I acknowledge with pleasure my use of the departmental X-ray crystallographic facility, which is supervised by Dr J. C. Gallucci. The diffractometer system was purchased with funds provided in part by an NIH grant.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FR1221). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1999). C**55**, 1859–1861

2′,5′-Dimethylbiphenyl-2-carboxylic acid

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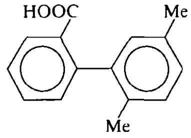
Abstract

The title acid, $C_{15}H_{14}O_2$, crystallized in the centrosymmetric space group $P2_1/n$ with one molecule in the asymmetric unit. There is a single hydrogen bond with $O_D\cdots O_A$ = 2.627 (2) Å and $O_D—H\cdots O_A$ = 174 (2)°. This bond forms an $R_2^2(8)$ cyclic dimer about a center of symmetry. The dihedral angle between the best-fit planes of the benzene rings within a molecule (biphenyl twist angle) is 85.4 (1)°. The dihedral angle between the carboxyl-group plane and the best-fit plane of the ring to which it is attached is 31.1 (3)°.

Comment

This report on 2′,5′-dimethylbiphenyl-2-carboxylic acid, (I), is one of a series on hydrogen bonding in carboxylic acids. It follows our reports on other biphenylcarboxylic acids, among which that on 6,6′-dimethylbiphenyl-2,2′-dicarboxylic acid (hereafter, DBDA; Gerkin, 1998) is of particular relevance here. Compound (I) crystallized in the centrosymmetric space group $P2_1/n$ with one molecule in the asymmetric unit. The refined molecule and the labeling scheme are shown in Fig. 1. There is a single hydrogen bond in this structure, in which $O_D—H$ = 0.98 (3) Å, $H\cdots O_A$ = 1.65 (3) Å, $O_D\cdots O_A$ = 2.627 (2) Å and $O_D—H\cdots O_A$ = 174 (2)°. The H and O atoms are ordered. This bond forms a first-level (Bernstein *et al.*, 1995) cyclic dimer with descriptor $R_2^2(8)$ about a center of symmetry; there are of course no higher-level graphs. One of the cyclic dimers appears in the packing diagram (Fig. 2).



(I)

The benzene rings in (I) are nearly planar, the maximum deviation of any of their atoms from the best-fit planes describing them being 0.002 (2) and 0.010 (2) Å, while the average deviations are 0.001 (2) and 0.006 (2) Å. These values are quite similar to the corresponding maximum and average deviations found in DBDA; 0.012 (2) Å for both rings and 0.007 (3) Å for both rings, respectively. The dihedral angle between

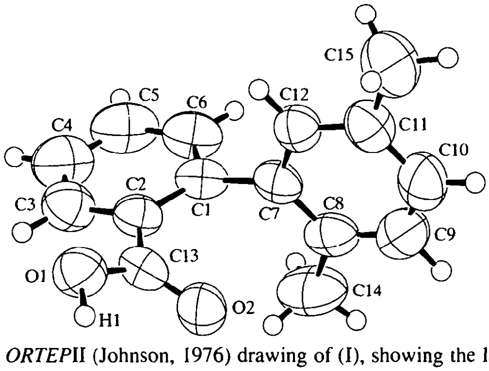


Fig. 1. *ORTEP*II (Johnson, 1976) drawing of (I), showing the labeling scheme. Displacement ellipsoids are drawn for 50% probability for non-H atoms; spheres of arbitrary small radii depict H atoms.

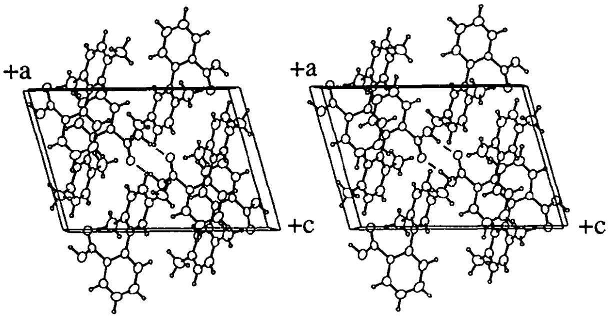


Fig. 2. *ORTEP*II (Johnson, 1976) packing stereodiagram of (I). Displacement ellipsoids are drawn for 20% probability for non-H atoms; spheres of arbitrary small radii depict H atoms. Hydrogen bonds are depicted by single dashes.

the benzene-ring planes within a molecule (biphenyl twist angle) in (I) is 85.4 (1)°; in DBDA, this angle is 84.9 (1)°. In contrast, for example, in 2-hydroxybiphenyl-3-carboxylic acid (Dobson & Gerkin, 1996), which has only a single 2-substituent, this angle is 40.52 (8)°. Twist angles in biphenyls unsubstituted at the positions *ortho* to the ring–ring bond have been discussed by Brock & Minton (1989). In (I), the dihedral angle between the plane of the carboxyl group and the plane of the ring to which it is attached is 31.1 (3)°. This value is quite similar to the mean of the corresponding values for the two carboxyl groups of DBDA, 36.7 (1) and 21.4 (1)°. In (I), the torsion angle C2—C1—C7—C8 is 97.6 (3)°, while C2—C1—C7—C12 is −84.7 (3)°.

Selected distances and angles are given in Table 1. All distances and angles fall within normal ranges. Excluding pairs of atoms in hydrogen-bonded carboxyl groups, the closest intermolecular approaches are between H14*C* and H15*B*$^{\rm i}$ [symmetry code: (i) $\frac{1}{2}+x$, $\frac{1}{2}-y$, $\frac{1}{2}+z$], and are 0.03 Å greater than the corresponding Bondi (1964) radius sum.

Experimental

The title acid was obtained from a sample in Dr M. S. Newman's chemical collection as a colorless chunk. It was used as found.

Crystal data

$C_{15}H_{14}O_2$
$M_r = 226.27$
Monoclinic
$P2_1/n$
$a = 9.374\,(2)$ Å
$b = 10.391\,(1)$ Å
$c = 13.410\,(1)$ Å
$\beta = 105.54\,(1)°$
$V = 1258.4\,(3)$ Å^3
$Z = 4$
$D_x = 1.194$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 13.6–17.3°$
$\mu = 0.078$ mm^{-1}
$T = 296$ K
Chunk
0.46 × 0.35 × 0.35 mm
Colorless

Data collection

AFC-5*S* diffractometer
ω scans
Absorption correction: none
3086 measured reflections
2914 independent reflections
1222 reflections with $I > 2\sigma I$
$R_{\rm int} = 0.015$
$\theta_{\rm max} = 27.56°$

$h = 0 \rightarrow 12$
$k = 0 \rightarrow 13$
$l = -17 \rightarrow 16$
6 standard reflections every 150 reflections intensity variation: ±1.4% (average maximum relative intensity)

Refinement

Refinement on F^2
$R(F) = 0.056$
$wR(F^2) = 0.063$
$S = 1.52$
2914 reflections
158 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2_{\rm cs} + (0.004I)^2]$
$(\Delta/\sigma)_{\rm max} = 0.0003$
$\Delta\rho_{\rm max} = 0.38$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.34$ e Å^{-3}
Extinction correction: none
Scattering factors from Stewart *et al.* (1965) (H) and Creagh & McAuley (1992) (C, O)

Table 1. *Selected geometric parameters (Å, °)*

O1—C13	1.304 (2)	C1—C7	1.492 (3)
O2—C13	1.236 (2)	C2—C13	1.484 (3)
C1—C2—C13	121.4 (2)	O1—C13—O2	122.9 (2)
C3—C2—C13	118.4 (2)		

The refined carboxyl group O—H distance is 0.98 (3) Å.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1995). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *TEXSAN*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *TEXSAN* and *PLATON* (Spek, 1990).

I acknowledge with pleasure my use of the departmental X-ray crystallographic facility, which is supervised by Dr J. C. Gallucci. The diffractometer system was purchased with funds provided in part by an NIH grant.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FR1218). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1999). C**55**, 1861–1862

Absolute configuration of 2,4,6-tri-*tert*-butyldiphenylselenonium-*N*-toluene-4′-sulfonimide

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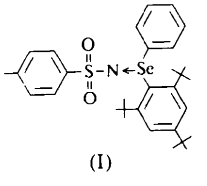
Abstract

The title compound, *N*-[phenyl(2,4,6-tri-*tert*-butylphenyl)selenonio]-4-toluenesulfonamidate, $C_{31}H_{41}NO_2SSe$, is an optically pure enantiomer which was separated by an optically active column. The absolute configuration of the selenium atom was determined as the *S* configuration.

Comment

In the course of our studies on the optical resolution of selenonium imides using optically active columns (Taka *et al.*, 1999), we found that the racemic title compound, (I), can be separated into optically pure enantiomers. To determine the absolute configuration of the selenium atom of the (−)-enantiomer, (I*a*), X-ray analysis was carried out.



(I)

Compound (I) contains a tricoordinate Se atom (Fig. 1) with an imide N atom, a phenyl group and a 2,4,6-tri-*tert*-butylphenyl group. Thus, the Se atom is a chiral centre, with a tetrahedral configuration including lone-paired electrons. The absolute configuration of the Se atom could be significantly determined as *S* by the structure refinement using Bijvoet-pair reflections. The degree of pyramidalization of the Se atom is less pronounced than that of the Se atom in *N*-phenylsulfonyl-*Se*,*Se*-diphenylselenimide, (II) (Furmanova *et al.*, 1984) and the S atom in *N*-*p*-tolylsulfonyl-*S*,*S*-diphenylsulfimide, (III) (Kálmán *et al.*, 1971). The sums of the bond angles at Se or S in (I*a*), (II) and (III) are 310.3 (3), 295.9 (12) and 308.5 (7)°, respectively.

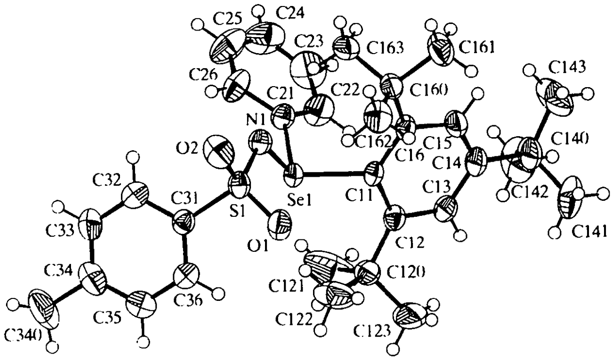


Fig. 1. The molecular structure of (I*a*) showing 50% probability displacement ellipsoids. H atoms are drawn as spheres with an arbitrary radius.

Although the length of the Se1—C21 bond corresponds with those found in (II) [1.93 (1)–1.95 (1) Å], the length of the Se1—C11 bond is significantly longer than those of Se1—C21 in (I*a*) and Se—C in (II). The deviation from planarity of C11 is more pronounced than those of C21 and C31. The sums of the bond angles at C11, C21 and C31 are 357.8 (4), 359.7 (6) and 360.0 (6)°, respectively. The less distinct pyramidal nature of the Se atom and the skewed bonds of C11 can be ascribed to the steric hindrance of two *tert*-butyl groups. No special intermolecular interactions except for van der Waals contacts were found in the crystal structure of (I*a*).

Experimental

The preparation and optical resolution of the (−)-enantiomer of (I) will be described in our related paper (Taka *et al.*, 1999). The crystals of (I*a*) used in the X-ray measurement were obtained by the slow evaporation of an acetonitrile solution in the dark.

Crystal data

$C_{31}H_{41}NO_2SSe$
$M_r = 570.67$
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å