C(6)	4.873	3.541	4.475
C(7)	3.473	3.316	2.432
C(8)	3.922	2.063	2.043
C(9)	3.263	1.329	1.091
C(10)	2.151	1.862	0.447
C(11)	1.686	3.099	0.842
C(12)	2.330	3.824	1.791
C(13)	1.499	1.070	-0.602

Discussion

It has been convenient in the analysis of similar compounds to define five angles of rotation. These are rotation of one ring with respect to the other by an angle Φ_1 , about C(1)-C(7); rotation of ring C(1)-C(6) by Φ_2 about an axis which is in the plane of that ring and passes through C(1) perpendicular to C(1)-C(7); rotation of ring C(7)-C(12) by angle Φ_3 about an axis which is in the plane of that ring and passes through C(7) perpendicular to C(1)-C(7); rotation of the car-

boxyl group by Φ_4 about C(10)–C(13) and finally a rotation of the carboxyl group by Φ_5 about an axis through C(10) in the plane of the group and perpendicular to C(10)–C(13).

Starting with a planar molecule and looking along the line from C(4) towards C(13), Φ_1 and Φ_4 are positive for clockwise rotations of C(2) and O(2). The signs of Φ_2 , Φ_3 and Φ_5 are positive for clockwise rotations of the respective rings when C(4) is to the left of the view point.

Although biphenyl in the solid state is planar the introduction of an iodine atom in the 2' position causes

a strain which results in a twisting of the rings through Φ_1 of 51.3° . When the substitution is in the 3' position this strain should not be present. However, a small angle of twist might be expected due to a relayed sterical effect. The angle of $+30.4 \pm 0.5^\circ$ between the two phenyl rings is considerably smaller than that observed for 2' substituted compounds and less than the $+38.6^\circ$ observed for 4-acetyl-3'-bromobiphenyl (Sutherland & Hoy, 1969).

In the present structure the angles Φ_2 and Φ_3 are respectively $+0.7 \pm 0.4$ and $-0.4 \pm 0.4^\circ$. Comparing these with the comparable values obtained in 4-acetyl-3'-bromobiphenyl it is found that Φ_2 is not significantly different from the value of $+1.2 \pm 0.7^\circ$ whereas Φ_3 is significantly smaller than $-4.1 \pm 0.7^\circ$. The reduced values of Φ_1 and Φ_3 in this investigation are probably

Table 4. Thermal parameters ($\text{\AA}^2 \times 10^3$) for the non-hydrogen atoms

	U_{11}	U_{22}	U_{33}	$2U_{12}$	$2U_{13}$	$2U_{23}$
I	55 (1)	47 (1)	39 (1)	-5 (11)	10 (1)	2 (1)
O(1)	65 (6)	43 (5)	59 (7)	37 (8)	-16 (9)	23 (8)
O(2)	60 (5)	38 (4)	70 (7)	35 (6)	6 (9)	2 (8)
C(1)	37 (5)	39 (5)	13 (7)	2 (7)	31 (7)	39 (8)
C(2)	29 (5)	46 (5)	23 (7)	7 (7)	17 (7)	13 (8)
C(3)	37 (5)	49 (5)	19 (7)	10 (8)	20 (8)	6 (9)
C(4)	30 (5)	67 (8)	20 (7)	20 (9)	7 (10)	16 (10)
C(5)	50 (6)	61 (6)	18 (8)	25 (10)	27 (8)	47 (10)
C(6)	53 (6)	37 (5)	51 (9)	40 (9)	45 (11)	42 (10)
C(7)	37 (5)	31 (4)	24 (7)	18 (7)	27 (8)	50 (8)
C(8)	46 (5)	30 (4)	43 (8)	22 (8)	36 (10)	63 (9)
C(9)	49 (6)	28 (4)	38 (8)	17 (8)	41 (10)	71 (9)
C(10)	39 (5)	31 (4)	34 (8)	8 (7)	26 (9)	52 (8)
C(11)	41 (5)	30 (4)	37 (8)	18 (7)	18 (9)	34 (8)
C(12)	43 (5)	33 (5)	30 (7)	20 (8)	30 (9)	43 (8)
C(13)	57 (6)	26 (4)	29 (8)	14 (8)	27 (10)	96 (8)

Table 5. Coordinates of the hydrogen atoms

H(1) is attached to O(2) and the numbering of the remaining hydrogen atoms corresponds to that of the carbon atoms to which they are attached.

	x	y	z
H(1)	-0.033	0.063	-0.470
H(2)	0.531	0.385	0.604
H(4)	0.960	0.420	1.470
H(5)	0.952	0.262	1.490
H(6)	0.745	0.171	1.114
H(8)	0.655	0.112	0.620
H(9)	0.451	0.025	0.190
H(11)	0.105	0.222	0.095
H(12)	0.310	0.307	0.499

Table 6. Intermolecular distances between atoms less than 3.6\AA apart

i	j	Equipoint indication	d_{ij}
O(1)	C(8)	$\bar{x} \bar{y} \bar{z}$	3.569 \AA
C(6)	C(7)	$1+x \ y \ 1+z$	3.519
C(9)	C(9)	$1-x \ \bar{y} \ 1-z$	3.556
O(1)	O(1)	$\bar{x} \ \bar{y} \ 1-z$	3.387
O(1)	C(13)	$\bar{x} \ \bar{y} \ 1-z$	3.392
O(1)	O(2)	$\bar{x} \ \bar{y} \ 1-z$	2.611
O(2)	C(13)	$\bar{x} \ \bar{y} \ 1-z$	3.417
C(1)	C(5)	$x \ y \ 1+z$	3.568
C(2)	C(4)	$x \ y \ 1+x$	3.578
C(2)	C(5)	$x \ y \ 1+z$	3.584
O(2)	O(2)	$\bar{x} \ \bar{y} \ 1-z$	3.463

Table 7. Bond lengths and angles with their e.s.d.'s

(a) Bond lengths		
I—C(2)		3.021 (10) \AA
I—C(3)		2.091 (11)
I—C(4)		3.020 (11)
O(1)—C(9)		2.773 (15)
O(1)—C(10)		2.360 (14)
O(1)—C(13)		1.246 (15)
O(2)—C(10)		2.345 (14)
O(2)—C(11)		2.790 (15)
O(2)—C(13)		1.283 (15)
C(1)—C(2)		1.386 (14)
C(1)—C(6)		1.371 (16)
C(1)—C(7)		1.471 (14)
C(2)—C(3)		1.357 (15)
C(3)—C(4)		1.413 (16)
C(4)—C(5)		1.408 (17)
C(5)—C(6)		1.359 (17)
C(7)—C(8)		1.386 (15)
C(7)—C(12)		1.406 (15)
C(8)—C(9)		1.371 (16)
C(9)—C(10)		1.391 (15)
C(10)—C(11)		1.379 (15)
C(10)—C(13)		1.468 (15)
C(11)—C(12)		1.357 (16)
(b) Bond angles		
I—C(3)—C(2)		120.9 (8)
I—C(3)—C(4)		117.8 (8)
C(1)—C(2)—C(3)		121.9 (1.0)
C(2)—C(3)—C(4)		121.3 (1.0)
C(3)—C(4)—C(5)		115.5 (1.0)
C(4)—C(5)—C(6)		122.1 (1.1)
C(1)—C(6)—C(5)		121.6 (1.1)
C(2)—C(1)—C(6)		117.6 (1.0)
C(6)—C(1)—C(7)		122.0 (9)
C(2)—C(1)—C(7)		120.4 (9)
C(1)—C(7)—C(8)		121.7 (9)
C(1)—C(7)—C(12)		120.7 (9)
C(8)—C(7)—C(12)		117.5 (1.0)
C(7)—C(8)—C(9)		121.5 (1.0)
C(8)—C(9)—C(10)		120.0 (1.0)
C(9)—C(10)—C(11)		118.8 (1.0)
C(9)—C(10)—C(13)		118.6 (1.0)
C(11)—C(10)—C(13)		122.6 (1.0)
C(10)—C(11)—C(12)		121.3 (1.0)
C(7)—C(12)—C(11)		120.8 (1.0)
O(1)—C(13)—O(2)		122.4 (1.1)
O(1)—C(13)—C(10)		120.6 (1.0)
O(2)—C(13)—C(10)		116.8 (1.0)

due to intermolecular forces. It is of interest that the molecules of 4-acetyl-3'-bromobiphenyl adopt a herring-bone configuration whereas the 3'-iodobiphenyl-4-carboxylic acid molecules lie in a series of chains. It is unlikely that the difference in the substituent in the 4 position will have such a large effect on Φ_1 and Φ_3 . There is for example no significant difference in Φ_1 or Φ_3 between 4-acetyl-2'-chlorobiphenyl (Suther-

land and Hoy, 1968) and 2'-chlorobiphenyl-4-carboxylic acid (Sutherland, 1969).

The angles Φ_4 of $+1.4 \pm 0.7$ and Φ_5 of $-0.4 \pm 0.4^\circ$ are smaller than the corresponding values $+2.9 \pm 1.2$ and $-2.1 \pm 0.8^\circ$ observed for 4-acetyl-3'-bromobiphenyl and apart from 4-acetyl-2'-fluorobiphenyl (Young, Tollin & Sutherland, 1968) are significantly smaller than obtained for 2 and 2' halogen substituted biphenyls. The magnitude of Φ_4 and Φ_5 would appear to depend on the position of the substituent; a 3' substituent might be expected to give a smaller relayed steric effect than a 2' substituent.

A stereo view of 3'-iodobiphenyl-4-carboxylic acid is shown in Fig. 2; from this it can be seen that the line of atoms C(4), C(1), C(7), C(10) and C(13) adopts a boat configuration.

The I-C(3) bond length of 2.091 ± 0.011 Å is possibly significantly longer than the value of 2.05 ± 0.01 Å quoted in *International Tables for X-ray Crystallography* (1962) but is not significantly different from the value of 2.122 ± 0.017 Å quoted for 2'-iodobiphenyl-4-carboxylic acid (Sutherland, 1970). The iodine atom is displaced by 0.082 Å from the plane of C(1)-C(6) representing an angle of 2.3° . A comparable displacement was observed for the bromine atom in 4-acetyl-3'-bromobiphenyl. Although several of the 2' halogen-substituted biphenyls also exhibited such a displacement, the iodine in 2'-iodobiphenyl-4-carboxylic acid remained in the plane of the ring with, however, a consequent increase in Φ_2 .

The exocyclic C(10)-C(13) bond of 1.468 ± 0.015 Å and the C(1)-C(7) bond length of 1.471 ± 0.014 Å are

Table 8. Mean planes of the ring systems

Plane I:	C(1)-C(6)
II:	C(7)-C(12)
III:	C(10), C(13), O(1) and O(2)
I	$0.8127X' - 0.0736Y' - 0.5780Z' = 1.1147$
II	$0.5778X' + 0.4192Y' - 0.7003Z' = 1.6937$
III	$0.6120X' + 0.4281Y' - 0.6649Z' = 1.8078$

Deviations from mean planes

	I	II	III
I	0.082 Å		
C(1)	0.004	0.050 Å	0.237 Å
C(2)	-0.006		
C(3)	0.005		
C(4)	-0.002	0.252	0.568
C(5)	0.001		
C(6)	-0.002		
C(7)	0.059	0.001	0.121
C(8)		0.006	0.117
C(9)		-0.015	0.033
C(10)	0.238	0.017	0.009
C(11)		-0.010	-0.009
C(12)		0.001	0.065
C(13)	0.373	0.042	-0.032
O(1)		0.089	0.008
O(2)		0.133	0.008

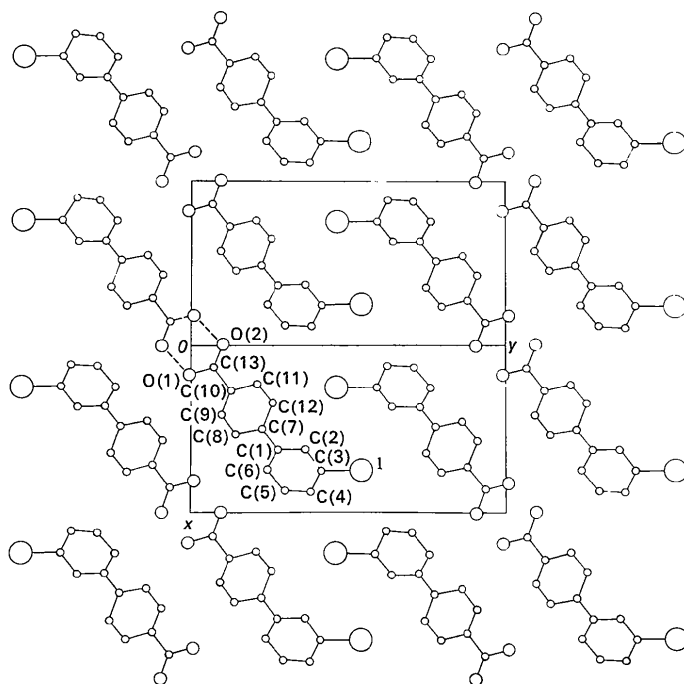


Fig. 1. The arrangement of the molecules in the unit cell viewed along the *c* axis.

in good agreement with the value of 1.477 Å quoted by Cruickshank & Sparks (1960) for the (sp^2) single-bond length between trigonally linked carbon atoms and do not differ significantly from the values obtained for other halogen substituted biphenyls.

The O(1)–C(13) and O(2)–C(13) bond lengths of 1.246 ± 0.015 and 1.283 ± 0.015 Å were respectively longer and shorter than had been expected. The bond lengths are similar to those obtained in 2'-iodobiphenyl-4-carboxylic acid of 1.261 ± 0.010 and 1.289 ± 0.010 Å respectively.

The O–H...O bond of 2.61 Å which is the shortest intermolecular distance, is similar to the value quoted

for 2'-iodobiphenyl-4-carboxylic acid of 2.60 Å. The bond angles C(10)–C(13)–O(1)–C(10)–C(13)–O(2) and O(1)–C(13)–O(2) are comparable with those found in similar compounds.

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References

- CRUICKSHANK, D. W. J., PILLING, D. E., BUJOSA, A., LOVELL, F. M. & TRUTER, M. R. (1961). In *Computing Methods and the Phase Problem in X-ray Crystal Analysis*. Oxford: Pergamon Press.
- CRUICKSHANK, D. W. J. & SPARKS, R. A. (1960). *Proc. Roy. Soc. A* **258**, 270.
- International Tables for X-ray Crystallography*. (1962). Vol. III. Birmingham: Kynoch Press.
- SUTHERLAND, H. H. (1969). *Acta Cryst.* **B25**, 171.
- SUTHERLAND, H. H. (1970). *Acta Cryst.* **B26**, 1217.
- SUTHERLAND, H. H. & HOY, T. G. (1968). *Acta Cryst.* **B24**, 1207.
- SUTHERLAND, H. H. & HOY, T. G. (1969). *Acta Cryst.* **B25**, 2385.
- YOUNG, D. W., TOLLIN, P. & SUTHERLAND, H. H. (1968). *Acta Cryst.* **B24**, 161.

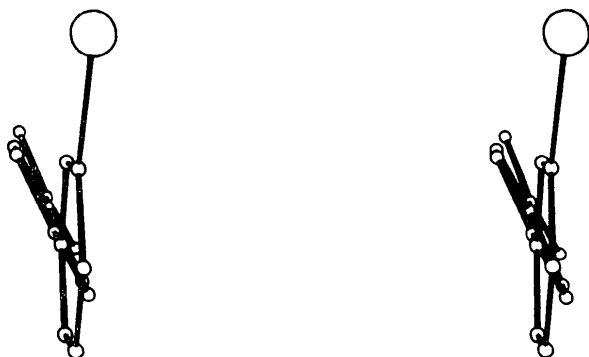


Fig. 2. A stereo view of 3'-iodobiphenyl-4-carboxylic acid.

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The Crystal and Molecular Structure of Potassium Hydrogen DL-Methylsuccinate

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The structure of potassium hydrogen DL-methylsuccinate [$\text{KO}_2\text{C}\cdot\text{CH}(\text{CH}_3)\cdot\text{CH}_2\cdot\text{CO}_2\text{H}$] was determined from three-dimensional X-ray data. The crystals are monoclinic with $a=11.927$, $b=6.217$, $c=9.731$ Å, $\beta=105.65^\circ$, $Z=4$, space group $P2_1/c$. The structure was solved by direct methods and was refined by a block-diagonal least-squares procedure to $R=0.040$. The succinic acid skeleton of the acid methylsuccinate ion is bent. The short intermolecular hydrogen bond with O(H)...O distance of 2.543(3) Å is acentric.

Introduction

We started this investigation because we were interested in the molecular conformation of the acid DL-methylsuccinate ion and in the intermolecular hydrogen bonding of acid salts of carboxylic acids in general.

In addition, knowledge of the molecular conformation might be helpful to our interpretation of the

Patterson synthesis of DL-methylsuccinic acid, the structure of which has proved to be difficult to determine because of poor crystals and the lack of reliable data.

Experimental

Crystals of potassium hydrogen DL-methylsuccinate were obtained by the slow evaporation, at room