



Crystal structure of tetrabutylammonium bromide–1,2-diiodo-3,4,5,6-tetrafluorobenzene–dichloromethane (2/2/1)

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The crystallization of a 1:1 molar solution of 1,2-diiodo-3,4,5,6-tetrafluorobenzene (*o*-DITFB) and tetrabutylammonium bromide (*n*-Bu₄NBr) from dichloromethane yielded pure white crystals of a halogen-bonded compound, C₁₆H₃₆N⁺·Br⁻·C₆F₄I₂·0.5CH₂Cl₂ or [(*n*-Bu₄NBr)(*o*-DITFB)]·0.5CH₂Cl₂. The compound may be described as a quaternary system and may be classified as a salt–cocrystal solvate. The asymmetric unit contains one molecule of solvent, two *o*-DITFB molecules, two cations (*n*-Bu₄N⁺) and two crystallographically distinct bromide ions [$\theta_{\text{I}\cdots\text{Br}\cdots\text{I}} = 144.18$ (1) and 135.35 (1)°]. The bromide ion is a bidentate halogen-bond acceptor which interacts with two covalently bonded iodines (*i.e.* halogen-bond donors), resulting in a one-dimensional polymeric zigzag chain network approximately along the *a* axis. The observed short contacts and angles are characteristic of the non-covalent interaction [$d_{\text{C}\cdots\text{I}\cdots\text{Br}} = 3.1593$ (4)–3.2590 (5) Å; $\theta_{\text{C}\cdots\text{I}\cdots\text{Br}} = 174.89$ (7) and 178.16 (7)°]. It is noted that iodine acts as both a halogen-bond donor and a weak CH hydrogen-bond acceptor, while the bromide ions act as acceptors for weak CH hydrogen bonds and halogen bonds.

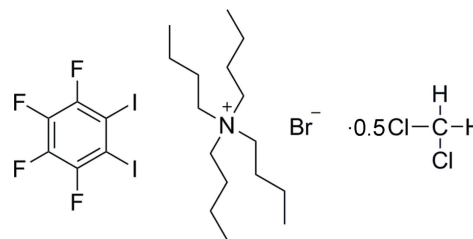
Keywords: crystal structure; halogen bonds; short contacts; noncovalent interaction.

CCDC reference: 1057419

1. Related literature

The halogen-bonding motif of a polymeric anionic zigzag chain has been described for halogen-bonded compounds of phosphonium halides and diiodoperfluorobenzenes, see: Abate *et al.* (2009). For the structure of *o*-DITFB, see: Viger-Gravel (2014) and of *n*-Bu₄NBr, see: Elsegood (2011). The title compound may be classified as a salt–cocrystal solvate,

see: Bond (2012). For weak hydrogen bonds, see: Desiraju & Steiner (1999).



2. Experimental

2.1. Crystal data

2C₁₆H₃₆N⁺·2Br⁻·2C₆F₄I₂·CH₂Cl₂
M_r = 1533.38
 Triclinic, *P* $\bar{1}$
a = 13.1654 (3) Å
b = 15.0483 (3) Å
c = 16.2559 (4) Å
 α = 66.668 (1)°
 β = 84.654 (1)°

γ = 80.842 (1)°
V = 2917.90 (12) Å³
Z = 2
 Mo *K*α radiation
 μ = 3.65 mm⁻¹
T = 200 K
 0.24 × 0.24 × 0.12 mm

2.2. Data collection

Bruker APEXII CCD diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2005)
T_{min} = 0.534, *T_{max}* = 0.746

42707 measured reflections
 14456 independent reflections
 12211 reflections with *I* > 2σ(*I*)
R_{int} = 0.022

2.3. Refinement

$R[F^2 > 2\sigma(F^2)] = 0.024$
 $wR(F^2) = 0.057$
S = 1.02
 14456 reflections

568 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.99 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.99 \text{ e \AA}^{-3}$

Table 1

Contacts below the sum of the van der Waals radii involving DITFB (Å, °).

| C—X···H | X···H | C—X···H |
|------------------------------|-------------|--------------|
| C30—F2···H30B ⁱ | 2.6538 (15) | 120.189 (17) |
| C33—F2···H33A ⁱ | 2.8610 (19) | 170.331 (18) |
| C31—F5···H31B ⁱ | 2.6222 (17) | 140.526 (17) |
| C25—F7···H25B | 2.518 (2) | 172.421 (16) |
| C45—F7···H45B ⁱⁱ | 2.4890 (15) | 149.87 (3) |
| C26—I2···H26A ⁱⁱⁱ | 3.0204 (3) | 115.88 (7) |

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

Table 2

Halogen-bond geometry (Å, °).

| C—X···Y | X···Y | C—X···Y | Y···X···Y | Y···X···Y |
|----------------------------|------------|------------|---------------|--------------|
| C1—I1···Br1 | 3.2582 (5) | 177.15 (8) | I1···Br1···I4 | 144.180 (13) |
| C2—I4···Br1 ^{iv} | 3.1593 (4) | 178.16 (7) | | |
| C1—I2···Br2 ⁱⁱⁱ | 3.2452 (4) | 176.83 (7) | I3···Br2···I2 | 134.350 (12) |
| C2—I3···Br2 ^v | 3.2590 (5) | 174.89 (7) | | |

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

Data collection: *APEX2*, Bruker (2005); cell refinement: *APEX2* and *SAINT* Bruker (2005); data reduction: *SAINT* and *XPREP* Bruker (2005); program(s) used to solve structure: *SHELXS03* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2015); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: GW2150).

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supporting information

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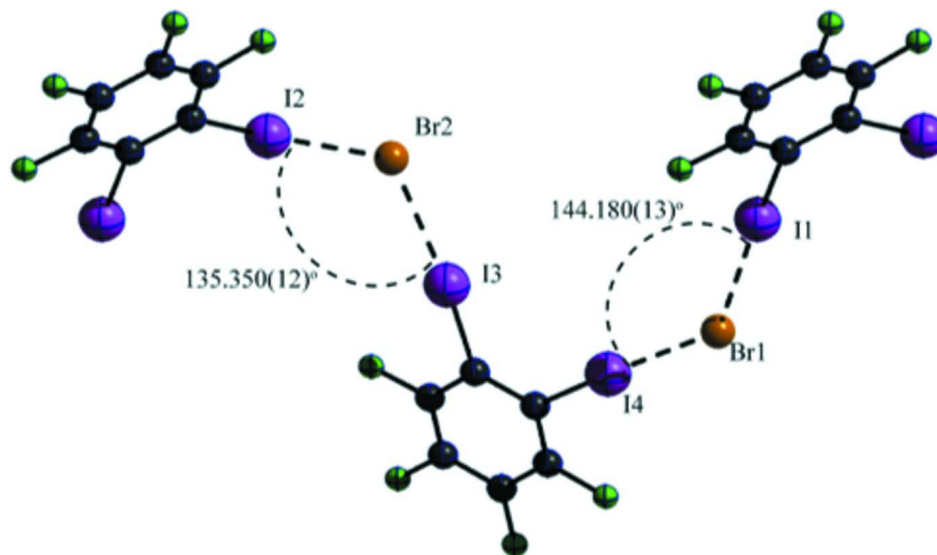
S1. Experimental

Data collection results for [(*n*-Bu₄NBr)(*o*-DITFB)]·CH₂Cl₂ represent the best data sets obtained in several trials. Crystals were mounted on thin glass fibers using paraffin oil. Prior to data collection, crystals were cooled to 200.15 °K. Data were collected on a Bruker AXS SMART single crystal diffractometer equipped with a sealed Mo tube source (wavelength 0.71073 Å) APEX II CCD detector. Raw data collection and processing were performed with the APEX II software package (BRUKER AXS, 2005). Due to lower symmetry in order to ensure adequate data completeness and redundancy, diffraction data were collected with a sequence of 0.3° ω scans at 0, 90, 180, and 270° in φ . Initial unit cell parameters were determined from 60 data frames with 0.3° ω scan each collected at the different sections of the Ewald sphere. Semi-empirical absorption corrections based on equivalent reflections were applied. In structural models for the compound, hydrogen atom positions were located from the differences in Fourier maps. However, after initial positioning, all hydrogen atoms were constrained to suitable geometries and subsequently treated as idealized contributions during the refinement. All scattering factors are contained in several versions of the SHELXTL program library, with the latest version used being v.6.12.

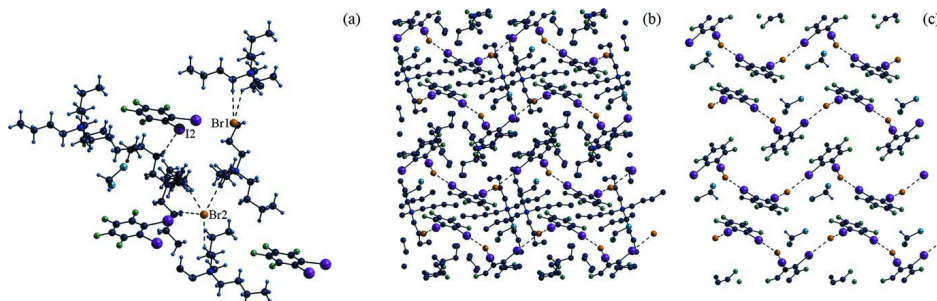
S2. Refinement details

Systematic absences in the diffraction data set and unit-cell parameters were consistent with the triclinic $P\bar{1}$ (No. 2) space group. Solutions in this centrosymmetric space group yielded chemically reasonable and computationally stable results of refinement. The structure was solved by direct methods, completed with difference Fourier synthesis, and refined with full-matrix least-squares procedures based on F^2 . In the structure all molecular fragments are located in general positions.

In the structural model hydrogen atom positions were located from the differences in Fourier maps. However, after initial positioning, all hydrogen atomic positions were constrained to suitable geometries and subsequently treated as idealized contributions. All scattering factors are contained in several versions of the SHELXTL program library, with the latest version used being v.6.12 (Sheldrick, 2008).

**Figure 1**

Halogen bonding environment of $[(n\text{-Bu}_4\text{NBr})(o\text{-DITFB})]\cdot\text{CH}_2\text{Cl}_2$, where iodine is in purple, carbon in black, fluorine in green, and bromine in orange.

**Figure 2**

(a) Detail of crystal structure showing selected weak hydrogen bond contacts to bromide and to iodine. (b, c) $2 \times 2 \times 2$ supercell of $[(n\text{-Bu}_4\text{NBr})(o\text{-DITFB})]\cdot\text{CH}_2\text{Cl}_2$ viewed along the b axis where in (b) the cation is present and in (c) is absent to clarify the image. Rows of the polymeric bromide anionic chains are separated by $n\text{-Bu}_4\text{N}^+$ cations. Hydrogen atoms are not shown for clarity, iodine is in purple, carbon in black, fluorine in green, bromine in orange, and chlorine in blue.

Tetrabutylammonium bromide–1,2-diiodo-3,4,5,6-tetrafluorobenzene–dichloromethane (2/2/1)

Crystal data

$2\text{C}_{16}\text{H}_{36}\text{N}^+\cdot 2\text{Br}^-\cdot 2\text{C}_6\text{F}_4\text{I}_2\cdot\text{CH}_2\text{Cl}_2$

$M_r = 1533.38$

Triclinic, $P\bar{1}$

$a = 13.1654 (3) \text{ \AA}$

$b = 15.0483 (3) \text{ \AA}$

$c = 16.2559 (4) \text{ \AA}$

$\alpha = 66.668 (1)^\circ$

$\beta = 84.654 (1)^\circ$

$\gamma = 80.842 (1)^\circ$

$V = 2917.90 (12) \text{ \AA}^3$

$Z = 2$

$F(000) = 1492$

$D_x = 1.745 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 9968 reflections

$\theta = 2.8\text{--}28.3^\circ$

$\mu = 3.65 \text{ mm}^{-1}$

$T = 200 \text{ K}$

Block, colourless

$0.24 \times 0.24 \times 0.12 \text{ mm}$

Data collection

Bruker APEXII CCD
diffractometer

φ and ω scans

Absorption correction: multi-scan
(SADABS; Bruker, 2009)

$T_{\min} = 0.534$, $T_{\max} = 0.746$

42707 measured reflections

14456 independent reflections

12211 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.022$

$\theta_{\max} = 28.4^\circ$, $\theta_{\min} = 2.0^\circ$

$h = -17 \rightarrow 17$

$k = -20 \rightarrow 20$

$l = -21 \rightarrow 21$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.024$

$wR(F^2) = 0.057$

$S = 1.02$

14456 reflections

568 parameters

0 restraints

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0216P)^2 + 2.2414P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.002$

$\Delta\rho_{\max} = 0.99 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.98 \text{ e } \text{\AA}^{-3}$

Special details

Experimental. Data collection is performed with four batch runs at $\phi = 0.00^\circ$ (600 frames), at $\phi = 90.00^\circ$ (600 frames), at $\phi = 180.00^\circ$ (600 frames), and at $\phi = 270.00^\circ$ (600 frames). A fifth batch run is collected at $\phi = 0.00^\circ$ (50 frames) to monitor crystal and diffractometer stability. Frame width = 0.30° in omega. Data is merged, corrected for decay (if any), and treated with multi-scan absorption corrections (if required). All symmetry-equivalent reflections are merged for centrosymmetric data. Friedel pairs are not merged for noncentrosymmetric data.

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 (\AA^2)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|---------------|---------------|----------------------------------|
| I1 | 0.24735 (2) | 0.72180 (2) | 0.14178 (2) | 0.04521 (5) |
| I2 | 0.01151 (2) | 0.59950 (2) | 0.19674 (2) | 0.03492 (4) |
| I3 | 0.70784 (2) | -0.18215 (2) | 0.37143 (2) | 0.03702 (4) |
| I4 | 0.44346 (2) | -0.05201 (2) | 0.31053 (2) | 0.03407 (4) |
| Br1 | 0.28482 (2) | 0.88557 (2) | 0.21586 (2) | 0.04897 (7) |
| Br2 | 0.81851 (2) | 0.63216 (2) | 0.32589 (2) | 0.03880 (6) |
| F1 | 0.03880 (12) | 0.46121 (11) | 0.09146 (11) | 0.0500 (4) |
| F2 | 0.17634 (14) | 0.43539 (12) | -0.03182 (12) | 0.0566 (4) |
| F3 | 0.34622 (15) | 0.52720 (15) | -0.07434 (13) | 0.0693 (5) |
| F4 | 0.37602 (14) | 0.64816 (16) | 0.00315 (14) | 0.0698 (5) |
| F5 | 0.42205 (13) | 0.12714 (11) | 0.36785 (11) | 0.0526 (4) |
| F6 | 0.55057 (16) | 0.19756 (12) | 0.43805 (12) | 0.0635 (5) |
| F7 | 0.74257 (15) | 0.10137 (13) | 0.48392 (12) | 0.0594 (5) |
| F8 | 0.80628 (12) | -0.06286 (13) | 0.45697 (12) | 0.0572 (4) |
| N1 | 0.91649 (15) | 0.31256 (13) | 0.48925 (12) | 0.0302 (4) |
| N2 | 0.68646 (16) | 0.83381 (15) | 0.02856 (14) | 0.0387 (5) |
| C1 | 0.2196 (2) | 0.62142 (19) | 0.08822 (17) | 0.0392 (5) |
| C2 | 0.13221 (19) | 0.57390 (17) | 0.11037 (16) | 0.0350 (5) |

| | | | | |
|------|--------------|---------------|---------------|-------------|
| C3 | 0.12048 (19) | 0.51035 (18) | 0.07065 (17) | 0.0383 (5) |
| C4 | 0.1907 (2) | 0.49549 (19) | 0.00793 (18) | 0.0429 (6) |
| C5 | 0.2762 (2) | 0.5427 (2) | -0.01434 (19) | 0.0483 (6) |
| C6 | 0.2901 (2) | 0.6044 (2) | 0.02608 (19) | 0.0468 (6) |
| C7 | 0.54822 (19) | -0.00556 (17) | 0.37100 (15) | 0.0329 (5) |
| C8 | 0.64711 (19) | -0.05500 (17) | 0.39466 (15) | 0.0334 (5) |
| C9 | 0.7104 (2) | -0.01755 (19) | 0.43258 (18) | 0.0408 (6) |
| C10 | 0.6785 (2) | 0.06638 (19) | 0.44735 (17) | 0.0438 (6) |
| C11 | 0.5823 (2) | 0.11516 (18) | 0.42431 (17) | 0.0437 (6) |
| C12 | 0.5173 (2) | 0.07841 (17) | 0.38746 (16) | 0.0385 (5) |
| C13 | 0.94820 (19) | 0.21590 (16) | 0.56604 (16) | 0.0336 (5) |
| H13A | 1.0118 | 0.1833 | 0.5469 | 0.040* |
| H13B | 0.8937 | 0.1738 | 0.5766 | 0.040* |
| C14 | 0.9674 (2) | 0.22143 (17) | 0.65406 (17) | 0.0369 (5) |
| H14A | 0.9033 | 0.2498 | 0.6766 | 0.044* |
| H14B | 1.0207 | 0.2644 | 0.6449 | 0.044* |
| C15 | 1.0030 (2) | 0.11948 (17) | 0.72286 (17) | 0.0389 (5) |
| H15A | 0.9492 | 0.0770 | 0.7321 | 0.047* |
| H15B | 1.0663 | 0.0910 | 0.6995 | 0.047* |
| C16 | 1.0244 (2) | 0.1222 (2) | 0.81190 (19) | 0.0517 (7) |
| H16A | 1.0463 | 0.0557 | 0.8546 | 0.077* |
| H16B | 0.9617 | 0.1500 | 0.8354 | 0.077* |
| H16C | 1.0791 | 0.1626 | 0.8033 | 0.077* |
| C17 | 0.81947 (19) | 0.36712 (16) | 0.51409 (16) | 0.0339 (5) |
| H17A | 0.8322 | 0.3769 | 0.5688 | 0.041* |
| H17B | 0.8065 | 0.4325 | 0.4653 | 0.041* |
| C18 | 0.7229 (2) | 0.31889 (19) | 0.53080 (19) | 0.0430 (6) |
| H18A | 0.7310 | 0.2568 | 0.5842 | 0.052* |
| H18B | 0.7115 | 0.3038 | 0.4786 | 0.052* |
| C19 | 0.6312 (2) | 0.3865 (2) | 0.5458 (2) | 0.0548 (7) |
| H19A | 0.6358 | 0.3885 | 0.6054 | 0.066* |
| H19B | 0.6347 | 0.4533 | 0.5002 | 0.066* |
| C20 | 0.5300 (2) | 0.3569 (2) | 0.5408 (3) | 0.0623 (8) |
| H20A | 0.4745 | 0.4035 | 0.5510 | 0.093* |
| H20B | 0.5251 | 0.2915 | 0.5867 | 0.093* |
| H20C | 0.5240 | 0.3564 | 0.4814 | 0.093* |
| C21 | 0.99919 (19) | 0.38009 (17) | 0.46600 (16) | 0.0351 (5) |
| H21A | 0.9698 | 0.4457 | 0.4245 | 0.042* |
| H21B | 1.0157 | 0.3861 | 0.5216 | 0.042* |
| C22 | 1.0985 (2) | 0.34942 (19) | 0.42390 (18) | 0.0403 (6) |
| H22A | 1.1265 | 0.2818 | 0.4621 | 0.048* |
| H22B | 1.0851 | 0.3508 | 0.3644 | 0.048* |
| C23 | 1.1766 (2) | 0.4183 (2) | 0.41343 (18) | 0.0458 (6) |
| H23A | 1.1958 | 0.4106 | 0.4736 | 0.055* |
| H23B | 1.1446 | 0.4865 | 0.3822 | 0.055* |
| C24 | 1.2730 (3) | 0.3988 (3) | 0.3611 (3) | 0.0737 (10) |
| H24A | 1.3207 | 0.4447 | 0.3559 | 0.111* |
| H24B | 1.2546 | 0.4074 | 0.3011 | 0.111* |

| | | | | |
|------|--------------|--------------|---------------|-------------|
| H24C | 1.3060 | 0.3318 | 0.3925 | 0.111* |
| C25 | 0.89861 (19) | 0.28809 (17) | 0.41017 (15) | 0.0336 (5) |
| H25A | 0.9643 | 0.2562 | 0.3934 | 0.040* |
| H25B | 0.8488 | 0.2402 | 0.4293 | 0.040* |
| C26 | 0.8586 (2) | 0.37464 (19) | 0.32801 (17) | 0.0445 (6) |
| H26A | 0.9047 | 0.4255 | 0.3113 | 0.053* |
| H26B | 0.7894 | 0.4028 | 0.3422 | 0.053* |
| C27 | 0.8526 (3) | 0.3456 (2) | 0.24908 (19) | 0.0537 (7) |
| H27A | 0.8369 | 0.4056 | 0.1948 | 0.064* |
| H27B | 0.9209 | 0.3127 | 0.2386 | 0.064* |
| C28 | 0.7739 (4) | 0.2797 (3) | 0.2609 (2) | 0.0872 (14) |
| H28A | 0.7751 | 0.2644 | 0.2075 | 0.131* |
| H28B | 0.7054 | 0.3123 | 0.2693 | 0.131* |
| H28C | 0.7895 | 0.2191 | 0.3136 | 0.131* |
| C29 | 0.6285 (2) | 0.8347 (2) | -0.04838 (18) | 0.0444 (6) |
| H29A | 0.5544 | 0.8540 | -0.0384 | 0.053* |
| H29B | 0.6514 | 0.8860 | -0.1043 | 0.053* |
| C30 | 0.6398 (2) | 0.7404 (2) | -0.06305 (18) | 0.0450 (6) |
| H30A | 0.6202 | 0.6872 | -0.0072 | 0.054* |
| H30B | 0.7124 | 0.7228 | -0.0790 | 0.054* |
| C31 | 0.5710 (2) | 0.7528 (2) | -0.13814 (19) | 0.0513 (7) |
| H31A | 0.4987 | 0.7708 | -0.1215 | 0.062* |
| H31B | 0.5903 | 0.8072 | -0.1932 | 0.062* |
| C32 | 0.5780 (3) | 0.6617 (3) | -0.1580 (2) | 0.0667 (9) |
| H32A | 0.5316 | 0.6739 | -0.2062 | 0.100* |
| H32B | 0.5582 | 0.6076 | -0.1040 | 0.100* |
| H32C | 0.6489 | 0.6447 | -0.1768 | 0.100* |
| C33 | 0.80217 (19) | 0.81006 (19) | 0.01584 (18) | 0.0413 (6) |
| H33A | 0.8166 | 0.7431 | 0.0168 | 0.050* |
| H33B | 0.8360 | 0.8098 | 0.0678 | 0.050* |
| C34 | 0.8511 (2) | 0.8777 (2) | -0.0685 (2) | 0.0566 (8) |
| H34A | 0.8227 | 0.8742 | -0.1213 | 0.068* |
| H34B | 0.8340 | 0.9457 | -0.0723 | 0.068* |
| C35 | 0.9686 (2) | 0.8509 (2) | -0.0705 (3) | 0.0621 (8) |
| H35A | 0.9985 | 0.9031 | -0.1214 | 0.075* |
| H35B | 0.9956 | 0.8485 | -0.0146 | 0.075* |
| C36 | 1.0036 (3) | 0.7566 (3) | -0.0790 (2) | 0.0651 (9) |
| H36A | 1.0789 | 0.7439 | -0.0787 | 0.098* |
| H36B | 0.9800 | 0.7591 | -0.1355 | 0.098* |
| H36C | 0.9749 | 0.7042 | -0.0287 | 0.098* |
| C37 | 0.6580 (2) | 0.93582 (19) | 0.02917 (18) | 0.0426 (6) |
| H37A | 0.6677 | 0.9840 | -0.0326 | 0.051* |
| H37B | 0.5839 | 0.9442 | 0.0458 | 0.051* |
| C38 | 0.7171 (2) | 0.9601 (2) | 0.0910 (2) | 0.0497 (7) |
| H38A | 0.7891 | 0.9661 | 0.0679 | 0.060* |
| H38B | 0.7184 | 0.9069 | 0.1513 | 0.060* |
| C39 | 0.6675 (2) | 1.0547 (2) | 0.0975 (2) | 0.0508 (7) |
| H39A | 0.6566 | 1.1049 | 0.0362 | 0.061* |

| | | | | |
|------|-------------|-------------|--------------|-------------|
| H39B | 0.5991 | 1.0451 | 0.1281 | 0.061* |
| C40 | 0.7296 (3) | 1.0921 (3) | 0.1474 (3) | 0.0656 (9) |
| H40A | 0.6921 | 1.1525 | 0.1505 | 0.098* |
| H40B | 0.7961 | 1.1053 | 0.1158 | 0.098* |
| H40C | 0.7409 | 1.0429 | 0.2082 | 0.098* |
| C41 | 0.6578 (2) | 0.7557 (2) | 0.11715 (17) | 0.0447 (6) |
| H41A | 0.6830 | 0.6911 | 0.1149 | 0.054* |
| H41B | 0.6947 | 0.7609 | 0.1651 | 0.054* |
| C42 | 0.5454 (2) | 0.7588 (3) | 0.1429 (2) | 0.0557 (8) |
| H42A | 0.5070 | 0.7522 | 0.0965 | 0.067* |
| H42B | 0.5187 | 0.8224 | 0.1469 | 0.067* |
| C43 | 0.5291 (3) | 0.6755 (3) | 0.2335 (2) | 0.0658 (9) |
| H43A | 0.5746 | 0.6788 | 0.2772 | 0.079* |
| H43B | 0.4572 | 0.6863 | 0.2549 | 0.079* |
| C44 | 0.5493 (4) | 0.5770 (3) | 0.2329 (3) | 0.1005 (15) |
| H44A | 0.5379 | 0.5288 | 0.2936 | 0.151* |
| H44B | 0.6207 | 0.5647 | 0.2129 | 0.151* |
| H44C | 0.5026 | 0.5717 | 0.1920 | 0.151* |
| C45 | 0.8462 (3) | 0.9249 (3) | 0.6717 (3) | 0.0774 (11) |
| H45A | 0.8378 | 0.9523 | 0.7185 | 0.093* |
| H45B | 0.7938 | 0.9625 | 0.6264 | 0.093* |
| Cl1 | 0.96601 (9) | 0.93975 (8) | 0.62159 (9) | 0.0951 (4) |
| Cl2 | 0.82419 (9) | 0.80409 (9) | 0.71994 (8) | 0.0888 (3) |

Atomic displacement parameters (Å²)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|--------------|--------------|--------------|---------------|---------------|---------------|
| I1 | 0.03980 (9) | 0.05145 (10) | 0.05094 (10) | -0.01695 (7) | 0.00179 (7) | -0.02342 (8) |
| I2 | 0.03671 (8) | 0.03464 (8) | 0.03288 (8) | -0.00851 (6) | 0.00126 (6) | -0.01166 (6) |
| I3 | 0.03450 (8) | 0.03702 (8) | 0.04262 (9) | -0.00278 (6) | -0.00354 (6) | -0.01896 (7) |
| I4 | 0.03396 (8) | 0.03321 (7) | 0.03537 (8) | -0.00373 (6) | -0.00246 (6) | -0.01365 (6) |
| Br1 | 0.04762 (15) | 0.04415 (14) | 0.05824 (17) | -0.00928 (12) | -0.01439 (13) | -0.01921 (13) |
| Br2 | 0.04265 (14) | 0.03591 (12) | 0.03926 (13) | -0.00107 (10) | -0.00096 (10) | -0.01773 (10) |
| F1 | 0.0502 (9) | 0.0472 (9) | 0.0607 (10) | -0.0161 (7) | 0.0022 (8) | -0.0267 (8) |
| F2 | 0.0659 (11) | 0.0518 (9) | 0.0629 (11) | 0.0038 (8) | -0.0069 (9) | -0.0369 (9) |
| F3 | 0.0623 (12) | 0.0857 (14) | 0.0656 (12) | -0.0023 (10) | 0.0197 (9) | -0.0428 (11) |
| F4 | 0.0482 (10) | 0.0924 (14) | 0.0811 (13) | -0.0296 (10) | 0.0273 (9) | -0.0455 (12) |
| F5 | 0.0615 (10) | 0.0444 (8) | 0.0529 (10) | 0.0109 (7) | -0.0088 (8) | -0.0251 (7) |
| F6 | 0.1002 (15) | 0.0406 (9) | 0.0601 (11) | -0.0073 (9) | -0.0051 (10) | -0.0307 (8) |
| F7 | 0.0777 (12) | 0.0600 (10) | 0.0569 (10) | -0.0328 (9) | -0.0055 (9) | -0.0305 (9) |
| F8 | 0.0427 (9) | 0.0709 (11) | 0.0721 (12) | -0.0089 (8) | -0.0122 (8) | -0.0397 (10) |
| N1 | 0.0384 (11) | 0.0249 (8) | 0.0298 (10) | -0.0065 (8) | 0.0003 (8) | -0.0128 (7) |
| N2 | 0.0352 (11) | 0.0435 (11) | 0.0329 (11) | 0.0073 (9) | -0.0077 (8) | -0.0135 (9) |
| C1 | 0.0386 (13) | 0.0418 (13) | 0.0379 (13) | -0.0072 (10) | -0.0002 (10) | -0.0156 (11) |
| C2 | 0.0347 (12) | 0.0338 (11) | 0.0327 (12) | -0.0040 (9) | 0.0000 (10) | -0.0094 (10) |
| C3 | 0.0371 (13) | 0.0334 (12) | 0.0420 (14) | -0.0015 (10) | -0.0053 (11) | -0.0126 (10) |
| C4 | 0.0492 (15) | 0.0374 (13) | 0.0424 (14) | 0.0044 (11) | -0.0077 (12) | -0.0182 (11) |
| C5 | 0.0466 (16) | 0.0520 (16) | 0.0424 (15) | 0.0030 (12) | 0.0055 (12) | -0.0194 (13) |

| | | | | | | |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| C6 | 0.0385 (14) | 0.0522 (16) | 0.0479 (16) | -0.0098 (12) | 0.0067 (12) | -0.0180 (13) |
| C7 | 0.0398 (13) | 0.0323 (11) | 0.0285 (11) | -0.0077 (10) | 0.0013 (9) | -0.0133 (9) |
| C8 | 0.0393 (13) | 0.0325 (11) | 0.0315 (12) | -0.0092 (10) | 0.0016 (10) | -0.0148 (9) |
| C9 | 0.0413 (14) | 0.0446 (14) | 0.0391 (13) | -0.0116 (11) | 0.0003 (11) | -0.0172 (11) |
| C10 | 0.0611 (17) | 0.0429 (14) | 0.0353 (13) | -0.0238 (13) | 0.0012 (12) | -0.0179 (11) |
| C11 | 0.0699 (19) | 0.0314 (12) | 0.0328 (13) | -0.0113 (12) | 0.0031 (12) | -0.0151 (10) |
| C12 | 0.0497 (15) | 0.0326 (12) | 0.0309 (12) | -0.0029 (11) | 0.0011 (10) | -0.0115 (10) |
| C13 | 0.0415 (13) | 0.0232 (10) | 0.0360 (12) | -0.0028 (9) | -0.0038 (10) | -0.0114 (9) |
| C14 | 0.0420 (13) | 0.0288 (11) | 0.0397 (13) | -0.0023 (10) | -0.0081 (11) | -0.0126 (10) |
| C15 | 0.0375 (13) | 0.0316 (11) | 0.0444 (14) | -0.0035 (10) | -0.0056 (11) | -0.0109 (10) |
| C16 | 0.0633 (19) | 0.0399 (14) | 0.0478 (16) | 0.0004 (13) | -0.0224 (14) | -0.0111 (12) |
| C17 | 0.0430 (13) | 0.0269 (10) | 0.0333 (12) | -0.0016 (9) | 0.0005 (10) | -0.0146 (9) |
| C18 | 0.0451 (15) | 0.0359 (12) | 0.0485 (15) | -0.0038 (11) | 0.0050 (12) | -0.0188 (11) |
| C19 | 0.0519 (17) | 0.0463 (15) | 0.0639 (19) | -0.0016 (13) | 0.0139 (14) | -0.0242 (14) |
| C20 | 0.0519 (18) | 0.0566 (18) | 0.076 (2) | 0.0023 (14) | 0.0054 (16) | -0.0281 (17) |
| C21 | 0.0461 (14) | 0.0296 (11) | 0.0352 (12) | -0.0133 (10) | 0.0016 (10) | -0.0161 (10) |
| C22 | 0.0419 (14) | 0.0409 (13) | 0.0433 (14) | -0.0118 (11) | -0.0003 (11) | -0.0194 (11) |
| C23 | 0.0490 (16) | 0.0524 (15) | 0.0399 (14) | -0.0205 (13) | -0.0037 (12) | -0.0166 (12) |
| C24 | 0.056 (2) | 0.100 (3) | 0.074 (2) | -0.034 (2) | 0.0123 (17) | -0.038 (2) |
| C25 | 0.0423 (13) | 0.0328 (11) | 0.0325 (12) | -0.0110 (10) | 0.0009 (10) | -0.0181 (10) |
| C26 | 0.0639 (18) | 0.0395 (13) | 0.0329 (13) | -0.0169 (12) | -0.0028 (12) | -0.0131 (11) |
| C27 | 0.072 (2) | 0.0612 (18) | 0.0331 (14) | -0.0214 (16) | 0.0030 (13) | -0.0207 (13) |
| C28 | 0.132 (4) | 0.093 (3) | 0.053 (2) | -0.062 (3) | -0.013 (2) | -0.026 (2) |
| C29 | 0.0418 (14) | 0.0528 (15) | 0.0347 (13) | 0.0069 (12) | -0.0113 (11) | -0.0157 (12) |
| C30 | 0.0450 (15) | 0.0517 (15) | 0.0356 (13) | 0.0007 (12) | -0.0046 (11) | -0.0161 (12) |
| C31 | 0.0551 (17) | 0.0572 (17) | 0.0408 (15) | -0.0060 (14) | -0.0102 (13) | -0.0169 (13) |
| C32 | 0.075 (2) | 0.075 (2) | 0.062 (2) | -0.0110 (18) | -0.0065 (17) | -0.0371 (18) |
| C33 | 0.0327 (13) | 0.0423 (13) | 0.0429 (14) | 0.0074 (10) | -0.0051 (11) | -0.0140 (11) |
| C34 | 0.0508 (17) | 0.0497 (16) | 0.0546 (18) | 0.0076 (13) | 0.0063 (14) | -0.0119 (14) |
| C35 | 0.0478 (17) | 0.0564 (18) | 0.076 (2) | -0.0086 (14) | 0.0156 (16) | -0.0225 (17) |
| C36 | 0.056 (2) | 0.068 (2) | 0.063 (2) | 0.0088 (16) | 0.0014 (16) | -0.0234 (17) |
| C37 | 0.0373 (13) | 0.0464 (14) | 0.0414 (14) | 0.0091 (11) | -0.0077 (11) | -0.0183 (12) |
| C38 | 0.0478 (16) | 0.0523 (16) | 0.0492 (16) | 0.0066 (13) | -0.0120 (13) | -0.0226 (13) |
| C39 | 0.0463 (16) | 0.0487 (15) | 0.0551 (17) | -0.0009 (12) | 0.0016 (13) | -0.0206 (13) |
| C40 | 0.066 (2) | 0.0583 (19) | 0.080 (2) | -0.0045 (16) | -0.0077 (18) | -0.0354 (18) |
| C41 | 0.0383 (14) | 0.0573 (16) | 0.0324 (13) | -0.0026 (12) | -0.0042 (10) | -0.0119 (12) |
| C42 | 0.0429 (16) | 0.074 (2) | 0.0429 (16) | -0.0010 (14) | 0.0027 (12) | -0.0188 (15) |
| C43 | 0.0457 (18) | 0.102 (3) | 0.0432 (17) | -0.0143 (18) | 0.0068 (13) | -0.0208 (18) |
| C44 | 0.089 (3) | 0.089 (3) | 0.095 (3) | -0.019 (2) | 0.030 (3) | -0.010 (3) |
| C45 | 0.096 (3) | 0.073 (2) | 0.057 (2) | 0.016 (2) | 0.0007 (19) | -0.0295 (18) |
| Cl1 | 0.0980 (8) | 0.0768 (6) | 0.1320 (10) | -0.0225 (6) | 0.0321 (7) | -0.0670 (7) |
| Cl2 | 0.0813 (7) | 0.1009 (8) | 0.1077 (8) | -0.0284 (6) | 0.0117 (6) | -0.0625 (7) |

Geometric parameters (Å, °)

| | | | |
|-------|-----------|---------|-----------|
| I1—C1 | 2.109 (3) | C9—C10 | 1.372 (4) |
| I2—C2 | 2.112 (2) | C10—C11 | 1.362 (4) |
| I3—C8 | 2.115 (2) | C11—C12 | 1.381 (4) |

| | | | |
|------------|-------------|-------------|-------------|
| I4—C7 | 2.112 (2) | C13—C14 | 1.512 (3) |
| F1—C3 | 1.344 (3) | C14—C15 | 1.528 (3) |
| F2—C4 | 1.346 (3) | C15—C16 | 1.517 (4) |
| F3—C5 | 1.342 (3) | C17—C18 | 1.513 (4) |
| F4—C6 | 1.348 (3) | C18—C19 | 1.517 (4) |
| F5—C12 | 1.345 (3) | C19—C20 | 1.489 (4) |
| F6—C11 | 1.341 (3) | C21—C22 | 1.510 (4) |
| F7—C10 | 1.351 (3) | C22—C23 | 1.526 (3) |
| F8—C9 | 1.349 (3) | C23—C24 | 1.515 (4) |
| N1—C17 | 1.517 (3) | C25—C26 | 1.514 (3) |
| N1—C13 | 1.521 (3) | C26—C27 | 1.522 (4) |
| N1—C25 | 1.517 (3) | C27—C28 | 1.495 (4) |
| N1—C21 | 1.527 (3) | C29—C30 | 1.512 (4) |
| N2—C33 | 1.522 (3) | C30—C31 | 1.522 (4) |
| N2—C29 | 1.520 (3) | C31—C32 | 1.515 (4) |
| N2—C41 | 1.516 (3) | C33—C34 | 1.507 (4) |
| N2—C37 | 1.525 (3) | C34—C35 | 1.537 (4) |
| C1—C6 | 1.379 (4) | C35—C36 | 1.477 (5) |
| C1—C2 | 1.395 (3) | C37—C38 | 1.509 (4) |
| C2—C3 | 1.382 (3) | C38—C39 | 1.508 (4) |
| C3—C4 | 1.372 (4) | C39—C40 | 1.508 (4) |
| C4—C5 | 1.371 (4) | C41—C42 | 1.499 (4) |
| C5—C6 | 1.373 (4) | C42—C43 | 1.534 (4) |
| C7—C12 | 1.385 (3) | C43—C44 | 1.468 (6) |
| C7—C8 | 1.398 (3) | C45—C12 | 1.733 (4) |
| C8—C9 | 1.383 (3) | C45—C11 | 1.711 (4) |
| C17—N1—C13 | 111.15 (18) | F8—C9—C8 | 121.0 (2) |
| C17—N1—C25 | 110.59 (18) | C10—C9—C8 | 121.7 (3) |
| C13—N1—C25 | 106.52 (16) | C11—C10—C9 | 120.0 (2) |
| C17—N1—C21 | 106.23 (17) | C11—C10—F7 | 119.9 (2) |
| C13—N1—C21 | 111.40 (18) | C9—C10—F7 | 120.1 (3) |
| C25—N1—C21 | 111.01 (17) | F6—C11—C10 | 120.5 (2) |
| C33—N2—C29 | 111.5 (2) | F6—C11—C12 | 120.2 (3) |
| C33—N2—C41 | 105.38 (18) | C10—C11—C12 | 119.3 (2) |
| C29—N2—C41 | 111.8 (2) | F5—C12—C7 | 120.7 (2) |
| C33—N2—C37 | 111.4 (2) | F5—C12—C11 | 117.6 (2) |
| C29—N2—C37 | 105.28 (18) | C7—C12—C11 | 121.7 (2) |
| C41—N2—C37 | 111.6 (2) | N1—C13—C14 | 116.31 (18) |
| C6—C1—C2 | 118.5 (2) | C13—C14—C15 | 110.04 (19) |
| C6—C1—I1 | 118.13 (19) | C16—C15—C14 | 111.6 (2) |
| C2—C1—I1 | 123.34 (19) | C18—C17—N1 | 116.13 (18) |
| C3—C2—C1 | 118.9 (2) | C17—C18—C19 | 109.8 (2) |
| C3—C2—I2 | 117.59 (18) | C20—C19—C18 | 113.7 (3) |
| C1—C2—I2 | 123.40 (18) | C22—C21—N1 | 116.31 (18) |
| F1—C3—C2 | 121.0 (2) | C23—C22—C21 | 110.0 (2) |
| F1—C3—C4 | 117.2 (2) | C22—C23—C24 | 112.3 (3) |
| C2—C3—C4 | 121.7 (2) | C26—C25—N1 | 114.90 (19) |

| | | | |
|--------------|--------------|-----------------|------------|
| F2—C4—C5 | 119.9 (2) | C25—C26—C27 | 111.6 (2) |
| F2—C4—C3 | 120.8 (3) | C28—C27—C26 | 114.6 (3) |
| C5—C4—C3 | 119.3 (2) | C30—C29—N2 | 116.9 (2) |
| F3—C5—C4 | 119.8 (3) | C29—C30—C31 | 109.6 (2) |
| F3—C5—C6 | 120.7 (3) | C32—C31—C30 | 113.3 (3) |
| C4—C5—C6 | 119.6 (3) | N2—C33—C34 | 116.3 (2) |
| F4—C6—C5 | 117.6 (3) | C35—C34—C33 | 111.4 (2) |
| F4—C6—C1 | 120.5 (3) | C36—C35—C34 | 114.1 (3) |
| C5—C6—C1 | 121.9 (3) | C38—C37—N2 | 116.5 (2) |
| C12—C7—C8 | 118.6 (2) | C37—C38—C39 | 110.2 (2) |
| C12—C7—I4 | 118.16 (18) | C40—C39—C38 | 113.8 (3) |
| C8—C7—I4 | 123.29 (17) | C42—C41—N2 | 116.1 (2) |
| C9—C8—C7 | 118.7 (2) | C41—C42—C43 | 109.7 (2) |
| C9—C8—I3 | 117.76 (19) | C44—C43—C42 | 115.3 (3) |
| C7—C8—I3 | 123.47 (16) | C12—C45—C11 | 113.4 (2) |
| F8—C9—C10 | 117.4 (2) | | |
| C6—C1—C2—C3 | 1.2 (4) | F6—C11—C12—F5 | -1.1 (4) |
| I1—C1—C2—C3 | 179.13 (18) | C10—C11—C12—F5 | 178.2 (2) |
| C6—C1—C2—I2 | -175.3 (2) | F6—C11—C12—C7 | 178.9 (2) |
| I1—C1—C2—I2 | 2.6 (3) | C10—C11—C12—C7 | -1.8 (4) |
| C1—C2—C3—F1 | 178.4 (2) | C17—N1—C13—C14 | -57.7 (3) |
| I2—C2—C3—F1 | -4.8 (3) | C25—N1—C13—C14 | -178.2 (2) |
| C1—C2—C3—C4 | -2.3 (4) | C21—N1—C13—C14 | 60.6 (3) |
| I2—C2—C3—C4 | 174.47 (19) | N1—C13—C14—C15 | -177.5 (2) |
| F1—C3—C4—F2 | 1.2 (4) | C13—C14—C15—C16 | 179.3 (2) |
| C2—C3—C4—F2 | -178.1 (2) | C13—N1—C17—C18 | -64.7 (3) |
| F1—C3—C4—C5 | -178.9 (2) | C25—N1—C17—C18 | 53.4 (3) |
| C2—C3—C4—C5 | 1.8 (4) | C21—N1—C17—C18 | 173.9 (2) |
| F2—C4—C5—F3 | -1.4 (4) | N1—C17—C18—C19 | -174.5 (2) |
| C3—C4—C5—F3 | 178.7 (2) | C17—C18—C19—C20 | 166.0 (3) |
| F2—C4—C5—C6 | 179.6 (2) | C17—N1—C21—C22 | -170.0 (2) |
| C3—C4—C5—C6 | -0.3 (4) | C13—N1—C21—C22 | 68.8 (3) |
| F3—C5—C6—F4 | 0.0 (4) | C25—N1—C21—C22 | -49.7 (3) |
| C4—C5—C6—F4 | 179.0 (3) | N1—C21—C22—C23 | -174.3 (2) |
| F3—C5—C6—C1 | -179.8 (3) | C21—C22—C23—C24 | -172.9 (3) |
| C4—C5—C6—C1 | -0.8 (4) | C17—N1—C25—C26 | 54.9 (3) |
| C2—C1—C6—F4 | -179.5 (2) | C13—N1—C25—C26 | 175.8 (2) |
| I1—C1—C6—F4 | 2.4 (4) | C21—N1—C25—C26 | -62.8 (3) |
| C2—C1—C6—C5 | 0.3 (4) | N1—C25—C26—C27 | 174.5 (2) |
| I1—C1—C6—C5 | -177.8 (2) | C25—C26—C27—C28 | 67.3 (4) |
| C12—C7—C8—C9 | -0.7 (3) | C33—N2—C29—C30 | 61.0 (3) |
| I4—C7—C8—C9 | 178.92 (18) | C41—N2—C29—C30 | -56.7 (3) |
| C12—C7—C8—I3 | -178.46 (17) | C37—N2—C29—C30 | -178.1 (2) |
| I4—C7—C8—I3 | 1.2 (3) | N2—C29—C30—C31 | 176.1 (2) |
| C7—C8—C9—F8 | 179.7 (2) | C29—C30—C31—C32 | 179.5 (3) |
| I3—C8—C9—F8 | -2.5 (3) | C29—N2—C33—C34 | 58.5 (3) |
| C7—C8—C9—C10 | 0.0 (4) | C41—N2—C33—C34 | -180.0 (2) |

| | | | |
|----------------|--------------|-----------------|------------|
| I3—C8—C9—C10 | 177.9 (2) | C37—N2—C33—C34 | -58.8 (3) |
| F8—C9—C10—C11 | -179.8 (2) | N2—C33—C34—C35 | 175.8 (3) |
| C8—C9—C10—C11 | -0.2 (4) | C33—C34—C35—C36 | 67.8 (4) |
| F8—C9—C10—F7 | 0.8 (4) | C33—N2—C37—C38 | -50.4 (3) |
| C8—C9—C10—F7 | -179.5 (2) | C29—N2—C37—C38 | -171.4 (2) |
| C9—C10—C11—F6 | -179.7 (2) | C41—N2—C37—C38 | 67.1 (3) |
| F7—C10—C11—F6 | -0.3 (4) | N2—C37—C38—C39 | -169.2 (2) |
| C9—C10—C11—C12 | 1.0 (4) | C37—C38—C39—C40 | -171.8 (3) |
| F7—C10—C11—C12 | -179.6 (2) | C33—N2—C41—C42 | -176.2 (3) |
| C8—C7—C12—F5 | -178.4 (2) | C29—N2—C41—C42 | -54.9 (3) |
| I4—C7—C12—F5 | 2.0 (3) | C37—N2—C41—C42 | 62.7 (3) |
| C8—C7—C12—C11 | 1.6 (4) | N2—C41—C42—C43 | 180.0 (3) |
| I4—C7—C12—C11 | -178.04 (19) | C41—C42—C43—C44 | -69.2 (4) |
