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N-(3-Chlorophenyl)-2-nitrobenzamide

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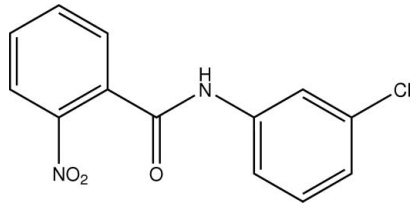
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.005 Å; R factor = 0.046; wR factor = 0.110; data-to-parameter ratio = 12.5.

Geometric parameters of the title compound, $C_{13}H_9ClN_2O_3$, are in the usual ranges. The dihedral angle between the two aromatic rings is 65.53 (7)°. The nitro group is twisted by 6.6 (5)° from the plane of the benzene ring to which it is attached. The crystal packing is stabilized by an N—H···O hydrogen bond.

Related literature

For related literature, see: Capdeville *et al.* (2002); Ho *et al.* (2002); Igawa *et al.* (1999); Jackson *et al.* (1994); Makino *et al.* (2001, 2003); Manley *et al.* (2002); Zhichkin *et al.* (2007).



Experimental

Crystal data

$C_{13}H_9ClN_2O_3$
M_r = 276.67
Orthorhombic, $Pna2_1$
a = 13.0699 (15) Å
b = 11.3731 (12) Å
c = 8.2215 (11) Å
V = 1222.1 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.32 mm^{-1}
T = 173 (2) K
0.21 × 0.15 × 0.13 mm

Data collection

Stoe IPDS II two-circle diffractometer
Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)
T_{min} = 0.936, T_{max} = 0.950
5123 measured reflections
2211 independent reflections
1912 reflections with $I > 2\sigma(I)$
R_{int} = 0.049

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.110
S = 1.10
2211 reflections
177 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}
Absolute structure: Flack (1983), with 1016 Friedel pairs
Flack parameter: −0.04 (10)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.92 (5)	2.02 (5)	2.901 (3)	159 (4)

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

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

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BX2116).

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supplementary materials

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N-(3-Chlorophenyl)-2-nitrobenzamide

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Comment

The benzanilide core is present in compounds with such a wide range of biological activities that it has been called a privileged structure. Benzanilides serve as intermediates towards benzothiadiazin-4-ones (Makino *et al.*, 2003), quinazoline-2,4-diones (Makino *et al.*, 2001) and benzodiazepine-2,5-diones (Ho *et al.*, 2002) and δ kinase inhibitors 2,3-disubstituted 3*H*-quinazoline-4-ones (Zhichkin *et al.*, 2007). Benzanilides have established their efficacy as centroid elements of ligands that bind to a wide variety of receptor types. Thus benzanilides containing aminoalkyl groups originally designed as a peptidomimetic, have been incorporated in an Arg-Gly-Asp cyclic peptide yielding a high affinity GPIIb/IIIa ligand (Jackson *et al.*, 1994). Imatinib is an ATP-site binding kinase inhibitor and platelet-derived growth factor receptor kinases (Capdeville *et al.*, 2002). Pyridylmethyl containing benzanilide are vascular endothelial growth factor receptor and tyrosine kinase inhibitor (Manley *et al.*, 2002). Furthermore, benzamides have been reported to have activities as acetyl-CoA carboxylase and farnesyl transferase inhibitors (Igawa *et al.*, 1999)

Geometric parameters of the title compound, $C_{13}H_9ClN_2O_3$, are in the usual ranges. The dihedral angle between the two aromatic rings is 65.53 (7)°. The nitro group is twisted by 6.6 (5)° from the plane of the phenyl ring to which it is attached. The crystal packing is stabilized by an N—H···O hydrogen bond.

Experimental

A mixture of 4-chloroaniline (10.0 g, 65.7 mmol), 2-nitrobenzoyl chloride (10 ml, 86.9 mmol), and pyridine (20 ml) was left at 25 °C for 15 h. Water (100 ml) was then added, and the resulting precipitates were collected. Recrystallization of the precipitates from benzene gave 12.6 g (75%) of title compound as yellow needles: mp 95–96 °C 1H NMR ($CDCl_3$) 7.23–8.30 (m, 8H, Ar—Hs), 11.36 (br s, 1H, NH).

Refinement

All H atoms were located in a difference map. Those bonded to C were geometrically positioned and refined with fixed individual displacement parameters [U(H) = 1.2 U_{eq}(C)] using a riding model with C—H = 0.95 Å. The H atom bonded to N was freely refined.

Figures

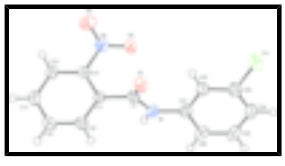


Fig. 1. Molecular structure of title compound with displacement ellipsoids at the 50% probability level.

N-(3-Chlorophenyl)-2-nitrobenzamide

Crystal data

$C_{13}H_9ClN_2O_3$	F_{000} = 568
M_r = 276.67	D_x = 1.504 Mg m^{-3}
Orthorhombic, *Pna*2$_1$	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: P 2c -2n	Cell parameters from 3179 reflections
a = 13.0699 (15) Å	θ = 3.5–24.9º
b = 11.3731 (12) Å	μ = 0.32 mm^{-1}
c = 8.2215 (11) Å	*T* = 173 (2) K
V = 1222.1 (3) Å^3	Block, colourless
Z = 4	0.21 × 0.15 × 0.13 mm

Data collection

STOE IPDS II two-circle-diffractometer	2211 independent reflections
Radiation source: fine-focus sealed tube	1912 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.049
T = 173(2) K	θ_{max} = 25.6º
ω scans	θ_{min} = 3.4º
Absorption correction: multi-scan (MULABS; Spek, 2003; Blessing, 1995)	*h* = −10→15
T_{min} = 0.936, T_{max} = 0.950	*k* = −13→13
5123 measured reflections	*l* = −9→10

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)]$ = 0.046	$w = 1/[\sigma^2(F_o^2) + (0.0624P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.110	$(\Delta/\sigma)_{max} < 0.001$
S = 1.10	$\Delta\rho_{max}$ = 0.18 e Å^{-3}
2211 reflections	$\Delta\rho_{min}$ = −0.25 e Å^{-3}
177 parameters	Extinction correction: SHELXL, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
1 restraint	Extinction coefficient: 0.017 (2)
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), 1016 Friedel pairs
Secondary atom site location: difference Fourier map	Flack parameter: −0.04 (10)

Special details

Experimental. ;

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 $F^2 > 2\text{sigma}(F^2)$ 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}
Cl1	0.93303 (7)	0.41919 (8)	0.65915 (12)	0.0466 (3)
C1	0.5135 (3)	0.4598 (3)	0.5810 (3)	0.0262 (7)
N1	0.5656 (2)	0.5412 (2)	0.4947 (3)	0.0272 (6)
H1	0.531 (4)	0.577 (4)	0.411 (6)	0.056 (12)*
O1	0.54763 (17)	0.4060 (2)	0.7001 (2)	0.0319 (5)
N2	0.4443 (2)	0.2770 (2)	0.3460 (3)	0.0309 (6)
O2	0.53558 (19)	0.3020 (2)	0.3575 (3)	0.0362 (6)
O3	0.4134 (3)	0.1931 (2)	0.2665 (4)	0.0594 (8)
C11	0.4028 (2)	0.4425 (2)	0.5315 (4)	0.0239 (6)
C12	0.3689 (2)	0.3512 (2)	0.4306 (4)	0.0254 (6)
C13	0.2658 (3)	0.3298 (3)	0.4017 (4)	0.0312 (7)
H13	0.2452	0.2664	0.3338	0.037*
C14	0.1933 (3)	0.4027 (3)	0.4738 (4)	0.0345 (8)
H14	0.1225	0.3896	0.4555	0.041*
C15	0.2251 (3)	0.4944 (3)	0.5724 (4)	0.0352 (8)
H15	0.1755	0.5444	0.6210	0.042*
C16	0.3285 (3)	0.5144 (3)	0.6014 (4)	0.0295 (7)
H16	0.3486	0.5778	0.6696	0.035*
C21	0.6723 (2)	0.5659 (3)	0.5030 (3)	0.0283 (7)
C22	0.7422 (3)	0.4869 (3)	0.5722 (4)	0.0309 (7)
H22	0.7196	0.4155	0.6200	0.037*
C23	0.8460 (3)	0.5159 (3)	0.5690 (4)	0.0349 (8)
C24	0.8818 (3)	0.6191 (3)	0.4996 (4)	0.0375 (8)
H24	0.9527	0.6373	0.4999	0.045*
C25	0.8107 (3)	0.6957 (3)	0.4291 (4)	0.0394 (8)
H25	0.8337	0.7662	0.3791	0.047*
C26	0.7070 (3)	0.6697 (3)	0.4315 (4)	0.0346 (8)
H26	0.6594	0.7229	0.3843	0.041*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0263 (4)	0.0597 (5)	0.0537 (5)	0.0065 (4)	−0.0018 (4)	0.0010 (5)
C1	0.0265 (17)	0.0272 (14)	0.0250 (15)	0.0020 (13)	0.0005 (12)	−0.0043 (12)
N1	0.0265 (15)	0.0260 (13)	0.0290 (13)	−0.0027 (11)	−0.0018 (11)	0.0000 (10)
O1	0.0272 (12)	0.0420 (12)	0.0265 (11)	0.0033 (9)	−0.0025 (9)	0.0061 (9)
N2	0.0328 (16)	0.0280 (13)	0.0319 (13)	0.0029 (12)	0.0026 (11)	−0.0049 (11)
O2	0.0271 (14)	0.0347 (12)	0.0466 (13)	0.0023 (10)	0.0045 (10)	−0.0071 (10)
O3	0.0520 (19)	0.0515 (16)	0.0746 (18)	−0.0034 (14)	0.0011 (14)	−0.0370 (16)
C11	0.0256 (16)	0.0217 (13)	0.0244 (13)	−0.0011 (12)	0.0002 (11)	0.0030 (11)
C12	0.0277 (17)	0.0216 (13)	0.0268 (13)	0.0015 (12)	0.0002 (13)	0.0004 (11)
C13	0.0286 (18)	0.0318 (17)	0.0331 (16)	−0.0049 (14)	−0.0035 (13)	−0.0009 (13)
C14	0.0234 (17)	0.0404 (19)	0.0396 (18)	−0.0021 (14)	0.0003 (13)	0.0026 (14)
C15	0.032 (2)	0.0349 (18)	0.0387 (17)	0.0081 (15)	0.0041 (15)	−0.0021 (15)
C16	0.0296 (17)	0.0286 (16)	0.0303 (14)	0.0018 (14)	−0.0007 (12)	−0.0028 (12)
C21	0.0273 (18)	0.0305 (15)	0.0272 (14)	−0.0012 (13)	−0.0010 (12)	−0.0050 (12)
C22	0.0281 (17)	0.0328 (16)	0.0318 (15)	−0.0018 (14)	0.0003 (13)	−0.0019 (13)
C23	0.029 (2)	0.0427 (18)	0.0327 (16)	0.0009 (16)	−0.0009 (14)	−0.0073 (15)
C24	0.0276 (18)	0.0450 (19)	0.0398 (18)	−0.0100 (15)	0.0026 (14)	−0.0085 (15)
C25	0.041 (2)	0.0372 (18)	0.0401 (17)	−0.0149 (15)	0.0036 (15)	−0.0043 (15)
C26	0.041 (2)	0.0275 (17)	0.0349 (16)	−0.0078 (15)	0.0003 (15)	−0.0009 (13)

Geometric parameters (Å, °)

Cl1—C23	1.747 (4)	C14—H14	0.9500
C1—O1	1.238 (3)	C15—C16	1.391 (5)
C1—N1	1.351 (4)	C15—H15	0.9500
C1—C11	1.515 (4)	C16—H16	0.9500
N1—C21	1.425 (4)	C21—C26	1.395 (5)
N1—H1	0.92 (5)	C21—C22	1.402 (5)
N2—O3	1.224 (4)	C22—C23	1.396 (5)
N2—O2	1.231 (4)	C22—H22	0.9500
N2—C12	1.472 (4)	C23—C24	1.386 (5)
C11—C12	1.401 (4)	C24—C25	1.399 (6)
C11—C16	1.394 (5)	C24—H24	0.9500
C12—C13	1.389 (4)	C25—C26	1.388 (5)
C13—C14	1.392 (5)	C25—H25	0.9500
C13—H13	0.9500	C26—H26	0.9500
C14—C15	1.385 (5)		
O1—C1—N1	124.9 (3)	C16—C15—H15	119.5
O1—C1—C11	119.5 (3)	C15—C16—C11	120.7 (3)
N1—C1—C11	115.4 (2)	C15—C16—H16	119.6
C1—N1—C21	127.2 (3)	C11—C16—H16	119.6
C1—N1—H1	117 (3)	C26—C21—C22	120.1 (3)
C21—N1—H1	115 (3)	C26—C21—N1	117.7 (3)
O3—N2—O2	122.7 (3)	C22—C21—N1	122.1 (3)

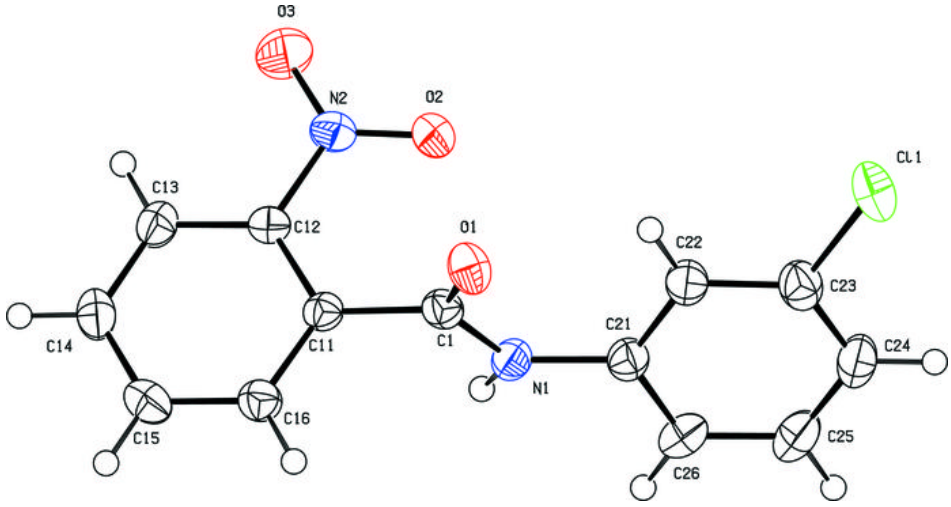
O3—N2—C12	118.6 (3)	C21—C22—C23	118.3 (3)
O2—N2—C12	118.7 (2)	C21—C22—H22	120.8
C12—C11—C16	117.3 (3)	C23—C22—H22	120.8
C12—C11—C1	123.9 (3)	C24—C23—C22	122.4 (3)
C16—C11—C1	118.6 (3)	C24—C23—Cl1	119.2 (3)
C13—C12—C11	122.6 (3)	C22—C23—Cl1	118.4 (3)
C13—C12—N2	117.9 (3)	C23—C24—C25	118.3 (3)
C11—C12—N2	119.5 (3)	C23—C24—H24	120.9
C12—C13—C14	118.8 (3)	C25—C24—H24	120.9
C12—C13—H13	120.6	C26—C25—C24	120.6 (3)
C14—C13—H13	120.6	C26—C25—H25	119.7
C15—C14—C13	119.6 (3)	C24—C25—H25	119.7
C15—C14—H14	120.2	C25—C26—C21	120.3 (3)
C13—C14—H14	120.2	C25—C26—H26	119.9
C14—C15—C16	121.0 (3)	C21—C26—H26	119.9
C14—C15—H15	119.5		
O1—C1—N1—C21	12.0 (5)	C13—C14—C15—C16	−0.4 (5)
C11—C1—N1—C21	−172.6 (3)	C14—C15—C16—C11	0.1 (5)
O1—C1—C11—C12	−85.3 (4)	C12—C11—C16—C15	0.7 (4)
N1—C1—C11—C12	99.0 (3)	C1—C11—C16—C15	−173.5 (3)
O1—C1—C11—C16	88.5 (3)	C1—N1—C21—C26	−166.0 (3)
N1—C1—C11—C16	−87.2 (3)	C1—N1—C21—C22	17.4 (5)
C16—C11—C12—C13	−1.2 (4)	C26—C21—C22—C23	0.7 (5)
C1—C11—C12—C13	172.7 (3)	N1—C21—C22—C23	177.3 (3)
C16—C11—C12—N2	176.4 (3)	C21—C22—C23—C24	−0.3 (5)
C1—C11—C12—N2	−9.7 (4)	C21—C22—C23—Cl1	178.6 (2)
O3—N2—C12—C13	−6.7 (4)	C22—C23—C24—C25	−0.7 (5)
O2—N2—C12—C13	173.0 (3)	Cl1—C23—C24—C25	−179.5 (3)
O3—N2—C12—C11	175.5 (3)	C23—C24—C25—C26	1.1 (5)
O2—N2—C12—C11	−4.8 (4)	C24—C25—C26—C21	−0.7 (5)
C11—C12—C13—C14	0.9 (5)	C22—C21—C26—C25	−0.3 (5)
N2—C12—C13—C14	−176.8 (3)	N1—C21—C26—C25	−177.0 (3)
C12—C13—C14—C15	−0.1 (5)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.92 (5)	2.02 (5)	2.901 (3)	159 (4)

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

Fig. 1


O3
N2
O2
C13
C12
O1
CL1
C22
C23
C14
C11
C1
C21
C24
N1
C16
C15
C25
C26