Refinement

Refinement on F^2
$R(F) = 0.039$
$wR(F^2) = 0.114$
$S = 1.026$
2007 reflections
200 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0734P)^2 + 0.275P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.033$
$\Delta\rho_{\max} = 0.254$ e Å^{-3}
$\Delta\rho_{\min} = -0.197$ e Å^{-3}
Extinction correction: *SHELXL*97 (Sheldrick, 1997*a*)
Extinction coefficient: 0.039 (3)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 3. *Selected geometric parameters (Å, °) for (II)*

O21—C2	1.220 (2)	N1—C2	1.373 (2)
O61—C6	1.214 (2)	N1—C6	1.376 (2)
C4—C3—C31	109.2 (1)	C2—C3—C1′	112.1 (1)
C2—C3—C4	110.6 (1)	C1′—C3—C4	106.9 (1)
C2—C3—C31	105.2 (1)	C1′—C3—C31	112.9 (1)
O22′—N21′—C2′—C3′	−130.4 (2)	C2—C3—C31—C32	66.1 (2)
O22′—N21′—C2′—C1′	47.3 (2)	C2—C3—C1′—C2′	34.8 (2)
O23′—N21′—C2′—C3′	47.5 (2)	C3—C4—C5—C6	−53.2 (2)
O23′—N21′—C2′—C1′	−134.8 (2)	C31—C3—C1′—C6′	−31.9 (2)
O42′—N41′—C4′—C3′	−1.3 (3)	C31—C3—C1′—C2′	153.4 (2)
O42′—N41′—C4′—C5′	−179.4 (2)	C31—C3—C4—C5	−66.9 (2)
O43′—N41′—C4′—C5′	0.2 (3)	C4—C3—C1′—C6′	88.2 (2)
O43′—N41′—C4′—C3′	178.3 (2)	C1′—C3—C4—C5	170.7 (2)
C2—C3—C4—C5	48.4 (2)		

Table 4. *Hydrogen-bonding geometry (Å, °) for (II)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O21$^{\mathrm{i}}$	0.86	2.13	2.951 (3)	160

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

For both compounds, data collection: *STADI*4 (Stoe & Cie, 1996*a*); cell refinement: *STADI*4; data reduction: *XRED* (Stoe & Cie, 1996*b*); program(s) used to solve structures: *SHELXS*97 (Sheldrick, 1997*b*); program(s) used to refine structures: *SHELXL*97 (Sheldrick, 1997*a*); molecular graphics: *ORTEP*III (Johnson & Burnett, 1997) and *PLUTON*97 (Spek, 1997); software used to prepare material for publication: *SHELXL*97.

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4,6-Bis(benzyloxy)-1,3,5-triazin-2-ylamine

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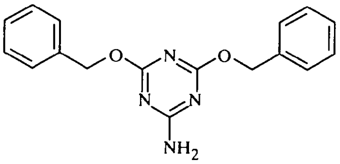
Abstract

The title compound, $C_{17}H_{16}N_4O_2$, was synthesized by the reaction of 4,6-dichloro-1,3,5-triazin-2-ylamine, obtained from cyanuric chloride, with powdered sodium hydroxide in benzyl alcohol. It crystallizes as a centrosymmetric hydrogen-bonded dimer, with only one of the two H atoms of the amino group involved in a hydrogen bond. Whereas one of the two phenyl rings is nearly coplanar with the triazine ring, the other one is nearly perpendicular to it.

Comment

DNA analogues are of considerable interest in medicinal chemistry and molecular biology, mainly because of their possible use as therapeutic agents and their potential applications in diagnostics and as biomolecular tools (Dueholm *et al.*, 1994). PNA (peptide nucleic acid) is a DNA mimic (Egholm *et al.*, 1992; Nielsen, 1996) in which the phosphate–sugar backbone has been replaced by a pseudo-peptide backbone. For the synthesis of an artificial self-pairing PNA comprising aspartic acid, in which a 1,3,5-triazine base is acylated to the aspartic acid side chain and glycine as a second amino acid, we used 1,3,5-triazine bases instead of natural nucleobases (Sommer, 1998). The ability of these compounds to build up self-pairing systems is mainly determined by formation of hydrogen bonds between the utilized 1,3,5-triazine bases. 4,6-Bis(benzyloxy)-1,3,5-triazin-2-ylamine was used as an intermediate in this PNA synthesis. The X-ray structure determination of the title compound, (I), was carried out in order to investigate its hydrogen-bonding pattern.



(I)

The bond lengths and angles in the title compound are in good agreement with values retrieved from the Cambridge Structural Database (Version 5.15 of April 1998; Allen & Kennard, 1993). Whereas the C33–C38 phenyl ring forms an angle of 16.6 (1)° with the triazine ring, the C13–C18 phenyl ring is nearly perpendicular [80.3 (1)°] to the triazine moiety.

Two molecules of the title compound form a centrosymmetric dimer, with only one of the two H atoms of the amino group involved in a hydrogen bond [N51—H51*B* 0.88 (2), H51*B*···N4 2.17 (2), N51···N4^{i} 3.041 (3) Å and N51—H51*B*···N4^{i} 175.1 (18)°; symmetry code: (i) $1 - x, 2 - y, 1 - z$]. For H51*A*, no short contact to a hydrogen-bond acceptor atom could be found.

Experimental

To a suspension of powdered NaOH (473 mg, 11 mmol) in benzyl alcohol (5 ml), 4,6-dichloro-1,3,5-triazin-2-ylamine (1 g, 5.9 mmol) was added and the solution was stirred for 30 min at 303–313 K. The solution was then heated to 363 K and stirred for 3 h. After adding diethyl ether to the mixture, the precipitated NaCl was filtered off. Crystals suitable for X-ray structure analysis were obtained from a diethyl ether/petroleum ether solution.

Crystal data

$C_{17}H_{16}N_4O_2$
$M_r = 308.34$
Triclinic
$P\bar{1}$
$a = 5.2214 (1)$ Å
$b = 13.1487 (2)$ Å
$c = 13.4792 (2)$ Å
$\alpha = 119.075 (1)°$
$\beta = 91.460 (1)°$
$\gamma = 90.094 (1)°$
$V = 808.43 (2)$ Å^3
$Z = 2$
$D_x = 1.267$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 4648 reflections
$\theta = 1–25°$
$\mu = 0.086$ mm^{-1}
$T = 293$ K
Block
0.70 × 0.40 × 0.40 mm
Colourless

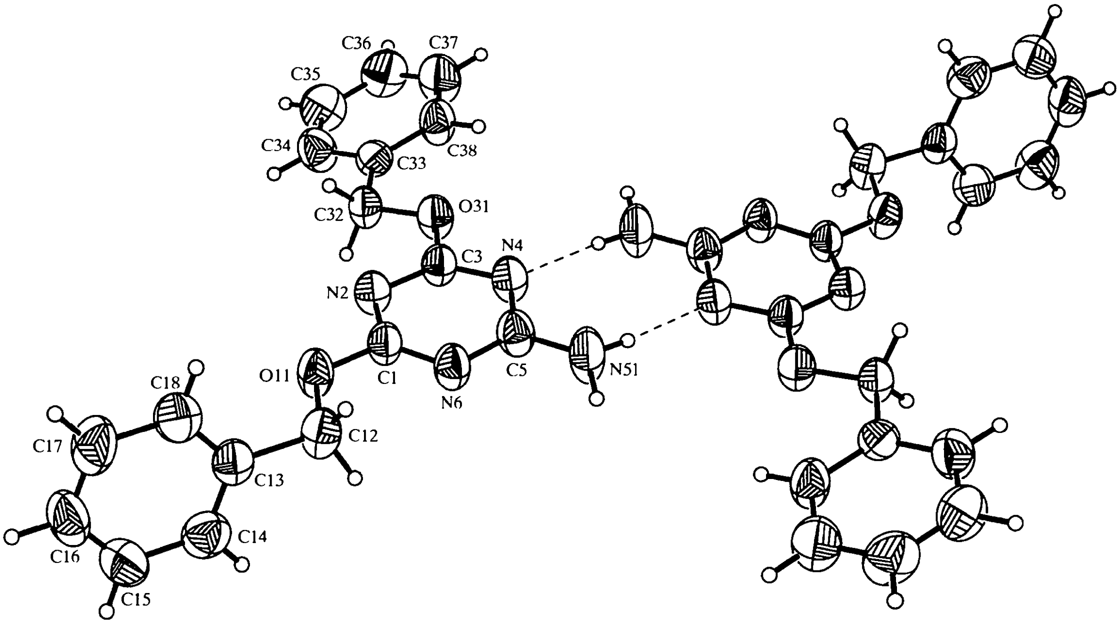


Fig. 1. A perspective view of two molecules of the title compound showing the atom-numbering scheme, the hydrogen-bond pattern and displacement ellipsoids at the 50% probability level.

Data collection

Siemens CCD three-circle diffractometer
ω scans
Absorption correction: none
7078 measured reflections
2781 independent reflections
2337 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.034$
$\theta_{\max} = 25.88°$
$h = -6 \rightarrow 6$
$k = -15 \rightarrow 16$
$l = -16 \rightarrow 16$
Intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.120$
$S = 1.130$
2781 reflections
217 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0501P)^2 + 0.1482P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.136$ e Å^{-3}
$\Delta\rho_{\min} = -0.117$ e Å^{-3}
Extinction correction: *SHELXL*97 (Sheldrick, 1997)
Extinction coefficient: 0.025 (4)
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

C1—N6	1.330 (2)	C3—N4	1.342 (2)
C1—N2	1.3541 (19)	N4—C5	1.360 (2)
N2—C3	1.343 (2)	C5—N6	1.370 (2)
N6—C1—N2	128.17 (14)	C3—N4—C5	113.71 (13)
C3—N2—C1	111.93 (13)	N4—C5—N6	124.86 (14)
N4—C3—N2	127.82 (14)	C1—N6—C5	113.43 (13)

H atoms were located by difference Fourier synthesis. Whereas the H atoms bonded to nitrogen were refined isotropically, the others were refined with fixed individual displacement parameters [$U(\text{H}) = 1.2U_{\text{eq}}(\text{C})$] using a riding model with C—H(secondary) = 0.97 Å or C—H(aromatic) = 0.93 Å.

Data collection: *SMART* (Siemens, 1995). Cell refinement: *SMART*. Data reduction: *SAINT* (Siemens, 1995). Program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997). Molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: JZ1354). Services for accessing these data are described at the back of the journal.

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2-(*p*-Toluoyl)benzoic acid

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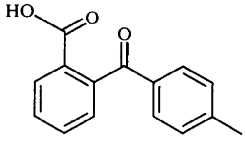
Abstract

The title compound, $C_{15}H_{12}O_3$, crystallizes as a centrosymmetric dimer hydrogen bonded *via* the carboxylic acid groups. The carboxylic acid group is nearly coplanar with the adjacent aromatic ring, which is almost perpendicular to the tolyl ring. In contrast to all comparable *o*-benzoylbenzoic acid structures retrieved from the Cambridge Structural Database, the torsion angle between the carbonyl group linking both aromatic rings and the phenyl ring carrying the carboxylic acid group is less than 90°, resulting in a shorter distance between the two carbonyl O atoms.

Comment

Substituted 2-benzoylbenzoic acids, such as the title compound, (I), are starting materials for the synthesis of 2,3-dihydrothiazolo[2,3-*a*]isoindol-5(9b*H*)-ones, compounds that inhibit reverse transcriptase of the human immune deficiency virus 1 (HIV-1) (Mertens *et al.*, 1993). The antiviral activity of these compounds depends on the substituent on the phenyl ring. 2-(*p*-Toluoyl)benzoic acid leads to a compound with an IC_{50} value of 0.77 µ*M*. The X-ray structure determination of (I) was carried out in order to elucidate the molecular conformation and the mode of hydrogen bonding.



(I)

The molecule consists of two substituted phenyl rings linked by a carbonyl group. The dihedral angle between the two aromatic rings is 89.86 (8)°. The carboxylic acid group is nearly coplanar with the adjacent aromatic ring, the dihedral angle between the two moieties being 5.2 (4)°. The carbonyl group is almost coplanar with the tolyl ring [O1—C1—C11—C16 16.3 (4)°], but it is nearly perpendicular [O1—C1—C21—C22 80.3 (3)°] to the other aromatic ring. As a result, the two C—C bonds adjacent to the carbonyl group display significantly

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