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Crystal structure of 4-amino-2,6-dichlorophenol

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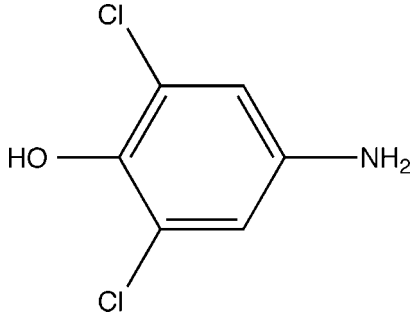
The title compound, $C_6H_5Cl_2NO$, has a single planar molecule in the asymmetric unit with the non-H atoms possessing a mean deviation from planarity of 0.020 Å. In the crystal, O—H···N hydrogen bonds lead to the formation of infinite chains along [101] which are further linked by N—H···O hydrogen bonds, forming (010) sheets.

Keywords: crystal structure; aminophenols; hydrogen bonding.

CCDC reference: 1400729

1. Related literature

For the crystal structure of the parent *p*-aminophenol, see: Brown (1951). For other related structures, see: Ermer & Eling (1994); Dey *et al.* (2005); Bacchi *et al.* (2009).



2. Experimental

2.1. Crystal data

$C_6H_5Cl_2NO$
M_r = 178.02
Monoclinic, $P2_1/n$
a = 4.6064 (5) Å
b = 11.7569 (12) Å
c = 13.2291 (13) Å
β = 96.760 (5)°
V = 711.47 (13) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 7.59 mm^{-1}
T = 120 K
0.4 × 0.2 × 0.1 mm

2.2. Data collection

Bruker D8 Venture CMOS diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2014)
T_{min} = 0.425, T_{max} = 0.754
7481 measured reflections
1402 independent reflections
1273 reflections with $I \geq 2\sigma(I)$
R_{int} = 0.043

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.091
S = 1.05
1402 reflections
99 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1^{i}	0.85 (2)	1.82 (2)	2.653 (2)	168 (2)
N1—H1a···O1^{ii}	0.87 (1)	2.05 (1)	2.921 (2)	177 (2)

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

Data collection: *APEX2* (Bruker, 2014); cell refinement: *SAINT* (Bruker, 2014); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015) and *OLEX2.refine* (Bourhis *et al.*, 2015); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2* and *publCIF* (Westrip, 2010).

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: FF2137).

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supporting information

Acta Cryst. (2015). E**71**, o406 [doi:10.1107/S2056989015009172]

Crystal structure of 4-amino-2,6-dichlorophenol

Kyle J. McDonald, Vasumathi Desikan, James A. Golen and David R. Manke

S1. Comment

The hydrogen bonding networks of aminophenols have been explored as hydroxy and amino groups are complementary hydrogen bonding donors and acceptors. This is exemplified in *p*-aminophenol, which exhibits a supertetrahedral hydrogen bonded architecture where all hydrogen bonding donors and acceptors are saturated (Brown, 1951; Ermer *et al.*, 1994). The mono-substitution in 4-amino-2-methylphenol and 4-amino-3-methylphenol yields a square motif structure that again exhibits saturation among hydrogen bonding donors and acceptors (Dey *et al.*, 2005). The more sterically encumbered substitution of 4-amino-2,6-diphenylphenol prevents the saturation in hydrogen bonding, with only O–H···N and N–H···aryl interactions observed (Bacchi *et al.*, 2009). The 2,6-dichloro substitution of the title compound also prevents saturation in its hydrogen bonding network.

The molecular structure of the title compound demonstrates a planar molecule with a mean deviation from the plane of the non-hydrogen atoms of 0.020 Å. Intermolecular hydrogen bonding between O1–H1···N1 results in infinite chains along [101] which combine with intermolecular hydrogen bonding between N1–H1a···O1 to give (010) sheets. The packing for the title compound indicating hydrogen bonding is shown in Figure 2.

S2. Experimental

A commercial sample (Aldrich) was used for the crystallization. Crystals suitable for single crystal X-ray analysis were grown by slow evaporation of a methanol solution.

S3. Refinement

All non-hydrogen atoms were refined anisotropically (Olex2) by full matrix least squares on F^2. Hydrogen atoms H1, H1a and H1b were found from a Fourier difference map. H1 was allowed to refine freely with an isotropic displacement parameter of 1.20 times U_{eq} of the parent O atom. H1a and H1b were refined with a fixed distance of 0.87 (0.005) Å and isotropic displacement parameters of 1.20 times U_{eq} of the parent N atom. The two remaining hydrogen atoms were placed in calculated positions and then refined with riding model with C–H lengths of 0.95 Å with isotropic displacement parameters set to 1.20 times U_{eq} of the parent C atom.

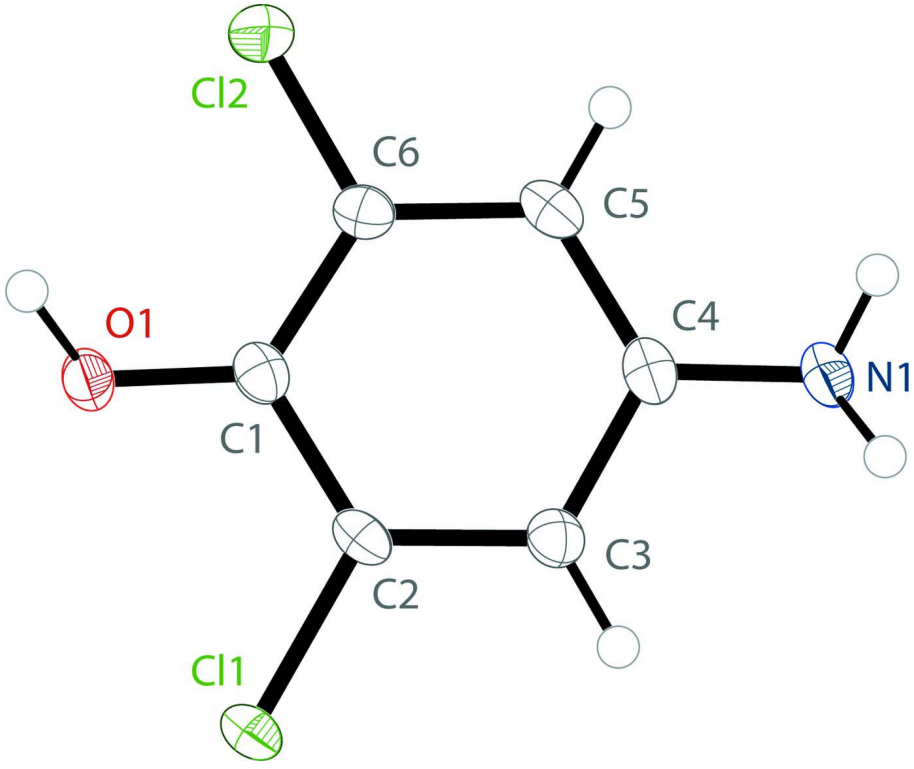

Figure 1

Molecular structure of the title compound, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are drawn as spheres of arbitrary radius.

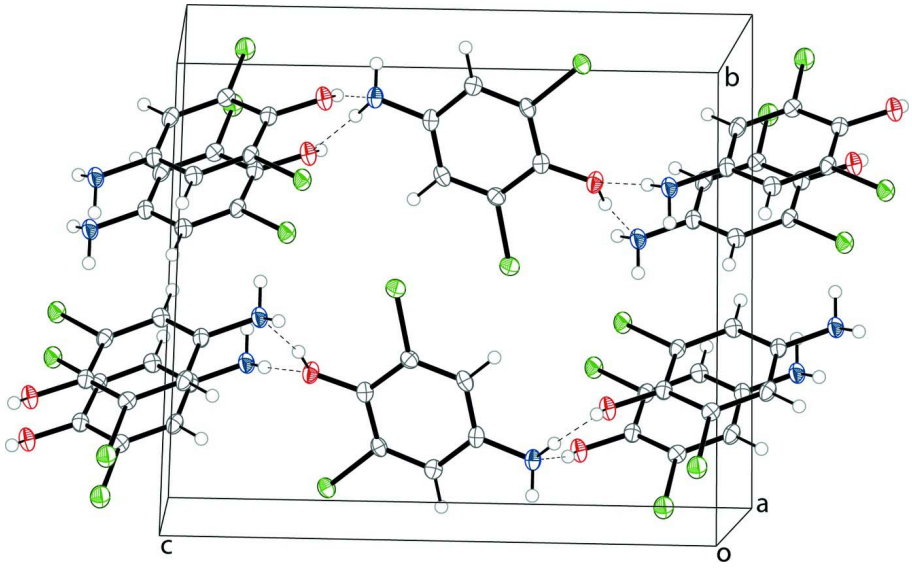

Figure 2

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

4-Amino-2,6-dichlorophenol

Crystal data

$C_6H_5Cl_2NO$
M_r = 178.02
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
a = 4.6064 (5) Å
b = 11.7569 (12) Å
c = 13.2291 (13) Å
β = 96.760 (5)°
V = 711.47 (13) Å^3
Z = 4
$F(000)$ = 363.7579
D_x = 1.662 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 5198 reflections
θ = 5.1–72.2°
μ = 7.59 mm^{-1}
T = 120 K
Plate, colourless
0.4 × 0.2 × 0.1 mm

Data collection

Bruker D8 Venture CMOS diffractometer
Radiation source: microfocus Cu
HELIOS MX monochromator
Detector resolution: 102.4 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2014)
T_{min} = 0.425, T_{max} = 0.754
7481 measured reflections
1402 independent reflections
1273 reflections with $I \geq 2\sigma(I)$
R_{int} = 0.043
θ_{max} = 72.2°, θ_{min} = 5.1°
h = −5→5
k = −14→14
l = −12→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.091
S = 1.05
1402 reflections
99 parameters
2 restraints
7 constraints
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0618P)^2 + 0.1912P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

Special details

Experimental. Absorption correction: SADABS-2014/4 (Bruker, 2014) was used for absorption correction. wR2(int) was 0.1370 before and 0.0641 after correction. The Ratio of minimum to maximum transmission is 0.5642. The $\lambda/2$ correction factor is 0.00150.

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

	x	y	z	U_{iso}*/U_{eq}
Cl1	0.10618 (10)	0.09750 (4)	0.70434 (3)	0.02213 (17)
Cl2	0.79187 (11)	0.44532 (4)	0.61176 (4)	0.02666 (18)
O1	0.4519 (3)	0.30303 (12)	0.74687 (10)	0.0214 (3)
N1	0.4522 (4)	0.13356 (14)	0.35171 (12)	0.0199 (4)
C1	0.4563 (4)	0.26523 (16)	0.65055 (14)	0.0176 (4)
C4	0.4607 (4)	0.17796 (16)	0.45234 (13)	0.0176 (4)
C5	0.6112 (4)	0.27756 (16)	0.48060 (14)	0.0196 (4)
H5	0.7170 (4)	0.31620 (16)	0.43359 (14)	0.0235 (5)*
C2	0.3011 (4)	0.16753 (16)	0.61822 (14)	0.0171 (4)
C3	0.3014 (4)	0.12337 (16)	0.52110 (14)	0.0182 (4)

H3	0.1938 (4)	0.05638 (16)	0.50165 (14)	0.0219 (5)*
C6	0.6052 (4)	0.31990 (16)	0.57788 (14)	0.0179 (4)
H1	0.615 (5)	0.329 (2)	0.7732 (18)	0.0215 (5)*
H1a	0.601 (3)	0.1551 (19)	0.3217 (15)	0.0215 (5)*
H1b	0.451 (5)	0.0598 (4)	0.3523 (17)	0.0215 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0232 (3)	0.0246 (3)	0.0204 (3)	−0.00409 (16)	0.00988 (19)	0.00083 (17)
Cl2	0.0334 (3)	0.0245 (3)	0.0225 (3)	−0.01014 (19)	0.0052 (2)	−0.00026 (18)
O1	0.0193 (6)	0.0298 (7)	0.0156 (7)	−0.0039 (6)	0.0046 (5)	−0.0042 (6)
N1	0.0206 (8)	0.0247 (9)	0.0151 (8)	0.0013 (6)	0.0057 (6)	−0.0009 (6)
C1	0.0146 (8)	0.0225 (9)	0.0159 (9)	0.0027 (7)	0.0023 (7)	0.0008 (7)
C4	0.0138 (8)	0.0248 (9)	0.0142 (9)	0.0050 (7)	0.0009 (7)	0.0005 (7)
C5	0.0177 (9)	0.0244 (10)	0.0173 (9)	0.0005 (7)	0.0048 (7)	0.0054 (7)
C2	0.0144 (8)	0.0215 (9)	0.0163 (9)	0.0011 (7)	0.0051 (7)	0.0035 (7)
C3	0.0148 (8)	0.0217 (9)	0.0185 (10)	0.0005 (7)	0.0029 (7)	−0.0007 (7)
C6	0.0158 (8)	0.0188 (9)	0.0194 (9)	−0.0007 (7)	0.0026 (7)	0.0016 (7)

Geometric parameters (Å, º)

Cl1—C2	1.7387 (18)	C1—C6	1.401 (3)
Cl2—C6	1.7389 (19)	C4—C5	1.389 (3)
O1—C1	1.352 (2)	C4—C3	1.391 (3)
O1—H1	0.85 (2)	C5—H5	0.9500
N1—C4	1.426 (2)	C5—C6	1.383 (3)
N1—H1a	0.870 (5)	C2—C3	1.386 (3)
N1—H1b	0.867 (5)	C3—H3	0.9500
C1—C2	1.393 (3)		
H1—O1—C1	113.3 (16)	C6—C5—C4	119.33 (17)
H1a—N1—C4	112.6 (15)	C6—C5—H5	120.34 (11)
H1b—N1—C4	110.9 (15)	C1—C2—Cl1	118.37 (14)
H1b—N1—H1a	108 (2)	C3—C2—Cl1	119.15 (14)
C2—C1—O1	119.75 (16)	C3—C2—C1	122.47 (16)
C6—C1—O1	123.97 (17)	C2—C3—C4	119.44 (18)
C6—C1—C2	116.26 (17)	H3—C3—C4	120.28 (11)
C5—C4—N1	121.18 (17)	H3—C3—C2	120.28 (11)
C3—C4—N1	118.87 (18)	C1—C6—Cl2	118.64 (14)
C3—C4—C5	119.85 (17)	C5—C6—Cl2	118.79 (14)
H5—C5—C4	120.34 (10)	C5—C6—C1	122.57 (17)
Cl1—C2—C3—C4	−178.98 (14)	C4—C5—C6—Cl2	−179.43 (14)
O1—C1—C2—Cl1	−0.1 (2)	C4—C5—C6—C1	1.0 (3)
O1—C1—C2—C3	−178.74 (17)	C5—C4—C3—C2	−1.7 (3)
O1—C1—C6—Cl2	−1.1 (3)	C2—C1—C6—Cl2	177.57 (13)
O1—C1—C6—C5	178.43 (17)	C2—C1—C6—C5	−2.9 (3)

N1—C4—C5—C6	177.70 (17)	C3—C4—C5—C6	1.3 (3)
N1—C4—C3—C2	−178.14 (17)	C6—C1—C2—Cl1	−178.80 (13)
C1—C2—C3—C4	−0.3 (3)	C6—C1—C2—C3	2.5 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1^{i}	0.85 (2)	1.82 (2)	2.653 (2)	168 (2)
N1—H1*a*···O1^{ii}	0.87 (1)	2.05 (1)	2.921 (2)	177 (2)

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