

2,9-Bis(trichloromethyl)-1,10-phenanthroline¹Hoong-Kun Fun,^{a*}§ Suchada Chantrapromma,^{b¶}
Annada C. Maity^c and Shyamaprosad Goswami^c^aX-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, ^bCrystal Materials Research Unit, Department of Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand, and ^cDepartment of Chemistry, Bengal Engineering and Science University, Shibpur, Howrah 711 103, India

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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.042; wR factor = 0.105; data-to-parameter ratio = 34.1.

The asymmetric unit of the title compound, $\text{C}_{14}\text{H}_6\text{Cl}_6\text{N}_2$, contains two crystallographically independent molecules, each of which is slightly twisted from planarity. The dihedral angles between the central ring and the two outer rings are 3.81 (7) and 4.30 (7)° in one molecule, and 4.13 (8) and 4.10 (7)° in the other. In the crystal structure, molecules are interlinked by $\text{C}-\text{Cl}\cdots\text{Cl}$ interactions into sheets parallel to the ac plane. These sheets are stacked along the b axis in such a way that the molecules are antiparallel; they are further connected into a supramolecular network. There are no classical hydrogen bonds. $\text{C}\cdots\text{Cl}$ [3.637 (2) Å], $\text{Cl}\cdots\text{Cl}$ [3.5639 (5)– 3.6807 (8) Å] and $\text{Cl}\cdots\text{N}$ [3.3802 (15)– 3.4093 (15) Å] short contacts and $\pi-\pi$ interactions, with centroid-centroid distances in the range 3.5868 (9)– 3.7844 (9) Å, are observed.

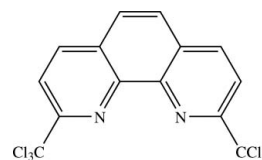
Related literature

For reference bond-length data, see: Allen *et al.* (1987). For background to and applications of 1,10-phenanthroline derivatives, see: Armaroli *et al.* 1992); Beer *et al.* (1993); Emmerling *et al.* (2007); Goswami *et al.* (2007); Wesselinova *et al.* (2009). For the stability of the temperature controller used in the data collection, see: Cosier & Glazer (1986).

¹This 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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Experimental

Crystal data

$\text{C}_{14}\text{H}_6\text{Cl}_6\text{N}_2$
 $M_r = 414.91$
 Monoclinic, $P2_1/c$
 $a = 24.3001$ (6) Å
 $b = 6.8825$ (2) Å
 $c = 20.3461$ (5) Å
 $\beta = 114.689$ (1)°

$V = 3091.74$ (14) Å³
 $Z = 8$
 Mo $K\alpha$ radiation
 $\mu = 1.11$ mm⁻¹
 $T = 100$ K
 $0.59 \times 0.36 \times 0.10$ mm

Data collection

Bruker APEXII CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2005)
 $T_{\min} = 0.561$, $T_{\max} = 0.898$

63570 measured reflections
 13520 independent reflections
 9474 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.054$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.042$
 $wR(F^2) = 0.105$
 $S = 1.06$
 13520 reflections

397 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.60$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.54$ 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: WN2373).

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2,9-Bis(trichloromethyl)-1,10-phenanthroline

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Comment

Trichloromethyl-substituted heterocyclic compounds are of great importance due to their broad spectrum of biological activities. 2,9-Bis(trichloromethyl)-1,10-phenanthroline is used as a potentially robust ligand for metal oxidation catalysts (Beer *et al.*, 1993). 1,10-phenanthroline derivatives also show antitumor (Wesselinova *et al.*, 2009) as well as luminescence properties (Armaroli *et al.*, 1992). Recently a series of trichloromethyl-substituted heterocyclic compounds has been synthesized (Goswami *et al.*, 2007) in good yield using *N*-chlorosuccinimide (NCS) and triphenylphosphine (PPh₃) in carbon tetrachloride. In supramolecular chemistry it is known that the self-association of individual molecules can lead to the formation of highly complex and fascinating supramolecular aggregates if halogen $\cdots\pi$ interactions contribute to the formation of specific motifs (Emmerling *et al.*, 2007). The title compound was synthesized in order to study its supramolecular structure.

The asymmetric unit (Fig. 1) contains two molecules, *A* and *B*, having slight differences in bond lengths and angles. The 1,10-phenanthroline unit is not strictly planar, with dihedral angles between the central ring and the C1–C4/C12/N1 and C7–C11/N2 rings of 3.81 (7) and 4.30 (7)°, respectively, for molecule *A* [the corresponding values for molecule *B* are 4.13 (8) and 4.10 (7)°]. In both molecules, *A* and *B*, none of the Cl atoms of the trichloromethyl substituent is coplanar with the 1,10-phenanthroline ring system. The bond distances adopt normal values (Allen *et al.*, 1987).

In the crystal structure (Fig. 2), non-covalent interactions play a significant role in the three-dimensional supramolecular architecture, in which the molecules are interlinked into sheets parallel to the *ac* plane. These sheets are stacked along the *b* axis in such a way that the molecules are antiparallel. These sheets are further connected into a supramolecular network. There are no classical hydrogen bonds. However, C \cdots Cl [3.637 (2) Å], Cl \cdots Cl [3.5639 (5)–3.6807 (8) Å] and Cl \cdots N [3.3802 (15)–3.4093 (15) Å] short contacts are present. π – π interactions are also observed, with distances of Cg₁ \cdots Cg₂ⁱ = 3.6610 (9) Å, Cg₁ \cdots Cg₂ⁱⁱ = 3.5868 (9) Å, Cg₁ \cdots Cg₃ⁱ = 3.7331 (10) Å, Cg₂ \cdots Cg₃ⁱⁱ = 3.7845 (9) Å, Cg₄ \cdots Cg₅ⁱⁱⁱ = 3.5949 (9) Å, Cg₄ \cdots Cg₅^{iv} = 3.6404 (9) Å, Cg₄ \cdots Cg₆ⁱⁱⁱ = 3.7417 (10) Å and Cg₅ \cdots Cg₆^{iv} = 3.7198 (9) Å (symmetry codes: (i) 1 - *x*, 1 - *y*, 1 - *z*; (ii) 1 - *x*, 2 - *y*, 1 - *z*; (iii) 2 - *x*, -*y*, 1 - *z* and (iv) 2 - *x*, 1 - *y*, 1 - *z*). Cg₁, Cg₂, Cg₃, Cg₄, Cg₅ and Cg₆ are the centroids of the rings C1A–C4A/C12A/N1A, C7A–C11A/N2A, C4A–C7A/C11A–C12A, C1B–C4B/C12B/N1B, C7B–C11B/N2B and C4B–C7B/C11B–C12B, respectively.

Experimental

A mixture of *N*-chlorosuccinimide (500 mg, 4.5 mmol) and triphenylphosphine (500 mg, 4.2 mmol) was moistened with CCl₄ (60 ml) in a round bottom flask and stirred at room temperature for 25 min. A solution of 2,9-dimethyl-1,10-phenanthroline (1 g, 5.2 mmol) was added to the suspension and the reaction mixture was stirred and heated under reflux for 7 h. The solution was cooled and filtered. The evaporated filtrate was washed with saturated aqueous Na₂CO₃ and extracted repeatedly with CHCl₃. Drying over anhydrous Na₂SO₄ was carried out, and the solvent was removed under reduced pressure. The crude product was purified with SiO₂ chromatography (eluted with 1% ethyl acetate in petroleum ether) to give

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the title compound as a white crystalline solid. Colorless plate-shaped single crystals suitable for X-ray structure determination were recrystallized from CH₂Cl₂:hexane (1:10, v/v) by slow evaporation of the solvent at room temperature over the course of a week.

Refinement

All H atoms were placed in calculated positions, with C—H = 0.93 Å, $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Figures

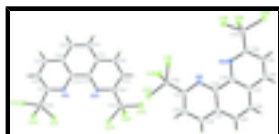


Fig. 1. The molecular structure of the asymmetric unit of the title compound, showing 50% probability displacement ellipsoids and the atom-numbering scheme. Hydrogen atoms are shown as spheres of arbitrary radius.

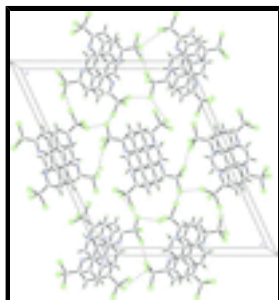


Fig. 2. The crystal packing of the title compound, viewed along the *b* axis. Cl...Cl contacts are shown as dashed lines.

2,9-Bis(trichloromethyl)-1,10-phenanthroline

Crystal data

C₁₄H₆Cl₆N₂

$M_r = 414.91$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 24.3001$ (6) Å

$b = 6.8825$ (2) Å

$c = 20.3461$ (5) Å

$\beta = 114.689$ (1)°

$V = 3091.74$ (14) Å³

$Z = 8$

$F(000) = 1648$

$D_x = 1.783$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 13520 reflections

$\theta = 0.9\text{--}35.0^\circ$

$\mu = 1.11$ mm⁻¹

$T = 100$ K

Plate, colorless

$0.59 \times 0.36 \times 0.10$ mm

Data collection

Bruker APEXII CCD area-detector diffractometer

Radiation source: sealed tube graphite

φ and ω scans

Absorption correction: multi-scan

13520 independent reflections

9474 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.054$

$\theta_{\text{max}} = 35.0^\circ$, $\theta_{\text{min}} = 0.9^\circ$

$h = -34 \rightarrow 39$

(SADABS; Bruker, 2005)
 $T_{\min} = 0.561$, $T_{\max} = 0.898$
 63570 measured reflections

$k = -11 \rightarrow 11$
 $l = -32 \rightarrow 32$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.042$

$wR(F^2) = 0.105$

$S = 1.06$

13520 reflections

397 parameters

0 restraints

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0401P)^2 + 1.5309P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.003$

$\Delta\rho_{\max} = 0.60 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.54 \text{ e } \text{\AA}^{-3}$

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C11A	0.334774 (19)	0.88628 (7)	0.25606 (2)	0.02086 (9)
C12A	0.242550 (19)	0.81155 (7)	0.30569 (2)	0.02246 (9)
C13A	0.301547 (19)	0.49030 (6)	0.26994 (2)	0.01983 (9)
C14A	0.55485 (2)	0.64013 (8)	0.29133 (3)	0.02921 (11)
C15A	0.679947 (19)	0.72747 (7)	0.37576 (2)	0.02217 (9)
C16A	0.59362 (2)	1.03890 (7)	0.31954 (3)	0.02459 (10)
N1A	0.41730 (6)	0.7423 (2)	0.39518 (8)	0.0142 (3)
N2A	0.53663 (6)	0.7752 (2)	0.41504 (8)	0.0150 (3)
C1A	0.36107 (7)	0.7161 (2)	0.38719 (9)	0.0148 (3)
C2A	0.34427 (8)	0.6758 (3)	0.44406 (9)	0.0170 (3)
H2AA	0.3041	0.6512	0.4352	0.020*
C3A	0.38910 (8)	0.6741 (3)	0.51314 (10)	0.0164 (3)
H3AA	0.3795	0.6497	0.5521	0.020*

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C4A	0.44947 (7)	0.7094 (2)	0.52491 (9)	0.0150 (3)
C5A	0.49733 (8)	0.7192 (2)	0.59632 (9)	0.0164 (3)
H5AA	0.4888	0.6984	0.6363	0.020*
C6A	0.55494 (8)	0.7584 (2)	0.60616 (9)	0.0165 (3)
H6AA	0.5852	0.7696	0.6528	0.020*
C7A	0.56969 (7)	0.7830 (2)	0.54558 (9)	0.0145 (3)
C8A	0.62916 (7)	0.8199 (2)	0.55361 (9)	0.0165 (3)
H8AA	0.6601	0.8368	0.5995	0.020*
C9A	0.64155 (8)	0.8310 (3)	0.49404 (10)	0.0170 (3)
H9AA	0.6805	0.8572	0.4985	0.020*
C10A	0.59347 (7)	0.8014 (2)	0.42572 (9)	0.0150 (3)
C11A	0.52411 (7)	0.7680 (2)	0.47387 (9)	0.0138 (3)
C12A	0.46148 (7)	0.7381 (2)	0.46339 (9)	0.0133 (3)
C13A	0.31304 (7)	0.7276 (2)	0.30927 (9)	0.0155 (3)
C14A	0.60462 (7)	0.8011 (3)	0.35693 (9)	0.0161 (3)
Cl1B	0.83130 (2)	0.36601 (7)	0.57545 (3)	0.02271 (9)
Cl2B	0.74008 (2)	0.25940 (8)	0.43624 (3)	0.02710 (11)
Cl3B	0.807802 (19)	-0.03889 (6)	0.53631 (2)	0.02007 (9)
Cl4B	1.05051 (2)	0.13826 (8)	0.76066 (2)	0.02696 (11)
Cl5B	1.177123 (19)	0.19488 (7)	0.80031 (2)	0.02159 (9)
Cl6B	1.09840 (2)	0.52724 (7)	0.77611 (2)	0.02242 (9)
N1B	0.91644 (6)	0.2259 (2)	0.52083 (8)	0.0149 (3)
N2B	1.03543 (6)	0.2648 (2)	0.62009 (8)	0.0143 (3)
C1B	0.86038 (7)	0.1972 (2)	0.47266 (9)	0.0150 (3)
C2B	0.84362 (8)	0.1635 (3)	0.39875 (9)	0.0175 (3)
H2BA	0.8037	0.1363	0.3676	0.021*
C3B	0.88840 (8)	0.1721 (3)	0.37394 (9)	0.0170 (3)
H3BA	0.8789	0.1524	0.3252	0.020*
C4B	0.94853 (7)	0.2108 (2)	0.42278 (9)	0.0149 (3)
C5B	0.99605 (8)	0.2322 (2)	0.39896 (9)	0.0163 (3)
H5BA	0.9876	0.2163	0.3503	0.020*
C6B	1.05313 (8)	0.2754 (3)	0.44695 (10)	0.0170 (3)
H6BA	1.0831	0.2953	0.4305	0.020*
C7B	1.06805 (7)	0.2908 (2)	0.52255 (9)	0.0148 (3)
C8B	1.12731 (8)	0.3302 (2)	0.57408 (10)	0.0166 (3)
H8BA	1.1580	0.3539	0.5591	0.020*
C9B	1.13989 (8)	0.3336 (3)	0.64646 (10)	0.0171 (3)
H9BA	1.1786	0.3611	0.6811	0.021*
C10B	1.09210 (7)	0.2938 (2)	0.66644 (9)	0.0146 (3)
C11B	1.02291 (7)	0.2647 (2)	0.54862 (9)	0.0139 (3)
C12B	0.96058 (7)	0.2317 (2)	0.49680 (9)	0.0137 (3)
C13B	0.81276 (7)	0.1983 (3)	0.50346 (9)	0.0159 (3)
C14B	1.10337 (7)	0.2871 (3)	0.74605 (9)	0.0159 (3)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1A	0.02068 (19)	0.0238 (2)	0.01618 (18)	-0.00383 (15)	0.00582 (15)	0.00361 (16)

C12A	0.01529 (18)	0.0299 (2)	0.0216 (2)	0.00706 (15)	0.00717 (16)	0.00172 (17)
C13A	0.01796 (18)	0.01901 (19)	0.0200 (2)	-0.00169 (14)	0.00547 (15)	-0.00399 (15)
C14A	0.0277 (2)	0.0436 (3)	0.0195 (2)	-0.0168 (2)	0.01296 (18)	-0.0111 (2)
C15A	0.01725 (19)	0.0279 (2)	0.0222 (2)	0.00705 (15)	0.00910 (16)	0.00110 (17)
C16A	0.0264 (2)	0.0251 (2)	0.0277 (2)	0.00896 (17)	0.01677 (19)	0.01139 (18)
N1A	0.0136 (6)	0.0136 (6)	0.0153 (6)	0.0006 (5)	0.0059 (5)	0.0001 (5)
N2A	0.0151 (6)	0.0146 (6)	0.0156 (6)	0.0006 (5)	0.0068 (5)	0.0008 (5)
C1A	0.0141 (7)	0.0134 (7)	0.0159 (7)	0.0004 (5)	0.0054 (6)	-0.0016 (6)
C2A	0.0139 (7)	0.0192 (8)	0.0185 (8)	-0.0009 (6)	0.0074 (6)	0.0001 (6)
C3A	0.0185 (8)	0.0171 (8)	0.0165 (7)	0.0004 (6)	0.0101 (6)	0.0003 (6)
C4A	0.0167 (7)	0.0123 (7)	0.0170 (7)	0.0019 (5)	0.0081 (6)	0.0004 (6)
C5A	0.0198 (8)	0.0158 (7)	0.0136 (7)	0.0021 (6)	0.0071 (6)	0.0005 (6)
C6A	0.0181 (8)	0.0151 (7)	0.0149 (7)	0.0012 (6)	0.0055 (6)	-0.0005 (6)
C7A	0.0149 (7)	0.0126 (7)	0.0156 (7)	0.0003 (5)	0.0058 (6)	0.0007 (6)
C8A	0.0150 (7)	0.0157 (7)	0.0165 (7)	-0.0015 (5)	0.0042 (6)	-0.0015 (6)
C9A	0.0145 (7)	0.0179 (8)	0.0183 (8)	-0.0027 (6)	0.0065 (6)	-0.0001 (6)
C10A	0.0149 (7)	0.0149 (7)	0.0154 (7)	0.0006 (5)	0.0063 (6)	0.0018 (6)
C11A	0.0136 (7)	0.0129 (7)	0.0148 (7)	0.0010 (5)	0.0057 (6)	0.0005 (6)
C12A	0.0148 (7)	0.0113 (7)	0.0143 (7)	0.0008 (5)	0.0065 (6)	-0.0002 (6)
C13A	0.0136 (7)	0.0162 (7)	0.0171 (7)	-0.0001 (5)	0.0070 (6)	-0.0006 (6)
C14A	0.0141 (7)	0.0182 (8)	0.0162 (7)	-0.0004 (5)	0.0066 (6)	0.0013 (6)
C11B	0.0236 (2)	0.0236 (2)	0.0259 (2)	-0.00425 (16)	0.01529 (18)	-0.00761 (17)
C12B	0.01594 (19)	0.0422 (3)	0.0212 (2)	0.01094 (18)	0.00581 (16)	0.0069 (2)
C13B	0.01827 (18)	0.01960 (19)	0.0235 (2)	-0.00159 (14)	0.00980 (16)	0.00239 (16)
C14B	0.0246 (2)	0.0411 (3)	0.0172 (2)	-0.01349 (19)	0.01067 (17)	-0.00318 (19)
C15B	0.01830 (19)	0.0254 (2)	0.0204 (2)	0.00703 (15)	0.00734 (16)	0.00478 (17)
C16B	0.0233 (2)	0.0233 (2)	0.01712 (19)	0.00652 (15)	0.00497 (16)	-0.00445 (16)
N1B	0.0153 (6)	0.0140 (6)	0.0156 (6)	0.0010 (5)	0.0067 (5)	0.0008 (5)
N2B	0.0145 (6)	0.0138 (6)	0.0152 (6)	0.0006 (5)	0.0068 (5)	-0.0012 (5)
C1B	0.0150 (7)	0.0141 (7)	0.0160 (7)	0.0011 (5)	0.0066 (6)	0.0013 (6)
C2B	0.0172 (8)	0.0176 (8)	0.0160 (7)	-0.0008 (6)	0.0053 (6)	0.0001 (6)
C3B	0.0204 (8)	0.0161 (8)	0.0139 (7)	0.0008 (6)	0.0064 (6)	-0.0006 (6)
C4B	0.0172 (7)	0.0121 (7)	0.0148 (7)	0.0016 (5)	0.0061 (6)	0.0016 (6)
C5B	0.0208 (8)	0.0166 (8)	0.0138 (7)	0.0020 (6)	0.0095 (6)	0.0021 (6)
C6B	0.0192 (8)	0.0164 (8)	0.0191 (8)	0.0018 (6)	0.0117 (7)	0.0026 (6)
C7B	0.0157 (7)	0.0118 (7)	0.0177 (7)	0.0005 (5)	0.0077 (6)	0.0002 (6)
C8B	0.0158 (7)	0.0161 (7)	0.0209 (8)	-0.0002 (5)	0.0105 (6)	-0.0001 (6)
C9B	0.0150 (7)	0.0173 (8)	0.0187 (8)	-0.0016 (6)	0.0068 (6)	-0.0020 (6)
C10B	0.0158 (7)	0.0138 (7)	0.0152 (7)	-0.0003 (5)	0.0074 (6)	-0.0017 (6)
C11B	0.0158 (7)	0.0111 (7)	0.0160 (7)	0.0005 (5)	0.0078 (6)	-0.0003 (6)
C12B	0.0149 (7)	0.0116 (7)	0.0154 (7)	0.0007 (5)	0.0070 (6)	0.0000 (6)
C13B	0.0127 (7)	0.0174 (7)	0.0161 (7)	0.0008 (5)	0.0045 (6)	0.0010 (6)
C14B	0.0124 (7)	0.0198 (8)	0.0145 (7)	-0.0002 (5)	0.0046 (6)	-0.0013 (6)

Geometric parameters (Å, °)

C11A—C13A	1.7667 (18)	C11B—C13B	1.7690 (18)
C12A—C13A	1.7800 (17)	C12B—C13B	1.7746 (17)
C13A—C13A	1.7887 (18)	C13B—C13B	1.7870 (18)

supplementary materials

C14A—C14A	1.7673 (18)	C14B—C14B	1.7625 (18)
C15A—C14A	1.7800 (17)	C15B—C14B	1.7834 (17)
C16A—C14A	1.7772 (18)	C16B—C14B	1.7839 (18)
N1A—C1A	1.319 (2)	N1B—C1B	1.319 (2)
N1A—C12A	1.354 (2)	N1B—C12B	1.352 (2)
N2A—C10A	1.318 (2)	N2B—C10B	1.319 (2)
N2A—C11A	1.353 (2)	N2B—C11B	1.355 (2)
C1A—C2A	1.406 (2)	C1B—C2B	1.403 (2)
C1A—C13A	1.529 (2)	C1B—C13B	1.529 (2)
C2A—C3A	1.372 (2)	C2B—C3B	1.379 (3)
C2A—H2AA	0.9300	C2B—H2BA	0.9300
C3A—C4A	1.405 (2)	C3B—C4B	1.408 (2)
C3A—H3AA	0.9300	C3B—H3BA	0.9300
C4A—C12A	1.413 (2)	C4B—C12B	1.416 (2)
C4A—C5A	1.434 (2)	C4B—C5B	1.434 (2)
C5A—C6A	1.356 (2)	C5B—C6B	1.354 (2)
C5A—H5AA	0.9300	C5B—H5BA	0.9300
C6A—C7A	1.430 (2)	C6B—C7B	1.429 (2)
C6A—H6AA	0.9300	C6B—H6BA	0.9300
C7A—C8A	1.408 (2)	C7B—C8B	1.409 (2)
C7A—C11A	1.420 (2)	C7B—C11B	1.415 (2)
C8A—C9A	1.368 (3)	C8B—C9B	1.373 (3)
C8A—H8AA	0.9300	C8B—H8BA	0.9300
C9A—C10A	1.408 (2)	C9B—C10B	1.408 (2)
C9A—H9AA	0.9300	C9B—H9BA	0.9300
C10A—C14A	1.533 (2)	C10B—C14B	1.526 (2)
C11A—C12A	1.461 (2)	C11B—C12B	1.457 (2)
C1A—N1A—C12A	117.40 (15)	C1B—N1B—C12B	117.69 (15)
C10A—N2A—C11A	117.80 (14)	C10B—N2B—C11B	117.80 (15)
N1A—C1A—C2A	124.57 (15)	N1B—C1B—C2B	124.64 (16)
N1A—C1A—C13A	115.06 (15)	N1B—C1B—C13B	114.72 (15)
C2A—C1A—C13A	120.35 (15)	C2B—C1B—C13B	120.63 (15)
C3A—C2A—C1A	117.74 (15)	C3B—C2B—C1B	117.71 (16)
C3A—C2A—H2AA	121.1	C3B—C2B—H2BA	121.1
C1A—C2A—H2AA	121.1	C1B—C2B—H2BA	121.1
C2A—C3A—C4A	119.90 (16)	C2B—C3B—C4B	119.67 (16)
C2A—C3A—H3AA	120.0	C2B—C3B—H3BA	120.2
C4A—C3A—H3AA	120.0	C4B—C3B—H3BA	120.2
C3A—C4A—C12A	117.41 (15)	C3B—C4B—C12B	117.55 (16)
C3A—C4A—C5A	121.86 (16)	C3B—C4B—C5B	121.71 (16)
C12A—C4A—C5A	120.72 (15)	C12B—C4B—C5B	120.73 (15)
C6A—C5A—C4A	120.61 (16)	C6B—C5B—C4B	120.36 (16)
C6A—C5A—H5AA	119.7	C6B—C5B—H5BA	119.8
C4A—C5A—H5AA	119.7	C4B—C5B—H5BA	119.8
C5A—C6A—C7A	120.75 (16)	C5B—C6B—C7B	121.01 (16)
C5A—C6A—H6AA	119.6	C5B—C6B—H6BA	119.5
C7A—C6A—H6AA	119.6	C7B—C6B—H6BA	119.5
C8A—C7A—C11A	117.04 (16)	C8B—C7B—C11B	117.17 (16)
C8A—C7A—C6A	122.40 (15)	C8B—C7B—C6B	122.41 (16)

C11A—C7A—C6A	120.55 (15)	C11B—C7B—C6B	120.41 (15)
C9A—C8A—C7A	120.16 (16)	C9B—C8B—C7B	120.10 (16)
C9A—C8A—H8AA	119.9	C9B—C8B—H8BA	119.9
C7A—C8A—H8AA	119.9	C7B—C8B—H8BA	119.9
C8A—C9A—C10A	117.89 (16)	C8B—C9B—C10B	117.73 (15)
C8A—C9A—H9AA	121.1	C8B—C9B—H9BA	121.1
C10A—C9A—H9AA	121.1	C10B—C9B—H9BA	121.1
N2A—C10A—C9A	124.26 (16)	N2B—C10B—C9B	124.30 (16)
N2A—C10A—C14A	114.98 (14)	N2B—C10B—C14B	115.15 (15)
C9A—C10A—C14A	120.75 (15)	C9B—C10B—C14B	120.54 (15)
N2A—C11A—C7A	122.63 (15)	N2B—C11B—C7B	122.70 (15)
N2A—C11A—C12A	118.78 (14)	N2B—C11B—C12B	118.42 (15)
C7A—C11A—C12A	118.57 (15)	C7B—C11B—C12B	118.86 (15)
N1A—C12A—C4A	122.83 (15)	N1B—C12B—C4B	122.58 (15)
N1A—C12A—C11A	118.56 (15)	N1B—C12B—C11B	118.99 (15)
C4A—C12A—C11A	118.60 (14)	C4B—C12B—C11B	118.42 (15)
C1A—C13A—C11A	111.92 (12)	C1B—C13B—C11B	111.59 (12)
C1A—C13A—C12A	111.35 (12)	C1B—C13B—C12B	111.42 (12)
C11A—C13A—C12A	107.65 (9)	C11B—C13B—C12B	108.11 (9)
C1A—C13A—C13A	109.01 (11)	C1B—C13B—C13B	109.25 (11)
C11A—C13A—C13A	108.73 (9)	C11B—C13B—C13B	108.67 (9)
C12A—C13A—C13A	108.08 (9)	C12B—C13B—C13B	107.68 (9)
C10A—C14A—C14A	111.35 (12)	C10B—C14B—C14B	112.10 (11)
C10A—C14A—C16A	109.72 (12)	C10B—C14B—C15B	110.87 (12)
C14A—C14A—C16A	108.72 (9)	C14B—C14B—C15B	107.66 (9)
C10A—C14A—C15A	111.23 (11)	C10B—C14B—C16B	109.13 (12)
C14A—C14A—C15A	107.56 (10)	C14B—C14B—C16B	108.79 (9)
C16A—C14A—C15A	108.16 (9)	C15B—C14B—C16B	108.17 (9)
C12A—N1A—C1A—C2A	-3.0 (2)	C12B—N1B—C1B—C2B	3.0 (2)
C12A—N1A—C1A—C13A	178.49 (14)	C12B—N1B—C1B—C13B	-178.33 (14)
N1A—C1A—C2A—C3A	3.8 (3)	N1B—C1B—C2B—C3B	-3.9 (3)
C13A—C1A—C2A—C3A	-177.75 (15)	C13B—C1B—C2B—C3B	177.48 (15)
C1A—C2A—C3A—C4A	-0.8 (3)	C1B—C2B—C3B—C4B	0.9 (3)
C2A—C3A—C4A—C12A	-2.4 (2)	C2B—C3B—C4B—C12B	2.5 (2)
C2A—C3A—C4A—C5A	176.67 (16)	C2B—C3B—C4B—C5B	-176.39 (16)
C3A—C4A—C5A—C6A	-178.26 (16)	C3B—C4B—C5B—C6B	177.95 (17)
C12A—C4A—C5A—C6A	0.8 (3)	C12B—C4B—C5B—C6B	-0.9 (3)
C4A—C5A—C6A—C7A	-2.6 (3)	C4B—C5B—C6B—C7B	3.2 (3)
C5A—C6A—C7A—C8A	-178.65 (17)	C5B—C6B—C7B—C8B	178.21 (17)
C5A—C6A—C7A—C11A	0.5 (3)	C5B—C6B—C7B—C11B	-1.2 (3)
C11A—C7A—C8A—C9A	-3.0 (2)	C11B—C7B—C8B—C9B	2.8 (2)
C6A—C7A—C8A—C9A	176.17 (16)	C6B—C7B—C8B—C9B	-176.64 (16)
C7A—C8A—C9A—C10A	-1.0 (3)	C7B—C8B—C9B—C10B	0.9 (3)
C11A—N2A—C10A—C9A	-2.9 (3)	C11B—N2B—C10B—C9B	3.1 (2)
C11A—N2A—C10A—C14A	178.02 (14)	C11B—N2B—C10B—C14B	-178.51 (14)
C8A—C9A—C10A—N2A	4.2 (3)	C8B—C9B—C10B—N2B	-4.1 (3)
C8A—C9A—C10A—C14A	-176.80 (16)	C8B—C9B—C10B—C14B	177.53 (16)
C10A—N2A—C11A—C7A	-1.5 (2)	C10B—N2B—C11B—C7B	1.2 (2)
C10A—N2A—C11A—C12A	-179.67 (15)	C10B—N2B—C11B—C12B	179.64 (15)

supplementary materials

C8A—C7A—C11A—N2A	4.4 (2)	C8B—C7B—C11B—N2B	-4.0 (2)
C6A—C7A—C11A—N2A	-174.77 (15)	C6B—C7B—C11B—N2B	175.45 (15)
C8A—C7A—C11A—C12A	-177.43 (15)	C8B—C7B—C11B—C12B	177.49 (15)
C6A—C7A—C11A—C12A	3.4 (2)	C6B—C7B—C11B—C12B	-3.0 (2)
C1A—N1A—C12A—C4A	-0.7 (2)	C1B—N1B—C12B—C4B	0.9 (2)
C1A—N1A—C12A—C11A	-179.56 (15)	C1B—N1B—C12B—C11B	179.52 (15)
C3A—C4A—C12A—N1A	3.3 (2)	C3B—C4B—C12B—N1B	-3.6 (2)
C5A—C4A—C12A—N1A	-175.76 (15)	C5B—C4B—C12B—N1B	175.35 (15)
C3A—C4A—C12A—C11A	-177.82 (15)	C3B—C4B—C12B—C11B	177.77 (15)
C5A—C4A—C12A—C11A	3.1 (2)	C5B—C4B—C12B—C11B	-3.3 (2)
N2A—C11A—C12A—N1A	-8.0 (2)	N2B—C11B—C12B—N1B	7.9 (2)
C7A—C11A—C12A—N1A	173.80 (15)	C7B—C11B—C12B—N1B	-173.53 (15)
N2A—C11A—C12A—C4A	173.16 (15)	N2B—C11B—C12B—C4B	-173.35 (15)
C7A—C11A—C12A—C4A	-5.1 (2)	C7B—C11B—C12B—C4B	5.2 (2)
N1A—C1A—C13A—C11A	-30.18 (18)	N1B—C1B—C13B—C11B	34.79 (18)
C2A—C1A—C13A—C11A	151.19 (14)	C2B—C1B—C13B—C11B	-146.50 (14)
N1A—C1A—C13A—C12A	-150.73 (13)	N1B—C1B—C13B—C12B	155.75 (13)
C2A—C1A—C13A—C12A	30.6 (2)	C2B—C1B—C13B—C12B	-25.5 (2)
N1A—C1A—C13A—C13A	90.12 (16)	N1B—C1B—C13B—C13B	-85.40 (16)
C2A—C1A—C13A—C13A	-88.50 (17)	C2B—C1B—C13B—C13B	93.31 (17)
N2A—C10A—C14A—C14A	-33.57 (19)	N2B—C10B—C14B—C14B	27.50 (19)
C9A—C10A—C14A—C14A	147.36 (14)	C9B—C10B—C14B—C14B	-154.01 (14)
N2A—C10A—C14A—C16A	86.84 (16)	N2B—C10B—C14B—C15B	147.86 (13)
C9A—C10A—C14A—C16A	-92.23 (17)	C9B—C10B—C14B—C15B	-33.7 (2)
N2A—C10A—C14A—C15A	-153.53 (13)	N2B—C10B—C14B—C16B	-93.09 (15)
C9A—C10A—C14A—C15A	27.4 (2)	C9B—C10B—C14B—C16B	85.40 (17)

Fig. 1

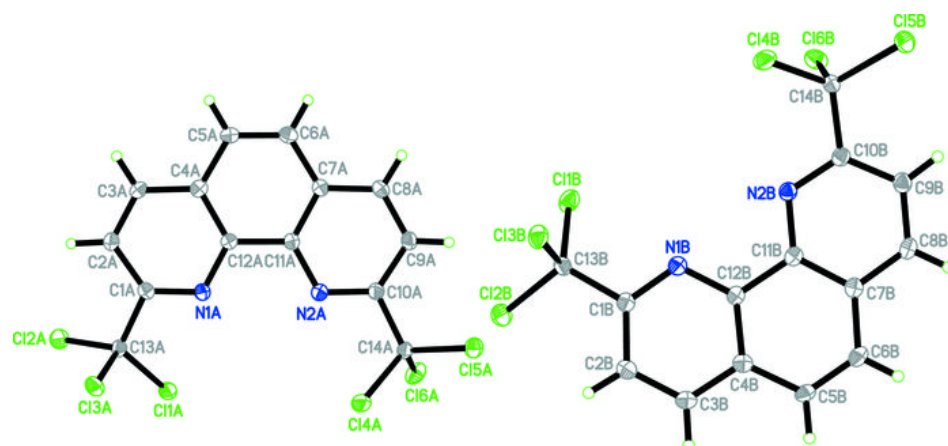


Fig. 2

