# The Role of Functional Nitro and Cyano Groups in the Self-Assembly of 1,3-Dithiole-2-thione–Halogen Adducts

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ABSTRACT: *The synthesis of five new dihalogen/interhalogen charge transfer adducts featuring cyano and nitro functionalized 1,3-dithiole-2-thiones have been prepared and characterized by x-ray crystallography. The structures feature a complex series of intermolecular close contacts between heteroatoms, as well as hydrogen bonding, to give highly ordered, polymeric assemblies. The adduct **4**.IBr crystallizes in a noncentrosymmetric chiral space group and features a stack of molecules assembled through hydrogen bonds. The functional groups of all adducts participate in networks of close contacts, but do not affect the coordination environment of the dihalogen/interhalogen species.* © 2007 Wiley Periodicals, Inc. Heteroatom Chem 18:176–184, 2007; Published online in Wiley InterScience (www.interscience.wiley.com). DOI 10.1002/hc.20327

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## INTRODUCTION

The reaction of dihalogen and interhalogen species with organic compounds bearing Lewis base functionalities results in the formation of charge-transfer adducts. The key interaction between reactants arises from a two-electron–three-centered noncovalent bond, in which the Lewis base donates electrons from a lone pair to the antibonding $\sigma$ orbital of the acceptor (the dihalogen or interhalogen). There are numerous examples of structures based on interactions with Group 15 and Group 16 atoms and the majority of cases represent phosphine, arsine, chalcogenoether, and chalcogenone functionalities [1–8]. In previous studies, we have investigated adducts of 1,3-dithiole-2-thione derivatives and assessed the role of redox active units and secondary functionalities attached to this heterocycle. For instance, the incorporation of ferrocene in **1** results in an open shell species with magnetic properties, due to the oxidation of the metallocene during adduct formation [6]. Functionalities that interact with the halogen adduct include esters (compound **2**) via hydrogen bonding [8,9] and methylene bromide side groups (in **3**) which form halogen–halogen contacts [10].

These interactions participate in the self-assembly of the adducts to form highly ordered and polymeric supramolecular structures in the solid state. In this work, we report on nitro and cyano derivatized analogues and comment on the role of these functional groups in the self-assembly of halogen adducts.
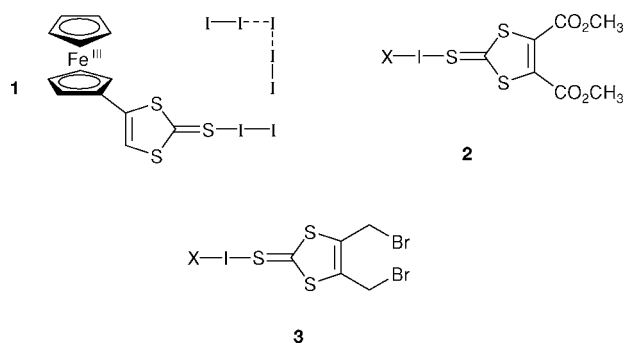


## SYNTHESIS

The 1,3-dithiole-2-thiones **5–7** were prepared according to Scheme 1. Compound **4** was treated with sodium ethoxide to furnish the corresponding thiolate species. The intermediates were allowed to react with the relevant alkyl halide to afford the nitro or cyano functionalized compounds. We attempted a range of reactions of compounds **4–7** with dibromine, diiodine, and iodine monobromide to obtain charge-transfer adducts. The reactions were carried out in the following manner: The donor molecules (**4–7**, 100 mg each) were dissolved in toluene (10 mL) and heated to 100°C. A solution of the corresponding halogen species ($I_2$, $Br_2$ or IBr, 1 eq in 10 mL of toluene) was added and the temperature maintained at 100°C for approximately 10 min. Upon slow cooling, each of the adducts crystallized and were isolated by filtration and washed with toluene and diethyl ether. However, only the following products were isolated in crystalline form: **5**.$I_2$, **6**.1.5$I_2$, **6**.1.5IBr, **4**.IBr, and **7**.2$I_2$. Most of the complexes lost the coordinated halogen on standing but the adducts **4**.IBr and **5**.$I_2$ were sufficiently stable to afford agreeable elemental analyses (Table 1). FTIR data show that the thiocarbonyl bond is weakened on formation of the adduct (C=S stretching frequency is lowered, Table 1) and, as expected, iodine monobromide has a stronger effect than diiodine owing to the polarity of the former (C=S 1013 cm$^{-1}$ for **6**.1.5$I_2$ and 1005 cm$^{-1}$ for
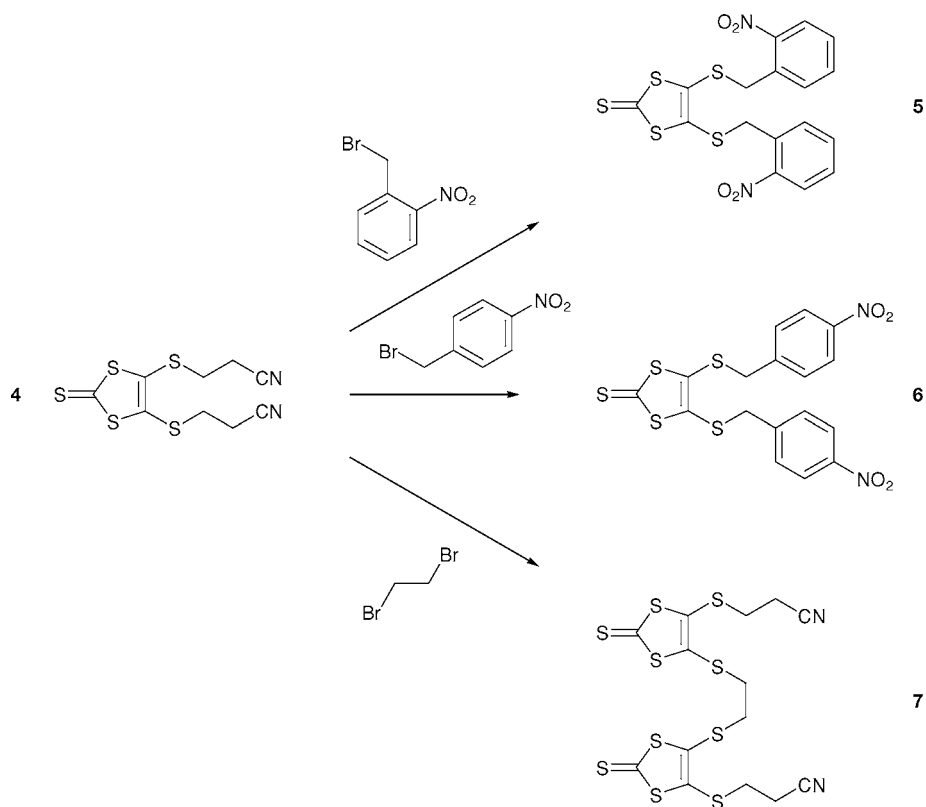


**SCHEME 1**    Reagents and conditions: (1) NaOEt (1.1 eq for **5** and **6**, 0.55 eq for **7**), EtOH, 0°C, 10 min, then (2) corresponding alkyl halide.

**TABLE 1** Selected Bond Lengths,ᵃ Angles (see Fig. 2), and Analytical Data for Crystalline Adducts

| | $C=S$ (Å) | $C-S_{hal}$ (Å) | $C-S$ (Å) | $S-I$ (Å) | $S{\cdots}I$ (Å) | $I-X$ (Å) | Hal–Hal "Free" (Å) | $S-I-X$ (°) | Elemental Analysis, Found (Calcd) | $I-X$ Raman (cm⁻¹) | $C=S$ IR (cm⁻¹) |
|---|---|---|---|---|---|---|---|---|---|---|---|
| **5**.I₂ | 1.673 (7) | 1.728 (9) | 1.707 (5) | 2.823 (5) | 3.463 (11) | 2.778 (5) | — | 176.68 (2) | C, 27.94 (28.26); H, 1.55 (1.67); N, 3.75 (3.88); S, 21.66 (22.19) | 160 | 1021 (1057 for **5**) |
| **6**.1.5I₂ | 1.686 (12) | 1.710 (12) | 1.702 (11) | 2.668 (6) | 3.375 (6) | 2.863 (5) | 2.736 (5) | 176.06 (7) | Unstable, loss of I₂ (and IBr in the case of **6**.1.5IBr) | 165 | 1013 (1005 for **6**.1.5IBr, 1052 for **6**) |
| **4**.IBr (A) | 1.661 (8) | 1.715 (8) | 1.722 (7) | 2.593 (2) | 3.384 (2) | 2.710 (1) | — | 177.23 (6) | C, 21.01 (21.14); H, 1.69 (1.58); N, 5.34 (5.48); S, 31.28 (31.36) | Fluoresces | 1017 (1071 for **4**) |
| **4**.IBr (B) | 1.685 (8) | 1.707 (9) | 1.711 (8) | 2.611 (2) | 3.399 (2) | 2.704 (1) | | 177.56 (6) | | | |
| **7**.2I₂ (A) | 1.681 (21) | 1.702 (22) | 1.699 (22) | 2.686 (7) | 3.546 (8) | 2.845 (3) | | 173.88 (17) | Unstable, loss of I₂ | 175 | 1032 (1057 for **7**) |
| **7**.2I₂ (B) | 1.703 (24) | 1.741 (22) | 1.662 (23) | 2.668 (7) | 3.473 (9) | 2.865 (3) | | 177.56 (18) | | | |
| **7**.2I₂ (C) | 1.686 (23) | 1.708 (22) | 1.701 (23) | 2.674 (7) | 3.432 (7) | 2.875 (3) | | 173.06 (16) | | | |
| **7**.2I₂ (D) | 1.707 (21) | 1.746 (22) | 1.673 (22) | 2.677 (7) | 3.670 (7) | 2.868 (3) | | 174.28 (16) | | | |

ᵃ Short contacts are referred to as interatomic distances less than the sum of the van der Waals radii of the corresponding atoms: H = 1.20, S = 1.80, N = 1.55, Cl = 1.75, Br = 1.85, and I = 1.98 Å.

**6**.1.5IBr). The same trend is observed in the Raman spectra of the I₂ adducts, but for the IBr analogs the low frequency peaks are obscured by fluorescence. The peaks for **5**.I₂ (160 cm⁻¹), **6**.1.5I₂ (165 cm⁻¹), and **7**.2.I₂ (175 cm⁻¹) are lower in frequency than that for uncoordinated diiodine (180 cm⁻¹), and thus show a decrease in bond order within the I₂ molecule.

## X-RAY CRYSTALLOGRAPHY

The asymmetric units of compounds **5**.I₂, **6**.1.5I₂, **4**.IBr, and **7**.2I₂ are shown in Figure 1, with corresponding crystallographic experimental data given in Table 2. In total, there are eight different cases where a 1,3-dithiole-2-thione heterocycle binds to a diiodine or iodine monobromide molecule. These associations allow a good comparison of bond lengths and angles between adducts. Selected data are presented in Table 1 using the identification scheme in Figure 2. As we have observed previously for related adducts, the sulfur atom of the thiocarbonyl functionality bonds to an iodine atom (even in the case of **4**.IBr), together with a secondary, weaker interaction involving one of the dithiole sulfurs. S···I distances between the thiocarbonyl functionality and the halogen species are in the range 2.59–2.82 Å, while for the dithiole species the parameters are 3.37–3.67 Å. The first of these contacts is therefore the predominant factor for the elongation of the dihalogen bond (injection of electron density into the $\sigma^*$ orbital). I—I bond lengths are within 2.77–2.88 Å, and for I—Br the distances are almost equal (2.704(1) and 2.710(1) Å). In comparison, the bond lengths for uncoordinated I₂ and IBr (solid state) are 2.715(6) [11] and 2.521(4) [12], respectively. Therefore, the greatest change in bond length is observed for IBr, which also features the shortest S···I contacts; this is to be expected for the more polarized I—Br molecule. Bond angles between the thiocarbonyl functionalities and the halogens are almost linear (S···I—X at 173.1°–177.6°). In the case of **6**.1.5I₂, an additional molecule of diiodine is coordinated between I₂ units of the adduct through the polarized iodine atom. In this "free" diiodine molecule, the bond length is 2.736(5) Å. Although the inclusion of additional halogen molecules in polyhalide chains has been seen in other systems [6,8–10], the remaining structures in this work are simple 1:1 adducts. The adduct **6**.1.5IBr is isostructural to **6**.1.5I₂, with the thiocarbonyl sulfur coordinating to the iodine atom of the interhalogen and the occupancy of the bromine/iodine atoms of the free molecule in a random distribution.

The structure of **7**.2I₂ has enabled us to identify any relationships between the bond length of the
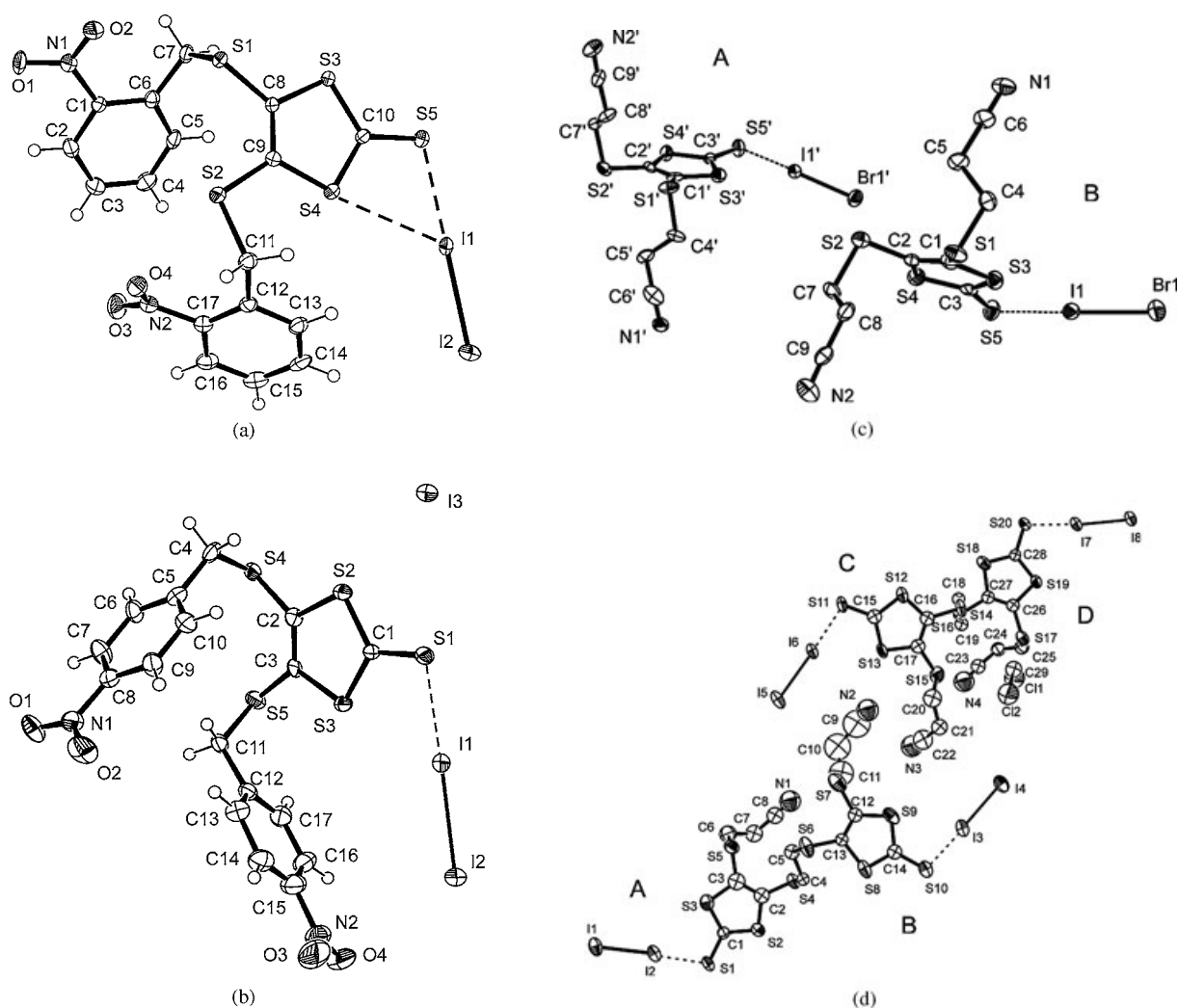
FIGURE 1   Asymmetric units of adducts (a) **5**.I$_2$, (b) **6**.1.5I$_2$, (c) **4**.IBr, and (d) **7**.2I$_2$.

diiodine molecule and the sulfur contacts of the dithiole–thione heterocycle, since the four adducts were found to possess the same molecular structure through crystallography. Comparison of the I—I bond length with the various bond lengths and angles from Table 2 did not yield any conclusive, direct correlation. For example, plots of the S(thiocarbonyl)⋯I distance versus the I—I bond length in adducts A, C, and D yield a linear plot (the shorter the S⋯I contact, the longer the I—I bond, $R > 0.999$), but the S⋯I distance for ring B is 0.197 Å shorter than this. For three adducts, similar linear correlations are observed for plots of I—I bond length versus S—I, C—S, C—S$_{hal}$, and C=S, but the anomalies are randomly distributed between adducts A, B, C, and D. The conclusion from this analysis is that the effects of the thiocarbonyl or dithiole sulfur contacts on the dihalogen

bond lengths are weak noncovalent interactions and likely to be influenced by packing forces in the crystal.

Numerous intermolecular contacts are evident in the crystal structures of the adducts; these interactions are responsible for linking the adducts together to give polymeric structures through noncovalent bonds. In the packing diagram of **5**.I$_2$ (Fig. 3a), the 1,3-dithiole-2-thione rings form dimers through three S⋯S intermolecular short contacts: S(3)⋯S(5′) and S(3′)⋯S(5) at 3.572(16) Å, and S(3)⋯S(3′) at 3.373(6) Å. Sets of dimers are linked in series through short S(1)⋯I(2) contacts (3.668(14) Å) to provide a polymeric chain of S⋯S and S⋯I associations. Between dimers, a weak interaction of I(1)⋯I(1′) exists at 3.976(13) Å. In the structure of **5**.I$_2$, the nitro groups participate in hydrogen bonding, rather than establishing any

**TABLE 2** Data Collection and Refinement Parameters for Compounds **5**.I$_2$, **6**.1.5IBr, **6**.1.5I$_2$, **4**.Ibr, and **7**.2I$_2$

| | *5.I$_2$* | *6.1.5IBr* | *6.1.5I$_2$* | *4.Ibr* | *7.2I$_2$* |
|---|---|---|---|---|---|
| Empirical formula | C$_{17}$H$_{12}$I$_2$N$_2$O$_4$S$_5$ | C$_{17}$H$_{12}$Br$_{1.50}$I$_{1.50}$N$_2$O$_4$S$_5$ | C$_{17}$H$_{12}$I$_3$N$_2$O$_4$S$_5$ | C$_9$H$_8$BrIN$_2$S$_5$ | C$_{29}$H$_{26}$C$_{12}$I$_8$N$_4$S$_{20}$ |
| Formula weight | 722.39 | 778.80 | 849.29 | 511.28 | 2157.84 |
| Space group | *P*−1 | *C*2/*c* | *C*2/*c* | P2$_1$2$_1$2$_1$ | P2$_1$/c |
| *a* (Å) | 5.0104 (4) | 25.7661 (17) | 25.7661 (17) | 8.50950 (10) | 29.9725 (11) |
| *b* (Å) | 11.8399 (9) | 5.6237 (4) | 5.6237 (4) | 13.5128 (2) | 14.7887 (5) |
| *c* (Å) | 19.5093 (13) | 37.861 (3) | 37.861 (3) | 27.7591 (5) | 13.6400 (4) |
| *α* (°) | 77.292 (7) | 90 | 90 | 90 | 90 |
| *β* (°) | 87.576 (7) | 102.350 (8) | 102.350 (8) | 90 | 95.0010 (12) |
| *γ* (°) | 86.124 (7) | 90 | 90 | 90 | 90 |
| Volume (Å$^3$) | 1125.96 (15) | 5359.1 (7) | 5359.1 (7) | 3191.94 (8) | 6023.0 (3) |
| *Z* | 2 | 8 | 8 | 8 | 4 |
| *D*$_c$ (mg/m$^3$) | 1.861 | 1.931 | 2.105 | 2.128 | 2.380 |
| *μ* (mm$^{-1}$) | 3.285 | 4.423 | 3.914 | 5.147 | 4.932 |
| Crystal size (mm$^3$) | 0.38 × 0.14 × 0.04 | 0.45 × 0.25 × 0.01 | 0.16 × 0.08 × 0.01 | 0.25 × 0.05 × 0.05 | 0.10 × 0.10 × 0.02 |
| *θ* range | 3.06–27.50 | 3.00−27.49 | 3.00−27.50 | 2.50−26.00 | 2.98–23.26 |
| Measured reflections | 21838 | 3246 | 24802 | 25989 | 40567 |
| Independent reflections [*I* > 2*σ*(*I*)] | 5150 | 2098 | 5904 | 6259 | 8382 |
| *R*$_{int}$ | 0.1043 | 0.0542 | 0.0654 | 0.1072 | 0.0789 |
| *R* indices [*F*$^2$ > 2*σ*F$^2$] | 0.0373/0.0848 | 0.1108/0.3239 | 0.0896/0.2518 | 0.0469/0.0687 | 0.1168/0.3427 |
| *R* indices [all data] | 0.0431/0.0896 | 0.1302/0.3578 | 0.1022/0.2746 | 0.1207/0.0778 | 0.1450/0.3489 |
| Goodness of fit | 1.012 | 1.499 | 1.053 | 0.888 | 2.292 |
| *ρ*$_{max}$/*ρ*$_{min}$ (e Å$^{-3}$) | 1.689 and −1.607 | 1.702 and −1.825 | 7.539 and −2.190 | 0.932 and −0.718 | 3.339 and −2.247 |

contacts with the dihalogen species (Figure 3b). Within each molecule, an intramolecular H-bond arises between O(4)⋯H(4) at 2.419(5) Å. Ten-membered rings are formed from intermolecular H-bonding between identical nitrobenzene rings (O(3)⋯H(16), 2.431(9) Å; O(1)⋯H(2), 2.548(11) Å) O(3)⋯H(16), while a third intermolecular close contact is observed in O(1)⋯H(15) at 2.563(10) Å.

The heterocyclic and diiodine units of **6**.1.5I$_2$ form polymeric, planar ribbons through close S⋯S, S⋯I, and I⋯I contacts (Fig. 4a). Pairs of the molecule **6**.1.5I$_2$ are linked through the coordination of diiodine I(3)−I(3′) via I(2)⋯I(3) contacts (3.397(6) Å). The latter is also coordinated by the S(2) atom of the dithiole ring, perpendicular to and on either side of the diiodine bond to form two fused triangles. The S(2)⋯I(3) contacts in each
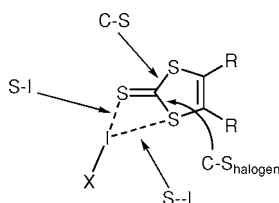
three-membered ring are asymmetric (3.555(10) and 3.62(1) Å). Five-membered rings share a common edge with this arrangement, through the sequence ⋯S(2)−C(1)−S(1)⋯I(1)−I(2)⋯I(3)⋯. Each pentagon is linked to another through a common, fused four-membered ring, comprising ⋯S(1)−I(1)⋯S(1′)−I(1′)⋯. The parallelogram has S(1)⋯I(1′) distances of 3.670(4) Å and angles S(1)⋯I(1′)−S(1′) and I(1)−S(1)⋯I(1′) at 98.16(8)° and 81.84(7)°, respectively. Once again, the nitro groups of the aryl units are associated with hydrogen bonding rather than iodine atoms (Fig. 4b). The atom O(1) forms H bonds with H(11B) (2.439(14) Å) and H(13) (2.554(10) Å), while a third contact exists in O(3)⋯H(4B) (2.427(17) Å).

The packing diagram of **4**.IBr reveals little participation of the cyanoethylene units toward the self-assembly of the compound. Interactions involving the nitrogen atom are limited to H bonding and are in the range 2.55–2.64 Å. The two different molecules of the asymmetric unit form their own separate rows (see Fig. 5a), and close contacts between S(4)⋯S(4′) (3.554 (3) Å) and I(1)⋯I(1′) (3.955(1) Å) hold two rows together (Fig. 5a). By viewing down the *a* axis, one can see that the molecules form stacks (Fig. 5b). Along this plane, each molecule forms an inverse of its adjacent partner. The molecules are held together
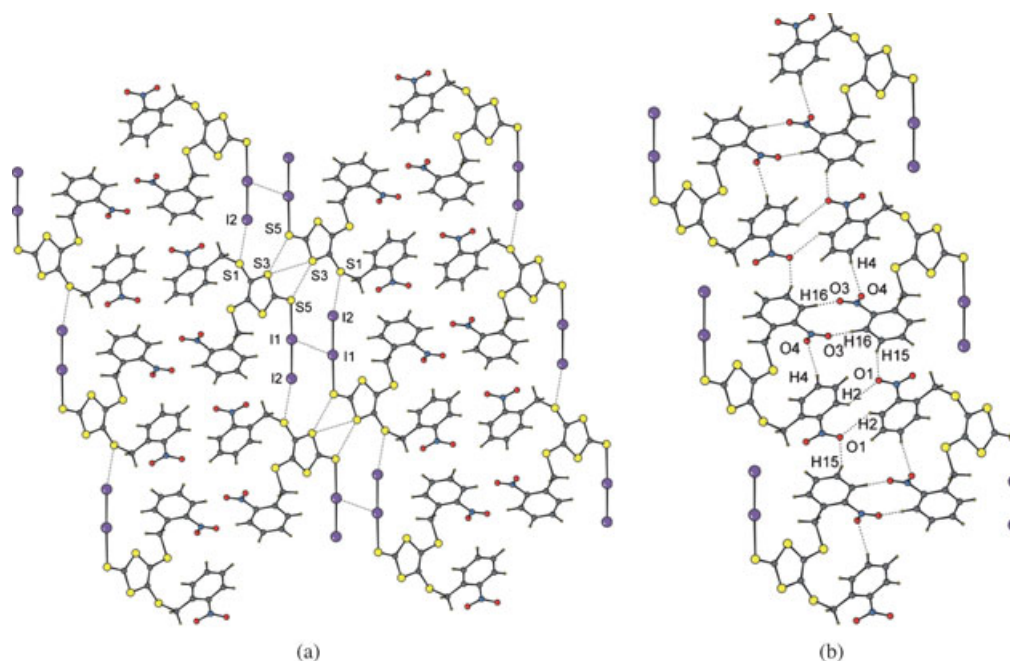


**FIGURE 2** Labeling scheme for Table 1.

**FIGURE 3** Crystal packing diagram of **5**.I$_2$ viewed along the *a* axis, showing (a) S⋯I and I⋯I contacts and (b) hydrogen bonding.

tightly through H bonding involving both bromine atoms. Each of the halogens participate in two such bonds, connecting to adjacent units either side of the molecule (Br(1)⋯H(5a′), 2.859(1) Å; Br(1)⋯H(7b′), 2.866(1) Å; Br(1′)⋯H(5a), 2.853(1) Å; Br(1′)⋯H(7b), 2.871(1) Å). Between stacks there is a significant contact in S(4′)⋯I(1′) at 3.718 (2) Å and a weaker association in S(4)⋯I(1) at 3.793 (2) Å (Fig. 5c).

In compound **7**.2I$_2$ numerous intermolecular contacts arise between S⋯S, S⋯I, and S⋯N pairs of atoms. Owing to the complexity of these interactions, selected data are presented in Table 3 and a single representation of the packing is given in Figure 6. Of particular note, there are only two close contacts involving the nitrogen atoms of the cyano groups.
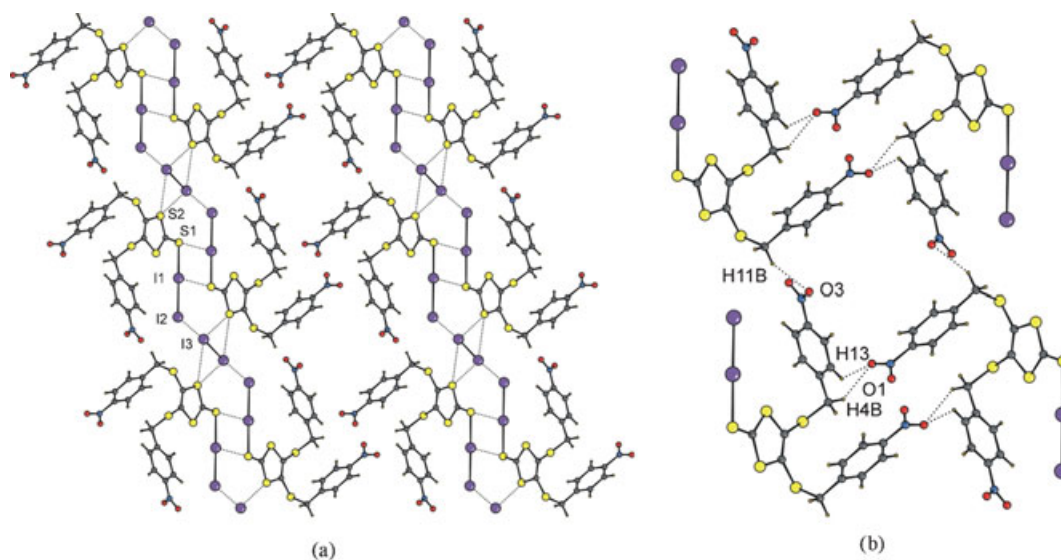


**FIGURE 4** Crystal packing diagram of **6**.1.5I$_2$ viewed along the *b* axis, showing (a) S⋯I and I⋯I contacts and (b) hydrogen bonding.
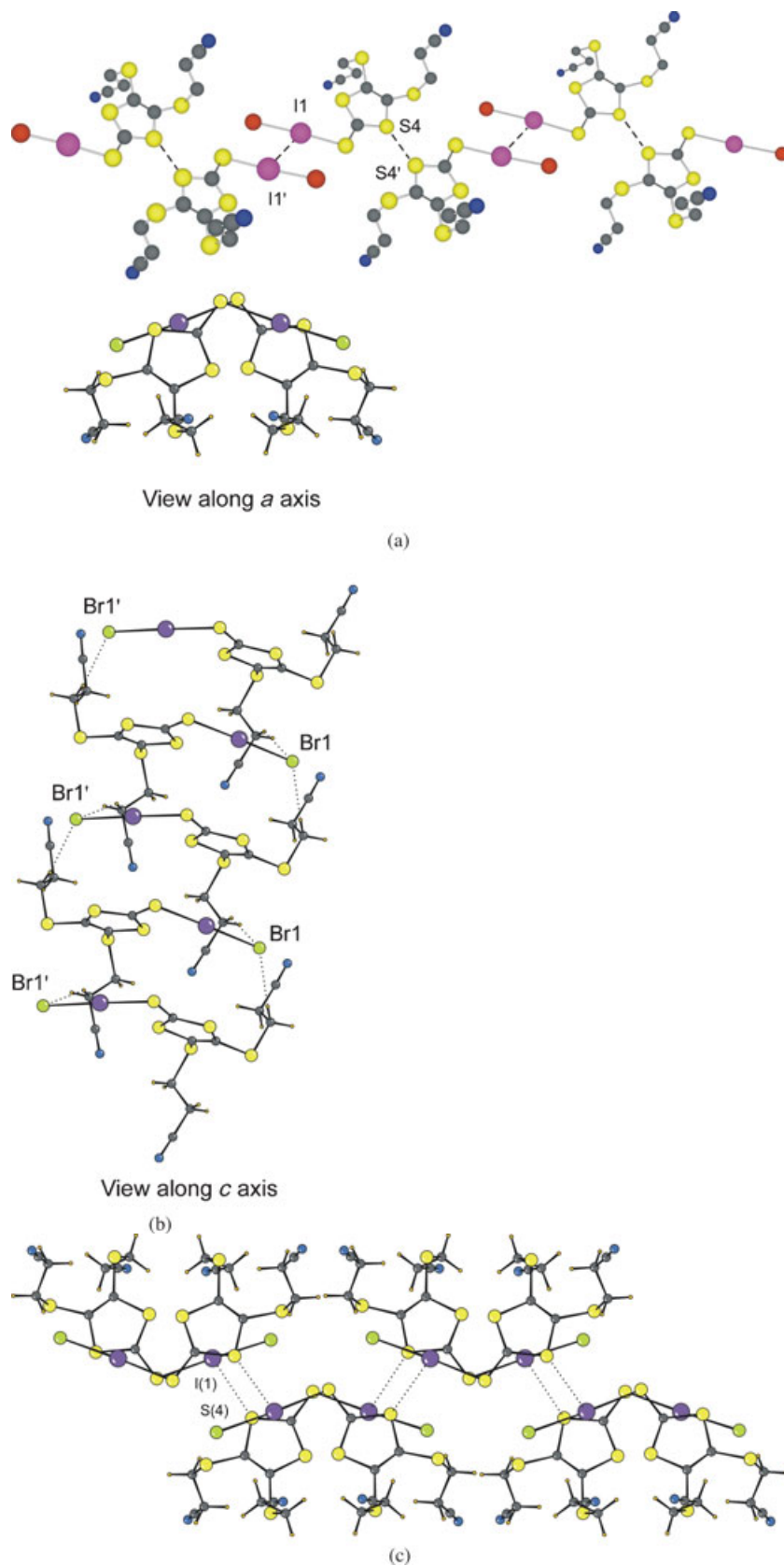
FIGURE 5   Crystal packing diagram of **4**.IBr showing (a) S···S and I···I contacts, (b) stacks of molecules viewed down the *a* and *c* axes, and (c) S···I contacts between stacks.

**TABLE 3**  Summary of Intermolecular Contacts in **7**.2I$_2$

| Contacts | | Contact Distance (Å) | $\Delta^a$ |
|---|---|---|---|
| I1 | S4 | 3.643 | −0.137 |
| I2 | S8 | 3.674 | −0.106 |
| I2 | S4 | 3.687 | −0.093 |
| I6 | S14 | 3.697 | −0.083 |
| I6 | S12 | 3.779 | −0.001 |
| I6 | S18 | 3.753 | −0.027 |
| I8 | S12 | 3.63 | −0.15 |
| I8 | S16 | 3.76 | −0.02 |
| N1 | S17 | 3.255 | −0.095 |
| N2 | S13 | 3.05 | −0.3 |
| S1 | S8 | 3.589 | −0.011 |
| S11 | S18 | 3.568 | −0.032 |

$^a$Difference between sum of van der Waals radii of X and Y and the contact distance.

Both involve interactions with sulfur atoms—one from the dithiole ring (S(13)···N(2)) and one from the sulfanyl substituent (S(17)···N(1)). There

are no associations between the halogen atoms and the cyano functionalities; the iodine atoms form close contacts exclusively with sulfur atoms.

## EXPERIMENTAL

Compounds **5** [13], **6** [14], and **7** [15] were prepared as described previously. *X-ray crystallography*: Data were collected at 120 K on a Nonius Kappa CCD area-detecting diffractometer located at the window of a Nonius FR591 rotating-anode x-ray generator, equipped with a molybdenum target ($\lambda$ Mo K$\alpha = 0.71073$ Å). Structures were solved and refined using the SHELX-97 suite of programs [16]. Data were corrected for absorption effects by means of comparison of equivalent reflections using the program SORTAV [17]. Non-hydrogen atoms were refined anisotropically, while hydrogen atoms were fixed in idealized positions, with their thermal parameters riding on the values of their parent atoms. CCDC
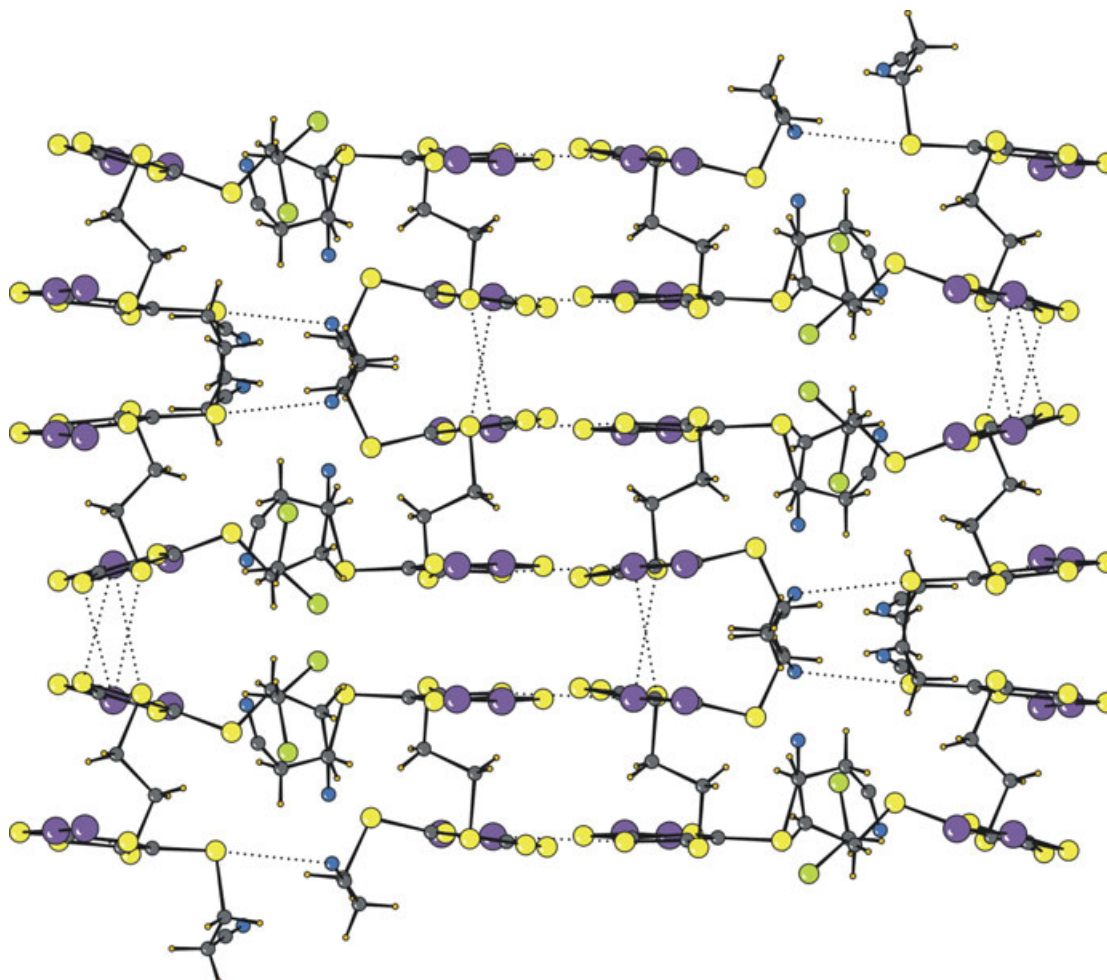


**FIGURE 6**  Packing diagram of **7**.2I$_2$ viewed along the *c* axis and showing intermolecular S···S, S···I, and S···N contacts.

223241 and CCDC 299125–299128 contain the supplementary crystallographic data for this paper. (These data can be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html [or from the Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336033; or deposit@ccdc.cam.ac. uk].)

## CONCLUSION

Five new charge-transfer adducts have been prepared and characterized by x-ray crystallography. Intermolecular close contacts between heteroatoms are assumed to be two-electron–three-centered noncovalent bonds. The heterocycles are functionalized with cyano or nitro groups and display hydrogen bonding and (in the case of **7**.2I$_2$) intermolecular S···N contacts. These interactions participate in the highly ordered, extensive polymeric nature of the adducts, but the functional groups do not interact directly with the halogen species.

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