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Crystal structure and Hirshfeld surface analysis of (*E*)-1-{2,2-dibromo-1-[4-(*tert*-butyl)phenyl]-ethenyl}-2-(3,4-dimethylphenyl)diazene

Namig G. Shikhaliyev,^a Jonathan Cisterna,^b Ayten M. Gajar,^c Ayten A. Niyazova,^d Ali N. Khalilov,^d Victor N. Khrustalev,^{e,f} Alebel N. Belay^{g,*} and Abel M. Maharramov^c

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^aDepartment of Chemical Engineering, Baku Engineering University, Hasan Aliyev Str. 120, AZ 0101 Baku, Azerbaijan,

^bDepartamento de Química, Universidad Católica del Norte, Av. Angamos 0610, Antofagasta, Chile, ^cDepartment of Chemistry, Baku State University, Z. Khalilov str. 23, Az 1148, Baku, Azerbaijan, ^d"Composite Materials" Scientific Research Center, Azerbaijan State Economic University (UNEC), Murtuza Mukhtarov str. 194, Az 1065, Baku, Azerbaijan,

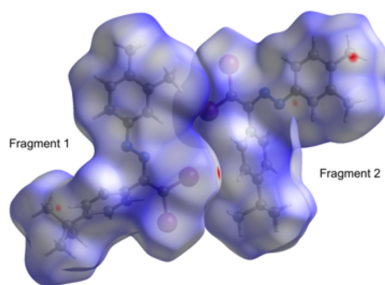
^ePeoples' Friendship University of Russia (RUDN University), Miklukho-Maklay St. 6, Moscow, 117198, Russian Federation, ^fN. D. Zelinsky Institute of Organic Chemistry RAS, Leninsky Prospekt. 47, Moscow, 119991, Russian Federation, and ^gDepartment of Chemistry, Bahir Dar University, PO Box 79, Bahir Dar, Ethiopia. *Correspondence e-mail: alebel.nibret@bdu.edu.et

The title azo compound, C₂₀H₂₂Br₂N₂, crystallizes in the triclinic space group *P* $\bar{1}$ (No. 2) with two independent molecules in the asymmetric unit. The molecular structure was analyzed using spectroscopic and crystallographic techniques, confirming the expected configuration and electronic environment. Hirshfeld surface analysis revealed key non-covalent interactions such as C—H... π , C—H...Br, π — π stacking, and halogen bonding, which consolidate the crystal structure. The study provides valuable insights into the structural features and intermolecular interactions of this brominated azo compound, which may have potential applications in optoelectronic materials, dyes, and molecular switches.

1. Chemical context

Azo compounds represent a significant class of organic molecules characterized by a diazenyl (—N=N—) functional group linking two aromatic systems (Shikhaliyev *et al.*, 2018; Naghiyev *et al.*, 2019). These compounds have garnered considerable interest due to their diverse applications in dyes, pigments, pharmaceuticals, and advanced materials, including liquid crystals and organic semiconductors (Shixaliyev *et al.*, 2013; Shikhaliyev *et al.*, 2019; Tahirli *et al.*, 2024). The introduction of various substituents on the aromatic rings can fine-tune their electronic, optical, and chemical properties, making them highly versatile for industrial and scientific applications (Gurbanov *et al.*, 2021; Mahmoudi *et al.*, 2021).

The structure under investigation features a dibromovinylidenebenzyl moiety and a methyl-substituted phenyl group linked *via* an azo (—N=N—) bridge (Israyilova *et al.*, 2016; Çelikesir *et al.*, 2022). The dibromoalkene moiety enhances the reactivity, allowing for further functionalization and potential applications in cross-coupling reactions, while alkyl substituents on the phenyl ring contribute to improved solubility and stability (Dobrounig *et al.*, 2017; Atioğlu *et al.*, 2022*a,b*; Khalilov *et al.*, 2022). Although the *tert*-butylphenyl ring is electronically decoupled due to the lack of conjugation with the azo and vinyl units, the compound still contains a conjugated π -system involving the diazo group and the *p*-methylphenyl ring. This partial conjugation may contribute to interesting photophysical and electronic properties, which are

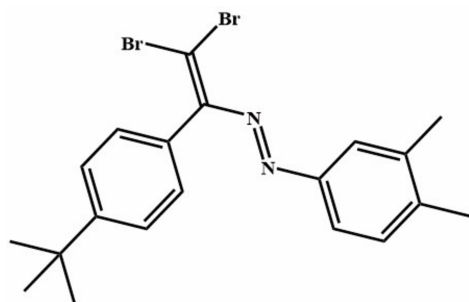


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relevant for potential applications in azo dyes, molecular switches, and optoelectronic materials, azo dyes, and molecular switches (Akkurt *et al.*, 2022; Shikhaliyev *et al.*, 2021).

While synthetic azo dyes have been widely studied and commercially utilized, naturally occurring dyes containing the azo ($-\text{N}=\text{N}-$) functional group are infrequent (Özkaraca *et al.*, 2020*a,b*). Most natural dyes are based on anthraquinone, flavonoid, or indigoid chromophores. Despite their limited natural occurrence, azo compounds are extensively synthesized because of their excellent color stability, tunability, and strong chromophoric properties (Magerramov *et al.*, 2018; Benkhaya *et al.*, 2020).



Given the significance of azo compounds in material science and organic synthesis, the present study reports the synthesis and crystal structure of (*E*)-1-[2,2-dibromo-1-[4-(*tert*-butyl)phenyl]ethenyl]-2-(3,4-dimethylphenyl)diazene in which the dibromovinyl and *tert*-butyl groups were incorporated to modulate the azo scaffold's reactivity and solubility profiles.

2. Structural commentary

The title compound crystallizes in the triclinic cell system with space group $P\bar{1}$ (No. 2), with two molecules in the asymmetric unit ($Z = 4$). The molecular structure of the compound agrees with the spectroscopic characterization and the proposed structure, in a centrosymmetric setting, with normal bond distances (Allen *et al.*, 1987) and angles, being the *E*-isomer confirmed as molecular structure within the crystal (see Fig. 1).

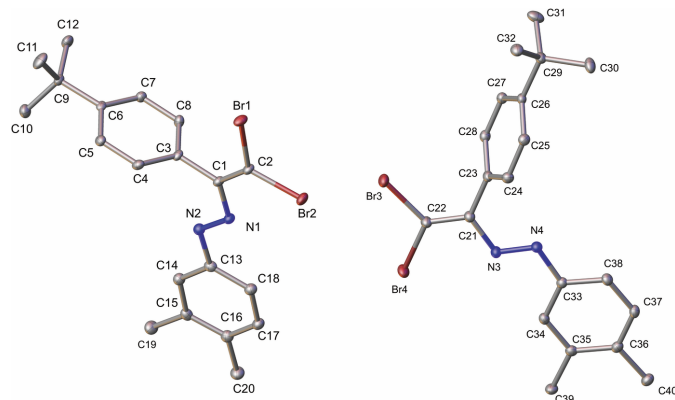


Figure 1
Ellipsoid plots (30% probability) of fragments 1(left) and 2 (right). Hydrogen atoms are omitted for the sake of clarity.

The independent molecules are labeled fragment 1 and 2, correlative and sorted atom labels, The diazo bond environments in each molecule are practically coplanar, apart from the 4-*tert*-butylphenyl moiety in both cases. There is a lack of conjugation between the *tert*-butylphenyl group and the adjacent vinyl group, with dihedral angles of 89.2 (2) and 66.8 (2)° for fragments 1 and 2, respectively. The dihedral angles between these two fragments indicate that in both cases the aromatic ring is twisted out of planarity relative to the vinyl moiety, effectively insulating it electronically from the azo group. This lack of conjugation may have important implications for the electronic distribution and potential reactivity of the molecule.

The origin of this non-conjugated geometry warrants further consideration. One possible explanation is steric hindrance introduced by the bulky *tert*-butyl substituent, which may prevent the coplanarity required for extended π -conjugation. Alternatively, this conformation could be stabilized or enforced by crystal-packing effects, as intermolecular forces may favor a geometry that minimizes unfavorable contacts or facilitates specific packing motifs, such as herringbone arrangements.

3. Supramolecular features

The crystal packing of the title compound does not exhibit geometrical parameters consistent with classical hydrogen bonds (Steiner, 2002). Instead, the structure is consolidated by intermolecular interactions such as $\text{H}\cdots\pi$, $\text{C}-\text{H}\cdots\text{Br}$, halogen bonds, and parallel-displaced π - π stacking (see Fig. 2). The π - π interactions exceed conventional parameters for this type of interaction (Carter-Fenk & Herbert, 2020). The centroid-centroid distances are 5.5060 (14) and 5.9474 (13) Å (red dotted lines), with perpendicular ring plane separations of 3.1104 (9) and 2.2010 (9) Å (blue dotted lines). The slip-page values are 4.543 and 5.060 Å (green dotted lines), observed between the C13-C18 ring and its symmetry equivalent at $1 - x, 1 - y, 2 - z$ and between the C13-C18 and C33-C38($x, 2 - y, 1 - z$) rings, respectively (Fig. 3).

It is worth noting that a Z' value of 2 for two conformationally identical molecules is uncommon. This appears to arise from the fact that, although the molecules adopt a herringbone packing motif, they cannot do so without a rela-

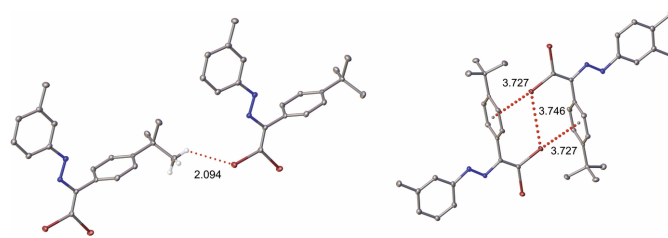


Figure 2
 $\text{C}-\text{H}\cdots\text{Br}$ interactions and halogen bonds found in the crystal structure of the title compound.

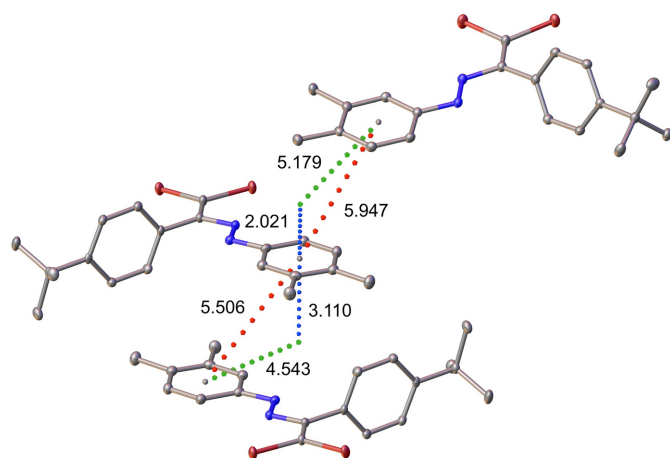


Figure 3
 π - π -like stacking in the title compound.

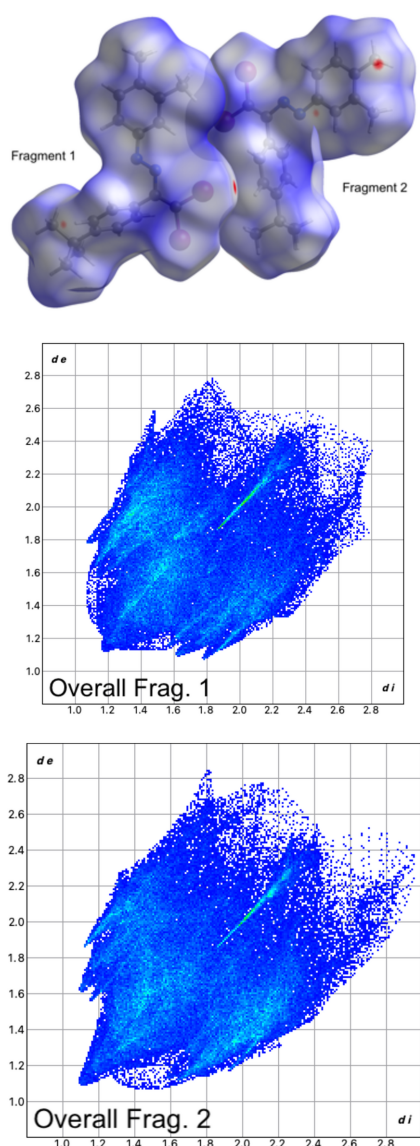


Figure 4
Overall Hirshfeld surface for fragments 1 and 2 and their respective overall fingerprint plots.

tive offset due to the dihedral angle. This observation suggests that the molecular conformation is particularly stable.

Hirshfeld surface analysis was conducted for each fragment individually to gain a deeper understanding of all contributions. These interactions are visualized as red ($d_{\text{norm}} < \text{vdW radii}$), white ($d_{\text{norm}} = \text{vdW radii}$), and blue ($d_{\text{norm}} > \text{vdW radii}$) spots on the d_{norm} surfaces for all compounds along with fingerprint plots mapped with d_{norm} (where $d_{\text{norm}} = d_i + d_e$) (Fig. 4). $\text{H} \cdots \text{Br}$ contacts are observed to contribute around 20% with $d_i + d_e \simeq 2.1 \text{ \AA}$, generating a dimeric setting between the two fragments within the crystal structure. This is observed in similar compounds with a similar behavior without the formation of supramolecular aggregates in the crystal structure (Maharramov *et al.* 2019). $\text{H} \cdots \pi$ contacts contribute around 20% with $d_i + d_e \simeq 3.0 \text{ \AA}$, generating the stacking between the fragments along the a -axis direction. Although weaker than conventional hydrogen bonds, these interactions can play a crucial role in crystal packing, molecular recognition, and other supramolecular processes (Nagarajan *et al.* 2014; Rajagopal *et al.* 2016). $\text{H} \cdots \text{H}$ contacts and π - π contacts make negligible contributions. $\text{Br} \cdots \text{Br}$ contacts are observed with a contribution of around 3.5%, and $d_i + d_e \simeq 3.7$ and $4.5 \text{ \AA}$, generating a tetramer in the crystal structure along the b -axis direction. These interactions, although often weak, are recognized for their capacity to influence the overall packing and consolidation of molecular crystals (Marek *et al.* 2018; Cavallo *et al.* 2016; Varadwaj *et al.* 2019).

It is also worth noting that the presence of electron-donating substituents on the molecule can modulate the electron-density distribution, potentially altering the nature and strength of interactions involving bromine atoms. This suggests that substituent effects may play a role not only in intramolecular properties, but also in dictating the intermolecular landscape of the crystal packing.

4. Database survey

A Cambridge Structural Database (CSD; 2022.3 Version, November 2024 update; Groom *et al.*, 2016) search for the (*E*)-1-[2,2-dibromo-1-[4-(*tert*-butyl)phenyl]vinyl]-2-(3,4-dimethylphenyl)diazene unit yielded 90 hits. Refining the search to include structures containing Br atoms gave just four compounds closely resembling the title compound, *viz.* ECUDAL [(I); Atioğlu *et al.*, 2022*b*], HEHKEO [(II); Akkurt *et al.*, 2022], PAXDOL [(III); Çelikesir *et al.*, 2022], and TAZDIL [(IV); Atioğlu *et al.*, 2022*a*].

In the crystal structure of (I), the molecules form inversion dimers *via* short halogen-halogen contacts [$\text{Br}3 \cdots \text{Br}3 = 4.0103(6) \text{ \AA}$, $\text{C}12-\text{Br}3 \cdots \text{Br}3 = 72.23(6)^\circ$], which are shorter than the van der Waals radius sum of $5.55 \text{ \AA}$ for two bromine atoms. Additional directional interactions include $\text{Br}1 \cdots \text{O}2$ contacts [$3.137(19) \text{ \AA}$] along the $[010]$ direction and π - π stacking interactions between aromatic rings are also present, with centroid-centroid distances of $3.7305(11) \text{ \AA}$ [perpendicular ring plane separations of $3.3964(8)$ and $3.1119(8) \text{ \AA}$, and slippage values of 2.057 and $1.543 \text{ \AA}$].

In (II), the structure features the same halogen contacts as in (I), but additionally presents $\pi \cdots \text{Br}$ contacts with a Br1–4-nitro mono-substituted ring distance of 3.546 (17) Å. Unlike in compound (I), no π – π stacking interactions are observed in this structure.

The crystal structure of (III) exhibits intermolecular hydrogen-bonding interactions between the aromatic rings and the oxygen of the nitro moiety [$D \cdots \text{H} = 2.51$ Å, $D \cdots \text{O} = 3.3244$ (18) Å, $D-\text{H} \cdots \text{O} = 144^\circ$. $\text{Br} \cdots \text{O}$ contacts (2.983 Å) are also present.

Finally in (IV), several hydrogen-bonding interactions consolidate the crystal structure due to the inversion center present in the unit cell. π – π stacking interactions are found, but at larger distances than conventional values (weighted distance = 4.079 Å). The structure also exhibits halogen– π interactions, with a mean distance of 3.503 Å.

5. Synthesis and crystallization

The title compound was obtained using a previously reported procedure (Shikhaliyev *et al.*, 2018), with appropriate modifications for the brominated analog. A 20 ml screw-neck vial was charged with DMSO (10 mL), (*E*)-1-[4-(*tert*-butyl)benzylidene]-2-(3,4-dimethylphenyl)hydrazine (281 mg, 1 mmol), tetramethylethylenediamine (TMEDA) (295 mg, 2.5 mmol), CuCl (2 mg, 0.02 mmol) and CBr_4 (3 mmol). After 1–3 h (until TLC analysis showed complete consumption of corresponding Schiff base), the reaction mixture was poured into an 0.01 M solution of HCl (100 ml, pH = 2), and extracted with dichloromethane (3×20 ml). The combined organic phase was washed with water (3×50 ml) and brine (30 ml), dried over anhydrous Na_2SO_4 and concentrated *in vacuo*. The residue was purified by column chromatography on silica gel using appropriate mixtures of hexane and dichloromethane (3:1, *v/v*). The resulting title compound was obtained as an orange crystalline solid, m.p. 388 K; yield: 34%.

^1H NMR (300 MHz, CDCl_3) δ 7.69 (*s*, 1H), 7.67 (*d*, $J = 8$ Hz, 1H), 7.54 (*d*, $J = 8.4$ Hz, 2H), 7.32 (*d*, $J = 8$ Hz, 1H), 7.21 (*d*, $J = 8.4$ Hz, 2H), 2.33 (*s*, 6H), 1.44 (*s*, 9H). ^{13}C NMR (75 MHz, CDCl_3) δ 153.7, 152.3, 151.9, 142.6, 138.5, 134.6, 132.5, 127.9, 127.7, 125.7, 123.8, 121.5, 34.5, 31.9, 20.7, 19.4.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. Hydrogen atoms were found in difference-Fourier maps, and included in the refinement using riding models, with constrained distances set to 0.95 Å (Csp^2-H) and 0.98 Å (RCH_3). $U_{\text{iso}}(\text{H})$ parameters were set to values of either 1.2 U_{eq} or 1.5 U_{eq} (RCH_3 only) of the attached atom.

Acknowledgements

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Table 1

Experimental details.

Crystal data	
Chemical formula	$\text{C}_{20}\text{H}_{22}\text{Br}_2\text{N}_2$
M_r	450.20
Crystal system, space group	Triclinic, $P\bar{1}$
Temperature (K)	100
a, b, c (Å)	9.89357 (5), 12.23384 (7), 17.12566 (9)
α, β, γ ($^\circ$)	76.6038 (5), 89.3055 (4), 73.3449 (5)
V (Å ³)	1928.56 (2)
Z	4
Radiation type	Cu $K\alpha$
μ (mm ^{−1})	5.34
Crystal size (mm)	0.31 $\times$ 0.25 $\times$ 0.21
Data collection	
Diffraction meter	XtaLAB Synergy, Dualflex, HyPix
Absorption correction	Multi-scan (CrysAlis PRO; Rigaku OD, 2021)
$T_{\text{min}}, T_{\text{max}}$	0.504, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	59739, 8330, 7876
R_{int}	0.049
$(\sin \theta/\lambda)_{\text{max}}$ (Å ^{−1})	0.638
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.035, 0.098, 1.09
No. of reflections	8330
No. of parameters	443
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å ^{−3})	1.01, −0.86

Computer programs: CrysAlis PRO (Rigaku OD, 2021), SHELXT (Sheldrick, 2015a), SHELXL (Sheldrick, 2015b) and SHELXTL (Sheldrick, 2008).

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Crystal structure and Hirshfeld surface analysis of (*E*)-1-{2,2-dibromo-1-[4-(*tert*-butyl)phenyl]ethenyl}-2-(3,4-dimethylphenyl)diazene

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Computing details

(*E*)-1-{2,2-Dibromo-1-[4-(*tert*-butyl)phenyl]ethenyl}-2-(3,4-dimethylphenyl)diazene

Crystal data

$C_{20}H_{22}Br_2N_2$

$M_r = 450.20$

Triclinic, $P\bar{1}$

$a = 9.89357(5) \text{ \AA}$

$b = 12.23384(7) \text{ \AA}$

$c = 17.12566(9) \text{ \AA}$

$\alpha = 76.6038(5)^\circ$

$\beta = 89.3055(4)^\circ$

$\gamma = 73.3449(5)^\circ$

$V = 1928.56(2) \text{ \AA}^3$

$Z = 4$

$F(000) = 904$

$D_x = 1.551 \text{ Mg m}^{-3}$

Cu $K\alpha$ radiation, $\lambda = 1.54184 \text{ \AA}$

Cell parameters from 36360 reflections

$\theta = 2.7\text{--}79.4^\circ$

$\mu = 5.34 \text{ mm}^{-1}$

$T = 100 \text{ K}$

Prism, red

$0.31 \times 0.25 \times 0.21 \text{ mm}$

Data collection

XtaLAB Synergy, Dualflex, HyPix
diffractometer

Radiation source: micro-focus sealed X-ray tube

Detector resolution: $0.78 \text{ pixels mm}^{-1}$

φ and ω scans

Absorption correction: multi-scan
(CrysAlisPro; Rigaku OD, 2021)

$T_{\min} = 0.504$, $T_{\max} = 1.000$

59739 measured reflections

8330 independent reflections

7876 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.049$

$\theta_{\max} = 79.6^\circ$, $\theta_{\min} = 2.7^\circ$

$h = -11 \rightarrow 12$

$k = -15 \rightarrow 15$

$l = -21 \rightarrow 21$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.035$

$wR(F^2) = 0.098$

$S = 1.09$

8330 reflections

443 parameters

0 restraints

Primary atom site location: difference Fourier
map

Secondary atom site location: difference Fourier
map

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0523P)^2 + 1.7466P]$

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

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 1.01 \text{ e \AA}^{-3}$

$\Delta\rho_{\min} = -0.86 \text{ e \AA}^{-3}$

Special details

Experimental. CrysAlisPro 1.171.41.117a (Rigaku Oxford Diffraction, 2021) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

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

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.39020 (3)	0.65399 (2)	0.49638 (2)	0.02673 (8)
Br2	0.34648 (3)	0.87116 (2)	0.57292 (2)	0.02590 (8)
N1	0.35259 (18)	0.69310 (16)	0.72925 (11)	0.0193 (3)
N2	0.34989 (19)	0.62342 (17)	0.79595 (11)	0.0209 (3)
C1	0.3699 (2)	0.63915 (18)	0.66366 (12)	0.0189 (4)
C2	0.3679 (2)	0.70933 (19)	0.59018 (13)	0.0210 (4)
C3	0.3979 (2)	0.50950 (18)	0.67648 (12)	0.0188 (4)
C4	0.2892 (2)	0.4581 (2)	0.67752 (13)	0.0216 (4)
H4	0.1939	0.5062	0.6681	0.026*
C5	0.3195 (2)	0.33693 (19)	0.69232 (13)	0.0208 (4)
H5	0.2441	0.3030	0.6934	0.025*
C6	0.4588 (2)	0.26337 (18)	0.70571 (11)	0.0177 (4)
C7	0.5661 (2)	0.31621 (19)	0.70445 (13)	0.0201 (4)
H7	0.6616	0.2685	0.7134	0.024*
C8	0.5364 (2)	0.43807 (19)	0.69034 (13)	0.0222 (4)
H8	0.6114	0.4722	0.6902	0.027*
C9	0.4882 (2)	0.12987 (18)	0.72170 (12)	0.0200 (4)
C10	0.4240 (3)	0.0863 (2)	0.80055 (15)	0.0304 (5)
H10A	0.4445	0.0007	0.8118	0.046*
H10B	0.3215	0.1227	0.7957	0.046*
H10C	0.4651	0.1076	0.8446	0.046*
C11	0.4221 (3)	0.0985 (2)	0.65293 (17)	0.0360 (6)
H11A	0.4594	0.1299	0.6021	0.054*
H11B	0.3193	0.1326	0.6502	0.054*
H11C	0.4453	0.0129	0.6624	0.054*
C12	0.6470 (3)	0.0659 (2)	0.72932 (16)	0.0295 (5)
H12A	0.6912	0.0835	0.7739	0.044*
H12B	0.6898	0.0923	0.6792	0.044*
H12C	0.6614	−0.0190	0.7397	0.044*
C13	0.3361 (2)	0.6775 (2)	0.86203 (13)	0.0221 (4)
C14	0.3347 (2)	0.6052 (2)	0.93741 (13)	0.0235 (4)
H14	0.3405	0.5252	0.9416	0.028*
C15	0.3249 (2)	0.6471 (2)	1.00761 (14)	0.0240 (4)
C16	0.3187 (2)	0.7646 (2)	1.00061 (14)	0.0247 (4)
C17	0.3173 (2)	0.8378 (2)	0.92485 (14)	0.0250 (4)
H17	0.3102	0.9181	0.9205	0.030*
C18	0.3259 (2)	0.7964 (2)	0.85567 (14)	0.0237 (4)

H18	0.3250	0.8476	0.8045	0.028*
C19	0.3217 (3)	0.5654 (2)	1.08795 (14)	0.0311 (5)
H19A	0.3261	0.4875	1.0802	0.047*
H19B	0.2341	0.5964	1.1132	0.047*
H19C	0.4030	0.5590	1.1227	0.047*
C20	0.3146 (3)	0.8120 (2)	1.07465 (15)	0.0323 (5)
H20A	0.3183	0.8934	1.0592	0.048*
H20B	0.3958	0.7640	1.1116	0.048*
H20C	0.2270	0.8095	1.1013	0.048*
Br3	0.24367 (3)	0.15502 (2)	0.41709 (2)	0.02955 (8)
Br4	0.19508 (3)	0.36576 (2)	0.50063 (2)	0.03296 (8)
N3	0.1094 (2)	0.51443 (17)	0.33545 (11)	0.0220 (4)
N4	0.07769 (19)	0.58727 (16)	0.26830 (11)	0.0204 (3)
C21	0.1507 (2)	0.39520 (19)	0.33196 (13)	0.0214 (4)
C22	0.1889 (2)	0.3189 (2)	0.40421 (13)	0.0241 (4)
C23	0.1561 (2)	0.35732 (18)	0.25522 (13)	0.0206 (4)
C24	0.0330 (2)	0.3772 (2)	0.20823 (14)	0.0233 (4)
H24	−0.0560	0.4162	0.2250	0.028*
C25	0.0405 (2)	0.3402 (2)	0.13751 (13)	0.0227 (4)
H25	−0.0442	0.3538	0.1065	0.027*
C26	0.1699 (2)	0.28308 (18)	0.11024 (12)	0.0194 (4)
C27	0.2918 (2)	0.26499 (19)	0.15739 (13)	0.0213 (4)
H27	0.3811	0.2274	0.1403	0.026*
C28	0.2851 (2)	0.30098 (19)	0.22908 (13)	0.0219 (4)
H28	0.3696	0.2869	0.2604	0.026*
C29	0.1723 (2)	0.2451 (2)	0.03092 (13)	0.0230 (4)
C30	0.1064 (3)	0.3543 (2)	−0.03732 (14)	0.0319 (5)
H30A	0.1140	0.3318	−0.0889	0.048*
H30B	0.1568	0.4128	−0.0383	0.048*
H30C	0.0067	0.3877	−0.0282	0.048*
C31	0.3219 (3)	0.1862 (3)	0.01054 (16)	0.0360 (6)
H31A	0.3653	0.1166	0.0536	0.054*
H31B	0.3782	0.2414	0.0050	0.054*
H31C	0.3185	0.1626	−0.0402	0.054*
C32	0.0853 (3)	0.1578 (2)	0.03751 (15)	0.0278 (5)
H32A	−0.0135	0.1969	0.0454	0.042*
H32B	0.1233	0.0909	0.0833	0.042*
H32C	0.0905	0.1299	−0.0120	0.042*
C33	0.0451 (2)	0.70618 (19)	0.27538 (13)	0.0203 (4)
C34	0.0515 (2)	0.7391 (2)	0.34773 (13)	0.0217 (4)
H34	0.0737	0.6807	0.3969	0.026*
C35	0.0256 (2)	0.8566 (2)	0.34845 (14)	0.0226 (4)
C36	−0.0068 (2)	0.9434 (2)	0.27575 (14)	0.0230 (4)
C37	−0.0160 (2)	0.9096 (2)	0.20417 (14)	0.0238 (4)
H37	−0.0399	0.9678	0.1550	0.029*
C38	0.0092 (2)	0.7919 (2)	0.20356 (13)	0.0230 (4)
H38	0.0019	0.7701	0.1544	0.028*
C39	0.0356 (3)	0.8902 (2)	0.42721 (14)	0.0281 (5)

H39A	0.0529	0.8201	0.4716	0.042*
H39B	−0.0533	0.9476	0.4341	0.042*
H39C	0.1135	0.9249	0.4270	0.042*
C40	−0.0267 (3)	1.0705 (2)	0.27440 (15)	0.0279 (5)
H40A	−0.1093	1.0992	0.3042	0.042*
H40B	−0.0414	1.1169	0.2186	0.042*
H40C	0.0575	1.0782	0.2995	0.042*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.04118 (15)	0.02158 (13)	0.01649 (12)	−0.00428 (10)	0.00179 (9)	−0.00884 (9)
Br2	0.04033 (14)	0.01623 (12)	0.02161 (12)	−0.00799 (9)	0.00190 (9)	−0.00576 (9)
N1	0.0230 (8)	0.0182 (8)	0.0171 (8)	−0.0051 (7)	−0.0006 (6)	−0.0062 (7)
N2	0.0222 (8)	0.0228 (9)	0.0183 (8)	−0.0068 (7)	−0.0011 (6)	−0.0056 (7)
C1	0.0225 (10)	0.0165 (9)	0.0177 (9)	−0.0035 (7)	−0.0009 (7)	−0.0066 (7)
C2	0.0279 (10)	0.0178 (10)	0.0188 (10)	−0.0057 (8)	0.0004 (8)	−0.0083 (8)
C3	0.0271 (10)	0.0158 (9)	0.0154 (9)	−0.0069 (8)	−0.0002 (7)	−0.0065 (7)
C4	0.0219 (10)	0.0219 (10)	0.0222 (10)	−0.0042 (8)	0.0011 (8)	−0.0105 (8)
C5	0.0234 (10)	0.0218 (10)	0.0210 (10)	−0.0095 (8)	0.0022 (8)	−0.0090 (8)
C6	0.0275 (10)	0.0178 (9)	0.0105 (8)	−0.0086 (8)	0.0029 (7)	−0.0060 (7)
C7	0.0206 (9)	0.0184 (10)	0.0204 (10)	−0.0041 (7)	0.0002 (7)	−0.0046 (8)
C8	0.0238 (10)	0.0200 (10)	0.0243 (10)	−0.0092 (8)	−0.0001 (8)	−0.0042 (8)
C9	0.0281 (10)	0.0153 (9)	0.0168 (9)	−0.0065 (8)	0.0013 (8)	−0.0041 (7)
C10	0.0379 (13)	0.0255 (11)	0.0283 (12)	−0.0130 (10)	0.0072 (10)	−0.0029 (9)
C11	0.0543 (16)	0.0242 (12)	0.0329 (13)	−0.0115 (11)	−0.0082 (11)	−0.0127 (10)
C12	0.0348 (12)	0.0180 (10)	0.0349 (13)	−0.0063 (9)	0.0047 (10)	−0.0065 (9)
C13	0.0197 (10)	0.0289 (11)	0.0184 (10)	−0.0064 (8)	−0.0009 (7)	−0.0078 (8)
C14	0.0246 (10)	0.0237 (10)	0.0215 (10)	−0.0066 (8)	−0.0006 (8)	−0.0048 (8)
C15	0.0212 (10)	0.0265 (11)	0.0213 (10)	−0.0055 (8)	0.0003 (8)	−0.0013 (8)
C16	0.0234 (10)	0.0279 (11)	0.0234 (11)	−0.0072 (8)	−0.0004 (8)	−0.0073 (9)
C17	0.0290 (11)	0.0234 (11)	0.0241 (11)	−0.0077 (9)	0.0019 (8)	−0.0089 (9)
C18	0.0249 (10)	0.0251 (11)	0.0214 (10)	−0.0063 (8)	−0.0005 (8)	−0.0074 (8)
C19	0.0415 (13)	0.0293 (12)	0.0204 (11)	−0.0097 (10)	0.0025 (9)	−0.0027 (9)
C20	0.0423 (13)	0.0351 (13)	0.0245 (11)	−0.0148 (11)	0.0055 (10)	−0.0125 (10)
Br3	0.04488 (15)	0.01919 (13)	0.02299 (13)	−0.00876 (10)	−0.00063 (10)	−0.00254 (9)
Br4	0.05341 (17)	0.02801 (14)	0.01713 (12)	−0.01054 (11)	−0.00191 (10)	−0.00619 (10)
N3	0.0276 (9)	0.0195 (9)	0.0194 (8)	−0.0081 (7)	0.0000 (7)	−0.0041 (7)
N4	0.0227 (8)	0.0203 (9)	0.0201 (8)	−0.0077 (7)	0.0003 (6)	−0.0066 (7)
C21	0.0249 (10)	0.0215 (10)	0.0190 (10)	−0.0080 (8)	0.0000 (8)	−0.0055 (8)
C22	0.0332 (11)	0.0212 (10)	0.0190 (10)	−0.0085 (9)	−0.0002 (8)	−0.0063 (8)
C23	0.0282 (10)	0.0171 (9)	0.0174 (9)	−0.0082 (8)	−0.0002 (8)	−0.0037 (8)
C24	0.0227 (10)	0.0237 (10)	0.0235 (10)	−0.0057 (8)	0.0018 (8)	−0.0073 (8)
C25	0.0230 (10)	0.0231 (10)	0.0222 (10)	−0.0060 (8)	−0.0027 (8)	−0.0063 (8)
C26	0.0241 (10)	0.0174 (9)	0.0156 (9)	−0.0060 (7)	−0.0001 (7)	−0.0019 (7)
C27	0.0221 (10)	0.0205 (10)	0.0205 (10)	−0.0058 (8)	0.0008 (8)	−0.0038 (8)
C28	0.0233 (10)	0.0211 (10)	0.0208 (10)	−0.0070 (8)	−0.0031 (8)	−0.0032 (8)
C29	0.0288 (11)	0.0231 (10)	0.0181 (10)	−0.0084 (8)	0.0001 (8)	−0.0059 (8)

C30	0.0501 (15)	0.0291 (12)	0.0187 (10)	−0.0178 (11)	−0.0060 (10)	−0.0016 (9)
C31	0.0335 (13)	0.0521 (16)	0.0266 (12)	−0.0105 (11)	0.0049 (10)	−0.0203 (12)
C32	0.0375 (12)	0.0253 (11)	0.0249 (11)	−0.0122 (9)	0.0007 (9)	−0.0103 (9)
C33	0.0211 (9)	0.0194 (10)	0.0211 (10)	−0.0066 (8)	0.0008 (7)	−0.0051 (8)
C34	0.0229 (10)	0.0230 (10)	0.0182 (10)	−0.0066 (8)	0.0009 (7)	−0.0033 (8)
C35	0.0208 (10)	0.0243 (11)	0.0230 (10)	−0.0047 (8)	0.0012 (8)	−0.0081 (9)
C36	0.0199 (10)	0.0210 (10)	0.0270 (11)	−0.0039 (8)	0.0006 (8)	−0.0063 (9)
C37	0.0264 (10)	0.0217 (10)	0.0212 (10)	−0.0073 (8)	−0.0027 (8)	−0.0005 (8)
C38	0.0257 (10)	0.0254 (11)	0.0185 (10)	−0.0083 (8)	−0.0018 (8)	−0.0051 (8)
C39	0.0366 (12)	0.0241 (11)	0.0232 (11)	−0.0043 (9)	0.0000 (9)	−0.0104 (9)
C40	0.0303 (11)	0.0209 (11)	0.0311 (12)	−0.0048 (9)	−0.0007 (9)	−0.0070 (9)

Geometric parameters (Å, °)

Br1—C2	1.870 (2)	Br3—C22	1.881 (2)
Br2—C2	1.882 (2)	Br4—C22	1.876 (2)
N1—N2	1.262 (3)	N3—N4	1.260 (3)
N1—C1	1.415 (3)	N3—C21	1.413 (3)
N2—C13	1.424 (3)	N4—C33	1.431 (3)
C1—C2	1.346 (3)	C21—C22	1.349 (3)
C1—C3	1.493 (3)	C21—C23	1.487 (3)
C3—C8	1.387 (3)	C23—C28	1.389 (3)
C3—C4	1.391 (3)	C23—C24	1.398 (3)
C4—C5	1.388 (3)	C24—C25	1.382 (3)
C4—H4	0.9500	C24—H24	0.9500
C5—C6	1.401 (3)	C25—C26	1.403 (3)
C5—H5	0.9500	C25—H25	0.9500
C6—C7	1.390 (3)	C26—C27	1.393 (3)
C6—C9	1.533 (3)	C26—C29	1.532 (3)
C7—C8	1.397 (3)	C27—C28	1.392 (3)
C7—H7	0.9500	C27—H27	0.9500
C8—H8	0.9500	C28—H28	0.9500
C9—C11	1.527 (3)	C29—C31	1.525 (3)
C9—C10	1.533 (3)	C29—C32	1.537 (3)
C9—C12	1.533 (3)	C29—C30	1.540 (3)
C10—H10A	0.9800	C30—H30A	0.9800
C10—H10B	0.9800	C30—H30B	0.9800
C10—H10C	0.9800	C30—H30C	0.9800
C11—H11A	0.9800	C31—H31A	0.9800
C11—H11B	0.9800	C31—H31B	0.9800
C11—H11C	0.9800	C31—H31C	0.9800
C12—H12A	0.9800	C32—H32A	0.9800
C12—H12B	0.9800	C32—H32B	0.9800
C12—H12C	0.9800	C32—H32C	0.9800
C13—C14	1.388 (3)	C33—C38	1.392 (3)
C13—C18	1.406 (3)	C33—C34	1.396 (3)
C14—C15	1.405 (3)	C34—C35	1.389 (3)
C14—H14	0.9500	C34—H34	0.9500

C15—C16	1.398 (3)	C35—C36	1.408 (3)
C15—C19	1.508 (3)	C35—C39	1.511 (3)
C16—C17	1.393 (3)	C36—C37	1.393 (3)
C16—C20	1.507 (3)	C36—C40	1.505 (3)
C17—C18	1.385 (3)	C37—C38	1.391 (3)
C17—H17	0.9500	C37—H37	0.9500
C18—H18	0.9500	C38—H38	0.9500
C19—H19A	0.9800	C39—H39A	0.9800
C19—H19B	0.9800	C39—H39B	0.9800
C19—H19C	0.9800	C39—H39C	0.9800
C20—H20A	0.9800	C40—H40A	0.9800
C20—H20B	0.9800	C40—H40B	0.9800
C20—H20C	0.9800	C40—H40C	0.9800
N2—N1—C1	113.51 (17)	N4—N3—C21	115.01 (18)
N1—N2—C13	113.24 (18)	N3—N4—C33	112.37 (18)
C2—C1—N1	116.73 (18)	C22—C21—N3	113.99 (19)
C2—C1—C3	121.99 (18)	C22—C21—C23	123.0 (2)
N1—C1—C3	121.17 (18)	N3—C21—C23	122.99 (19)
C1—C2—Br1	122.95 (16)	C21—C22—Br4	123.41 (17)
C1—C2—Br2	123.01 (16)	C21—C22—Br3	122.78 (17)
Br1—C2—Br2	114.03 (11)	Br4—C22—Br3	113.81 (12)
C8—C3—C4	119.14 (19)	C28—C23—C24	118.8 (2)
C8—C3—C1	118.81 (18)	C28—C23—C21	120.03 (19)
C4—C3—C1	122.02 (19)	C24—C23—C21	121.2 (2)
C5—C4—C3	120.2 (2)	C25—C24—C23	120.2 (2)
C5—C4—H4	119.9	C25—C24—H24	119.9
C3—C4—H4	119.9	C23—C24—H24	119.9
C4—C5—C6	121.55 (19)	C24—C25—C26	121.8 (2)
C4—C5—H5	119.2	C24—C25—H25	119.1
C6—C5—H5	119.2	C26—C25—H25	119.1
C7—C6—C5	117.50 (19)	C27—C26—C25	117.30 (19)
C7—C6—C9	122.46 (19)	C27—C26—C29	123.09 (19)
C5—C6—C9	120.04 (18)	C25—C26—C29	119.61 (18)
C6—C7—C8	121.32 (19)	C28—C27—C26	121.3 (2)
C6—C7—H7	119.3	C28—C27—H27	119.3
C8—C7—H7	119.3	C26—C27—H27	119.3
C3—C8—C7	120.33 (19)	C23—C28—C27	120.64 (19)
C3—C8—H8	119.8	C23—C28—H28	119.7
C7—C8—H8	119.8	C27—C28—H28	119.7
C11—C9—C6	110.07 (18)	C31—C29—C26	112.15 (18)
C11—C9—C10	109.4 (2)	C31—C29—C32	108.6 (2)
C6—C9—C10	109.04 (18)	C26—C29—C32	109.08 (18)
C11—C9—C12	108.1 (2)	C31—C29—C30	108.6 (2)
C6—C9—C12	111.90 (18)	C26—C29—C30	108.85 (18)
C10—C9—C12	108.28 (19)	C32—C29—C30	109.58 (19)
C9—C10—H10A	109.5	C29—C30—H30A	109.5
C9—C10—H10B	109.5	C29—C30—H30B	109.5

H10A—C10—H10B	109.5	H30A—C30—H30B	109.5
C9—C10—H10C	109.5	C29—C30—H30C	109.5
H10A—C10—H10C	109.5	H30A—C30—H30C	109.5
H10B—C10—H10C	109.5	H30B—C30—H30C	109.5
C9—C11—H11A	109.5	C29—C31—H31A	109.5
C9—C11—H11B	109.5	C29—C31—H31B	109.5
H11A—C11—H11B	109.5	H31A—C31—H31B	109.5
C9—C11—H11C	109.5	C29—C31—H31C	109.5
H11A—C11—H11C	109.5	H31A—C31—H31C	109.5
H11B—C11—H11C	109.5	H31B—C31—H31C	109.5
C9—C12—H12A	109.5	C29—C32—H32A	109.5
C9—C12—H12B	109.5	C29—C32—H32B	109.5
H12A—C12—H12B	109.5	H32A—C32—H32B	109.5
C9—C12—H12C	109.5	C29—C32—H32C	109.5
H12A—C12—H12C	109.5	H32A—C32—H32C	109.5
H12B—C12—H12C	109.5	H32B—C32—H32C	109.5
C14—C13—C18	119.1 (2)	C38—C33—C34	119.8 (2)
C14—C13—N2	116.2 (2)	C38—C33—N4	115.74 (19)
C18—C13—N2	124.7 (2)	C34—C33—N4	124.42 (19)
C13—C14—C15	121.8 (2)	C35—C34—C33	120.5 (2)
C13—C14—H14	119.1	C35—C34—H34	119.7
C15—C14—H14	119.1	C33—C34—H34	119.7
C16—C15—C14	118.5 (2)	C34—C35—C36	119.9 (2)
C16—C15—C19	121.8 (2)	C34—C35—C39	119.7 (2)
C14—C15—C19	119.7 (2)	C36—C35—C39	120.4 (2)
C17—C16—C15	119.7 (2)	C37—C36—C35	119.0 (2)
C17—C16—C20	120.0 (2)	C37—C36—C40	120.1 (2)
C15—C16—C20	120.3 (2)	C35—C36—C40	120.9 (2)
C18—C17—C16	121.6 (2)	C38—C37—C36	121.1 (2)
C18—C17—H17	119.2	C38—C37—H37	119.5
C16—C17—H17	119.2	C36—C37—H37	119.5
C17—C18—C13	119.2 (2)	C37—C38—C33	119.7 (2)
C17—C18—H18	120.4	C37—C38—H38	120.2
C13—C18—H18	120.4	C33—C38—H38	120.2
C15—C19—H19A	109.5	C35—C39—H39A	109.5
C15—C19—H19B	109.5	C35—C39—H39B	109.5
H19A—C19—H19B	109.5	H39A—C39—H39B	109.5
C15—C19—H19C	109.5	C35—C39—H39C	109.5
H19A—C19—H19C	109.5	H39A—C39—H39C	109.5
H19B—C19—H19C	109.5	H39B—C39—H39C	109.5
C16—C20—H20A	109.5	C36—C40—H40A	109.5
C16—C20—H20B	109.5	C36—C40—H40B	109.5
H20A—C20—H20B	109.5	H40A—C40—H40B	109.5
C16—C20—H20C	109.5	C36—C40—H40C	109.5
H20A—C20—H20C	109.5	H40A—C40—H40C	109.5
H20B—C20—H20C	109.5	H40B—C40—H40C	109.5
C1—N1—N2—C13	178.45 (17)	C21—N3—N4—C33	−176.50 (17)

N2—N1—C1—C2	177.35 (19)	N4—N3—C21—C22	177.8 (2)
N2—N1—C1—C3	−6.3 (3)	N4—N3—C21—C23	−0.2 (3)
N1—C1—C2—Br1	179.63 (14)	N3—C21—C22—Br4	−2.6 (3)
C3—C1—C2—Br1	3.3 (3)	C23—C21—C22—Br4	175.41 (16)
N1—C1—C2—Br2	1.0 (3)	N3—C21—C22—Br3	178.31 (15)
C3—C1—C2—Br2	−175.32 (15)	C23—C21—C22—Br3	−3.7 (3)
C2—C1—C3—C8	89.0 (3)	C22—C21—C23—C28	−64.6 (3)
N1—C1—C3—C8	−87.1 (3)	N3—C21—C23—C28	113.2 (2)
C2—C1—C3—C4	−93.1 (3)	C22—C21—C23—C24	115.3 (3)
N1—C1—C3—C4	90.7 (3)	N3—C21—C23—C24	−66.9 (3)
C8—C3—C4—C5	0.0 (3)	C28—C23—C24—C25	0.6 (3)
C1—C3—C4—C5	−177.83 (19)	C21—C23—C24—C25	−179.4 (2)
C3—C4—C5—C6	−0.6 (3)	C23—C24—C25—C26	−0.5 (3)
C4—C5—C6—C7	0.6 (3)	C24—C25—C26—C27	−0.1 (3)
C4—C5—C6—C9	−179.88 (19)	C24—C25—C26—C29	−179.4 (2)
C5—C6—C7—C8	0.0 (3)	C25—C26—C27—C28	0.7 (3)
C9—C6—C7—C8	−179.51 (19)	C29—C26—C27—C28	179.9 (2)
C4—C3—C8—C7	0.6 (3)	C24—C23—C28—C27	0.0 (3)
C1—C3—C8—C7	178.49 (19)	C21—C23—C28—C27	180.0 (2)
C6—C7—C8—C3	−0.6 (3)	C26—C27—C28—C23	−0.7 (3)
C7—C6—C9—C11	−124.5 (2)	C27—C26—C29—C31	0.0 (3)
C5—C6—C9—C11	56.0 (3)	C25—C26—C29—C31	179.2 (2)
C7—C6—C9—C10	115.5 (2)	C27—C26—C29—C32	120.3 (2)
C5—C6—C9—C10	−64.0 (2)	C25—C26—C29—C32	−60.5 (3)
C7—C6—C9—C12	−4.3 (3)	C27—C26—C29—C30	−120.2 (2)
C5—C6—C9—C12	176.27 (19)	C25—C26—C29—C30	59.0 (3)
N1—N2—C13—C14	−179.04 (18)	N3—N4—C33—C38	179.90 (19)
N1—N2—C13—C18	0.2 (3)	N3—N4—C33—C34	1.9 (3)
C18—C13—C14—C15	−0.7 (3)	C38—C33—C34—C35	−1.6 (3)
N2—C13—C14—C15	178.53 (19)	N4—C33—C34—C35	176.37 (19)
C13—C14—C15—C16	−1.0 (3)	C33—C34—C35—C36	−0.2 (3)
C13—C14—C15—C19	179.2 (2)	C33—C34—C35—C39	−178.9 (2)
C14—C15—C16—C17	2.3 (3)	C34—C35—C36—C37	1.7 (3)
C19—C15—C16—C17	−177.9 (2)	C39—C35—C36—C37	−179.6 (2)
C14—C15—C16—C20	−177.3 (2)	C34—C35—C36—C40	−176.3 (2)
C19—C15—C16—C20	2.5 (3)	C39—C35—C36—C40	2.4 (3)
C15—C16—C17—C18	−1.9 (3)	C35—C36—C37—C38	−1.3 (3)
C20—C16—C17—C18	177.7 (2)	C40—C36—C37—C38	176.7 (2)
C16—C17—C18—C13	0.1 (3)	C36—C37—C38—C33	−0.4 (3)
C14—C13—C18—C17	1.2 (3)	C34—C33—C38—C37	1.9 (3)
N2—C13—C18—C17	−178.0 (2)	N4—C33—C38—C37	−176.22 (19)