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2,7-Bis(trichloromethyl)-1,8-naphthyridine[1]

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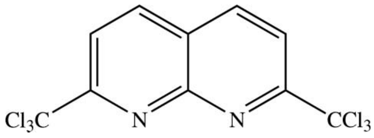
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.035; wR factor = 0.089; data-to-parameter ratio = 44.1.

The complete molecule of the title compound, $C_{10}H_4Cl_6N_2$, is generated by crystallographic twofold symmetry, with two C atoms lying on the rotation axis; the 1,8-naphthyridine ring is almost planar with an r.m.s. deviation of 0.0002 Å. In the crystal structure, the molecules are stacked in an antiparallel manner along [001]. Short Cl···Cl [3.3502 (4)] and Cl···N [3.2004 (11)–3.2220 (10) Å] contacts are observed in the crystal structure.

Related literature

For bond-length data, see: Allen *et al.* (1987). For graph-set notation of hydrogen-bond motifs, see: Bernstein *et al.* (1995). For related structures, see: Fun *et al.* (2009); Wang *et al.* (2008). For background to the properties and applications of 1,8-naphthyridines, see: Braccio *et al.* (2008); Chen *et al.* (2001); Ferrarini *et al.* (1998; 2000). For the stability of the temperature controller used in the data collection, see: Cosier & Glazer (1986).



Experimental

Crystal data

$C_{10}H_4Cl_6N_2$	V = 1295.73 (4) Å^3
M_r = 364.85	Z = 4
Monoclinic, $C2/c$	Mo $K\alpha$ radiation
a = 19.9154 (4) Å	μ = 1.30 mm^{-1}
b = 6.5977 (1) Å	T = 100 K
c = 10.5975 (2) Å	0.40 × 0.26 × 0.05 mm
β = 111.483 (2)°	

Data collection

Bruker APEXII CCD diffractometer	28872 measured reflections
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)	4010 independent reflections
T_{min} = 0.624, T_{max} = 0.944	3136 reflections with $I > 2\sigma(I)$
	R_{int} = 0.053

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035	91 parameters
$wR(F^2)$ = 0.089	All H-atom parameters refined
S = 1.05	$\Delta\rho_{max}$ = 0.64 e Å^{-3}
4010 reflections	$\Delta\rho_{min}$ = −0.56 e Å^{-3}

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5307).

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[1] This paper is dedicated to His Majesty King Bhumibol Adulyadej of Thailand (King Rama IX) for his sustainable development of the country.
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supplementary materials

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2,7-Bis(trichloromethyl)-1,8-naphthyridine

H.-K. Fun, S. Chantrapromma, A. C. Maity and **S. Goswami**

Comment

The substituted 1,8-naphthyridine compounds have been studied for their chemical and biological activities for a long time. They show various biological activities such as antibacterial (Chen *et al.*, 2001; Ferrarini *et al.*, 1998), anti-inflammatory (Braccio *et al.*, 2008) as well as antihypertensive (Ferrarini *et al.*, 2000) properties. Trichloromethyl-substituted heterocyclic compounds are of great importance due to their broad spectrum biological activities. These interesting properties prompt us to synthesise the title compound (I) and its crystal structure was reported.

The asymmetric unit of the title molecule (Fig. 1), $C_{10}H_4Cl_6N_2$, contains one half-molecule with two shared C atoms (C3 and C4) lying on a twofold rotation axis. The 1,8-naphthyridine ring is planar with the r.m.s. deviation of 0.0002 (2) Å. One Cl atom (Cl3) of the trichloromethyl substitutent is co-planar with the 1,8-naphthyridine ring which can be indicated by the torsion angle C1–C2–C6–Cl3 = 1.85 (14) Å whereas the other two Cl atoms are in the (+)-anti-clinal and (-)-anti-clinal configurations with the torsion angles C1–C2–C6–Cl1 = 122.04 (10)° and C1–C2–C6–Cl2 = -119.02 (10)°, respectively. The C1—H1···Cl3 intramolecular interaction (Table 1) generates S(5) ring motif (Bernstein *et al.*, 1995). The bond distances are of normal values (Allen *et al.*, 1987) and are comparable with related structures (Fun *et al.*, 2009; Wang *et al.*, 2008).

In the crystal structure (Fig. 2), the non-covalent interactions play a significant role in the three-dimensional supramolecular architecture in which the molecules are stacked in an antiparallel manner along the [0 0 1] direction and the neighbouring molecules are interlinked by C—Cl···Cl interactions into polymeric chains along the [0 1 0] direction. The molecules are also consolidated by Cl···Cl [3.3502 (4)Å] and Cl···N [3.2004 (11)–3.2220 (10) Å] short contacts. $\pi\cdots\pi$ interactions were observed with the distances of $Cg_1\cdots Cg_1$ = 4.2360 (6) Å (symmetry code: $-x$, $1 - y$, $2 - z$) and $Cg_2\cdots Cg_2$ = 4.2360 (6) Å (symmetry code: $-x$, $1 - y$, $1 - z$): Cg_1 and Cg_2 are the centroids of C1–C4/C5A/N1 and C1A–C2A/C3–C5/N1A, respectively. All these interactions connect the molecules into a three-dimensional supramolecular network.

Experimental

A mixture of *N*-chlorosuccinimide (500 mg, 4.5 mmol) and triphenylphosphine (500 mg, 4.2 mmol) was moistened with CCl_4 (60 ml) in a round bottom flask and stirred at room temperature for 25 min. A solution of 2,7-dimethyl-1,8-naphthyridine (0.9 g, 5.25 mmol) was added to the suspension and the reaction mixture was stirred and heated under reflux for 7 hr. The solution was cooled and filtered. The evaporated filtrate was washed with saturated aqueous Na_2CO_3 and extracted repeatedly with $CHCl_3$. Drying over anhydrous Na_2SO_4, the solvent was removed under reduced pressure. The crude product was purified with SiO_2 chromatography (eluted with 1% ethylacetate in petroleum ether) to give the title compound as a white crystalline solid. Colorless slabs of (I) were recrystalized from CH_2Cl_2:hexane (1:10, *v*/*v*) by the slow evaporation of the solvent at room temperature after a week.

Refinement

H atoms were located in a difference maps and refined isotropically. The highest residual electron density peak is located at 1.72 Å from H6 and the deepest hole is located at 0.65 Å from Cl2.

Figures

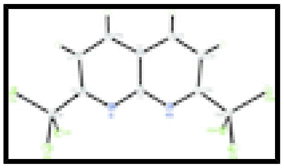

Fig. 1. The molecular structure of (I) showing 50% probability displacement ellipsoids. Atoms with suffix A were generated by symmetry code -*x*, *y*, -1/2 - *z*.

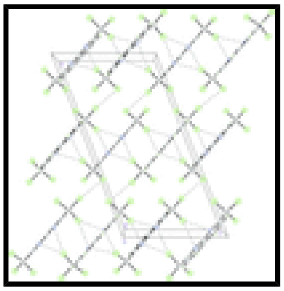

Fig. 2. The crystal packing of the title compound viewed along the *b* axis, N···Cl and C···.Cl short contacts are shown as dashed lines.

2,7-Bis(trichloromethyl)-1,8-naphthyridine

Crystal data

$C_{10}H_4Cl_6N_2$

M_r = 364.85

Monoclinic, *C*2/*c*

Hall symbol: -C 2yc

a = 19.9154 (4) Å

b = 6.5977 (1) Å

c = 10.5975 (2) Å

β = 111.483 (2)°

V = 1295.73 (4) Å^3

Z = 4

F(000) = 720

D_x = 1.870 Mg m^{-3}

Mo *K*α radiation, λ = 0.71073 Å

Cell parameters from 4010 reflections

θ = 2.2–40.0°

μ = 1.30 mm^{-1}

T = 100 K

Slab, colorless

0.40 × 0.26 × 0.05 mm

Data collection

Bruker APEXII CCD diffractometer

Radiation source: sealed tube

graphite

φ and ω scans

Absorption correction: multi-scan (SADABS; Bruker, 2005)

T_{min} = 0.624, T_{max} = 0.944

4010 independent reflections

3136 reflections with $I > 2\sigma(I)$

R_{int} = 0.053

θ_{max} = 40.0°, θ_{min} = 2.2°

$h = -36 \rightarrow 34$

$k = -11 \rightarrow 11$

28872 measured reflections	$l = -18 \rightarrow 19$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.035$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.089$	All H-atom parameters refined
$S = 1.05$	$w = 1/[\sigma^2(F_o^2) + (0.0403P)^2 + 0.889P]$ where $P = (F_o^2 + 2F_c^2)/3$
4010 reflections	$(\Delta/\sigma)_{max} = 0.002$
91 parameters	$\Delta\rho_{max} = 0.64$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.56$ e Å^{-3}

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 120.0 (1) K.

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}
Cl1	0.090117 (15)	0.01827 (4)	1.12884 (3)	0.01585 (6)
Cl2	0.194324 (15)	0.02817 (4)	0.99732 (3)	0.01645 (6)
Cl3	0.202697 (15)	0.31574 (4)	1.20815 (3)	0.01848 (6)
N1	0.04657 (5)	0.21807 (14)	0.85379 (9)	0.01358 (15)
C1	0.09721 (6)	0.53166 (17)	0.96675 (12)	0.01599 (18)
C2	0.09266 (6)	0.31794 (16)	0.95656 (11)	0.01302 (16)
C3	0.0000	0.3281 (2)	0.7500	0.0124 (2)
C4	0.0000	0.5430 (2)	0.7500	0.0136 (2)
C5	−0.05049 (6)	0.64457 (17)	0.63741 (11)	0.01611 (18)
C6	0.14272 (6)	0.17959 (16)	1.06796 (10)	0.01333 (16)
H6	−0.0513 (10)	0.788 (3)	0.6355 (18)	0.021 (4)*
H1	0.1292 (10)	0.598 (3)	1.038 (2)	0.024 (5)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.01798 (11)	0.01518 (11)	0.01362 (10)	−0.00263 (8)	0.00487 (8)	0.00164 (8)
Cl2	0.01578 (11)	0.01700 (12)	0.01533 (11)	0.00202 (8)	0.00425 (8)	0.00040 (8)
Cl3	0.02005 (12)	0.01629 (11)	0.01330 (11)	−0.00383 (9)	−0.00076 (8)	−0.00181 (8)
N1	0.0155 (4)	0.0114 (3)	0.0115 (3)	−0.0004 (3)	0.0021 (3)	0.0002 (3)
C1	0.0186 (4)	0.0110 (4)	0.0152 (4)	−0.0022 (3)	0.0025 (4)	−0.0012 (3)
C2	0.0145 (4)	0.0113 (4)	0.0123 (4)	−0.0008 (3)	0.0038 (3)	0.0001 (3)
C3	0.0137 (5)	0.0101 (5)	0.0117 (5)	0.000	0.0027 (4)	0.000
C4	0.0173 (6)	0.0095 (5)	0.0132 (5)	0.000	0.0045 (5)	0.000
C5	0.0198 (5)	0.0104 (4)	0.0155 (4)	0.0011 (3)	0.0033 (4)	0.0009 (3)
C6	0.0148 (4)	0.0122 (4)	0.0113 (4)	−0.0015 (3)	0.0028 (3)	−0.0007 (3)

Geometric parameters (Å, °)

Cl1—C6	1.7725 (11)	C2—C6	1.5358 (15)
Cl2—C6	1.7827 (11)	C3—N1$^{\mathrm{i}}$	1.3602 (12)
Cl3—C6	1.7723 (10)	C3—C4	1.418 (2)
N1—C2	1.3156 (14)	C4—C5	1.4160 (13)
N1—C3	1.3602 (12)	C4—C5$^{\mathrm{i}}$	1.4160 (13)
C1—C5$^{\mathrm{i}}$	1.3736 (16)	C5—C1$^{\mathrm{i}}$	1.3737 (16)
C1—C2	1.4145 (15)	C5—H6	0.95 (2)
C1—H1	0.90 (2)		
C2—N1—C3	117.68 (10)	C5—C4—C3	118.24 (7)
C5$^{\mathrm{i}}$—C1—C2	118.28 (10)	C5$^{\mathrm{i}}$—C4—C3	118.24 (7)
C5$^{\mathrm{i}}$—C1—H1	118.1 (14)	C1$^{\mathrm{i}}$—C5—C4	118.91 (11)
C2—C1—H1	123.6 (14)	C1$^{\mathrm{i}}$—C5—H6	121.6 (11)
N1—C2—C1	124.62 (10)	C4—C5—H6	119.5 (11)
N1—C2—C6	113.48 (9)	C2—C6—Cl3	113.05 (7)
C1—C2—C6	121.90 (9)	C2—C6—Cl1	109.44 (7)
N1—C3—N1$^{\mathrm{i}}$	115.46 (13)	Cl3—C6—Cl1	107.82 (6)
N1—C3—C4	122.27 (6)	C2—C6—Cl2	108.79 (7)
N1$^{\mathrm{i}}$—C3—C4	122.27 (6)	Cl3—C6—Cl2	108.72 (6)
C5—C4—C5$^{\mathrm{i}}$	123.51 (14)	Cl1—C6—Cl2	108.95 (6)
C3—N1—C2—C1	0.00 (16)	N1$^{\mathrm{i}}$—C3—C4—C5$^{\mathrm{i}}$	179.98 (8)
C3—N1—C2—C6	−179.95 (8)	C5$^{\mathrm{i}}$—C4—C5—C1$^{\mathrm{i}}$	180.00 (13)
C5$^{\mathrm{i}}$—C1—C2—N1	−0.02 (19)	C3—C4—C5—C1$^{\mathrm{i}}$	0.00 (13)
C5$^{\mathrm{i}}$—C1—C2—C6	179.92 (11)	N1—C2—C6—Cl3	−178.20 (8)
C2—N1—C3—N1$^{\mathrm{i}}$	−179.98 (11)	C1—C2—C6—Cl3	1.85 (14)
C2—N1—C3—C4	0.02 (11)	N1—C2—C6—Cl1	−58.01 (11)
N1—C3—C4—C5	179.98 (8)	C1—C2—C6—Cl1	122.04 (10)
N1$^{\mathrm{i}}$—C3—C4—C5	−0.02 (8)	N1—C2—C6—Cl2	60.93 (11)
N1—C3—C4—C5$^{\mathrm{i}}$	−0.02 (8)	C1—C2—C6—Cl2	−119.02 (10)

Symmetry codes: (i) $-x$, y, $-z+3/2$.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1···Cl3	0.90 (2)	2.63 (2)	3.0085 (12)	106.0 (14)

Fig. 1

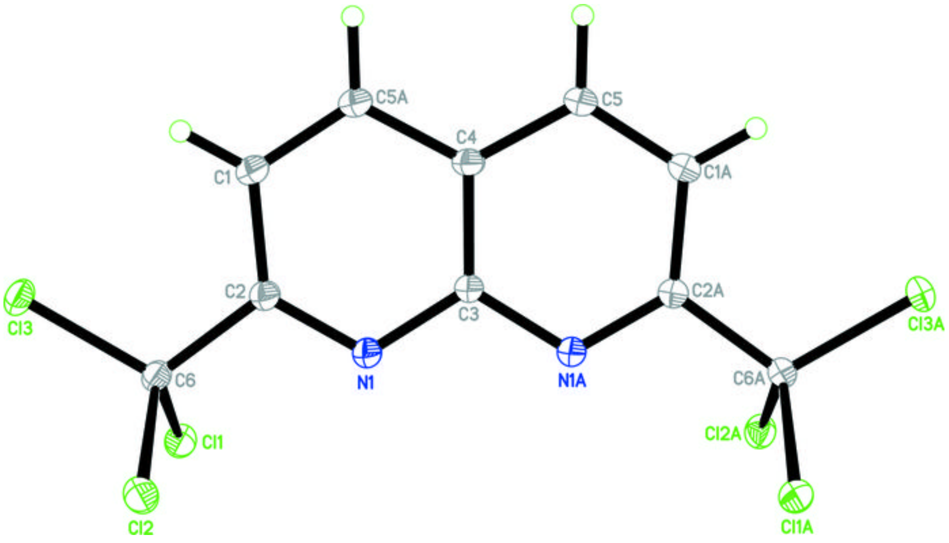
C5A
C5
C4
C1
C1A
C2
C3
C2A
Cl3
Cl3A
C6
N1
N1A
C6A
Cl1
Cl2A
Cl2
Cl1A

Fig. 2