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2-Chloro-*N*-(3-nitrophenyl)acetamide

B. Thimme Gowda,[a]* Sabine Foro[b] and Hartmut Fuess[b]

[a]Department of Chemistry, Mangalore University, Mangalagangotri 574 199, Mangalore, India, and [b]Institute of Materials Science, Darmstadt University of Technology, Petersenstrasse 23, D-64287 Darmstadt, Germany
Correspondence e-mail: gowdabt@yahoo.com

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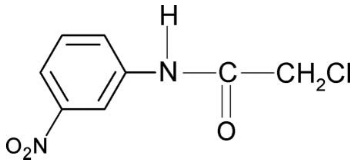
Key indicators: single-crystal X-ray study; T = 299 K; mean σ(C–C) = 0.010 Å; R factor = 0.065; wR factor = 0.188; data-to-parameter ratio = 6.3.

The conformation of the N—H bond in the structure of the title compound, $C_8H_7ClN_2O_3$, is *anti* to the *meta*-nitro group, in contrast with the *syn* conformation observed with respect to the *ortho*-nitro substituent in 2-chloro-*N*-(2-nitrophenyl)-acetamide. The geometric parameters of the title compound are similar to those of 2-chloro-*N*-(4-nitrophenyl)acetamide and other acetanilides. Intermolecular N—H···O hydrogen bonds link the molecules into zigzag chains running along the *a* and *b* axes.

Related literature

For related literature, see: Gowda & Weiss (1994); Gowda *et al.* (2007, 2007*a*, 2007*b*, 2007*c*).



Experimental

Crystal data

$C_8H_7ClN_2O_3$
M_r = 214.61
Tetragonal, $P4_3$
a = 4.999 (2) Å
c = 35.876 (6) Å
V = 896.5 (5) Å^3

Z = 4
Cu $K\alpha$ radiation
μ = 3.67 mm^{-1}
T = 299 (2) K
0.60 × 0.60 × 0.45 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min}$ = 0.220, $T_{\max}$ = 0.276 (expected range = 0.153–0.192)
1784 measured reflections

810 independent reflections
686 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.162
3 standard reflections
frequency: 120 min
intensity decay: 1.0%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.066
$wR(F^2)$ = 0.188
S = 1.13
810 reflections
128 parameters
1 restraint

H-atom parameters constrained
$\Delta\rho_{\max}$ = 0.31 e Å^{-3}
$\Delta\rho_{\min}$ = −0.40 e Å^{-3}
Absolute structure: Flack (1983), with no Friedel pairs
Flack parameter: 0.03 (4)

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

D—H···A	D—H	H···A	D···A	D—H···A
N5—H5N···O4$^{\rm i}$	0.86	2.14	2.904 (7)	147

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

Data collection: *CAD-4-PC Software* (Enraf–Nonius, 1996); cell refinement: *CAD-4-PC Software*; data reduction: *REDU4* (Stoe & Cie, 1987); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); 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: BT2406).

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supplementary materials

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2-Chloro-*N*-(3-nitrophenyl)acetamide

B. T. Gowda, S. Foro and H. Fuess

Comment

The amide moiety is an important constituent of many biologically significant compounds. The structural studies of amides are therefore of interest. As part of a study of the effect of ring and side chain substitutions on the solid state structures of this class of compounds (Gowda *et al.*, 2007*a*,*b*,*c*,*d*), the crystal structure of *N*-(3-nitrophenyl)-2-chloroacetamide has been determined to explore the effects of polar substituent groups on the structures of N-aromatic amides. The conformation of the N—H bond (Fig. 1) is *anti* to the *meta* nitro group, in contrast to the *syn* conformation observed with respect to *ortho* nitro substituent in *N*-(2-nitrophenyl)-2-chloroacetamide (Gowda *et al.*, 2007*a*). The geometric parameters of are similar to those of *N*-(2-nitrophenyl)-2-chloroacetamide (Gowda *et al.*, 2007*a*), *N*-(4-nitrophenyl)-2-chloroacetamide (Gowda *et al.*, 2007*c*) and other acetanilides (Gowda *et al.*, 2007*b*, Gowda *et al.*, 2007). The molecular skeleton is essentially planar. Intermolecular N—H···O hydrogen bonds (Table 1) link the molecules into zigzag chains running along the *a* and *b* axis (Fig. 2).

Experimental

The title compound was prepared according to the literature method (Gowda & Weiss, 1994). The purity of the compound was checked by determining its melting point. It was characterized by recording its infrared and NQR spectra (Gowda & Weiss, 1994). Single crystals of the title compound were obtained from an ethanolic solution and used for X-ray diffraction studies at room temperature.

Refinement

The H atoms were positioned with idealized geometry using a riding model with N—H = 0.86 Å and C—H = 0.93–0.97 Å]. U_{iso}(H) values were set equal to 1.2U_{eq} of the parent atom.

Figures

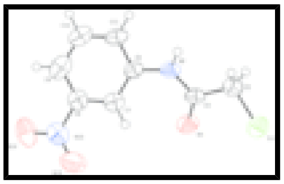

Fig. 1. Molecular structure of the title compound showing the atom labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small spheres of arbitrary radii.

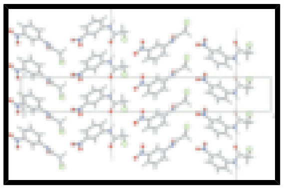

Fig. 2. Molecular packing of (I) with hydrogen bonding shown as dashed lines.

2-Chloro-*N*-(3-nitrophenyl)acetamide

Crystal data

$C_8H_7ClN_2O_3$
$M_r = 214.61$
Tetragonal, $P4_3$
Hall symbol: P 4cw
$a = 4.999$ (2) Å
$b = 4.999$ (2) Å
$c = 35.876$ (6) Å
$\alpha = 90°$
$\beta = 90°$
$\gamma = 90°$
$V = 896.5$ (5) Å^3

$Z = 4$
$F_{000} = 440$
$D_x = 1.590$ Mg m^{-3}
Cu *K*α radiation
$\lambda = 1.54180$ Å
Cell parameters from 25 reflections
$\theta = 9.0–25.8°$
$\mu = 3.67$ mm^{-1}
$T = 299$ (2) K
Prism, dark orange
0.60 × 0.60 × 0.45 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Monochromator: graphite
$T = 299(2)$ K
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.220$, $T_{max} = 0.276$
1784 measured reflections
810 independent reflections
686 reflections with $I > 2\sigma(I)$

$R_{int} = 0.162$
$\theta_{max} = 67.1°$
$\theta_{min} = 4.9°$
$h = 0 \rightarrow 5$
$k = -5 \rightarrow 5$
$l = -42 \rightarrow 0$
3 standard reflections
every 120 min
intensity decay: 1.0%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.066$
$wR(F^2) = 0.188$
$S = 1.13$
810 reflections
128 parameters
1 restraint
Primary atom site location: structure-invariant direct methods

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.078P)^2 + 0.1695P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.31$ e Å^{-3}
$\Delta\rho_{min} = -0.40$ e Å^{-3}
Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.022 (5)
Absolute structure: Flack (1983), with no Friedel pairs

Secondary atom site location: difference Fourier map Flack parameter: 0.03 (4)

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 $F^2 > 2\text{sigma}(F^2)$ 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}
C2	−0.3115 (17)	0.3054 (16)	0.15620 (18)	0.058 (2)
H2B	−0.3817	0.4858	0.1543	0.070*
H2A	−0.1869	0.3003	0.1769	0.070*
C3	−0.1668 (14)	0.2336 (13)	0.12061 (17)	0.0444 (16)
C6	0.1238 (13)	0.4335 (12)	0.07257 (17)	0.0414 (14)
C7	0.0760 (14)	0.2529 (14)	0.04397 (18)	0.0487 (16)
H7	−0.0619	0.1287	0.0457	0.058*
C8	0.2381 (14)	0.2624 (15)	0.01301 (17)	0.0484 (16)
C9	0.4455 (15)	0.4393 (16)	0.0093 (3)	0.062 (2)
H9	0.5530	0.4392	−0.0118	0.074*
C10	0.4895 (17)	0.6160 (16)	0.0377 (2)	0.061 (2)
H10	0.6290	0.7382	0.0358	0.073*
C11	0.3318 (14)	0.6165 (12)	0.0690 (2)	0.0505 (16)
H11	0.3641	0.7397	0.0879	0.061*
Cl1	−0.5750 (3)	0.0811 (4)	0.16453 (4)	0.0687 (8)
N5	−0.0402 (12)	0.4416 (10)	0.10456 (15)	0.0455 (14)
H5N	−0.0616	0.5954	0.1149	0.055*
N12	0.1788 (14)	0.0720 (13)	−0.01703 (17)	0.0581 (16)
O4	−0.1610 (12)	0.0095 (10)	0.10761 (15)	0.0587 (14)
O13	−0.0164 (16)	−0.0715 (14)	−0.01436 (19)	0.083 (2)
O14	0.3341 (15)	0.0670 (15)	−0.04350 (16)	0.084 (2)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C2	0.065 (5)	0.049 (4)	0.061 (4)	−0.003 (4)	0.008 (3)	−0.008 (3)
C3	0.045 (4)	0.031 (3)	0.058 (3)	−0.001 (2)	−0.003 (3)	−0.007 (3)
C6	0.039 (3)	0.030 (3)	0.055 (3)	0.003 (2)	0.000 (3)	0.002 (3)
C7	0.043 (4)	0.043 (3)	0.060 (3)	0.006 (3)	0.000 (3)	−0.001 (3)
C8	0.047 (4)	0.044 (3)	0.054 (3)	0.012 (3)	−0.001 (3)	0.000 (3)
C9	0.040 (4)	0.062 (5)	0.082 (4)	0.012 (4)	0.008 (3)	0.021 (4)

C10	0.049 (4)	0.048 (4)	0.086 (5)	−0.003 (4)	0.002 (4)	0.010 (4)
C11	0.045 (4)	0.027 (3)	0.079 (4)	0.002 (3)	−0.006 (3)	−0.001 (3)
Cl1	0.0534 (13)	0.0763 (15)	0.0765 (11)	−0.0104 (8)	0.0105 (8)	−0.0069 (9)
N5	0.053 (3)	0.028 (3)	0.055 (3)	−0.001 (2)	0.001 (3)	−0.008 (2)
N12	0.061 (4)	0.054 (4)	0.059 (3)	0.019 (3)	−0.006 (3)	−0.002 (3)
O4	0.067 (3)	0.035 (3)	0.074 (3)	0.000 (2)	0.011 (3)	−0.004 (2)
O13	0.084 (4)	0.077 (5)	0.086 (4)	−0.012 (4)	−0.007 (4)	−0.022 (3)
O14	0.083 (4)	0.103 (5)	0.067 (3)	0.029 (4)	0.022 (3)	−0.014 (3)

Geometric parameters (Å, °)

C2—C3	1.510 (9)	C8—C9	1.369 (11)
C2—Cl1	1.756 (8)	C8—N12	1.468 (10)
C2—H2B	0.9700	C9—C10	1.365 (13)
C2—H2A	0.9700	C9—H9	0.9300
C3—O4	1.214 (8)	C10—C11	1.372 (12)
C3—N5	1.347 (9)	C10—H10	0.9300
C6—C7	1.388 (9)	C11—H11	0.9300
C6—C11	1.391 (9)	N5—H5N	0.8600
C6—N5	1.411 (9)	N12—O13	1.215 (10)
C7—C8	1.376 (9)	N12—O14	1.227 (9)
C7—H7	0.9300		

C3—C2—Cl1	110.6 (5)	C7—C8—N12	116.8 (7)
C3—C2—H2B	109.5	C10—C9—C8	117.9 (8)
Cl1—C2—H2B	109.5	C10—C9—H9	121.1
C3—C2—H2A	109.5	C8—C9—H9	121.1
Cl1—C2—H2A	109.5	C9—C10—C11	121.3 (8)
H2B—C2—H2A	108.1	C9—C10—H10	119.4
O4—C3—N5	122.5 (6)	C11—C10—H10	119.4
O4—C3—C2	123.7 (6)	C10—C11—C6	120.3 (7)
N5—C3—C2	113.8 (6)	C10—C11—H11	119.9
C7—C6—C11	119.2 (6)	C6—C11—H11	119.9
C7—C6—N5	121.3 (6)	C3—N5—C6	126.8 (5)
C11—C6—N5	119.4 (6)	C3—N5—H5N	116.6
C8—C7—C6	118.2 (6)	C6—N5—H5N	116.6
C8—C7—H7	120.9	O13—N12—O14	123.9 (7)
C6—C7—H7	120.9	O13—N12—C8	119.1 (6)
C9—C8—C7	123.1 (7)	O14—N12—C8	117.0 (7)
C9—C8—N12	120.0 (7)		

Cl1—C2—C3—O4	−25.7 (9)	C7—C6—C11—C10	−0.7 (10)
Cl1—C2—C3—N5	155.1 (5)	N5—C6—C11—C10	−178.4 (6)
C11—C6—C7—C8	0.1 (9)	O4—C3—N5—C6	−3.3 (11)
N5—C6—C7—C8	177.7 (6)	C2—C3—N5—C6	175.9 (7)
C6—C7—C8—C9	0.8 (10)	C7—C6—N5—C3	32.2 (10)
C6—C7—C8—N12	−178.9 (6)	C11—C6—N5—C3	−150.1 (6)
C7—C8—C9—C10	−0.9 (11)	C9—C8—N12—O13	−175.2 (8)
N12—C8—C9—C10	178.8 (7)	C7—C8—N12—O13	4.4 (10)
C8—C9—C10—C11	0.1 (11)	C9—C8—N12—O14	5.4 (9)
C9—C10—C11—C6	0.6 (11)	C7—C8—N12—O14	−175.0 (6)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N5—H5N···O4^{i}	0.86	2.14	2.904 (7)	147

Symmetry codes: (i) x, y+1, z.

Fig. 1

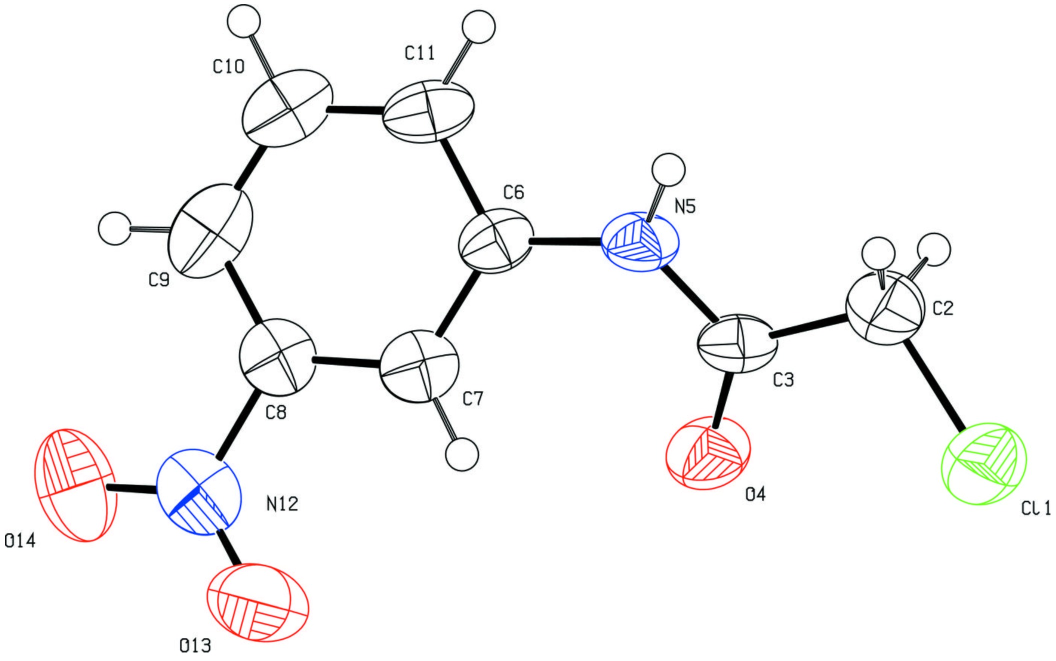

Fig. 2