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Crystal structures of 2,4,6-triiodobenzonitrile and 2,4,6-triiodophenyl isocyanide

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The title crystals, $C_7H_2I_3N$, are isomorphous. Both molecules lie across two crystallographic mirror planes and a twofold axis. The principal supramolecular interaction is centric $R_2^2(10)$ CN/NC$\cdots$I short contacts involving both *ortho* I atoms, with two contacts bisecting each cyano and isocyano group. These form ribbons along [010] and give rise to a planar sheet structure parallel to (100). All pairs of adjacent sheets have centric stacking, a mode not previously reported for sheets of this type. This study completes the series of *homo*-2,4,6-trihalobenzonitriles, in which I atoms give the strongest CN$\cdots$*X* and NC$\cdots$*X* interactions (*X* = F, Cl, Br, I).

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CCDC references: 1580006; 1581218

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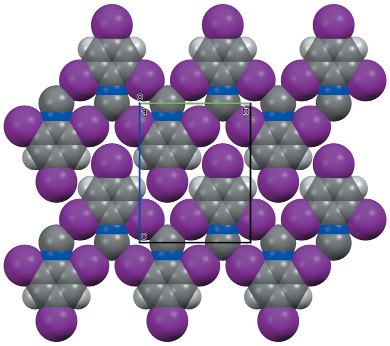

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1. Chemical context

The strength of cyano–halo interactions tends to increase with increasing polarizability, or the elemental period, of the halogen. Structure-directing CN$\cdots$F interactions are usually not observed (Bond *et al.*, 2001). In crystals of the other 4-halobenzonitriles (*X* = Cl, Br, I), parallel or antiparallel $C_1^1(7)$ CN$\cdots$*X* chains dominate the secondary structures (Fig. 1; Desiraju & Harlow, 1989). When the halo atom is moved to the 2-position, $R_2^2(10)$ CN$\cdots$*X* rings can form, usually as inversion dimers. Halogenation at both *ortho* positions allows the formation of CN$\cdots$*X*-derived ribbons or sheets. The aforementioned periodic trend is exhibited by the *homo*-2,4,6-trihalobenzonitriles. No CN$\cdots$F contacts are observed in 2,4,6-trifluorobenzonitrile (F3CN). Instead, each CN group is bisected by two CN$\cdots$H contacts (Fig. 2*a*; Britton, 2008). In 2,4,6-trichlorobenzonitrile (Cl3CN), half of these have been replaced by CN$\cdots$Cl contacts (Fig. 2*b*; Pink *et al.*, 2000). In 2,4,6-tribromobenzonitrile (Br3CN), no CN$\cdots$H contacts are found, and each CN group is bisected by two CN$\cdots$Br contacts (Fig. 2*c*; Britton *et al.*, 2016).

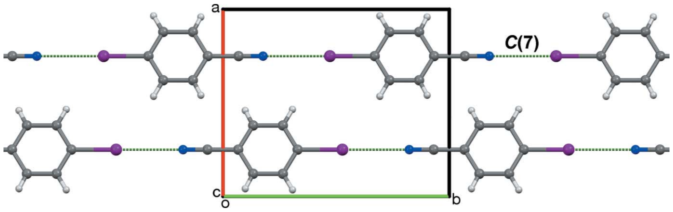
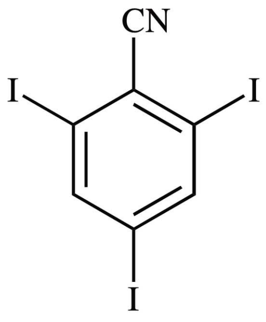
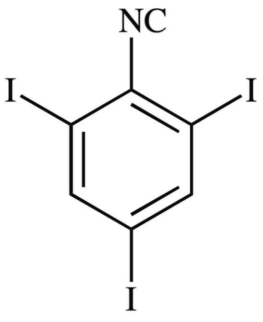


Figure 1
Several molecules in the crystal of 4-iodobenzonitrile (4ICN), viewed along [001]. Dashed green lines represent CN$\cdots$I short contacts, which collectively form a *C*(7) chain motif along [010]. All previously reported 4-iodobenzonitriles form similar chains.



I3CN



I3NC

2. Database survey

No entries were found in the most recent update of the Cambridge Structural Database (Version 5.37, May 2017; Groom *et al.*, 2016) that have I atoms at both *ortho* positions of a benzonitrile. Four of the five crystalline 2-iodobenzonitriles have CN···I contacts (Britton, 2001, 2004; Ketels *et al.*, 2017; Lam & Britton, 1974); the fifth is a cyano alcohol that forms O—H···NC hydrogen bonds (Salvati *et al.*, 2008). The 3-iodo analogs do not pack as efficiently. Three of the four examples feature I···I contacts (Britton, 2006; Merz, 2006); packing in the fourth example is directed by hydrogen bonding between acetamido groups (Garden *et al.*, 2007). All five reported 4-iodobenzonitriles form $C(7)$ CN···I chains (Fig. 1; Bond *et al.*, 2001; Britton, 2004; Desiraju & Harlow, 1989; Gleason & Britton, 1978). It is pertinent to determine the crystal structure of 2,4,6-triiodobenzonitrile (I3CN) to complete the series of *homo*-2,4,6-trihalobenzonitriles, and to determine whether the primary packing interaction is CN···I-derived $C(7)$ chains, $R_2^2(10)$ rings, or another motif. 2,4,6-Triiodophenyl isocyanide (I3NC) is included to contribute to the library of corresponding halogenated nitrile-isocyanide crystal pairs.

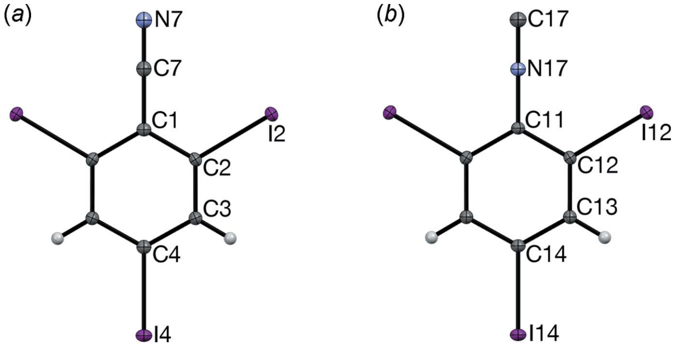


Figure 3
The molecular structures of (*a*) I3CN and (*b*) I3NC, with atom labeling and displacement ellipsoids at the 50% probability level. Unlabeled atoms are generated by the symmetry operation $(1 - x, \frac{3}{2} - y, z)$.

3. Structural commentary

Molecules of I3CN and I3NC (Fig. 3) lie about a twofold axis and two orthogonal vertical mirror planes. Thus, they have crystallographically-imposed C_{2v} symmetry and are planar, with the *para* I atom (I4; I14) collinear with the CN and NC groups. All of the aryl bond angles are roughly 120°. The *ortho* I atoms (I2, I2′; I12, I12′) are scissored slightly toward the *ipso* C atom (C1; C11), which is probably caused by the intermolecular CN···I and NC···I short contacts. The bond lengths are typical for their respective functional groups.

4. Supramolecular features

Crystals of I3CN and I3NC are isomorphous. The CN and NC groups are bisected by C7≡N7···I2 and N17≡C17···I12 contacts (Table 1), forming ribbons of $R_2^2(10)$ rings parallel to (100) along [010]. Adjacent ribbons translate along [001]. The resulting planar sheet structure (Fig. 4) matches that observed in Br3CN and the corresponding isocyanide (Br3NC) (Britton *et al.*, 2016), and the 4-chloro (Britton, 2005) and 4-nitro

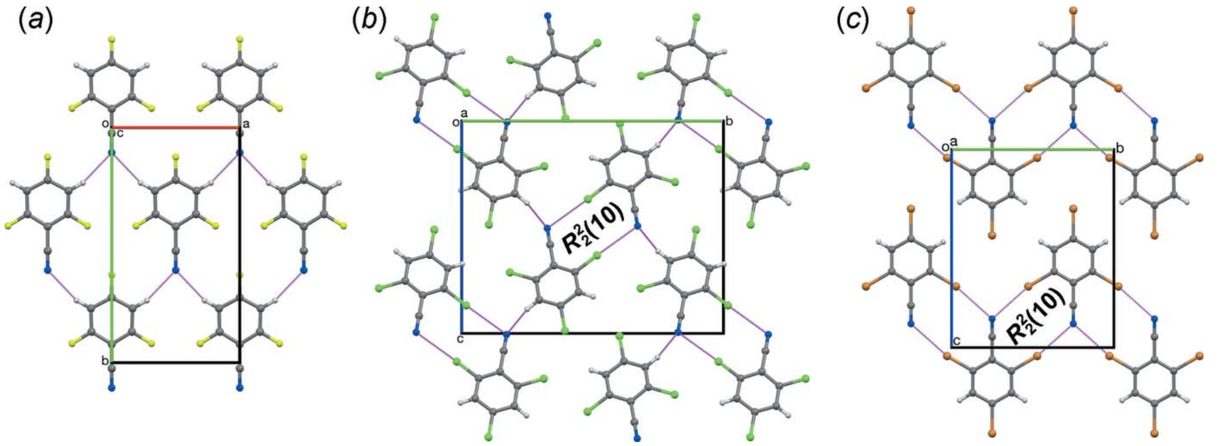


Figure 2
(*a*) A sheet in a crystal of F3CN, showing two CN···H contacts per CN group, viewed along [001]; (*b*) A sheet in a crystal of Cl3CN, showing one CN···H and one CN···Cl contact per CN group, viewed along [100]; (*c*) A sheet in the $Z = 8$ polytype of Br3CN, showing two CN···Br contacts per CN group, viewed along [100]. Dashed magenta lines represent short contacts.

Table 1
Contact geometry (Å, °) for I3CN and I3NC.

$A \equiv B \cdots$I	$A \equiv B$	$B \cdots$I	$A \equiv B \cdots$I
C7$\equiv$N7$\cdots$I2$^{\rm i}$	1.151 (3)	3.074 (2)	132.85 (3)
N17$\equiv$C17$\cdots$I12$^{\rm i}$	1.164 (3)	3.106 (2)	134.18 (3)

Symmetry code: (i) $-x + 1, y - \frac{1}{2}, -z + 2$

Table 2
Mean CN$\cdots X$ contact lengths (Å) in 4-halobenzonitriles (4*X*CN) and 2,4,6-trihalobenzonitriles (*X*3CN).

X	4*X*CN	*X*3CN	*r* [N + *X*] (Å)	[*r*/*X*3CN]
Cl	3.370 (4)	3.153 (2)	3.35	1.06
Br	3.249 (5)	3.064 (4)	3.46	1.13
I	3.127 (4)	3.074 (2)	3.61	1.17

Table 3
Mean NC$\cdots X$ contact lengths (Å) in 2,4,6-trihalophenyl isocyanides (*X*3NC).

X	*X*3NC	*r* [C + *X*]	[*r*/*X*3NC]
Cl	3.245 (3)	3.49	1.08
Br	3.151 (4)	3.60	1.14
I	3.106 (2)	3.75	1.21

(Noland & Tritch, 2017) analogs of Br3CN. In crystals of I3CN and I3NC, all pairs of adjacent sheets have centric stacking along [100] (Fig. 5), with molecules stacked about a glide plane and an inversion center. In the polytypes of Br3CN and Br3NC, adjacent sheets had combinations of centric and translational stacking, but not solely centric stacking. The 4-chloro analog had translational stacking. The 4-nitro analog had glide stacking, with no inversion center between stacked molecules. Thus, the all-centric stacking of I3CN and I3NC can be regarded as a new polytype in this series.

The mean CN$\cdots X$ contact lengths can be compared for X = Cl, Br, and I (Table 2). For 4-chlorobenzonitrile (4ClCN), 4-bromobenzonitrile (4BrCN), and 4-iodobenzonitrile (4ICN) (Table 2, col. 2), the contact distance decreases with increasing halogen size, highlighting the increase in contact strength (Desiraju & Harlow, 1989). This trend is essentially mirrored among 2,4,6-trihalobenzonitriles (Table 2, col. 3), although the contact distance in I3CN is 0.01 Å larger than in Br3CN. The N$\cdots X$ non-bonded contact radii are listed (Table 2, col. 4; Rowland & Taylor, 1996). The 'shortness' of contacts in 2,4,6-trihalobenzonitriles is expressed as the ratios of contact radii to the respective contact distances (Table 2, col. 5). A similar comparison of NC$\cdots X$ contact lengths in the corresponding trihalo isocyanides also shows decreasing contact length with increasing halogen size (Table 3, col. 2). The NC$\cdots X$ contacts have slightly greater shortness (Table 3, col. 4) than the corresponding CN$\cdots X$ contacts. The N17$\equiv$C17$\cdots$I12 contacts in I3NC are the strongest cyano/isocyano–halo interactions in this series.

5. Synthesis and crystallization

2,4,6-Triiodoaniline (I3NH2), adapted from the work of Jackson & Whitmore (1915): Aniline (1.0 mL) and hydrochloric acid (0.7 *M*, 850 mL) were combined and stirred in a round-bottomed flask. Iodine monochloride (8.2 g) was placed in a separate flask and then warmed to 315 K. The two flasks were connected with a glass bridge. A slow stream of nitrogen was passed through the headspace in the second vessel so that the iodine monochloride was gradually swept into the first

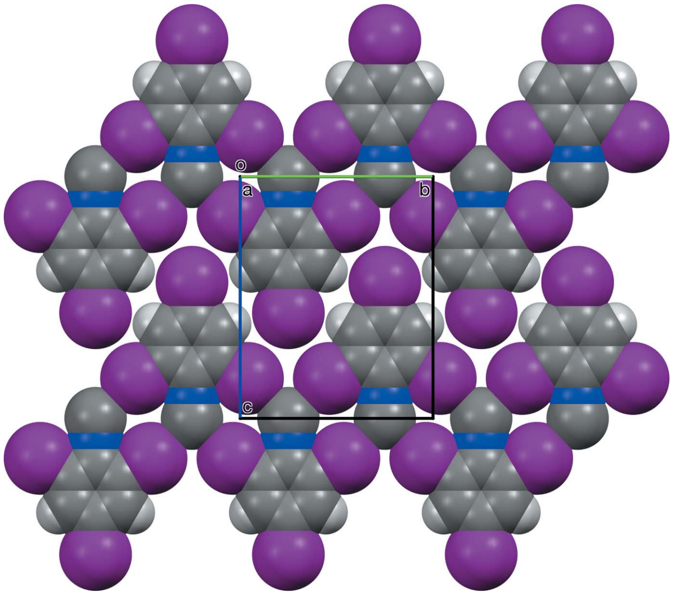


Figure 4
A space-filling drawing of the sheet structure of I3NC, viewed along [100].

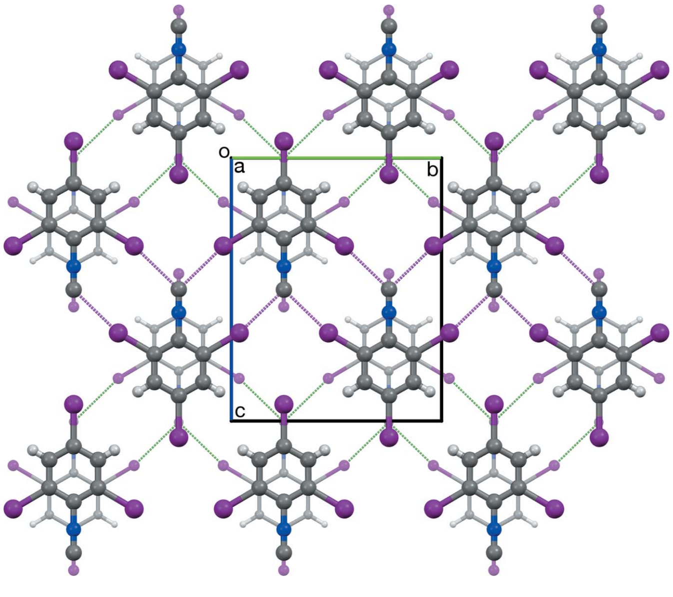


Figure 5
Two adjacent sheets in I3NC, viewed along [100], illustrating the centric stacking mode. Dashed magenta lines represent short contacts in the front layer. Molecules in the rear layer are drawn with smaller balls and sticks, lower opacity, and green dashed lines representing short contacts.

Table 4
Experimental details.

	I3CN	I3NC
Crystal data		
Chemical formula	$C_7H_2I_3N$	$C_7H_2I_3N$
M_r	480.80	480.80
Crystal system, space group	Orthorhombic, *Imma*	Orthorhombic, *Imma*
Temperature (K)	100	100
a, *b*, *c* (Å)	7.0593 (4), 10.5346 (5), 13.0658 (6)	7.0552 (3), 10.4947 (5), 13.1557 (5)
V (Å^3)	971.66 (8)	974.08 (7)
Z	4	4
Radiation type	Mo *Kα*	Mo *Kα*
μ (mm^{-1})	9.59	9.56
Crystal size (mm)	0.12 × 0.10 × 0.10	0.15 × 0.09 × 0.07
Data collection		
Diffractometer	Bruker VENTURE PHOTON-II	Bruker VENTURE PHOTON-II
Absorption correction	Multi-scan (*SADABS*; Sheldrick, 1996)	Multi-scan (*SADABS*; Sheldrick, 1996)
T_{min}, T_{max}	0.281, 0.344	0.251, 0.344
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	8436, 1303, 1261	13040, 1314, 1267
R_{int}	0.024	0.026
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.835	0.834
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.013, 0.030, 1.18	0.011, 0.024, 1.14
No. of reflections	1303	1314
No. of parameters	40	40
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.89, −0.60	0.72, −0.48

Computer programs: *APEX3* (Bruker, 2012), *SAINT* (Bruker, 2012), *SHELXT2014* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*), *Mercury* (Macrae *et al.*, 2008), *publCIF* (Westrip, 2010).

vessel over 2–4 d. After the transfer was complete, the reaction mixture was neutralized with $NaHCO_3$ solution, followed by reduction of excess iodine by washing with $Na_2S_2O_3$ solution. Dichloromethane (approx. 100 mL) was added, with stirring, until nearly all solids had dissolved. The organic portion was filtered through silica gel (3 cm H × 4 cm D), and then the filter was washed with dichloromethane (3 × 20 mL). The filtrate was placed in a loosely-covered beaker. After most of the dichloromethane had evaporated, beige needles were collected by suction filtration (4.48 g, 89%). M.p. 459–460 K (lit. 459); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.864 (*s*, 2H), 4.658 (*s*, 2H); ^{13}C NMR (75 MHz, $(CD_3)_2SO$) δ 147.0 (1C), 145.4 (2C), 83.0 (2C), 78.8 (1C); IR (KBr, cm^{-1}) 3417, 3056, 2987, 1632, 1422, 1265, 741, 704; MS (EI, *m*/*z*) $[M]^+$ calculated for $C_6H_4I_3N$ 470.7472, found 470.7470.

2,4,6-Triiodobenzonitrile (I3CN), was prepared from I3NH2 (101 mg; Fig. 6) based on the Sandmeyer procedure described by Britton *et al.* (2016). Ethyl acetate (20 mL), toluene-4-sulfonic acid monohydrate (77 mg), and isoamyl nitrite (60 µL) were used in place of water, acetic and sulfuric acids, and sodium nitrite, respectively. The desired chromatographic fraction ($R_f = 0.44$ in 4:1 hexane–ethyl acetate) was concentrated on a rotary evaporator, giving a beige powder (67 mg, 65%). M.p. 517–518 K; ^{1}H NMR (500 MHz, $(CD_3)_2SO$) δ 8.431 (*s*, 2H, H3); ^{13}C NMR (126 MHz, CD_2Cl_2) δ 147.5 (2C, C3), 127.3 (1C, C1), 120.7 (1C, C7), 101.1 (1C, C4), 99.6 (2C, C2); IR (NaCl, cm^{-1}) 3070, 2227, 1532, 1359, 1206, 1081, 861, 787, 706; MS (ESI, *m*/*z*) $[M + Na]^+$ calculated for $C_7H_2I_3N$ 503.7213, found 503.7216.

2,4,6-Triiodoformanilide (I3FA) was prepared from I3NH2 (1.01 g) according to the formylation procedure described by Britton *et al.* (2016), with 1,2-dichloroethane (10 mL and

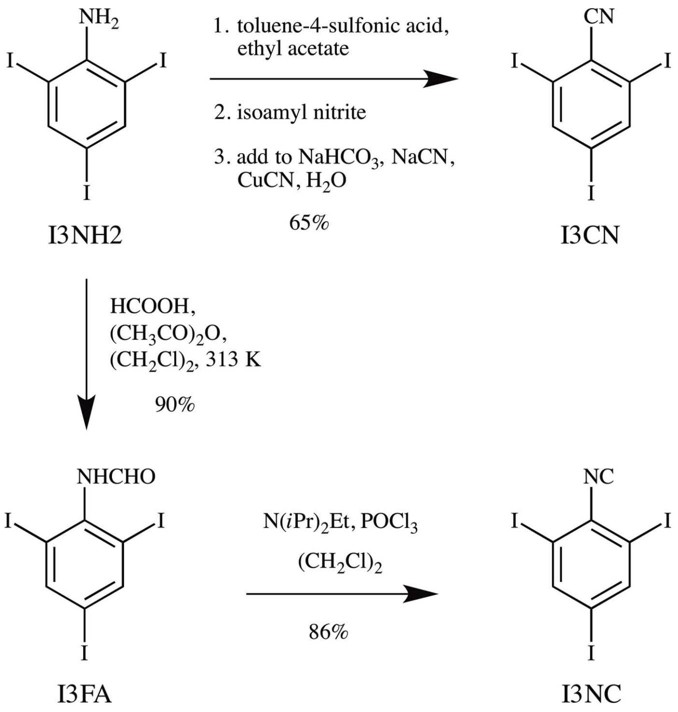


Figure 6
The synthesis of I3CN and I3NC.

100 mL) in place of tetrahydrofuran, giving white needles (962 mg, 90%). M.p. 557–558 K; ^{1}H NMR (300 MHz, $(CD_3)_2SO$; 2 conformers observed) δ 10.089 (*s*, 1H; major), 9.655 (*s*, 1H; minor), 8.303 (*s*, 1H; major), 8.278 (*s*, 2H; minor), 8.233 (*s*, 2H; major), 7.978 (*s*, 1H; minor); ^{13}C NMR (126 MHz, $(CD_3)_2SO$; 2 conformers observed) δ 164.4 (1C; minor), 159.4 (1C; major), 146.4 (2C; minor), 146.1 (2C; major), 141.0 (1C; major), 140.4 (1C; minor), 102.4 (2C; minor), 101.9 (2C; major), 95.9 (1C; minor), 95.6 (1C; major); IR (NaCl, cm^{-1}) 3221, 3076, 2919, 1637, 1490, 1380, 1232, 1143, 857, 794, 703, 682; MS (ESI, *m*/*z*) [M - H]$^-$ calculated for $C_7H_4I_3NO$ 497.7354, found 497.7365.

2,4,6-Triiodophenyl isocyanide (I3NC) was prepared from I3FA (397 mg) according to the dehydration procedure described by Britton *et al.* (2016), giving a white powder (330 mg, 86%). M.p. 467–468 K; ^{1}H NMR (300 MHz, $CDCl_3$) 8.198 (*s*, 2H, H13); ^{13}C NMR (126 MHz, $(CD_3)_2SO$) δ 170.0 (1C, C17), 146.2 (2C, C13), 133.8 (1C, C11), 98.8 (1C, C14), 97.7 (2C, C12); IR (KBr, cm^{-1}) 3073, 3037, 2920, 2126, 1529, 1079, 861, 704; MS (ESI, *m*/*z*) [*M* - H]$^-$ calculated for $C_7H_2I_3N$ 479.7249, found 479.7226.

Crystallization: Crystals of I3CN and I3NC were prepared by slow evaporation of acetonitrile solutions at ambient temperature, followed by decantation and then washing with pentane.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 4. A direct-methods solution was calculated, followed by full-matrix least squares/difference-Fourier cycles. All H atoms were placed in calculated positions (C—H = 0.95 Å) and refined as riding atoms with $U_{\rm iso}$(H) set to 1.2$U_{\rm eq}$(C).

Acknowledgements

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supporting information

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Crystal structures of 2,4,6-triiodobenzonitrile and 2,4,6-triiodophenyl isocyanide

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Computing details

For both structures, data collection: *APEX3* (Bruker, 2012); cell refinement: *SAINT* (Bruker, 2012); data reduction: *SAINT* (Bruker, 2012); program(s) used to solve structure: SHELXT2014 (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015b); molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *publCIF* (Westrip, 2010).

2,4,6-Triiodobenzonitrile (I3CN)

Crystal data

$C_7H_2I_3N$
$M_r = 480.80$
Orthorhombic, *Imma*
$a = 7.0593\ (4)$ Å
$b = 10.5346\ (5)$ Å
$c = 13.0658\ (6)$ Å
$V = 971.66\ (8)$ Å^3
$Z = 4$
$F(000) = 840$
$D_x = 3.287$ Mg m^{-3}
Melting point: 517 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2721 reflections
$\theta = 2.5$–$36.4°$
$\mu = 9.59$ mm^{-1}
$T = 100$ K
Square bipyramid, colorless
0.12 × 0.10 × 0.10 mm

Data collection

Bruker VENTURE PHOTON-II diffractometer
Radiation source: micro-focus
φ and ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
$T_{min} = 0.281$, $T_{max} = 0.344$
8436 measured reflections
1303 independent reflections
1261 reflections with $I > 2\sigma(I)$
$R_{int} = 0.024$
$\theta_{max} = 36.4°$, $\theta_{min} = 2.5°$
$h = -9 \rightarrow 11$
$k = -17 \rightarrow 17$
$l = -21 \rightarrow 21$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.013$
$wR(F^2) = 0.030$
$S = 1.18$
1303 reflections
40 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0078P)^2 + 1.2371P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.89$ e Å^{-3}
$\Delta\rho_{min} = -0.60$ e Å^{-3}

Extinction correction: SHELXL2014 (Sheldrick, 2015b), $Fc^{*}=kFc[1+0.001xFc^{2}\lambda^{3}/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.00199 (9)

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*	$U_{\rm iso}$*/$U_{\rm eq}$
I2	0.5000	0.46395 (2)	0.83491 (2)	0.01267 (4)
I4	0.5000	0.7500	0.43429 (2)	0.01359 (4)
N7	0.5000	0.7500	1.00506 (18)	0.0167 (4)
C1	0.5000	0.7500	0.80692 (18)	0.0103 (4)
C2	0.5000	0.63456 (15)	0.75302 (13)	0.0105 (3)
C3	0.5000	0.63428 (16)	0.64671 (13)	0.0117 (3)
H3	0.5000	0.5565	0.6100	0.014*
C4	0.5000	0.7500	0.59458 (19)	0.0113 (4)
C7	0.5000	0.7500	0.9170 (2)	0.0130 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I2	0.01764 (6)	0.00914 (5)	0.01122 (5)	0.000	0.000	0.00198 (3)
I4	0.01801 (8)	0.01463 (7)	0.00812 (6)	0.000	0.000	0.000
N7	0.0218 (11)	0.0137 (9)	0.0145 (9)	0.000	0.000	0.000
C1	0.0104 (9)	0.0113 (9)	0.0094 (8)	0.000	0.000	0.000
C2	0.0123 (6)	0.0088 (6)	0.0104 (6)	0.000	0.000	0.0017 (5)
C3	0.0157 (7)	0.0095 (6)	0.0099 (6)	0.000	0.000	0.0002 (5)
C4	0.0121 (9)	0.0115 (9)	0.0104 (9)	0.000	0.000	0.000
C7	0.0137 (10)	0.0120 (9)	0.0133 (10)	0.000	0.000	0.000

Geometric parameters (Å, º)

I2—C2	2.0916 (16)	C1—C7	1.438 (3)
I4—C4	2.094 (2)	C2—C3	1.389 (2)
N7—C7	1.151 (3)	C3—C4	1.396 (2)
C1—C2	1.405 (2)	C3—H3	0.9500
C1—C2$^{\rm i}$	1.405 (2)	C4—C3$^{\rm i}$	1.396 (2)
C2—C1—C2$^{\rm i}$	119.9 (2)	C2—C3—H3	120.5
C2—C1—C7	120.07 (11)	C4—C3—H3	120.5
C2$^{\rm i}$—C1—C7	120.07 (11)	C3$^{\rm i}$—C4—C3	121.6 (2)
C3—C2—C1	120.19 (16)	C3$^{\rm i}$—C4—I4	119.19 (11)
C3—C2—I2	120.65 (12)	C3—C4—I4	119.19 (11)

C1—C2—I2	119.16 (13)	N7—C7—C1	180.0
C2—C3—C4	119.07 (16)		

C2^i—C1—C2—C3	0.000 (1)	C1—C2—C3—C4	0.000 (1)
C7—C1—C2—C3	180.000 (1)	I2—C2—C3—C4	180.000 (1)
C2^i—C1—C2—I2	180.000 (1)	C2—C3—C4—C3^i	0.000 (1)
C7—C1—C2—I2	0.000 (1)	C2—C3—C4—I4	180.000 (1)

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

2,4,6-Triiodophenyl isocyanide (I3NC)

Crystal data

$C_7H_2I_3N$	D_x = 3.279 Mg m^{-3}
M_r = 480.80	Melting point: 467 K
Orthorhombic, *Imma*	Mo $K\alpha$ radiation, λ = 0.71073 Å
a = 7.0552 (3) Å	Cell parameters from 2833 reflections
b = 10.4947 (5) Å	θ = 2.5–36.3°
c = 13.1557 (5) Å	μ = 9.56 mm^{-1}
V = 974.08 (7) Å^3	*T* = 100 K
Z = 4	Block, colourless
F(000) = 840	0.14 × 0.09 × 0.07 mm

Data collection

Bruker VENTURE PHOTON-II diffractometer	1314 independent reflections
Radiation source: micro-focus	1267 reflections with $I > 2\sigma(I)$
φ and ω scans	R_{int} = 0.026
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	θ_{max} = 36.3°, θ_{min} = 2.5°
T_{min} = 0.251, T_{max} = 0.344	$h = -11\rightarrow11$
13040 measured reflections	$k = -17\rightarrow12$
	$l = -21\rightarrow21$

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0044P)^2 + 1.2499P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.011	$(\Delta/\sigma)_{max}$ = 0.001
$wR(F^2)$ = 0.024	$\Delta\rho_{max}$ = 0.72 e Å^{-3}
S = 1.14	$\Delta\rho_{min}$ = −0.48 e Å^{-3}
1314 reflections	Extinction correction: SHELXL2014 (Sheldrick, 2015b), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
40 parameters	Extinction coefficient: 0.00312 (10)
0 restraints	
Hydrogen site location: inferred from neighbouring sites	

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	$U_{\rm iso}$*/$U_{\rm eq}$
I12	0.5000	0.46223 (2)	0.83472 (2)	0.01190 (3)
I14	0.5000	0.7500	0.43715 (2)	0.01245 (4)
N17	0.5000	0.7500	0.91223 (14)	0.0120 (3)
C11	0.5000	0.7500	0.80681 (15)	0.0098 (3)
C12	0.5000	0.63389 (13)	0.75389 (11)	0.0102 (2)
C13	0.5000	0.63411 (14)	0.64790 (11)	0.0112 (2)
H13	0.5000	0.5561	0.6113	0.013*
C14	0.5000	0.7500	0.59640 (15)	0.0108 (3)
C17	0.5000	0.7500	1.00074 (17)	0.0150 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I12	0.01610 (5)	0.00910 (4)	0.01051 (4)	0.000	0.000	0.00184 (3)
I14	0.01561 (6)	0.01413 (6)	0.00761 (5)	0.000	0.000	0.000
N17	0.0134 (7)	0.0125 (7)	0.0102 (7)	0.000	0.000	0.000
C11	0.0106 (8)	0.0106 (7)	0.0081 (7)	0.000	0.000	0.000
C12	0.0121 (5)	0.0086 (5)	0.0098 (5)	0.000	0.000	0.0012 (4)
C13	0.0138 (6)	0.0096 (5)	0.0101 (5)	0.000	0.000	0.0000 (4)
C14	0.0124 (8)	0.0116 (8)	0.0086 (7)	0.000	0.000	0.000
C17	0.0174 (9)	0.0148 (9)	0.0127 (8)	0.000	0.000	0.000

Geometric parameters (Å, º)

I12—C12	2.0920 (14)	C11—C12	1.4035 (17)
I14—C14	2.095 (2)	C12—C13	1.394 (2)
N17—C17	1.164 (3)	C13—C14	1.3922 (17)
N17—C11	1.387 (3)	C13—H13	0.9500
C11—C12$^{\rm i}$	1.4035 (17)	C14—C13$^{\rm i}$	1.3922 (17)
C17—N17—C11	180.0	C14—C13—C12	119.21 (14)
N17—C11—C12$^{\rm i}$	119.75 (9)	C14—C13—H13	120.4
N17—C11—C12	119.74 (9)	C12—C13—H13	120.4
C12$^{\rm i}$—C11—C12	120.51 (18)	C13$^{\rm i}$—C14—C13	121.76 (18)
C13—C12—C11	119.65 (13)	C13$^{\rm i}$—C14—I14	119.12 (9)
C13—C12—I12	120.65 (10)	C13—C14—I14	119.12 (9)
C11—C12—I12	119.70 (11)		
N17—C11—C12—C13	180.000 (1)	C11—C12—C13—C14	0.000 (1)
C12$^{\rm i}$—C11—C12—C13	0.000 (1)	I12—C12—C13—C14	180.000 (1)
N17—C11—C12—I12	0.000 (1)	C12—C13—C14—C13$^{\rm i}$	0.000 (1)
C12$^{\rm i}$—C11—C12—I12	180.000 (1)	C12—C13—C14—I14	180.000 (1)

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