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N-Phenylanthranilic anhydride

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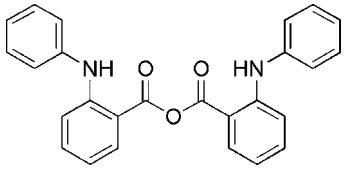
Key indicators: single-crystal X-ray study; T = 93 K; mean σ(C–C) = 0.002 Å; R factor = 0.039; wR factor = 0.111; data-to-parameter ratio = 15.7.

The complete molecule of the title compound, $C_{26}H_{20}N_2O_3$, is generated by crystallographic twofold symmetry, with the central O atom lying on the rotation axis. The conformation is stabilized by an intramolecular N—H···O hydrogen bond. The dihedral angle between the inner and outer aromatic ring planes is 61.12 (5)°.

Related literature

For the synthesis, see: Martín *et al.* (2006); Wiklund *et al.* (2004). For related structures, see: Duesler *et al.* (1981); Huelgas *et al.* (2006).



Experimental

Crystal data

$C_{26}H_{20}N_2O_3$
M_r = 408.44
Monoclinic, $C2/c$
a = 9.090 (3) Å
b = 21.056 (6) Å
c = 10.623 (3) Å
β = 100.594 (3)°
V = 1998.5 (10) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 93 K
0.33 × 0.30 × 0.18 mm

Data collection

Rigaku Spider diffractometer
Absorption correction: none
8102 measured reflections
2275 independent reflections
2000 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.111
S = 1.00
2275 reflections
145 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*N*···O1	0.903 (16)	1.966 (15)	2.6629 (14)	132.7 (13)

Data collection: *RAPID-AUTO* (Rigaku/MSC, 2004); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2927).

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supplementary materials

Acta Cryst. (2009). E**65**, o1043 [**doi:10.1107/S1600536809013002**]

N-Phenylanthranilic anhydride

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Comment

N-Phenylanthranilic acid derivatives display a antipyretic acitivity, its RhI complex is known as a remarkably active hydrogenation catalyst. *N*-phenyl anthranilic acid anhydride, which is considered as an important reaction intermediate, We here report the crystal structure of the title compound, (I).

Bond lengths and angles in (I) (Fig. 1) are within their normal ranges. In each independent molecule, the arrangement of N—H···O hydrogen bond and the planar (O=C—O) group is almost coplanar with respect to its carrier benzene ring, with dihedral angle of 6.63 (1)°, but the two benzene rings in the diphenylamine units are twisted with a dihedral angle of 61.12 (4)°. The structure is stabilized by an intramolecular hydrogen bond from an H atom of a amido N atom to a carbonyl O atom on the six-membered ring.

Experimental

The title compound was prepared according to the reported procedure of Martín *et al.* (2006) and Wiklund *et al.* (2004). Colourless chunks of (I) were obtained by recrystallization from ethyl acetate.

Refinement

The N-bound N atom was located in a difference map and its position and U_{iso} value were freely refined. The C-bound H atoms were placed in calculated positions with C—H = 0.95 Å and refined as riding with $U_{iso}(H) = 1.2U_{eq}(C)$.

Figures

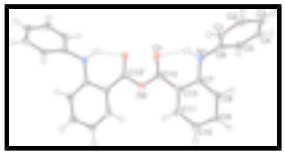

Fig. 1. The molecular structure of (I) showing 50% probability displacement ellipsoids for the non-hydrogen atoms. Symmetry code: (i) –x, y, 1/2–y.

N-Phenylanthranilic anhydride

Crystal data

$C_{26}H_{20}N_2O_3$	F_{000} = 856
M_r = 408.44	D_x = 1.358 Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -C 2yc	Cell parameters from 3250 reflections
a = 9.090 (3) Å	θ = 3.4–27.5º

b = 21.056 (6) Å	μ = 0.09 mm^{-1}
c = 10.623 (3) Å	T = 93 K
β = 100.594 (3)º	Chunk, colourless
V = 1998.5 (10) Å^3	0.33 × 0.30 × 0.18 mm
Z = 4	

Data collection

Rigaku Spider diffractometer	2000 reflections with $I > 2\sigma(I)$
Radiation source: Rotating Anode	R_{int} = 0.023
Monochromator: graphite	θ_{max} = 27.5º
T = 93 K	θ_{min} = 3.4º
ω scans	h = −11→11
Absorption correction: none	k = −27→27
8102 measured reflections	l = −10→13
2275 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.039	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.111	$w = 1/[\sigma^2(F_o^2) + (0.0706P)^2 + 0.28P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.00	$(\Delta/\sigma)_{max} < 0.001$
2275 reflections	$\Delta\rho_{max}$ = 0.25 e Å^{-3}
145 parameters	$\Delta\rho_{min}$ = −0.20 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}

O1	0.11910 (9)	0.38137 (4)	0.34545 (8)	0.0229 (2)
O2	0.0000	0.29421 (5)	0.2500	0.0225 (3)
N1	0.41159 (11)	0.37188 (5)	0.43582 (9)	0.0204 (2)
C1	0.56426 (13)	0.43835 (6)	0.59357 (11)	0.0236 (3)
H1	0.4845	0.4399	0.6401	0.028*
C2	0.69505 (14)	0.47174 (6)	0.63703 (12)	0.0275 (3)
H2	0.7045	0.4964	0.7130	0.033*
C3	0.81191 (14)	0.46930 (6)	0.57006 (13)	0.0274 (3)
H3	0.9020	0.4918	0.6006	0.033*
C4	0.79712 (13)	0.43398 (5)	0.45834 (12)	0.0246 (3)
H4	0.8771	0.4324	0.4121	0.030*
C5	0.66575 (13)	0.40089 (5)	0.41392 (11)	0.0215 (3)
H5	0.6555	0.3771	0.3369	0.026*
C6	0.54946 (12)	0.40262 (5)	0.48214 (11)	0.0185 (3)
C7	0.39399 (12)	0.30870 (5)	0.40566 (10)	0.0176 (2)
C8	0.51448 (12)	0.26579 (6)	0.43627 (11)	0.0207 (3)
H8	0.6099	0.2811	0.4764	0.025*
C9	0.49596 (13)	0.20234 (6)	0.40898 (11)	0.0233 (3)
H9	0.5787	0.1744	0.4313	0.028*
C10	0.35825 (13)	0.17796 (6)	0.34912 (11)	0.0234 (3)
H10	0.3469	0.1339	0.3305	0.028*
C11	0.23933 (13)	0.21874 (5)	0.31759 (11)	0.0206 (3)
H11	0.1453	0.2025	0.2763	0.025*
C12	0.25345 (12)	0.28396 (5)	0.34495 (10)	0.0179 (3)
C13	0.12427 (12)	0.32625 (5)	0.31547 (10)	0.0183 (2)
H1N	0.3269 (17)	0.3947 (7)	0.4330 (15)	0.039 (4)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0191 (4)	0.0209 (4)	0.0273 (5)	0.0013 (3)	0.0006 (3)	−0.0033 (3)
O2	0.0175 (6)	0.0198 (6)	0.0272 (6)	0.000	−0.0037 (5)	0.000
N1	0.0157 (5)	0.0197 (5)	0.0248 (5)	0.0012 (4)	0.0014 (4)	−0.0022 (4)
C1	0.0231 (6)	0.0258 (6)	0.0215 (6)	0.0022 (5)	0.0033 (5)	−0.0004 (5)
C2	0.0299 (7)	0.0248 (6)	0.0244 (6)	0.0003 (5)	−0.0037 (5)	−0.0046 (5)
C3	0.0219 (6)	0.0198 (6)	0.0367 (7)	−0.0020 (4)	−0.0045 (5)	0.0005 (5)
C4	0.0199 (6)	0.0200 (6)	0.0340 (7)	0.0010 (4)	0.0051 (5)	0.0036 (5)
C5	0.0225 (6)	0.0199 (6)	0.0215 (6)	0.0012 (4)	0.0027 (5)	−0.0015 (4)
C6	0.0171 (6)	0.0170 (5)	0.0197 (5)	0.0008 (4)	−0.0009 (4)	0.0014 (4)
C7	0.0181 (6)	0.0189 (5)	0.0158 (5)	−0.0003 (4)	0.0033 (4)	0.0015 (4)
C8	0.0170 (6)	0.0234 (6)	0.0212 (6)	0.0002 (4)	0.0019 (4)	0.0035 (4)
C9	0.0218 (6)	0.0230 (6)	0.0256 (6)	0.0058 (4)	0.0059 (5)	0.0055 (5)
C10	0.0260 (6)	0.0179 (5)	0.0268 (6)	0.0004 (5)	0.0064 (5)	0.0007 (5)
C11	0.0206 (6)	0.0210 (6)	0.0203 (6)	−0.0019 (4)	0.0039 (4)	0.0002 (4)
C12	0.0181 (6)	0.0202 (6)	0.0157 (5)	0.0005 (4)	0.0037 (4)	0.0014 (4)
C13	0.0173 (6)	0.0197 (5)	0.0175 (5)	−0.0021 (4)	0.0023 (4)	0.0002 (4)

Geometric parameters (Å, °)

O1—C13	1.2067 (14)	C4—H4	0.9500
O2—C13^{i}	1.3877 (12)	C5—C6	1.3876 (17)
O2—C13	1.3877 (12)	C5—H5	0.9500
N1—C7	1.3708 (15)	C7—C8	1.4109 (15)
N1—C6	1.4159 (14)	C7—C12	1.4199 (15)
N1—H1N	0.903 (16)	C8—C9	1.3708 (17)
C1—C2	1.3853 (18)	C8—H8	0.9500
C1—C6	1.3880 (16)	C9—C10	1.3936 (17)
C1—H1	0.9500	C9—H9	0.9500
C2—C3	1.3838 (19)	C10—C11	1.3728 (16)
C2—H2	0.9500	C10—H10	0.9500
C3—C4	1.3860 (18)	C11—C12	1.4047 (15)
C3—H3	0.9500	C11—H11	0.9500
C4—C5	1.3881 (16)	C12—C13	1.4610 (15)
C13^{i}—O2—C13	121.84 (12)	N1—C7—C8	121.02 (10)
C7—N1—C6	125.64 (9)	N1—C7—C12	121.25 (10)
C7—N1—H1N	116.5 (10)	C8—C7—C12	117.72 (10)
C6—N1—H1N	117.6 (10)	C9—C8—C7	121.02 (10)
C2—C1—C6	120.13 (11)	C9—C8—H8	119.5
C2—C1—H1	119.9	C7—C8—H8	119.5
C6—C1—H1	119.9	C8—C9—C10	121.37 (11)
C3—C2—C1	120.18 (11)	C8—C9—H9	119.3
C3—C2—H2	119.9	C10—C9—H9	119.3
C1—C2—H2	119.9	C11—C10—C9	118.80 (11)
C2—C3—C4	119.82 (11)	C11—C10—H10	120.6
C2—C3—H3	120.1	C9—C10—H10	120.6
C4—C3—H3	120.1	C10—C11—C12	121.54 (11)
C3—C4—C5	120.16 (12)	C10—C11—H11	119.2
C3—C4—H4	119.9	C12—C11—H11	119.2
C5—C4—H4	119.9	C11—C12—C7	119.53 (10)
C6—C5—C4	119.98 (11)	C11—C12—C13	120.82 (10)
C6—C5—H5	120.0	C7—C12—C13	119.62 (10)
C4—C5—H5	120.0	O1—C13—O2	122.15 (10)
C5—C6—C1	119.73 (10)	O1—C13—C12	126.76 (10)
C5—C6—N1	121.13 (10)	O2—C13—C12	111.05 (10)
C1—C6—N1	119.01 (10)		
C6—C1—C2—C3	−0.43 (18)	C8—C9—C10—C11	−0.18 (18)
C1—C2—C3—C4	0.82 (18)	C9—C10—C11—C12	−0.38 (17)
C2—C3—C4—C5	−0.28 (18)	C10—C11—C12—C7	0.54 (17)
C3—C4—C5—C6	−0.66 (17)	C10—C11—C12—C13	−177.48 (11)
C4—C5—C6—C1	1.05 (16)	N1—C7—C12—C11	−178.92 (10)
C4—C5—C6—N1	176.88 (10)	C8—C7—C12—C11	−0.14 (16)
C2—C1—C6—C5	−0.51 (17)	N1—C7—C12—C13	−0.87 (16)
C2—C1—C6—N1	−176.42 (10)	C8—C7—C12—C13	177.90 (10)
C7—N1—C6—C5	56.75 (16)	C13^{i}—O2—C13—O1	18.14 (8)

C7—N1—C6—C1	−127.39 (12)	C13^{i}—O2—C13—C12	−164.03 (10)
C6—N1—C7—C8	9.20 (17)	C11—C12—C13—O1	172.29 (11)
C6—N1—C7—C12	−172.07 (10)	C7—C12—C13—O1	−5.73 (18)
N1—C7—C8—C9	178.37 (10)	C11—C12—C13—O2	−5.43 (14)
C12—C7—C8—C9	−0.41 (17)	C7—C12—C13—O2	176.56 (9)
C7—C8—C9—C10	0.58 (18)		

Symmetry codes: (i) −*x*, *y*, −*z*+1/2.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1N···O1	0.903 (16)	1.966 (15)	2.6629 (14)	132.7 (13)

Fig. 1