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ISSN 2414-3146

2-Chloro-*N*-(4-hydroxyphenyl)acetamide

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Received 14 October 2024
Accepted 17 October 2024

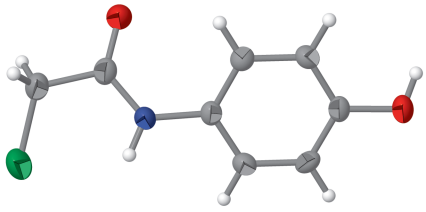
Keywords: crystal structure; acetamide; hydrogen-bonding.

CCDC reference: 2392239

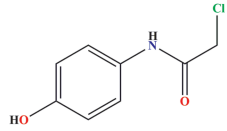
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_8H_8ClNO_2$, is significantly distorted from planarity, with a twist angle between the planes through the hydroxybenzene and acetamide groups being 23.5 (2)°. This conformation is supported by intramolecular C—H···O and N—H···Cl contacts. In the crystal, N—H···O hydrogen-bonding contacts between acetamide groups and O—H···O contacts between hydroxyl groups form tapes propagating parallel to [$\bar{1}$03].

3D view



Chemical scheme



Structure description

N-arylacetamides are intermediates for the synthesis of medicinal, agrochemical and pharmaceutical compounds (Missioui *et al.*, 2021). As part of our ongoing studies of these systems (Missioui *et al.*, 2022), we now describe the synthesis and structure of the title compound, $C_8H_8ClNO_2$.

The molecule, Fig. 1, is almost planar as indicated by a twist angle between the planes through the hydroxybenzene (C1–C6, O1) and acetamide (C7, C8, N1, O2) groups being 23.5 (2)°; the acetamide group has an *anti* conformation. The chloro substituent deviates only slightly from the plane of the acetamide group as indicated by the N1—C7—C8—Cl1 torsion angle of 15.4 (4)°.

Two types of close intramolecular contacts occur within the molecule. The first contact is of the type C—H···O with a C3—H3···O2 angle of 116° and a C3···O2 distance of 2.873 (4) Å, Table 1. Similar contacts are observed in related structures including 2-chloro-*N*-(4-fluorophenyl)acetamide (Kang *et al.*, 2008), 2-chloro-*N*-phenylacetamide (Gowda *et al.*, 2008) and 2-chloro-*N*-(4-chlorophenyl)acetamide (Gowda *et al.*, 2007). The second contact is of the type N—H···Cl and has a N1—H1···Cl1 angle of 115° and a N1···Cl1 distance of 2.999 (2) Å.

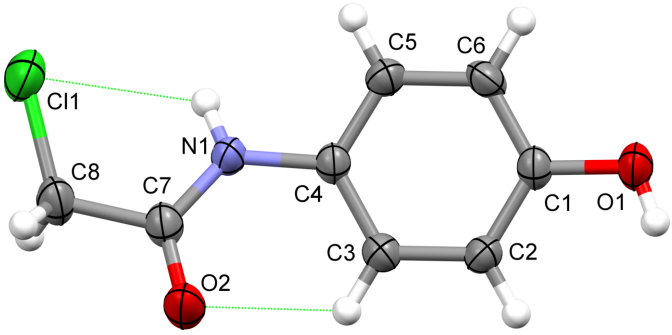


Figure 1
The molecule of 2-chloro-*N*-(4-hydroxyphenyl)acetamide showing the atom-numbering scheme and displacement parameters at the 50% probability level. The intramolecular C—H···O and N—H···Cl contacts are shown as green dotted lines.

In the crystal, neighbouring molecules are linked by N—H···O hydrogen-bonding between translationally related acetamide groups with a N1—H1···O2^{i} [symmetry code: (i) x, $y-1$, z] angle of 145° and a N1···O2^{i} distance of 3.025 (3) Å, Table 1, to form linear chains parallel to the b axis (Fig. 2). The molecules are also bridged by O—H···O contacts with a O1—H1A···O1^{ii} [symmetry code: (ii) $-x+2$, $y+\frac{1}{2}$, $-z+2$] angle of 166° and an O1···O1^{ii} distance of 2.8585 (17) Å which, by themselves assemble molecules along the 2_1 screw axis in the b-axis direction. The combined hydrogen-bonding interactions result in almost flat tapes of molecules parallel to [$\bar{1}$03].

Synthesis and crystallization

4-Aminophenol (1 mmol) was dissolved in pure acetic acid (30 ml) and placed in an ice-bath. Subsequently, chloroacetyl chloride (1.2 mmol) was added portion-wise under stirring. At the end of the reaction, a solution of sodium acetate (25 ml) was added, and a solid precipitate formed after 30 min of stirring at room temperature. The resulting solid was filtered, washed with cold water, dried and recrystallized from its ethanol solution to yield the title compound as colourless crystals.

Yield = 89%, colour:colourless, m.p. = 413–415 K. FT–IR (ATR, ν, cm^{-1}): 3385 (OH), 3200 (NH), 1640 (C═O). ^{1}H NMR (500 MHz, DMSO-d$_6$): δ p.p.m. 4.21 (*s*, 2H, CH$_2$), 6.76–7.34 (*m*, 4H, Ar—H), 9.20 (*s*, 1H, OH), 10.23 (*s*, 1H, NH). ^{13}C NMR (500 MHz, DMSO-d_6): 43.42 (**C**H$_2$); 117.68, 122.20, 131.50, 132.63, 153.68 (C—Ar); 164.76 (**C**═O). HRMS (ESI): calculated for $C_8H_8ClNO_2$ [M - H]$^{+}$ 186.0224, found 186.0328.

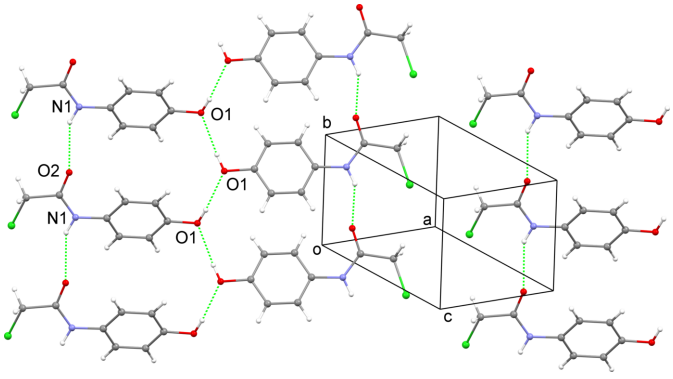


Figure 2
A segment of the packing in the crystal of 2-chloro-*N*-(4-hydroxyphenyl) acetamide showing the intermolecular N—H···O and O—H···O hydrogen bonds as green dotted lines.

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···O2	0.93	2.34	2.873 (4)	116
N1—H1···Cl1	0.86	2.53	2.999 (2)	115
N1—H1···O2^{i}	0.86	2.28	3.025 (3)	145
O1—H1A···O1^{ii}	0.82	2.06	2.8585 (17)	166

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_8H_8ClNO_2$
M_r	185.60
Crystal system, space group	Monoclinic, $P2_1$
Temperature (K)	296
a, b, c (Å)	6.5088 (6), 5.1758 (5), 12.2175 (14)
β (°)	101.649 (10)
V (Å^3)	403.11 (7)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.43
Crystal size (mm)	0.45 × 0.20 × 0.07
Data collection	
Diffractometer	SuperNova, Dual, Cu at home/near, Atlas
Absorption correction	Gaussian (*CrysAlis PRO*; Rigaku OD, 2023)
T_{min}, T_{max}	0.642, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	3642, 1902, 1487
R_{int}	0.028
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.697
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.039, 0.081, 1.06
No. of reflections	1902
No. of parameters	110
No. of restraints	1
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.16, −0.19
Absolute structure	Flack x determined using 510 quotients [$(I^+)-(I^-)$]/[$(I^+)+(I^-)$] (Parsons *et al.*, 2013)
Absolute structure parameter	0.11 (5)

Computer programs: *CrysAlis PRO* (Rigaku OD, 2023), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2019/2* (Sheldrick, 2015*b*), *Mercury* (Macrae *et al.*, 2020) and *WinGX* (Farrugia, 2012).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

YR is thankful to the National Center for Scientific and Technical Research of Morocco (CNRST) for its continuous support. The contributions of the authors are as follows: conceptualization, YR; methodology, AA; investigation, AEMAA and IAEH; writing (original draft), AEMAA; writing (review and editing of the manuscript), YR; formal analysis, YR and BMK; supervision, YR; crystal structure determination, BMK.

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full crystallographic data

IUCrData (2024). **9**, x241015 [https://doi.org/10.1107/S2414314624010150]

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2-Chloro-*N*-(4-hydroxyphenyl)acetamide

Crystal data

$C_8H_8ClNO_2$
M_r = 185.60
Monoclinic, $P2_1$
a = 6.5088 (6) Å
b = 5.1758 (5) Å
c = 12.2175 (14) Å
β = 101.649 (10)°
V = 403.11 (7) Å^3
Z = 2
$F(000)$ = 192
D_x = 1.529 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1576 reflections
θ = 3.9–28.2°
μ = 0.43 mm^{-1}
T = 296 K
Plate, colourless
0.45 × 0.20 × 0.07 mm

Data collection

SuperNova, Dual, Cu at home/near, Atlas diffractometer
ω scans
Absorption correction: gaussian (CrysAlis Pro; Rigaku OD, 2023)
T_{min} = 0.642, T_{max} = 1.000
3642 measured reflections
1902 independent reflections
1487 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 29.7°, θ_{min} = 3.3°
h = −8→8
k = −7→7
l = −16→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.081
S = 1.06
1902 reflections
110 parameters
1 restraint
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0294P)^2 + 0.0021P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Absolute structure: Flack x determined using 510 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: 0.11 (5)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.7301 (4)	0.3699 (6)	0.8980 (2)	0.0311 (7)
C2	0.5982 (4)	0.5654 (6)	0.9179 (3)	0.0334 (7)
H2	0.645995	0.688449	0.972562	0.040*
C3	0.3940 (5)	0.5786 (6)	0.8563 (3)	0.0337 (7)
H3	0.305127	0.710757	0.869335	0.040*
C4	0.3236 (4)	0.3932 (6)	0.7754 (2)	0.0290 (7)
C5	0.4562 (5)	0.1964 (6)	0.7575 (3)	0.0335 (8)
H5	0.408280	0.070786	0.703994	0.040*
C6	0.6593 (5)	0.1844 (6)	0.8184 (3)	0.0348 (8)
H6	0.747984	0.051642	0.805778	0.042*
C7	−0.0065 (5)	0.6041 (7)	0.6835 (3)	0.0349 (7)
C8	−0.2181 (5)	0.5695 (7)	0.6051 (3)	0.0442 (9)
H8A	−0.238963	0.713348	0.553173	0.053*
H8B	−0.326264	0.580067	0.649112	0.053*
N1	0.1164 (3)	0.3959 (5)	0.7083 (2)	0.0331 (6)
H1	0.065971	0.250333	0.681401	0.040*
O1	0.9342 (3)	0.3507 (4)	0.9570 (2)	0.0433 (6)
H1A	0.967245	0.484830	0.991810	0.065*
O2	0.0363 (3)	0.8215 (4)	0.7186 (2)	0.0500 (6)
Cl1	−0.25580 (12)	0.27889 (19)	0.52655 (7)	0.0555 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0250 (14)	0.0296 (17)	0.0360 (17)	−0.0017 (13)	−0.0002 (12)	0.0054 (14)
C2	0.0334 (17)	0.0289 (17)	0.0344 (17)	−0.0011 (14)	−0.0014 (13)	−0.0042 (14)
C3	0.0304 (16)	0.0303 (17)	0.0383 (18)	0.0034 (14)	0.0022 (13)	−0.0025 (14)
C4	0.0251 (15)	0.0268 (15)	0.0329 (16)	−0.0010 (13)	0.0006 (12)	0.0036 (13)
C5	0.0326 (16)	0.0266 (17)	0.0386 (18)	−0.0022 (13)	0.0007 (13)	−0.0037 (13)
C6	0.0293 (15)	0.0254 (16)	0.050 (2)	0.0063 (12)	0.0079 (14)	−0.0008 (14)
C7	0.0279 (16)	0.0337 (18)	0.0412 (18)	0.0000 (14)	0.0022 (13)	0.0041 (15)
C8	0.0340 (17)	0.0336 (19)	0.058 (2)	0.0014 (15)	−0.0066 (15)	0.0046 (17)
N1	0.0279 (13)	0.0259 (13)	0.0407 (15)	−0.0005 (11)	−0.0047 (11)	−0.0030 (12)
O1	0.0293 (10)	0.0387 (15)	0.0545 (14)	0.0005 (10)	−0.0093 (9)	−0.0015 (12)
O2	0.0407 (12)	0.0297 (14)	0.0705 (16)	0.0019 (11)	−0.0105 (11)	−0.0033 (12)
Cl1	0.0505 (5)	0.0519 (5)	0.0530 (5)	−0.0009 (5)	−0.0155 (4)	−0.0056 (5)

Geometric parameters (Å, º)

C1—C6	1.378 (4)	C5—H5	0.9300
C1—C2	1.379 (4)	C6—H6	0.9300
C1—O1	1.381 (3)	C7—O2	1.216 (4)
C2—C3	1.391 (4)	C7—N1	1.340 (4)
C2—H2	0.9300	C7—C8	1.520 (4)
C3—C4	1.387 (4)	C8—Cl1	1.774 (4)

C3—H3	0.9300	C8—H8A	0.9700
C4—C5	1.381 (4)	C8—H8B	0.9700
C4—N1	1.430 (3)	N1—H1	0.8600
C5—C6	1.382 (4)	O1—H1A	0.8200
C6—C1—C2	120.3 (3)	C1—C6—H6	120.1
C6—C1—O1	117.8 (3)	C5—C6—H6	120.1
C2—C1—O1	121.9 (3)	O2—C7—N1	125.5 (3)
C1—C2—C3	120.1 (3)	O2—C7—C8	116.4 (3)
C1—C2—H2	119.9	N1—C7—C8	118.1 (3)
C3—C2—H2	119.9	C7—C8—Cl1	116.7 (2)
C4—C3—C2	119.6 (3)	C7—C8—H8A	108.1
C4—C3—H3	120.2	Cl1—C8—H8A	108.1
C2—C3—H3	120.2	C7—C8—H8B	108.1
C5—C4—C3	119.8 (3)	Cl1—C8—H8B	108.1
C5—C4—N1	117.6 (3)	H8A—C8—H8B	107.3
C3—C4—N1	122.6 (3)	C7—N1—C4	126.0 (3)
C4—C5—C6	120.6 (3)	C7—N1—H1	117.0
C4—C5—H5	119.7	C4—N1—H1	117.0
C6—C5—H5	119.7	C1—O1—H1A	109.5
C1—C6—C5	119.7 (3)		
N1—C7—C8—Cl1	−15.4 (4)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O2	0.93	2.34	2.873 (4)	116
N1—H1···Cl1	0.86	2.53	2.999 (2)	115
N1—H1···O2^{i}	0.86	2.28	3.025 (3)	145
O1—H1*A*···O1^{ii}	0.82	2.06	2.8585 (17)	166

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