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4-Pyrrolidinopyridine as halogen-bond acceptor in cocrystals

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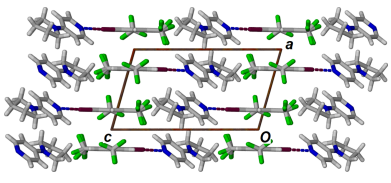
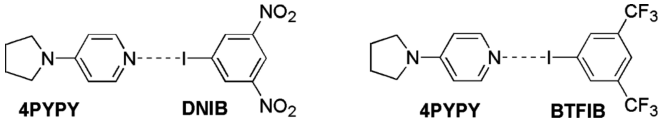
The potential of 4-pyrrolidinopyridine as halogen-bond acceptor is further explored and the structures of 1:1 cocrystals 1-iodo-3,5-dinitrobenzene–4-pyrrolidinopyridine, $C_6H_3IN_2O_4{\cdot}C_9H_{12}N_2$, and 1-iodo-3,5-bis(trifluoromethyl)-benzene–4-pyrrolidinopyridine $C_8H_3F_6I{\cdot}C_9H_{12}N_2$, are reported. This is the first reported halogen-bonded cocrystal with 1-iodo-3,5-bis(trifluoromethyl)benzene. The halogen bonds in these structures have similar I$\cdots$N separations of 2.871 (2) and 2.858 (4) Å, respectively, with C—I$\cdots$N angles of 172.91 (9) and 171.38 (14)°, respectively. The components within the cocrystal 1-iodo-3,5-bisdinitrobenzene–4-pyrrolidinopyridine are coplanar and form sheets that π-stack with donor–acceptor, donor–donor and acceptor–acceptor interactions within the layers. In contrast, the components in the cocrystal 1-iodo-3,5-bis(trifluoromethyl)benzene–4-pyrrolidinopyridine are twisted with a dihedral angle of 44.052 (2)° between the pyridine and benzene rings and the donor and acceptor molecules are individually π-stacked with no donor–acceptor stacking interactions. Analysis of the intermolecular interaction energy between molecules within the crystal structures reveals that in cocrystal 1-iodo-3,5-dinitrobenzene–4-pyrrolidinopyridine the donor-acceptor π-stacking is the strongest interaction whereas in cocrystal 1-iodo-3,5-bis(trifluoromethyl)benzene–4-pyrrolidinopyridine, the head-to-tail π-stacking of the pyrrolidinopyridine molecules is the strongest interaction.

1. Chemical context

Halogen bonding is now an accepted intermolecular force with a multitude of applications in the general field of supramolecular chemistry (Metrangolo & Resnati, 2008; Cavallo *et al.*, 2016; Liu & Yang (2025). Electron-poor iodoarenes, for example polyfluoroiodobenzenes, are common halogen-bond donors often coupled with pyridines as halogen-bond acceptor. 4-(*N*,*N*-Dimethylamino)pyridine (**4DMAP**) is a strong base and in general forms stronger halogen bonds than other pyridines with lower I$\cdots$N separations (Präsang *et al.*, 2009). Thus, the I$\cdots$N separation in the cocrystals formed between 1,2,4,5-tetrafluoro-3,6-diiodobenzene and **4DMAP** and 4,4-bipyridine are 2.664 and 2.854 Å, respectively (Roper *et al.*, 2010; Walsh *et al.*, 2001). 4-Pyrrolidinopyridine, **4PYPY**, is reported to be a slightly stronger base than **4DMAP** (Kaljurand *et al.*, 2005); however, few halogen-bonded cocrystals featuring **4PYPY** as halogen-bond acceptor have been reported. These include the 1:1 cocrystal of **4PYPY** with 4-bromo-2,2′,3,3′,5,5′,6,6′-octafluoro-4′-iodobiphenyl (Aakeroy *et al.*, 2013) and the 1:1 and 1:2 halogen-bonded cocrystals of 1,3-diiodo-5,5-dimethylimidazolidine-2,4-dione (Nicolas *et al.*, 2016). Recently, Rissanen and coworkers reported the complex of **4PYPY** with *N*-iodosaccharin (Schumacher *et al.*, 2024) and the complex 1-{[(diphenylphosphoryl)oxy]iodanyl}-4-(pyrrolidin-1-yl)-1-pyridine (Mo-



han *et al.*, 2024) with exceptionally short I···N (**4PYPY**) distances. Here we report two cocrystals formed between halogen-bond donors 1-iodo-3,5-dinitrobenzene (**DNIB**) and 1-iodo-3,5-bis(trifluoromethyl)benzene (**BTFIB**) with **4PYPY** as halogen-bond acceptor.



2. Molecular electrostatic potentials

The molecular electrostatic potentials (MEP) of the molecules in this study were calculated using the program *Spartan '20* version 1.1.4 (Wavefunction, 2020) with density functional theory at the B3LYP-D3/6-311+G** level with an isovalue of 0.2 electrons bohr^{-3}. The molecular electrostatic potentials for **DNIB** and **BTFIB** revealed that the σ-hole on the iodine atom in **DNIB** at 173.2 kJ mol^{-1} is similar to that calculated for iodopentafluorobenzene using the same experimental conditions (174.6 kJ mol^{-1}). In contrast, the calculated σ-hole on the iodine atom in **BTFIB** at 146.6 kJ mol^{-1} is similar to the weaker halogen-bond donors iodopentachlorobenezne (154.9 kJ mol^{-1}) and bromopentafluorobenzene (144.5 kJ mol^{-1}). The minimum negative electrostatic potential on the pyridine N atom in **4PYPY** at −228 kJ mol^{-1} is slightly more negative than that on **4DMAP**, −217.4 kJ mol^{-1} (Fig. 1).

3. Structural commentary

The 1:1 cocrystal **DNIB·4PYPY** crystallizes in the triclinic space group $P\overline{1}$ with one molecule of each component in the asymmetric unit as shown in Fig. 2. The halogen-bond donor and acceptor moieties are essentially coplanar with a dihedral angle of 7.38 (14)° between the pyridyl and benzene rings. The I···N separation is 2.871 (2) Å, 77.6% of the sum of the van der Waals radii (Alvarez, 2013) and the C—I···N angle is essentially linear at 172.91 (8)°.

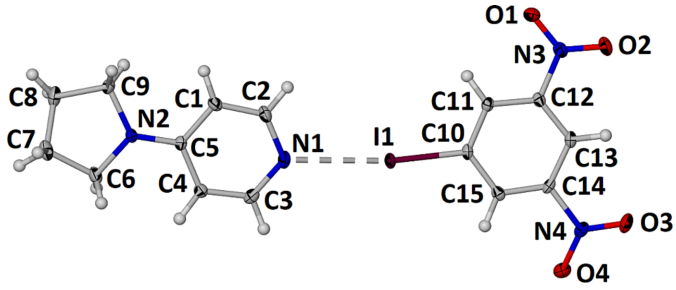


Figure 2
Asymmetric unit of the cocrystal **DNIB·4PYPY** with displacement ellipsoids drawn at the 50% level and the halogen bond shown as a grey dashed line.

The 1:1 cocrystal **BTFIB·4PYPY** crystallizes in the triclinic space group $P\overline{1}$ with one molecule of each component in the asymmetric unit as shown in Fig. 3. The two moieties are not coplanar with a dihedral angle of 44.052 (2)° between the pyridyl and benzene rings. The I···N separation is 2.858 (4) Å, 77.2% of the sum of the van der Waals radii and the C—I···N angle is 171.38 (14)°.

These cocrystals confirm the viability of **4PYPY** as a good halogen-bond acceptor. It is interesting, however, to note that the halogen-bond distance, or I···N separation, in the two complexes is similar for the halogen bond donors, at 2.871 (2) and 2.858 (4) Å, despite the large variation in the σ-hole on these halogen-bond donors of 173.2 and 146.6 kJ mol^{-1}, respectively. This reasonably highlights the role that other crystal packing interactions have in modulating the I···N separation. Also, comparison of the cocrystal **DNIB·4PYPY** with the previously reported cocrystal **DNIB·DMAP** (Nwachukwu *et al.*, 2018) reveals that the I···N separation in **DNIB·DMAP** is slightly longer at 2.8936 (16) Å than those reported here for **DNIB·4PYPY**, in line with the more negative electrostatic potential of **4PYPY**.

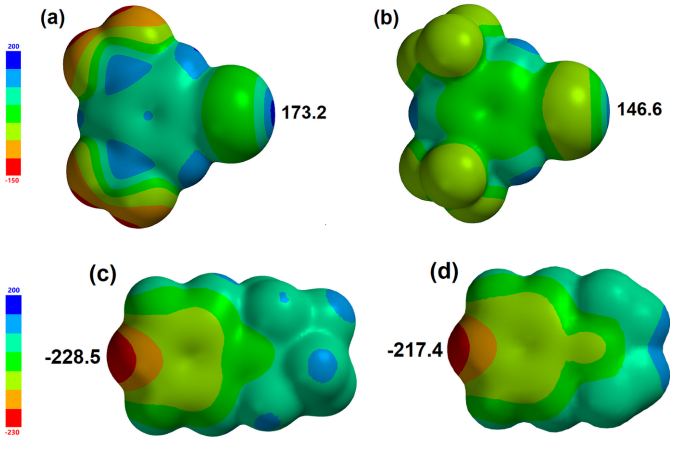


Figure 1
Molecular electrostatic potential plots drawn over the 0.002 isodensity surface for (*a*) **DNIB**, (*b*) **BTFIB**, (*c*) **4PYPY**, and (*d*) **DMAP**. Maxima are annotated for (*a*) and (*b*) and minima annotated for (*c*) and (*d*) in kJ mol^{-1}.

4. Supramolecular features

The halogen-bonded pairs of molecules in cocrystal **DNIB·4-PYPY** pack side-by-side to form planar sheets as shown in Fig. 4. The weak bifurcated C—H···O(nitro) interaction

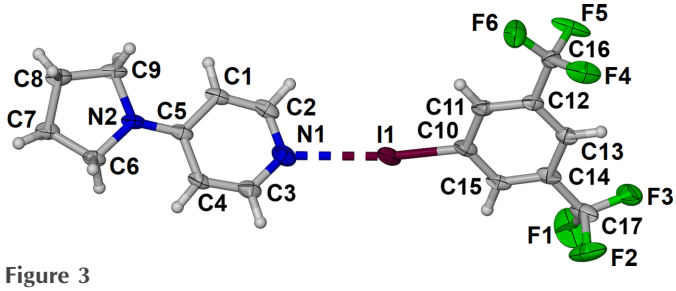


Figure 3
Asymmetric unit of the cocrystal **TFMIB·4PYPY** with displacement ellipsoids drawn at the 50% level and the halogen bond shown as a dashed line.

Table 1
Hydrogen-bond geometry (Å, °) for **DNIB·4PYPY**.

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···O1^{i}	0.95	2.61	3.499 (3)	156
C7—H7B···I1^{ii}	0.99	3.22	4.104 (3)	150
C8—H8A···O2^{iii}	0.99	2.55	3.301 (4)	133
C8—H8A···O4^{iv}	0.99	2.61	3.420 (4)	140
C9—H9A···O4^{v}	0.99	2.61	3.186 (3)	117

Symmetry codes: (i) $x, y-1, z$; (ii) $-x, -y, -z+1$; (iii) $-x+1, -y+1, -z+1$; (iv) $-x+1, -y, -z+1$; (v) $x-1, y, z-1$.

between adjacent molecules within each plane that is parallel to the *b*-axis (Bosch *et al.*, 2022).

The halogen-bonded complexes are head-to-tail π-stacked where the closest interaction is between a halogen-bonded donor–acceptor (*DA*) pair with a centroid–centroid distance and closest perpendicular distances between the pyridyl and phenyl groups of 3.9166 (16) and 3.3735 (10) Å, respectively. Each halogen-bonded **4PYPY** molecule is also offset π-stacked in a head-to-tail orientation with another **4PYPY** with closest perpendicular distance and centroid-to-centroid distances of 3.8823 (10) and 4.9846 (17) Å, respectively. Accordingly, each **DNIB** molecule is also offset π-stacked in a head-to-tail orientation with another **DNIB** molecule with a

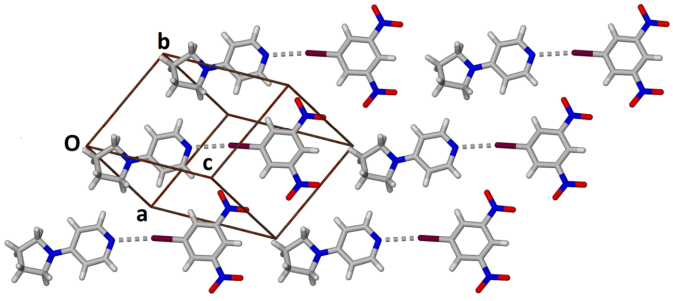


Figure 4
Partial view of one of the planar sheets within the cocrystal **DNIB·4-PYPY**. The halogen bond is shown as a grey dashed line.

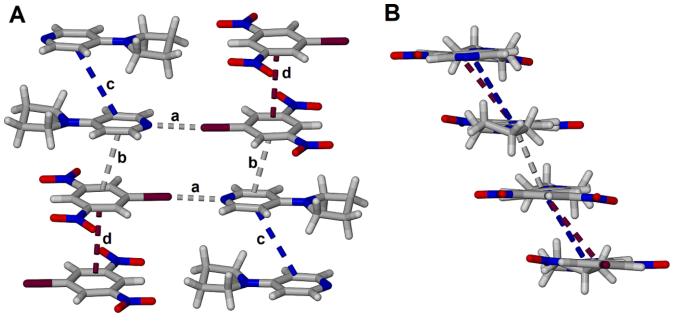


Figure 5
(A) Oblique partial view of the π-stacking within cocrystal **DNIB·4-PYPY**. The halogen bond and the **DNIB**···**4PYPY** centroid-to-centroid π-stacking interaction are shown as grey dashed lines and labelled a and b, respectively. Centroid-to-centroid connections are also shown as blue dashed lines for π-stacking interactions between two **4PYPY** molecules and maroon dashed line between two **DNIB** molecules. (B) Side view of the molecules in (A) with the molecules rotated into the horizontal plane.

Table 2
Hydrogen-bond geometry (Å, °) for **BTFIB·4PYPY**.

D—H···A	D—H	H···A	D···A	D—H···A
C6—H6A···F6^{i}	0.99	2.54	3.465 (5)	156
C1—H1···F5^{ii}	0.95	2.59	3.265 (5)	128

Symmetry codes: (i) $x, y, z+1$; (ii) $-x, -y, -z$.

centroid-to-centroid distance of 4.4801 (16) Å (Fig. 5). Thus, while all halogen-bonded pairs of molecules within each plane have the same orientation, the orientation of the molecules in the π-stacked sheets alternates. Details of hydrogen-bonding interactions are given in Table 1.

The crystal packing within cocrystal **BTFIB·4PYPY** is significantly different to that within **DNIB·4PYPY** with the two components separately π-stacking while forming corrugated sheets. The individual molecules in each stack are offset π-stacked, with a head-to-tail arrangement as shown with the view along the *b*-axis direction (Fig. 6). There are two unique π-stacking interactions within the stacks of each component of cocrystal **BTFIB·4PYPY**. With respect to the **4PYPY** molecules the separations are similar, with pyridine centroid-to-centroid distances of 4.512 (3) and 4.796 (3) Å, and interplanar distances of 3.5831 (18) and 3.5377 (18) Å. The **BTFIB** molecules are also π-stacked with two distinct stacking arrangements with interplanar distances of 3.6688 (17) and 3.9066 (17) Å. The first of these π-stacked molecules is rotated through 180° with a benzene centroid-to-centroid distance of 3.670 (2) Å indicating no shift with maximal surface–surface interaction. The second π-stacked **BTFIB** molecule while also rotated through 180° is shifted with a higher benzene centroid-to-centroid distance of 4.684 (2) Å. There are two close C—H···F interactions with H···F separations marginally less than the sum of the van der Waals radii. Details of hydrogen-bonding interactions are given in Table 2.

5. Hirshfeld surface analysis

The program *CrystalExplorer21* (Spackman *et al.*, 2021) was used to calculate the Hirshfeld surface using density functional theory at the B3LYP/DGDZVP level. The fingerprint plots derived from the Hirshfeld surface provide a breakdown of the intermolecular contacts in terms of the atoms within the Hirshfeld surface and atoms outside the surface.

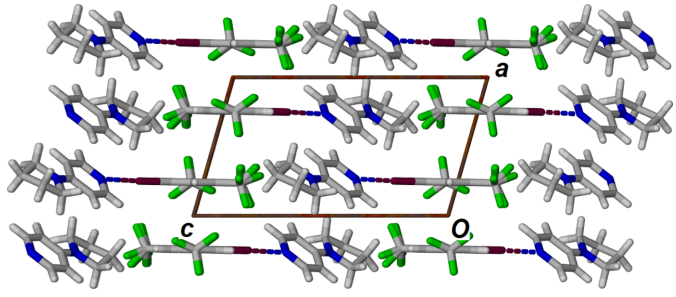


Figure 6
Partial view of the packing within cocrystal **BTFIB·4PYPY** viewed along the *b*-axis direction.

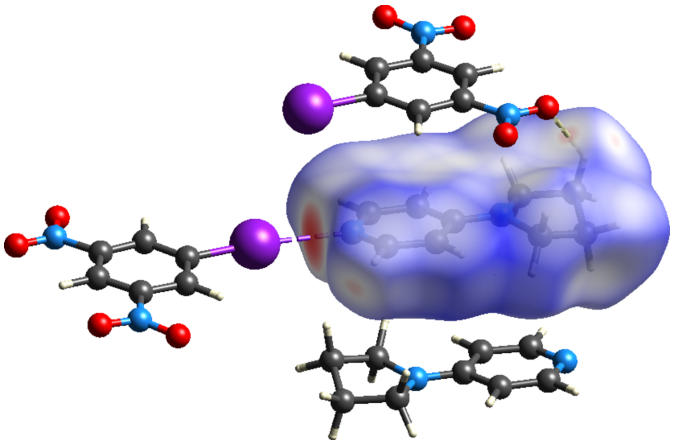


Figure 7
Plot of the Hirshfeld surface of **4PYPY** molecule within the **DNIB·4-PYPY** cocrystal with d_{norm} mapped over the surface. Included are three molecules with strong interactions with the central **4PYPY** molecule with the N···I halogen bond and the H···O interaction shown as purple and grey dashed lines, respectively.

The Hirshfeld surface analysis of a **4PYPY** molecule within the **DNIB·4PYPY** cocrystal is shown in Fig. 7. The red colour indicates an interaction in which the atom-to-atom separation is less than the sum of the van der Waals radii. The N···I halogen bond represents the closest interaction, while the pale-red areas correspond to C—H···O interactions. Based on fingerprint analysis wherein the atom-to-atom surface contacts are analysed by element, the N···I contact corresponds to 3.3% of the surface area of the **4PYPY** molecule. The most common atom-to-atom interaction is H···H, accounting for 46.2% of surface-to-surface interactions with the H···O and H···C interactions accounting for 20.9 and 14.3% of the surface area, respectively.

6. Intermolecular energy of interaction

The program *CrystalExplorer21* was also used to calculate the intermolecular energies of interaction within each crystal

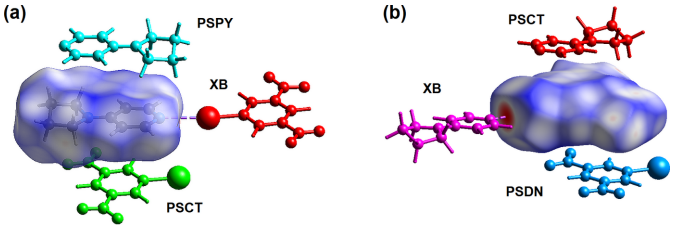


Figure 8
(*a*) Plot showing the three molecules with the strongest intermolecular interaction energy to the central **4PYPY** molecule, shown with the Hirshfeld surface, in cocrystal **DNIB·4PYPY**. The halogen bonded molecule is shown in red and labelled **XB**, π-stacked **DNIB** shown green and labelled **PSCT**, and the head-to-tail π-stacked **4PYPY** shown in blue labelled **PSPY**. (*b*) Plot showing the three molecules with the strongest intermolecular interaction energy to the central **DNIB** molecule in cocrystal **DNIB·4PYPY**. Note that colours in (*b*) are not related to colours in (*a*). The turquoise molecule labelled **PSDN** corresponds to an offset π-stacked **DNIB** molecule.

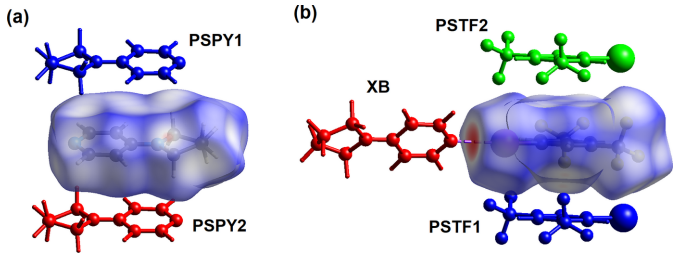


Figure 9
(*a*) Plot showing the two unique **4PYPY** π-stacking interactions to the central **4PYPY** molecule within cocrystal **BTFIB·4PYPY. PSPY1** corresponds to the molecule with the higher degree of overlap. (*b*) Plot showing the three molecules with the strongest intermolecular interaction energy to the central **BTFIB** molecule in cocrystal **BTFIB·4PYPY**. Note that colours in (*b*) are not related to colours in (*a*). The dark-blue molecule labelled **PSTF1** corresponds to the molecule with higher overlap, and the molecule **XB** is the **4PYPY** molecule halogen bonded to the central **BTFIB**.

structure (Mackenzie *et al.*, 2017). The strongest intermolecular interaction in the cocrystal **DNIB·4PYPY** is the π-stacking between the two components. In Fig. 8(*a*) this is shown as the interaction between the central **4PYPY** molecule and the green molecule labelled **PSCT**. This interaction exhibits the highest dispersion component and moderate electrostatic component to the overall intermolecular interaction energy of −45.5 kJ mol^{-1}. The head-to-tail π-stacking between two **4PYPY** molecules, shown in Fig. 8(*a*) as the interaction between the central molecule and the molecule labelled **PSPY**, turquoise, has lower dispersion and electrostatic components with a total intermolecular interaction energy of −42.5 kJ mol^{-1}. In contrast, the intermolecular interaction between the halogen bonded molecules has the lowest surface area contact resulting in the lowest dispersion component. This interaction does have the highest electrostatic component with an overall intermolecular interaction energy of −29.9 kJ mol^{-1}. The π-stacking between two **DNIB** molecules has interaction energy of −26.1 kJ mol^{-1} shown as **PSDN** in Fig. 8(*b*).

The intermolecular interaction energies within the **BTFIB·4PYPY** cocrystal were similarly calculated individually for each component. In this cocrystal, the two unique **4PYPY** π-stacking interactions, labelled **PSPY1** and **PSPY2** in Fig. 9(*a*), are the strongest with interaction energies of −43.4 and −40.5 kJ mol^{-1}, respectively. The π-stacked **BTFIB** molecules with maximum overlap have interaction energy of −38.4 kJ mol^{-1} illustrated as **PSTF1** in Fig. 9(*b*). The offset π-stacked **BTFIB**···**BTFIB** interaction, **PSTF2** in Fig. 9(*b*), has lower electrostatic and dispersion components to the overall energy of interaction of −26.7 kJ mol^{-1}. The halogen-bond interaction, **XB** in Fig. 9(*b*), has the highest electrostatic component of the intermolecular interactions with an overall energy of interaction of −24.3 kJ mol^{-1}.

7. Database survey

While 1-iodo-3,5-dinitrobenzene is not a common halogen-bond donor, a search of the Cambridge Crystallographic

Table 3
Experimental details.

	DNIB·4PYPY	**BTFIB·4PYPY**
Crystal data		
Chemical formula	$C_6H_3IN_2O_4·C_9H_{12}N_2$	$C_8H_3F_6I·C_9H_{12}N_2$
M_r	442.21	488.21
Crystal system, space group	Triclinic, $P\overline{1}$	Triclinic, $P\overline{1}$
Temperature (K)	100	100
a, b, c (Å)	7.9441 (14), 8.4866 (15), 12.139 (2)	7.9777 (4), 8.5138 (4), 14.1679 (7)
α, β, γ (°)	82.209 (2), 87.800 (2), 83.219 (2)	101.579 (1), 103.264 (1), 100.370 (1)
V (Å^3)	805.0 (2)	891.40 (8)
Z	2	2
Radiation type	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	2.02	1.86
Crystal size (mm)	0.18 × 0.05 × 0.04	0.41 × 0.20 × 0.11
Data collection		
Diffractometer	Bruker APEXI CCD	Bruker APEXI CCD
Absorption correction	Multi-scan (*SADABS*; Krause *et al.*, 2015)	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.565, 0.746	0.669, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	8115, 3527, 3181	11576, 3979, 3517
R_{int}	0.025	0.021
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.641	0.644
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.024, 0.057, 1.04	0.042, 0.110, 1.03
No. of reflections	3527	3979
No. of parameters	217	235
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.91, −0.53	2.23, −1.21

Computer programs: *SMART* and *SAINT* (Bruker, 2014), *SHELXT2018/2* (Sheldrick, 2015*a*), *SHELXL2019/2* (Sheldrick, 2015*b*) and *X-SEED-4* (Barbour, 2020).

Database (CSD, Version 6.0.1, Nov 2025; Groom *et al.*, 2016) using Conquest Version 2025.3.0, Build 466532 (Bruno *et al.*, 2002) for structures containing the **DNIB** halogen bonded to an amine yielded eight structures. Thus in 2009, Rissanen reported polymorphism in the 2:1 halogen-bonded cocrystals of **DNIB** with 1,4-diazabicyclo[2.2.2]octane (Raatikainen & Rissanen, 2009). This study also reported the 1:1 cocrystal **DNIB** with 4,4-bipyridine that featured both a halogen bond and a C—H···N hydrogen bond to the more acidic H atom between the two nitro substituents. In 2018, 1:1 halogen-bonded cocrystals of **DNIB** with the thiophene-substituted pyridines 4-([2,2′-bithiophen]-5-yl)pyridine and 4-[5-(furan-2-yl)thiophen-2-yl]pyridine were reported as part of a computational study of substituent and hybridization effects on halogen bonding (Nguyen *et al.*, 2018). In the same year, we reported the structure of the cocrystal of **DNIB** with 4-(*N*,*N*-dimethylamino)pyridine (Nwachukwu *et al.*, 2018). Desiraju and coworkers also incorporated halogen bonding to **DNIB** in their elegant study of cocrystallization and the formation of ternary cocrystals (Jain *et al.*, 2021). In contrast, there are no halogen-bonding studies with 1-iodo-3,5-bis(trifluoromethyl) benzene, **BTFIB**, prior to this report.

8. Synthesis and crystallization

The compounds and solvents used in this study are commercially available and were used without purification. Equimolar amounts, 0.1 mmol, of each component were weighed and placed in a small screw-cap vial and dichloromethane added to effect complete solution of both compounds. The lid was loosely attached to permit slow evaporation of the solvent. Once crystals formed, the remaining solvent was removed and the crystals removed for X-ray studies. The mixture of 1-iodo-3,5-dinitrobenzene and 4-pyrrolidinopyridine, formed a mass of orange-coloured crystals, **DNIB·4PYPY**, the mixture 1-iodo-3,5-bis(trifluoromethyl)benzene and 4-pyrrolidinopyridine formed colourless crystals, **BTFIB·4PYPY**.

9. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. The structure of **BTFIB·4PYPY** was solved with *SHELXT* in space group $P\overline{1}$ with two molecules of each component in the asymmetric unit. *PLATON* ADDSYM (Spek, 2003) was used to transform this to the structure containing only one molecule of each component in the same space group. H atoms were positioned geometrically (C—H = 0.95–0.99 Å) and refined as riding with U_{iso}(H) = 1.2–1.5U_{eq}(C).

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supporting information

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4-Pyrrolidinopyridine as halogen-bond acceptor in cocrystals

Eric Bosch

Computing details

4-(Pyrrolidin-1-yl)pyridine–1-iodo-3,5-dinitrobenzene (1/1) (DNIB4PYPY)

Crystal data

$C_6H_3IN_2O_4·C_9H_{12}N_2$
M_r = 442.21
Triclinic, $P\overline{1}$
a = 7.9441 (14) Å
b = 8.4866 (15) Å
c = 12.139 (2) Å
α = 82.209 (2)°
β = 87.800 (2)°
γ = 83.219 (2)°
V = 805.0 (2) Å^3
Z = 2
$F(000)$ = 436
D_x = 1.824 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4152 reflections
θ = 2.4–27.1°
μ = 2.02 mm^{-1}
T = 100 K
Block, red
0.18 × 0.05 × 0.04 mm

Data collection

Bruker APEXI CCD diffractometer
Detector resolution: 8.3660 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
T_{min} = 0.565, T_{max} = 0.746
8115 measured reflections
3527 independent reflections
3181 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 27.1°, θ_{min} = 2.4°
h = −10→10
k = −10→10
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.024
$wR(F^2)$ = 0.057
S = 1.04
3527 reflections
217 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0285P)^2 + 0.2789P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.91 e Å^{-3}
$\Delta\rho_{min}$ = −0.53 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	0.39367 (2)	0.48998 (2)	0.65240 (2)	0.01837 (6)
O2	0.6369 (3)	1.0467 (2)	0.92236 (17)	0.0283 (5)
O3	0.8608 (3)	0.5175 (2)	1.08618 (16)	0.0275 (4)
O4	0.7828 (3)	0.3099 (2)	1.02383 (17)	0.0296 (5)
O1	0.4709 (3)	1.0748 (2)	0.78321 (19)	0.0346 (5)
N1	0.2416 (3)	0.3150 (3)	0.50366 (19)	0.0227 (5)
N2	0.0925 (3)	0.0008 (3)	0.29720 (19)	0.0192 (5)
N3	0.5626 (3)	0.9924 (3)	0.85229 (19)	0.0225 (5)
N4	0.7850 (3)	0.4544 (3)	1.02106 (19)	0.0209 (5)
C5	0.1385 (3)	0.1016 (3)	0.3650 (2)	0.0159 (5)
C10	0.5159 (3)	0.5906 (3)	0.7724 (2)	0.0163 (5)
C15	0.6085 (3)	0.4906 (3)	0.8549 (2)	0.0161 (5)
H15	0.617536	0.377528	0.857578	0.019*
C3	0.2955 (4)	0.1573 (3)	0.5187 (2)	0.0223 (6)
H3	0.370755	0.118433	0.577767	0.027*
C1	0.0823 (3)	0.2664 (3)	0.3493 (2)	0.0189 (5)
H1	0.007726	0.310176	0.290738	0.023*
C4	0.2489 (3)	0.0479 (3)	0.4543 (2)	0.0207 (5)
H4	0.290598	−0.062168	0.469952	0.025*
C6	0.1261 (4)	−0.1737 (3)	0.3195 (2)	0.0227 (6)
H6A	0.103579	−0.212086	0.398760	0.027*
H6B	0.245087	−0.210446	0.299989	0.027*
C11	0.5032 (3)	0.7561 (3)	0.7701 (2)	0.0174 (5)
H11	0.441806	0.825365	0.713548	0.021*
C12	0.5819 (3)	0.8174 (3)	0.8518 (2)	0.0174 (5)
C14	0.6874 (3)	0.5607 (3)	0.9333 (2)	0.0173 (5)
C2	0.1363 (3)	0.3640 (3)	0.4197 (2)	0.0213 (6)
H2	0.095426	0.474592	0.407427	0.026*
C13	0.6760 (3)	0.7230 (3)	0.9350 (2)	0.0191 (5)
H13	0.729706	0.768028	0.989943	0.023*
C7	0.0035 (4)	−0.2332 (4)	0.2450 (2)	0.0283 (6)
H7A	0.056018	−0.330624	0.214921	0.034*
H7B	−0.101840	−0.257661	0.286700	0.034*
C8	−0.0334 (4)	−0.0957 (4)	0.1520 (2)	0.0285 (6)
H8A	0.053908	−0.100886	0.091809	0.034*
H8B	−0.146104	−0.097701	0.120528	0.034*
C9	−0.0290 (4)	0.0531 (4)	0.2081 (2)	0.0256 (6)
H9A	0.010003	0.141619	0.155250	0.031*
H9B	−0.142273	0.088875	0.238628	0.031*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.02050 (9)	0.02056 (9)	0.01580 (9)	−0.00533 (6)	−0.00338 (6)	−0.00528 (6)
O2	0.0354 (12)	0.0230 (10)	0.0305 (11)	−0.0106 (9)	−0.0011 (9)	−0.0109 (9)

O3	0.0299 (11)	0.0341 (11)	0.0204 (10)	−0.0045 (9)	−0.0075 (8)	−0.0076 (9)
O4	0.0407 (12)	0.0213 (10)	0.0257 (11)	0.0004 (9)	−0.0106 (9)	−0.0003 (8)
O1	0.0449 (14)	0.0203 (10)	0.0383 (13)	−0.0011 (10)	−0.0136 (11)	−0.0015 (9)
N1	0.0267 (13)	0.0237 (12)	0.0204 (12)	−0.0061 (10)	−0.0014 (9)	−0.0089 (10)
N2	0.0217 (11)	0.0183 (11)	0.0183 (11)	−0.0015 (9)	−0.0070 (9)	−0.0044 (9)
N3	0.0264 (12)	0.0165 (11)	0.0258 (13)	−0.0042 (9)	0.0002 (10)	−0.0056 (10)
N4	0.0203 (11)	0.0249 (12)	0.0175 (11)	−0.0008 (9)	−0.0019 (9)	−0.0038 (9)
C5	0.0156 (12)	0.0175 (12)	0.0156 (12)	−0.0027 (10)	0.0004 (10)	−0.0045 (10)
C10	0.0152 (12)	0.0207 (12)	0.0146 (12)	−0.0046 (10)	0.0006 (9)	−0.0066 (10)
C15	0.0170 (12)	0.0165 (12)	0.0157 (12)	−0.0038 (10)	0.0018 (9)	−0.0045 (10)
C3	0.0222 (14)	0.0278 (14)	0.0182 (13)	−0.0031 (11)	−0.0057 (11)	−0.0055 (11)
C1	0.0199 (13)	0.0190 (13)	0.0175 (13)	−0.0004 (10)	−0.0020 (10)	−0.0030 (10)
C4	0.0199 (13)	0.0221 (13)	0.0195 (13)	0.0008 (11)	−0.0052 (10)	−0.0022 (11)
C6	0.0305 (15)	0.0175 (13)	0.0216 (14)	−0.0053 (11)	−0.0051 (11)	−0.0046 (11)
C11	0.0174 (13)	0.0219 (13)	0.0130 (12)	−0.0039 (10)	0.0006 (10)	−0.0012 (10)
C12	0.0197 (13)	0.0133 (12)	0.0204 (13)	−0.0040 (10)	0.0035 (10)	−0.0061 (10)
C14	0.0170 (12)	0.0208 (13)	0.0145 (12)	−0.0014 (10)	−0.0007 (10)	−0.0039 (10)
C2	0.0244 (14)	0.0169 (13)	0.0227 (14)	−0.0027 (11)	0.0027 (11)	−0.0035 (11)
C13	0.0193 (13)	0.0219 (13)	0.0181 (13)	−0.0061 (10)	−0.0009 (10)	−0.0066 (10)
C7	0.0324 (16)	0.0304 (15)	0.0265 (15)	−0.0126 (13)	0.0002 (12)	−0.0123 (13)
C8	0.0247 (15)	0.0373 (17)	0.0263 (16)	−0.0060 (13)	−0.0058 (12)	−0.0104 (13)
C9	0.0252 (15)	0.0287 (15)	0.0241 (15)	−0.0002 (12)	−0.0098 (12)	−0.0076 (12)

Geometric parameters (Å, º)

I1—C10	2.106 (2)	C1—C2	1.380 (4)
O2—N3	1.222 (3)	C1—H1	0.9500
O3—N4	1.225 (3)	C4—H4	0.9500
O4—N4	1.225 (3)	C6—C7	1.525 (4)
O1—N3	1.221 (3)	C6—H6A	0.9900
N1—C2	1.334 (4)	C6—H6B	0.9900
N1—C3	1.346 (4)	C11—C12	1.382 (3)
N2—C5	1.353 (3)	C11—H11	0.9500
N2—C6	1.464 (3)	C12—C13	1.384 (4)
N2—C9	1.465 (3)	C14—C13	1.373 (4)
N3—C12	1.476 (3)	C2—H2	0.9500
N4—C14	1.479 (3)	C13—H13	0.9500
C5—C1	1.407 (4)	C7—C8	1.519 (4)
C5—C4	1.414 (4)	C7—H7A	0.9900
C10—C11	1.393 (4)	C7—H7B	0.9900
C10—C15	1.394 (3)	C8—C9	1.518 (4)
C15—C14	1.393 (3)	C8—H8A	0.9900
C15—H15	0.9500	C8—H8B	0.9900
C3—C4	1.382 (4)	C9—H9A	0.9900
C3—H3	0.9500	C9—H9B	0.9900
C2—N1—C3	115.5 (2)	C12—C11—C10	118.5 (2)
C5—N2—C6	123.9 (2)	C12—C11—H11	120.7

C5—N2—C9	122.4 (2)	C10—C11—H11	120.7
C6—N2—C9	112.3 (2)	C11—C12—C13	123.5 (2)
O1—N3—O2	123.7 (2)	C11—C12—N3	118.6 (2)
O1—N3—C12	117.8 (2)	C13—C12—N3	117.9 (2)
O2—N3—C12	118.5 (2)	C13—C14—C15	123.4 (2)
O4—N4—O3	124.9 (2)	C13—C14—N4	118.3 (2)
O4—N4—C14	117.5 (2)	C15—C14—N4	118.2 (2)
O3—N4—C14	117.6 (2)	N1—C2—C1	125.1 (2)
N2—C5—C1	122.0 (2)	N1—C2—H2	117.5
N2—C5—C4	121.9 (2)	C1—C2—H2	117.5
C1—C5—C4	116.1 (2)	C14—C13—C12	116.1 (2)
C11—C10—C15	120.1 (2)	C14—C13—H13	121.9
C11—C10—I1	120.25 (19)	C12—C13—H13	121.9
C15—C10—I1	119.66 (18)	C8—C7—C6	104.7 (2)
C14—C15—C10	118.3 (2)	C8—C7—H7A	110.8
C14—C15—H15	120.8	C6—C7—H7A	110.8
C10—C15—H15	120.8	C8—C7—H7B	110.8
N1—C3—C4	124.6 (2)	C6—C7—H7B	110.8
N1—C3—H3	117.7	H7A—C7—H7B	108.9
C4—C3—H3	117.7	C9—C8—C7	104.1 (2)
C2—C1—C5	119.4 (2)	C9—C8—H8A	110.9
C2—C1—H1	120.3	C7—C8—H8A	110.9
C5—C1—H1	120.3	C9—C8—H8B	110.9
C3—C4—C5	119.3 (2)	C7—C8—H8B	110.9
C3—C4—H4	120.3	H8A—C8—H8B	109.0
C5—C4—H4	120.3	N2—C9—C8	103.6 (2)
N2—C6—C7	104.0 (2)	N2—C9—H9A	111.0
N2—C6—H6A	111.0	C8—C9—H9A	111.0
C7—C6—H6A	111.0	N2—C9—H9B	111.0
N2—C6—H6B	111.0	C8—C9—H9B	111.0
C7—C6—H6B	111.0	H9A—C9—H9B	109.0
H6A—C6—H6B	109.0		

C6—N2—C5—C1	−170.4 (3)	O1—N3—C12—C13	−175.5 (3)
C9—N2—C5—C1	−4.8 (4)	O2—N3—C12—C13	3.5 (4)
C6—N2—C5—C4	11.0 (4)	C10—C15—C14—C13	1.3 (4)
C9—N2—C5—C4	176.6 (2)	C10—C15—C14—N4	179.8 (2)
C11—C10—C15—C14	−0.4 (4)	O4—N4—C14—C13	174.3 (2)
I1—C10—C15—C14	−179.76 (18)	O3—N4—C14—C13	−5.0 (4)
C2—N1—C3—C4	−0.1 (4)	O4—N4—C14—C15	−4.2 (4)
N2—C5—C1—C2	−178.6 (2)	O3—N4—C14—C15	176.4 (2)
C4—C5—C1—C2	0.1 (4)	C3—N1—C2—C1	−0.5 (4)
N1—C3—C4—C5	0.7 (4)	C5—C1—C2—N1	0.5 (4)
N2—C5—C4—C3	178.1 (3)	C15—C14—C13—C12	−0.9 (4)
C1—C5—C4—C3	−0.6 (4)	N4—C14—C13—C12	−179.3 (2)
C5—N2—C6—C7	161.7 (3)	C11—C12—C13—C14	−0.5 (4)
C9—N2—C6—C7	−5.2 (3)	N3—C12—C13—C14	178.4 (2)
C15—C10—C11—C12	−0.9 (4)	N2—C6—C7—C8	24.1 (3)

I1—C10—C11—C12	178.47 (19)	C6—C7—C8—C9	−33.9 (3)
C10—C11—C12—C13	1.4 (4)	C5—N2—C9—C8	177.2 (2)
C10—C11—C12—N3	−177.5 (2)	C6—N2—C9—C8	−15.7 (3)
O1—N3—C12—C11	3.4 (4)	C7—C8—C9—N2	30.1 (3)
O2—N3—C12—C11	−177.6 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O1^{i}	0.95	2.61	3.499 (3)	156
C7—H7*B*···I1^{ii}	0.99	3.22	4.104 (3)	150
C8—H8*A*···O2^{iii}	0.99	2.55	3.301 (4)	133
C8—H8*A*···O4^{iv}	0.99	2.61	3.420 (4)	140
C9—H9*A*···O4^{v}	0.99	2.61	3.186 (3)	117

Symmetry codes: (i) *x*, *y*−1, *z*; (ii) −*x*, −*y*, −*z*+1; (iii) −*x*+1, −*y*+1, −*z*+1; (iv) −*x*+1, −*y*, −*z*+1; (v) *x*−1, *y*, *z*−1.

4-(Pyrrolidin-1-yl)pyridine–1-iodo-3,5-bis(trifluoromethyl)benzene (1/1) (BTFIB4PYPY)

Crystal data

$C_8H_3F_6I·C_9H_{12}N_2$
M_r = 488.21
Triclinic, $P\overline{1}$
a = 7.9777 (4) Å
b = 8.5138 (4) Å
c = 14.1679 (7) Å
α = 101.579 (1)°
β = 103.264 (1)°
γ = 100.370 (1)°
V = 891.40 (8) Å^3
Z = 2
F(000) = 476
D_x = 1.819 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 7472 reflections
θ = 2.5–27.2°
μ = 1.86 mm^{-1}
T = 100 K
Cut, colourless
0.41 × 0.20 × 0.11 mm

Data collection

Bruker APEXI CCD diffractometer
Detector resolution: 8.3660 pixels mm^{-1}
φ and *ω* scans
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
T_{min} = 0.669, T_{max} = 0.746
11576 measured reflections
3979 independent reflections
3517 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
θ_{max} = 27.2°, θ_{min} = 1.5°
h = −10→10
k = −10→10
l = −18→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.110
S = 1.03
3979 reflections
235 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0623P)^2 + 1.7968P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 2.23 e Å^{-3}
$\Delta\rho_{min}$ = −1.21 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	0.25075 (3)	0.40397 (3)	0.25028 (2)	0.04031 (12)
F3	0.1815 (5)	0.9266 (4)	−0.0400 (2)	0.0699 (10)
F2	0.4058 (5)	0.9949 (4)	0.0867 (4)	0.0981 (16)
F6	0.3083 (8)	0.2154 (6)	−0.1345 (3)	0.1099 (19)
F5	0.0807 (4)	0.2808 (6)	−0.2053 (3)	0.1068 (19)
F1	0.1466 (8)	0.9537 (6)	0.1061 (3)	0.1139 (19)
F4	0.3331 (6)	0.4094 (4)	−0.1980 (3)	0.0930 (15)
C14	0.2424 (5)	0.7210 (5)	0.0425 (3)	0.0371 (9)
C11	0.2431 (5)	0.3922 (5)	0.0328 (3)	0.0348 (8)
H11	0.242389	0.279409	0.029139	0.042*
C12	0.2414 (5)	0.4538 (5)	−0.0508 (3)	0.0311 (8)
C15	0.2465 (5)	0.6632 (5)	0.1279 (3)	0.0348 (9)
H15	0.249785	0.735595	0.189071	0.042*
C10	0.2459 (5)	0.4985 (5)	0.1227 (3)	0.0347 (8)
C16	0.2362 (5)	0.3388 (5)	−0.1481 (3)	0.0318 (8)
C13	0.2399 (5)	0.6171 (5)	−0.0480 (3)	0.0343 (8)
H13	0.237243	0.657398	−0.106043	0.041*
C17	0.2452 (7)	0.8988 (6)	0.0480 (4)	0.0541 (13)
N2	0.2661 (4)	−0.1161 (4)	0.5764 (3)	0.0356 (7)
N1	0.2667 (5)	0.2330 (5)	0.4051 (3)	0.0431 (9)
C4	0.3814 (5)	0.1550 (5)	0.5581 (3)	0.0351 (8)
H4	0.461763	0.186897	0.623266	0.042*
C5	0.2673 (5)	−0.0044 (5)	0.5219 (3)	0.0326 (8)
C9	0.1582 (5)	−0.2865 (5)	0.5391 (3)	0.0414 (10)
H9A	0.177820	−0.341771	0.475465	0.050*
H9B	0.030462	−0.289137	0.528127	0.050*
C6	0.3750 (5)	−0.0824 (5)	0.6811 (3)	0.0335 (8)
H6A	0.368445	0.024343	0.721668	0.040*
H6B	0.500531	−0.080878	0.684121	0.040*
C2	0.1594 (6)	0.0792 (6)	0.3706 (3)	0.0412 (10)
H2	0.081913	0.051437	0.304785	0.049*
C7	0.2906 (6)	−0.2267 (5)	0.7170 (3)	0.0408 (9)
H7A	0.191991	−0.201739	0.744282	0.049*
H7B	0.379140	−0.252966	0.769246	0.049*
C1	0.1534 (5)	−0.0393 (6)	0.4230 (3)	0.0390 (9)
H1	0.073671	−0.144412	0.393517	0.047*
C3	0.3751 (5)	0.2644 (6)	0.4979 (3)	0.0384 (9)
H3	0.453833	0.370463	0.524397	0.046*
C8	0.2226 (6)	−0.3690 (6)	0.6224 (4)	0.0461 (10)

H8A	0.318785	−0.422350	0.610027	0.055*
H8B	0.124415	−0.453024	0.627894	0.055*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.03090 (16)	0.05218 (19)	0.02766 (16)	−0.00391 (11)	0.00906 (11)	−0.00150 (11)
F3	0.095 (3)	0.0434 (16)	0.0525 (18)	0.0249 (16)	−0.0135 (17)	0.0018 (13)
F2	0.079 (2)	0.0322 (15)	0.132 (4)	0.0066 (15)	−0.045 (2)	−0.0038 (18)
F6	0.204 (5)	0.118 (3)	0.0405 (18)	0.122 (4)	0.039 (2)	0.0124 (19)
F5	0.0302 (15)	0.164 (4)	0.067 (2)	−0.0023 (19)	0.0075 (15)	−0.075 (3)
F1	0.192 (5)	0.102 (3)	0.087 (3)	0.112 (4)	0.059 (3)	0.019 (2)
F4	0.126 (3)	0.068 (2)	0.081 (3)	−0.018 (2)	0.081 (3)	−0.0143 (18)
C14	0.0242 (18)	0.040 (2)	0.037 (2)	0.0097 (16)	−0.0009 (15)	−0.0041 (17)
C11	0.0293 (19)	0.0337 (19)	0.034 (2)	−0.0036 (15)	0.0115 (16)	−0.0022 (16)
C12	0.0241 (17)	0.0339 (19)	0.0293 (18)	0.0017 (14)	0.0082 (14)	−0.0017 (15)
C15	0.0208 (17)	0.042 (2)	0.0309 (19)	0.0047 (15)	0.0046 (14)	−0.0097 (16)
C10	0.0225 (17)	0.046 (2)	0.0267 (18)	−0.0022 (15)	0.0087 (14)	−0.0025 (16)
C16	0.0313 (19)	0.0316 (19)	0.0319 (19)	0.0047 (15)	0.0129 (15)	0.0045 (15)
C13	0.0252 (18)	0.039 (2)	0.0322 (19)	0.0077 (15)	0.0016 (15)	0.0008 (16)
C17	0.052 (3)	0.049 (3)	0.045 (3)	0.025 (2)	−0.011 (2)	−0.010 (2)
N2	0.0256 (15)	0.0365 (17)	0.0321 (17)	−0.0010 (13)	0.0048 (13)	−0.0084 (14)
N1	0.0373 (19)	0.057 (2)	0.0322 (18)	0.0114 (17)	0.0122 (15)	0.0012 (16)
C4	0.0291 (18)	0.044 (2)	0.0239 (18)	0.0019 (16)	0.0063 (15)	−0.0040 (16)
C5	0.0246 (17)	0.041 (2)	0.0257 (18)	0.0056 (15)	0.0079 (14)	−0.0058 (15)
C9	0.029 (2)	0.038 (2)	0.043 (2)	−0.0012 (16)	0.0085 (17)	−0.0123 (18)
C6	0.0290 (18)	0.036 (2)	0.0283 (19)	0.0026 (15)	0.0090 (15)	−0.0044 (15)
C2	0.031 (2)	0.056 (3)	0.0280 (19)	0.0111 (19)	0.0056 (16)	−0.0055 (18)
C7	0.037 (2)	0.040 (2)	0.038 (2)	0.0008 (17)	0.0137 (18)	−0.0025 (18)
C1	0.0275 (19)	0.047 (2)	0.031 (2)	0.0051 (17)	0.0041 (16)	−0.0080 (17)
C3	0.033 (2)	0.046 (2)	0.030 (2)	0.0012 (17)	0.0130 (16)	−0.0024 (17)
C8	0.037 (2)	0.039 (2)	0.048 (3)	−0.0032 (18)	0.0093 (19)	−0.0060 (19)

Geometric parameters (Å, º)

I1—C10	2.116 (4)	N1—C3	1.343 (6)
F3—C17	1.319 (6)	N1—C2	1.354 (6)
F2—C17	1.316 (6)	C4—C3	1.383 (6)
F6—C16	1.311 (5)	C4—C5	1.413 (6)
F5—C16	1.264 (5)	C4—H4	0.9500
F1—C17	1.338 (7)	C5—C1	1.424 (5)
F4—C16	1.309 (5)	C9—C8	1.528 (7)
C14—C15	1.391 (6)	C9—H9A	0.9900
C14—C13	1.397 (6)	C9—H9B	0.9900
C14—C17	1.496 (6)	C6—C7	1.527 (6)
C11—C12	1.388 (6)	C6—H6A	0.9900
C11—C10	1.400 (5)	C6—H6B	0.9900
C11—H11	0.9500	C2—C1	1.367 (7)

C12—C13	1.385 (6)	C2—H2	0.9500
C12—C16	1.508 (5)	C7—C8	1.529 (6)
C15—C10	1.388 (6)	C7—H7A	0.9900
C15—H15	0.9500	C7—H7B	0.9900
C13—H13	0.9500	C1—H1	0.9500
N2—C5	1.340 (6)	C3—H3	0.9500
N2—C9	1.470 (5)	C8—H8A	0.9900
N2—C6	1.478 (5)	C8—H8B	0.9900
C15—C14—C13	121.3 (4)	N2—C5—C4	122.5 (4)
C15—C14—C17	119.5 (4)	N2—C5—C1	122.0 (4)
C13—C14—C17	119.2 (4)	C4—C5—C1	115.5 (4)
C12—C11—C10	119.1 (4)	N2—C9—C8	104.0 (3)
C12—C11—H11	120.5	N2—C9—H9A	111.0
C10—C11—H11	120.5	C8—C9—H9A	111.0
C13—C12—C11	121.7 (4)	N2—C9—H9B	111.0
C13—C12—C16	119.2 (4)	C8—C9—H9B	111.0
C11—C12—C16	119.1 (3)	H9A—C9—H9B	109.0
C10—C15—C14	119.3 (3)	N2—C6—C7	103.0 (3)
C10—C15—H15	120.3	N2—C6—H6A	111.2
C14—C15—H15	120.3	C7—C6—H6A	111.2
C15—C10—C11	120.3 (4)	N2—C6—H6B	111.2
C15—C10—I1	120.8 (3)	C7—C6—H6B	111.2
C11—C10—I1	118.8 (3)	H6A—C6—H6B	109.1
F5—C16—F4	107.9 (4)	N1—C2—C1	125.3 (4)
F5—C16—F6	108.4 (5)	N1—C2—H2	117.3
F4—C16—F6	101.9 (4)	C1—C2—H2	117.3
F5—C16—C12	112.6 (3)	C6—C7—C8	103.7 (4)
F4—C16—C12	112.6 (3)	C6—C7—H7A	111.0
F6—C16—C12	112.7 (3)	C8—C7—H7A	111.0
C12—C13—C14	118.3 (4)	C6—C7—H7B	111.0
C12—C13—H13	120.9	C8—C7—H7B	111.0
C14—C13—H13	120.9	H7A—C7—H7B	109.0
F2—C17—F3	107.1 (5)	C2—C1—C5	119.6 (4)
F2—C17—F1	106.4 (5)	C2—C1—H1	120.2
F3—C17—F1	106.1 (4)	C5—C1—H1	120.2
F2—C17—C14	112.5 (4)	N1—C3—C4	125.3 (4)
F3—C17—C14	113.2 (4)	N1—C3—H3	117.3
F1—C17—C14	111.1 (5)	C4—C3—H3	117.3
C5—N2—C9	124.1 (3)	C9—C8—C7	103.9 (4)
C5—N2—C6	123.8 (3)	C9—C8—H8A	111.0
C9—N2—C6	112.1 (3)	C7—C8—H8A	111.0
C3—N1—C2	114.7 (4)	C9—C8—H8B	111.0
C3—C4—C5	119.4 (4)	C7—C8—H8B	111.0
C3—C4—H4	120.3	H8A—C8—H8B	109.0
C5—C4—H4	120.3		
C10—C11—C12—C13	−0.9 (6)	C15—C14—C17—F1	−38.9 (6)

C10—C11—C12—C16	−179.3 (3)	C13—C14—C17—F1	142.6 (4)
C13—C14—C15—C10	−0.9 (6)	C9—N2—C5—C4	−175.8 (4)
C17—C14—C15—C10	−179.3 (4)	C6—N2—C5—C4	3.6 (6)
C14—C15—C10—C11	0.7 (6)	C9—N2—C5—C1	4.0 (6)
C14—C15—C10—I1	−179.7 (3)	C6—N2—C5—C1	−176.6 (3)
C12—C11—C10—C15	0.1 (6)	C3—C4—C5—N2	−179.7 (4)
C12—C11—C10—I1	−179.5 (3)	C3—C4—C5—C1	0.5 (5)
C13—C12—C16—F5	−83.0 (5)	C5—N2—C9—C8	172.2 (4)
C11—C12—C16—F5	95.5 (5)	C6—N2—C9—C8	−7.3 (4)
C13—C12—C16—F4	39.3 (5)	C5—N2—C6—C7	165.2 (4)
C11—C12—C16—F4	−142.2 (4)	C9—N2—C6—C7	−15.4 (4)
C13—C12—C16—F6	153.9 (4)	C3—N1—C2—C1	1.0 (6)
C11—C12—C16—F6	−27.6 (6)	N2—C6—C7—C8	31.7 (4)
C11—C12—C13—C14	0.8 (6)	N1—C2—C1—C5	−0.3 (6)
C16—C12—C13—C14	179.2 (3)	N2—C5—C1—C2	179.7 (4)
C15—C14—C13—C12	0.1 (6)	C4—C5—C1—C2	−0.5 (5)
C17—C14—C13—C12	178.6 (4)	C2—N1—C3—C4	−0.9 (6)
C15—C14—C17—F2	80.2 (6)	C5—C4—C3—N1	0.2 (6)
C13—C14—C17—F2	−98.3 (5)	N2—C9—C8—C7	27.0 (4)
C15—C14—C17—F3	−158.2 (4)	C6—C7—C8—C9	−36.7 (4)
C13—C14—C17—F3	23.3 (6)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6*A*···F6^{i}	0.99	2.54	3.465 (5)	156
C1—H1···F5^{ii}	0.95	2.59	3.265 (5)	128

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

supporting information

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4-Pyrrolidinopyridine as halogen-bond acceptor in cocrystals

Eric Bosch

Computing details

4-(Pyrrolidin-1-yl)pyridine–1-iodo-3,5-dinitrobenzene (1/1) (DNIB4PYPY)

Crystal data

$C_6H_3IN_2O_4·C_9H_{12}N_2$
M_r = 442.21
Triclinic, $P\overline{1}$
a = 7.9441 (14) Å
b = 8.4866 (15) Å
c = 12.139 (2) Å
α = 82.209 (2)°
β = 87.800 (2)°
γ = 83.219 (2)°
V = 805.0 (2) Å^3
Z = 2
$F(000)$ = 436
D_x = 1.824 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4152 reflections
θ = 2.4–27.1°
μ = 2.02 mm^{-1}
T = 100 K
Block, red
0.18 × 0.05 × 0.04 mm

Data collection

Bruker APEXI CCD diffractometer
Detector resolution: 8.3660 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
T_{min} = 0.565, T_{max} = 0.746
8115 measured reflections
3527 independent reflections
3181 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 27.1°, θ_{min} = 2.4°
h = −10→10
k = −10→10
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.024
$wR(F^2)$ = 0.057
S = 1.04
3527 reflections
217 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0285P)^2 + 0.2789P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.91 e Å^{-3}
$\Delta\rho_{min}$ = −0.53 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	0.39367 (2)	0.48998 (2)	0.65240 (2)	0.01837 (6)
O2	0.6369 (3)	1.0467 (2)	0.92236 (17)	0.0283 (5)
O3	0.8608 (3)	0.5175 (2)	1.08618 (16)	0.0275 (4)
O4	0.7828 (3)	0.3099 (2)	1.02383 (17)	0.0296 (5)
O1	0.4709 (3)	1.0748 (2)	0.78321 (19)	0.0346 (5)
N1	0.2416 (3)	0.3150 (3)	0.50366 (19)	0.0227 (5)
N2	0.0925 (3)	0.0008 (3)	0.29720 (19)	0.0192 (5)
N3	0.5626 (3)	0.9924 (3)	0.85229 (19)	0.0225 (5)
N4	0.7850 (3)	0.4544 (3)	1.02106 (19)	0.0209 (5)
C5	0.1385 (3)	0.1016 (3)	0.3650 (2)	0.0159 (5)
C10	0.5159 (3)	0.5906 (3)	0.7724 (2)	0.0163 (5)
C15	0.6085 (3)	0.4906 (3)	0.8549 (2)	0.0161 (5)
H15	0.617536	0.377528	0.857578	0.019*
C3	0.2955 (4)	0.1573 (3)	0.5187 (2)	0.0223 (6)
H3	0.370755	0.118433	0.577767	0.027*
C1	0.0823 (3)	0.2664 (3)	0.3493 (2)	0.0189 (5)
H1	0.007726	0.310176	0.290738	0.023*
C4	0.2489 (3)	0.0479 (3)	0.4543 (2)	0.0207 (5)
H4	0.290598	−0.062168	0.469952	0.025*
C6	0.1261 (4)	−0.1737 (3)	0.3195 (2)	0.0227 (6)
H6A	0.103579	−0.212086	0.398760	0.027*
H6B	0.245087	−0.210446	0.299989	0.027*
C11	0.5032 (3)	0.7561 (3)	0.7701 (2)	0.0174 (5)
H11	0.441806	0.825365	0.713548	0.021*
C12	0.5819 (3)	0.8174 (3)	0.8518 (2)	0.0174 (5)
C14	0.6874 (3)	0.5607 (3)	0.9333 (2)	0.0173 (5)
C2	0.1363 (3)	0.3640 (3)	0.4197 (2)	0.0213 (6)
H2	0.095426	0.474592	0.407427	0.026*
C13	0.6760 (3)	0.7230 (3)	0.9350 (2)	0.0191 (5)
H13	0.729706	0.768028	0.989943	0.023*
C7	0.0035 (4)	−0.2332 (4)	0.2450 (2)	0.0283 (6)
H7A	0.056018	−0.330624	0.214921	0.034*
H7B	−0.101840	−0.257661	0.286700	0.034*
C8	−0.0334 (4)	−0.0957 (4)	0.1520 (2)	0.0285 (6)
H8A	0.053908	−0.100886	0.091809	0.034*
H8B	−0.146104	−0.097701	0.120528	0.034*
C9	−0.0290 (4)	0.0531 (4)	0.2081 (2)	0.0256 (6)
H9A	0.010003	0.141619	0.155250	0.031*
H9B	−0.142273	0.088875	0.238628	0.031*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.02050 (9)	0.02056 (9)	0.01580 (9)	−0.00533 (6)	−0.00338 (6)	−0.00528 (6)
O2	0.0354 (12)	0.0230 (10)	0.0305 (11)	−0.0106 (9)	−0.0011 (9)	−0.0109 (9)

O3	0.0299 (11)	0.0341 (11)	0.0204 (10)	−0.0045 (9)	−0.0075 (8)	−0.0076 (9)
O4	0.0407 (12)	0.0213 (10)	0.0257 (11)	0.0004 (9)	−0.0106 (9)	−0.0003 (8)
O1	0.0449 (14)	0.0203 (10)	0.0383 (13)	−0.0011 (10)	−0.0136 (11)	−0.0015 (9)
N1	0.0267 (13)	0.0237 (12)	0.0204 (12)	−0.0061 (10)	−0.0014 (9)	−0.0089 (10)
N2	0.0217 (11)	0.0183 (11)	0.0183 (11)	−0.0015 (9)	−0.0070 (9)	−0.0044 (9)
N3	0.0264 (12)	0.0165 (11)	0.0258 (13)	−0.0042 (9)	0.0002 (10)	−0.0056 (10)
N4	0.0203 (11)	0.0249 (12)	0.0175 (11)	−0.0008 (9)	−0.0019 (9)	−0.0038 (9)
C5	0.0156 (12)	0.0175 (12)	0.0156 (12)	−0.0027 (10)	0.0004 (10)	−0.0045 (10)
C10	0.0152 (12)	0.0207 (12)	0.0146 (12)	−0.0046 (10)	0.0006 (9)	−0.0066 (10)
C15	0.0170 (12)	0.0165 (12)	0.0157 (12)	−0.0038 (10)	0.0018 (9)	−0.0045 (10)
C3	0.0222 (14)	0.0278 (14)	0.0182 (13)	−0.0031 (11)	−0.0057 (11)	−0.0055 (11)
C1	0.0199 (13)	0.0190 (13)	0.0175 (13)	−0.0004 (10)	−0.0020 (10)	−0.0030 (10)
C4	0.0199 (13)	0.0221 (13)	0.0195 (13)	0.0008 (11)	−0.0052 (10)	−0.0022 (11)
C6	0.0305 (15)	0.0175 (13)	0.0216 (14)	−0.0053 (11)	−0.0051 (11)	−0.0046 (11)
C11	0.0174 (13)	0.0219 (13)	0.0130 (12)	−0.0039 (10)	0.0006 (10)	−0.0012 (10)
C12	0.0197 (13)	0.0133 (12)	0.0204 (13)	−0.0040 (10)	0.0035 (10)	−0.0061 (10)
C14	0.0170 (12)	0.0208 (13)	0.0145 (12)	−0.0014 (10)	−0.0007 (10)	−0.0039 (10)
C2	0.0244 (14)	0.0169 (13)	0.0227 (14)	−0.0027 (11)	0.0027 (11)	−0.0035 (11)
C13	0.0193 (13)	0.0219 (13)	0.0181 (13)	−0.0061 (10)	−0.0009 (10)	−0.0066 (10)
C7	0.0324 (16)	0.0304 (15)	0.0265 (15)	−0.0126 (13)	0.0002 (12)	−0.0123 (13)
C8	0.0247 (15)	0.0373 (17)	0.0263 (16)	−0.0060 (13)	−0.0058 (12)	−0.0104 (13)
C9	0.0252 (15)	0.0287 (15)	0.0241 (15)	−0.0002 (12)	−0.0098 (12)	−0.0076 (12)

Geometric parameters (Å, º)

I1—C10	2.106 (2)	C1—C2	1.380 (4)
O2—N3	1.222 (3)	C1—H1	0.9500
O3—N4	1.225 (3)	C4—H4	0.9500
O4—N4	1.225 (3)	C6—C7	1.525 (4)
O1—N3	1.221 (3)	C6—H6A	0.9900
N1—C2	1.334 (4)	C6—H6B	0.9900
N1—C3	1.346 (4)	C11—C12	1.382 (3)
N2—C5	1.353 (3)	C11—H11	0.9500
N2—C6	1.464 (3)	C12—C13	1.384 (4)
N2—C9	1.465 (3)	C14—C13	1.373 (4)
N3—C12	1.476 (3)	C2—H2	0.9500
N4—C14	1.479 (3)	C13—H13	0.9500
C5—C1	1.407 (4)	C7—C8	1.519 (4)
C5—C4	1.414 (4)	C7—H7A	0.9900
C10—C11	1.393 (4)	C7—H7B	0.9900
C10—C15	1.394 (3)	C8—C9	1.518 (4)
C15—C14	1.393 (3)	C8—H8A	0.9900
C15—H15	0.9500	C8—H8B	0.9900
C3—C4	1.382 (4)	C9—H9A	0.9900
C3—H3	0.9500	C9—H9B	0.9900
C2—N1—C3	115.5 (2)	C12—C11—C10	118.5 (2)
C5—N2—C6	123.9 (2)	C12—C11—H11	120.7

C5—N2—C9	122.4 (2)	C10—C11—H11	120.7
C6—N2—C9	112.3 (2)	C11—C12—C13	123.5 (2)
O1—N3—O2	123.7 (2)	C11—C12—N3	118.6 (2)
O1—N3—C12	117.8 (2)	C13—C12—N3	117.9 (2)
O2—N3—C12	118.5 (2)	C13—C14—C15	123.4 (2)
O4—N4—O3	124.9 (2)	C13—C14—N4	118.3 (2)
O4—N4—C14	117.5 (2)	C15—C14—N4	118.2 (2)
O3—N4—C14	117.6 (2)	N1—C2—C1	125.1 (2)
N2—C5—C1	122.0 (2)	N1—C2—H2	117.5
N2—C5—C4	121.9 (2)	C1—C2—H2	117.5
C1—C5—C4	116.1 (2)	C14—C13—C12	116.1 (2)
C11—C10—C15	120.1 (2)	C14—C13—H13	121.9
C11—C10—I1	120.25 (19)	C12—C13—H13	121.9
C15—C10—I1	119.66 (18)	C8—C7—C6	104.7 (2)
C14—C15—C10	118.3 (2)	C8—C7—H7A	110.8
C14—C15—H15	120.8	C6—C7—H7A	110.8
C10—C15—H15	120.8	C8—C7—H7B	110.8
N1—C3—C4	124.6 (2)	C6—C7—H7B	110.8
N1—C3—H3	117.7	H7A—C7—H7B	108.9
C4—C3—H3	117.7	C9—C8—C7	104.1 (2)
C2—C1—C5	119.4 (2)	C9—C8—H8A	110.9
C2—C1—H1	120.3	C7—C8—H8A	110.9
C5—C1—H1	120.3	C9—C8—H8B	110.9
C3—C4—C5	119.3 (2)	C7—C8—H8B	110.9
C3—C4—H4	120.3	H8A—C8—H8B	109.0
C5—C4—H4	120.3	N2—C9—C8	103.6 (2)
N2—C6—C7	104.0 (2)	N2—C9—H9A	111.0
N2—C6—H6A	111.0	C8—C9—H9A	111.0
C7—C6—H6A	111.0	N2—C9—H9B	111.0
N2—C6—H6B	111.0	C8—C9—H9B	111.0
C7—C6—H6B	111.0	H9A—C9—H9B	109.0
H6A—C6—H6B	109.0		

C6—N2—C5—C1	−170.4 (3)	O1—N3—C12—C13	−175.5 (3)
C9—N2—C5—C1	−4.8 (4)	O2—N3—C12—C13	3.5 (4)
C6—N2—C5—C4	11.0 (4)	C10—C15—C14—C13	1.3 (4)
C9—N2—C5—C4	176.6 (2)	C10—C15—C14—N4	179.8 (2)
C11—C10—C15—C14	−0.4 (4)	O4—N4—C14—C13	174.3 (2)
I1—C10—C15—C14	−179.76 (18)	O3—N4—C14—C13	−5.0 (4)
C2—N1—C3—C4	−0.1 (4)	O4—N4—C14—C15	−4.2 (4)
N2—C5—C1—C2	−178.6 (2)	O3—N4—C14—C15	176.4 (2)
C4—C5—C1—C2	0.1 (4)	C3—N1—C2—C1	−0.5 (4)
N1—C3—C4—C5	0.7 (4)	C5—C1—C2—N1	0.5 (4)
N2—C5—C4—C3	178.1 (3)	C15—C14—C13—C12	−0.9 (4)
C1—C5—C4—C3	−0.6 (4)	N4—C14—C13—C12	−179.3 (2)
C5—N2—C6—C7	161.7 (3)	C11—C12—C13—C14	−0.5 (4)
C9—N2—C6—C7	−5.2 (3)	N3—C12—C13—C14	178.4 (2)
C15—C10—C11—C12	−0.9 (4)	N2—C6—C7—C8	24.1 (3)

I1—C10—C11—C12	178.47 (19)	C6—C7—C8—C9	−33.9 (3)
C10—C11—C12—C13	1.4 (4)	C5—N2—C9—C8	177.2 (2)
C10—C11—C12—N3	−177.5 (2)	C6—N2—C9—C8	−15.7 (3)
O1—N3—C12—C11	3.4 (4)	C7—C8—C9—N2	30.1 (3)
O2—N3—C12—C11	−177.6 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O1^{i}	0.95	2.61	3.499 (3)	156
C7—H7*B*···I1^{ii}	0.99	3.22	4.104 (3)	150
C8—H8*A*···O2^{iii}	0.99	2.55	3.301 (4)	133
C8—H8*A*···O4^{iv}	0.99	2.61	3.420 (4)	140
C9—H9*A*···O4^{v}	0.99	2.61	3.186 (3)	117

Symmetry codes: (i) *x*, *y*−1, *z*; (ii) −*x*, −*y*, −*z*+1; (iii) −*x*+1, −*y*+1, −*z*+1; (iv) −*x*+1, −*y*, −*z*+1; (v) *x*−1, *y*, *z*−1.

4-(Pyrrolidin-1-yl)pyridine–1-iodo-3,5-bis(trifluoromethyl)benzene (1/1) (BTFIB4PYPY)

Crystal data

$C_8H_3F_6I·C_9H_{12}N_2$
M_r = 488.21
Triclinic, $P\overline{1}$
a = 7.9777 (4) Å
b = 8.5138 (4) Å
c = 14.1679 (7) Å
α = 101.579 (1)°
β = 103.264 (1)°
γ = 100.370 (1)°
V = 891.40 (8) Å^3
Z = 2
F(000) = 476
D_x = 1.819 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 7472 reflections
θ = 2.5–27.2°
μ = 1.86 mm^{-1}
T = 100 K
Cut, colourless
0.41 × 0.20 × 0.11 mm

Data collection

Bruker APEXI CCD diffractometer
Detector resolution: 8.3660 pixels mm^{-1}
φ and *ω* scans
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
T_{min} = 0.669, T_{max} = 0.746
11576 measured reflections
3979 independent reflections
3517 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
θ_{max} = 27.2°, θ_{min} = 1.5°
h = −10→10
k = −10→10
l = −18→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.110
S = 1.03
3979 reflections
235 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0623P)^2 + 1.7968P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 2.23 e Å^{-3}
$\Delta\rho_{min}$ = −1.21 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	0.25075 (3)	0.40397 (3)	0.25028 (2)	0.04031 (12)
F3	0.1815 (5)	0.9266 (4)	−0.0400 (2)	0.0699 (10)
F2	0.4058 (5)	0.9949 (4)	0.0867 (4)	0.0981 (16)
F6	0.3083 (8)	0.2154 (6)	−0.1345 (3)	0.1099 (19)
F5	0.0807 (4)	0.2808 (6)	−0.2053 (3)	0.1068 (19)
F1	0.1466 (8)	0.9537 (6)	0.1061 (3)	0.1139 (19)
F4	0.3331 (6)	0.4094 (4)	−0.1980 (3)	0.0930 (15)
C14	0.2424 (5)	0.7210 (5)	0.0425 (3)	0.0371 (9)
C11	0.2431 (5)	0.3922 (5)	0.0328 (3)	0.0348 (8)
H11	0.242389	0.279409	0.029139	0.042*
C12	0.2414 (5)	0.4538 (5)	−0.0508 (3)	0.0311 (8)
C15	0.2465 (5)	0.6632 (5)	0.1279 (3)	0.0348 (9)
H15	0.249785	0.735595	0.189071	0.042*
C10	0.2459 (5)	0.4985 (5)	0.1227 (3)	0.0347 (8)
C16	0.2362 (5)	0.3388 (5)	−0.1481 (3)	0.0318 (8)
C13	0.2399 (5)	0.6171 (5)	−0.0480 (3)	0.0343 (8)
H13	0.237243	0.657398	−0.106043	0.041*
C17	0.2452 (7)	0.8988 (6)	0.0480 (4)	0.0541 (13)
N2	0.2661 (4)	−0.1161 (4)	0.5764 (3)	0.0356 (7)
N1	0.2667 (5)	0.2330 (5)	0.4051 (3)	0.0431 (9)
C4	0.3814 (5)	0.1550 (5)	0.5581 (3)	0.0351 (8)
H4	0.461763	0.186897	0.623266	0.042*
C5	0.2673 (5)	−0.0044 (5)	0.5219 (3)	0.0326 (8)
C9	0.1582 (5)	−0.2865 (5)	0.5391 (3)	0.0414 (10)
H9A	0.177820	−0.341771	0.475465	0.050*
H9B	0.030462	−0.289137	0.528127	0.050*
C6	0.3750 (5)	−0.0824 (5)	0.6811 (3)	0.0335 (8)
H6A	0.368445	0.024343	0.721668	0.040*
H6B	0.500531	−0.080878	0.684121	0.040*
C2	0.1594 (6)	0.0792 (6)	0.3706 (3)	0.0412 (10)
H2	0.081913	0.051437	0.304785	0.049*
C7	0.2906 (6)	−0.2267 (5)	0.7170 (3)	0.0408 (9)
H7A	0.191991	−0.201739	0.744282	0.049*
H7B	0.379140	−0.252966	0.769246	0.049*
C1	0.1534 (5)	−0.0393 (6)	0.4230 (3)	0.0390 (9)
H1	0.073671	−0.144412	0.393517	0.047*
C3	0.3751 (5)	0.2644 (6)	0.4979 (3)	0.0384 (9)
H3	0.453833	0.370463	0.524397	0.046*
C8	0.2226 (6)	−0.3690 (6)	0.6224 (4)	0.0461 (10)

H8A	0.318785	−0.422350	0.610027	0.055*
H8B	0.124415	−0.453024	0.627894	0.055*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.03090 (16)	0.05218 (19)	0.02766 (16)	−0.00391 (11)	0.00906 (11)	−0.00150 (11)
F3	0.095 (3)	0.0434 (16)	0.0525 (18)	0.0249 (16)	−0.0135 (17)	0.0018 (13)
F2	0.079 (2)	0.0322 (15)	0.132 (4)	0.0066 (15)	−0.045 (2)	−0.0038 (18)
F6	0.204 (5)	0.118 (3)	0.0405 (18)	0.122 (4)	0.039 (2)	0.0124 (19)
F5	0.0302 (15)	0.164 (4)	0.067 (2)	−0.0023 (19)	0.0075 (15)	−0.075 (3)
F1	0.192 (5)	0.102 (3)	0.087 (3)	0.112 (4)	0.059 (3)	0.019 (2)
F4	0.126 (3)	0.068 (2)	0.081 (3)	−0.018 (2)	0.081 (3)	−0.0143 (18)
C14	0.0242 (18)	0.040 (2)	0.037 (2)	0.0097 (16)	−0.0009 (15)	−0.0041 (17)
C11	0.0293 (19)	0.0337 (19)	0.034 (2)	−0.0036 (15)	0.0115 (16)	−0.0022 (16)
C12	0.0241 (17)	0.0339 (19)	0.0293 (18)	0.0017 (14)	0.0082 (14)	−0.0017 (15)
C15	0.0208 (17)	0.042 (2)	0.0309 (19)	0.0047 (15)	0.0046 (14)	−0.0097 (16)
C10	0.0225 (17)	0.046 (2)	0.0267 (18)	−0.0022 (15)	0.0087 (14)	−0.0025 (16)
C16	0.0313 (19)	0.0316 (19)	0.0319 (19)	0.0047 (15)	0.0129 (15)	0.0045 (15)
C13	0.0252 (18)	0.039 (2)	0.0322 (19)	0.0077 (15)	0.0016 (15)	0.0008 (16)
C17	0.052 (3)	0.049 (3)	0.045 (3)	0.025 (2)	−0.011 (2)	−0.010 (2)
N2	0.0256 (15)	0.0365 (17)	0.0321 (17)	−0.0010 (13)	0.0048 (13)	−0.0084 (14)
N1	0.0373 (19)	0.057 (2)	0.0322 (18)	0.0114 (17)	0.0122 (15)	0.0012 (16)
C4	0.0291 (18)	0.044 (2)	0.0239 (18)	0.0019 (16)	0.0063 (15)	−0.0040 (16)
C5	0.0246 (17)	0.041 (2)	0.0257 (18)	0.0056 (15)	0.0079 (14)	−0.0058 (15)
C9	0.029 (2)	0.038 (2)	0.043 (2)	−0.0012 (16)	0.0085 (17)	−0.0123 (18)
C6	0.0290 (18)	0.036 (2)	0.0283 (19)	0.0026 (15)	0.0090 (15)	−0.0044 (15)
C2	0.031 (2)	0.056 (3)	0.0280 (19)	0.0111 (19)	0.0056 (16)	−0.0055 (18)
C7	0.037 (2)	0.040 (2)	0.038 (2)	0.0008 (17)	0.0137 (18)	−0.0025 (18)
C1	0.0275 (19)	0.047 (2)	0.031 (2)	0.0051 (17)	0.0041 (16)	−0.0080 (17)
C3	0.033 (2)	0.046 (2)	0.030 (2)	0.0012 (17)	0.0130 (16)	−0.0024 (17)
C8	0.037 (2)	0.039 (2)	0.048 (3)	−0.0032 (18)	0.0093 (19)	−0.0060 (19)

Geometric parameters (Å, º)

I1—C10	2.116 (4)	N1—C3	1.343 (6)
F3—C17	1.319 (6)	N1—C2	1.354 (6)
F2—C17	1.316 (6)	C4—C3	1.383 (6)
F6—C16	1.311 (5)	C4—C5	1.413 (6)
F5—C16	1.264 (5)	C4—H4	0.9500
F1—C17	1.338 (7)	C5—C1	1.424 (5)
F4—C16	1.309 (5)	C9—C8	1.528 (7)
C14—C15	1.391 (6)	C9—H9A	0.9900
C14—C13	1.397 (6)	C9—H9B	0.9900
C14—C17	1.496 (6)	C6—C7	1.527 (6)
C11—C12	1.388 (6)	C6—H6A	0.9900
C11—C10	1.400 (5)	C6—H6B	0.9900
C11—H11	0.9500	C2—C1	1.367 (7)

C12—C13	1.385 (6)	C2—H2	0.9500
C12—C16	1.508 (5)	C7—C8	1.529 (6)
C15—C10	1.388 (6)	C7—H7A	0.9900
C15—H15	0.9500	C7—H7B	0.9900
C13—H13	0.9500	C1—H1	0.9500
N2—C5	1.340 (6)	C3—H3	0.9500
N2—C9	1.470 (5)	C8—H8A	0.9900
N2—C6	1.478 (5)	C8—H8B	0.9900
C15—C14—C13	121.3 (4)	N2—C5—C4	122.5 (4)
C15—C14—C17	119.5 (4)	N2—C5—C1	122.0 (4)
C13—C14—C17	119.2 (4)	C4—C5—C1	115.5 (4)
C12—C11—C10	119.1 (4)	N2—C9—C8	104.0 (3)
C12—C11—H11	120.5	N2—C9—H9A	111.0
C10—C11—H11	120.5	C8—C9—H9A	111.0
C13—C12—C11	121.7 (4)	N2—C9—H9B	111.0
C13—C12—C16	119.2 (4)	C8—C9—H9B	111.0
C11—C12—C16	119.1 (3)	H9A—C9—H9B	109.0
C10—C15—C14	119.3 (3)	N2—C6—C7	103.0 (3)
C10—C15—H15	120.3	N2—C6—H6A	111.2
C14—C15—H15	120.3	C7—C6—H6A	111.2
C15—C10—C11	120.3 (4)	N2—C6—H6B	111.2
C15—C10—I1	120.8 (3)	C7—C6—H6B	111.2
C11—C10—I1	118.8 (3)	H6A—C6—H6B	109.1
F5—C16—F4	107.9 (4)	N1—C2—C1	125.3 (4)
F5—C16—F6	108.4 (5)	N1—C2—H2	117.3
F4—C16—F6	101.9 (4)	C1—C2—H2	117.3
F5—C16—C12	112.6 (3)	C6—C7—C8	103.7 (4)
F4—C16—C12	112.6 (3)	C6—C7—H7A	111.0
F6—C16—C12	112.7 (3)	C8—C7—H7A	111.0
C12—C13—C14	118.3 (4)	C6—C7—H7B	111.0
C12—C13—H13	120.9	C8—C7—H7B	111.0
C14—C13—H13	120.9	H7A—C7—H7B	109.0
F2—C17—F3	107.1 (5)	C2—C1—C5	119.6 (4)
F2—C17—F1	106.4 (5)	C2—C1—H1	120.2
F3—C17—F1	106.1 (4)	C5—C1—H1	120.2
F2—C17—C14	112.5 (4)	N1—C3—C4	125.3 (4)
F3—C17—C14	113.2 (4)	N1—C3—H3	117.3
F1—C17—C14	111.1 (5)	C4—C3—H3	117.3
C5—N2—C9	124.1 (3)	C9—C8—C7	103.9 (4)
C5—N2—C6	123.8 (3)	C9—C8—H8A	111.0
C9—N2—C6	112.1 (3)	C7—C8—H8A	111.0
C3—N1—C2	114.7 (4)	C9—C8—H8B	111.0
C3—C4—C5	119.4 (4)	C7—C8—H8B	111.0
C3—C4—H4	120.3	H8A—C8—H8B	109.0
C5—C4—H4	120.3		
C10—C11—C12—C13	−0.9 (6)	C15—C14—C17—F1	−38.9 (6)

C10—C11—C12—C16	−179.3 (3)	C13—C14—C17—F1	142.6 (4)
C13—C14—C15—C10	−0.9 (6)	C9—N2—C5—C4	−175.8 (4)
C17—C14—C15—C10	−179.3 (4)	C6—N2—C5—C4	3.6 (6)
C14—C15—C10—C11	0.7 (6)	C9—N2—C5—C1	4.0 (6)
C14—C15—C10—I1	−179.7 (3)	C6—N2—C5—C1	−176.6 (3)
C12—C11—C10—C15	0.1 (6)	C3—C4—C5—N2	−179.7 (4)
C12—C11—C10—I1	−179.5 (3)	C3—C4—C5—C1	0.5 (5)
C13—C12—C16—F5	−83.0 (5)	C5—N2—C9—C8	172.2 (4)
C11—C12—C16—F5	95.5 (5)	C6—N2—C9—C8	−7.3 (4)
C13—C12—C16—F4	39.3 (5)	C5—N2—C6—C7	165.2 (4)
C11—C12—C16—F4	−142.2 (4)	C9—N2—C6—C7	−15.4 (4)
C13—C12—C16—F6	153.9 (4)	C3—N1—C2—C1	1.0 (6)
C11—C12—C16—F6	−27.6 (6)	N2—C6—C7—C8	31.7 (4)
C11—C12—C13—C14	0.8 (6)	N1—C2—C1—C5	−0.3 (6)
C16—C12—C13—C14	179.2 (3)	N2—C5—C1—C2	179.7 (4)
C15—C14—C13—C12	0.1 (6)	C4—C5—C1—C2	−0.5 (5)
C17—C14—C13—C12	178.6 (4)	C2—N1—C3—C4	−0.9 (6)
C15—C14—C17—F2	80.2 (6)	C5—C4—C3—N1	0.2 (6)
C13—C14—C17—F2	−98.3 (5)	N2—C9—C8—C7	27.0 (4)
C15—C14—C17—F3	−158.2 (4)	C6—C7—C8—C9	−36.7 (4)
C13—C14—C17—F3	23.3 (6)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6*A*···F6^{i}	0.99	2.54	3.465 (5)	156
C1—H1···F5^{ii}	0.95	2.59	3.265 (5)	128

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