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2-Chloro-4-iodoaniline

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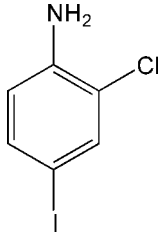
Key indicators: single-crystal X-ray study; T = 90 K; mean σ(C–C) = 0.005 Å; R factor = 0.024; wR factor = 0.046; data-to-parameter ratio = 19.1.

The title dihaloaniline, C_6H_5ClIN, shows no significant hydrogen bonds nor the commonly observed I···I interactions in the crystal structure, although an amino group and an I atom are available for such contacts. The crystal structure is stabilized by weak interactions involving the amine functionality as donor group and N or halogen atoms as acceptors.

Related literature

The title compound was first synthesized 90 years ago (Dains *et al.*, 1918). For structures of halogenated anilines, see: Cox (2001); Dey *et al.* (2003); Dou *et al.* (1993); Fukuyo *et al.* (1982); Goubitz *et al.* (2001); Parkin *et al.* (2005); Sakurai *et al.* (1963).



Experimental

Crystal data

C_6H_5ClIN
M_r = 253.46
Orthorhombic, $P2_12_12_1$
a = 5.6277 (2) Å
b = 8.7859 (3) Å
c = 14.9217 (5) Å
V = 737.79 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 4.61 mm^{-1}
T = 90.0 (2) K
0.22 × 0.15 × 0.10 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.424, T_{max} = 0.630
5635 measured reflections
1696 independent reflections
1587 reflections with $I > 2\sigma(I)$
R_{int} = 0.033

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.024
$wR(F^2)$ = 0.046
S = 1.14
1696 reflections
89 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 1.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.76 e Å^{-3}
Absolute structure: Flack (1983), 681 Friedel pairs
Flack parameter: −0.03 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···N1^{i}	0.82 (3)	2.61 (3)	3.359 (4)	153 (4)
N1—H1N···Cl1^{ii}	0.82 (3)	2.94 (4)	3.515 (4)	129 (4)
N1—H2N···I1^{iii}	0.81 (3)	3.16 (3)	3.807 (4)	139 (4)

Symmetry codes: (i) $x+\frac{1}{2}, -y+\frac{3}{2}, -z+1$; (ii) $x+1, y, z$; (iii) $-x+\frac{3}{2}, -y+1, z-\frac{1}{2}$.

Data collection: *COLLECT* (Nonius, 2002); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO–SMN* (Otwinowski & Minor, 1997); 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: *SHELXL97* and local procedures.

Y-HX thanks Dr Sihui Long for helpful discussions and invaluable suggestions.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BH2201).

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supporting information

Acta Cryst. (2008). E**64**, o2300 [doi:10.1107/S1600536808036076]

2-Chloro-4-iodoaniline

Yun-Hua Xu, Can Wang and Fanqi Qu

S1. Comment

Although structurally simple and readily available, few crystal structures of dihaloanilines have been measured. A total of 10 structures were found in the 2007 CSD; the refcodes are CAJWEQ, CAJWEQ01 (Goubitz *et al.*, 2001), DCHLAN, DCHLAN01 (Sakurai *et al.*, 1963), KUMTER (Cox, 2001), WEMDAT, WEMDEX, WEMDIB, WEMDOH, WEMDUN (Dou *et al.*, 1993). 2-Chloro-4-iodoaniline, (I), an aniline with two different halogen substituents, was first synthesized 90 years ago (Dains *et al.*, 1918), yet its crystal structure is reported here for the first time.

The asymmetric unit contains one molecule (Fig. 1). The N atom is not coplanar with the aromatic ring; H atoms of the amino group are also out of the halogenated benzene ring, but in the opposite direction to that of the N atom. So, the C(Ar)NH_2 group has a pyramidal shape. This is similar to the structure of aniline at 252 K (Fukuyo *et al.*, 1982), 2-iodoaniline at 100 K (Parkin *et al.*, 2005) and 4-iodoaniline at 203 K (Dey *et al.*, 2003).

Despite the presence of amino, chloro and iodo groups, no classic interactions associated with them, such as hydrogen bonds, Cl···Cl, or I···I contacts were observed in the crystal structure of (I). Instead, weak interactions such as N—H···N, N—H···I, and N—H···Cl are found to provide stability to the crystal (Fig. 2).

S2. Experimental

The compound was purchased from TCI America Laboratory Chemicals as colorless block crystals suitable for single-crystal X-ray diffraction measurement.

S3. Refinement

H atoms were found in a difference map and those on the aromatic ring subsequently placed in idealized positions with C—H distances of 0.95 Å and isotropic displacement parameters equal to $1.2U_{eq}$ of the carrier C atom. Amine H atoms H1N and H2N were refined freely but were restrained to converge to the same N—H bond lengths, with a standard deviation of 0.02 Å. Isotropic displacement parameters for H1N and H2N were computed as $1.5U_{eq}$(N1)

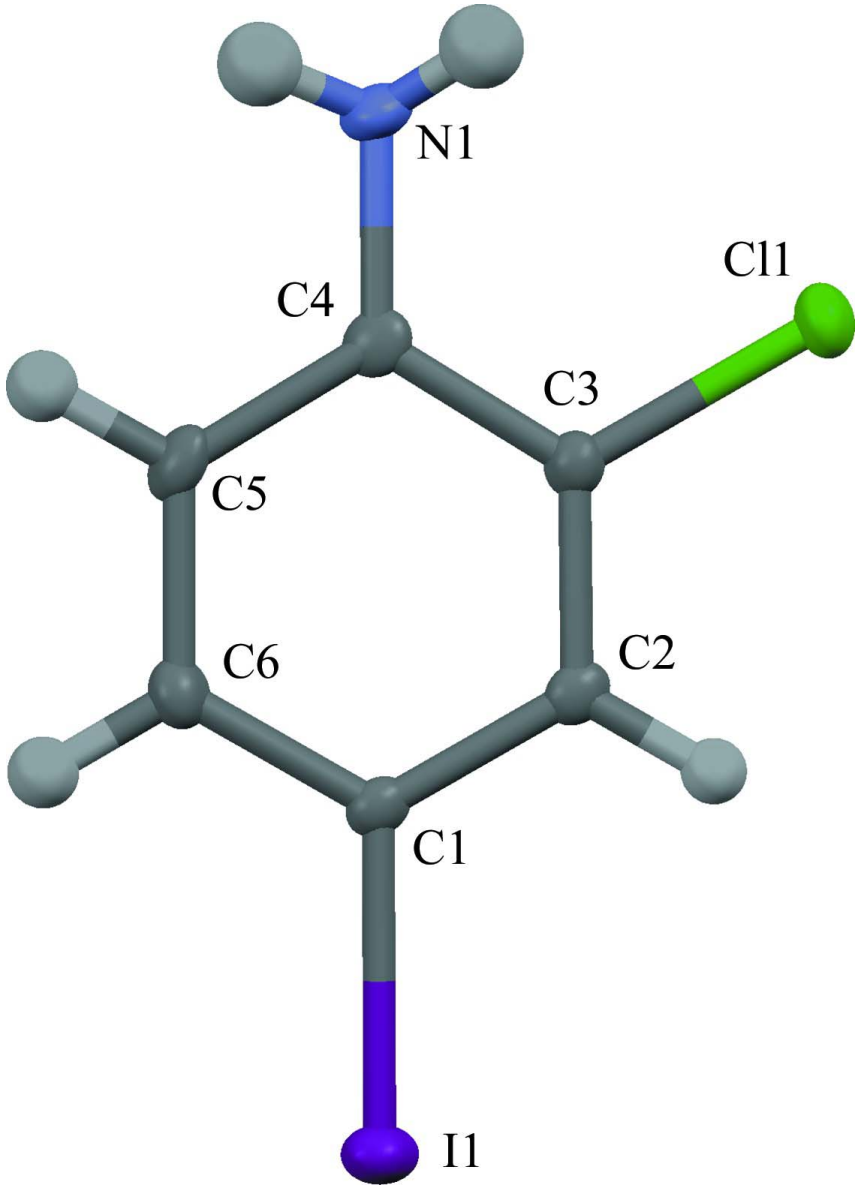

Figure 1

The molecular structure of (I), with displacement ellipsoids drawn at the 50% probability level (arbitrary spheres for the H atoms).

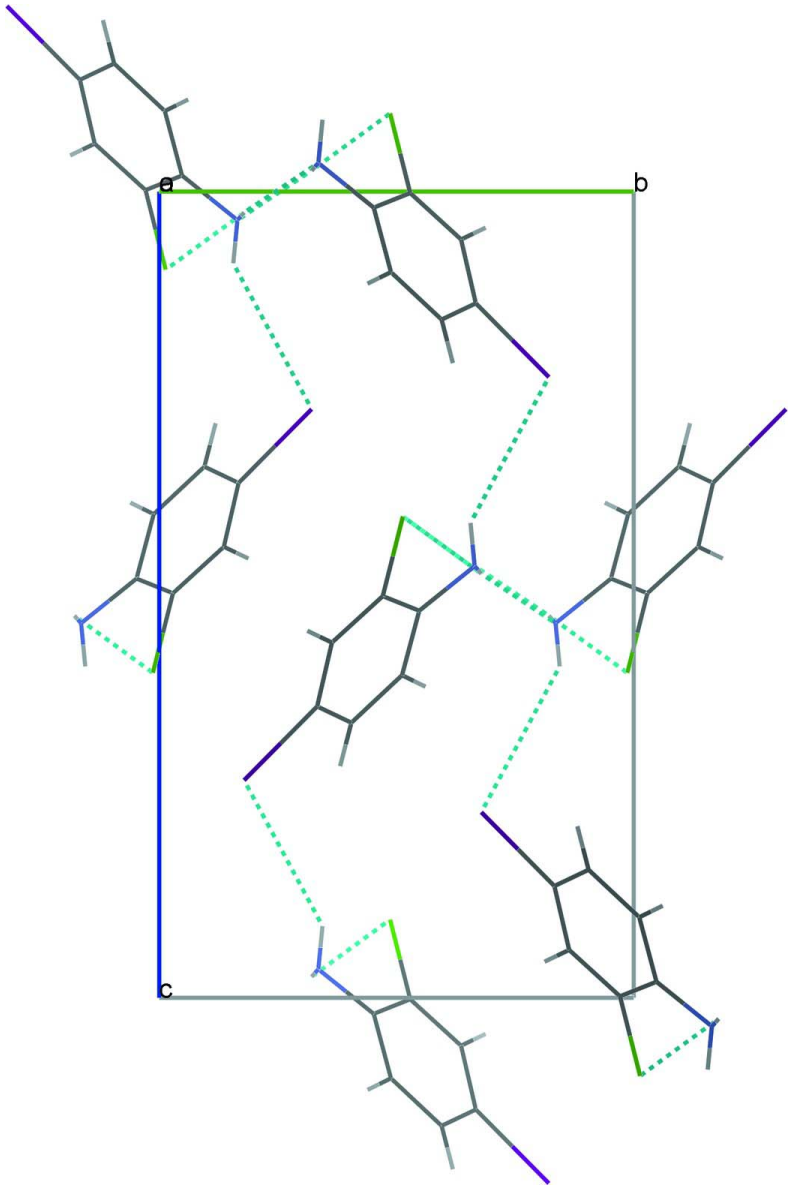

Figure 2

A packing diagram of (I) down the *a* axis.

2-Chloro-4-iodoaniline

Crystal data

C_6H_5ClIN
$M_r = 253.46$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 5.6277\ (2)$ Å
$b = 8.7859\ (3)$ Å
$c = 14.9217\ (5)$ Å
$V = 737.79\ (4)$ Å^3
$Z = 4$
$F(000) = 472$
$D_x = 2.282$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1019 reflections
$\theta = 1.0$–$27.5°$
$\mu = 4.61$ mm^{-1}
$T = 90$ K
Rounded block, colourless
0.22 × 0.15 × 0.10 mm

Data collection

Nonius KappaCCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 18 pixels mm^{-1}
ω scans at fixed χ = 55°
Absorption correction: multi-scan (*SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.424, T_{max} = 0.630
5635 measured reflections
1696 independent reflections
1587 reflections with $I > 2\sigma(I)$
R_{int} = 0.033
θ_{max} = 27.5°, θ_{min} = 2.7°
h = −7→7
k = −11→11
l = −19→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.024
$wR(F^2)$ = 0.046
S = 1.14
1696 reflections
89 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
$w = 1/[\sigma^2(F_o^2) + (0.P)^2 + 0.4678P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 1.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.76 e Å^{-3}
Extinction correction: *SHELXL97*, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0021 (3)
Absolute structure: Flack (1983), 681 Friedel pairs
Absolute structure parameter: −0.03 (3)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
I1	0.48590 (4)	0.17882 (2)	0.730162 (15)	0.01928 (9)
Cl1	0.46148 (17)	0.51349 (10)	0.40305 (6)	0.0194 (2)
N1	0.9067 (6)	0.6647 (5)	0.4646 (2)	0.0188 (8)
H1N	1.048 (5)	0.679 (5)	0.474 (3)	0.028*
H2N	0.882 (7)	0.656 (5)	0.411 (2)	0.028*
C1	0.6370 (6)	0.3328 (4)	0.6386 (2)	0.0133 (7)
C2	0.5194 (7)	0.3639 (3)	0.5594 (2)	0.0138 (7)
H2	0.3764	0.3121	0.5448	0.017*
C3	0.6128 (6)	0.4711 (4)	0.5018 (2)	0.0145 (8)
C4	0.8253 (6)	0.5480 (4)	0.5199 (3)	0.0152 (8)
C5	0.9422 (6)	0.5117 (4)	0.5999 (2)	0.0166 (8)
H5	1.0875	0.5611	0.6142	0.020*
C6	0.8494 (7)	0.4049 (4)	0.6588 (3)	0.0163 (8)
H6	0.9312	0.3812	0.7127	0.020*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.02354 (13)	0.01764 (13)	0.01666 (14)	−0.00271 (12)	0.00379 (13)	0.00106 (9)
Cl1	0.0189 (5)	0.0231 (4)	0.0163 (4)	0.0003 (4)	−0.0036 (4)	−0.0007 (3)
N1	0.0163 (16)	0.0192 (18)	0.0208 (18)	−0.0046 (15)	0.0035 (15)	0.0025 (15)
C1	0.0136 (16)	0.0101 (17)	0.0161 (19)	0.0001 (15)	0.0027 (15)	−0.0029 (16)

C2	0.0142 (18)	0.0107 (15)	0.0165 (17)	0.0005 (16)	0.003 (2)	−0.0046 (13)
C3	0.0134 (18)	0.0133 (17)	0.0169 (19)	0.0020 (16)	−0.0006 (16)	−0.0017 (16)
C4	0.0121 (18)	0.0129 (18)	0.020 (2)	0.0043 (15)	0.0034 (16)	−0.0031 (17)
C5	0.0096 (18)	0.0154 (17)	0.025 (2)	0.0007 (14)	0.0002 (16)	−0.0053 (15)
C6	0.0172 (19)	0.0182 (19)	0.0133 (18)	0.0019 (16)	−0.0003 (16)	−0.0034 (16)

Geometric parameters (Å, º)

I1—C1	2.103 (4)	C2—C3	1.379 (5)
Cl1—C3	1.742 (4)	C2—H2	0.9500
N1—C4	1.394 (5)	C3—C4	1.400 (5)
N1—H1N	0.82 (3)	C4—C5	1.400 (5)
N1—H2N	0.81 (3)	C5—C6	1.387 (5)
C1—C2	1.382 (5)	C5—H5	0.9500
C1—C6	1.386 (5)	C6—H6	0.9500
C4—N1—H1N	110 (3)	C4—C3—Cl1	118.6 (3)
C4—N1—H2N	117 (3)	N1—C4—C5	121.2 (3)
H1N—N1—H2N	110 (5)	N1—C4—C3	121.4 (3)
C2—C1—C6	120.6 (3)	C5—C4—C3	117.2 (3)
C2—C1—I1	119.3 (3)	C6—C5—C4	121.1 (3)
C6—C1—I1	120.1 (3)	C6—C5—H5	119.4
C3—C2—C1	119.0 (3)	C4—C5—H5	119.4
C3—C2—H2	120.5	C1—C6—C5	119.7 (3)
C1—C2—H2	120.5	C1—C6—H6	120.1
C2—C3—C4	122.3 (3)	C5—C6—H6	120.1
C2—C3—Cl1	119.1 (3)		
C6—C1—C2—C3	−1.9 (5)	Cl1—C3—C4—C5	179.8 (3)
I1—C1—C2—C3	176.5 (2)	N1—C4—C5—C6	174.2 (3)
C1—C2—C3—C4	1.2 (5)	C3—C4—C5—C6	−0.5 (5)
C1—C2—C3—Cl1	−178.6 (3)	C2—C1—C6—C5	1.5 (5)
C2—C3—C4—N1	−174.7 (3)	I1—C1—C6—C5	−176.9 (2)
Cl1—C3—C4—N1	5.1 (5)	C4—C5—C6—C1	−0.3 (5)
C2—C3—C4—C5	0.0 (5)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···N1^{i}	0.82 (3)	2.61 (3)	3.359 (4)	153 (4)
N1—H1*N*···Cl1^{ii}	0.82 (3)	2.94 (4)	3.515 (4)	129 (4)
N1—H2*N*···I1^{iii}	0.81 (3)	3.16 (3)	3.807 (4)	139 (4)

Symmetry codes: (i) *x*+1/2, −*y*+3/2, −*z*+1; (ii) *x*+1, *y*, *z*; (iii) −*x*+3/2, −*y*+1, *z*−1/2.