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N-(4-Chlorophenyl)acetamide: a redetermination

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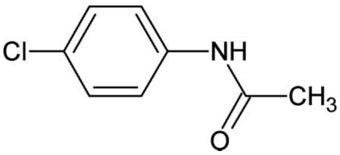
Key indicators: single-crystal X-ray study; T = 299 K; mean σ(C–C) = 0.004 Å; R factor = 0.030; wR factor = 0.084; data-to-parameter ratio = 7.6.

The structure of the title compound, C_8H_8ClNO, has already been reported [Subramanian (1966). *Z. Kristallogr.* **123**, 222–234], but the positions of the H atoms have not been determined. It has now been refined with new intensity data to significantly higher precision and with all H atoms. The structure is closely related to *N*-(2-chlorophenyl)acetamide, *N*-(3-chlorophenyl)acetamide and other related amides, with similar bond parameters. The molecules are linked into chains through N—H···O hydrogen bonds.

Related literature

For related literature, see: Gowda *et al.* (2000, 2003); Gowda, Foro & Fuess (2007); Gowda, Kozisek, Svoboda & Fuess (2007); Gowda, Kožíšek, Tokarčík & Fuess (2007); Gowda, Svoboda & Fuess (2007).



Experimental

Crystal data

C_8H_8ClNO
M_r = 169.60
Orthorhombic, $Pna2_1$
a = 9.7163 (7) Å
b = 12.8528 (9) Å
c = 6.5282 (6) Å
V = 815.25 (11) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 3.65 mm^{-1}
T = 299 (2) K
0.33 × 0.10 × 0.08 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.359, T_{max} = 0.776
1260 measured reflections
793 independent reflections
706 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
3 standard reflections
frequency: 120 min
intensity decay: 1.0%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.084
S = 1.04
793 reflections
105 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}
Absolute structure: Flack (1983), no Friedel pairs
Flack parameter: −0.02 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4N···O3^{i}	0.87 (3)	1.98 (3)	2.853 (3)	179 (3)

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

Data collection: *CAD-4-PC* (Enraf–Nonius, 1996); cell refinement: *CAD-4-PC*; 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: BT2422).

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supplementary materials

Acta Cryst. (2007). E**63**, o3392 [**doi:10.1107/S1600536807031893**]

N-(4-Chlorophenyl)acetamide: a redetermination

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

Comment

The structure of *N*-(4-chlorophenyl)-acetamide has been refined as part of a study on the systematization of the crystal structures of N-aromatic amides (Gowda *et al.*, 2000; Gowda, Foro & Fuess, 2007; Gowda, Kozisek, Svoboda & Fuess, 2007; Gowda, Kožíšek, Tokarčík & Fuess, 2007; Gowda, Svoboda & Fuess, 2007). The purpose is to have accurate values of the bond parameters for comparison with the other structures which have recently been determined. The structure of the title compound (Fig. 1) is closely related to *N*-(2-chlorophenyl)- acetamide, *N*-(3-chlorophenyl)-acetamide and other related amides, with similar bond parameters (Gowda *et al.*, 2000; Gowda, Foro & Fuess, 2007; Gowda, Kozisek, Svoboda & Fuess, 2007; Gowda, Kožíšek, Tokarčík & Fuess, 2007; Gowda, Svoboda & Fuess, 2007). The molecules are linked into chains through N—H···O hydrogen bonds (Fig. 2 & Table 1).

Experimental

The title compound was prepared according to the literature method (Gowda *et al.*, 2003). The purity of the compound was checked by determining its melting point. It was characterized by recording its infrared and NMR spectra (Gowda *et al.*, 2003). Single crystals of the title compound were obtained from an ethanolic solution.

Refinement

The H atom of the NH group was located in a diffrerence map and its coordinates have been refined with U_{iso}(H) = 1.2 U_{eq}(N). The carbon-bound H atoms were positioned with idealized geometry and refined using a riding model, with C—H distances in the range 0.93–0.96 Å, with U_{iso}(H) = 1.2 U_{eq}(C).

Figures

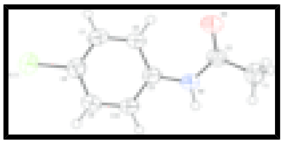

Fig. 1. Molecular structure of the title compound showing the atom labeling scheme. The displacement ellipsoids are drawn at the 50% probability level.

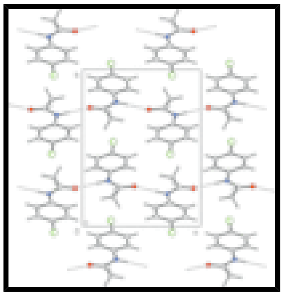

Fig. 2. Molecular packing of the title compound with hydrogen bonding shown as dashed lines.

N-(4-Chlorophenyl)acetamide

Crystal data

C_8H_8ClNO

$M_r = 169.60$

Orthorhombic, $Pna2_1$

Hall symbol: P 2c -2n

$a = 9.7163\ (7)$ Å

$b = 12.8528\ (9)$ Å

$c = 6.5282\ (6)$ Å

$V = 815.25\ (11)$ Å^3

$Z = 4$

$F_{000} = 352$

$D_x = 1.382$ Mg m^{-3}

Cu $K\alpha$ radiation
$\lambda = 1.54180$ Å

Cell parameters from 25 reflections

$\theta = 8.9$–25.3°

$\mu = 3.65$ mm^{-1}

$T = 299\ (2)$ K

Rod, colourless

0.33 × 0.10 × 0.08 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: Psi-scan
(North *et al.*, 1968)

$T_{min} = 0.359$, $T_{max} = 0.776$

1260 measured reflections

793 independent reflections

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

$R_{int} = 0.027$

$\theta_{max} = 66.8$°

$\theta_{min} = 5.7$°

$h = 0 \rightarrow 11$

$k = -7 \rightarrow 15$

$l = -7 \rightarrow 0$

3 standard reflections

every 120 min

intensity decay: 1.0%

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.030$

$wR(F^2) = 0.084$

$S = 1.04$

793 reflections

105 parameters

1 restraint

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of
independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0574P)^2 + 0.0284P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.006$

$\Delta\rho_{max} = 0.17$ e Å^{-3}

$\Delta\rho_{min} = -0.23$ e Å^{-3}

Extinction correction: SHELXL97,
$Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0070 (10)

Absolute structure: Flack (1983), no Friedel pairs

Flack parameter: −0.02 (2)

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}
Cl1	0.25877 (7)	0.53871 (6)	0.69773 (15)	0.0595 (3)
O3	0.43707 (19)	0.2559 (2)	−0.1534 (4)	0.0730 (8)
N4	0.2176 (2)	0.29176 (19)	−0.0645 (5)	0.0432 (5)
H4N	0.132 (3)	0.277 (2)	−0.092 (6)	0.052*
C1	0.2594 (3)	0.1900 (3)	−0.3668 (6)	0.0545 (8)
H1A	0.1632	0.1758	−0.3474	0.065*
H1B	0.3085	0.1256	−0.3811	0.065*
H1C	0.2715	0.2311	−0.4883	0.065*
C2	0.3137 (3)	0.2483 (2)	−0.1857 (5)	0.0460 (7)
C5	0.2355 (2)	0.3493 (2)	0.1167 (4)	0.0396 (6)
C6	0.3619 (2)	0.3811 (2)	0.1933 (5)	0.0468 (7)
H6	0.4424	0.3643	0.1237	0.056*
C7	0.3682 (2)	0.4379 (2)	0.3731 (5)	0.0504 (8)
H7	0.4532	0.4580	0.4255	0.061*
C8	0.2495 (2)	0.4648 (2)	0.4747 (6)	0.0448 (7)
C9	0.1231 (2)	0.4346 (2)	0.4013 (5)	0.0495 (7)
H9	0.0431	0.4525	0.4710	0.059*
C10	0.1167 (2)	0.3772 (2)	0.2229 (5)	0.0471 (7)
H10	0.0313	0.3567	0.1725	0.057*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0594 (4)	0.0668 (5)	0.0524 (4)	−0.0019 (3)	−0.0033 (5)	−0.0122 (4)
O3	0.0373 (11)	0.1145 (18)	0.0672 (16)	0.0193 (11)	−0.0051 (11)	−0.0167 (15)
N4	0.0293 (10)	0.0544 (12)	0.0459 (13)	0.0007 (9)	−0.0018 (10)	−0.0021 (11)
C1	0.0610 (17)	0.0520 (15)	0.0506 (18)	0.0043 (12)	−0.0012 (14)	−0.0032 (15)
C2	0.0379 (13)	0.0543 (14)	0.0458 (16)	0.0066 (13)	0.0005 (14)	0.0073 (14)
C5	0.0329 (11)	0.0440 (13)	0.0419 (14)	−0.0015 (10)	−0.0015 (11)	0.0052 (12)
C6	0.0302 (11)	0.0603 (16)	0.0499 (16)	−0.0028 (9)	0.0021 (15)	−0.0046 (16)
C7	0.0323 (11)	0.0624 (16)	0.057 (2)	−0.0061 (11)	−0.0061 (13)	−0.0020 (16)
C8	0.0456 (15)	0.0441 (14)	0.0448 (16)	0.0003 (10)	−0.0019 (12)	0.0032 (13)

C9	0.0345 (11)	0.0619 (16)	0.0521 (17)	0.0016 (11)	0.0060 (13)	−0.0049 (15)
C10	0.0293 (11)	0.0583 (15)	0.0536 (19)	−0.0001 (10)	−0.0020 (11)	−0.0043 (15)

Geometric parameters (Å, °)

Cl11—C8	1.741 (4)	C5—C10	1.393 (3)
O3—C2	1.221 (3)	C6—C7	1.384 (5)
N4—C2	1.345 (4)	C6—H6	0.9300
N4—C5	1.406 (4)	C7—C8	1.375 (4)
N4—H4N	0.87 (3)	C7—H7	0.9300
C1—C2	1.496 (5)	C8—C9	1.374 (4)
C1—H1A	0.9600	C9—C10	1.380 (5)
C1—H1B	0.9600	C9—H9	0.9300
C1—H1C	0.9600	C10—H10	0.9300
C5—C6	1.389 (3)		
C2—N4—C5	128.9 (2)	C7—C6—C5	120.0 (2)
C2—N4—H4N	117 (2)	C7—C6—H6	120.0
C5—N4—H4N	114 (3)	C5—C6—H6	120.0
C2—C1—H1A	109.5	C8—C7—C6	120.3 (2)
C2—C1—H1B	109.5	C8—C7—H7	119.8
H1A—C1—H1B	109.5	C6—C7—H7	119.8
C2—C1—H1C	109.5	C9—C8—C7	120.7 (3)
H1A—C1—H1C	109.5	C9—C8—Cl11	119.5 (2)
H1B—C1—H1C	109.5	C7—C8—Cl11	119.8 (2)
O3—C2—N4	123.1 (3)	C8—C9—C10	119.1 (3)
O3—C2—C1	121.5 (3)	C8—C9—H9	120.5
N4—C2—C1	115.3 (2)	C10—C9—H9	120.5
C6—C5—C10	118.6 (3)	C9—C10—C5	121.3 (2)
C6—C5—N4	124.6 (2)	C9—C10—H10	119.3
C10—C5—N4	116.9 (2)	C5—C10—H10	119.3
C5—N4—C2—O3	−1.7 (5)	C6—C7—C8—C9	1.0 (5)
C5—N4—C2—C1	178.8 (3)	C6—C7—C8—Cl11	−178.6 (2)
C2—N4—C5—C6	7.1 (5)	C7—C8—C9—C10	−0.5 (5)
C2—N4—C5—C10	−174.1 (3)	Cl11—C8—C9—C10	179.2 (2)
C10—C5—C6—C7	1.0 (4)	C8—C9—C10—C5	0.2 (5)
N4—C5—C6—C7	179.7 (3)	C6—C5—C10—C9	−0.5 (4)
C5—C6—C7—C8	−1.3 (5)	N4—C5—C10—C9	−179.3 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H4N···O3^{i}	0.87 (3)	1.98 (3)	2.853 (3)	179 (3)

Symmetry codes: (i) $x-1/2, -y+1/2, z$.

Fig. 1

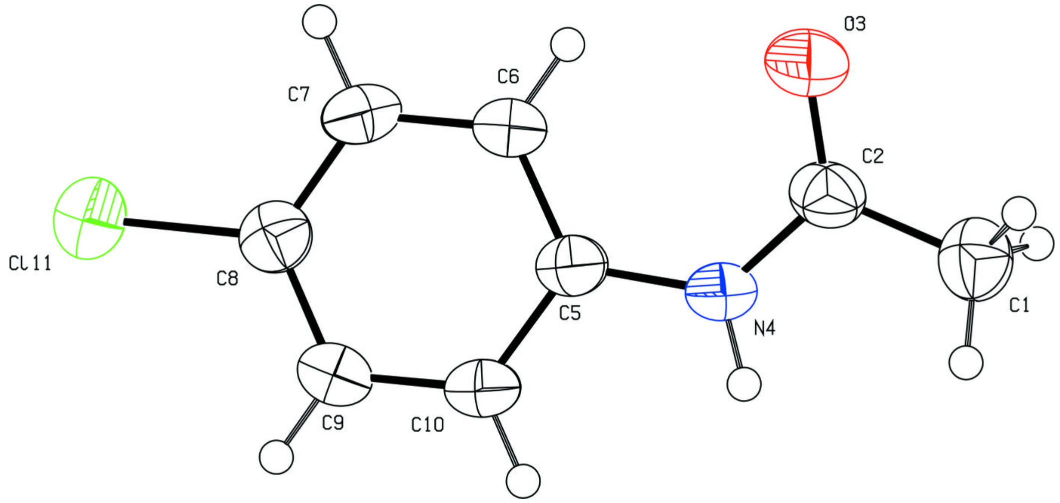

Fig. 2