Halogen bonding in a series of Br(CF₂)_nBr-DABCO adducts (n = 4, 6, 8)

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Halogen bonding in a series of $\text{Br}(\text{CF}_2)_n\text{Br}$ -DABCO adducts ($n = 4, 6, 8$).

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Abstract

Halogen bonding (XB) is a highly-directional class of intermolecular interactions that has been used as a powerful tool to drive the design of crystals in the solid phase. To date the majority of the XB-donors have been iodine-containing compounds, with many fewer involving brominated analogues. We report the formation of adducts in the vapour phase from a series of dibromo-perfluoroalkyl compounds $\text{BrCF}_2(\text{CF}_2)_n\text{CF}_2\text{Br}$ ($n=2, 4, 6$) and 1,4-diazabicyclo[2.2.2]octane (DABCO). Single crystal X-ray diffraction studies of the colourless crystals identified $\text{Br}_2\text{C}_4\text{F}_8 \cdot \text{C}_6\text{H}_{12}\text{N}_2$ (I), $\text{Br}_2\text{C}_6\text{F}_{12} \cdot \text{C}_6\text{H}_{12}\text{N}_2$ (II) and $\text{Br}_2\text{C}_8\text{F}_{16} \cdot \text{C}_6\text{H}_{12}\text{N}_2$ (III), each of which display one-dimensional halogen-bonded networks. All three adducts exhibit $\text{N} \cdots \text{Br}$ distances less than the sum of the van der Waals radii, with $\text{Br}_2\text{C}_4\text{F}_8 \cdot \text{C}_6\text{H}_{12}\text{N}_2$ showing the shortest $\text{N} \cdots \text{Br}$ halogen bond distances yet reported between a bromoperfluorocarbon and a nitrogen base [2.809 (3) and 2.818 (3) Å], which are 0.58 and 0.59 Å shorter than the sum of the van der Waals radii.

Keywords: crystal structure, halogen bonding, dibromoperfluoroalkanes, DABCO, adducts

99scheme1.tif

1. Introduction

The attractive, halogen bonding interactions between a halogen atom and a Lewis base, such as an amine, has been known for nearly 150 years (Guthrie, 1863; Mulliken, 1950; Mulliken, 1952; Mulliken, 1952; Hassel and Hvoslef, 1954; Hassel, 1970). It has been rationalized as an $n \rightarrow \sigma^*$ donation process (Forster, 1969) and recognized as being similar in strength to the more widely-known hydrogen bonding interaction (Steiner, 2002; Prins *et al.*, 2001). On bonding a

halogen atom, X , to a carbon framework, and especially if the fragment is electron withdrawing, distortion of the halogen electron distribution occurs resulting in the formation of an area of reduced electron density referred to as a σ -hole with which a Lewis base (B) may interact. Because the depletion occurs on the opposite side of the halogen (C— X) bond, halogen bond interactions are highly directional ($150^\circ < \text{C—}X\cdots\text{B} < 180^\circ$) and most commonly associated with compounds of the most readily polarized halogens, *i.e.* iodine.

The majority of halogen-bonded co-crystals have therefore been assembled using $\text{N}\cdots\text{I}$ interactions between iodo-PFCs donors (PFCs = perfluorocarbons) such as diiodoperfluoroalkanes and –arenes and nitrogen acceptors, such as aliphatic or aromatic amines and pyridine derivatives (Cardillo *et al.*, 2000; Messina *et al.*, 2001; Corradi *et al.*, 1999; Farina *et al.*, 1999). In this way the rational design of a wide range of halogen bonded adducts has been undertaken, leading to applications in fields ranging from crystal engineering (Desiraju, 2010; Cavallo *et al.*, 2010) to converting volatile molecules into more easily handled solid adducts (Aakeröy *et al.*, 2015).

By comparison with the number of iodo-perfluorocarbon derived structures, relatively few co-crystals with bromo-perfluorocarbons have been reported. A search of the Cambridge Structural Database (CSD Version 5.38, Groom *et al.*, 2014) using Conquest (Bruno *et al.*, 2002) shows that there are nearly 400 reported single-crystal structures featuring near-linear ($150 < \text{N}\cdots\text{I—C} < 180$) $\text{N}\cdots\text{I}$ contacts less than the sum of the van der Waals radii derived from an iodo-fluorocarbon and a nitrogen base. Of these 87 involve fluoroalkyl or fluoroalkenyl systems and 299 are based on iodo-fluoroaromatic compounds. By contrast the analogous search of $\text{N}\cdots\text{Br}$ systems reveals just 42 hits of which 39 are based on bromo-fluoroaromatic systems, and just 3 involve bromo-fluoroalkanes.

One of the three previously reported bromo-fluoroalkyl systems is the adduct obtained by the vapour-phase diffusion of 1,4-diazabicyclo[2.2.2]octane (DABCO) with dibromotetrafluoroethane which results in trapping the volatile, environmentally harmful, ozone-depleting bromo-fluorocarbon, $\text{C}_2\text{F}_4\text{Br}_2$, as a solid 1:1 adduct (Brisdon *et al.*, 2015). The crystals displayed infinite one-dimensional chains linked by $\text{N}\cdots\text{Br}$ halogen bonds ($d = 2.829(3) \text{ \AA}$), which are substantially shorter than the sum of the van der Waals radii of the involved nuclei ($1.55 \text{ \AA}$ for nitrogen and $1.85 \text{ \AA}$ for bromine, sum = $3.40 \text{ \AA}$, (Bondi, 1964)). Given the limited amount of data in the area of halogen bonded bromo-fluoroalkyl amine adducts, in this paper we report the results of a systematic investigation of the crystal structures of the halogen bonded adducts of 1,4-diazabicyclo[2.2.2]octane (DABCO) with 1,4-dibromoperfluorobutane, 1,6-dibromoperfluorohexane and 1,8-dibromoperfluorooctane.

2. Experimental

$\text{Br}(\text{CF}_2)_4\text{Br}$, $\text{Br}(\text{CF}_2)_6\text{Br}$, $\text{Br}(\text{CF}_2)_8\text{Br}$ and 1,4-diazabicyclo[2.2.2]octane (DABCO) were obtained from commercial sources and were used without further purification.

2.1. Synthesis and crystallization

Preparation of the title compounds was by vapour diffusion in a sealed system consisting of two concentric glass vials. DABCO (0.1 g) was placed in the smaller, inner vial with the appropriate dibromoperfluorocarbon (0.5 ml for I and II, and 0.1 g for III) in the outer vial. Crystals suitable for X-ray diffraction studies formed within 24 h on the walls of the inner vial at room temperature. The crystals were subjected to single-crystal X-ray analysis (see below) and IR spectroscopy: Adduct (I) formed between DABCO and 1,4-dibromoperfluorobutane: IR: $\nu(\text{cm}^{-1})$ 2936, 2872 (C—H), 1190, 1129 (C—F). Adduct (II) formed from DABCO with 1,6-dibromoperfluorohexane: IR: $\nu(\text{cm}^{-1})$ 2936, 2873 (C—H), 1201, 1140 (C—F). Adduct (III) formed from DABCO and 1,8-dibromoperfluorooctane: IR: $\nu(\text{cm}^{-1})$ 2937, 2873 (C—H), 1204, 1146 (C—F).

2.2. Refinement

Suitable crystals of (I), (II) and (III) were selected and mounted on SuperNova Eos or Xcalibur Atlas diffractometers employing Mo K_α ($\lambda = 0.71073$ Å) radiation. The crystals were kept at low temperature (100, or 150 K) during data collection. Crystal, data collection and structure refinement details are summarized in Table 1. Non-hydrogen atoms were refined anisotropically; hydrogen atoms were treated as riding atoms with $d(\text{C—H}) = 0.97$ Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. Using Olex2 (Dolomanov *et al.*, 2009), the structure of adduct (I) was solved with *SHELXS* (Sheldrick, 2008) and refined with *SHELXL* (Sheldrick, 2015) using Least Squares minimization. While for adduct (II) solution of the data used *ShelXT* (Sheldrick, 2015) and *SHELXL*, and for adduct (III) a solution was obtained using *Superflip* (Palatinus & Chapuis, 2007; Palatinus & van der Lee, 2008; Palatinus *et al.*, 2012) prior to refinement with *SHELXL*. For adduct (II) two reflections were omitted from the final refinement cycle.

3. Results and discussion

Adduct (I) crystallizes in the $P2_1/c$ space group, and the asymmetric unit consists of one molecule of DABCO and one molecule of $\text{Br}(\text{CF}_2)_4\text{Br}$ (Fig. 1). The bond lengths and angles within each molecule (Table 2) are unexceptional. Although there is no structural data available for this dibromoperfluoroalkane, it is worth noting that the C—Br distances in (I) are slightly longer [1.933 (4) and 1.928 (4)] than the C—Br distances observed for $\text{BrCF}_2\text{CF}_2\text{Br}$ recorded under pressure in a diamond anvil cell [$d(\text{C—Br}) = 1.918$ (8) Å] (Olejniczak *et al.*, 2009). The average N—C and C—C bond lengths in adduct (I) are 1.471 (5) and 1.545 (6) Å respectively, which are comparable with the values reported for the low-temperature phase of DABCO (Sauvajol, 1980) of 1.483 (10) and 1.536 (10) Å.

In the extended structure, the two parent molecules are linked by near-linear halogen bonds [C—Br $\cdots$ N = 174.0 (2) and 177.2 (2)° with N $\cdots$ Br distances of 2.809 (3) and 2.818 (3) Å] to yield 1-D infinite chains of alternating DABCO and $\text{Br}(\text{CF}_2)_4\text{Br}$ molecules parallel to the *a*-axis (Fig 2). The N $\cdots$ Br distances are 0.58 (17.1%) and 0.59 Å (17.4%) shorter than the sum of the van der Waals radii for the two atoms (3.40 Å) (Bondi, 1964), which places these N $\cdots$ Br distances at the shorter end of the values previously reported in the related crystal structures of bromoperfluorocarbon-containing type I halogen bonded adducts, such as $\text{BrCF}_2(\text{CF}_2)_6\text{CF}_2\text{Br}\cdot\text{N,N,N,N}$ -tetramethyl-1,4-phenylenediamine (2.922 (2) Å) (Liantonio *et al.*, 2003), $\text{BrCF}_2\text{CF}_2\text{Br}\cdot\text{N,N,N,N}$ -tetramethyl-ethylenediamine (2.863 (5) Å) (Huang *al.*, 2006), ($\text{BrCF}_2\text{CF}_2\text{Br}\cdot\text{DABCO}$) (2.829 (3) Å) (Brisdon *et al.*, 2015).

The dominant interactions in adduct (I) are the halogen bonds, there are no classical hydrogen bonds formed, but several weak F $\cdots$ H contacts are observed; the shortest of which is a pair of contacts between two H atoms on adjacent carbons of DABCO with two fluorines of a neighbouring dibromoperfluorobutane molecule, F3 $\cdots$ H7Aⁱ, 2.60 Å and F7 $\cdots$ H10Bⁱ, 2.62 Å (symmetry code, i: 1 - *x*, -1/2 + *y*, 1/2 - *z*), which are just less than the sum of the respective van der Waals radii at 2.67 Å (Bondi, 1964). These data suggest that the N $\cdots$ Br halogen bond interaction is the more significant, and is therefore primarily responsible for the observed extended structure. Further confirmation of the halogen bonding interaction comes from the IR spectrum of the solid crystals. The two most notable C—F absorptions of pure $\text{Br}(\text{CF}_2)_4\text{Br}$ are observed at 1193.5 and 1141.4 cm⁻¹, but appear at lower frequencies (1189.8 and 1129.0 cm⁻¹) in adduct (I), with notably decreased intensities.

Adduct (II) crystallizes in the triclinic space group $P\bar{1}$, the asymmetric unit consists of one molecule of both DABCO and $\text{Br}(\text{CF}_2)_6\text{Br}$, as shown in Fig. 3. The observed bond lengths are given in Table 3, and while the average C—N and C—C distances of the DABCO molecule at $d(\text{C—N}) = 1.476$ (7) and $d(\text{C—C}) = 1.546$ (7) Å are similar to those observed in adduct (I), there are no reported structural data for $\text{Br}(\text{CF}_2)_6\text{Br}$ with which to make a direct comparison.

The extended structure displays near-linear interactions [C—Br $\cdots$ N angles of 171.8 (2) and 172.6 (2)°] between the nitrogen atoms of the donor DABCO molecules and the bromine atoms of $\text{Br}(\text{CF}_2)_6\text{Br}$, to give infinite one-dimensional

chains, (Fig. 4), similar to those observed in adduct (I). The N $\cdots$ Br distances [2.940 (4) and 2.958 (4) Å] are a little longer than found in (I), but still 0.44 (12.9%) and 0.45 Å (13.2%) shorter than the sum of the van der Waals radii of 3.40 Å. Taken together these data are consistent with the presence of a halogen bond interaction.

Once again, other, weak interactions are apparent in the extended structure. One of the fluorine atoms on the β -carbon of the dibromoperfluorohexane chain, F9, acts as a hydrogen bond acceptor to a hydrogen of DABCO, d(F9 $\cdots$ H9Bⁱⁱ) at a bond distance of 2.63 Å [symmetry code, ii: 1 - x, 1 - y, 1 - z]. While the other fluorine of the β -CF₂, F10, generates an F $\cdots$ F contact of 2.825 (5) Å, just less than twice the van der Waals radius of fluorine (2.94 Å, Bondi, 1964) with F6ⁱⁱⁱ, with a central fluorine of an adjacent chain, [symmetry code, iii: 1 - x, -y, 1 - z]. The reciprocal arrangement is generated from the second chain, F10ⁱⁱⁱ $\cdots$ F6. A further pair of F $\cdots$ F interactions are observed between F3 and F5ⁱⁱ, and F5 and F3ⁱⁱ, d = 2.872 (6) Å of adjacent chains. In this way a total of five secondary interactions are generated by fluorines of the perfluorinated chains.

Adduct (III) also crystallizes in the triclinic $P\bar{1}$ space group, and the asymmetric unit consists of a molecule of DABCO and Br(CF₂)₈Br (Fig. 5). The observed bond distance within each molecule of the adduct are consistent with those found in adducts (I) and (II), Table 4. The extended structure again shows the presence of N $\cdots$ Br halogen bonds involving the two nitrogen atoms of the DABCO molecules and both bromine atoms of Br(CF₂)₈Br, resulting in 1-D infinite chains (Fig. 6). The N $\cdots$ Br distances are 2.945 (3) and 2.971 (3) Å, which are 0.42 Å (12.4%) and 0.45 Å (13.2%) less than the sum of the van der Waals radii, and similar to the distances observed in adduct (II). The halogen bonds formed possess near linear N $\cdots$ Br—C angles of 172.6 (2) and 173.0 (2)°. Other non-covalent interactions observed in the extended structure of (III) include an H $\cdots$ F interaction between a fluorine of one of the β -CF₂ groups, F14, of the dibromoperfluorooctane chain and a hydrogen (H3A^{iv}, symmetry code, iv: x, y, 1+z) on DABCO, at a distance of 2.66 Å. As was observed in (II), the other fluorine atom of this CF₂ group is involved in an F $\cdots$ F interaction, d(F13 $\cdots$ F5^v) = 2.852 (5) Å, [symmetry code, v: 2 - x, 2 - y, 1 - z], as are the fluorines of alternate CF₂ groups, d(F9 $\cdots$ F9^v) = 2.882 (4) Å and F5 $\cdots$ F13^v), while a pair of F $\cdots$ F interactions arises between F4 and F10 on the other side of the fluorinated chain, d(F4 $\cdots$ F10^{iv}) = d(F10 $\cdots$ F4^{iv}) = 2.864 (4) Å [symmetry code, iv: 2 - x, 1 - y, 1 - z]. Thus, the 1-D halogen-bonded chains are cross-linked alternately by three F $\cdots$ F interactions or two F $\cdots$ F and one F $\cdots$ H interactions.

Thus, in all three new structures reported here, (I), (II) and (III), the dominant non-covalent interaction appears to be the N $\cdots$ Br halogen bonds, the distance of which lie between 2.809 (3) and 2.971 (3) Å, and all of the interactions are approximately linear, with C—Br $\cdots$ N angles ranging from 171.8 (2) to 177.2 (2)°. Values for these parameters for (I), (II) and (III), as well as those of related bromofluoroalkyl halogen bonded systems, are listed in Table 5.

The C—N $\cdots$ Br distances of the previously reported systems (CSD identifiers KURMAN (Brisdon *et al.*, 2015), REMBOB (Huang *et al.*, 2006) and VAQROV (Liantonio *et al.*, 2003)) lie between 2.829 and 2.965 Å, with the mean being 2.871 Å. The distances determined for adduct (I) of 2.809 (3) and 2.818 (3) Å, average 2.814 (3) Å, are shorter than those previously reported. However, it is worth noting that even shorter N $\cdots$ Br distances have been reported in other systems, for example 2.752 Å in the adduct of 4-bromo-2,3,5,6-tetrafluorophenyl)-2,3,5,6-tetrafluoro-4-iodobenzamide and 1,2-bis(4-pyridyl)-ethene (Aakeröy *et al.*, 2013) and 2.76 (1) Å for the adduct of DABCO and CBr₄ (Blackstock *et al.*, 1987). Interestingly, the N $\cdots$ Br distances observed in our systems increase in the order (I) < (II) < (III), that is the shorter dibromo-PFC chains give rise to shorter halogen bond distances than for the long chained adducts. However, this trend does not extend to the previously reported adduct of Br(CF₂)₂Br with DABCO, where the N $\cdots$ Br distance was found to be 2.829 (3) Å (Brisdon *et al.*, 2015), which is significantly longer than the equivalent distance in (I).

It is noteworthy that the 1-D halogen-bonded chains formed between DABCO and the two shorter dibromoperfluoroalkyl materials, *i.e.* KURMAN (Brisdon *et al.*, 2015) and adduct (I) align so that a pair of weak H $\cdots$ F interactions are generated between two DABCO H atoms and two fluorines on different carbons, but on the same side of the perfluoro-

alkyl chain. Whilst adducts (II) and (III) derived from the longer perfluoroalkyl chained starting materials, display both $\text{H}\cdots\text{F}$ and $\text{F}\cdots\text{F}$ interactions that predominantly involve both fluorine atoms of alternating CF_2 groups along the perfluoroalkyl chain. A summary of these interactions is given in Table 6.

In conclusion, attractive intermolecular $\text{N}\cdots\text{Br}$ halogen bonds have been detected in three new crystal structures containing DABCO and 1,4-dibromoperfluorobutane, 1,6-dibromoperfluorohexane and 1,8-dibromoperfluorooctane, respectively, which were formed by vapour diffusion. The presence of halogen donor-acceptor interactions at either ends of both molecules gives rise to extended one-dimensional chain structures. The observed $\text{N}\cdots\text{Br}$ bond distances are found to be in the order (I) < (II) < (III) (Table 2), ranging from *ca.* 17% to 12% less than the sum of the relevant van der Waals radii. Based on a search of the CSD the $\text{N}\cdots\text{Br}$ bond distance in (I) at 2.809 (3) Å is the shortest halogen bonded distance so far observed between a nitrogen atom and a bromine atom attached to a fluorocarbon system. Alternate CF_2 units of the perfluoroalkyl chains are involved in $\text{F}\cdots\text{F}$ and $\text{H}\cdots\text{F}$ interactions which act to crosslink the 1-D polymeric chains.

fig1.tif

Figure 1

A view of the structure of halogen bonded adduct (I), showing the atomic numbering scheme. The blue dotted line indicates halogen bonding.

fig2.tif

Figure 2

A view of the intermolecular halogen bonds (dotted lines) in the crystal structure of (I). Colours are as follows: grey, carbon; blue, nitrogen; brown, bromine; and yellow, fluorine.

fig3.tif

160 **Figure 3**

161 A view of the structure of halogen-bonded adduct (II), showing the atomic numbering scheme. The blue dotted line
162 indicates halogen bonding.

fig4.tif

163 **Figure 4**

164 A view of the intermolecular halogen bonds (dotted lines) in the crystal structure of (II). Colours are as follows: grey,
165 carbon; blue, nitrogen; brown, bromine; and yellow, fluorine.

fig5.tif

166 **Figure 5**

167 A view of the structure of halogen bonded adduct (III), showing the atom numbering scheme. The blue dotted line
168 indicates halogen bonding.

fig6.tif

169 **Figure 6**

170 A view of the intermolecular halogen bonds (dotted lines) in the crystal structure of (III). Colours are as follows: grey,
171 carbon; blue, nitrogen; brown, bromine; and green, fluorine.

Table 1

Experimental details

	(I)	(II)	(III)
Crystal data			
Chemical formula	C ₄ Br ₂ F ₈ ·C ₆ H ₁₂ N ₂	C ₆ Br ₂ F ₁₂ ·C ₆ H ₁₂ N ₂	C ₈ Br ₂ F ₁₆ ·C ₆ H ₁₂ N ₂
M_r	472.04	572.06	672.08
Crystal system, space group	Monoclinic, $P2_1/c$	Triclinic, $P\bar{1}$	Triclinic, $P\bar{1}$
Temperature (K)	150	100	150
a, b, c (Å)	13.4659 (6), 11.0854 (6), 10.7955 (6)	6.0096 (4), 10.4941 (8), 14.5016 (11)	6.03367 (19), 10.4836 (5), 16.4471 (9)
α, β, γ (°)	90, 106.271 (5), 90	92.051 (6), 101.375 (6), 90.507 (6)	88.785 (4), 84.126 (3), 89.724 (3)
V (Å ³)	1546.96 (15)	895.89 (11)	1034.65 (8)
Z	4	2	2
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$
μ (mm ⁻¹)	5.32	4.64	4.07
Crystal size (mm)	0.2 × 0.2 × 0.15	0.18 × 0.16 × 0.12	0.14 × 0.08 × 0.04
Data collection			
Diffractometer	SuperNova, Single source at offset, Eos	Xcalibur, Atlas	SuperNova, Single source at offset, Eos
Absorption correction	Multi-scan <i>CrysAlis PRO</i> 1.171.38.41 (Rigaku Oxford Diffraction, 2015) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.	Multi-scan <i>CrysAlis PRO</i> , Agilent Technologies, Version 1.171.37.33 (release 27-03-2014 <i>CrysAlis171 .NET</i>) (compiled Mar 27 2014, 17:12:48) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.	Multi-scan <i>CrysAlis PRO</i> , Agilent Technologies, Version 1.171.37.33 (release 27-03-2014 <i>CrysAlis171 .NET</i>) (compiled Mar 27 2014, 17:12:48) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.
$T_{\min}, T_{\max}$	0.025, 1.000	0.769, 1.000	0.622, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	6191, 3034, 2260	12221, 3504, 2686	14918, 4680, 3723
R_{int}	0.046	0.053	0.042
$(\sin \theta/\lambda)_{\text{max}}$ (Å ⁻¹)	0.617	0.617	0.650
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.041, 0.064, 0.93	0.049, 0.104, 1.13	0.050, 0.122, 1.07
No. of reflections	3034	3504	4680
No. of parameters	199	253	307
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å ⁻³)	0.43, -0.51	0.94, -0.72	1.00, -0.69

Computer programs: *CrysAlis PRO* 1.171.38.41 (Rigaku OD, 2015), *CrysAlis PRO*, Agilent Technologies, Version 1.171.37.33 (release 27-03-2014
CrysAlis171 .NET) (compiled Mar 27 2014, 17:12:48), *SHELXS* (Sheldrick, 2008), *ShelXT* (Sheldrick, 2015), *Superflip* (Palatinus & Chapuis,
2007;Palatinus & van der Lee, 2008; Palatinus *et al.*, 2012), *SHELXL* (Sheldrick, 2015), *SHELXL* (Sheldrick, 2008), *Olex2* (Dolomanov *et al.*, 2009).

Table 2

Selected bond lengths (Å) for (I)

Br1—C1	1.933 (4)	C4—F7	1.328 (4)
Br2—C4	1.928 (4)	C4—F8	1.334 (5)
C1—C2	1.523 (5)	C5—C8	1.546 (6)
C1—F1	1.336 (5)	C5—N1	1.465 (5)
C1—F2	1.328 (5)	C6—C9	1.547 (6)
C2—C3	1.562 (5)	C6—N1	1.472 (5)
C2—F3	1.331 (4)	C7—C10	1.541 (6)
C2—F4	1.333 (4)	C7—N1	1.477 (4)
C3—C4	1.551 (5)	C8—N2	1.471 (5)
C3—F5	1.334 (5)	C9—N2	1.475 (4)
C3—F6	1.333 (4)	C10—N2	1.465 (5)

Table 3

Selected bond lengths (Å) for (II)

Br1—C1	1.930 (6)	C1—C2	1.541 (8)
Br2—C6	1.937 (6)	C2—C3	1.560 (8)
F1—C1	1.349 (7)	C3—C4	1.545 (8)
F2—C1	1.328 (7)	C4—C5	1.553 (8)
F3—C2	1.313 (6)	C5—C6	1.537 (8)
F4—C2	1.343 (6)	N1—C7	1.482 (6)
F5—C3	1.341 (7)	N1—C9	1.479 (7)
F6—C3	1.334 (7)	N1—C11	1.472 (7)
F7—C4	1.341 (7)	N2—C8	1.468 (7)
F8—C4	1.346 (6)	N2—C10	1.476 (7)
F9—C5	1.337 (6)	N2—C12	1.477 (7)
F10—C5	1.318 (7)	C7—C8	1.545 (7)
F11—C6	1.327 (7)	C9—C10	1.545 (7)
F12—C6	1.351 (8)	C11—C12	1.549 (7)

Table 4

Selected bond lengths (Å) for (III)

Br1—C7	1.915 (5)	F16—C14	1.314 (6)
Br2—C14	1.921 (5)	C7—C8	1.547 (6)
F1—C7	1.321 (6)	C8—C9	1.547 (6)
F2—C7	1.347 (6)	C9—C10	1.562 (6)
F3—C8	1.321 (5)	C10—C11	1.549 (6)
F4—C8	1.326 (5)	C11—C12	1.555 (6)
F5—C9	1.325 (5)	C12—C13	1.560 (6)
F6—C9	1.346 (5)	C13—C14	1.537 (6)

244	F7—C10	1.334 (5)	N1—C1	1.469 (5)
245	F8—C10	1.336 (5)	N1—C2	1.469 (5)
246	F9—C11	1.323 (5)	N1—C3	1.472 (5)
247	F10—C11	1.333 (5)	N2—C4	1.466 (5)
248	F11—C12	1.343 (5)	N2—C5	1.474 (5)
249	F12—C12	1.323 (5)	N2—C6	1.471 (5)
250	F13—C13	1.320 (5)	C1—C6	1.543 (6)
251	F14—C13	1.323 (5)	C2—C5	1.536 (5)
252	F15—C14	1.348 (7)	C3—C4	1.554 (6)

253 **Table 5**

254 N···Br halogen bond distances in the current work and previously reported structures.

255	Entry	Donor	Acceptor	d(N···Br)/Å	Refcode	Reference
256	1	Br(CF ₂) ₈ Br	Me ₂ N-C ₆ H ₄ -NMe ₂	2.922 (3)	VAQROV	Liantonio <i>et al.</i> , 2003
257	2	Br(CF ₂) ₂ Br	Me ₂ NCH ₂ CH ₂ NMe ₂	2.862 (5)	REMBOB	Huang <i>et al.</i> 2006
258	3	Br(CF ₂) ₂ Br	DABCO	2.829 (3)	KURMAN	Brisdon <i>et al.</i> , 2015
259	4	Br(CF ₂) ₄ Br	DABCO	2.814 (3) _{av}	-	this work
260	5	Br(CF ₂) ₆ Br	DABCO	2.949 (4) _{av}	-	this work
261	6	Br(CF ₂) ₈ Br	DABCO	2.958 (3) _{av}	-	this work

262 **Table 6**

263 Intermolecular F···H and F···F contact distances less than the sum of van der Waals radii and their difference.

264	Adduct	X	Y	d(X···Y)/Å	Difference/Å
265	KURMAN	F1	H2A	2.66	-0.01
266	(I)	F3	H7A ⁱ	2.60	-0.08
267		F7	H10B ⁱ	2.62	-0.05
268	(II)	F9	H9B ⁱⁱ	2.63	-0.04
269		F3	F5 ⁱⁱ	2.872 (6)	-0.068
270		F6	F10 ⁱⁱⁱ	2.825 (5)	-0.115
271	(III)	F14	H3A ^{iv}	2.66	-0.01
272		F5	F13 ^v	2.852 (5)	-0.090
273		F9	F9 ^v	2.882 (4)	-0.058
274		F4	F10 ^{vi}	2.864 (4)	-0.080

275 Symmetry codes, i: 1 - x, -1/2 + y, 1/2 - z; ii: 1 - x, 1 - y, 1 - z; iii: 1 - x, -y, 1 - z; iv: x, y, 1 + z; v: 2 - x, 2 - y, 1 - z; vi: 2 - x, 1 - y, 1 - z;

276 **Acknowledgements**277 We thank the Libyan Government for support of AM. We acknowledge the EPSRC for support of departmental X-ray
278 diffraction facilities (grant number EP/K039547/1).279 **References**

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checkCIF/PLATON results for paper uk3139

checkCIF/PLATON results

Ellipsoid plot

checkCIF/PLATON results

No syntax errors found.

CIF dictionary

Interpreting this report

Datablock: I

Bond precision: C-C = 0.0065 Å Wavelength=0.71073

Cell: a=13.4659(6) b=11.0854(6) c=10.7955(6)

alpha=90 beta=106.271(5) gamma=90

Temperature: 150 K

	Calculated	Reported
	-----	-----
Volume	1546.95(14)	1546.96(15)
Space group	P 21/c	P 1 21/c 1
Hall group	-P 2ybc	-P 2ybc
Moiety formula	C4 Br2 F8, C6 H12 N2	C4 Br2 F8, C6 H12 N2
Sum formula	C10 H12 Br2 F8 N2	C10 H12 Br2 F8 N2
Mr	472.02	472.04
Dx, g cm-3	2.027	2.027
Z	4	4
Mu (mm-1)	5.320	5.320
F000	912.0	912.0
F000'	910.47	
h,k,lmax	16,13,13	16,13,13
Nref	3040	3034
Tmin,Tmax	0.359,0.450	0.025,1.000
Tmin'	0.332	

Correction method= # Reported T Limits: Tmin=0.025

Tmax=1.000 AbsCorr = MULTI-SCAN

Data completeness= 0.998

Theta(max)= 25.997

R(reflections)= 0.0408(2260) wR2(reflections)= 0.0644(3034)

S = 0.934 Npar= 199

Alert level C

PLAT213_ALERT_2_C Atom F2	has ADP max/min Ratio	3.1 prolat
PLAT250_ALERT_2_C Large U3/U1 Ratio for Average U(i,j) Tensor		2.5 Note
PLAT341_ALERT_3_C Low Bond Precision on C-C Bonds		0.0065 Ang.
PLAT906_ALERT_3_C Large K value in the Analysis of Variance		3.920 Check
PLAT910_ALERT_3_C Missing # of FCF Reflection(s) Below Theta(Min).		5 Note
PLAT978_ALERT_2_C Number C-C Bonds with Positive Residual Density.		0 Info

Alert level G

PLAT012_ALERT_1_G No _shelx_res_checksum found in CIF		Please Check
PLAT431_ALERT_2_G Short Inter HL..A Contact Br1 .. N1 ..		2.82 Ang.
PLAT431_ALERT_2_G Short Inter HL..A Contact Br2 .. N2 ..		2.81 Ang.
PLAT432_ALERT_2_G Short Inter X...Y Contact Br1 .. C6 ..		3.34 Ang.
PLAT899_ALERT_4_G SHELXL97 is Deprecated and Succeeded by SHELXL		2016 Note

0 **ALERT level A** = Most likely a serious problem - resolve or explain

0 **ALERT level B** = A potentially serious problem, consider carefully

6 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight

5 **ALERT level G** = General information/check it is not something unexpected

1 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
6 ALERT type 2 Indicator that the structure model may be wrong or deficient
3 ALERT type 3 Indicator that the structure quality may be low
1 ALERT type 4 Improvement, methodology, query or suggestion
0 ALERT type 5 Informative message, check

Datablock: II

Bond precision: C-C = 0.0080 Å Wavelength=0.71073
Cell: a=6.0096(4) b=10.4941(8) c=14.5016(11)
alpha=92.051(6) beta=101.375(6) gamma=90.507(6)
Temperature: 100 K

	Calculated	Reported
	-----	-----
Volume	895.89(11)	895.89(11)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	C6 Br2 F12, C6 H12 N2	C6 Br2 F12, C6 H12 N2
Sum formula	C12 H12 Br2 F12 N2	C12 H12 Br2 F12 N2
Mr	572.04	572.06
Dx,g cm-3	2.121	2.121
Z	2	2
Mu (mm-1)	4.644	4.644
F000	552.0	552.0
F000'	551.36	
h,k,lmax	7,12,17	7,12,17
Nref	3517	3504
Tmin,Tmax	0.438,0.573	0.769,1.000
Tmin'	0.429	

Correction method= # Reported T Limits: Tmin=0.769 Tmax=1.000 AbsCorr = MULTI-SCAN
Data completeness= 0.996
Theta(max)= 25.998
R(reflections)= 0.0493(2686) wR2(reflections)= 0.1042(3504)
S = 1.128 Npar= 253

Alert level C

PLAT213_ALERT_2_C Atom F2	has ADP max/min Ratio	3.1 prolat
PLAT213_ALERT_2_C Atom F11	has ADP max/min Ratio	3.5 prolat
PLAT242_ALERT_2_C Low 'MainMol' Ueq as Compared to Neighbors of		C2 Check
PLAT250_ALERT_2_C Large U3/U1 Ratio for Average U(i,j) Tensor		2.7 Note
PLAT341_ALERT_3_C Low Bond Precision on C-C Bonds		0.008 Ang.
PLAT906_ALERT_3_C Large K value in the Analysis of Variance		4.258 Check
PLAT910_ALERT_3_C Missing # of FCF Reflection(s) Below Theta(Min).		5 Note
PLAT911_ALERT_3_C Missing # FCF Refl Between THmin & STh/L= 0.600		2 Report
PLAT978_ALERT_2_C Number C-C Bonds with Positive Residual Density.		0 Info

Alert level G

PLAT154_ALERT_1_G The s.u.'s on the Cell Angles are Equal ..(Note)		0.006 Degree
PLAT431_ALERT_2_G Short Inter HL..A Contact Br1 .. N1 ..		2.94 Ang.
PLAT431_ALERT_2_G Short Inter HL..A Contact Br2 .. N2 ..		2.96 Ang.
PLAT432_ALERT_2_G Short Inter X...Y Contact Br2 .. C12 ..		3.34 Ang.
PLAT434_ALERT_2_G Short Inter HL..HL Contact F6 .. F10 ..		2.83 Ang.
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L= 0.600		6 Note
PLAT933_ALERT_2_G Number of OMIT Records in Embedded .res File ...		2 Note

0 **ALERT level A** = Most likely a serious problem - resolve or explain
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9 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
7 **ALERT level G** = General information/check it is not something unexpected

1 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
10 ALERT type 2 Indicator that the structure model may be wrong or deficient
4 ALERT type 3 Indicator that the structure quality may be low
1 ALERT type 4 Improvement, methodology, query or suggestion
0 ALERT type 5 Informative message, check

Datablock: III

Bond precision: C-C = 0.0064 Å Wavelength=0.71073
Cell: a=6.03367(19) b=10.4836(5) c=16.4471(9)
alpha=88.785(4) beta=84.126(3) gamma=89.724(3)
Temperature: 150 K

	Calculated	Reported
	-----	-----
Volume	1034.66(8)	1034.65(8)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	C8 Br2 F16, C6 H12 N2	C8 Br2 F16, C6 H12 N2
Sum formula	C14 H12 Br2 F16 N2	C14 H12 Br2 F16 N2
Mr	672.06	672.08
Dx,g cm-3	2.157	2.157
Z	2	2
Mu (mm-1)	4.065	4.065
F000	648.0	648.0
F000'	647.49	
h,k,lmax	7,13,21	7,13,21
Nref	4755	4680
Tmin,Tmax	0.684,0.850	0.622,1.000
Tmin'	0.560	

Correction method= # Reported T Limits: Tmin=0.622 Tmax=1.000 AbsCorr = MULTI-SCAN
Data completeness= 0.984
Theta(max)= 27.497
R(reflections)= 0.0497(3723) wR2(reflections)= 0.1222(4680)
S = 1.069 Npar= 307

Alert level C

PLAT213_ALERT_2_C Atom F1	has ADP max/min Ratio	3.3 prolat
PLAT213_ALERT_2_C Atom F10	has ADP max/min Ratio	3.1 prolat
PLAT213_ALERT_2_C Atom F16	has ADP max/min Ratio	3.6 prolat
PLAT242_ALERT_2_C Low 'MainMol' Ueq as Compared to Neighbors of		C7 Check
PLAT242_ALERT_2_C Low 'MainMol' Ueq as Compared to Neighbors of		C8 Check
PLAT242_ALERT_2_C Low 'MainMol' Ueq as Compared to Neighbors of		C13 Check
PLAT242_ALERT_2_C Low 'MainMol' Ueq as Compared to Neighbors of		C14 Check
PLAT250_ALERT_2_C Large U3/U1 Ratio for Average U(i,j) Tensor		3.2 Note
PLAT341_ALERT_3_C Low Bond Precision on C-C Bonds		0.0064 Ang.
PLAT906_ALERT_3_C Large K value in the Analysis of Variance		5.573 Check
PLAT910_ALERT_3_C Missing # of FCF Reflection(s) Below Theta(Min).		7 Note
PLAT911_ALERT_3_C Missing # FCF Refl Between THmin & STh/L= 0.600		3 Report
PLAT978_ALERT_2_C Number C-C Bonds with Positive Residual Density.		0 Info

Alert level G

PLAT012_ALERT_1_G No _shelx_res_checksum found in CIF		Please Check
PLAT112_ALERT_2_G ADDSYM Detects New (Pseudo) Symm. Elem. sub		82 %Fit
PLAT431_ALERT_2_G Short Inter HL..A Contact Br1 .. N1 ..		2.94 Ang.

0 **ALERT level A** = Most likely a serious problem - resolve or explain
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12 ALERT type 2 Indicator that the structure model may be wrong or deficient
4 ALERT type 3 Indicator that the structure quality may be low
1 ALERT type 4 Improvement, methodology, query or suggestion
0 ALERT type 5 Informative message, check

database duplication summary

Datablock: I

- **Chemical name** =
- *R* factor = 0.041
- Space group =
- Formula = C10 H12 Br2 F8 N2
- a=13.4659 b=11.0854 c=10.7955
- alpha=90 beta=106.271 gamma=90

Datablock: II

- **Chemical name** =
- *R* factor = 0.049
- Space group =
- Formula = C12 H12 Br2 F12 N2
- a=6.0096 b=10.4941 c=14.5016
- alpha=92.051 beta=101.375 gamma=90.507

Datablock: III

- **Chemical name** =
- *R* factor = 0.050
- Space group =
- Formula = C14 H12 Br2 F16 N2
- a=6.03367 b=10.4836 c=16.4471
- alpha=88.785 beta=84.126 gamma=89.724

No duplication found.

reference checking results

The following reference was not checked in detail as it was not recognized as a journal reference

Forster, R. Organic Charge-Transfer Complexes; Academic Press, London, 1969.

The following reference does not appear to be cited

Forster, R. Organic Charge-Transfer Complexes; Academic Press, London, 1969.

Citation comments

4 reference(s) in data_global _publ_body_contents should be checked as possibly cited ambiguously (or simply identified with less confidence)

1 date found in data_global _publ_body_contents that could be part of a citation but not found in reference list: 1969

2 reference(s) in data_global _publ_body_contents should be checked as possibly cited ambiguously (or simply identified with less confidence)

1 date found in data_global _publ_body_contents that could be part of a citation but not found in reference list: 2006

1 date found in data_I _exptl_absorpt_process_details that could be part of a citation but not found in reference list: 2015

1 date found in data_I _computing_data_collection that could be part of a citation but not found in reference list: 2015

1 date found in data_I _computing_data_reduction that could be part of a citation but not found in reference list: 2015

1 date found in data_I _computing_cell_refinement that could be part of a citation but not found in reference list: 2015

2 reference(s) in data_I _computing_structure_solution should be checked as possibly cited ambiguously (or simply identified with less confidence)

1 date found in data_II _exptl_absorpt_process_details that could be part of a citation but not found in reference list: 2014

1 date found in data_II _computing_data_collection that could be part of a citation but not found in reference list: 2014

1 date found in data_II _computing_data_reduction that could be part of a citation but not found in reference list: 2014

1 date found in data_II _computing_cell_refinement that could be part of a citation but not found in reference list: 2014

1 date found in data_III _exptl_absorpt_process_details that could be part of a citation but not found in reference list: 2014

1 date found in data_III _computing_data_collection that could be part of a citation but not found in reference list: 2014

1 date found in data_III _computing_data_reduction that could be part of a citation but not found in reference list: 2014

1 date found in data_III _computing_cell_refinement that could be part of a citation but not found in reference list: 2014

2 reference(s) in data_III _computing_structure_refinement should be checked as possibly cited ambiguously (or simply identified with less confidence)

1 supporting information

2 Halogen bonding in a series of Br(CF₂)_nBr-DABCO adducts (n = 4, 6, 8).

3 Alan K. Brisdon,* Abeer M. T. Muneer and Robin G Pritchard

4 Computing details

5 Data collection: *CrysAlis PRO* 1.171.38.41 (Rigaku OD, 2015) for (I); *CrysAlis PRO*, Agilent Technologies, Version
 6 1.171.37.33 (release 27-03-2014 CrysAlis171 .NET) (compiled Mar 27 2014,17:12:48) for (II), (III). Cell refinement:
 7 *CrysAlis PRO* 1.171.38.41 (Rigaku OD, 2015) for (I); *CrysAlis PRO*, Agilent Technologies, Version 1.171.37.33 (release
 8 27-03-2014 CrysAlis171 .NET) (compiled Mar 27 2014,17:12:48) for (II), (III). Data reduction: *CrysAlis PRO*
 9 1.171.38.41 (Rigaku OD, 2015) for (I); *CrysAlis PRO*, Agilent Technologies, Version 1.171.37.33 (release 27-03-2014
 10 CrysAlis171 .NET) (compiled Mar 27 2014,17:12:48) for (II), (III). Program(s) used to solve structure: *SHELXS*
 11 (Sheldrick, 2008) for (I); *ShelXT* (Sheldrick, 2015) for (II); *Superflip* (Palatinus & Chapuis, 2007;Palatinus & van der
 12 Lee, 2008; Palatinus *et al.*, 2012) for (III). Program(s) used to refine structure: *SHELXL* (Sheldrick, 2015) for (I), (II);
 13 *SHELXL* (Sheldrick, 2008) for (III). For all structures, molecular graphics: *Olex2* (Dolomanov *et al.*, 2009); software
 14 used to prepare material for publication: *Olex2* (Dolomanov *et al.*, 2009).

15 (I)

16 Crystal data

17 C₄Br₂F₈·C₆H₁₂N₂
 18 *M_r* = 472.04
 19 Monoclinic, *P*2₁/*c*
 20 *a* = 13.4659 (6) Å
 21 *b* = 11.0854 (6) Å
 22 *c* = 10.7955 (6) Å
 23 β = 106.271 (5)°
 24 *V* = 1546.96 (15) Å³
 25 *Z* = 4

F(000) = 912
D_x = 2.027 Mg m⁻³
 Mo *K*α radiation, λ = 0.71073 Å
 Cell parameters from 1842 reflections
 θ = 3.7–27.3°
 μ = 5.32 mm⁻¹
T = 150 K
 Block, colourless
 0.2 × 0.2 × 0.15 mm

26 Data collection

27 SuperNova, Single source at offset, Eos
 diffractometer
 28 Radiation source: micro-focus sealed X-ray
 tube, SuperNova (Mo) X-ray Source
 29 Mirror monochromator
 30 Detector resolution: 8.0714 pixels mm⁻¹
 31 ω scans

Absorption correction: multi-scan
CrysAlis PRO 1.171.38.41 (Rigaku Oxford
 Diffraction, 2015) Empirical absorption
 correction using spherical harmonics,
 implemented in SCALE3 ABSPACK scaling
 algorithm.
T_{min} = 0.025, *T_{max}* = 1.000
 6191 measured reflections
 3034 independent reflections
 2260 reflections with *I* > 2σ(*I*)
R_{int} = 0.046
 θ_{max} = 26.0°, θ_{min} = 3.4°
h = -14→16
k = -13→12
l = -13→13

32 *Refinement*33 Refinement on F^2

34 Least-squares matrix: full

35 $R[F^2 > 2\sigma(F^2)] = 0.041$ 36 $wR(F^2) = 0.064$ 37 $S = 0.93$

38 3034 reflections

39 199 parameters

40 0 restraints

Primary atom site location: structure-invariant
direct methodsHydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0076P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} = 0.001$ $\Delta\rho_{\max} = 0.43 \text{ e } \text{\AA}^{-3}$ $\Delta\rho_{\min} = -0.51 \text{ e } \text{\AA}^{-3}$ 41 *Special details*

42 **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.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
45 Br1	0.47057 (3)	0.36072 (4)	0.13908 (5)	0.02488 (13)
46 Br2	-0.07338 (3)	0.33779 (4)	-0.18506 (5)	0.02532 (13)
47 C1	0.3492 (3)	0.3537 (4)	-0.0077 (5)	0.0282 (12)
48 C2	0.2501 (3)	0.3426 (4)	0.0330 (4)	0.0202 (10)
49 C3	0.1478 (3)	0.3489 (4)	-0.0796 (5)	0.0208 (10)
50 C4	0.0488 (3)	0.3080 (4)	-0.0446 (5)	0.0240 (11)
51 F1	0.35763 (18)	0.2579 (3)	-0.0792 (3)	0.0613 (10)
52 F2	0.34425 (18)	0.4521 (3)	-0.0793 (3)	0.0598 (10)
53 F3	0.25256 (16)	0.2386 (2)	0.0954 (3)	0.0434 (8)
54 F4	0.24725 (17)	0.4318 (2)	0.1146 (3)	0.0456 (8)
55 F5	0.15754 (17)	0.2775 (2)	-0.1749 (3)	0.0408 (8)
56 F6	0.13500 (16)	0.4625 (2)	-0.1215 (3)	0.0398 (8)
57 F7	0.05584 (16)	0.1913 (2)	-0.0158 (3)	0.0429 (8)
58 F8	0.04251 (17)	0.3683 (2)	0.0599 (3)	0.0387 (7)
59 C5	0.6632 (3)	0.2423 (4)	0.4059 (5)	0.0264 (11)
60 H5A	0.6997	0.1996	0.3510	0.032*
61 H5B	0.5974	0.1996	0.3992	0.032*
62 C6	0.5907 (3)	0.4310 (4)	0.4455 (4)	0.0297 (12)
63 H6A	0.5239	0.3917	0.4415	0.036*
64 H6B	0.5764	0.5153	0.4154	0.036*
65 C7	0.7404 (3)	0.4287 (4)	0.3691 (4)	0.0255 (11)
66 H7A	0.7268	0.5133	0.3400	0.031*
67 H7B	0.7763	0.3881	0.3122	0.031*
68 C8	0.7308 (3)	0.2414 (4)	0.5477 (5)	0.0291 (12)
69 H8A	0.6929	0.2007	0.6023	0.035*
70 H8B	0.7950	0.1953	0.5541	0.035*
71 C9	0.6604 (3)	0.4305 (4)	0.5864 (5)	0.0317 (12)
72 H9A	0.6763	0.5145	0.6166	0.038*
73 H9B	0.6233	0.3912	0.6428	0.038*

74	C10	0.8092 (3)	0.4258 (4)	0.5097 (5)	0.0309 (12)
75	H10A	0.8745	0.3831	0.5132	0.037*
76	H10B	0.8266	0.5094	0.5403	0.037*
77	N1	0.6416 (2)	0.3667 (3)	0.3606 (3)	0.0222 (9)
78	N2	0.7574 (2)	0.3650 (3)	0.5949 (4)	0.0219 (9)

79 *Atomic displacement parameters ($\text{\AA}^2$)*

80		U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
81	Br1	0.0157 (2)	0.0345 (3)	0.0208 (3)	−0.00078 (18)	−0.0007 (2)	0.0013 (2)
82	Br2	0.0159 (2)	0.0321 (3)	0.0241 (3)	−0.00058 (18)	−0.0007 (2)	−0.0016 (2)
83	C1	0.018 (2)	0.043 (3)	0.020 (3)	0.001 (2)	0.000 (2)	0.000 (3)
84	C2	0.022 (2)	0.020 (2)	0.015 (3)	−0.0026 (18)	−0.001 (2)	−0.004 (2)
85	C3	0.021 (2)	0.022 (2)	0.017 (3)	0.0005 (19)	0.001 (2)	0.002 (2)
86	C4	0.020 (2)	0.023 (2)	0.025 (3)	0.0026 (19)	0.001 (2)	0.002 (2)
87	F1	0.0250 (15)	0.104 (3)	0.049 (2)	0.0131 (15)	0.0010 (15)	−0.044 (2)
88	F2	0.0290 (15)	0.093 (2)	0.048 (2)	−0.0155 (15)	−0.0049 (15)	0.051 (2)
89	F3	0.0267 (14)	0.0503 (17)	0.043 (2)	−0.0060 (12)	−0.0069 (14)	0.0314 (17)
90	F4	0.0265 (14)	0.0638 (19)	0.040 (2)	0.0065 (13)	−0.0023 (14)	−0.0322 (18)
91	F5	0.0251 (14)	0.075 (2)	0.0210 (18)	0.0005 (13)	0.0039 (13)	−0.0220 (17)
92	F6	0.0230 (13)	0.0348 (15)	0.052 (2)	−0.0015 (11)	−0.0045 (14)	0.0251 (16)
93	F7	0.0246 (14)	0.0327 (15)	0.066 (3)	−0.0047 (12)	0.0033 (15)	0.0202 (16)
94	F8	0.0263 (14)	0.070 (2)	0.0196 (17)	0.0058 (13)	0.0058 (13)	−0.0084 (17)
95	C5	0.027 (2)	0.028 (3)	0.022 (3)	−0.001 (2)	0.003 (2)	−0.003 (2)
96	C6	0.026 (2)	0.041 (3)	0.021 (3)	0.008 (2)	0.003 (2)	0.003 (3)
97	C7	0.022 (2)	0.031 (3)	0.022 (3)	−0.004 (2)	0.004 (2)	0.000 (2)
98	C8	0.031 (2)	0.027 (3)	0.025 (3)	0.002 (2)	−0.001 (2)	−0.003 (2)
99	C9	0.026 (2)	0.046 (3)	0.022 (3)	0.010 (2)	0.006 (2)	−0.002 (3)
100	C10	0.023 (2)	0.036 (3)	0.030 (3)	−0.005 (2)	0.003 (2)	0.004 (3)
101	N1	0.0214 (18)	0.030 (2)	0.014 (2)	0.0003 (16)	0.0032 (17)	−0.0002 (19)
102	N2	0.0183 (18)	0.027 (2)	0.019 (2)	−0.0008 (15)	0.0032 (17)	−0.0009 (19)

103 *Geometric parameters ($\text{\AA}$, $^\circ$)*

104	Br1—C1	1.933 (4)	C6—H6A	0.9900
105	Br2—C4	1.928 (4)	C6—H6B	0.9900
106	C1—C2	1.523 (5)	C6—C9	1.547 (6)
107	C1—F1	1.336 (5)	C6—N1	1.472 (5)
108	C1—F2	1.328 (5)	C7—H7A	0.9900
109	C2—C3	1.562 (5)	C7—H7B	0.9900
110	C2—F3	1.331 (4)	C7—C10	1.541 (6)
111	C2—F4	1.333 (4)	C7—N1	1.477 (4)
112	C3—C4	1.551 (5)	C8—H8A	0.9900
113	C3—F5	1.334 (5)	C8—H8B	0.9900
114	C3—F6	1.333 (4)	C8—N2	1.471 (5)
115	C4—F7	1.328 (4)	C9—H9A	0.9900
116	C4—F8	1.334 (5)	C9—H9B	0.9900
117	C5—H5A	0.9900	C9—N2	1.475 (4)

118	C5—H5B	0.9900	C10—H10A	0.9900
119	C5—C8	1.546 (6)	C10—H10B	0.9900
120	C5—N1	1.465 (5)	C10—N2	1.465 (5)
121				
122	C2—C1—Br1	112.0 (3)	N1—C6—H6A	109.5
123	F1—C1—Br1	108.5 (3)	N1—C6—H6B	109.5
124	F1—C1—C2	109.0 (3)	N1—C6—C9	110.5 (3)
125	F2—C1—Br1	109.7 (3)	H7A—C7—H7B	108.2
126	F2—C1—C2	109.2 (3)	C10—C7—H7A	109.8
127	F2—C1—F1	108.4 (4)	C10—C7—H7B	109.8
128	C1—C2—C3	115.2 (4)	N1—C7—H7A	109.8
129	F3—C2—C1	108.4 (3)	N1—C7—H7B	109.8
130	F3—C2—C3	108.9 (3)	N1—C7—C10	109.5 (3)
131	F3—C2—F4	108.0 (4)	C5—C8—H8A	109.5
132	F4—C2—C1	108.7 (3)	C5—C8—H8B	109.5
133	F4—C2—C3	107.4 (3)	H8A—C8—H8B	108.1
134	C4—C3—C2	115.4 (4)	N2—C8—C5	110.8 (3)
135	F5—C3—C2	108.5 (3)	N2—C8—H8A	109.5
136	F5—C3—C4	107.0 (3)	N2—C8—H8B	109.5
137	F6—C3—C2	107.9 (3)	C6—C9—H9A	109.6
138	F6—C3—C4	108.7 (3)	C6—C9—H9B	109.6
139	F6—C3—F5	109.3 (4)	H9A—C9—H9B	108.1
140	C3—C4—Br2	111.2 (3)	N2—C9—C6	110.2 (4)
141	F7—C4—Br2	109.9 (3)	N2—C9—H9A	109.6
142	F7—C4—C3	109.4 (3)	N2—C9—H9B	109.6
143	F7—C4—F8	107.9 (4)	C7—C10—H10A	109.3
144	F8—C4—Br2	109.7 (3)	C7—C10—H10B	109.3
145	F8—C4—C3	108.8 (3)	H10A—C10—H10B	108.0
146	H5A—C5—H5B	108.2	N2—C10—C7	111.6 (3)
147	C8—C5—H5A	109.6	N2—C10—H10A	109.3
148	C8—C5—H5B	109.6	N2—C10—H10B	109.3
149	N1—C5—H5A	109.6	C5—N1—C6	109.4 (3)
150	N1—C5—H5B	109.6	C5—N1—C7	109.0 (3)
151	N1—C5—C8	110.1 (3)	C6—N1—C7	107.7 (3)
152	H6A—C6—H6B	108.1	C8—N2—C9	108.1 (3)
153	C9—C6—H6A	109.5	C10—N2—C8	108.8 (3)
154	C9—C6—H6B	109.5	C10—N2—C9	107.7 (3)
155				
156	Br1—C1—C2—C3	174.3 (3)	F5—C3—C4—Br2	−65.9 (4)
157	Br1—C1—C2—F3	−63.4 (4)	F5—C3—C4—F7	55.6 (5)
158	Br1—C1—C2—F4	53.8 (4)	F5—C3—C4—F8	173.2 (3)
159	C1—C2—C3—C4	166.2 (4)	F6—C3—C4—Br2	51.9 (4)
160	C1—C2—C3—F5	46.2 (5)	F6—C3—C4—F7	173.4 (4)
161	C1—C2—C3—F6	−72.1 (4)	F6—C3—C4—F8	−69.0 (5)
162	C2—C3—C4—Br2	173.3 (3)	C5—C8—N2—C9	60.2 (4)
163	C2—C3—C4—F7	−65.2 (5)	C5—C8—N2—C10	−56.5 (4)
164	C2—C3—C4—F8	52.4 (5)	C6—C9—N2—C8	−58.5 (5)
165	F1—C1—C2—C3	−65.6 (5)	C6—C9—N2—C10	59.0 (4)

166	F1—C1—C2—F3	56.7 (5)	C7—C10—N2—C8	58.4 (4)
167	F1—C1—C2—F4	173.9 (4)	C7—C10—N2—C9	−58.6 (4)
168	F2—C1—C2—C3	52.6 (5)	C8—C5—N1—C6	−57.3 (4)
169	F2—C1—C2—F3	174.9 (4)	C8—C5—N1—C7	60.3 (4)
170	F2—C1—C2—F4	−67.9 (5)	C9—C6—N1—C5	58.8 (4)
171	F3—C2—C3—C4	44.2 (5)	C9—C6—N1—C7	−59.5 (4)
172	F3—C2—C3—F5	−75.8 (4)	C10—C7—N1—C5	−58.5 (4)
173	F3—C2—C3—F6	165.9 (3)	C10—C7—N1—C6	60.0 (4)
174	F4—C2—C3—C4	−72.6 (4)	N1—C5—C8—N2	−2.2 (5)
175	F4—C2—C3—F5	167.4 (3)	N1—C6—C9—N2	−0.3 (5)
176	F4—C2—C3—F6	49.1 (4)	N1—C7—C10—N2	−1.0 (5)

177 (II)

178 *Crystal data*179 $\text{C}_6\text{Br}_2\text{F}_{12} \cdot \text{C}_6\text{H}_{12}\text{N}_2$ 180 $M_r = 572.06$ 181 Triclinic, $P\bar{1}$ 182 $a = 6.0096$ (4) Å183 $b = 10.4941$ (8) Å184 $c = 14.5016$ (11) Å185 $\alpha = 92.051$ (6)°186 $\beta = 101.375$ (6)°187 $\gamma = 90.507$ (6)°188 $V = 895.89$ (11) Å³ $Z = 2$ $F(000) = 552$ $D_x = 2.121$ Mg m^{−3}Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 4386 reflections

 $\theta = 3.9$ – 27.1 ° $\mu = 4.64$ mm^{−1} $T = 100$ K

Block, colourless

 $0.18 \times 0.16 \times 0.12$ mm189 *Data collection*190 Xcalibur, Atlas
diffractometer

191 Radiation source: Enhance (Mo) X-ray Source

192 Graphite monochromator

193 Detector resolution: 5.1636 pixels mm^{−1}194 ω scans

Absorption correction: multi-scan

CrysAlis PRO, Agilent Technologies, Version

1.171.37.33 (release 27-03-2014 CrysAlis171

.NET) (compiled Mar 27 2014, 17:12:48)

Empirical absorption correction using spherical
harmonics, implemented in SCALE3

ABSPACK scaling algorithm.

 $T_{\min} = 0.769$, $T_{\max} = 1.000$

12221 measured reflections

3504 independent reflections

2686 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.053$ $\theta_{\max} = 26.0^\circ$, $\theta_{\min} = 3.4^\circ$ $h = -7 \rightarrow 7$ $k = -12 \rightarrow 12$ $l = -17 \rightarrow 17$ 195 *Refinement*196 Refinement on F^2

197 Least-squares matrix: full

198 $R[F^2 > 2\sigma(F^2)] = 0.049$ 199 $wR(F^2) = 0.104$ 200 $S = 1.13$

201 3504 reflections

202 253 parameters

203 0 restraints

Primary atom site location: structure-invariant
direct methodsHydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.021P)^2 + 2.803P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} = 0.001$

$$\Delta\rho_{\max} = 0.94 \text{ e } \text{\AA}^{-3} \qquad \Delta\rho_{\min} = -0.72 \text{ e } \text{\AA}^{-3}$$

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 ($\text{\AA}^2$)

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
Br1	0.69172 (9)	0.45860 (5)	0.79186 (4)	0.01867 (17)
Br2	−0.13706 (9)	0.04259 (5)	0.20985 (4)	0.01959 (17)
F1	0.7975 (6)	0.4096 (5)	0.6225 (3)	0.0554 (14)
F2	0.7593 (7)	0.2410 (4)	0.6992 (3)	0.0600 (15)
F3	0.3387 (6)	0.4527 (4)	0.6086 (3)	0.0399 (10)
F4	0.3091 (7)	0.2778 (4)	0.6799 (2)	0.0442 (11)
F5	0.5053 (6)	0.3254 (4)	0.4718 (2)	0.0383 (10)
F6	0.4927 (6)	0.1494 (3)	0.5435 (3)	0.0388 (10)
F7	0.0458 (6)	0.3529 (3)	0.4598 (3)	0.0350 (10)
F8	0.0337 (6)	0.1758 (4)	0.5308 (2)	0.0326 (9)
F9	0.2375 (7)	0.2223 (4)	0.3250 (3)	0.0463 (12)
F10	0.2026 (6)	0.0481 (4)	0.3960 (3)	0.0392 (10)
F11	−0.2157 (8)	0.2589 (4)	0.3024 (3)	0.0672 (16)
F12	−0.2544 (6)	0.0894 (5)	0.3777 (3)	0.0560 (14)
C1	0.6716 (10)	0.3545 (6)	0.6780 (4)	0.0257 (15)
C2	0.4245 (9)	0.3390 (5)	0.6239 (4)	0.0166 (12)
C3	0.3941 (10)	0.2621 (6)	0.5280 (4)	0.0197 (13)
C4	0.1466 (9)	0.2400 (6)	0.4751 (4)	0.0182 (13)
C5	0.1192 (9)	0.1627 (6)	0.3800 (4)	0.0182 (13)
C6	−0.1253 (10)	0.1463 (6)	0.3240 (4)	0.0263 (15)
N1	0.7560 (7)	0.6409 (4)	0.9540 (3)	0.0157 (10)
N2	0.8071 (7)	0.8576 (4)	1.0479 (3)	0.0135 (10)
C7	0.9959 (8)	0.6855 (5)	0.9717 (4)	0.0154 (12)
H7A	1.0448	0.6926	0.9122	0.019*
H7B	1.0910	0.6238	1.0084	0.019*
C8	1.0238 (9)	0.8163 (6)	1.0254 (4)	0.0185 (13)
H8A	1.1358	0.8103	1.0831	0.022*
H8B	1.0779	0.8791	0.9871	0.022*
C9	0.6153 (9)	0.7392 (5)	0.9010 (4)	0.0175 (13)
H9A	0.4574	0.7116	0.8877	0.021*
H9B	0.6623	0.7507	0.8415	0.021*
C10	0.6408 (9)	0.8669 (5)	0.9587 (4)	0.0184 (13)
H10A	0.6897	0.9332	0.9218	0.022*
H10B	0.4947	0.8906	0.9724	0.022*
C11	0.6861 (9)	0.6296 (5)	1.0452 (4)	0.0166 (12)
H11A	0.7724	0.5633	1.0803	0.020*
H11B	0.5265	0.6057	1.0346	0.020*
C12	0.7259 (9)	0.7576 (5)	1.1030 (4)	0.0190 (13)

247	H12A	0.5852	0.7841	1.1206	0.023*
248	H12B	0.8372	0.7462	1.1602	0.023*

249 *Atomic displacement parameters ($\text{\AA}^2$)*

		U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
251	Br1	0.0226 (3)	0.0185 (4)	0.0139 (3)	−0.0010 (2)	0.0020 (2)	−0.0039 (2)
252	Br2	0.0238 (3)	0.0191 (4)	0.0138 (3)	0.0006 (2)	−0.0006 (2)	−0.0040 (2)
253	F1	0.029 (2)	0.109 (4)	0.030 (2)	−0.026 (2)	0.0163 (19)	−0.030 (2)
254	F2	0.063 (3)	0.039 (3)	0.057 (3)	0.034 (2)	−0.037 (2)	−0.027 (2)
255	F3	0.048 (2)	0.027 (2)	0.035 (2)	0.0166 (18)	−0.0128 (19)	−0.0137 (18)
256	F4	0.047 (2)	0.068 (3)	0.018 (2)	−0.033 (2)	0.0093 (18)	−0.006 (2)
257	F5	0.033 (2)	0.067 (3)	0.0161 (19)	−0.0233 (19)	0.0109 (16)	−0.0084 (19)
258	F6	0.042 (2)	0.023 (2)	0.041 (2)	0.0170 (17)	−0.0157 (18)	−0.0147 (18)
259	F7	0.038 (2)	0.027 (2)	0.032 (2)	0.0178 (16)	−0.0109 (17)	−0.0101 (17)
260	F8	0.030 (2)	0.053 (3)	0.0147 (18)	−0.0173 (17)	0.0070 (16)	−0.0038 (17)
261	F9	0.054 (3)	0.067 (3)	0.021 (2)	−0.036 (2)	0.0193 (19)	−0.011 (2)
262	F10	0.048 (2)	0.025 (2)	0.034 (2)	0.0192 (17)	−0.0139 (18)	−0.0131 (18)
263	F11	0.078 (3)	0.043 (3)	0.056 (3)	0.043 (2)	−0.043 (3)	−0.032 (2)
264	F12	0.030 (2)	0.107 (4)	0.032 (2)	−0.023 (2)	0.0144 (19)	−0.033 (2)
265	C1	0.030 (3)	0.025 (4)	0.021 (3)	0.004 (3)	0.003 (3)	−0.007 (3)
266	C2	0.020 (3)	0.015 (3)	0.015 (3)	0.001 (2)	0.004 (2)	0.002 (2)
267	C3	0.024 (3)	0.015 (3)	0.022 (3)	0.001 (2)	0.010 (3)	−0.002 (3)
268	C4	0.019 (3)	0.019 (3)	0.017 (3)	−0.001 (2)	0.003 (2)	0.003 (3)
269	C5	0.017 (3)	0.022 (4)	0.016 (3)	0.000 (2)	0.004 (2)	0.001 (3)
270	C6	0.024 (3)	0.028 (4)	0.025 (4)	0.007 (3)	0.001 (3)	−0.006 (3)
271	N1	0.016 (2)	0.017 (3)	0.013 (2)	−0.0002 (19)	0.001 (2)	−0.004 (2)
272	N2	0.016 (2)	0.008 (3)	0.016 (2)	−0.0036 (18)	0.0005 (19)	−0.0035 (19)
273	C7	0.012 (3)	0.019 (3)	0.014 (3)	0.003 (2)	0.001 (2)	−0.002 (2)
274	C8	0.017 (3)	0.023 (4)	0.014 (3)	−0.001 (2)	−0.001 (2)	0.001 (2)
275	C9	0.020 (3)	0.013 (3)	0.016 (3)	0.001 (2)	−0.003 (2)	−0.006 (2)
276	C10	0.022 (3)	0.016 (3)	0.016 (3)	0.005 (2)	0.001 (2)	−0.001 (2)
277	C11	0.019 (3)	0.012 (3)	0.019 (3)	−0.006 (2)	0.006 (2)	−0.002 (2)
278	C12	0.017 (3)	0.020 (3)	0.020 (3)	0.000 (2)	0.004 (2)	−0.001 (3)

279 *Geometric parameters ($\text{\AA}$, $^\circ$)*

280	Br1—C1	1.930 (6)	N1—C9	1.479 (7)
281	Br2—C6	1.937 (6)	N1—C11	1.472 (7)
282	F1—C1	1.349 (7)	N2—C8	1.468 (7)
283	F2—C1	1.328 (7)	N2—C10	1.476 (7)
284	F3—C2	1.313 (6)	N2—C12	1.477 (7)
285	F4—C2	1.343 (6)	C7—H7A	0.9700
286	F5—C3	1.341 (7)	C7—H7B	0.9700
287	F6—C3	1.334 (7)	C7—C8	1.545 (7)
288	F7—C4	1.341 (7)	C8—H8A	0.9700
289	F8—C4	1.346 (6)	C8—H8B	0.9700
290	F9—C5	1.337 (6)	C9—H9A	0.9700

291	F10—C5	1.318 (7)	C9—H9B	0.9700
292	F11—C6	1.327 (7)	C9—C10	1.545 (7)
293	F12—C6	1.351 (8)	C10—H10A	0.9700
294	C1—C2	1.541 (8)	C10—H10B	0.9700
295	C2—C3	1.560 (8)	C11—H11A	0.9700
296	C3—C4	1.545 (8)	C11—H11B	0.9700
297	C4—C5	1.553 (8)	C11—C12	1.549 (7)
298	C5—C6	1.537 (8)	C12—H12A	0.9700
299	N1—C7	1.482 (6)	C12—H12B	0.9700
300				
301	F1—C1—Br1	108.9 (4)	C8—N2—C10	108.2 (4)
302	F1—C1—C2	109.2 (5)	C8—N2—C12	107.9 (4)
303	F2—C1—Br1	109.5 (4)	C10—N2—C12	108.0 (4)
304	F2—C1—F1	107.4 (5)	N1—C7—H7A	109.5
305	F2—C1—C2	110.1 (5)	N1—C7—H7B	109.5
306	C2—C1—Br1	111.6 (4)	N1—C7—C8	110.8 (4)
307	F3—C2—F4	109.0 (5)	H7A—C7—H7B	108.1
308	F3—C2—C1	108.7 (5)	C8—C7—H7A	109.5
309	F3—C2—C3	108.7 (5)	C8—C7—H7B	109.5
310	F4—C2—C1	107.0 (5)	N2—C8—C7	110.8 (4)
311	F4—C2—C3	108.3 (4)	N2—C8—H8A	109.5
312	C1—C2—C3	115.0 (5)	N2—C8—H8B	109.5
313	F5—C3—C2	107.9 (4)	C7—C8—H8A	109.5
314	F5—C3—C4	108.0 (5)	C7—C8—H8B	109.5
315	F6—C3—F5	107.7 (5)	H8A—C8—H8B	108.1
316	F6—C3—C2	108.4 (5)	N1—C9—H9A	109.6
317	F6—C3—C4	108.9 (4)	N1—C9—H9B	109.6
318	C4—C3—C2	115.7 (5)	N1—C9—C10	110.1 (4)
319	F7—C4—F8	107.2 (5)	H9A—C9—H9B	108.1
320	F7—C4—C3	109.3 (4)	C10—C9—H9A	109.6
321	F7—C4—C5	109.0 (5)	C10—C9—H9B	109.6
322	F8—C4—C3	108.4 (4)	N2—C10—C9	111.3 (4)
323	F8—C4—C5	107.5 (4)	N2—C10—H10A	109.4
324	C3—C4—C5	115.1 (5)	N2—C10—H10B	109.4
325	F9—C5—C4	108.4 (5)	C9—C10—H10A	109.4
326	F9—C5—C6	106.9 (5)	C9—C10—H10B	109.4
327	F10—C5—F9	108.8 (5)	H10A—C10—H10B	108.0
328	F10—C5—C4	109.1 (5)	N1—C11—H11A	109.5
329	F10—C5—C6	107.8 (5)	N1—C11—H11B	109.5
330	C6—C5—C4	115.6 (5)	N1—C11—C12	110.6 (4)
331	F11—C6—Br2	109.5 (4)	H11A—C11—H11B	108.1
332	F11—C6—F12	107.1 (5)	C12—C11—H11A	109.5
333	F11—C6—C5	110.6 (5)	C12—C11—H11B	109.5
334	F12—C6—Br2	108.7 (4)	N2—C12—C11	110.8 (4)
335	F12—C6—C5	109.4 (5)	N2—C12—H12A	109.5
336	C5—C6—Br2	111.5 (4)	N2—C12—H12B	109.5
337	C9—N1—C7	108.0 (4)	C11—C12—H12A	109.5
338	C11—N1—C7	108.5 (4)	C11—C12—H12B	109.5

339	C11—N1—C9	108.1 (4)	H12A—C12—H12B	108.1
340				
341	Br1—C1—C2—F3	54.7 (6)	F10—C5—C6—Br2	−55.4 (6)
342	Br1—C1—C2—F4	−62.8 (5)	F10—C5—C6—F11	−177.4 (5)
343	Br1—C1—C2—C3	176.8 (4)	F10—C5—C6—F12	64.9 (6)
344	F1—C1—C2—F3	−65.8 (6)	C1—C2—C3—F5	−61.8 (6)
345	F1—C1—C2—F4	176.7 (5)	C1—C2—C3—F6	54.6 (6)
346	F1—C1—C2—C3	56.3 (7)	C1—C2—C3—C4	177.2 (5)
347	F2—C1—C2—F3	176.6 (5)	C2—C3—C4—F7	57.3 (6)
348	F2—C1—C2—F4	59.1 (6)	C2—C3—C4—F8	−59.3 (6)
349	F2—C1—C2—C3	−61.3 (7)	C2—C3—C4—C5	−179.7 (5)
350	F3—C2—C3—F5	60.3 (6)	C3—C4—C5—F9	−56.7 (6)
351	F3—C2—C3—F6	176.6 (4)	C3—C4—C5—F10	61.6 (6)
352	F3—C2—C3—C4	−60.8 (6)	C3—C4—C5—C6	−176.8 (5)
353	F4—C2—C3—F5	178.6 (5)	C4—C5—C6—Br2	−177.7 (4)
354	F4—C2—C3—F6	−65.1 (6)	C4—C5—C6—F11	60.3 (7)
355	F4—C2—C3—C4	57.5 (6)	C4—C5—C6—F12	−57.4 (7)
356	F5—C3—C4—F7	−63.7 (6)	N1—C7—C8—N2	2.6 (6)
357	F5—C3—C4—F8	179.7 (5)	N1—C9—C10—N2	3.2 (6)
358	F5—C3—C4—C5	59.3 (6)	N1—C11—C12—N2	4.1 (6)
359	F6—C3—C4—F7	179.6 (5)	C7—N1—C9—C10	−60.1 (5)
360	F6—C3—C4—F8	63.0 (6)	C7—N1—C11—C12	55.6 (6)
361	F6—C3—C4—C5	−57.4 (6)	C8—N2—C10—C9	56.6 (6)
362	F7—C4—C5—F9	66.4 (6)	C8—N2—C12—C11	−61.1 (5)
363	F7—C4—C5—F10	−175.2 (4)	C9—N1—C7—C8	57.2 (5)
364	F7—C4—C5—C6	−53.6 (7)	C9—N1—C11—C12	−61.3 (5)
365	F8—C4—C5—F9	−177.6 (5)	C10—N2—C8—C7	−59.6 (6)
366	F8—C4—C5—F10	−59.3 (6)	C10—N2—C12—C11	55.7 (6)
367	F8—C4—C5—C6	62.3 (7)	C11—N1—C7—C8	−59.7 (6)
368	F9—C5—C6—Br2	61.5 (6)	C11—N1—C9—C10	57.1 (5)
369	F9—C5—C6—F11	−60.6 (7)	C12—N2—C8—C7	57.0 (6)
370	F9—C5—C6—F12	−178.3 (5)	C12—N2—C10—C9	−60.0 (6)

371 (III)

372 *Crystal data*373 $\text{C}_8\text{Br}_2\text{F}_{16}\cdot\text{C}_6\text{H}_{12}\text{N}_2$ 374 $M_r = 672.08$ 375 Triclinic, $P\bar{1}$ 376 $a = 6.03367 (19) \text{ \AA}$ 377 $b = 10.4836 (5) \text{ \AA}$ 378 $c = 16.4471 (9) \text{ \AA}$ 379 $\alpha = 88.785 (4)^\circ$ 380 $\beta = 84.126 (3)^\circ$ 381 $\gamma = 89.724 (3)^\circ$ 382 $V = 1034.65 (8) \text{ \AA}^3$ $Z = 2$ $F(000) = 648$ $D_x = 2.157 \text{ Mg m}^{-3}$ Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 4902 reflections

 $\theta = 3.7\text{--}28.5^\circ$ $\mu = 4.07 \text{ mm}^{-1}$ $T = 150 \text{ K}$

Plate, colourless

 $0.14 \times 0.08 \times 0.04 \text{ mm}$

383 *Data collection*

384 SuperNova, Single source at offset, Eos
 diffractometer
 385 Radiation source: SuperNova (Mo) X-ray
 Source
 386 Mirror monochromator
 387 Detector resolution: 8.0714 pixels mm⁻¹
 388 ω scans

Absorption correction: multi-scan
CrysAlis PRO, Agilent Technologies, Version
 1.171.37.33 (release 27-03-2014 CrysAlis171
 .NET) (compiled Mar 27 2014,17:12:48)
 Empirical absorption correction using spherical
 harmonics, implemented in SCALE3
 ABSPACK scaling algorithm.
 $T_{\min} = 0.622$, $T_{\max} = 1.000$
 14918 measured reflections
 4680 independent reflections
 3723 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.042$
 $\theta_{\max} = 27.5^\circ$, $\theta_{\min} = 3.4^\circ$
 $h = -7 \rightarrow 7$
 $k = -13 \rightarrow 13$
 $l = -20 \rightarrow 21$

389 *Refinement*

390 Refinement on F^2
 391 Least-squares matrix: full
 392 $R[F^2 > 2\sigma(F^2)] = 0.050$
 393 $wR(F^2) = 0.122$
 394 $S = 1.07$
 395 4680 reflections
 396 307 parameters
 397 0 restraints
 398 Primary atom site location: iterative

Hydrogen site location: inferred from
 neighbouring sites
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0543P)^2 + 1.8052P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 1.00 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.69 \text{ e } \text{\AA}^{-3}$

399 *Special details*

400 **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.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
403 Br1	1.23740 (7)	0.52567 (4)	0.18081 (3)	0.02372 (14)
404 Br2	0.31570 (7)	0.97270 (4)	0.81774 (3)	0.02532 (14)
405 F1	1.3167 (7)	0.7383 (4)	0.2593 (3)	0.0833 (16)
406 F2	1.3831 (5)	0.5669 (5)	0.3265 (2)	0.0725 (13)
407 F3	0.8768 (5)	0.6934 (4)	0.28024 (19)	0.0543 (10)
408 F4	0.9322 (5)	0.5136 (3)	0.34060 (19)	0.0451 (8)
409 F5	1.0979 (5)	0.8127 (3)	0.3973 (2)	0.0527 (10)
410 F6	1.1252 (5)	0.6296 (3)	0.45864 (18)	0.0488 (9)
411 F7	0.6478 (5)	0.7905 (3)	0.40831 (18)	0.0478 (9)
412 F8	0.6682 (5)	0.6037 (3)	0.4658 (2)	0.0432 (8)
413 F9	0.8583 (5)	0.8949 (3)	0.5291 (2)	0.0516 (10)
414 F10	0.8853 (5)	0.7085 (3)	0.58647 (18)	0.0528 (10)
415 F11	0.4022 (5)	0.8649 (4)	0.53924 (19)	0.0505 (9)
416 F12	0.4369 (5)	0.6839 (3)	0.6011 (2)	0.0546 (10)

417	F13	0.5997 (6)	0.9860 (3)	0.6536 (2)	0.0494 (9)
418	F14	0.6676 (6)	0.8086 (4)	0.71488 (19)	0.0603 (11)
419	F15	0.1540 (5)	0.9307 (5)	0.6751 (2)	0.0803 (15)
420	F16	0.2275 (8)	0.7598 (4)	0.7402 (3)	0.098 (2)
421	C7	1.2412 (8)	0.6223 (5)	0.2782 (3)	0.0330 (11)
422	C8	1.0085 (6)	0.6313 (4)	0.3270 (3)	0.0208 (9)
423	C9	1.0034 (7)	0.6992 (4)	0.4097 (3)	0.0228 (9)
424	C10	0.7661 (7)	0.7177 (4)	0.4556 (3)	0.0206 (9)
425	C11	0.7649 (7)	0.7810 (4)	0.5399 (3)	0.0225 (9)
426	C12	0.5298 (7)	0.7974 (4)	0.5869 (3)	0.0242 (9)
427	C13	0.5292 (7)	0.8682 (4)	0.6694 (3)	0.0219 (9)
428	C14	0.3021 (8)	0.8752 (5)	0.7208 (3)	0.0378 (13)
429	N1	0.7315 (5)	0.6447 (3)	−0.0400 (2)	0.0189 (7)
430	N2	0.7042 (5)	0.8534 (3)	0.0414 (2)	0.0189 (7)
431	C1	0.8227 (7)	0.6268 (4)	0.0389 (3)	0.0238 (9)
432	H1A	0.9790	0.6037	0.0296	0.029*
433	H1B	0.7448	0.5575	0.0696	0.029*
434	C2	0.4988 (6)	0.6868 (4)	−0.0243 (3)	0.0217 (9)
435	H2A	0.4133	0.6216	0.0075	0.026*
436	H2B	0.4356	0.6983	−0.0759	0.026*
437	C3	0.8580 (7)	0.7475 (4)	−0.0859 (3)	0.0238 (9)
438	H3A	0.7961	0.7640	−0.1372	0.029*
439	H3B	1.0119	0.7211	−0.0981	0.029*
440	C4	0.8483 (7)	0.8712 (4)	−0.0353 (3)	0.0243 (9)
441	H4A	0.9972	0.8935	−0.0233	0.029*
442	H4B	0.7914	0.9409	−0.0670	0.029*
443	C5	0.4823 (6)	0.8127 (4)	0.0220 (3)	0.0217 (9)
444	H5A	0.4188	0.8783	−0.0111	0.026*
445	H5B	0.3845	0.8016	0.0723	0.026*
446	C6	0.7980 (7)	0.7497 (4)	0.0891 (3)	0.0242 (9)
447	H6A	0.7012	0.7336	0.1390	0.029*
448	H6B	0.9427	0.7750	0.1040	0.029*

449 *Atomic displacement parameters ($\text{\AA}^2$)*

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
450						
451	Br1	0.0275 (2)	0.0276 (3)	0.0159 (2)	0.00098 (16)	−0.00033 (16)
452	Br2	0.0299 (2)	0.0282 (3)	0.0170 (3)	−0.00127 (17)	0.00269 (17)
453	F1	0.106 (3)	0.060 (2)	0.071 (3)	−0.059 (2)	0.064 (2)
454	F2	0.0323 (17)	0.149 (4)	0.040 (2)	0.029 (2)	−0.0150 (15)
455	F3	0.059 (2)	0.087 (3)	0.0182 (17)	0.0405 (18)	−0.0105 (14)
456	F4	0.064 (2)	0.0318 (16)	0.0356 (19)	−0.0178 (14)	0.0164 (15)
457	F5	0.0584 (19)	0.0421 (18)	0.052 (2)	−0.0298 (15)	0.0295 (16)
458	F6	0.0370 (16)	0.093 (3)	0.0180 (16)	0.0294 (16)	−0.0096 (12)
459	F7	0.0454 (17)	0.080 (2)	0.0193 (16)	0.0333 (16)	−0.0079 (13)
460	F8	0.0454 (17)	0.0393 (17)	0.0413 (19)	−0.0196 (13)	0.0169 (14)
461	F9	0.0539 (18)	0.0433 (18)	0.052 (2)	−0.0283 (14)	0.0289 (16)
462	F10	0.0505 (18)	0.092 (3)	0.0169 (16)	0.0429 (18)	−0.0122 (13)

463	F11	0.0364 (16)	0.093 (3)	0.0236 (17)	0.0310 (16)	−0.0106 (13)	−0.0121 (17)
464	F12	0.063 (2)	0.0417 (18)	0.052 (2)	−0.0320 (15)	0.0318 (17)	−0.0249 (16)
465	F13	0.072 (2)	0.0342 (17)	0.0369 (19)	−0.0213 (15)	0.0241 (16)	−0.0136 (14)
466	F14	0.062 (2)	0.101 (3)	0.0196 (17)	0.049 (2)	−0.0153 (15)	−0.0143 (18)
467	F15	0.0312 (17)	0.165 (4)	0.049 (2)	0.033 (2)	−0.0147 (16)	−0.055 (3)
468	F16	0.133 (4)	0.071 (3)	0.075 (3)	−0.073 (3)	0.077 (3)	−0.050 (2)
469	C7	0.032 (2)	0.046 (3)	0.021 (3)	−0.009 (2)	0.0049 (19)	−0.012 (2)
470	C8	0.021 (2)	0.023 (2)	0.018 (2)	−0.0001 (16)	−0.0047 (16)	−0.0028 (18)
471	C9	0.021 (2)	0.029 (2)	0.018 (2)	−0.0047 (17)	−0.0010 (16)	−0.0028 (18)
472	C10	0.023 (2)	0.026 (2)	0.013 (2)	−0.0003 (16)	−0.0043 (16)	−0.0019 (17)
473	C11	0.022 (2)	0.027 (2)	0.019 (2)	−0.0018 (16)	−0.0033 (17)	−0.0048 (19)
474	C12	0.025 (2)	0.028 (2)	0.019 (2)	−0.0044 (17)	0.0010 (17)	−0.0046 (19)
475	C13	0.023 (2)	0.024 (2)	0.019 (2)	−0.0018 (16)	−0.0033 (16)	0.0005 (18)
476	C14	0.035 (3)	0.047 (3)	0.029 (3)	−0.012 (2)	0.012 (2)	−0.017 (2)
477	N1	0.0191 (16)	0.0215 (18)	0.0164 (19)	−0.0004 (13)	−0.0025 (13)	−0.0072 (15)
478	N2	0.0208 (17)	0.0172 (17)	0.018 (2)	0.0011 (13)	−0.0004 (13)	−0.0043 (14)
479	C1	0.030 (2)	0.021 (2)	0.022 (2)	0.0070 (17)	−0.0097 (18)	−0.0025 (18)
480	C2	0.0173 (19)	0.025 (2)	0.023 (2)	−0.0029 (15)	−0.0036 (16)	−0.0046 (18)
481	C3	0.026 (2)	0.025 (2)	0.019 (2)	−0.0050 (17)	0.0036 (17)	−0.0069 (18)
482	C4	0.025 (2)	0.022 (2)	0.024 (3)	−0.0053 (16)	0.0036 (17)	−0.0060 (18)
483	C5	0.0177 (19)	0.027 (2)	0.021 (2)	0.0033 (16)	−0.0028 (16)	−0.0040 (18)
484	C6	0.031 (2)	0.023 (2)	0.020 (2)	0.0058 (17)	−0.0081 (18)	−0.0033 (18)

485 *Geometric parameters (Å, °)*

486	Br1—C7	1.915 (5)	C12—C13	1.560 (6)
487	Br2—C14	1.921 (5)	C13—C14	1.537 (6)
488	F1—C7	1.321 (6)	N1—C1	1.469 (5)
489	F2—C7	1.347 (6)	N1—C2	1.469 (5)
490	F3—C8	1.321 (5)	N1—C3	1.472 (5)
491	F4—C8	1.326 (5)	N2—C4	1.466 (5)
492	F5—C9	1.325 (5)	N2—C5	1.474 (5)
493	F6—C9	1.346 (5)	N2—C6	1.471 (5)
494	F7—C10	1.334 (5)	C1—H1A	0.9700
495	F8—C10	1.336 (5)	C1—H1B	0.9700
496	F9—C11	1.323 (5)	C1—C6	1.543 (6)
497	F10—C11	1.333 (5)	C2—H2A	0.9700
498	F11—C12	1.343 (5)	C2—H2B	0.9700
499	F12—C12	1.323 (5)	C2—C5	1.536 (5)
500	F13—C13	1.320 (5)	C3—H3A	0.9700
501	F14—C13	1.323 (5)	C3—H3B	0.9700
502	F15—C14	1.348 (7)	C3—C4	1.554 (6)
503	F16—C14	1.314 (6)	C4—H4A	0.9700
504	C7—C8	1.547 (6)	C4—H4B	0.9700
505	C8—C9	1.547 (6)	C5—H5A	0.9700
506	C9—C10	1.562 (6)	C5—H5B	0.9700
507	C10—C11	1.549 (6)	C6—H6A	0.9700
508	C11—C12	1.555 (6)	C6—H6B	0.9700

509				
510	F1—C7—Br1	109.7 (3)	F16—C14—F15	106.7 (5)
511	F1—C7—F2	107.2 (5)	F16—C14—C13	110.3 (4)
512	F1—C7—C8	109.4 (4)	C13—C14—Br2	112.0 (3)
513	F2—C7—Br1	109.2 (3)	C1—N1—C3	108.0 (3)
514	F2—C7—C8	108.7 (4)	C2—N1—C1	108.3 (3)
515	C8—C7—Br1	112.5 (3)	C2—N1—C3	108.0 (3)
516	F3—C8—F4	108.8 (4)	C4—N2—C5	108.6 (3)
517	F3—C8—C7	107.4 (4)	C4—N2—C6	108.2 (3)
518	F3—C8—C9	108.9 (3)	C6—N2—C5	107.8 (3)
519	F4—C8—C7	107.8 (3)	N1—C1—H1A	109.5
520	F4—C8—C9	108.7 (4)	N1—C1—H1B	109.5
521	C9—C8—C7	115.1 (3)	N1—C1—C6	110.9 (3)
522	F5—C9—F6	108.2 (4)	H1A—C1—H1B	108.0
523	F5—C9—C8	108.9 (4)	C6—C1—H1A	109.5
524	F5—C9—C10	108.4 (3)	C6—C1—H1B	109.5
525	F6—C9—C8	108.1 (3)	N1—C2—H2A	109.4
526	F6—C9—C10	108.0 (4)	N1—C2—H2B	109.4
527	C8—C9—C10	115.0 (3)	N1—C2—C5	111.1 (3)
528	F7—C10—F8	108.4 (4)	H2A—C2—H2B	108.0
529	F7—C10—C9	108.0 (4)	C5—C2—H2A	109.4
530	F7—C10—C11	108.6 (3)	C5—C2—H2B	109.4
531	F8—C10—C9	108.4 (3)	N1—C3—H3A	109.6
532	F8—C10—C11	109.1 (4)	N1—C3—H3B	109.6
533	C11—C10—C9	114.2 (3)	N1—C3—C4	110.4 (3)
534	F9—C11—F10	109.2 (4)	H3A—C3—H3B	108.1
535	F9—C11—C10	108.7 (4)	C4—C3—H3A	109.6
536	F9—C11—C12	108.4 (3)	C4—C3—H3B	109.6
537	F10—C11—C10	108.2 (3)	N2—C4—C3	110.6 (3)
538	F10—C11—C12	107.6 (4)	N2—C4—H4A	109.5
539	C10—C11—C12	114.7 (4)	N2—C4—H4B	109.5
540	F11—C12—C11	108.5 (4)	C3—C4—H4A	109.5
541	F11—C12—C13	107.7 (3)	C3—C4—H4B	109.5
542	F12—C12—F11	107.7 (4)	H4A—C4—H4B	108.1
543	F12—C12—C11	109.4 (3)	N2—C5—C2	110.6 (3)
544	F12—C12—C13	109.1 (4)	N2—C5—H5A	109.5
545	C11—C12—C13	114.2 (4)	N2—C5—H5B	109.5
546	F13—C13—F14	109.1 (4)	C2—C5—H5A	109.5
547	F13—C13—C12	108.6 (4)	C2—C5—H5B	109.5
548	F13—C13—C14	108.0 (4)	H5A—C5—H5B	108.1
549	F14—C13—C12	108.7 (4)	N2—C6—C1	110.5 (4)
550	F14—C13—C14	107.0 (4)	N2—C6—H6A	109.5
551	C14—C13—C12	115.4 (4)	N2—C6—H6B	109.5
552	F15—C14—Br2	108.7 (3)	C1—C6—H6A	109.5
553	F15—C14—C13	108.7 (4)	C1—C6—H6B	109.5
554	F16—C14—Br2	110.2 (4)	H6A—C6—H6B	108.1
555				
556	Br1—C7—C8—F3	−63.1 (4)	F13—C13—C14—Br2	−55.0 (5)

557	Br1—C7—C8—F4	54.0 (5)	F13—C13—C14—F15	65.2 (5)
558	Br1—C7—C8—C9	175.5 (3)	F13—C13—C14—F16	−178.2 (5)
559	F1—C7—C8—F3	59.2 (5)	F14—C13—C14—Br2	62.4 (5)
560	F1—C7—C8—F4	176.2 (4)	F14—C13—C14—F15	−177.5 (4)
561	F1—C7—C8—C9	−62.3 (6)	F14—C13—C14—F16	−60.8 (6)
562	F2—C7—C8—F3	175.9 (4)	C7—C8—C9—F5	53.5 (5)
563	F2—C7—C8—F4	−67.0 (5)	C7—C8—C9—F6	−63.8 (5)
564	F2—C7—C8—C9	54.5 (5)	C7—C8—C9—C10	175.4 (4)
565	F3—C8—C9—F5	−67.1 (5)	C8—C9—C10—F7	−61.4 (5)
566	F3—C8—C9—F6	175.6 (4)	C8—C9—C10—F8	55.9 (5)
567	F3—C8—C9—C10	54.8 (5)	C8—C9—C10—C11	177.7 (4)
568	F4—C8—C9—F5	174.5 (3)	C9—C10—C11—F9	59.7 (5)
569	F4—C8—C9—F6	57.1 (4)	C9—C10—C11—F10	−58.8 (5)
570	F4—C8—C9—C10	−63.6 (5)	C9—C10—C11—C12	−178.8 (3)
571	F5—C9—C10—F7	60.8 (5)	C10—C11—C12—F11	−56.5 (5)
572	F5—C9—C10—F8	178.0 (4)	C10—C11—C12—F12	60.7 (5)
573	F5—C9—C10—C11	−60.1 (5)	C10—C11—C12—C13	−176.7 (4)
574	F6—C9—C10—F7	177.8 (4)	C11—C12—C13—F13	64.0 (5)
575	F6—C9—C10—F8	−64.9 (4)	C11—C12—C13—F14	−54.6 (5)
576	F6—C9—C10—C11	56.9 (5)	C11—C12—C13—C14	−174.8 (4)
577	F7—C10—C11—F9	−60.9 (5)	C12—C13—C14—Br2	−176.6 (3)
578	F7—C10—C11—F10	−179.4 (3)	C12—C13—C14—F15	−56.4 (5)
579	F7—C10—C11—C12	60.6 (5)	C12—C13—C14—F16	60.2 (6)
580	F8—C10—C11—F9	−178.8 (3)	N1—C1—C6—N2	−3.9 (5)
581	F8—C10—C11—F10	62.7 (4)	N1—C2—C5—N2	−2.6 (5)
582	F8—C10—C11—C12	−57.3 (5)	N1—C3—C4—N2	−3.7 (5)
583	F9—C11—C12—F11	65.1 (5)	C1—N1—C2—C5	59.6 (4)
584	F9—C11—C12—F12	−177.6 (4)	C1—N1—C3—C4	−56.4 (4)
585	F9—C11—C12—C13	−55.0 (5)	C2—N1—C1—C6	−55.8 (4)
586	F10—C11—C12—F11	−176.9 (4)	C2—N1—C3—C4	60.5 (4)
587	F10—C11—C12—F12	−59.6 (5)	C3—N1—C1—C6	60.9 (4)
588	F10—C11—C12—C13	62.9 (5)	C3—N1—C2—C5	−57.1 (4)
589	F11—C12—C13—F13	−56.6 (4)	C4—N2—C5—C2	59.9 (4)
590	F11—C12—C13—F14	−175.2 (4)	C4—N2—C6—C1	−56.5 (4)
591	F11—C12—C13—C14	64.6 (5)	C5—N2—C4—C3	−56.1 (4)
592	F12—C12—C13—F13	−173.3 (4)	C5—N2—C6—C1	60.8 (4)
593	F12—C12—C13—F14	68.1 (5)	C6—N2—C4—C3	60.7 (4)
594	F12—C12—C13—C14	−52.0 (5)	C6—N2—C5—C2	−57.2 (4)

595 **other supporting information**

596 Crystallographic Information File. uk3139.cif

597 Structure factors. uk3139Isup2.hkl

598 MDL mol file. uk3139Isup5.mol

599 Structure factors. uk3139IIsup3.hkl

600 MDL mol file. uk3139IIsup6.mol

601 Structure factors. uk3139IIIsup4.hkl

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