organic compounds

2,3,6-Trichloro-5-(trichloromethyl)-pyridine

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The title compound, C_6HCl_6N, lies on a mirror plane, the asymmetric unit conataining a half-molecule. Weak intramolecular C—H···Cl contacts are observed.

Related literature

For biological background, see: Okorley & Dietsche (1988). For the synthetic procedure, see: Allphin et al. (1993); For a related structure, see: Fun et al. (2011).
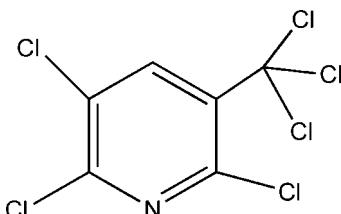
Experimental

Crystal data

C_6HCl_6N	$V = 1034.6$ (4) Å^3
$M_r = 299.78$	$Z = 4$
Orthorhombic, $Pbcm$	Mo $K\alpha$ radiation
$a = 8.3100$ (17) Å	$\mu = 1.61$ mm^{-1}
$b = 17.018$ (3) Å	$T = 293$ K
$c = 7.3160$ (15) Å	$0.30 \times 0.20 \times 0.20$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer	1033 independent reflections
Absorption correction: ψ scan (North et al., 1968)	779 reflections with $I > 2\sigma(I)$
$T_{min} = 0.644$, $T_{max} = 0.739$	$R_{int} = 0.063$
1985 measured reflections	3 standard reflections every 200 reflections
	intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.040$	77 parameters
$wR(F^2) = 0.123$	H-atom parameters constrained
$S = 1.01$	$\Delta\rho_{max} = 0.26$ e Å^{-3}
1033 reflections	$\Delta\rho_{min} = -0.39$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1A···Cl5	0.93	2.48	2.944 (5)	111

Data collection: *CAD-4 Software* (Enraf–Nonius, 1985); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); 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: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2577).

References

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supporting information

2,3,6-Trichloro-5-(trichloromethyl)pyridine

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S1. Comment

Polychloropyridines derivatives are useful as intermediates for production of biological compounds (Okorley & Dietsche, (1988). Herein, we report the crystal structure of the title compound (Fig. 1). The bond distances and angles in the title compound agree very well with the corresponding bond distances and angles reported in a closely related compound (Fun *et al.*, 2011).

S2. Experimental

The title compound was synthesized by the chlorination of 2-chloro-5-chloromethyl pyridine using chlorine gas as a chlorinating agent in the presence of ultraviolet radiation and in the presence of WCl_6 for 6.0 h by following a reported synthetic procedure (Allphin *et al.*, 1993). The crystals of the title compound were obtained from a solution of 1,2-di-chloroethane by evaporating the solvent slowly at room temperature in about 5 d.

S3. Refinement

The only H atom was positioned geometrically and constrained to ride on C1 with C—H = 0.93 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.
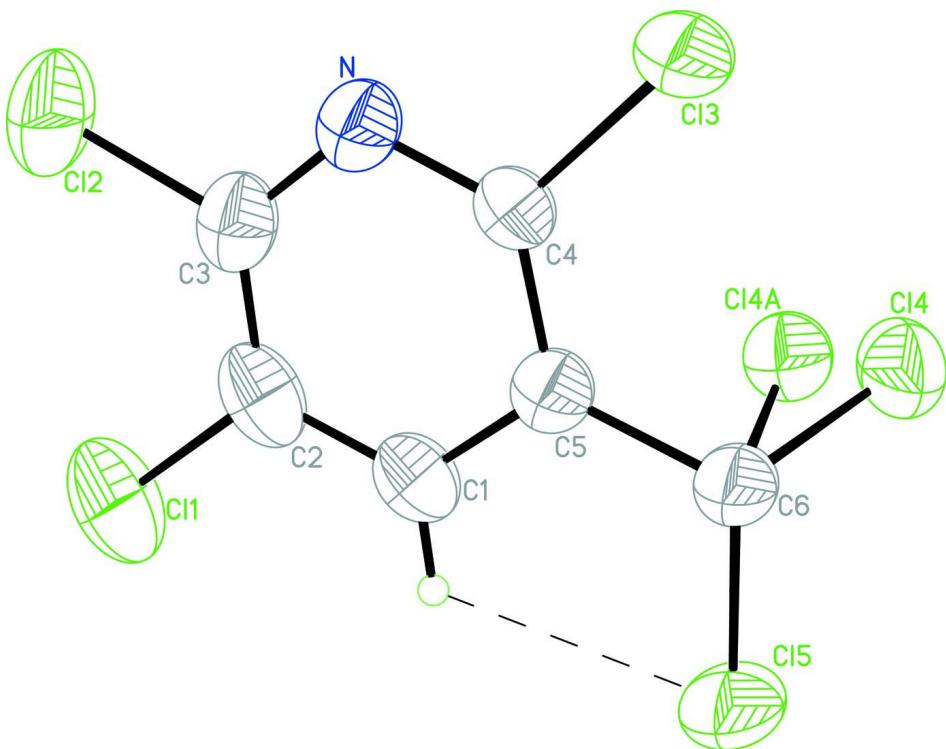
Figure 1

The molecular structure of the title compound with the atom numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atom ise presented as small spheres of arbitrary radius. [Symmetry code A: x, y, 1/2-z]
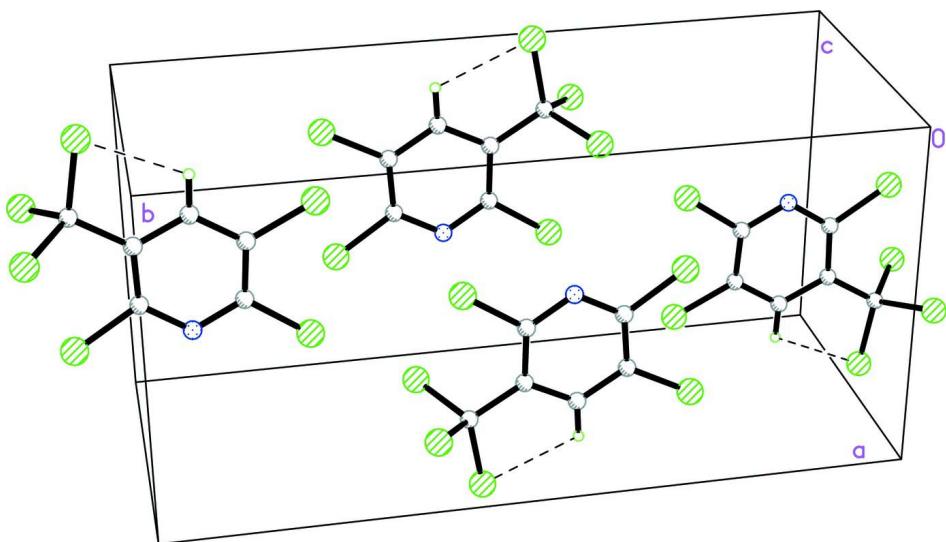
Figure 2

A packing diagram for the title compound showing C—H···Cl intra-molecular hydrogen bonds (dashed lines).

2,3,6-Trichloro-5-(trichloromethyl)pyridine

Crystal data

C_6HCl_6N	$F(000) = 584$
$M_r = 299.78$	$D_x = 1.925$ Mg m^{-3}
Orthorhombic, *Pbcm*	Mo *Kα* radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2c 2b	Cell parameters from 25 reflections
$a = 8.3100$ (17) Å	$\theta = 10–13°$
$b = 17.018$ (3) Å	$\mu = 1.61$ mm^{-1}
$c = 7.3160$ (15) Å	$T = 293$ K
$V = 1034.6$ (4) Å^3	Block, colorless
$Z = 4$	$0.30 \times 0.20 \times 0.20$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer	1033 independent reflections
Radiation source: fine-focus sealed tube	779 reflections with $I > 2\sigma(I)$
Graphite monochromator	$R_{int} = 0.063$
$\omega/2\theta$ scans	$\theta_{max} = 25.4°$, $\theta_{min} = 2.4°$
Absorption correction: ψ scan (North *et al.*, 1968)	$h = -10 \rightarrow 10$
	$k = -20 \rightarrow 0$
$T_{min} = 0.644$, $T_{max} = 0.739$	$l = -8 \rightarrow 0$
1985 measured reflections	3 standard reflections every 200 reflections
	intensity decay: 1%

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.040$	$w = 1/[\sigma^2(F_o^2) + (0.078P)^2]$
$wR(F^2) = 0.123$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.00$	$(\Delta/\sigma)_{max} = 0.001$
1033 reflections	$\Delta\rho_{max} = 0.26$ e Å^{-3}
77 parameters	$\Delta\rho_{min} = -0.38$ e Å^{-3}
0 restraints	Extinction correction: *SHELXL97* (Sheldrick, 2008), Fc*=kFc[1+0.001xFc$^2\lambda^3$/sin(2θ)]$^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.077 (6)
Secondary atom site location: difference Fourier map	

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 $F^2 > \sigma(F^2)$ 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}
N	0.5025 (5)	0.4324 (2)	0.2500	0.0467 (9)
Cl1	0.8417 (2)	0.26884 (8)	0.2500	0.0806 (6)

C1	0.8339 (6)	0.4268 (3)	0.2500	0.0478 (11)
H1A	0.9457	0.4241	0.2500	0.057*
Cl2	0.45826 (19)	0.28195 (8)	0.2500	0.0753 (5)
C2	0.7445 (7)	0.3584 (3)	0.2500	0.0537 (12)
Cl3	0.47402 (13)	0.58193 (7)	0.2500	0.0569 (4)
C3	0.5783 (6)	0.3642 (3)	0.2500	0.0486 (11)
Cl4	0.81252 (10)	0.63147 (5)	0.05227 (15)	0.0619 (4)
C4	0.5902 (5)	0.4969 (2)	0.2500	0.0399 (10)
Cl5	1.06686 (13)	0.55710 (8)	0.2500	0.0672 (5)
C5	0.7586 (5)	0.4991 (2)	0.2500	0.0418 (10)
C6	0.8554 (5)	0.5748 (2)	0.2500	0.0451 (11)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N	0.0472 (18)	0.042 (2)	0.051 (2)	−0.0058 (15)	0.000	0.000
Cl1	0.1128 (13)	0.0423 (7)	0.0867 (11)	0.0263 (7)	0.000	0.000
C1	0.051 (3)	0.042 (2)	0.051 (3)	0.0113 (19)	0.000	0.000
Cl2	0.0977 (11)	0.0433 (7)	0.0849 (10)	−0.0220 (6)	0.000	0.000
C2	0.075 (3)	0.042 (2)	0.044 (2)	0.014 (2)	0.000	0.000
Cl3	0.0372 (6)	0.0463 (7)	0.0872 (10)	0.0073 (4)	0.000	0.000
C3	0.061 (3)	0.043 (2)	0.042 (2)	−0.009 (2)	0.000	0.000
Cl4	0.0572 (5)	0.0552 (6)	0.0734 (7)	0.0006 (3)	0.0064 (5)	0.0182 (4)
C4	0.039 (2)	0.034 (2)	0.046 (2)	0.0056 (17)	0.000	0.000
Cl5	0.0334 (6)	0.0666 (8)	0.1018 (12)	0.0007 (5)	0.000	0.000
C5	0.041 (2)	0.040 (2)	0.044 (2)	−0.0008 (18)	0.000	0.000
C6	0.036 (2)	0.039 (2)	0.061 (3)	0.0008 (17)	0.000	0.000

Geometric parameters (Å, º)

N—C4	1.318 (6)	C2—C3	1.385 (8)
N—C3	1.321 (6)	Cl3—C4	1.739 (4)
Cl1—C2	1.724 (5)	Cl4—C6	1.774 (3)
C1—C2	1.381 (7)	C4—C5	1.400 (6)
C1—C5	1.381 (6)	Cl5—C6	1.783 (5)
C1—H1A	0.9300	C5—C6	1.519 (6)
Cl2—C3	1.719 (4)	C6—Cl4i	1.774 (3)
C4—N—C3	118.0 (4)	N—C4—Cl3	112.7 (3)
C2—C1—C5	120.5 (4)	C5—C4—Cl3	122.2 (3)
C2—C1—H1A	119.7	C1—C5—C4	115.4 (4)
C5—C1—H1A	119.7	C1—C5—C6	121.1 (4)
C1—C2—C3	118.4 (4)	C4—C5—C6	123.5 (4)
C1—C2—Cl1	119.6 (4)	C5—C6—Cl4	110.77 (18)
C3—C2—Cl1	122.0 (4)	C5—C6—Cl4i	110.77 (18)
N—C3—C2	122.6 (4)	Cl4—C6—Cl4i	109.2 (2)
N—C3—Cl2	116.0 (4)	C5—C6—Cl5	112.2 (3)
C2—C3—Cl2	121.4 (4)	Cl4—C6—Cl5	106.84 (17)

N—C4—C5	125.1 (4)	Cl4ⁱ—C6—Cl5	106.84 (17)
C5—C1—C2—C3	0.0	C2—C1—C5—C6	180.0
C5—C1—C2—Cl1	180.0	N—C4—C5—C1	0.0
C4—N—C3—C2	0.0	Cl3—C4—C5—C1	180.0
C4—N—C3—Cl2	180.0	N—C4—C5—C6	180.0
C1—C2—C3—N	0.0	Cl3—C4—C5—C6	0.0
Cl1—C2—C3—N	180.0	C1—C5—C6—Cl4	119.32 (19)
C1—C2—C3—Cl2	180.0	C4—C5—C6—Cl4	−60.68 (19)
Cl1—C2—C3—Cl2	0.0	C1—C5—C6—Cl4ⁱ	−119.32 (19)
C3—N—C4—C5	0.0	C4—C5—C6—Cl4ⁱ	60.68 (19)
C3—N—C4—Cl3	180.0	C1—C5—C6—Cl5	0.0
C2—C1—C5—C4	0.0	C4—C5—C6—Cl5	180.0

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

Hydrogen-bond geometry (Å, º)

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C1—H1$A\cdots$Cl5	0.93	2.48	2.944 (5)	111