C2—C3—C6	111.5 (1)	C6—C7—C8	120.9 (1)
C5—C4—C6	121.5 (1)	C7^{i}—C7—C8	119.6 (1)
C4—C5—C8^{i}	121.6 (1)	C1—C8—C5^{i}	121.5 (1)
C3—C6—C4	121.7 (1)	C1—C8—C7	119.7 (1)
C3—C6—C7	119.3 (1)		

Symmetry code: (i) $1 - x, 1 - y, 1 - z$.

Each of the two subsets of data [subset 1: indices $h,k,\pm l$; subset 2, collected immediately following collection of subset 1: indices $h,-k,\pm l$] was corrected to account for decay; -1.9% for subset 1 and -2.3% for subset 2. Following this, the subsets were merged, with the inclusion of a second (refined) scale factor, to form the final data file. The greatest positive residual electron density occurred ~ 0.7 Å from the C7 and C8 atoms, while the greatest negative density occurred near the centers of the unsaturated rings.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1989). 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*.

The authors thank R. H. McElheny for recrystallizing the sample and Dr J. C. Gallucci for help of various sorts. The diffractometer was purchased with funds provided in part by an NIH grant.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: FG1217). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1996). C**52**, 3088–3091

2-Hydroxybiphenyl-3-carboxylic Acid (3-Phenylsalicylic Acid)

Allison J. Dobson and Roger E. Gerkin

Department of Chemistry, The Ohio State University, Columbus, Ohio 43210, USA. E-mail: rgerkin@magnus.acs.ohio-state.edu

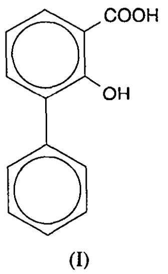
(Received 21 May 1996; accepted 12 August 1996)

Abstract

In the title compound, $C_{13}H_{10}O_3$, there is a single type of intermolecular hydrogen bond. From this is formed a cyclic dimer about a twofold axis. In addition, there is an intramolecular hydrogen bond which is virtually identical to that observed in salicylic acid.

Comment

This structural study of 2-hydroxybiphenyl-3-carboxylic acid, (I), is one of a continuing series on hydrogen bonding in carboxylic acids. Of additional interest was the potential involvement of the hydroxyl group in the hydrogen-bonding scheme; since in this molecule, the carboxyl and hydroxyl functions are located *ortho* with respect to one another, a likely possibility was that a carboxyl O atom would act as an acceptor of an intramolecular hydrogen bond from the hydroxyl group (see, for example, Blackburn, Dobson & Gerkin, 1996*a*, and references therein).



(I)

In the title acid (Figs. 1 and 2), there is a single type of intermolecular hydrogen bond, which forms cyclic dimers about a twofold symmetry axis. Also, as anticipated, an intramolecular hydrogen bond as described above is present; geometric details are given in Table 3. Distances and angles involving the carboxylic group atoms show that these O atoms are ordered and that the carboxylic H atom is also ordered (Tables 2 and 3).

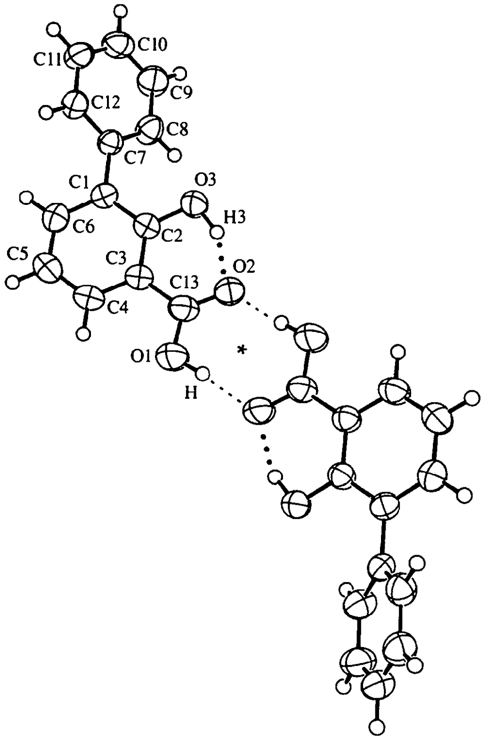


Fig. 1. An *ORTEP*II (Johnson, 1976) drawing of the 2-hydroxybiphenyl-3-carboxylic acid dimer showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level for all atoms except H atoms, for which they have been set artificially small. Intermolecular hydrogen bonds are drawn as dashed lines and intramolecular hydrogen bonds as dotted lines. The asterisk indicates the twofold symmetry axis.

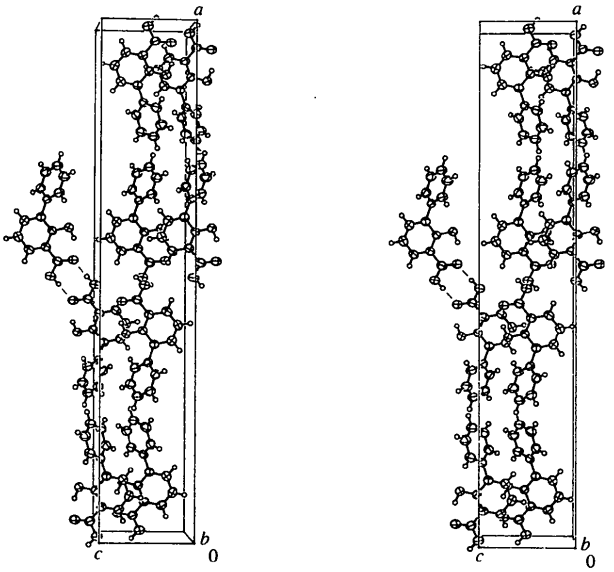


Fig. 2. An *ORTEP*II (Johnson, 1976) packing diagram of 2-hydroxybiphenyl-3-carboxylic acid. Displacement ellipsoids are drawn at the 50% probability level for all atoms except H atoms, for which they have been set artificially small. Intermolecular hydrogen bonds are shown as dashes. Intramolecular hydrogen bonds have been omitted for clarity.

The geometric parameters characterizing the intramolecular hydrogen bond may be compared to those in salicylic acid as determined most recently (X-ray) by Sundaralingam & Jensen (1965): $O_D\cdots O_A$ 2.620 (5) (X-ray) and 2.612 (3) Å (this study); O_D—$H\cdots O_A$ 146 (4) (X-ray) and 153 (3)° (this study). This close agreement indicates that the intramolecular hydrogen bonding is little affected by the presence of the additional substituent in the title acid. Moreover, there is good agreement with the two similar intermolecular hydrogen bonds found in 2,6-dihydroxybenzoic acid (MacGillivray & Zaworotko, 1994), where the $O_D\cdots O_A$ distances are 2.584 (3) and 2.603 (4) Å, and the O_D—$H\cdots O_A$ angles are 134 (3) and 146 (3)°. In contrast, whereas the carboxyl groups in the cyclic dimer in the present case are ordered, those in 2,6-dihydroxybenzoic acid are not.

Each of the phenyl rings is close to planarity. The average deviation of atoms C1–C6 from their least-squares best-fit plane is 0.003 (2) Å, while for atoms C7–C12, the corresponding value is 0.002 (2) Å. The dihedral angle between the phenyl-ring planes is 40.52 (8)°, a value similar to that found for biphenyl-3-carboxylic acid [31.78 (8)°; Blackburn, Dobson & Gerkin, 1996*b*]. The dihedral angle between the carboxyl group plane and the plane of the phenyl ring to which it is bonded is 4.4 (3)°, which is also quite similar to the corresponding value for biphenyl-3-carboxylic acid [3.1 (3)°].

Excepting approaches directly associated with the hydrogen bonding, the closest intermolecular approach involves $O1\cdots H4^{i}$ and falls short of the Bondi (1964) radius sum by 0.15 Å [symmetry code: (i) $-x, y, -\frac{1}{2}-z$]. Additional close approaches which fall short of their Bondi sums (by 0.05 Å or less) involve $C6\cdots H8^{ii}$, $H3\cdots H5^{iii}$ and $C2\cdots C5^{iv}$ [symmetry codes: (ii) $x, -y, -\frac{1}{2}+z$; (iii) $x, y, 1+z$; (iv) $x, 1-y, \frac{1}{2}+z$].

Experimental

2-Hydroxybiphenyl-3-carboxylic acid was obtained from the Sigma–Aldrich Chemical Company. The experimental sample was selected from the material as received.

Crystal data

$C_{13}H_{10}O_3$
$M_r = 214.22$
Orthorhombic
Pbcn
$a = 40.877\ (2)$ Å
$b = 6.960\ (3)$ Å
$c = 7.371\ (2)$ Å
$V = 2097\ (1)$ Å^3
$Z = 8$
$D_x = 1.357$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 14.3$–$17.3°$
$\mu = 0.090$ mm^{-1}
$T = 296$ K
Prism
0.31 × 0.31 × 0.31 mm
Colorless

Data collection

Rigaku AFC-5*S* diffractometer
ω scans
Absorption correction: none
5716 measured reflections
2858 independent reflections
1164 observed reflections [$I > 3\sigma(I)$]
$R_{\rm int} = 0.030$

$\theta_{\rm max} = 27.5°$
$h = 0 \rightarrow 53$
$k = -9 \rightarrow 9$
$l = 0 \rightarrow 8$
6 standard reflections monitored every 150 reflections intensity variation: ±1.6% (average maximum relative intensity)

Refinement

Refinement on F
$R = 0.037$
$wR = 0.046$
$S = 1.79$
1164 reflections
154 parameters
$w = 1/\sigma_F^2$
$(\Delta/\sigma)_{\rm max} = 0.01$
$\Delta\rho_{\rm max} = 0.17$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.16$ e Å^{-3}

Extinction correction: Zachariasen (1963, 1968)
Extinction coefficient: $4.2(9) \times 10^{-7}$
Atomic scattering factors from Stewart, Davidson & Simpson (1965) for H atoms and Cromer & Waber (1974) for C and O atoms

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
O1	0.00781 (4)	0.2301 (3)	0.0090 (3)	0.0814 (7)
O2	0.04016 (4)	0.1927 (3)	0.2496 (2)	0.0658 (6)
O3	0.10349 (4)	0.1963 (3)	0.2027 (2)	0.0587 (5)
C1	0.12335 (5)	0.2640 (3)	−0.0924 (3)	0.0460 (6)
C2	0.09662 (5)	0.2308 (3)	0.0254 (3)	0.0461 (6)
C3	0.06437 (5)	0.2404 (3)	−0.0400 (3)	0.0483 (7)
C4	0.05870 (6)	0.2802 (3)	−0.2230 (3)	0.0562 (7)
C5	0.08434 (6)	0.3115 (3)	−0.3383 (3)	0.0593 (8)
C6	0.11611 (6)	0.3039 (3)	−0.2727 (3)	0.0531 (7)
C7	0.15794 (5)	0.2605 (3)	−0.0290 (3)	0.0465 (6)
C8	0.16928 (5)	0.1234 (3)	0.0924 (3)	0.0588 (7)
C9	0.20180 (6)	0.1214 (4)	0.1455 (4)	0.0693 (9)
C10	0.22328 (6)	0.2558 (4)	0.0784 (4)	0.0675 (9)
C11	0.21235 (6)	0.3925 (4)	−0.0407 (4)	0.0663 (8)
C12	0.17989 (6)	0.3959 (3)	−0.0942 (3)	0.0565 (7)
C13	0.03678 (6)	0.2183 (3)	0.0843 (4)	0.0553 (8)

Table 2. *Selected geometric parameters (Å, °)*

C1—C2	1.415 (3)	C8—C9	1.386 (3)
C1—C6	1.390 (3)	C9—C10	1.375 (3)
C1—C7	1.489 (3)	C10—C11	1.370 (3)
C2—C3	1.406 (3)	C11—C12	1.384 (3)
C3—C4	1.397 (3)	C2—O3	1.358 (3)
C4—C5	1.367 (3)	C3—C13	1.461 (3)
C5—C6	1.387 (3)	C13—O1	1.310 (3)
C7—C8	1.388 (3)	C13—O2	1.239 (3)
C7—C12	1.387 (3)		
C13—O1—H	109 (1)	C1—C6—C5	122.7 (2)
C2—O3—H3	102 (1)	C1—C7—C8	122.1 (2)
C2—C1—C6	117.1 (2)	C1—C7—C12	119.6 (2)
C2—C1—C7	122.5 (2)	C8—C7—C12	118.3 (2)
C6—C1—C7	120.4 (2)	C7—C8—C9	120.6 (2)
O3—C2—C1	117.4 (2)	C8—C9—C10	120.3 (2)
O3—C2—C3	122.2 (2)	C9—C10—C11	119.6 (2)
C1—C2—C3	120.4 (2)	C10—C11—C12	120.5 (2)
C2—C3—C4	119.8 (2)	C7—C12—C11	120.7 (2)
C2—C3—C13	120.3 (2)	C3—C13—O1	115.2 (2)
C4—C3—C13	119.9 (2)	C3—C13—O2	123.1 (2)
C3—C4—C5	120.3 (2)	O1—C13—O2	121.8 (2)
C4—C5—C6	119.7 (2)		

Table 3. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H···O2$^{\rm i}$	0.92 (3)	1.75 (3)	2.661 (2)	176 (2)
O3—H3···O2	0.96 (3)	1.72 (3)	2.612 (3)	153 (3)

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

Scan widths were $(1.30 + 0.35\tan\theta)°$ in ω, with a background/scan time ratio of 0.5. The data were corrected for Lorentz and polarization effects. The Laue group assignment, systematic absences and the centrosymmetry indicated by the intensity statistics led to unique assignment of the space group as *Pbcn* (No. 60); since refinement proceeded well it was adopted. Fourier difference methods were used to locate the initial H-atom positions. In the later stages of the refinement, the ring H atoms were made canonical, with C—H distances of 0.98 Å and $U_{\rm iso}$ values $1.2U_{\rm eq}$ of the attached C atom. The H and H3 atoms were refined isotropically. The maximum effect of extinction was 10.8% of F_o for 020. The maximum peak in the final difference map occurred approximately midway between the C1 and C7 atoms; the maximum negative peak occurred near the center of the C7–C12 ring.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1989). 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*.

It is a pleasure to acknowledge the partial support provided to AJD by a National Needs Fellowship. We thank Dr J. C. Gallucci for help of various sorts. The diffractometer was purchased with funds provided in part by an NIH grant.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BK1266). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1996). C**52**, 3091-3093

9,10-Bis(1-pyridiniomethyl)anthracene Dichloride–Methanol–Water (1/2/1)

Jeffrey C. Bryan, Andrei A. Gakh and Richard A. Sachleben

Chemical and Analytical Sciences Division, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6119, USA. E-mail: bryanjc@ornl.gov

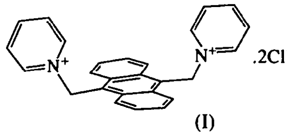
(*Received 6 June 1996; accepted 16 September 1996*)

Abstract

The title compound, 9,10-bis(1-pyridiniomethyl)anthracene dichloride, crystallizes as the methanol/water solvate $C_{26}H_{22}N_2^{2+}.2Cl^-.2CH_3OH.H_2O$, with a one-dimensional network of hydrogen bonds. Two formula units make up the asymmetric unit. The pyridine rings are on the same side of the anthracene plane.

Comment

The structure of the title compound, (I), was determined as part of our continuing group interest in molecular-recognition phenomena (Moyer & Bonnesen, 1997) and as part of a growing interest in anion recognition (Seel, Galán & de Mendoza, 1995). The title compound represents a new topography for a large diffuse cation, one that may exhibit anion selectivity.



$.2Cl^-.2CH_3OH.H_2O$

(I)

The asymmetric unit for the title compound contains two formula units and is shown in Fig. 1. All bond lengths and angles are in good agreement with standard values (Allen *et al.*, 1987). For example, the aromatic C—C bonds vary from 1.354 (3) to 1.440 (3) Å, with an average of 1.396 (3) Å. The anthracene units stack roughly along the *c* axis, with an interplanar distance of approximately 3.40 Å. One-dimensional chains are formed along the *a* axis by hydrogen bonding between two of the chloride ions (Cl1 and Cl2) and the two water solvate molecules (O5 and O6). Two of the methanol solvate molecules (containing O1 and O2) hydrogen bond to this chain at Cl1 and O6. The remaining two methanol solvate molecules (O3 and O4) hydrogen bond to Cl3 and Cl4, respectively.

The pyridyl groups are located on the same side of the anthracene ring, forming an electropositive cavity. Two methanol molecules fit head-to-tail into these cavities, with the oxygen lone pairs pointing into the

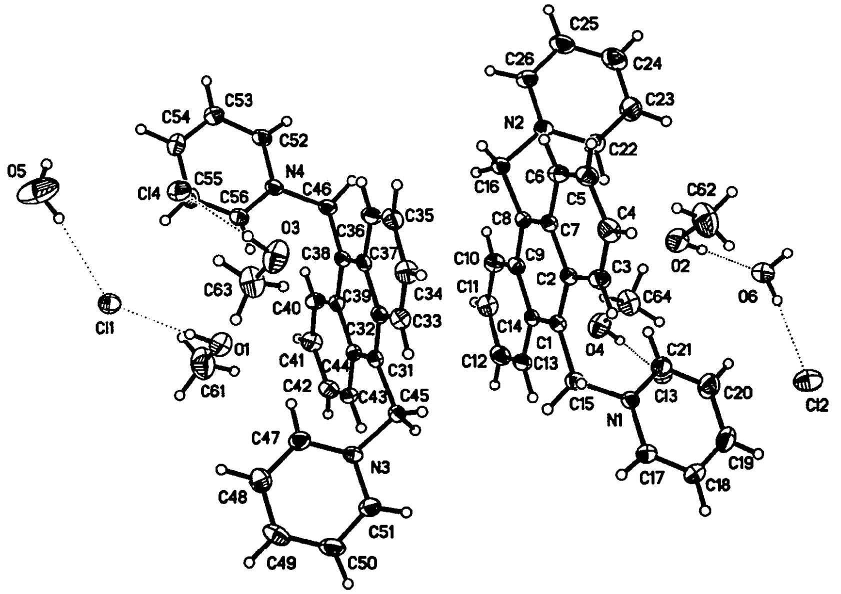


Fig. 1. The contents of the asymmetric unit shown with 50% probability displacement ellipsoids. H atoms are represented as open circles for clarity.