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## Structure Reports

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# The halogen-bonded adduct 1,4-bis(pyridin-4-yl)buta-1,3-diyne–1,1,2,2,3,3,4,4-,5,5,6,6,7,7,8,8-hexadecafluoro-1,8-diiodooctane (1/1)

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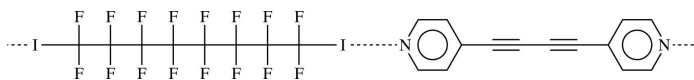
Received 23 November 2012; accepted 18 January 2013

Key indicators: single-crystal X-ray study;  $T = 295$  K; mean  $\sigma(\text{C}-\text{C}) = 0.007$  Å;R factor = 0.039;  $wR$  factor = 0.114; data-to-parameter ratio = 16.1.

In the crystal structure of the title compound,  $\text{C}_8\text{F}_{16}\text{I}_2\text{-C}_{14}\text{H}_8\text{N}_2$ , the molecules form infinite chains parallel to  $[2\bar{1}1]$  through two symmetry-independent  $\text{C}-\text{I}\cdots\text{N}$  halogen bonds (XBs). As commonly found, the perfluoroalkyl molecules segregate from the hydrocarbon ones, forming a layered structure. Apart from the XB, the only contact below the sum of van der Waals radii is a weak  $\text{H}\cdots\text{F}$  contact. The topology of the network is a nice example of the paradigm of the expansion of ditopic starting modules; the XB leads to the construction of infinite supramolecular chains along  $[2\bar{1}1]$  formed by alternating XB donors and acceptors.

## Related literature

For the use of bis-(4-pyridyl)buta-1,3-diyne in crystal engineering based on hydrogen bonding and transition metal binding, see: Nakamura *et al.* (2003); Curtis *et al.* (2005); Maekawa *et al.* (2000); Badruz Zaman *et al.* (2001); Allan *et al.* (1988). For  $\text{N}\cdots\text{I}$  halogen bonds based on  $\alpha,\omega$ -diiodoperfluorocarbons, see: Neukirch *et al.* (2005); Navarrini *et al.* (2000); Liantonio *et al.* (2003); Bertani *et al.* (2002); Metrangolo *et al.* (2004, 2008); Fox *et al.* (2004); Dey *et al.* (2009). For segregation of perfluoroalkyl chains, see: Fox *et al.* (2008). For chirality and order/disorder of long perfluoroalkyl chains, see: Monde *et al.* (2006). For the synthesis of bis-(4-pyridyl)buta-1,3-diyne, see: Della Ciana & Haim (1984).



## Experimental

### Crystal data

 $\text{C}_8\text{F}_{16}\text{I}_2\cdot\text{C}_{14}\text{H}_8\text{N}_2$  $M_r = 858.10$ Triclinic,  $P\bar{1}$  $a = 5.4849$  (11) Å $b = 14.302$  (2) Å $c = 18.354$  (3) Å $\alpha = 111.40$  (2)° $\beta = 90.35$  (2)° $\gamma = 94.03$  (2)° $V = 1336.4$  (4) Å<sup>3</sup> $Z = 2$ Mo  $K\alpha$  radiation $\mu = 2.48$  mm<sup>-1</sup> $T = 295$  K $0.36 \times 0.12 \times 0.10$  mm

### Data collection

Bruker SMART CCD area detector diffractometer

Absorption correction: multi-scan (*SADABS*; Bruker, 2010) $T_{\min} = 0.734$ ,  $T_{\max} = 1.000$ 

15067 measured reflections

6100 independent reflections

4600 reflections with  $I > 2\sigma(I)$  $R_{\text{int}} = 0.026$ 

### Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.039$  $wR(F^2) = 0.114$  $S = 1.02$ 

6100 reflections

379 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.90$  e Å<sup>-3</sup> $\Delta\rho_{\text{min}} = -0.57$  e Å<sup>-3</sup>

**Table 1**

Halogen and hydrogen-bonding contacts (Å, °).

| $\text{C}-\text{X}\cdots\text{Y}$                | $\text{X}\cdots\text{Y}$ | $\text{C}-\text{X}\cdots\text{Y}$ |
|--|--------------------------|-----------------------------------|
| $\text{C1}-\text{I1}\cdots\text{N1}$             | 2.863 (4)                | 177.93 (16)                       |
| $\text{C8}-\text{I2}\cdots\text{N2}^i$           | 2.887 (4)                | 175.39 (16)                       |
| $\text{C1}-\text{F1}\cdots\text{H9}^{\text{ii}}$ | 2.60                     | 145.3                             |

Symmetry codes: (i) =  $-2 + x, 1 + y, -1 + z$ ; (ii) =  $-x, 1 - y, 1 - z$ .

Data collection: *APEX2* (Bruker, 2010); cell refinement: *SAINT* (Bruker, 2010); data reduction: *SAINT*; program(s) used to solve structure: *SIR2002* (Burla *et al.*, 2003); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *enCIFer* (Allen *et al.*, 2004).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FY2080).

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## supporting information

*Acta Cryst.* (2013). E69, o328–o329 [doi:10.1107/S1600536813001888]

## The halogen-bonded adduct 1,4-bis(pyridin-4-yl)buta-1,3-diyne– 1,1,2,2,3,3,4,4,5,5,6,6,7,7,8,8-hexadecafluoro-1,8-diiodooctane (1/1)

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### S1. Comment

Bis-(4-pyridyl)buta-1,3-diyne (1) (Allan *et al.*, 1988) has been used as ditopic hydrogen bonding (HB) acceptor in crystal engineering (Nakamura *et al.*, 2003; Curtis *et al.*, 2005) and in transition metals complexes (Badruz Zaman *et al.*, 2001; Maekawa *et al.*, 2000), but it has never been used in halogen bonding (XB) adducts formation. Our group has shown that  $\alpha,\omega$ -diiodoperfluorocarbons are very good ditopic XB donors, both towards neutral (Fox *et al.*, 2004) and ionic (Metrangolo *et al.*, 2008) electron-donors. As expected, when solutions of (1) and 1,1,2,2,3,3,4,4,5,5,6,6,7,7,8,8-hexadecafluoro-1,8-diiodooctane (2), are mixed, the (1)⋯(2) adduct quickly precipitates (Fig. 1). It forms an infinite one-dimensional and non-covalent polymer through short XBs (Table 1). In our experience, this kind of nearly linear adduct has normally  $Z' = 1/2$ , that is, both molecules lie on crystallographic elements of symmetry, more frequently  $C_i$ , but also  $C_2$  (see Table 2). Here instead, both molecules are in general position and  $Z'$  is 1. The cause of the molecular symmetry breaking is an F⋯H contact, the only interaction shorter than the sum of the van der Waals radii beside the I⋯N XBs. (Table 1). As happens in most structures containing perfluorocarbons and hydrocarbons moieties (see for example Fox *et al.*, 2008), the two components segregate and a layered structure is formed (Fig. 2).

### S2. Experimental

(1) was synthesized according to Della Ciana & Haim (1984); (2) was from Aldrich. The adduct was obtained by slow evaporation from a 1:1 solution of the two components in chloroform.

### S3. Refinement

The lowest energy conformation of long perfluoroalkanes is chiral in due to the sterically hindered F⋯F contacts between 1,3 positioned  $CF_2$  groups (Monde *et al.*, 2006). Their crystals frequently show a superposition, in the same crystallographic site, of the two more common conformers: all-trans<sup>+</sup> and all-trans<sup>-</sup> (see, for example, Dey <i>1,4-bis-(pyridin-4-yl)buta-1,3-diyne–1,1,2,2,3,3,4,4,5,5,6,6,7,7,8,8-hexadecafluoro-1,8-diiodooctane (1/1)> et al., 2009), which in some cases have unequal occupancy factors. This is particularly visible for  $-CF_3$  terminated chains, that is, for  $\alpha,\omega$ -dihaloperfluorocarbons, which have the two endings strongly interacting with any electron-donor site. This type of disorder is difficult to observe, at least at room temperature, because it is masked by the large ADPs of perfluoroalkyl chains. This is due to the very weak interactions that their chains give with any environment. In the present study, splitting of some fluorine atoms was suggested by SHELXL but did not give good results. In spite of the use of a lot of restraints and constraints, at the price of a large increase of refined parameters, the final  $R_1$ ,  $wR_2$  and  $\Delta\rho$  did not change significantly. The correlations between couples of parameters involving split atoms were very high, many of them being in the range 0.95–0.99. For these reasons we decided to use the ordered model of refinement. All H atoms were placed in geometrically calculated positions with C—H = 0.93 Å and  $U_{iso}(H) = 1.2 U_{eq}(C)$ .

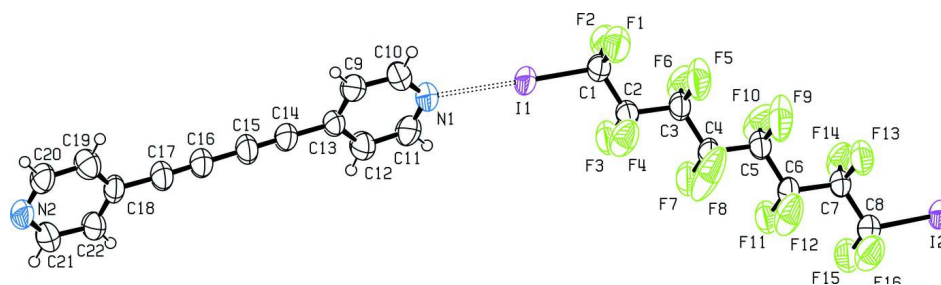


Figure 1

The asymmetric unit of the title compound. Displacement ellipsoids are drawn at 50% probability level.

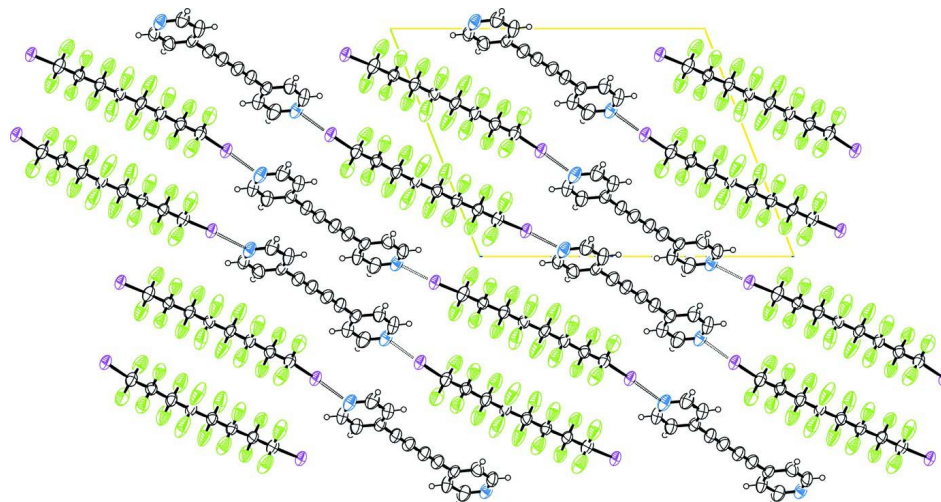


Figure 2

Crystal packing of the title compound viewed along the *a* axis, showing the alternating perfluorocarbon/hydrocarbon layers.

### 1,4-Bis(pyridin-4-yl)buta-1,3-diyne-1,1,2,2,3,3,4,4,5,5,6,6,7,7,8,8-hexadecafluoro-1,8-diiodooctane (1/1)

#### Crystal data

$C_8F_{16}I_2 \cdot C_{14}H_8N_2$

$M_r = 858.10$

Triclinic,  $P\bar{1}$

$a = 5.4849$  (11) Å

$b = 14.302$  (2) Å

$c = 18.354$  (3) Å

$\alpha = 111.40$  (2)°

$\beta = 90.35$  (2)°

$\gamma = 94.03$  (2)°

$V = 1336.4$  (4) Å<sup>3</sup>

$Z = 2$

$F(000) = 808$

$D_x = 2.132$  Mg m<sup>-3</sup>

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

Cell parameters from 981 reflections

$\theta = 2.4$ – $27.4$ °

$\mu = 2.48$  mm<sup>-1</sup>

$T = 295$  K

Elongated prism, colourless

$0.36 \times 0.12 \times 0.10$  mm

#### Data collection

Bruker SMART CCD area detector  
diffractometer

$\omega$  and  $\phi$  scans

Absorption correction: multi-scan  
(*SADABS*; Bruker, 2010)

$T_{\min} = 0.734$ ,  $T_{\max} = 1.000$

15067 measured reflections

6100 independent reflections

4600 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.026$

$\theta_{\max} = 27.5^\circ$ ,  $\theta_{\min} = 2.3^\circ$   
 $h = -7 \rightarrow 7$

$k = -18 \rightarrow 18$   
 $l = -23 \rightarrow 23$

*Refinement*

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.039$   
 $wR(F^2) = 0.114$   
 $S = 1.02$   
 6100 reflections  
 379 parameters  
 0 restraints

Hydrogen site location: inferred from  
 neighbouring sites  
 H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0662P)^2 + 0.5679P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.002$   
 $\Delta\rho_{\max} = 0.90 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.57 \text{ e } \text{\AA}^{-3}$

*Special details*

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

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

|     | x            | y           | z             | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|-------------|---------------|----------------------------------|
| I1  | 0.11335 (6)  | 0.46492 (2) | 0.32421 (2)   | 0.06421 (11)                     |
| C1  | -0.0850 (8)  | 0.5404 (4)  | 0.2631 (3)    | 0.0616 (10)                      |
| F1  | -0.1957 (7)  | 0.6162 (3)  | 0.3136 (2)    | 0.1019 (12)                      |
| F2  | -0.2603 (6)  | 0.4751 (3)  | 0.2165 (2)    | 0.0938 (10)                      |
| C2  | 0.0789 (7)   | 0.5809 (3)  | 0.2117 (2)    | 0.0525 (9)                       |
| F3  | 0.2403 (6)   | 0.6512 (3)  | 0.25989 (19)  | 0.0936 (10)                      |
| F4  | 0.1996 (6)   | 0.5068 (2)  | 0.16550 (18)  | 0.0852 (9)                       |
| C3  | -0.0565 (8)  | 0.6289 (3)  | 0.1613 (2)    | 0.0598 (10)                      |
| F5  | -0.1913 (8)  | 0.6966 (3)  | 0.2048 (2)    | 0.1254 (16)                      |
| F6  | -0.2101 (6)  | 0.5560 (3)  | 0.1115 (2)    | 0.1027 (12)                      |
| C4  | 0.1092 (8)   | 0.6720 (3)  | 0.1114 (2)    | 0.0558 (9)                       |
| F7  | 0.2517 (11)  | 0.7470 (4)  | 0.1598 (3)    | 0.170 (3)                        |
| F8  | 0.2464 (7)   | 0.6031 (4)  | 0.0697 (3)    | 0.1272 (17)                      |
| C5  | -0.0268 (8)  | 0.7128 (3)  | 0.0561 (2)    | 0.0569 (10)                      |
| F9  | -0.1635 (10) | 0.7821 (4)  | 0.0985 (2)    | 0.152 (2)                        |
| F10 | -0.1735 (7)  | 0.6388 (3)  | 0.0088 (2)    | 0.1142 (14)                      |
| C6  | 0.1400 (8)   | 0.7555 (3)  | 0.0062 (2)    | 0.0546 (9)                       |
| F11 | 0.2902 (7)   | 0.8298 (3)  | 0.0525 (2)    | 0.1111 (13)                      |
| F12 | 0.2741 (6)   | 0.6821 (3)  | -0.0373 (2)   | 0.0963 (11)                      |
| C7  | 0.0006 (7)   | 0.7947 (3)  | -0.0502 (2)   | 0.0519 (9)                       |
| F13 | -0.1322 (6)  | 0.8675 (2)  | -0.00739 (17) | 0.0850 (9)                       |
| F14 | -0.1497 (6)  | 0.7197 (2)  | -0.09729 (17) | 0.0826 (9)                       |
| C8  | 0.1623 (9)   | 0.8356 (4)  | -0.1016 (3)   | 0.0651 (11)                      |
| F15 | 0.3155 (6)   | 0.9102 (3)  | -0.0564 (2)   | 0.1065 (12)                      |
| F16 | 0.2988 (7)   | 0.7622 (3)  | -0.1455 (2)   | 0.1068 (13)                      |
| I2  | -0.04797 (6) | 0.88682 (2) | -0.17707 (2)  | 0.06339 (11)                     |
| C9  | 0.4885 (9)   | 0.3202 (4)  | 0.5119 (3)    | 0.0669 (12)                      |
| H1B | 0.4645       | 0.3284      | 0.5639        | 0.080*                           |

|     |             |             |            |             |
|-----|-------------|-------------|------------|-------------|
| C10 | 0.3452 (9)  | 0.3656 (4)  | 0.4747 (3) | 0.0758 (13) |
| H10 | 0.2236      | 0.4041      | 0.5031     | 0.091*      |
| N1  | 0.3700 (8)  | 0.3578 (3)  | 0.4013 (3) | 0.0730 (11) |
| C11 | 0.5425 (10) | 0.3021 (5)  | 0.3621 (3) | 0.0775 (14) |
| H11 | 0.5607      | 0.2952      | 0.3101     | 0.093*      |
| C12 | 0.6971 (9)  | 0.2536 (4)  | 0.3932 (3) | 0.0689 (12) |
| H12 | 0.8175      | 0.2160      | 0.3634     | 0.083*      |
| C13 | 0.6682 (7)  | 0.2623 (3)  | 0.4706 (2) | 0.0553 (9)  |
| C14 | 0.8229 (8)  | 0.2136 (4)  | 0.5068 (3) | 0.0616 (10) |
| C15 | 0.9543 (9)  | 0.1761 (4)  | 0.5378 (3) | 0.0651 (11) |
| C16 | 1.1066 (9)  | 0.1343 (4)  | 0.5753 (3) | 0.0646 (11) |
| C17 | 1.2428 (9)  | 0.0982 (4)  | 0.6076 (3) | 0.0654 (11) |
| C18 | 1.3964 (8)  | 0.0545 (3)  | 0.6481 (2) | 0.0565 (10) |
| C19 | 1.3530 (9)  | 0.0648 (4)  | 0.7250 (3) | 0.0678 (11) |
| H19 | 1.2238      | 0.0998      | 0.7514     | 0.081*      |
| C20 | 1.5051 (9)  | 0.0220 (4)  | 0.7606 (3) | 0.0674 (11) |
| H20 | 1.4754      | 0.0291      | 0.8121     | 0.081*      |
| N2  | 1.6933 (7)  | -0.0291 (3) | 0.7268 (2) | 0.0699 (10) |
| C21 | 1.7289 (9)  | -0.0398 (4) | 0.6527 (3) | 0.0743 (13) |
| H21 | 1.8567      | -0.0769     | 0.6274     | 0.089*      |
| C22 | 1.5882 (9)  | 0.0006 (4)  | 0.6113 (3) | 0.0706 (13) |
| H22 | 1.6214      | -0.0082     | 0.5597     | 0.085*      |

*Atomic displacement parameters (Å<sup>2</sup>)*

|     | $U^{11}$     | $U^{22}$    | $U^{33}$     | $U^{12}$     | $U^{13}$      | $U^{23}$     |
|-----|--------------|-------------|--------------|--------------|---------------|--------------|
| II  | 0.06808 (19) | 0.0752 (2)  | 0.06554 (19) | 0.00504 (14) | -0.00501 (13) | 0.04518 (15) |
| C1  | 0.065 (3)    | 0.071 (3)   | 0.062 (2)    | 0.017 (2)    | 0.009 (2)     | 0.037 (2)    |
| F1  | 0.132 (3)    | 0.111 (2)   | 0.094 (2)    | 0.070 (2)    | 0.056 (2)     | 0.0628 (19)  |
| F2  | 0.0727 (18)  | 0.119 (3)   | 0.116 (3)    | -0.0210 (18) | -0.0270 (17)  | 0.079 (2)    |
| C2  | 0.054 (2)    | 0.057 (2)   | 0.052 (2)    | 0.0110 (18)  | 0.0002 (17)   | 0.0243 (18)  |
| F3  | 0.108 (2)    | 0.100 (2)   | 0.084 (2)    | -0.0334 (19) | -0.0355 (18)  | 0.0554 (18)  |
| F4  | 0.103 (2)    | 0.101 (2)   | 0.0805 (18)  | 0.0584 (18)  | 0.0315 (16)   | 0.0569 (16)  |
| C3  | 0.068 (3)    | 0.067 (2)   | 0.056 (2)    | 0.021 (2)    | 0.006 (2)     | 0.034 (2)    |
| F5  | 0.171 (4)    | 0.146 (3)   | 0.112 (3)    | 0.114 (3)    | 0.073 (3)     | 0.091 (2)    |
| F6  | 0.091 (2)    | 0.135 (3)   | 0.110 (3)    | -0.040 (2)   | -0.0437 (19)  | 0.087 (2)    |
| C4  | 0.061 (2)    | 0.058 (2)   | 0.060 (2)    | 0.0006 (19)  | -0.0104 (18)  | 0.0354 (19)  |
| F7  | 0.238 (5)    | 0.165 (4)   | 0.139 (3)    | -0.132 (4)   | -0.127 (4)    | 0.120 (3)    |
| F8  | 0.122 (3)    | 0.180 (4)   | 0.158 (3)    | 0.098 (3)    | 0.080 (3)     | 0.138 (3)    |
| C5  | 0.067 (2)    | 0.060 (2)   | 0.049 (2)    | 0.021 (2)    | 0.0048 (18)   | 0.0245 (18)  |
| F9  | 0.222 (5)    | 0.187 (4)   | 0.122 (3)    | 0.158 (4)    | 0.109 (3)     | 0.118 (3)    |
| F10 | 0.107 (3)    | 0.161 (3)   | 0.101 (2)    | -0.058 (2)   | -0.046 (2)    | 0.092 (3)    |
| C6  | 0.061 (2)    | 0.057 (2)   | 0.053 (2)    | 0.0073 (18)  | -0.0054 (18)  | 0.0279 (18)  |
| F11 | 0.130 (3)    | 0.118 (3)   | 0.105 (2)    | -0.055 (2)   | -0.068 (2)    | 0.077 (2)    |
| F12 | 0.101 (2)    | 0.128 (3)   | 0.102 (2)    | 0.069 (2)    | 0.0459 (18)   | 0.081 (2)    |
| C7  | 0.057 (2)    | 0.054 (2)   | 0.050 (2)    | 0.0100 (17)  | -0.0028 (17)  | 0.0253 (17)  |
| F13 | 0.105 (2)    | 0.098 (2)   | 0.0755 (17)  | 0.0553 (18)  | 0.0257 (16)   | 0.0516 (16)  |
| F14 | 0.095 (2)    | 0.0869 (19) | 0.0769 (18)  | -0.0257 (16) | -0.0342 (16)  | 0.0497 (15)  |

|     |              |            |              |              |               |              |
|-----|--------------|------------|--------------|--------------|---------------|--------------|
| C8  | 0.062 (3)    | 0.079 (3)  | 0.070 (3)    | 0.009 (2)    | -0.001 (2)    | 0.045 (2)    |
| F15 | 0.093 (2)    | 0.131 (3)  | 0.123 (3)    | -0.044 (2)   | -0.047 (2)    | 0.089 (2)    |
| F16 | 0.110 (2)    | 0.147 (3)  | 0.109 (2)    | 0.073 (2)    | 0.051 (2)     | 0.090 (2)    |
| I2  | 0.07009 (19) | 0.0747 (2) | 0.06057 (18) | 0.00691 (14) | -0.00418 (13) | 0.04264 (15) |
| C9  | 0.061 (2)    | 0.090 (3)  | 0.062 (3)    | 0.006 (2)    | 0.000 (2)     | 0.042 (2)    |
| C10 | 0.066 (3)    | 0.093 (3)  | 0.088 (3)    | 0.026 (3)    | 0.008 (2)     | 0.052 (3)    |
| N1  | 0.064 (2)    | 0.091 (3)  | 0.086 (3)    | 0.011 (2)    | -0.005 (2)    | 0.058 (2)    |
| C11 | 0.078 (3)    | 0.110 (4)  | 0.066 (3)    | 0.006 (3)    | -0.002 (2)    | 0.057 (3)    |
| C12 | 0.068 (3)    | 0.087 (3)  | 0.063 (3)    | 0.017 (2)    | 0.004 (2)     | 0.038 (2)    |
| C13 | 0.050 (2)    | 0.064 (2)  | 0.060 (2)    | 0.0001 (18)  | -0.0102 (17)  | 0.034 (2)    |
| C14 | 0.062 (2)    | 0.073 (3)  | 0.062 (2)    | 0.003 (2)    | -0.0062 (19)  | 0.039 (2)    |
| C15 | 0.067 (3)    | 0.069 (3)  | 0.070 (3)    | 0.010 (2)    | -0.009 (2)    | 0.038 (2)    |
| C16 | 0.067 (3)    | 0.071 (3)  | 0.067 (3)    | 0.008 (2)    | -0.006 (2)    | 0.038 (2)    |
| C17 | 0.066 (3)    | 0.071 (3)  | 0.069 (3)    | 0.010 (2)    | -0.008 (2)    | 0.038 (2)    |
| C18 | 0.061 (2)    | 0.057 (2)  | 0.062 (2)    | 0.0009 (18)  | -0.0117 (19)  | 0.0348 (19)  |
| C19 | 0.070 (3)    | 0.076 (3)  | 0.057 (2)    | 0.012 (2)    | -0.007 (2)    | 0.023 (2)    |
| C20 | 0.075 (3)    | 0.080 (3)  | 0.055 (2)    | 0.002 (2)    | -0.010 (2)    | 0.036 (2)    |
| N2  | 0.064 (2)    | 0.086 (3)  | 0.077 (3)    | -0.001 (2)   | -0.0163 (19)  | 0.051 (2)    |
| C21 | 0.066 (3)    | 0.091 (3)  | 0.081 (3)    | 0.020 (3)    | -0.002 (2)    | 0.047 (3)    |
| C22 | 0.064 (3)    | 0.104 (4)  | 0.062 (3)    | 0.016 (3)    | 0.000 (2)     | 0.051 (3)    |

*Geometric parameters (Å, °)*

|        |           |         |           |
|--------|-----------|---------|-----------|
| II—C1  | 2.154 (4) | C9—C10  | 1.376 (6) |
| C1—F1  | 1.330 (5) | C9—C13  | 1.377 (7) |
| C1—F2  | 1.345 (6) | C9—H1B  | 0.9300    |
| C1—C2  | 1.540 (6) | C10—N1  | 1.320 (7) |
| C2—F4  | 1.316 (5) | C10—H10 | 0.9300    |
| C2—F3  | 1.338 (5) | N1—C11  | 1.321 (7) |
| C2—C3  | 1.552 (5) | C11—C12 | 1.376 (7) |
| C3—F5  | 1.290 (5) | C11—H11 | 0.9300    |
| C3—F6  | 1.344 (6) | C12—C13 | 1.390 (6) |
| C3—C4  | 1.545 (6) | C12—H12 | 0.9300    |
| C4—F8  | 1.298 (6) | C13—C14 | 1.436 (6) |
| C4—F7  | 1.315 (5) | C14—C15 | 1.185 (6) |
| C4—C5  | 1.552 (6) | C15—C16 | 1.375 (6) |
| C5—F9  | 1.303 (5) | C16—C17 | 1.204 (6) |
| C5—F10 | 1.312 (6) | C17—C18 | 1.434 (6) |
| C5—C6  | 1.546 (6) | C18—C22 | 1.378 (7) |
| C6—F11 | 1.319 (5) | C18—C19 | 1.389 (6) |
| C6—F12 | 1.336 (5) | C19—C20 | 1.362 (6) |
| C6—C7  | 1.563 (5) | C19—H19 | 0.9300    |
| C7—F13 | 1.322 (5) | C20—N2  | 1.330 (7) |
| C7—F14 | 1.330 (5) | C20—H20 | 0.9300    |
| C7—C8  | 1.536 (6) | N2—C21  | 1.329 (6) |
| C8—F15 | 1.324 (6) | C21—C22 | 1.372 (6) |
| C8—F16 | 1.346 (6) | C21—H21 | 0.9300    |
| C8—I2  | 2.151 (4) | C22—H22 | 0.9300    |

|             |           |               |           |
|-------------|-----------|---------------|-----------|
| F1—C1—F2    | 107.3 (4) | F15—C8—F16    | 107.0 (4) |
| F1—C1—C2    | 109.0 (4) | F15—C8—C7     | 109.4 (4) |
| F2—C1—C2    | 108.1 (4) | F16—C8—C7     | 108.8 (4) |
| F1—C1—I1    | 110.5 (3) | F15—C8—I2     | 109.8 (3) |
| F2—C1—I1    | 108.9 (3) | F16—C8—I2     | 109.3 (3) |
| C2—C1—I1    | 112.9 (3) | C7—C8—I2      | 112.5 (3) |
| F4—C2—F3    | 108.4 (4) | C10—C9—C13    | 118.7 (4) |
| F4—C2—C1    | 108.5 (3) | C10—C9—H1B    | 120.6     |
| F3—C2—C1    | 107.0 (3) | C13—C9—H1B    | 120.6     |
| F4—C2—C3    | 109.1 (3) | N1—C10—C9     | 123.9 (5) |
| F3—C2—C3    | 108.2 (3) | N1—C10—H10    | 118.1     |
| C1—C2—C3    | 115.4 (3) | C9—C10—H10    | 118.1     |
| F5—C3—F6    | 106.3 (4) | C10—N1—C11    | 117.0 (4) |
| F5—C3—C4    | 110.6 (4) | N1—C11—C12    | 124.2 (5) |
| F6—C3—C4    | 107.1 (3) | N1—C11—H11    | 117.9     |
| F5—C3—C2    | 110.1 (4) | C12—C11—H11   | 117.9     |
| F6—C3—C2    | 107.0 (4) | C11—C12—C13   | 118.1 (5) |
| C4—C3—C2    | 115.2 (3) | C11—C12—H12   | 120.9     |
| F8—C4—F7    | 108.2 (5) | C13—C12—H12   | 120.9     |
| F8—C4—C3    | 109.2 (3) | C9—C13—C12    | 118.1 (4) |
| F7—C4—C3    | 107.6 (4) | C9—C13—C14    | 120.8 (4) |
| F8—C4—C5    | 108.6 (4) | C12—C13—C14   | 121.1 (4) |
| F7—C4—C5    | 107.7 (4) | C15—C14—C13   | 178.1 (5) |
| C3—C4—C5    | 115.4 (4) | C14—C15—C16   | 178.8 (6) |
| F9—C5—F10   | 107.2 (5) | C17—C16—C15   | 179.1 (6) |
| F9—C5—C6    | 109.6 (4) | C16—C17—C18   | 177.5 (5) |
| F10—C5—C6   | 108.5 (3) | C22—C18—C19   | 118.6 (4) |
| F9—C5—C4    | 108.2 (4) | C22—C18—C17   | 120.8 (4) |
| F10—C5—C4   | 107.9 (4) | C19—C18—C17   | 120.6 (4) |
| C6—C5—C4    | 115.2 (4) | C20—C19—C18   | 118.1 (5) |
| F11—C6—F12  | 108.1 (4) | C20—C19—H19   | 121.0     |
| F11—C6—C5   | 109.6 (4) | C18—C19—H19   | 121.0     |
| F12—C6—C5   | 107.9 (3) | N2—C20—C19    | 124.4 (4) |
| F11—C6—C7   | 108.4 (3) | N2—C20—H20    | 117.8     |
| F12—C6—C7   | 108.0 (3) | C19—C20—H20   | 117.8     |
| C5—C6—C7    | 114.7 (3) | C21—N2—C20    | 116.6 (4) |
| F13—C7—F14  | 108.4 (4) | N2—C21—C22    | 123.8 (5) |
| F13—C7—C8   | 108.1 (3) | N2—C21—H21    | 118.1     |
| F14—C7—C8   | 107.8 (3) | C22—C21—H21   | 118.1     |
| F13—C7—C6   | 108.2 (3) | C21—C22—C18   | 118.4 (4) |
| F14—C7—C6   | 108.5 (3) | C21—C22—H22   | 120.8     |
| C8—C7—C6    | 115.6 (3) | C18—C22—H22   | 120.8     |
|             |           |               |           |
| F1—C1—C2—F4 | 176.0 (4) | F10—C5—C6—F12 | -62.3 (5) |
| F2—C1—C2—F4 | -67.7 (4) | C4—C5—C6—F12  | 58.8 (5)  |
| I1—C1—C2—F4 | 52.8 (4)  | F9—C5—C6—C7   | -58.7 (5) |
| F1—C1—C2—F3 | 59.2 (5)  | F10—C5—C6—C7  | 58.1 (5)  |



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|               |            |                 |            |
|---------------|------------|-----------------|------------|
| F2—C1—C2—F3   | 175.5 (4)  | C4—C5—C6—C7     | 179.1 (3)  |
| I1—C1—C2—F3   | -64.0 (4)  | F11—C6—C7—F13   | -63.1 (5)  |
| F1—C1—C2—C3   | -61.3 (5)  | F12—C6—C7—F13   | -179.9 (4) |
| F2—C1—C2—C3   | 55.0 (5)   | C5—C6—C7—F13    | 59.8 (5)   |
| I1—C1—C2—C3   | 175.5 (3)  | F11—C6—C7—F14   | 179.6 (4)  |
| F4—C2—C3—F5   | 175.0 (4)  | F12—C6—C7—F14   | 62.7 (5)   |
| F3—C2—C3—F5   | -67.2 (5)  | C5—C6—C7—F14    | -57.6 (5)  |
| C1—C2—C3—F5   | 52.6 (6)   | F11—C6—C7—C8    | 58.3 (5)   |
| F4—C2—C3—F6   | 59.9 (5)   | F12—C6—C7—C8    | -58.5 (5)  |
| F3—C2—C3—F6   | 177.6 (4)  | C5—C6—C7—C8     | -178.8 (4) |
| C1—C2—C3—F6   | -62.6 (5)  | F13—C7—C8—F15   | 63.4 (5)   |
| F4—C2—C3—C4   | -59.1 (5)  | F14—C7—C8—F15   | -179.7 (4) |
| F3—C2—C3—C4   | 58.7 (5)   | C6—C7—C8—F15    | -58.0 (5)  |
| C1—C2—C3—C4   | 178.5 (4)  | F13—C7—C8—F16   | 180.0 (4)  |
| F5—C3—C4—F8   | 178.5 (4)  | F14—C7—C8—F16   | -63.1 (4)  |
| F6—C3—C4—F8   | -66.0 (5)  | C6—C7—C8—F16    | 58.5 (5)   |
| C2—C3—C4—F8   | 52.9 (5)   | F13—C7—C8—I2    | -58.8 (4)  |
| F5—C3—C4—F7   | 61.3 (6)   | F14—C7—C8—I2    | 58.1 (4)   |
| F6—C3—C4—F7   | 176.8 (4)  | C6—C7—C8—I2     | 179.7 (3)  |
| C2—C3—C4—F7   | -64.3 (5)  | C13—C9—C10—N1   | -0.5 (8)   |
| F5—C3—C4—C5   | -58.9 (5)  | C9—C10—N1—C11   | 0.6 (8)    |
| F6—C3—C4—C5   | 56.6 (5)   | C10—N1—C11—C12  | -0.8 (8)   |
| C2—C3—C4—C5   | 175.5 (4)  | N1—C11—C12—C13  | 1.0 (9)    |
| F8—C4—C5—F9   | -179.8 (4) | C10—C9—C13—C12  | 0.6 (7)    |
| F7—C4—C5—F9   | -62.9 (6)  | C10—C9—C13—C14  | 179.8 (5)  |
| C3—C4—C5—F9   | 57.2 (6)   | C11—C12—C13—C9  | -0.8 (7)   |
| F8—C4—C5—F10  | 64.5 (5)   | C11—C12—C13—C14 | 179.9 (5)  |
| F7—C4—C5—F10  | -178.6 (4) | C22—C18—C19—C20 | 0.9 (7)    |
| C3—C4—C5—F10  | -58.5 (5)  | C17—C18—C19—C20 | -179.8 (5) |
| F8—C4—C5—C6   | -56.9 (5)  | C18—C19—C20—N2  | 0.0 (8)    |
| F7—C4—C5—C6   | 60.1 (6)   | C19—C20—N2—C21  | -1.3 (8)   |
| C3—C4—C5—C6   | -179.8 (3) | C20—N2—C21—C22  | 1.7 (8)    |
| F9—C5—C6—F11  | 63.5 (5)   | N2—C21—C22—C18  | -0.8 (9)   |
| F10—C5—C6—F11 | -179.7 (4) | C19—C18—C22—C21 | -0.5 (7)   |
| C4—C5—C6—F11  | -58.7 (5)  | C17—C18—C22—C21 | -179.8 (5) |
| F9—C5—C6—F12  | -179.0 (4) |                 |            |

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