21562 measured reflections

 $R_{\rm int} = 0.053$

3111 independent reflections

2424 reflections with $I > 2\sigma(I)$

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N-(2-Chloro-4-nitrophenyl)-2-nitrobenzamide

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Key indicators: single-crystal X-ray study; T = 120 K; mean σ (C–C) = 0.002 Å; R factor = 0.043; wR factor = 0.108; data-to-parameter ratio = 15.6.

In the title compound, $C_{13}H_8CIN_3O_5$, the dihedral angle between the two aromatic rings is 70.74 (6)°. The nitro groups of the Cl-substituted and benzamide benzene rings are twisted by 2.6 (1) and 31.3 (2)°, respectively. The crystal packing shows intermolecular $C-H \cdots O$ hydrogen bonds that link molecules into sheets stacked along [010].

Related literature

For the biological activities of benzanilides and related compounds, see: Makino *et al.* (2003); Ho *et al.* (2002); Zhichkin *et al.* (2007); Jackson *et al.* (1994); Capdeville *et al.* (2002); Igawa *et al.* (1999). For related structures, see: Di Rienzo *et al.* (1980); Batsanov & Lyubchik (2003).



Experimental

Crystal data

 $\begin{array}{l} C_{13}H_8{\rm CIN}_3{\rm O}_5 \\ M_r = 321.67 \\ {\rm Orthorhombic}, Pbca \\ a = 7.8053 \ (9) \ {\rm \AA} \\ b = 13.8621 \ (17) \ {\rm \AA} \\ c = 24.101 \ (3) \ {\rm \AA} \end{array}$

 $V = 2607.7 (5) Å^{3}$ Z = 8Mo K\alpha radiation $\mu = 0.32 \text{ mm}^{-1}$ T = 120 (2) K $0.47 \times 0.20 \times 0.14 \text{ mm}$

Data collection

Bruker SMART APEX

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diffractometer
Absorption correction: multi-scan
(SADABS; Sheldrick, 2004)
T_{min} = 0.863, T_{max} = 0.956
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Refinement

$R[F^2 > 2\sigma(F^2)] = 0.043$	199 parameters
$wR(F^2) = 0.107$	H-atom parameters constrained
S = 1.04	$\Delta \rho_{\rm max} = 0.39 \ {\rm e} \ {\rm \AA}^{-3}$
3111 reflections	$\Delta \rho_{\rm min} = -0.23 \ {\rm e} \ {\rm \AA}^{-3}$

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	D-H	$H \cdots A$	$D \cdots A$	$D - \mathbf{H} \cdots A$
$C13-H13A\cdots O1$	0.95	2.24	2.848 (2)	121
$C10-H10A\cdots O4^{i}$	0.95	2.35	3.246 (2)	157
$C11-H11A\cdots O2^{ii}$	0.95	2.55	3.202 (2)	126

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

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; 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: SI2073).

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supplementary materials

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N-(2-Chloro-4-nitrophenyl)-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), benzodiazepine-2,5-diones (Ho *et al.*, 2002), and 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)·Benzamides have activities as acetyl-CoA carboxylase and farnesyl transferase inhibitors (Igawa *et al.*, 1999) The literature is full of the function of the 2-chloro-4-nitrophenyl group (CNP) and also structures of nitrobenzamide (NB) and related compounds (Di Rienzo *et al.*, 1980; Batsanov & Lyubchik, 2003). The aim of the present work was to combine CNP and NB in a single structure which is not well known in the literature.

Geometric parameters of the title compound, $C_{13}H_8CIN_3O_5$, are in the usual ranges. The dihedral angle between the two aromatic rings is 70.74 (6)°. The N2 nitro group is twisted by 31.3 (2)° from the plane of the C2–C7 phenyl ring, and the N3 group 2.6 (2)° from the C8–C13 plane, respectively. The crystal packing shows intermolecular C–H…O hydrogen bonds, from the Cl-phenyl group to both nitro groups. Details are depicted in Table 1. By these hydrogen bonds molecules are linked to endless sheets that are stacked along [010]. Additionally, stacking of molecules along [100] can be recognized. The intramolecular C13–H13A…O1 interaction is a common feature for this molecule with an almost planar O1–C1–N1–C8–C13 arrangement. The corresponding torsion angles are C8–N1–C1–O1 6.7 (3)° and C1–N1–C8–C13 – 7.6 (3)°, respectively.

Experimental

2-Nitrobenzoyl chloride (5.4 mmol) in CHCl₃ was treated with 2-chloro-4-nitroaniline (21.6 mmol) under a nitrogen atmosphere at reflux for 3 h. Upon cooling, the reaction mixture was diluted with CHCl₃ and washed consecutively with aq 1 *M* HCl and saturated aq NaHCO₃. The organic layer was dried over anhydrous sodium sulfate and concentrated under reduced pressure. Crystallization of the residue in CHCl₃ afforded the title compound (84%) as white needles: IR (KBr) 3226, 1665, 1616, 1520, 1352 cm-1; 1H NMR (CDCl₃, 400 MHz) ? 8.13 (d, J) 8 Hz, 1H), 7.81 (d, J) 8 Hz, 1H), 7.51 (dd, J) 8 Hz, 1H), 7.66 (dd, J) 8 Hz, 1H), 7.43 (d, J) 8 Hz, 2H), 7.36 (br s, 1H), 7.25 (d, J) 8 Hz, 1H); 13 C NMR (100 MHz) ? 164.7, 147.8, 134.6, 134.4, 132.7, 132.1, 130.3, 129.9, 129.3, 125.0. Anal. Calcd. For $C_{13}H_9N_3O_5$, C, 48.54; H, 2.51; Cl, 11.02; N, 13.06 found C, 48.12; H, 2.31; Cl, 11.3; N, 12.94.

Refinement

Hydrogen atoms were located in difference syntheses, refined at idealized positions riding on the carbon or nitrogen atoms with isotropic displacement parameters $U_{iso}(H) = 1.2U_{eq}(C \text{ or } N)$.

Figures



Fig. 1. Molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level.



Fig. 2. Crystal packing viewed along [100] with intermolecular hydrogen bonding pattern indicated as dashed lines. H-atoms not involved in hydrogen bonding are omitted.

N-(2-Chloro-4-nitrophenyl)-2-nitrobenzamide

Crystal data	
C ₁₃ H ₈ ClN ₃ O ₅	$F_{000} = 1312$
$M_r = 321.67$	$D_{\rm x} = 1.639 {\rm ~Mg~m}^{-3}$
Orthorhombic, Pbca	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -P 2ac 2ab	Cell parameters from 769 reflections
a = 7.8053 (9) Å	$\theta = 2.9 - 25.7^{\circ}$
b = 13.8621 (17) Å	$\mu = 0.32 \text{ mm}^{-1}$
c = 24.101 (3) Å	T = 120 (2) K
$V = 2607.7 (5) \text{ Å}^3$	Prism, colourless
<i>Z</i> = 8	$0.47\times0.20\times0.14~mm$
Data collection	
Bruker SMART APEX diffractometer	3111 independent reflections

2424 reflections with $I > 2\sigma(I)$ $R_{int} = 0.053$ $\theta_{max} = 27.9^{\circ}$ $\theta_{min} = 1.7^{\circ}$ $h = -10 \rightarrow 10$ $k = -18 \rightarrow 16$ $l = -31 \rightarrow 31$

Refinement

T = 120(2) K

 ϕ and ω scans

Refinement on F^2 Least-squares matrix: full $R[F^2 > 2\sigma(F^2)] = 0.043$ $wR(F^2) = 0.107$

Radiation source: sealed tube

Absorption correction: multi-scan

(SADABS; Sheldrick, 2004) $T_{\text{min}} = 0.863$, $T_{\text{max}} = 0.956$

21562 measured reflections

Monochromator: graphite

Secondary atom site location: difference Fourier map Hydrogen site location: difference Fourier map H-atom parameters constrained $w = 1/[\sigma^2(F_o^2) + (0.0553P)^2 + 0.7975P]$ where $P = (F_o^2 + 2F_c^2)/3$

<i>S</i> = 1.04	$(\Delta/\sigma)_{max} < 0.001$
3111 reflections	$\Delta \rho_{max} = 0.39 \text{ e} \text{ Å}^{-3}$
199 parameters	$\Delta \rho_{min} = -0.23 \text{ e } \text{\AA}^{-3}$

Primary atom site location: structure-invariant direct 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 $F^2 > 2 \operatorname{sigma}(F^2)$ 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 (A^2)

	x	У	Ζ	$U_{\rm iso}*/U_{\rm eq}$
Cl1	1.13944 (6)	0.38512 (3)	0.446787 (18)	0.02271 (13)
01	0.49413 (17)	0.41462 (11)	0.39481 (5)	0.0317 (3)
O2	0.6129 (2)	0.24007 (10)	0.33719 (6)	0.0374 (4)
O3	0.4575 (2)	0.23191 (11)	0.26277 (7)	0.0428 (4)
O4	0.38629 (18)	0.37084 (12)	0.57016 (6)	0.0388 (4)
O5	0.54709 (19)	0.33143 (11)	0.63925 (5)	0.0345 (4)
N1	0.7841 (2)	0.41233 (11)	0.40825 (6)	0.0221 (3)
H1A	0.8816	0.4222	0.3908	0.027*
N2	0.5594 (2)	0.27328 (11)	0.29309 (7)	0.0269 (4)
N3	0.5267 (2)	0.35472 (11)	0.59071 (6)	0.0229 (3)
C1	0.6395 (2)	0.41795 (13)	0.37682 (7)	0.0208 (4)
C2	0.6749 (2)	0.43395 (13)	0.31581 (7)	0.0191 (4)
C3	0.7458 (2)	0.52025 (13)	0.29780 (7)	0.0226 (4)
H3A	0.7797	0.5675	0.3242	0.027*
C4	0.7676 (3)	0.53806 (14)	0.24147 (7)	0.0253 (4)
H4A	0.8179	0.5969	0.2296	0.030*
C5	0.7163 (3)	0.47032 (14)	0.20255 (7)	0.0254 (4)
H5A	0.7310	0.4831	0.1641	0.030*
C6	0.6437 (2)	0.38416 (14)	0.21950 (7)	0.0237 (4)
H6A	0.6073	0.3376	0.1931	0.028*
C7	0.6253 (2)	0.36732 (13)	0.27579 (7)	0.0200 (4)
C8	0.7961 (2)	0.39263 (12)	0.46521 (7)	0.0190 (4)
C9	0.9581 (2)	0.37737 (12)	0.48863 (7)	0.0196 (4)
C10	0.9792 (2)	0.35510 (13)	0.54414 (7)	0.0216 (4)
H10A	1.0907	0.3443	0.5587	0.026*
C11	0.8378 (2)	0.34852 (13)	0.57846 (7)	0.0211 (4)
H11A	0.8497	0.3338	0.6168	0.025*

supplementary materials

C12	0.6785 (2)	0.36410 (12)	0.55510 (7)	0.0192 (4)
C13	0.6534 (2)	0.38633 (12)	0.49987 (7)	0.0194 (4)
H13A	0.5414	0.3971	0.4858	0.023*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0156 (2)	0.0279 (2)	0.0246 (2)	0.00115 (17)	0.00315 (15)	0.00015 (17)
01	0.0189 (7)	0.0559 (10)	0.0203 (6)	0.0026 (7)	0.0019 (5)	0.0048 (6)
02	0.0461 (10)	0.0290 (8)	0.0371 (8)	-0.0004 (7)	0.0064 (7)	0.0124 (6)
03	0.0422 (10)	0.0316 (8)	0.0545 (10)	-0.0107 (7)	-0.0013 (8)	-0.0119 (7)
04	0.0163 (7)	0.0739 (12)	0.0262 (7)	-0.0008 (7)	-0.0005 (6)	0.0062 (7)
05	0.0305 (8)	0.0551 (10)	0.0178 (6)	0.0020 (7)	0.0025 (5)	0.0071 (6)
N1	0.0153 (8)	0.0339 (9)	0.0172 (7)	0.0003 (7)	0.0018 (6)	0.0034 (6)
N2	0.0255 (9)	0.0226 (8)	0.0325 (9)	0.0008 (7)	0.0081 (7)	-0.0029 (7)
N3	0.0203 (8)	0.0300 (9)	0.0182 (7)	-0.0007 (7)	0.0008 (6)	-0.0010 (6)
C1	0.0205 (9)	0.0234 (9)	0.0186 (8)	0.0022 (7)	0.0016 (7)	0.0004 (7)
C2	0.0138 (9)	0.0243 (9)	0.0191 (8)	0.0035 (7)	0.0011 (6)	0.0020 (7)
C3	0.0195 (10)	0.0239 (9)	0.0245 (9)	0.0002 (8)	0.0013 (7)	-0.0004 (7)
C4	0.0238 (10)	0.0243 (9)	0.0278 (9)	0.0030 (8)	0.0062 (8)	0.0069 (8)
C5	0.0252 (10)	0.0331 (11)	0.0178 (8)	0.0079 (8)	0.0043 (7)	0.0052 (8)
C6	0.0237 (10)	0.0276 (10)	0.0197 (8)	0.0048 (8)	-0.0005 (7)	-0.0048 (7)
C7	0.0162 (9)	0.0212 (9)	0.0226 (9)	0.0026 (7)	0.0028 (7)	0.0002 (7)
C8	0.0182 (9)	0.0197 (9)	0.0191 (8)	0.0001 (7)	-0.0012 (7)	0.0003 (7)
C9	0.0162 (9)	0.0172 (9)	0.0254 (9)	0.0003 (7)	0.0029 (7)	-0.0016 (7)
C10	0.0164 (9)	0.0249 (10)	0.0236 (9)	0.0019 (7)	-0.0038 (7)	0.0003 (7)
C11	0.0217 (9)	0.0230 (9)	0.0187 (8)	0.0008 (8)	-0.0030(7)	0.0015 (7)
C12	0.0196 (9)	0.0186 (8)	0.0192 (8)	-0.0008 (7)	0.0018 (7)	-0.0015 (7)
C13	0.0153 (9)	0.0238 (9)	0.0191 (8)	-0.0009(7)	-0.0012 (6)	-0.0010(7)

Geometric parameters (Å, °)

Cl1—C9	1.7411 (18)	C4—C5	1.386 (3)
01—C1	1.216 (2)	C4—H4A	0.9500
O2—N2	1.231 (2)	C5—C6	1.383 (3)
O3—N2	1.223 (2)	C5—H5A	0.9500
O4—N3	1.223 (2)	C6—C7	1.384 (2)
O5—N3	1.2240 (19)	C6—H6A	0.9500
N1-C1	1.362 (2)	C8—C13	1.395 (2)
N1—C8	1.403 (2)	C8—C9	1.401 (2)
N1—H1A	0.8800	C9—C10	1.383 (2)
N2—C7	1.462 (2)	C10—C11	1.383 (3)
N3—C12	1.469 (2)	C10—H10A	0.9500
C1—C2	1.512 (2)	C11—C12	1.381 (3)
C2—C3	1.388 (3)	C11—H11A	0.9500
C2—C7	1.391 (3)	C12—C13	1.380 (2)
C3—C4	1.390 (2)	C13—H13A	0.9500
С3—НЗА	0.9500		

C1—N1—C8	127.64 (15)	C5—C6—C7		118.53 (17)
C1—N1—H1A	116.2	С5—С6—Н6А		120.7
C8—N1—H1A	116.2	С7—С6—Н6А		120.7
O3—N2—O2	124.13 (17)	С6—С7—С2		122.60 (17)
O3—N2—C7	118.45 (16)	C6—C7—N2		117.81 (16)
O2—N2—C7	117.41 (16)	C2—C7—N2		119.51 (16)
O4—N3—O5	123.50 (16)	С13—С8—С9		118.04 (16)
O4—N3—C12	118.03 (14)	C13—C8—N1		123.03 (16)
O5—N3—C12	118.46 (15)	C9—C8—N1		118.92 (16)
01—C1—N1	124.98 (16)	С10—С9—С8		122.07 (16)
O1—C1—C2	121.51 (16)	C10-C9-Cl1		118.51 (14)
N1—C1—C2	113.45 (15)	C8—C9—Cl1		119.41 (13)
C3—C2—C7	117.83 (16)	С11—С10—С9		119.91 (17)
C3—C2—C1	120.22 (16)	C11-C10-H10A		120.0
C7—C2—C1	121.73 (16)	C9-C10-H10A		120.0
C2—C3—C4	120.48 (17)	C12-C11-C10		117.65 (16)
С2—С3—НЗА	119.8	C12-C11-H11A		121.2
С4—С3—НЗА	119.8	C10-C11-H11A		121.2
C5—C4—C3	120.34 (17)	C13—C12—C11		123.77 (17)
C5—C4—H4A	119.8	C13-C12-N3		117.93 (16)
C3—C4—H4A	119.8	C11-C12-N3		118.28 (15)
C6—C5—C4	120.21 (16)	C12—C13—C8		118.55 (17)
С6—С5—Н5А	119.9	C12-C13-H13A		120.7
C4—C5—H5A	119.9	C8—C13—H13A		120.7
C8—N1—C1—O1	6.7 (3)	O2—N2—C7—C2		-29.6 (2)
C8—N1—C1—C2	-176.12 (17)	C1-N1-C8-C13		-7.6 (3)
O1—C1—C2—C3	110.1 (2)	C1—N1—C8—C9		171.65 (18)
N1—C1—C2—C3	-67.2 (2)	C13—C8—C9—C10		1.0 (3)
O1—C1—C2—C7	-64.4 (3)	N1-C8-C9-C10		-178.25 (16)
N1—C1—C2—C7	118.28 (19)	C13—C8—C9—Cl1		-179.73 (13)
C7—C2—C3—C4	-0.8 (3)	N1-C8-C9-Cl1		1.0 (2)
C1—C2—C3—C4	-175.50 (17)	C8—C9—C10—C11		-0.8 (3)
C2—C3—C4—C5	1.0 (3)	Cl1—C9—C10—C11		179.93 (14)
C3—C4—C5—C6	-0.3 (3)	C9—C10—C11—C12		0.5 (3)
C4—C5—C6—C7	-0.5 (3)	C10-C11-C12-C13		-0.4 (3)
C5—C6—C7—C2	0.8 (3)	C10-C11-C12-N3		178.34 (16)
C5—C6—C7—N2	-175.84 (17)	O4—N3—C12—C13		-2.9 (2)
C3—C2—C7—C6	-0.1 (3)	O5—N3—C12—C13		177.27 (17)
C1—C2—C7—C6	174.54 (17)	O4—N3—C12—C11		178.28 (18)
C3—C2—C7—N2	176.43 (16)	O5—N3—C12—C11		-1.6 (3)
C1—C2—C7—N2	-8.9 (3)	C11—C12—C13—C8		0.7 (3)
O3—N2—C7—C6	-31.8 (2)	N3—C12—C13—C8		-178.11 (15)
O2—N2—C7—C6	147.07 (18)	C9—C8—C13—C12		-0.9 (2)
O3—N2—C7—C2	151.52 (17)	N1—C8—C13—C12		178.32 (16)
Hydrogen-bond geometry (Å, °)				
D—H···A	<i>D</i> —Н	H···A	$D \cdots A$	D—H···A
C13—H13A…O1	0.95	2.24	2.848 (2)	121

supplementary materials

C10—H10A···O4 ⁱ	0.95	2.35	3.246 (2)	157	
C11—H11A···O2 ⁱⁱ	0.95	2.55	3.202 (2)	126	
Symmetry codes: (i) $x+1$, y , z ; (ii) $x+1/2$, $-y+1/2$, $-z+1$.					



Fig. 1

Fig. 2



addenda and errata

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N-(2-Chloro-4-nitrophenyl)-2-nitrobenzamide. Corrigendum

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The title and the chemical diagram of the paper by Saeed, Hussain & Flörke [*Acta Cryst.* (2008), E64, o705] are corrected.

In the paper by Saeed, Hussain & Flörke [Acta Cryst. (2008), E64, o705], the title and the chemical diagram are incorrect. The correct structure is shown below and the correct title of the original paper should be 'N-(2-Chloro-5-nitrophenyl)-2-nitrobenzamide'.

