Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	H···*A*	*D*···*A*	*D*—H···*A*
O—H1O···Cl	2.73	3.197 (6)	118
O*W*—H1O*W*···Cl	2.52	3.168 (6)	134
N1—H1N···O*W*^{i}	2.01	2.785 (8)	150
O*W*—H2O*W*···Cl^{i}	2.18	3.165 (7)	166
N2—H2N···Cl^{ii}	2.41	3.163 (7)	146

Symmetry codes: (i) $\frac{1}{2}+x, y, \frac{1}{2}-z$; (ii) $-\frac{3}{2}-x, y-\frac{3}{2}, z$.

With the poor crystal quality, data collection had to be restricted to $\theta = 50°$, beyond which intensity decreased rapidly. All of the H atoms were placed in geometrically calculated positions (with average distances C—H 0.961, N—H 0.86 and O—H 0.82 Å), except for the two H atoms of the solvent water molecule which were located from a difference Fourier map. All hydrogen bond calculations were made using *PARST* (Nardelli, 1983).

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software* (Enraf–Nonius, 1989). Data reduction: *SDP* (Enraf–Nonius, 1985). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEX* (McArdle, 1993). Software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: VJ1028). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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4,4′-Dichloro-2,2′-iminodibenzoic Acid

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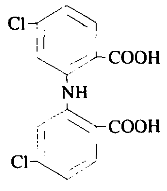
Abstract

Both rings in the title compound, $C_{14}H_9Cl_2NO_4$, are essentially planar, the r.m.s. deviation being 0.007 Å. The dihedral angle between the two planes is 44.8 (3)°. Dimerization occurs through hydrogen bonding of the carboxylic groups.

Comment

Lobenzarit acid (4-chloro-2,2′-iminodibenzoic acid) is an intermediate compound in the synthesis of lobenzarit disodium (CCA, disodium 4-chloro-2,2′-iminodibenzoate) which is an anti-rheumatic drug (Suzuki *et al.*, 1984; Pellón, 1990, 1993). We have carried out the crystallographic characterization of both compounds (Novoa, Duque, Pomés & Pellón, 1995) in the course of a crystallographic investigation of CCA analogues. Although the pharmacological activity of the title compound, (I), has not been tested, the substituents bonded to the diphenylamine skeleton makes this compound an analogue of lobenzarit acid.



(I)

Fig. 1 shows the atom-numbering scheme used. The aromatic rings are planar and the dihedral angle between the two planes is 44.8 (3)°. An internal N—H···O bifurcated hydrogen bond with the imino N atom as donor and carbonyl O atoms as acceptors is present [H(1)···O(1) 2.12 (6) Å, N(1)—H(1)···O(1) 129 (6)° and H(1)···O(4) 2.16 Å, N(1)—H(1)···O(4) 124 (6)°]. The imino group is not involved in intermolecular interactions, which is a common feature of related compounds such as fenamates (Dhanaraj & Vijayan,

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1988). The carboxyl group is, therefore, the only site for intermolecular interactions. Dimerization occurs through hydrogen bonding of the carboxylic groups [H(3a)···O(3)($2-x, y, \frac{1}{2}-z$) 1.298 (7), H(4)···O(4)($2-x, y, \frac{1}{2}-z$) 1.346 (6) Å]. Bond lengths are in good agreement with the average values given by Allen *et al.* (1987).

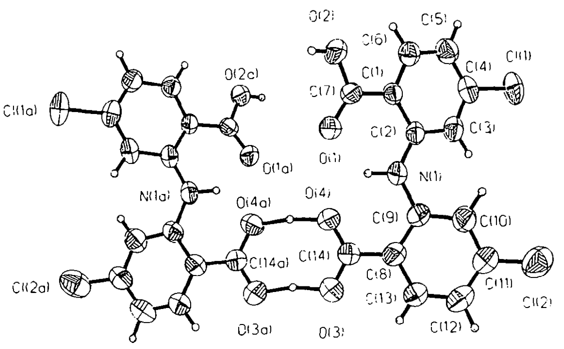

Fig. 1. Displacement ellipsoid plot (*SHELXTL-Plus*; Sheldrick, 1991) of the title compound. Ellipsoids are scaled to enclose 50% probability; H atoms are represented as spheres of arbitrary radii.

Experimental

The title compound was obtained from the reaction of a mixture of 2,4-dichlorobenzoic acid and anthranilic acid. Details of the synthetic work will be published elsewhere (Pellón, 1995). Crystals were obtained by recrystallization from a mixture of acetone and ethanol (1:1).

Crystal data

$C_{14}H_9Cl_2NO_4$
$M_r = 326.13$
Orthorhombic
Pbcn
$a = 8.653$ (2) Å
$b = 20.225$ (4) Å
$c = 14.724$ (3) Å
$V = 2576.8$ (9) Å^3
$Z = 8$
$D_x = 1.682$ Mg m^{-3}

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 25 reflections
$\theta = 4$–15°
$\mu = 4.77$ mm^{-1}
$T = 293$ (2) K
Plate
0.54 × 0.30 × 0.02 mm
Colourless

Data collection

Siemens *P*3/*PC* diffractometer
$2\theta/\theta$ scans
Absorption correction: refined from ΔF (Walker & Stuart, 1983)
$T_{\min} = 0.461$, $T_{\max} = 0.868$
1241 measured reflections
1241 independent reflections

1046 observed reflections [$I > 3\sigma(I)$]
$\theta_{\max} = 25°$
$h = 0 \rightarrow 8$
$k = -20 \rightarrow 0$
$l = 0 \rightarrow 14$
3 standard reflections monitored every 50 reflections intensity decay: 3%

Refinement

Refinement on F^2
$R(F) = 0.056$
$wR(F^2) = 0.0747$
$S = 1.68$
1241 reflections
200 parameters
Only coordinates of H atoms refined
$w = 1/[\sigma^2(F) + 0.0012F^2]$
$(\Delta/\sigma)_{\max} = 0.05$

$\Delta\rho_{\max} = 0.30$ e Å^{-3}
$\Delta\rho_{\min} = -0.33$ e Å^{-3}
Extinction correction: Zachariasen (1963)
Extinction coefficient: 0.0018 (5)
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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

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

	x	y	z	U_{eq}
Cl(1)	0.1559 (2)	0.4789 (1)	0.2993 (1)	0.076 (1)
Cl(2)	0.1727 (13)	0.7549 (5)	0.4365 (6)	0.096 (5)
O(1)	0.8734 (3)	0.5495 (2)	0.4506 (2)	0.059 (1)
O(2)	0.8472 (3)	0.4415 (2)	0.4742 (2)	0.062 (1)
O(3)	0.8644 (4)	0.7824 (2)	0.2866 (3)	0.091 (2)
O(4)	0.8636 (4)	0.6738 (2)	0.2935 (2)	0.083 (2)
N(1)	0.6295 (4)	0.6140 (2)	0.3818 (3)	0.060 (2)
C(1)	0.6385 (4)	0.4960 (2)	0.4060 (3)	0.042 (1)
C(2)	0.5573 (6)	0.5533 (2)	0.3796 (3)	0.047 (2)
C(3)	0.4075 (5)	0.5455 (3)	0.3460 (3)	0.055 (2)
C(4)	0.3426 (5)	0.4839 (3)	0.3396 (3)	0.056 (2)
C(5)	0.4216 (5)	0.4275 (2)	0.3631 (3)	0.059 (2)
C(6)	0.5692 (5)	0.4347 (2)	0.3958 (3)	0.051 (2)
C(7)	0.7946 (5)	0.4988 (2)	0.4447 (3)	0.045 (2)
C(8)	0.6454 (5)	0.7329 (2)	0.3505 (3)	0.058 (2)
C(9)	0.5613 (5)	0.6765 (2)	0.3815 (3)	0.056 (2)
C(10)	0.4130 (6)	0.6870 (2)	0.4175 (3)	0.067 (2)
C(11)	0.3493 (6)	0.7488 (3)	0.4242 (4)	0.076 (2)
C(12)	0.4350 (7)	0.8037 (3)	0.3955 (4)	0.083 (2)
C(13)	0.5796 (6)	0.7946 (3)	0.3590 (3)	0.072 (2)
C(14)	0.7995 (6)	0.7284 (3)	0.3091 (3)	0.062 (2)

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

Cl(1)—C(4)	1.724 (4)	Cl(2)—C(11)	1.54 (1)
O(1)—C(7)	1.234 (5)	O(2)—C(7)	1.317 (5)
O(3)—C(14)	1.272 (6)	O(4)—C(14)	1.257 (6)
N(1)—C(2)	1.379 (6)	N(1)—C(9)	1.395 (6)
C(1)—C(2)	1.410 (6)	C(1)—C(6)	1.385 (6)
C(1)—C(7)	1.467 (6)	C(2)—C(3)	1.396 (6)
C(3)—C(4)	1.369 (7)	C(4)—C(5)	1.376 (7)
C(5)—C(6)	1.373 (6)	C(8)—C(9)	1.427 (6)
C(8)—C(13)	1.377 (7)	C(8)—C(14)	1.469 (7)
C(9)—C(10)	1.404 (7)	C(10)—C(11)	1.368 (7)
C(11)—C(12)	1.402 (8)	C(12)—C(13)	1.374 (8)
C(2)—N(1)—C(9)	128.0 (4)	C(2)—C(1)—C(6)	119.4 (4)
C(2)—C(1)—C(7)	122.3 (4)	C(6)—C(1)—C(7)	118.3 (4)
N(1)—C(2)—C(1)	120.0 (4)	N(1)—C(2)—C(3)	122.0 (4)
C(1)—C(2)—C(3)	117.9 (4)	C(2)—C(3)—C(4)	120.5 (4)
Cl(1)—C(4)—C(3)	117.6 (4)	Cl(1)—C(4)—C(5)	120.2 (4)
C(3)—C(4)—C(5)	122.3 (4)	C(4)—C(5)—C(6)	117.6 (4)
C(1)—C(6)—C(5)	122.4 (4)	O(1)—C(7)—O(2)	121.1 (4)
O(1)—C(7)—C(1)	124.6 (4)	O(2)—C(7)—C(1)	114.4 (4)
C(9)—C(8)—C(13)	118.9 (4)	C(9)—C(8)—C(14)	123.1 (4)
C(13)—C(8)—C(14)	118.0 (4)	N(1)—C(9)—C(8)	120.5 (4)
N(1)—C(9)—C(10)	121.5 (4)	C(8)—C(9)—C(10)	117.8 (4)
C(9)—C(10)—C(11)	122.2 (4)	Cl(2)—C(11)—C(10)	118.7 (6)
Cl(2)—C(11)—C(12)	119.7 (6)	C(10)—C(11)—C(12)	119.3 (5)
C(11)—C(12)—C(13)	119.5 (5)	C(8)—C(13)—C(12)	122.3 (5)
O(3)—C(14)—O(4)	120.8 (4)	O(3)—C(14)—C(8)	117.1 (4)
O(4)—C(14)—C(8)	122.1 (4)		

The disordered H atom of the carboxylic group C(14), O(3), O(4) was split between two positions on the twofold axis ($1, y, \frac{1}{4}$) with occupancy 0.5, *i.e.* only the y coordinates were refined. The structure was solved by direct methods and refined successfully in the space group *Pbcn*. Full-matrix least-squares refinement was carried out by minimizing $w(\Delta F)^2$.

Data reduction, structure solution and refinement were carried out using *SHELXTL-Plus* (Sheldrick, 1991).

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: AB1148). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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2-Phenylmaleic Anhydride

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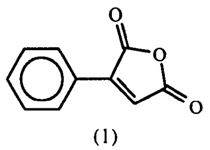
Abstract

The title compound, $C_{10}H_6O_3$, has been studied by both X-ray diffraction and molecular-mechanics calculations to determine the ring–ring dihedral angle. Calculated and observed values of 5.4 and 5.53 (6)° for this dihedral angle in the crystal were found to be in good agreement; these values were quite different from the calculated value of 40° for the gas-phase structure.

Comment

The structure of (1) is particularly important in relation to its use as a dienophile in Diels–Alder chemistry. In this context, possible differences between the solid-state and gas-phase structures are of considerable interest. The present X-ray crystallographic results show the solid-state structure of (1) to be almost planar [ring–ring dihedral angle = 5.53 (6)°]. In general, bond lengths and bond angles within (1) are similar to those found for 1,4-phenylenebis(phenylmaleic anhydride) (Fields *et al.*, 1990).



(1)

Force-field calculations were carried out to determine both the minimized crystal and gas-phase structures of (1). The *POLYGRAF* software (POLYGRAF, 1992) with the Dreiding 11 (Mayo, Olafson & Goddard, 1990) force field was employed for all molecular-mechanics calculations. The partial atomic charges in (1) for both the crystal and the gas-phase calculations were estimated by Del Re's method (Del Re, Pullman & Yonezawa, 1963) as implemented in the *POLYGRAF* software. Within *POLYGRAF*, the crystal field was provided by both van der Waals and electrostatic Ewald sums as previously described (Karasawa & Goddard, 1989).

The observed solid-state bond lengths and angles are compared with the calculated values in Table 2. Since the computed bond distances are based on empirically fit radii, there is some imperfection in the agreement between the Dreiding-based bond lengths and the experimental results, but the overall agreement with the experimental results is quite reasonable. The r.m.s. deviation between the solid-state observed and calculated values was found to be 0.05 Å for the bond lengths and 2.7° for the bond angles. The ring–ring dihedral angle calculated for the *POLYGRAF*-minimized solid-state structure was 5.4°, which is in excellent agreement with the corresponding observed value (see above). The ring–ring dihedral angle for the gas-phase molecule, as minimized by using *POLYGRAF*, was estimated to be 40°. The difference between the almost planar solid-state structure of (1) and the distinctly non-planar gas-phase structure must be attributable to the sum of the intermolecular forces acting on the molecules in the solid

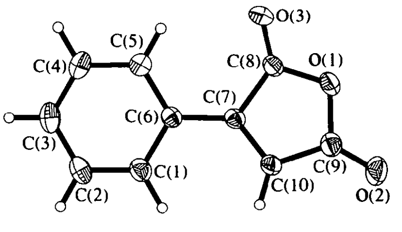


Fig. 1. The structure of (1) (50% probability thermal ellipsoids). H atoms are included as spheres of arbitrary radius.