

4-Nitro-*N*-[4-(4-nitrophenoxy)phenyl]benzamide

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Key indicators

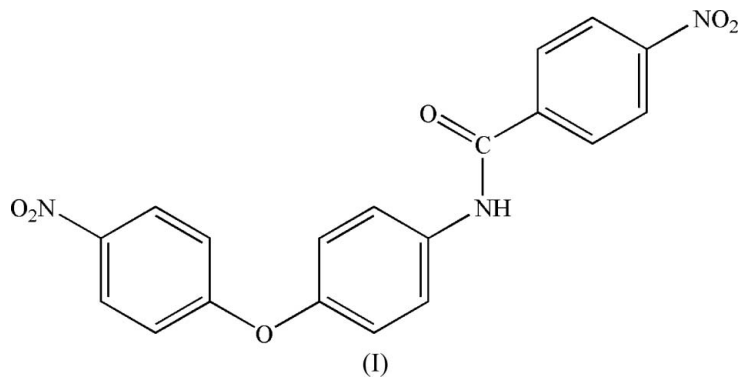
Single-crystal X-ray study
T = 173 K
Mean $\sigma(\text{C}-\text{C})$ = 0.002 Å
R factor = 0.029
wR factor = 0.079
Data-to-parameter ratio = 8.8

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The title compound, C₁₉H₁₃N₃O₆, is a precursor for the synthesis of polymers. The molecule is not planar and the terminal benzene rings are almost perpendicular to each other, at an angle of 85.19 (7)°. In the crystal structure, the molecules are linked by C—H···O and N—H···O intermolecular hydrogen bonds to form a one-dimensional network parallel to the *bc* face.

Comment

High-temperature polymers have received much attention due to the increasing demands for the replacement of ceramics and metals (Ataei *et al.*, 2005). However, in many cases, they are insoluble and do not melt below their decomposition temperature, which restricts their applications (Im & Jung, 2000). Thus, many studies have focused on obtaining aromatic polymers that are processable by conventional techniques (Yang *et al.*, 2002).



The title compound, (I), is a precursor for an attempt to synthesize polymers having excellent thermal and mechanical properties. The whole molecule is not planar (Fig. 1) although all the benzene rings and the N2/C1/O1 amide fragment are individually planar with a maximum deviation of 0.021 (1) Å for atom C14 from the least-squares plane of the C11–C16 ring. The C11–C16 and C21–C26 benzene rings are nearly coplanar, with a dihedral angle of 3.27 (9)° but make dihedral angles with the amide fragment, N2/C1/O1, of 25.27 (8) and 22.61 (8)°, respectively. The terminal C31–C36 and C11–C16 benzene rings are almost perpendicular to each other, at an angle of 85.19 (7)°. The bond lengths and angles are in normal ranges (Allen *et al.*, 1987). In the crystal structure, the molecules are linked by C—H···O and N—H···O intermolecular hydrogen bonds (Table 2) to form a one-dimensional network parallel to the *bc* face (Fig. 2).

Received 28 November 2006

Accepted 11 December 2006

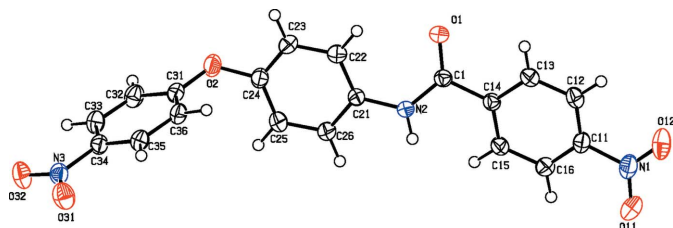


Figure 1
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

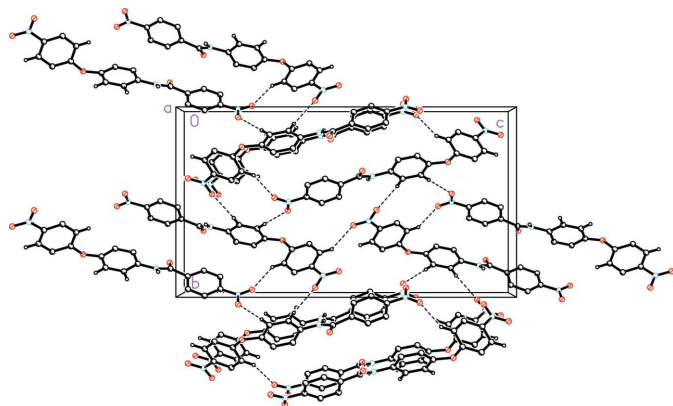


Figure 2
Packing diagram of (I) viewed down the *a* axis. The dashed lines denoted the C—H...O and N—H...O hydrogen bonds. Non-hydrogen-bonded H atoms have been omitted for clarity.

Experimental

A mixture of *p*-aminophenol (10.9 g, 0.10 mol), *p*-nitrobenzoyl chloride (18.5 g, 0.10 mol) and triethylamine (101 g, 0.10 mol) in anhydrous toluene (150 ml) was stirred in two-necked round-bottomed flask under a nitrogen atmosphere for 3 h and then allowed to stand for 16 h at room temperature. The resulting solids (mixture of the product and triethyl ammonium chloride) were filtered off and washed with water to remove the salt. In the second step a mixture of the solid product (5.0 g, 0.019 mol), anhydrous K_2CO_3 (2.62 g, 0.019 mol) and 4-chloronitrobenzene (3.0 g, 0.019 mol) in a two-necked round-bottomed flask containing 70 ml of dimethylacetamide was heated at 373 K for 20 h under a nitrogen atmosphere. The colour of the solution changed from yellow to dark brown as the reaction proceeded. After cooling to room temperature the reaction mixture was poured into 800 ml of water to form a brown solid which was washed thoroughly with water and then separated by filtration. The crude product was recrystallized from toluene. Yield 87% (6.26 g), m.p 491 K.

Crystal data

$C_{19}H_{13}N_3O_6$ $Z = 4$
 $M_r = 379.32$ $D_x = 1.501 \text{ Mg m}^{-3}$
 Orthorhombic, $P2_12_12_1$ Mo $K\alpha$ radiation
 $a = 5.0815 (2) \text{ \AA}$ $\mu = 0.11 \text{ mm}^{-1}$
 $b = 13.5782 (8) \text{ \AA}$ $T = 173 (2) \text{ K}$
 $c = 24.3279 (12) \text{ \AA}$ Rod, light brown
 $V = 1678.57 (14) \text{ \AA}^3$ $0.38 \times 0.18 \times 0.14 \text{ mm}$

Data collection

Stoe IPDSII two-circle
 diffractometer
 ω scans
 Absorption correction: none
 19554 measured reflections

2264 independent reflections
 2180 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.055$
 $\theta_{\text{max}} = 27.5^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.029$
 $wR(F^2) = 0.079$
 $S = 1.06$
 2264 reflections
 258 parameters
 H atoms treated by a mixture of
 independent and constrained
 refinement

$w = 1/[\sigma^2(F_o^2) + (0.0528P)^2 + 0.1513P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.20 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.14 \text{ e \AA}^{-3}$
 Extinction correction: *SHELXL97*
 Extinction coefficient: 0.047 (4)

Table 1

Selected bond and torsion angles ($^\circ$).

N2—C1—C14	114.66 (13)	C31—O2—C24	117.97 (12)
C1—N2—C21	127.14 (13)		
C24—O2—C31—C36	32.7 (2)	C24—O2—C31—C32	−150.29 (15)

Table 2

Hydrogen-bond geometry (\AA , $^\circ$).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C22—H22...O1	0.95	2.36	2.8988 (18)	116
N2—H2...O1 ⁱ	0.84 (3)	2.28 (3)	3.0449 (17)	152 (2)
C25—H25...O11 ⁱⁱ	0.95	2.32	3.240 (2)	164
C26—H26...O31 ⁱⁱⁱ	0.95	2.52	3.396 (2)	153
C33—H33...O32 ^{iv}	0.95	2.51	3.297 (2)	141
C36—H36...O12 ^v	0.95	2.54	3.2329 (19)	130

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

H atoms were located in a difference map, but those bonded to C were placed geometrically and allowed to ride on their parent C atoms at distances of 0.95 \AA , and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The H atom bonded to N2 was refined freely. In the absence of significant anomalous scattering effects, Friedel pairs were merged.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97*.

The authors are grateful to the Department of Chemistry, Quaid-I-Azam University, Islamabad, Pakistan for providing laboratory and analytical facilities.

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