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Structural and luminescent properties of co-crystals of tetraiodoethylene with two azaphenanthrenes

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CCDC references: 1899078; 1899077

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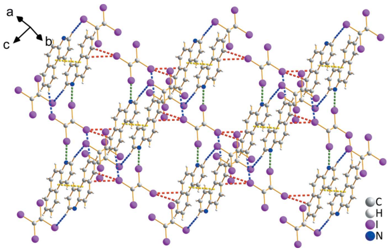
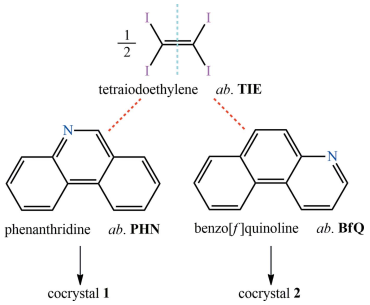
Two new co-crystals, tetraiodoethylene–phenanthridine (1/2), $0.5C_2I_4\cdot C_{13}H_9N$ (**1**) and tetraiodoethylene–benzo[*f*]quinoline (1/2), $0.5C_2I_4\cdot C_{13}H_9N$ (**2**), were obtained from tetraiodoethylene and azaphenanthrenes, and characterized by IR and fluorescence spectroscopy, elemental analysis and X-ray crystallography. In the crystal structures, $C—I\cdots\pi$ and $C—I\cdots N$ halogen bonds link the independent molecules into one-dimensional chains and two-dimensional networks with subloops. In addition, the planar azaphenanthrenes lend themselves to π–π stacking and $C—H\cdots\pi$ interactions, leading to a diversity of supramolecular three-dimensional structural motifs being formed by these interactions. Luminescence studies show that co-crystals **1** and **2** exhibit distinctly different luminescence properties in the solid state at room temperature.



1. Chemical context

A halogen bond is an attractive non-covalent interaction between an electrophilic region in a covalently bonded halogen atom and a Lewis base. Halogen bonding (XB) is a powerful tool to assemble supramolecular materials and to promote chemical or biological molecular recognition (Desiraju *et al.*, 2013; Cavallo *et al.*, 2016; Gilday *et al.*, 2015; Wang *et al.*, 2016). Over the past few years, XB has been used successfully to assemble luminescent co-crystals (Liu *et al.*, 2017*a*; d'Agostino *et al.*, 2015; Ventura *et al.*, 2014; Bolton *et al.*, 2011). XB can play multiple roles in co-crystals, for example, as cement to assemble XB donors and acceptors together (Metrangolo *et al.*, 2005), and, importantly, as a heavy-atom source to enhance phosphorescence or delayed fluorescence by efficient spin-orbital coupling (Gao *et al.*, 2012). Phosphorescence or delayed fluorescence materials are very popular for preparing light devices because of the higher internal quantum efficiency of triplet excitons (Brown *et al.*, 1993; Baldo *et al.*, 1999).



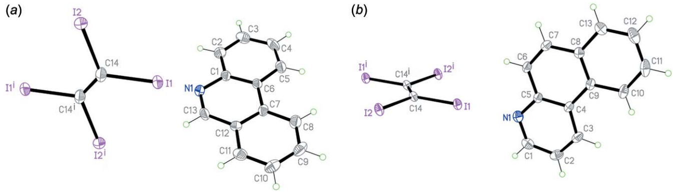


Figure 1
The molecular structures of co-crystals **1** and **2**, showing the atom-labelling scheme and displacement ellipsoids at the 30% probability level [Symmetry codes: (i) $-x + \frac{3}{2}, -y + \frac{3}{2}, -z + 1$ for co-crystal **1**; (i) $-x, -y + 1, -z + 2$ for co-crystal **2**].

Nitrogen heteroaromatic rings are a common type of luminescence or luminescent precursor materials. However, in general, it is difficult to use them to generate phosphorescence or delayed fluorescence. Haloperfluorobenzenes, as XB donors, have been used in attempts to assemble luminescence co-crystals with azaphenanthrenes (Gao *et al.*, 2017; Wang *et al.*, 2014, 2016; Wang & Jin, 2017; Liu *et al.*, 2017*b*). We report herein the use of tetraiodoethylene (TIE) as a new XB donor in the assembly of co-crystals with two different azaphenanthrenes, namely phenanthridine (PHN) and benzo[*f*]quinoline (BfQ), which is expected to tune their luminescence behaviour *via* a change of the co-crystal structures. Single crystal X-ray diffraction (XRD) data reveal that the two co-crystals of TIE with PHN and BfQ reported here have interesting structural properties and exhibit different luminescence behaviour from previous reports. TIE as a quadridentate XB donor allows the formation of three-dimensional halogen-bonded networks with XB acceptors, PHN and BfQ. Using the conventional solution-based method, yellow co-crystals suitable for XRD measurement were obtained. The crystal structures of the co-crystals are mainly constructed by C—I$\cdots\pi$ and C—I$\cdots$N halogen bonds. Other multiple intermolecular interactions, such as π–π stacking, C—H$\cdots\pi$, C—H$\cdots$I as well as C—H$\cdots$H—C interactions, are also observed in the co-crystals.

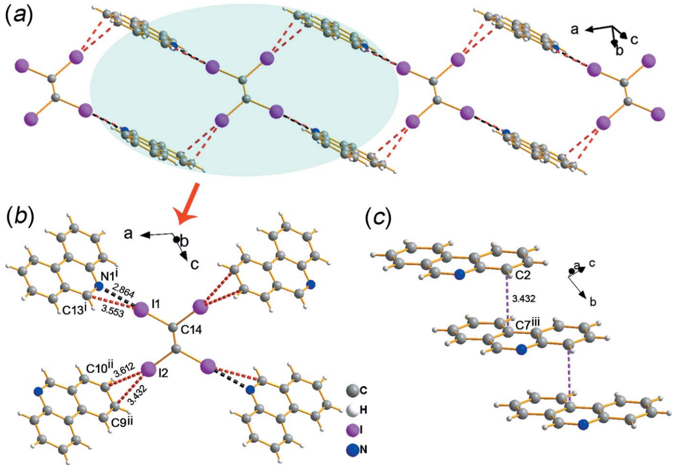


Figure 2
Crystal packing of **1**. (*a*) The two-dimensional network structure formed by C—I$\cdots$N, C—I$\cdots$C and C—I$\cdots\pi$ halogen bonds. (*b*) The structural motif extracted from the two-dimensional network. (*c*) The π–π stacking interactions extracted from the three-dimensional structure.

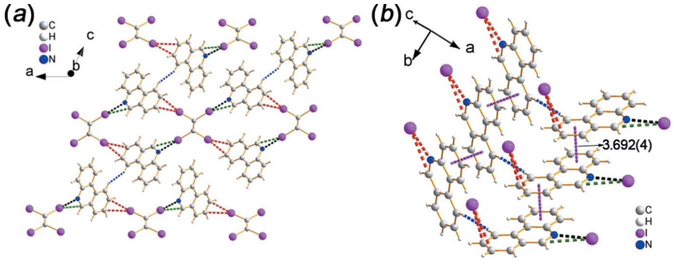


Figure 3
Crystal packing of **1**. (*a*) The two-dimensional network extends along the *a*- and *c*-axis directions, and two directions connect by C—H$\cdots$H—C interactions. (*b*) The two-dimensional networks connected by π–π stacking and C—H$\cdots$H—C interactions to form a three-dimensional structure.

2. Structural commentary

The asymmetric units of co-crystals **1** and **2** each comprise one half TIE molecule lying about an inversion centre and one PHN or BfQ molecule in a general position, hence the co-crystals have a 1:2 stoichiometry (Fig. 1). Co-crystal **1** crystallizes in the monoclinic space group $C2/c$ while **2** crystallizes in the triclinic space group $P\overline{1}$.

3. Supramolecular features

In the crystal of **1**, C—I$\cdots$N, C—I$\cdots$C and C—I$\cdots\pi$ halogen bonds lead to the formation of a two-dimensional network structure in which the rectangular motif has a $D\cdots 2A\cdots D\cdots 2A$ arrangement, as shown in Fig. 2*a*. The I1 atom of the TIE molecule interacts with the N1 atom of a PHN molecule, forming a C1—I1$\cdots$N1 halogen bond (Fig. 2*b*). The I1$\cdots$N1^{i} distance is 2.864 (7) Å and the corresponding C14–I1$\cdots$N1^{i} angle is 172.8 (2)° [symmetry code: (i) $x, y + 1, z$]. The strong C14—I1$\cdots$N1 halogen bond results in a I1$\cdots$C13^{i} distance [3.553 (8) Å] shorter than the sum of the van der Waals radii, which indicates a C1—I1$\cdots$C13 halogen inter-

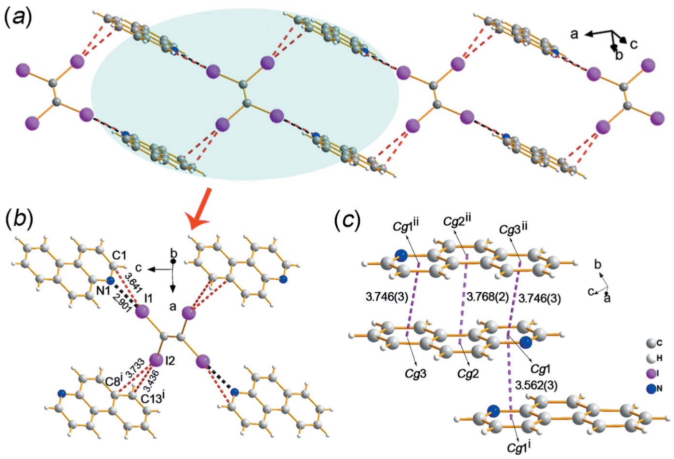


Figure 4
Crystal packing of **2**. (*a*) The two-dimensional network structure formed by C—I$\cdots$N and C—I$\cdots\pi$ halogen bonds. (*b*) The structural motif of the two-dimensional network. (*c*) The π–π stacking interactions in the three-dimensional structure.

Table 1
Hydrogen-bond geometry (Å, °) for (2).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C1—H1$\cdots$I2^{i}	0.93	3.16	4.019 (5)	155
C6—H6$\cdots$I1	0.93	3.31	3.945 (4)	127

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

action. In addition, the C14—I2$\cdots$C9^{ii}/C10^{ii} C—I$\cdots\pi$ separations [Fig. 2*b*; symmetry code: (ii) $\frac{1}{2} - x, \frac{1}{2} - y, z$] are 3.432 (9) and 3.612 (8) Å, and the corresponding bond angles are 165.6 (2) and 156.7 (2)°, respectively. Furthermore, π–π stacking [Fig. 2*c*; $Cg1\cdots Cg2^{iii}$ = 3.692 (4) Å, $Cg3\cdots Cg2^{iii}$ = 3.626 (4) Å; $Cg1$, $Cg2$ and $Cg3$ are the centroids of rings C1–C6, C7–C12 and N1/C1/C6/C7/C12/C13, respectively; symmetry code: (iii) $x, -1 + y, z$] and C—H$\cdots$H—C interactions between two adjacent PHN molecules contribute to the extension of the two-dimensional network into a three-dimensional supramolecular structure (Fig. 3).

The two-dimensional network of co-crystal **2** is similar to that of **1**, as shown in Fig. 4*a*. Both of them are constructed by the same halogen-bonded synthon, *i.e.*, C—I$\cdots$N, C—I$\cdots$C and C—I$\cdots\pi$ halogen bonds, but the bonding characteristics are slightly different. In general, the distances of the C—I$\cdots$N, C—I$\cdots$C and C—I$\cdots\pi$ interactions [I1$\cdots$N1 = 2.901 (4), I1$\cdots$C1 = 3.641 (5), I2$\cdots$C13($-x, 1 - y, 1 - z$) = 3.436 (5) and I2$\cdots$C8($-x, 1 - y, 1 - z$) = 3.733 (4) Å, respectively] in co-crystal **2** are all a little longer (0.004–0.121 Å) than in **1** (Fig. 4*b*). In addition, the two-dimensional network (Fig. 5) is extended to a three-dimensional supramolecular structure by π–π stacking (Fig. 4*c* and 5; $Cg1\cdots Cg1^{i}$ = 3.562 (3) Å, $Cg1\cdots Cg2^{ii}$ = 3.963 (2) Å, $Cg1\cdots Cg3^{ii}$ = 3.746 (3) Å, $Cg2\cdots Cg2^{ii}$ = 3.768 (2) Å; $Cg1$, $Cg2$ and $Cg3$ are the centroids of rings N1/C1–C5, C4–C9 and C8–C13, respectively; symmetry codes: (i) $1 - x, 1 - y, 1 - z$; (ii) $1 - x, -y, 1 - z$] and C—H$\cdots$I hydrogen bonds (Table 1).

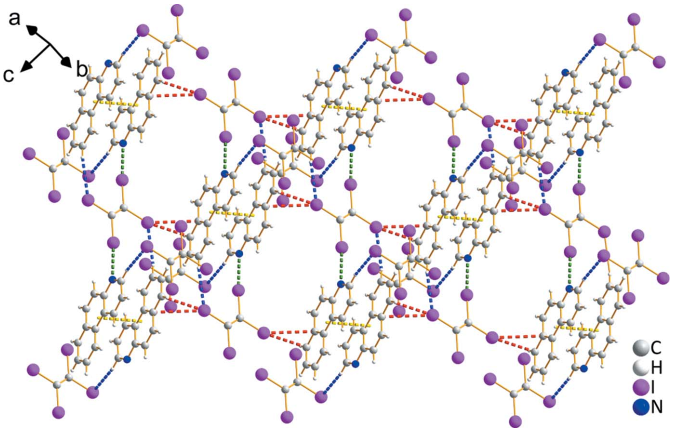


Figure 5
Crystal packing of **2**. The two-dimensional network extends along two directions, by C—I2$\cdots$H7 interactions and π–π stacking in one direction, and by C—I2$\cdots$H1 interactions and π–π stacking in the other.

Table 2
Phosphorescent characteristics of co-crystals at room temperature.

		TIE–PHN	TIE–BfQ
Total luminescent spectra	λ_{ex}/nm	300	300
	λ_{em} /nm	375, 484, 578	368, 438, 452, 480
	$\tau_{average}$/ ns	11.49	9.29
DF and phosphorescent spectra	λ_{ex}/nm	330	330
	λ_{em} /nm	375, 489, 600	430, 489, 596, 654
	$\tau_{average}$/ µs	4.36	6.45

4. Powder X-ray diffraction pattern

The powder X-ray diffraction (PXRD) experiments were carried out for the title co-crystals using a Bruker D8-ADVANCE X-ray diffractometer (Cu $K\alpha$, λ = 1.5418 Å) in the 2θ range of 5 to 50°. As shown in Fig. 6, the experimental patterns for **1** and **2** match well with the spectra simulated from the XRD data, which confirms the purity of **1** and **2**.

5. Luminescence behavior of co-crystals 1 and 2

As shown in Fig. 7, the two co-crystals fluoresce with some vibrational fine structure (see also spectroscopic data in Table 2). The two co-crystals also show delayed fluorescence (Fig. 8). For both co-crystals, the emission bands in the region of 450–480 nm should be relative to the π–π stacking patterns. Luminescence from the excimer is possible because of the close π–π stacking distances as shown in Figs. 2–5, besides luminescence from a monomer. Furthermore, TIE–PHN and TIE–BfQ produce weak phosphorescence. The strong XB interaction between the iodine atoms of TIE and the non-bonding orbitals of the azaphenanthrene N atoms should cause the energy of the lowest $^{1}(n, \pi^{*})$ state to drop below that of the $^{3}(\pi, \pi^{*})$ state. It is supposed that for the singlet states the 0–0 transition of emitters in co-crystals is localized at 375 nm and 450 nm, respectively, and for triplet states the 0–0 transition is at about 600 nm. The energy gap between S_1 and T_1 is largely greater than 20 kJ mol^{-1}, so the delayed fluorescence most likely originates from the triplet–triplet annihilation process, named *P*-type delayed fluorescence (P-DF). Both delayed fluorescence and phosphorescence are relative to triplet states, so they should be significant for

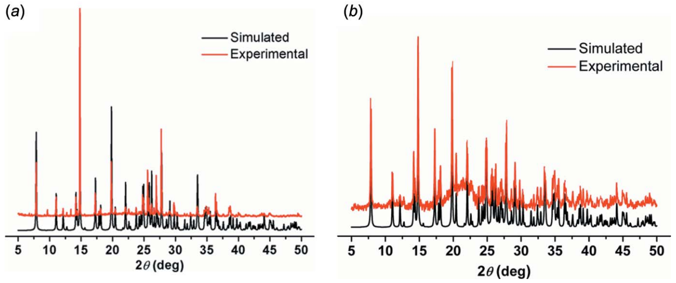


Figure 6
Powder X-ray diffraction pattern of co-crystals (*a*) **1** and (*b*) **2**.

Table 3
Experimental details.

	1	2
Crystal data		
Chemical formula	$0.5C_2I_4 \cdot C_{13}H_9N$	$0.5C_2I_4 \cdot C_{13}H_9N$
M_r	445.02	445.02
Crystal system, space group	Monoclinic, $C2/c$	Triclinic, $P\overline{1}$
Temperature (K)	296	296
a, b, c (Å)	24.2920 (18), 4.8348 (4), 24.8761 (16)	7.3179 (4), 8.1089 (5), 11.3252 (7)
α, β, γ (°)	90, 116.272 (2), 90	97.050 (2), 92.059 (2), 95.579 (2)
V (Å^3)	2619.8 (3)	663.02 (7)
Z	8	2
Radiation type	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	4.78	4.72
Crystal size (mm)	0.30 × 0.25 × 0.25	0.35 × 0.32 × 0.30
Data collection		
Diffractometer	Bruker APEXII CCD	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2012)	Multi-scan (*SADABS*; Bruker, 2012)
T_{min}, T_{max}	0.489, 0.745	0.638, 0.745
No. of measured, independent and observed $[I > 2\sigma(I)]$ reflections	12094, 2635, 1782	8567, 2701, 2099
R_{int}	0.045	0.026
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.624	0.625
Refinement		
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.043, 0.078, 1.03	0.028, 0.058, 1.08
No. of reflections	2635	2701
No. of parameters	154	154
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å^{-3})	1.08, −0.62	0.80, −0.74

Computer programs: *APEX2* and *SAINT* (Bruker, 2012), *SHELXS97* and *SHELXTL* (Sheldrick, 2008), *SHELXL2018/3* (Sheldrick, 2015), (Sheldrick, 2008), *DIAMOND* (Brandenburg, 2005) and *publCIF* (Westrip, 2010).

improving the exciton emission efficiency of luminescence materials (Adachi *et al.*, 2001).

For the luminescence decay, all singlet state decay lifetimes (11.49 ns for **1** and 9.29 ns for **2**) are about 10 ns, while the delayed fluorescence lifetime (4.36 µs for **1** and 6.45 µs for **2**) is less than the 10 µs level because of the strong heavy-atom effect leading to a faster decay of the triplet state. Additionally, the phosphorescence is too weak to measure its decay lifetime. However, the phosphorescence lifetime can be estimated to be about 20 µs based on the relationship between P-DF and the accompanying phosphorescence (Parker *et al.*, 1962, 1965).

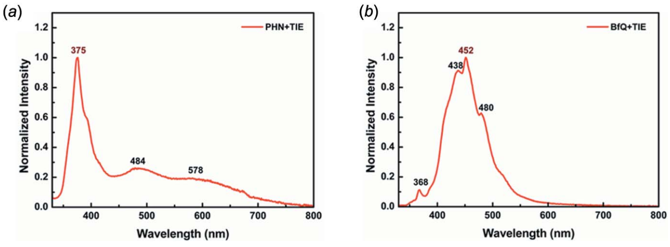


Figure 7
Total luminescence spectra of co-crystals (*a*) **1** and (*b*) **2** (excitation at 300 nm) measured under fluorescence mode.

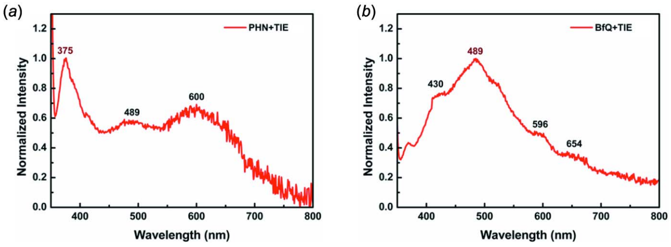


Figure 8
Luminescence spectra of co-crystals (*a*) **1** and (*b*) **2** (excitation at 330 nm) measured under phosphorescence mode.

6. Synthesis and crystallization

0.1 mmol of PHN/BfQ and 0.05 mmol of TIE were dissolved in an acetone/chloroform (2:1) mixture in a glass vial. Well–formed co-crystals **1** and **2** suitable for single-crystal X–ray diffraction (XRD) measurements were obtained by slow evaporation of the solvent at room temperature after about two weeks. Elemental analysis (%, EA) calculated for $C_{14}H_9NI_2$ (445.02): C 37.78, H 2.04, N 3.15. Found: C 37.54, H 2.31, N 3.26. For co-crystal **1**, and C 37.85, H 2.16, N 3.04 for co-crystal **2**. IR (KBr, ν, cm^{-1}) For **1**: 3048(*w*), 1603(*w*), 1572(*w*), 1494(*m*), 1446(*w*), 1382(*m*), 1293(*m*), 1267(*m*), 1189(*m*), 1089(*m*), 948(*m*), 870(*s*), 832(*s*), 802(*s*), 749(*s*), 707(*s*), 615(*m*), 538(*m*), 487(*m*), 435(*m*). For **2**: 3048(*w*), 1611(*w*), 1576(*s*), 1522(*w*), 1486(*w*), 1458(*m*), 1440(*m*), 1238(*m*), 1132(*m*), 1032(*m*), 953(*m*), 924(*m*), 889(*s*), 745(*s*), 714(*s*), 610(*m*), 552(*m*), 448(*m*), 423(*m*).

7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. H atoms attached to C atoms were positioned geometrically and refined as riding on their parent atoms, with C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C).

Funding information

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supporting information

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Structural and luminescent properties of co-crystals of tetraiodoethylene with two azaphenanthrenes

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Computing details

For both structures, data collection: *APEX2* (Bruker, 2012); cell refinement: *SAINT* (Bruker, 2012); data reduction: *SAINT* (Bruker, 2012); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2018/3* (Sheldrick, 2015). Molecular graphics: *SHELXTL* (Sheldrick, 2008), *DIAMOND* (Brandenburg, 2005) for (1); *SHELXTL* (Sheldrick, 2008), *DIAMOND* (Brandenburg, 2005), for (2). For both structures, software used to prepare material for publication: *publCIF* (Westrip, 2010).

Tetraiodoethylene–phenanthridine (1/2) (1)

Crystal data

$0.5C_2I_4\cdot C_{13}H_9N$
$M_r = 445.02$
Monoclinic, $C2/c$
$a = 24.2920\ (18)$ Å
$b = 4.8348\ (4)$ Å
$c = 24.8761\ (16)$ Å
$\beta = 116.272\ (2)°$
$V = 2619.8\ (3)$ Å^3
$Z = 8$
$F(000) = 1648$
$D_x = 2.257$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5311 reflections
$\theta = 3.1–26.3°$
$\mu = 4.78$ mm^{-1}
$T = 296$ K
Block, yellow
0.30 × 0.25 × 0.25 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: sealed tube
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2012)
$T_{min} = 0.489$, $T_{max} = 0.745$
12094 measured reflections
2635 independent reflections
1782 reflections with $I > 2\sigma(I)$
$R_{int} = 0.045$
$\theta_{max} = 26.3°$, $\theta_{min} = 3.1°$
$h = -30\rightarrow29$
$k = -6\rightarrow6$
$l = -31\rightarrow30$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.078$
$S = 1.03$
2635 reflections
154 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0187P)^2 + 28.9715P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.08$ e Å^{-3}
$\Delta\rho_{min} = -0.61$ e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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

	x	y	z	$U_{\rm iso}$*/$U_{\rm eq}$
I1	0.70990 (2)	0.95088 (10)	0.57060 (2)	0.04011 (16)
I2	0.85225 (2)	0.96131 (11)	0.56611 (2)	0.04316 (16)
N1	0.6417 (3)	0.1927 (12)	0.6276 (2)	0.0377 (14)
C1	0.6440 (3)	0.1453 (14)	0.6836 (3)	0.0339 (16)
C2	0.6849 (3)	−0.0548 (15)	0.7193 (3)	0.0442 (18)
H2	0.708323	−0.152775	0.704671	0.053*
C3	0.6910 (4)	−0.1093 (16)	0.7755 (3)	0.053 (2)
H3	0.719345	−0.239978	0.799494	0.064*
C4	0.6547 (4)	0.0319 (17)	0.7965 (3)	0.054 (2)
H4	0.658318	−0.006933	0.834537	0.065*
C5	0.6141 (3)	0.2253 (16)	0.7623 (3)	0.0452 (19)
H5	0.590382	0.318582	0.777344	0.054*
C6	0.6070 (3)	0.2880 (14)	0.7044 (3)	0.0328 (16)
C7	0.5640 (3)	0.4901 (13)	0.6650 (3)	0.0342 (16)
C8	0.5240 (3)	0.6464 (16)	0.6801 (3)	0.0475 (19)
H8	0.524161	0.622630	0.717264	0.057*
C9	0.4853 (3)	0.8313 (17)	0.6406 (4)	0.054 (2)
H9	0.459174	0.933001	0.651309	0.065*
C10	0.4834 (3)	0.8741 (16)	0.5846 (4)	0.051 (2)
H10	0.456128	1.001451	0.558148	0.061*
C11	0.5219 (3)	0.7271 (15)	0.5691 (3)	0.0455 (19)
H11	0.521208	0.755053	0.531834	0.055*
C12	0.5627 (3)	0.5329 (14)	0.6091 (3)	0.0345 (16)
C13	0.6035 (3)	0.3779 (15)	0.5941 (3)	0.0405 (18)
H13	0.602707	0.412409	0.557015	0.049*
C14	0.7603 (3)	0.8233 (15)	0.5244 (3)	0.0411 (18)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.0411 (3)	0.0521 (3)	0.0327 (3)	0.0057 (2)	0.0213 (2)	−0.0022 (2)
I2	0.0332 (3)	0.0582 (3)	0.0364 (3)	−0.0048 (2)	0.0139 (2)	−0.0059 (2)
N1	0.034 (3)	0.046 (4)	0.037 (3)	0.000 (3)	0.019 (3)	−0.004 (3)
C1	0.034 (4)	0.036 (4)	0.033 (4)	−0.005 (3)	0.016 (3)	−0.005 (3)
C2	0.042 (4)	0.043 (4)	0.046 (4)	0.001 (4)	0.018 (4)	0.001 (4)
C3	0.060 (5)	0.049 (5)	0.041 (5)	0.000 (4)	0.013 (4)	0.011 (4)
C4	0.067 (6)	0.066 (6)	0.027 (4)	−0.015 (5)	0.019 (4)	−0.001 (4)
C5	0.048 (5)	0.053 (5)	0.041 (4)	−0.010 (4)	0.026 (4)	−0.008 (4)
C6	0.031 (4)	0.038 (4)	0.030 (4)	−0.008 (3)	0.014 (3)	−0.007 (3)

C7	0.032 (4)	0.034 (4)	0.041 (4)	−0.006 (3)	0.020 (3)	−0.008 (3)
C8	0.045 (5)	0.053 (5)	0.049 (5)	−0.002 (4)	0.026 (4)	−0.005 (4)
C9	0.031 (5)	0.051 (5)	0.081 (6)	−0.003 (4)	0.025 (4)	−0.015 (5)
C10	0.038 (5)	0.040 (5)	0.070 (6)	0.006 (4)	0.019 (4)	0.005 (4)
C11	0.042 (5)	0.049 (5)	0.039 (4)	−0.012 (4)	0.012 (4)	−0.003 (4)
C12	0.030 (4)	0.036 (4)	0.033 (4)	−0.003 (3)	0.010 (3)	−0.001 (3)
C13	0.046 (5)	0.052 (5)	0.030 (4)	−0.005 (4)	0.023 (4)	−0.002 (4)
C14	0.045 (5)	0.044 (5)	0.043 (4)	0.007 (4)	0.028 (4)	0.003 (3)

Geometric parameters (Å, º)

I1—C14	2.108 (6)	C6—C7	1.449 (9)
I2—C14	2.111 (7)	C7—C12	1.394 (9)
N1—C13	1.293 (8)	C7—C8	1.406 (9)
N1—C1	1.390 (8)	C8—C9	1.353 (10)
C1—C2	1.389 (9)	C8—H8	0.9300
C1—C6	1.400 (9)	C9—C10	1.387 (11)
C2—C3	1.364 (10)	C9—H9	0.9300
C2—H2	0.9300	C10—C11	1.360 (10)
C3—C4	1.389 (11)	C10—H10	0.9300
C3—H3	0.9300	C11—C12	1.408 (9)
C4—C5	1.352 (10)	C11—H11	0.9300
C4—H4	0.9300	C12—C13	1.416 (9)
C5—C6	1.406 (9)	C13—H13	0.9300
C5—H5	0.9300	C14—C14$^{\rm i}$	1.301 (13)
C13—N1—C1	117.3 (6)	C9—C8—C7	120.1 (7)
N1—C1—C2	117.1 (6)	C9—C8—H8	120.0
N1—C1—C6	122.8 (6)	C7—C8—H8	120.0
C2—C1—C6	120.1 (6)	C8—C9—C10	122.1 (7)
C3—C2—C1	120.8 (7)	C8—C9—H9	119.0
C3—C2—H2	119.6	C10—C9—H9	119.0
C1—C2—H2	119.6	C11—C10—C9	119.0 (7)
C2—C3—C4	119.4 (7)	C11—C10—H10	120.5
C2—C3—H3	120.3	C9—C10—H10	120.5
C4—C3—H3	120.3	C10—C11—C12	120.4 (7)
C5—C4—C3	120.8 (7)	C10—C11—H11	119.8
C5—C4—H4	119.6	C12—C11—H11	119.8
C3—C4—H4	119.6	C7—C12—C11	120.1 (6)
C4—C5—C6	121.2 (7)	C7—C12—C13	118.4 (6)
C4—C5—H5	119.4	C11—C12—C13	121.6 (6)
C6—C5—H5	119.4	N1—C13—C12	125.7 (6)
C1—C6—C5	117.7 (6)	N1—C13—H13	117.1
C1—C6—C7	118.1 (6)	C12—C13—H13	117.1
C5—C6—C7	124.2 (6)	C14$^{\rm i}$—C14—I2	121.2 (7)
C12—C7—C8	118.3 (7)	C14$^{\rm i}$—C14—I1	126.2 (7)
C12—C7—C6	117.7 (6)	I2—C14—I1	112.5 (3)
C8—C7—C6	124.0 (6)		

C13—N1—C1—C2	179.7 (6)
C13—N1—C1—C6	0.2 (10)
N1—C1—C2—C3	178.4 (7)
C6—C1—C2—C3	−2.1 (11)
C1—C2—C3—C4	1.8 (12)
C2—C3—C4—C5	−1.0 (12)
C3—C4—C5—C6	0.5 (12)
N1—C1—C6—C5	−179.0 (6)
C2—C1—C6—C5	1.5 (10)
N1—C1—C6—C7	1.0 (10)
C2—C1—C6—C7	−178.5 (6)
C4—C5—C6—C1	−0.7 (10)
C4—C5—C6—C7	179.3 (7)
C1—C6—C7—C12	−0.9 (9)
C5—C6—C7—C12	179.1 (7)
C1—C6—C7—C8	179.7 (6)
C5—C6—C7—C8	−0.3 (11)
C12—C7—C8—C9	0.5 (11)
C6—C7—C8—C9	179.9 (7)
C7—C8—C9—C10	0.0 (12)
C8—C9—C10—C11	−0.5 (12)
C9—C10—C11—C12	0.5 (11)
C8—C7—C12—C11	−0.5 (10)
C6—C7—C12—C11	−179.9 (6)
C8—C7—C12—C13	179.3 (6)
C6—C7—C12—C13	−0.2 (10)
C10—C11—C12—C7	0.0 (10)
C10—C11—C12—C13	−179.7 (7)
C1—N1—C13—C12	−1.4 (10)
C7—C12—C13—N1	1.5 (11)
C11—C12—C13—N1	−178.8 (7)

Symmetry code: (i) $-x+3/2$, $-y+3/2$, $-z+1$.

Tetraiodoethylene–benzo[*f*]quinoline (1/2) (2)

Crystal data

$0.5\mathrm{C_2I_4}\cdot\mathrm{C_{13}H_9N}$
$M_r = 445.02$
Triclinic, $P\bar{1}$
$a = 7.3179$ (4) Å
$b = 8.1089$ (5) Å
$c = 11.3252$ (7) Å
$\alpha = 97.050$ (2)°
$\beta = 92.059$ (2)°
$\gamma = 95.579$ (2)°
$V = 663.02$ (7) Å^3
$Z = 2$
$F(000) = 412$
$D_x = 2.229$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4699 reflections
$\theta = 2.8$–26.4°
$\mu = 4.72$ mm^{-1}
$T = 296$ K
Block, yellow
0.35 × 0.32 × 0.30 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: sealed tube
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2012)
$T_{\min} = 0.638$, $T_{\max} = 0.745$
8567 measured reflections
2701 independent reflections
2099 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.026$
$\theta_{\max} = 26.4°$, $\theta_{\min} = 2.5°$
$h = -9 \rightarrow 9$
$k = -10 \rightarrow 10$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.028$
$wR(F^2) = 0.058$
$S = 1.08$
2701 reflections
154 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0189P)^2 + 0.7413P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.80$ e Å^{-3}
$\Delta\rho_{\min} = -0.74$ e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
I1	0.20988 (4)	0.45307 (3)	0.82595 (2)	0.04767 (10)
I2	−0.08934 (4)	0.75374 (3)	0.91391 (3)	0.05167 (11)
N1	0.4565 (5)	0.3596 (5)	0.6400 (3)	0.0518 (9)
C1	0.6267 (7)	0.4270 (6)	0.6615 (4)	0.0597 (13)
H1	0.655445	0.495377	0.733212	0.072*
C2	0.7655 (7)	0.4022 (6)	0.5841 (5)	0.0613 (13)
H2	0.883874	0.453358	0.603495	0.074*
C3	0.7274 (6)	0.3026 (6)	0.4794 (5)	0.0541 (12)
H3	0.819507	0.286190	0.425958	0.065*
C4	0.5492 (5)	0.2240 (5)	0.4514 (4)	0.0399 (10)
C5	0.4171 (5)	0.2587 (5)	0.5350 (4)	0.0389 (9)
C6	0.2327 (6)	0.1869 (5)	0.5119 (4)	0.0470 (11)
H6	0.144788	0.211154	0.566982	0.056*
C7	0.1844 (6)	0.0845 (5)	0.4114 (4)	0.0471 (11)
H7	0.062732	0.038787	0.398340	0.057*
C8	0.3128 (6)	0.0431 (5)	0.3238 (4)	0.0424 (10)
C9	0.4960 (6)	0.1144 (5)	0.3424 (4)	0.0418 (10)
C10	0.6199 (7)	0.0731 (6)	0.2544 (4)	0.0561 (12)
H10	0.741510	0.119847	0.264207	0.067*
C11	0.5643 (9)	−0.0348 (7)	0.1546 (5)	0.0730 (16)
H11	0.648615	−0.060734	0.097457	0.088*
C12	0.3842 (9)	−0.1063 (7)	0.1373 (4)	0.0716 (16)
H12	0.347907	−0.180218	0.069304	0.086*
C13	0.2612 (7)	−0.0676 (6)	0.2203 (4)	0.0555 (12)
H13	0.140145	−0.115380	0.208420	0.067*
C14	0.0238 (6)	0.5360 (5)	0.9536 (4)	0.0449 (10)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.0579 (2)	0.04585 (18)	0.04064 (16)	0.01385 (14)	0.01030 (14)	0.00193 (12)
I2	0.0700 (2)	0.04158 (18)	0.04756 (18)	0.02041 (15)	0.00980 (15)	0.00849 (13)
N1	0.058 (3)	0.051 (2)	0.049 (2)	0.0117 (19)	0.003 (2)	0.0083 (18)
C1	0.068 (3)	0.052 (3)	0.058 (3)	0.007 (3)	−0.016 (3)	0.006 (2)
C2	0.042 (3)	0.067 (3)	0.076 (4)	−0.002 (2)	−0.010 (3)	0.024 (3)
C3	0.039 (3)	0.055 (3)	0.073 (3)	0.006 (2)	0.013 (2)	0.026 (3)
C4	0.036 (2)	0.041 (2)	0.049 (2)	0.0109 (18)	0.008 (2)	0.0221 (19)
C5	0.041 (2)	0.036 (2)	0.043 (2)	0.0108 (18)	0.0065 (19)	0.0135 (18)
C6	0.042 (2)	0.054 (3)	0.048 (3)	0.008 (2)	0.015 (2)	0.009 (2)

C7	0.036 (2)	0.051 (3)	0.056 (3)	0.003 (2)	0.006 (2)	0.016 (2)
C8	0.051 (3)	0.036 (2)	0.044 (2)	0.0087 (19)	0.006 (2)	0.0144 (18)
C9	0.046 (2)	0.042 (2)	0.044 (2)	0.0161 (19)	0.014 (2)	0.0199 (19)
C10	0.059 (3)	0.066 (3)	0.052 (3)	0.023 (2)	0.021 (2)	0.022 (2)
C11	0.099 (5)	0.080 (4)	0.051 (3)	0.043 (4)	0.031 (3)	0.018 (3)
C12	0.115 (5)	0.059 (3)	0.044 (3)	0.025 (3)	0.007 (3)	0.004 (2)
C13	0.072 (3)	0.047 (3)	0.049 (3)	0.009 (2)	−0.003 (3)	0.010 (2)
C14	0.054 (3)	0.039 (2)	0.041 (2)	0.011 (2)	0.002 (2)	−0.0017 (18)

Geometric parameters (Å, º)

I1—C14	2.116 (4)	C6—H6	0.9300
I2—C14	2.111 (4)	C7—C8	1.423 (6)
N1—C1	1.313 (6)	C7—H7	0.9300
N1—C5	1.363 (5)	C8—C13	1.402 (6)
C1—C2	1.379 (7)	C8—C9	1.405 (6)
C1—H1	0.9300	C9—C10	1.403 (6)
C2—C3	1.354 (7)	C10—C11	1.365 (7)
C2—H2	0.9300	C10—H10	0.9300
C3—C4	1.402 (6)	C11—C12	1.384 (8)
C3—H3	0.9300	C11—H11	0.9300
C4—C5	1.401 (5)	C12—C13	1.355 (7)
C4—C9	1.448 (6)	C12—H12	0.9300
C5—C6	1.418 (6)	C13—H13	0.9300
C6—C7	1.339 (6)	C14—C14$^{\mathrm{i}}$	1.305 (8)
C1—N1—C5	117.4 (4)	C13—C8—C9	119.4 (4)
N1—C1—C2	123.8 (5)	C13—C8—C7	121.7 (4)
N1—C1—H1	118.1	C9—C8—C7	118.9 (4)
C2—C1—H1	118.1	C10—C9—C8	118.0 (4)
C3—C2—C1	119.2 (4)	C10—C9—C4	123.0 (4)
C3—C2—H2	120.4	C8—C9—C4	119.0 (4)
C1—C2—H2	120.4	C11—C10—C9	120.9 (5)
C2—C3—C4	120.1 (4)	C11—C10—H10	119.6
C2—C3—H3	120.0	C9—C10—H10	119.6
C4—C3—H3	120.0	C10—C11—C12	121.0 (5)
C3—C4—C5	116.5 (4)	C10—C11—H11	119.5
C3—C4—C9	124.0 (4)	C12—C11—H11	119.5
C5—C4—C9	119.5 (4)	C13—C12—C11	119.5 (5)
N1—C5—C4	123.0 (4)	C13—C12—H12	120.3
N1—C5—C6	117.1 (4)	C11—C12—H12	120.3
C4—C5—C6	119.9 (4)	C12—C13—C8	121.3 (5)
C7—C6—C5	120.4 (4)	C12—C13—H13	119.4
C7—C6—H6	119.8	C8—C13—H13	119.4
C5—C6—H6	119.8	C14$^{\mathrm{i}}$—C14—I2	121.4 (4)
C6—C7—C8	122.3 (4)	C14$^{\mathrm{i}}$—C14—I1	126.2 (4)
C6—C7—H7	118.9	I2—C14—I1	112.33 (18)
C8—C7—H7	118.9		

C5—N1—C1—C2	0.5 (7)	C13—C8—C9—C10	1.1 (6)
N1—C1—C2—C3	−0.3 (8)	C7—C8—C9—C10	−179.4 (4)
C1—C2—C3—C4	−0.9 (7)	C13—C8—C9—C4	−177.9 (4)
C2—C3—C4—C5	1.7 (6)	C7—C8—C9—C4	1.6 (6)
C2—C3—C4—C9	−179.4 (4)	C3—C4—C9—C10	1.3 (6)
C1—N1—C5—C4	0.5 (6)	C5—C4—C9—C10	−179.8 (4)
C1—N1—C5—C6	−179.7 (4)	C3—C4—C9—C8	−179.7 (4)
C3—C4—C5—N1	−1.6 (6)	C5—C4—C9—C8	−0.9 (6)
C9—C4—C5—N1	179.5 (4)	C8—C9—C10—C11	−0.9 (6)
C3—C4—C5—C6	178.6 (4)	C4—C9—C10—C11	178.1 (4)
C9—C4—C5—C6	−0.3 (6)	C9—C10—C11—C12	0.1 (8)
N1—C5—C6—C7	−179.0 (4)	C10—C11—C12—C13	0.4 (8)
C4—C5—C6—C7	0.8 (6)	C11—C12—C13—C8	−0.2 (7)
C5—C6—C7—C8	−0.1 (7)	C9—C8—C13—C12	−0.5 (6)
C6—C7—C8—C13	178.4 (4)	C7—C8—C13—C12	180.0 (4)
C6—C7—C8—C9	−1.1 (6)		

Symmetry code: (i) −*x*, −*y*+1, −*z*+2.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1···I2^{ii}	0.93	3.16	4.019 (5)	155
C6—H6···I1	0.93	3.31	3.945 (4)	127

Symmetry code: (ii) *x*+1, *y*, *z*.