Crystal structure and Hirshfeld surface analysis of 2-[(E)-(3-cyclobutyl-1H-1,2,4-triazol-5-yl)imino]-methylphenol

Mustafa Kemal Gumus,a Fatih Sen,b* Sevgi Kansiz,c Necmi Dege,d and Eiad Sai‡f,e*

aArtvin Coruh University, Science-Technology Research and Application Center, 08000, Artvin, Turkey,
bYozgat Bozok University, Sorgun Vocational School, 66100, Yozgat, Turkey, cSamsun University, Faculty of Engineering, Department of Fundamental Sciences, 55420, Samsun, Turkey, dOndokuz Mayis University, Faculty of Arts and Sciences, Department of Physics, 55139, Samsun, Turkey, eDepartment of Computer and Electronic Engineering Technology, Sanaa Community College, Sanaa, Yemen, and fDepartment of Electrical and Electronic Engineering, Faculty of Engineering, Ondokuz Mayis University, 55139, Samsun, Turkey. *Correspondence e-mail: fatih.sen@yobu.edu.tr, eiad.sai@ssc.edu.ye

The title compound, C_{13}H_{14}N_{4}O, was developed using the reaction of salicylaldehyde and 3-amino-5-cyclobutyl-1,2,4-triazole in ethanol under microwave irradiation. This eco-friendly microwave-promoted method proved to be efficient in the synthesis of 2-[(E)-(3-cyclobutyl-1H-1,2,4-triazol-5-yl)imino]-methylphenol in good yields and purity. The title compound is a Schiff base that exists in the phenol–imine tautomeric form and adopts an E configuration. The three independent molecules in the asymmetric unit (A, B and C) are not planar, the cyclobutyl and the phenol-imine rings are twisted to each other making a dihedral angle of 67.8 (4)° in molecule A, 69.1 (2)° in molecule B and 89.1 (2)° in molecule C. In each molecule an intramolecular O—H···N hydrogen bond is present, forming an S(6) ring motif. A Hirshfeld surface analysis was performed to investigate the contributions of the different intermolecular contacts within the supramolecular structure. The major interactions are H···H (53%), C···H (19%) and N···H (17%) for molecule A, H···H (50%), N···H (20%) and C···H (20%) for molecule B and H···H (57%), C···H (14%) and N···H (13%) for molecule C.

1. Chemical context

Imines (Schiff bases) have been extensively used as analytical and medicinal materials (Bülbüld et al., 2017; Singh, 2021). 1,2,4-Triazoles possess a number of medicinal attributes (Aggarwal & Sumran, 2020). Taking into account the above considerations, it was decided to merge the chemistry of both parts by reacting 3-amino-5-cyclobutyl-1,2,4-triazole with salicylaldehyde to develop an efficient green protocol for the synthesis of 2-[(E)-(3-cyclobutyl-1H-1,2,4-triazol-3-ylimino)-methyl]phenol. In this work, an eco-friendly protocol for the synthesis of Schiff bases from 3-amino-5-cyclobutyl-1,2,4-triazole and salicylaldehyde in ethanol under microwave irradiation was developed. In addition, 2-[(E)-(3-cyclobutyl-1H-1,2,4-triazol-3-ylimino)methyl]phenol was characterized by single crystal X-ray diffraction and investigated using Hirshfeld surface analysis.
2. Structural commentary

The molecular structure of the title compound, (I), with the atomic numbering scheme is shown in Fig. 1. The asymmetric unit contains three non-planar molecules. The 1,2,4-triazole and phenol-imine rings are twisted with respect to each other, making a dihedral angle of 18.1 (3)° for molecule A. The cyclobutyl ring is twisted by 73.9 (3)° and 67.8 (4)°, with respect to the 1,2,4-triazole, and phenol-imine rings in molecule A. The corresponding angles in molecule B are 18.7 (3), 74.6 (2)° and 69.1 (2)°, and in molecule C, 18.7 (3), 74.6 (2)° and 89.1 (2)°. When these angles for the three molecules are compared, it is observed that there is a harmony between them, as well as significant differences, especially in the angles between the phenol-imine and 1,2,4-triazole rings. These values and other bond lengths previously reported for C=N and O—C bonds (Bülbul et al., 2019; Demircioğlu et al., 2019). The average triazole N—N bond length is 1.353 Å. This length is quite close to the corresponding values reported by Al-Karawi and co-workers [1.343 (4) and 1.353 (6) Å; Al-Karawi et al., 2021a,b]. In each molecule, the hydroxyl H atom is involved in a strong intramolecular O—H···N hydrogen bond (O1—H1···N1, O2—H2···N5 and O3—H3···N9; Table 2) forming an S(6) ring motif.

3. Supramolecular features

In the crystal, intermolecular hydrogen bonds N3—H3···N10a, N7—H7D···N2, N11—H11···N6 and C10—H10···O3i (symmetry codes as in Table 2) link the molecules into [100] chains. A view of the crystal packing of the structure is shown in Fig. 2.

4. Database survey

There are no direct precedents for the structure of (I) in the crystallographic literature (CSD Version 5.42, update of May 2021; Groom et al., 2016). However, several related compounds have been reported that include (E)-N-benzylidene-1H-1,2,4-triazol-5-amine as the main skeleton, viz. 5-methyl-2-[(1H-1,2,4-triazol-3-yl)mino]methyl]phenol (PEVXAS; Brink et al., 2018), 1-(4-bromophenyl)-N-(1H-1,2,4-triazol-3-yl)methanimine (TIVDUA; Kolodziej et al., 2019), 5-bromo-2-[[1H-1,2,4-triazol-3-yl]mimo]methylphenol (TIVFAI; Kolodziej et al., 2019), 4-bromo-2-[[1H-1,2,4-triazol-3-ylmino]methyl]phenol (UZOKIE; Chohan & Hanif, 2011) and 3,5-bis(salicylideneamino)-1H-1,2,4-triazole methanol solvate.
(WEFTUX; Cheng et al., 2006). In addition, 1-[(1H-1,2,4-triazol-3-ylimino)methyl]-2-naphthol (GILYUX; Jia et al., 2013), which contains a naphthalene fragment instead of benzene, has been reported. In UZOKIE, the hydroxyl-C2 group makes a dihedral angle of 4.48 (3)° with the plane of the 1,2,4-triazole ring system. In addition, there are intramolecular O—H···N contacts in the molecule. Similarly, in WEFTUX, the hydroxyl H atom is involved in an intramolecular O—H···N hydrogen bond, forming an S(6) ring motif as in the title compound. The two benzene rings (1 and 3) and the triazole ring (2) in WEFTUX, are almost in the same plane, the angles between rings 1 and 2, and between rings 2 and 3 being 3.7 (2)° and 3.3 (2)°, respectively. This latter angle is 4.58 (8)° in PEVXAS. In the structures mentioned above, the twist angles between triazole and phenyl rings are quite small, as in molecule C of (I) [3.2 (4)°]; however, for molecules A and B of the title compound, these angles are over 18°. All compounds were isolated as the phenol-imine (O—H···N) tautomeric form, as in (I). The bond lengths of the triazole ring in the studied compound are very similar to those in the other 1H-1,2,4-triazole derivatives mentioned above.

5. Hirshfeld surface analysis

We performed a Hirshfeld surface analysis and generated the associated two-dimensional fingerprint plots (Spackman & Jayatilaka, 2009) with CrystalExplorer17 (Turner et al., 2017). Hirshfeld surface (HS) analysis is a valuable tool for assessing the strength of intermolecular interactions and for predicting the properties of a crystal and its potential applications (Demir Kanmazalp et al., 2019; Al-Resayes et al., 2020). The Hirshfeld surfaces were generated using a standard (high) surface resolution with the three-dimensional $d_{norm}$ surface mapped over fixed colour scales of −0.6059 (red) to 1.5176 Å (blue) (molecule A), −0.6084 (red) to 1.2881 Å (blue) (molecule B) and −0.6060 (red) to 1.5351 Å (blue) (molecule C), respectively. In Fig. 3, the red circle on the $d_{norm}$ surface of molecules A, B and C represents the N—H···N interactions. The major interactions of the compound (Fig. 4) are H···H (53%), C···H (19%) and N···H (17%) for molecule A, H···H (57%), C···H (14%) and N···H (13%) for molecule B.
6. Synthesis and crystallization
Salicylaldehyde (1.0 mmol), 3-amino-5-cyclobutyl-1,2,4-triazole (1.0 mmol) and absolute EtOH (2.0 ml) were mixed in a microwave process vial (10 ml), then a 4 N solution of HCl in dioxane (one drop) was added. The mixtures were irradiated at 393 K for 30 min. The precipitated solid was filtered, washed with cold ethanol and dried at 353 K. The title compound was obtained in the form of a pale-yellow solid in 92% yield. It was recrystallized from ethanol (m.p. 448–449 K). The reaction scheme is shown in Fig. 4. The microwave experiment was carried out using a monomode Anton Paar Monowave 300 microwave reactor (2.45 GHz) in a G10 sealed microwave process vial (10 ml). The reaction temperatures were monitored by an IR sensor. After completion of the reaction, the vial was cooled to 323 K by air jet cooling. IR (Shimadzu Prestige–21 Fourier spectrometer, ATR, cm⁻¹): 759, 991, 1030, 1076, 1276, 1562, 1612, 2986, 3040.

1H NMR (Nanalysis Benchtop NMR spectrometer, 60 MHz, DMSO-d₆, ppm): 13.73 (s, 1H, NH), 12.60 (s, 1H, OH), 9.34 (s, 1H, CH=N), 7.90–6.80 (m, 4H, aromatic H), 3.70–3.30 (m, 1H, cyc-butyl, CH), 2.55–1.75 (m, 6H, cyc-butyl, CH₂).

Elemental analysis (Vario MACRO cube CHNS elemental analyzer): Found, %: C 64.31; H 5.64; N 23.79. C₁₃H₁₄N₄O. Calculated, %: C, 64.45; H, 5.82; N, 23.13.

7. Refinement
Crystal data, data collection and structure refinement details are summarized in Table 3. The O-bound H atom was located in a difference-Fourier map and refined with O—H = 0.82 Å, and with Uiso(H) = 1.5Ueq(O). The N-bound H atom was located in a difference-Fourier map. Its parameters were adjusted to give N—H = 0.86 Å and it was then refined as riding with Uiso(H) = 1.2Ueq(N). The C-bound H atoms were positioned geometrically and refined using a riding model with C—H = 0.93 Å and Uiso(H) = 1.2Ueq(C) for aromatic and other H atoms, and with C—H = 0.97 Å and Uiso(H) = 1.5Ueq(C) for methylene H atoms. The crystal studied was refined as a two-component inversion twin.

| Table 3 | Experimental details. |
|---------|-----------------------|
| Crystal data | C₁₃H₁₄N₄O |
| Chemical formula | C₁₃H₁₄N₄O |
| Mᵣ | 242.28 |
| Crystal system, space group | Monoclinic, P2₁ |
| Temperature (K) | 296 |
| a, b, c (Å) | 5.2717 (3), 24.9066 (14), 14.8628 (7) |
| β (°) | 96.214 (4) |
| V (Å³) | 1940.02 (18) |
| Z | 6 |
| Radiation type | Mo Ka |
| μ (mm⁻¹) | 0.08 |
| Crystal size (mm) | 0.76 × 0.52 × 0.30 |

Data collection
Diffractometer | Stoe IPDS 2 |
Absorption correction | Integration (X-RED32; Stoe & Cie, 2002) |
Tmin, Tmax | 0.938, 0.980 |
No. of measured, independent and observed [I > 2σ(I)] reflections | 15228, 8333, 5374 |
R(M) | 0.096 |
(σ(θ))max (Å⁻¹) | 0.637 |

Refinement
R[F² > 2σ(F²)], wR(F²), S | 0.058, 0.153, 0.94 |
No. of reflections | 8333 |
No. of parameters | 493 |
No. of restraints | 1 |
H-atom treatment | H atoms treated by a mixture of independent and constrained refinement |
Δρ max, Δρ min (e Å⁻³) | 0.16, −0.19 |
Absolute structure | Refined as an inversion twin (2) |
Absolute structure parameter | |

Acknowledgements
Author contributions are as follows. Conceptualization, SK, ND and ES; synthesis, MKG; writing (review and editing of the manuscript), MKG, SK and FS; formal analysis, MKG and SK; crystal-structure determination, SK, ND and FS; validation, MKG, FS, SK and ES; project administration, MKG, FS and SK.

Funding information
This study was supported financially by the Project Coordination Application and Research Center in Yozgat Bozok University (project No. 6602a-SMYO/19-273).

References
Aggarwal, R. & Sumran, G. (2020). Eur. J. Med. Chem. 205, 112652.
Al-Karawi, A. J. M., OmarAli, A. B., Dege, N. & Kansiz, S. (2021b). Chem. Pap. 75, 3901–3914.
Al-Karawi, A. J. M., OmarAli, A. B., Mangelsen, S., Dege, N., Kansiz, S., Breuninger, P., Baydere, C. & OmarAli, O. B. (2021a). Polyhedron, 198, 115084.
Al-Resayes, S. I., Azam, M., Trzesowska-Kruszynska, A., Kruszynski, R., Soliman, S. M., Mohapatra, R. K. & Khan, Z. (2020). Am. Chem. Soc. Omega, 5, 27227–27234.
Brink, A., Kroon, R. E., Visser, H. G., van Rensburg, C. E. J. & Roodt, A. (2018). New J. Chem. 42, 5193–5203.
Bülbül, H., Köysal, Y., Doğan, O. E., Dege, N. & Ağar, E. (2019). Crystallogr. Rep. 64, 403–406.
Bülbül, H., Köysal, Y., Macit, M., Yaman, R. & Dege, N. (2017). Z. Kristallogr. New Crystal. Struct. 232, 135–136.
Cheng, R.-M., Li, Y.-Z., Ou, S.-J. & Chen, X.-T. (2006). Acta Crystal. E62, o1424–o1425.
Chohan, Z. H. & Hanif, M. (2011). Appl. Organomet. Chem. 25, 753–760.
Demircioğlu, Z., Kaştaş, G., Kaştaş, Ç. A. & Frank, R. (2019). J. Mol. Struct. 1191, 129–137.
Demir Kanmazalp, S., Doğan, O. E., Dege, N., Ağar, E., Bulbul, H. & Golenya, I. A. (2019). Acta Crystal. E75, 470–474.
Farrugia, L. J. (2012). J. Appl. Cristal. 45, 849–854.
Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). Acta Crystal. B72, 171–179.
Jia, T.-J., Cao, W., Zheng, X.-J. & Jin, L.-P. (2013). Tetrahedron Lett. 54, 3471–3474.
Kołodziej, B., Morawiak, M., Schilf, W. & Kamieński, B. (2019). J. Mol. Struct. 1184, 207–218.
Sheldrick, G. M. (2015a). Acta Crystal. A71, 3–8.
Sheldrick, G. M. (2015b). Acta Crystal. C71, 3–8.
Singh, G. S. (2021). In Green Synthetic Approaches for Biologically Relevant Heterocycles, pp. 655–687. Amsterdam: Elsevier.
Spackman, M. A. & Jayatilaka, D. (2009). CrystEngComm, 11, 19–32.
Spek, A. L. (2020). Acta Crystal. E76, 1–11.
Stoe & Cie (2002). X-AREA and X-RED32. Stoe & Cie GmbH, Darmstadt, Germany.
Turner, M. J., MacKinnon, J. J., Wolff, S. K., Grimwood, D. J., Spackman, P. R., Jayatilaka, D. & Spackman, M. A. (2017). CrystalExplorer17.5. University of Western Australia. http://hirshfeldsurface.net.
supporting information

Acta Cryst. (2021). E77, 1267-1271  [https://doi.org/10.1107/S2056989021011658]

Crystal structure and Hirshfeld surface analysis of 2-[[((E)-(3-cyclobutyl-1H-1,2,4-triazol-5-yl)imino)methyl]phenol

Mustafa Kemal Gumus, Fatih Sen, Sevgi Kansiz, Necmi Dege and Eiad Saif

Computing details

Data collection: X-AREA (Stoe & Cie, 2002); cell refinement: X-AREA (Stoe & Cie, 2002); data reduction: X-RED (Stoe & Cie, 2002); program(s) used to solve structure: SHELXT2017/1 (Sheldrick, 2015a); program(s) used to refine structure: SHELXL2017/1 (Sheldrick, 2015b); molecular graphics: PLATON (Spek, 2020); software used to prepare material for publication: WinGX (Farrugia, 2012).

2-(((E)-(3-Cyclobutyl-1H-1,2,4-triazol-5-yl)imino)methyl]phenol

Crystal data

C13H14N4O  
Mr = 242.28  
Monoclinic, P21  
a = 5.2717 (3) Å  
b = 24.9066 (14) Å  
c = 14.8628 (7) Å  
β = 96.214 (4)°  
V = 1940.02 (18) Å³  
Z = 6  

F(000) = 768  
Dₐ = 1.244 Mg m⁻³  
Mo Kα radiation, λ = 0.71073 Å  
Cell parameters from 17085 reflections  
θ = 1.4–27.4°  
μ = 0.08 mm⁻¹  
T = 296 K  
Stick, yellow  
0.76 × 0.52 × 0.30 mm

Data collection

Stoe IPDS 2  
diffractometer  
Radiation source: sealed X-ray tube, 12 x 0.4 mm long-focus  
Detector resolution: 6.67 pixels mm⁻¹  
rotation method scans  
Absorption correction: integration  
(X-RED32; Stoe & Cie, 2002)  
Tmin = 0.938, Tmax = 0.980

Refinement

Refinement on F²  
Least-squares matrix: full  
R[F² > 2σ(F²)] = 0.058  
wR(F²) = 0.153  
S = 0.94  
8333 reflections  
493 parameters  
1 restraint  

Hydrogen site location: inferred from 
neighbouring sites  
H atoms treated by a mixture of independent 
and constrained refinement  
w = 1/[σ²(Fc) + (0.084P)²]  
where P = (Fc² + 2Fc) / 3  
(Δ/σ)max < 0.001  
Δρmax = 0.16 e Å⁻³
$\Delta \rho_{\text{min}} = -0.19 \text{ e Å}^{-3}$

Absolute structure: Refined as an inversion twin

**Special details**

**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.

**Refinement.** Refined as a two-component inversion twin.

**Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)**

| x          | y          | z          | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------------|------------|------------|----------------------------------|
| N11        | -0.7970 (7)| -0.44524 (14) | -0.4433 (2)  | 0.0699 (9) |
| H11        | -0.897406 | -0.472528 | -0.444919 | 0.084* |
| N6         | -1.0215 (7)| -0.54788 (13) | -0.4431 (2) | 0.0652 (9) |
| N5         | -0.8325 (6) | -0.56917 (14) | -0.5739 (2) | 0.0616 (8) |
| N9         | -0.3235 (7) | -0.36402 (14) | -0.3466 (2) | 0.0699 (9) |
| N12        | -0.6141 (7) | -0.37213 (14) | -0.4855 (2) | 0.0698 (9) |
| O2         | -0.4440 (6) | -0.51862 (14) | -0.6305 (2) | 0.0792 (8) |
| H2         | -0.548954 | -0.525821 | -0.595446 | 0.119* |
| N10        | -0.6221 (8) | -0.43214 (14) | -0.3725 (2) | 0.0709 (9) |
| N7         | -1.1902 (7) | -0.57344 (14) | -0.3961 (2) | 0.0667 (9) |
| H7D        | -1.247216 | -0.560968 | -0.348178 | 0.088 (16)* |
| O1         | -0.7626 (7) | -0.65935 (15) | -0.2019 (2) | 0.0900 (10) |
| H1         | -0.871881 | -0.635799 | -0.204508 | 0.135* |
| N8         | -1.1368 (7) | -0.62721 (14) | -0.5071 (2) | 0.0645 (8) |
| N3         | -1.5053 (9) | -0.49514 (16) | -0.2114 (2) | 0.0809 (11) |
| H3D        | -1.563359 | -0.470387 | -0.248339 | 0.077 (14)* |
| N1         | -1.1418 (7) | -0.60443 (14) | -0.1485 (2) | 0.0673 (9) |
| C21        | -0.9973 (8) | -0.58229 (16) | -0.5094 (3) | 0.0597 (9) |
| N4         | -1.4393 (8) | -0.54232 (16) | -0.0881 (2) | 0.0779 (10) |
| N2         | -1.3418 (8) | -0.53404 (15) | -0.2310 (2) | 0.0756 (10) |
| C20        | -0.8486 (8) | -0.59720 (17) | -0.6470 (3) | 0.0615 (9) |
| H20        | -0.967147 | -0.624910 | -0.654904 | 0.074* |
| C35        | -0.7894 (9) | -0.40930 (16) | -0.5098 (3) | 0.0654 (10) |
| C34        | -0.5166 (9) | -0.38873 (16) | -0.4011 (3) | 0.0630 (10) |
| O3         | -0.0219 (9) | -0.35762 (17) | -0.1976 (3) | 0.1155 (15) |
| H3         | -0.124553 | -0.371866 | -0.235635 | 0.173* |
| C8         | -1.3077 (9) | -0.56122 (17) | -0.1547 (3) | 0.0673 (10) |
| C7         | -1.1444 (9) | -0.63770 (19) | -0.0833 (3) | 0.0709 (11) |
| H7         | -1.257747 | -0.631869 | -0.040517 | 0.085* |
| C32        | -0.0082 (8) | -0.29555 (16) | -0.3204 (3) | 0.0637 (10) |
| C22        | -1.2564 (8) | -0.62051 (16) | -0.4337 (3) | 0.0633 (10) |
| C33        | -0.2150 (9) | -0.32178 (18) | -0.3745 (3) | 0.0674 (11) |
| H33        | -0.271873 | -0.307826 | -0.431097 | 0.081* |
| C19        | -0.6849 (8) | -0.58627 (17) | -0.7172 (3) | 0.0607 (9) |
| C31        | 0.1081 (9) | -0.2502 (2) | -0.3523 (3) | 0.0764 (12) |
| Atomic     | x       | y       | z       | Ueq |
|------------|---------|---------|---------|-----|
| H31        | 0.048601| -0.236737| -0.409058| 0.092* |
| C23        | -1.4205 (9) | -0.66013 (18) | -0.3935 (3) | 0.0721 (12) |
| H23        | -1.533394| -0.642112| -0.354781| 0.087* |
| C1         | -0.7985 (8) | -0.69374 (19) | -0.1338 (3) | 0.0713 (11) |
| C6         | -0.9800 (8) | -0.68365 (18) | -0.0732 (3) | 0.0646 (10) |
| C9         | -1.5652 (11) | -0.5004 (2) | -0.1259 (3) | 0.0799 (13) |
| C15        | -0.3418 (9) | -0.5393 (2) | -0.7774 (3) | 0.0825 (13) |
| H15        | -0.211309| -0.513991| -0.70918  | 0.099* |
| C14        | -0.4932 (7) | -0.54795 (18) | -0.7067 (3) | 0.0634 (10) |
| C16        | -0.3863 (11) | -0.5680 (3) | -0.8555 (3) | 0.0887 (15) |
| H16        | -0.285384| -0.561698| -0.901974| 0.106* |
| C30        | 0.3059 (10) | -0.2248 (2) | -0.3032 (4) | 0.0835 (13) |
| H30        | 0.377953 | -0.194138 | -0.325682 | 0.100* |
| C24        | -1.2859 (11) | -0.7076 (2) | -0.3438 (4) | 0.0945 (16) |
| H24A       | -1.275498| -0.704915| -0.278389| 0.113* |
| H24B       | -1.120686| -0.715851| -0.363368| 0.113* |
| C5         | -1.0037 (10) | -0.7208 (2) | -0.0045 (3) | 0.0838 (13) |
| H5         | -1.123504| -0.714299| 0.036028 | 0.101* |
| C27        | 0.0858 (9) | -0.3150 (2) | -0.2347 (3) | 0.0773 (13) |
| C36        | -0.9562 (9) | -0.41109 (19) | -0.5962 (3) | 0.0741 (11) |
| H36        | -1.079123| -0.440553| -0.595341| 0.089* |
| C18        | -0.7251 (9) | -0.6154 (2) | -0.7978 (3) | 0.0758 (12) |
| H18        | -0.852386| -0.641378| -0.805055| 0.091* |
| C2         | -0.6502 (10) | -0.7394 (2) | -0.1235 (4) | 0.0910 (15) |
| H2A        | -0.529395| -0.745790| -0.163398| 0.109* |
| C4         | -0.8513 (11) | -0.7665 (2) | 0.0046 (4) | 0.0957 (16) |
| H4         | -0.867080| -0.790781| 0.051120 | 0.115* |
| C26        | -1.5682 (9) | -0.7009 (2) | -0.4542 (4) | 0.0835 (14) |
| H26A       | -1.749634| -0.693456| -0.464529| 0.100* |
| H26B       | -1.497645| -0.706884| -0.510986| 0.100* |
| C3         | -0.6767 (12) | -0.7752 (3) | -0.0564 (4) | 0.0997 (17) |
| H3A        | -0.575938| -0.805905| -0.051536| 0.120* |
| C10        | -1.7219 (14) | -0.4618 (2) | -0.0811 (4) | 0.106 (2) |
| H10        | -1.841507| -0.443208| -0.125701| 0.127* |
| C28        | 0.2882 (11) | -0.2889 (3) | -0.1857 (4) | 0.0950 (16) |
| H28        | 0.350184| -0.301504| -0.128671| 0.114* |
| C17        | -0.5736 (11) | -0.6054 (3) | -0.8673 (3) | 0.0888 (15) |
| H17        | -0.600936| -0.624342| -0.921459| 0.107* |
| C29        | 0.3970 (10) | -0.2450 (2) | -0.2204 (4) | 0.0878 (14) |
| H29        | 0.535293| -0.228508| -0.187238| 0.105* |
| C39        | -1.0923 (12) | -0.3597 (3) | -0.6292 (4) | 0.0994 (17) |
| H39A       | -1.271579| -0.358844| -0.619836| 0.119* |
| H39B       | -1.006271| -0.327158| -0.606644| 0.119* |
| C25        | -1.5013 (11) | -0.7440 (2) | -0.3854 (4) | 0.0948 (16) |
| H25A       | -1.442921| -0.777109| -0.410537| 0.114* |
| H25B       | -1.632828| -0.750774| -0.345929| 0.114* |
| C37        | -0.8362 (12) | -0.4113 (3) | -0.6856 (3) | 0.1036 (18) |
| H37A       | -0.667032| -0.395503| -0.681379| 0.124* |
H37B  -0.839005  -0.446067  -0.715000  0.124*
C38  -1.0446 (15)  -0.3741 (3)  -0.7240 (4)  0.121 (2)
H38A  -0.984828  -0.344306  -0.758075  0.145*
H38B  -0.984828  -0.344306  -0.758075  0.145*
C11  -1.5830 (19)  -0.4224 (4)  -0.0169 (8)  0.167 (4)
H11A  -1.414523  -0.434387  0.007721  0.200*
H11B  -1.576586  -0.386355  0.177 (4)
H12A  -1.907206  -0.0113 (5)  0.212*
H12B  -1.706473  -0.433875  0.212*
C13  -1.8566 (14)  -0.4810 (4)  -0.0013 (5)  0.150*
H13A  -2.039826  -0.484886  0.150*
H13B  -1.781036  -0.512679  0.150*

Atomic displacement parameters (Å²)

|      | U¹¹ | U²² | U³³ | U¹² | U¹³ | U²³ |
|------|-----|-----|-----|-----|-----|-----|
| N11  | 0.097 (2) | 0.0553 (18) | 0.0566 (19) | -0.0203 (18) | 0.0055 (17) | -0.0031 (15) |
| N6   | 0.092 (2) | 0.0555 (18) | 0.0513 (17) | -0.0169 (17) | 0.0211 (16) | -0.0036 (15) |
| N5   | 0.0716 (18) | 0.0616 (18) | 0.0541 (17) | -0.0049 (16) | 0.0186 (14) | -0.0007 (15) |
| N9   | 0.096 (3) | 0.059 (2) | 0.0516 (17) | -0.0067 (19) | -0.0049 (17) | 0.0045 (15) |
| N12  | 0.093 (2) | 0.060 (2) | 0.0533 (18) | -0.0144 (18) | -0.0069 (16) | 0.0124 (15) |
| O2   | 0.088 (2) | 0.086 (2) | 0.0657 (18) | -0.0154 (17) | 0.0176 (14) | -0.0036 (16) |
| N10  | 0.105 (3) | 0.0541 (19) | 0.0528 (18) | -0.0139 (18) | 0.0040 (18) | 0.0043 (15) |
| N7   | 0.097 (2) | 0.0576 (19) | 0.0495 (17) | -0.0128 (18) | 0.0262 (17) | -0.0061 (15) |
| O1   | 0.097 (2) | 0.092 (2) | 0.086 (2) | 0.0105 (19) | 0.0338 (18) | 0.0083 (19) |
| N8   | 0.078 (2) | 0.0558 (19) | 0.0634 (19) | -0.0051 (17) | 0.0253 (16) | -0.0067 (15) |
| N3   | 0.133 (3) | 0.063 (2) | 0.0467 (18) | 0.016 (2) | 0.0101 (19) | 0.0084 (16) |
| N1   | 0.089 (2) | 0.063 (2) | 0.0517 (18) | 0.0084 (18) | 0.0155 (16) | 0.0017 (15) |
| C21  | 0.073 (2) | 0.055 (2) | 0.053 (2) | -0.0082 (19) | 0.0154 (17) | -0.0015 (17) |
| N4   | 0.120 (3) | 0.073 (2) | 0.0427 (16) | 0.028 (2) | 0.0158 (17) | 0.0011 (16) |
| N2   | 0.121 (3) | 0.061 (2) | 0.0473 (18) | 0.007 (2) | 0.0200 (18) | 0.0027 (15) |
| C20  | 0.071 (2) | 0.058 (2) | 0.058 (2) | -0.0010 (19) | 0.0162 (18) | -0.0006 (17) |
| C35  | 0.085 (3) | 0.053 (2) | 0.057 (2) | -0.010 (2) | -0.0011 (19) | 0.0053 (18) |
| C34  | 0.088 (3) | 0.052 (2) | 0.047 (2) | -0.008 (2) | -0.0001 (18) | 0.0050 (16) |
| O3   | 0.158 (4) | 0.100 (3) | 0.077 (2) | -0.045 (3) | -0.040 (2) | 0.035 (2) |
| C8   | 0.098 (3) | 0.062 (2) | 0.045 (2) | 0.007 (2) | 0.0171 (19) | -0.0001 (17) |
| C7   | 0.087 (3) | 0.076 (3) | 0.051 (2) | 0.008 (2) | 0.0143 (19) | -0.004 (2) |
| C32  | 0.078 (2) | 0.058 (2) | 0.053 (2) | 0.000 (2) | -0.0030 (18) | 0.0000 (18) |
| C22  | 0.078 (2) | 0.055 (2) | 0.060 (2) | -0.008 (2) | 0.0214 (19) | -0.0068 (18) |
| C33  | 0.089 (3) | 0.060 (2) | 0.050 (2) | -0.002 (2) | -0.0051 (19) | 0.0058 (18) |
| C19  | 0.066 (2) | 0.066 (2) | 0.052 (2) | 0.0133 (19) | 0.0129 (17) | 0.0042 (17) |
| C31  | 0.089 (3) | 0.071 (3) | 0.065 (3) | -0.010 (2) | -0.008 (2) | 0.009 (2) |
| C23  | 0.083 (3) | 0.065 (2) | 0.075 (3) | -0.015 (2) | 0.035 (2) | -0.010 (2) |
| C1   | 0.069 (2) | 0.074 (3) | 0.070 (3) | 0.000 (2) | 0.000 (2) | -0.006 (2) |
| C6   | 0.072 (2) | 0.064 (2) | 0.056 (2) | 0.004 (2) | -0.0003 (18) | -0.0023 (18) |
| C9   | 0.121 (4) | 0.075 (3) | 0.044 (2) | 0.019 (3) | 0.008 (2) | -0.003 (2) |
| C15  | 0.078 (3) | 0.098 (3) | 0.074 (3) | -0.004 (3) | 0.025 (2) | 0.018 (3) |
|   |   |   |   |   |   |   |
|---|---|---|---|---|---|---|
|   |   |   |   |   |   |   |
| C14 | 0.065 (2) | 0.074 (2) | 0.052 (2) | 0.007 (2) | 0.0095 (17) | 0.0100 (18) |
| C16 | 0.097 (3) | 0.113 (4) | 0.060 (3) | 0.019 (3) | 0.028 (2) | 0.013 (3) |
| C30 | 0.088 (3) | 0.075 (3) | 0.085 (3) | −0.011 (3) | −0.004 (2) | 0.004 (2) |
| C24 | 0.097 (3) | 0.100 (4) | 0.084 (3) | −0.030 (3) | −0.002 (3) | 0.025 (3) |
| C5  | 0.096 (3) | 0.089 (3) | 0.065 (3) | 0.011 (3)  | 0.001 (2)  | 0.014 (2) |
| C27 | 0.098 (3) | 0.071 (3) | 0.059 (3) | −0.008 (2) | −0.011 (2) | 0.008 (2) |
| C36 | 0.084 (3) | 0.068 (3) | 0.068 (3) | −0.017 (2) | −0.005 (2) | 0.003 (2) |
| C18 | 0.084 (3) | 0.086 (3) | 0.059 (2) | 0.007 (2)  | 0.017 (2)  | −0.007 (2) |
| C2  | 0.083 (3) | 0.099 (4) | 0.091 (4) | 0.020 (3)  | 0.006 (3)  | 0.001 (3) |
| C4  | 0.114 (4) | 0.086 (3) | 0.084 (4) | 0.013 (3)  | −0.008 (3) | 0.023 (3) |
| C26 | 0.075 (3) | 0.084 (3) | 0.093 (3) | −0.019 (2) | 0.013 (2)  | −0.003 (3) |
| C3  | 0.102 (4) | 0.093 (4) | 0.100 (4) | 0.035 (3)  | −0.008 (3) | 0.000 (3) |
| C10 | 0.161 (5) | 0.091 (4) | 0.064 (3) | 0.059 (4)  | 0.001 (3)  | 0.002 (3) |
| C28 | 0.114 (4) | 0.099 (4) | 0.064 (3) | −0.013 (3) | −0.027 (3) | 0.006 (3) |
| C17 | 0.097 (3) | 0.117 (4) | 0.055 (2) | 0.015 (3)  | 0.019 (2)  | −0.004 (3) |
| C29 | 0.089 (3) | 0.088 (3) | 0.082 (3) | −0.016 (3) | −0.011 (3) | −0.009 (3) |
| C39 | 0.099 (4) | 0.100 (4) | 0.093 (4) | 0.017 (3)  | −0.016 (3) | −0.006 (3) |
| C25 | 0.103 (4) | 0.073 (3) | 0.110 (4) | −0.020 (3) | 0.021 (3)  | −0.009 (3) |
| C37 | 0.113 (4) | 0.136 (5) | 0.058 (3) | 0.020 (4)  | −0.006 (3) | −0.006 (3) |
| C38 | 0.142 (5) | 0.139 (6) | 0.077 (4) | 0.015 (5)  | −0.010 (3) | 0.022 (4) |
| C11 | 0.174 (7) | 0.119 (6) | 0.216 (10) | −0.016 (6) | 0.064 (7)  | −0.104 (7) |
| C12 | 0.224 (10) | 0.199 (10) | 0.109 (6) | 0.031 (9)  | 0.022 (6)  | −0.063 (7) |
| C13 | 0.120 (5)  | 0.137 (6)  | 0.126 (6) | 0.026 (4)  | 0.043 (4)  | −0.021 (5) |

**Geometric parameters (Å, º)**

|   |   |   |   |   |   |   |
|---|---|---|---|---|---|---|
| N11—C35 | 1.338 (5) | C15—C16 | 1.361 (8) |
| N11—N10  | 1.361 (5) | C15—C14  | 1.403 (6) |
| N11—H11  | 0.8600   | C15—H15  | 0.9300   |
| N6—C21   | 1.322 (5) | C16—C17  | 1.356 (8) |
| N6—N7    | 1.349 (5) | C16—H16  | 0.9300   |
| N5—C20   | 1.287 (5) | C30—C29  | 1.369 (7) |
| N5—C21   | 1.400 (5) | C30—H30  | 0.9300   |
| N9—C33   | 1.287 (6) | C24—C25  | 1.530 (8) |
| N9—C34   | 1.376 (5) | C24—H24A | 0.9700   |
| N12—C35  | 1.330 (5) | C24—H24B | 0.9700   |
| N12—C34  | 1.368 (5) | C5—C4    | 1.391 (8) |
| O2—C14   | 1.349 (5) | C5—H5    | 0.9300   |
| O2—H2    | 0.8200   | C27—C28  | 1.386 (7) |
| N10—C34  | 1.308 (5) | C36—C39  | 1.523 (7) |
| N7—C22   | 1.330 (5) | C36—C37  | 1.533 (8) |
| N7—H7D   | 0.8600   | C36—H36  | 0.9800   |
| O1—C1    | 1.355 (6) | C18—C17  | 1.395 (7) |
| O1—H1    | 0.8200   | C18—H18  | 0.9300   |
| N8—C22   | 1.328 (5) | C2—C3    | 1.355 (9) |
| N8—C21   | 1.341 (5) | C2—H2A   | 0.9300   |
| N3—C9    | 1.347 (6) | C4—C3    | 1.377 (8) |
| N3—N2    | 1.349 (6) | C4—H4    | 0.9300   |

*Acta Cryst. (2021). E77, 1267-1271*
| Bond | Distance (Å) | Bond | Distance (Å) | Bond | Distance (Å) |
|------|-------------|------|-------------|------|-------------|
| N3—H3D | 0.8600 | C26—C25 | 1.498 (8) |
| N1—C7 | 1.276 (6) | C26—H26A | 0.9700 |
| N1—C8 | 1.383 (6) | C26—H26B | 0.9700 |
| N4—C9 | 1.329 (6) | C3—H3A | 0.9300 |
| N4—C8 | 1.353 (5) | C10—C11 | 1.503 (10) |
| N2—C8 | 1.316 (5) | C10—C13 | 1.524 (10) |
| C20—C19 | 1.451 (5) | C10—H10 | 0.9800 |
| C20—H20 | 0.9300 | C28—C29 | 1.361 (8) |
| C35—C36 | 1.476 (6) | C28—H28 | 0.9300 |
| O3—C27 | 1.351 (6) | C17—H17 | 0.9300 |
| O3—H3 | 0.8200 | C29—H29 | 0.9300 |
| C7—C6 | 1.434 (6) | C39—C38 | 1.502 (9) |
| C7—H7 | 0.9300 | C39—H39A | 0.9700 |
| C32—C31 | 1.393 (6) | C39—H39B | 0.9700 |
| C32—C27 | 1.402 (6) | C25—H25A | 0.9700 |
| C32—C33 | 1.439 (6) | C25—H25B | 0.9700 |
| C22—C23 | 1.481 (6) | C37—C38 | 1.501 (9) |
| C33—H33 | 0.9300 | C37—H37A | 0.9700 |
| C19—C14 | 1.386 (6) | C37—H37B | 0.9700 |
| C19—C18 | 1.396 (6) | C38—H38A | 0.9700 |
| C31—C30 | 1.362 (7) | C38—H38B | 0.9700 |
| C31—H31 | 0.9300 | C11—C12 | 1.450 (13) |
| C23—C26 | 1.517 (7) | C11—H11A | 0.9700 |
| C23—C24 | 1.527 (8) | C11—H11B | 0.9700 |
| C34—N11—N10 | 109.8 (3) | C12—C13 | 1.493 (13) |
| C5—N11—H11 | 125.1 | C12—H12A | 0.9700 |
| C21—N6—N7 | 101.7 (3) | C12—H12B | 0.9700 |
| C20—N5—C21 | 117.5 (3) | C3—C2—C1 | 121.4 (5) |
| C33—N9—C34 | 120.1 (3) | C3—C2—H2A | 119.3 |
| C35—N12—C34 | 102.7 (3) | C3—C2—H2A | 119.3 |
| C14—O2—H2 | 109.5 | C3—C2—H2A | 119.3 |
| C34—N10—N11 | 103.0 (3) | C39—C36—C37 | 119.4 (4) |
| C22—N7—N6 | 110.8 (3) | C39—C36—C37 | 119.4 (4) |
| C22—N7—H7D | 124.6 | C39—C36—C37 | 119.4 (4) |
| N6—N7—H7D | 124.6 | C39—C36—C37 | 119.4 (4) |
| C1—O1—H1 | 109.5 | C17—C18—C19 | 119.8 (5) |
| C22—N8—C21 | 103.1 (3) | C17—C18—H18 | 120.1 |
| C9—N3—N2 | 110.8 (4) | C19—C18—H18 | 120.1 |
| C9—N3—H3D | 124.6 | C3—C2—C1 | 121.4 (5) |
| N2—N3—H3D | 124.6 | C3—C2—H2A | 119.3 |
| C7—N1—C8 | 119.8 (4) | C1—C2—H2A | 119.3 |
| Bond | Angle | Torsion | Angle | Torsion |
|------|-------|---------|-------|---------|
| N6—C21—N8 | 115.1 (3) | C3—C4—C5 | 119.0 (5) | |
| N6—C21—N5 | 118.4 (3) | C3—C4—H4 | 120.5 | |
| N8—C21—N5 | 126.5 (3) | C5—C4—H4 | 120.5 | |
| C9—N4—C8 | 103.7 (4) | C25—C26—C23 | 90.3 (4) | |
| C8—N2—N3 | 102.4 (3) | C25—C26—H26A | 113.6 | |
| N5—C20—C19 | 121.1 (4) | C23—C26—H26A | 113.6 | |
| N5—C20—H20 | 119.5 | C25—C26—H26B | 113.6 | |
| C19—C20—H20 | 119.5 | C23—C26—H26B | 113.6 | |
| N12—C35—N11 | 109.9 (3) | H26A—C26—H26B | 110.9 | |
| N12—C35—C36 | 126.0 (4) | C2—C3—C4 | 120.7 (5) | |
| N11—C35—C36 | 124.1 (4) | C2—C3—H3A | 119.7 | |
| N10—C34—N12 | 114.6 (4) | C4—C3—H3A | 119.7 | |
| N10—C34—N9 | 119.5 (4) | C9—C10—C11 | 117.0 (6) | |
| N12—C34—N9 | 125.9 (4) | C9—C10—C13 | 118.6 (5) | |
| C27—O3—H3 | 109.5 | C11—C10—C13 | 86.9 (6) | |
| N2—C8—N4 | 114.7 (4) | C9—C10—H10 | 110.8 | |
| N2—C8—N1 | 118.9 (3) | C11—C10—H10 | 110.8 | |
| N4—C8—N1 | 126.4 (4) | C13—C10—H10 | 110.8 | |
| N1—C7—C6 | 122.8 (4) | C29—C28—C27 | 120.6 (5) | |
| N1—C7—H7 | 118.6 | C29—C28—H28 | 119.7 | |
| C6—C7—H7 | 118.6 | C27—C28—H28 | 119.7 | |
| C31—C32—C27 | 117.6 (4) | C16—C17—C18 | 119.6 (5) | |
| C31—C32—C33 | 120.9 (4) | C16—C17—H17 | 120.2 | |
| C27—C32—C33 | 121.5 (4) | C18—C17—H17 | 120.2 | |
| N8—C22—N7 | 109.3 (3) | C28—C29—C30 | 121.0 (5) | |
| N8—C22—C23 | 126.8 (4) | C28—C29—H29 | 119.5 | |
| N7—C22—C23 | 123.7 (4) | C30—C29—H29 | 119.5 | |
| N9—C33—C32 | 121.9 (4) | C38—C39—C36 | 88.9 (5) | |
| N9—C33—H33 | 119.1 | C38—C39—H39A | 113.8 | |
| C32—C33—H33 | 119.1 | C36—C39—H39A | 113.8 | |
| C14—C19—C18 | 119.7 (4) | C38—C39—H39B | 113.8 | |
| C14—C19—C20 | 122.0 (4) | C36—C39—H39B | 113.8 | |
| C18—C19—C20 | 118.2 (4) | H39A—C39—H39B | 111.1 | |
| C30—C31—C32 | 122.3 (4) | C26—C25—C24 | 87.8 (4) | |
| C30—C31—H31 | 118.8 | C26—C25—H25A | 114.0 | |
| C32—C31—H31 | 118.8 | C24—C25—H25A | 114.0 | |
| C22—C23—C26 | 119.4 (4) | C26—C25—H25B | 114.0 | |
| C22—C23—C24 | 116.7 (4) | C24—C25—H25B | 114.0 | |
| C26—C23—C24 | 87.2 (4) | H25A—C25—H25B | 111.2 | |
| C22—C23—H23 | 110.5 | C38—C37—C36 | 88.5 (5) | |
| C26—C23—H23 | 110.5 | C38—C37—H37A | 113.9 | |
| C24—C23—H23 | 110.5 | C36—C37—H37A | 113.9 | |
| O1—C1—C2 | 118.8 (5) | C38—C37—H37B | 113.9 | |
| O1—C1—C6 | 121.7 (4) | C36—C37—H37B | 113.9 | |
| C2—C1—C6 | 119.6 (5) | H37A—C37—H37B | 111.1 | |
| C5—C6—C1 | 118.2 (4) | C37—C38—C39 | 88.9 (4) | |
| C5—C6—C7 | 120.4 (4) | C37—C38—H38A | 113.8 | |
| C1—C6—C7 | 121.4 (4) | C39—C38—H38A | 113.8 | |
| Bond                 | Angle (°) (e) | Bond                 | Angle (°) (e) |
|---------------------|--------------|---------------------|--------------|
| N4—C9—N3            | 108.5 (4)    | C37—C38—H38B        | 113.8        |
| N4—C9—C10           | 126.9 (4)    | C39—C38—H38B        | 113.8        |
| N3—C9—C10           | 124.3 (4)    | H38A—C38—H38B       | 111.1        |
| C16—C15—C14         | 119.9 (5)    | C12—C11—C10         | 89.7 (7)     |
| C16—C15—H15         | 120.1        | C12—C11—H11A        | 113.7        |
| C14—C15—H15         | 120.1        | C10—C11—H11A        | 113.7        |
| O2—C14—C19          | 122.7 (4)    | C12—C11—H11B        | 113.7        |
| O2—C14—C15          | 118.2 (4)    | C10—C11—H11B        | 113.7        |
| C19—C14—C15         | 119.2 (4)    | H11A—C11—H11B       | 110.9        |
| C17—C16—C15         | 121.9 (5)    | C11—C12—C13         | 90.0 (6)     |
| C17—C16—H16         | 119.1        | C11—C12—H12A        | 113.6        |
| C15—C16—H16         | 119.1        | C13—C12—H12A        | 113.6        |
| C31—C30—C29         | 119.0 (5)    | C11—C12—H12B        | 113.6        |
| C31—C30—H30         | 120.5        | C13—C12—H12B        | 113.6        |
| C29—C30—H30         | 120.5        | H12A—C12—H12B       | 110.9        |
| C23—C24—C25         | 88.7 (4)     | C12—C13—C10         | 87.3 (7)     |
| C23—C24—H24A        | 113.9        | C12—C13—H13A        | 114.1        |
| C25—C24—H24A        | 113.9        | C10—C13—H13A        | 114.1        |
| C23—C24—H24B        | 113.9        | C12—C13—H13B        | 114.1        |
| C25—C24—H24B        | 113.9        | C10—C13—H13B        | 114.1        |
| H24A—C24—H24B       | 111.1        | H13A—C13—H13B       | 111.3        |

| Bond                 | Bond                 | Bond                 | Bond                 |
|---------------------|---------------------|---------------------|---------------------|
| C35—N11—N10—C34    | -0.4 (5)            | C20—C19—C14—O2     | 0.6 (6)             |
| C21—N6—N7—C22      | 0.6 (5)             | C18—C19—C14—C15    | -0.3 (6)            |
| N7—N6—C21—N8       | -0.2 (5)            | C20—C19—C14—C15    | -179.9 (4)          |
| N7—N6—C21—N5       | 180.0 (4)           | C16—C15—C14—O2     | -179.7 (5)          |
| C22—N8—C21—N6      | -0.3 (5)            | C16—C15—C14—C19    | 0.7 (7)             |
| C22—N8—C21—N5      | 179.5 (4)           | C14—C15—C16—C17    | -0.4 (8)            |
| C20—N5—C21—N6      | -166.4 (4)          | C32—C31—C30—C29    | 1.2 (8)             |
| C20—N5—C21—N8      | 13.8 (6)            | C22—C23—C24—C25    | -139.8 (4)          |
| C9—N3—N2—C8        | -0.6 (5)            | C26—C23—C24—C25    | -18.1 (4)           |
| C21—N5—C20—C19     | -180.0 (4)          | C1—C6—C5—C4        | 0.4 (7)             |
| C34—N12—C35—N11    | 0.7 (5)             | C7—C6—C5—C4        | 178.0 (5)           |
| C34—N12—C35—C36    | 179.8 (5)           | C31—C32—C27—O3     | 177.7 (5)           |
| N10—N11—C35—N12    | -0.2 (5)            | C33—C32—C27—O3     | -3.1 (8)            |
| N10—N11—C35—C36    | -179.3 (4)          | C31—C32—C27—C28    | -0.3 (7)            |
| N11—N10—C34—N9     | 0.9 (5)             | C33—C32—C27—C28    | 178.9 (5)           |
| N11—N10—C34—N9     | -179.0 (4)          | N12—C35—C36—C39    | -47.2 (7)           |
| C35—N12—C34—N10    | -1.1 (5)            | N11—C35—C36—C39    | 131.7 (5)           |
| C35—N12—C34—N9     | 178.9 (5)           | N12—C35—C36—C37    | 56.2 (7)            |
| C33—N9—C34—N10     | 178.8 (4)           | N11—C35—C36—C37    | -124.8 (5)          |
| C33—N9—C34—N12     | -1.1 (7)            | C14—C19—C18—C17    | -0.5 (7)            |
| N3—N2—C8—N4        | 0.3 (5)             | C20—C19—C18—C17    | 179.1 (4)           |
| N3—N2—C8—N1        | -178.3 (4)          | O1—C1—C2—C3        | 179.8 (5)           |
| C9—N4—C8—N2        | 0.2 (6)             | C6—C1—C2—C3        | 0.5 (8)             |
| C9—N4—C8—N1        | 178.6 (5)           | C6—C5—C4—C3        | -0.8 (9)            |
| C7—N1—C8—N2        | -165.7 (4)          | C22—C23—C26—C25    | 137.8 (5)           |
| C7—N1—C8—N4        | 15.9 (7)            | C24—C23—C26—C25    | 18.5 (4)            |
C8—N1—C7—C6 179.3 (4)  C1—C2—C3—C4  1.1 (9)
C21—N8—C22—N7  0.6 (5)   C5—C4—C3—C2  72.4 (9)
C21—N8—C22—C23  174.3 (4)  N4—C9—C10—C11  100.3 (8)
N6—N7—C22—N8  0.8 (5)  N3—C9—C10—C13  29.6 (10)
N6—N7—C22—C23  174.3 (4)  N3—C9—C10—C13  157.6 (6)
C34—N9—C33—C32  179.4 (4)  O3—C27—C28—C29  178.4 (6)
C21—N8—C22—C23  0.2 (7)  C32—C27—C28—C29  0.5 (9)
N6—N7—C22—C23  179.4 (4)  N4—C9—C10—C13  157.6 (6)
N5—C20—C19—C14  4.4 (6)  C15—C16—C17—C18  0.4 (8)
N5—C20—C19—C18  175.2 (4)  C19—C18—C17—C16  9.8 (8)
C27—C32—C31—C30  0.1 (7)  C27—C28—C29—C30  1.6 (9)
C33—C32—C31—C30  179.3 (5)  C31—C30—C29—C28  1.9 (9)
N8—C22—C23—C26  156.2 (4)  C35—C36—C39—C38  19.3 (5)
N7—C22—C23—C26  73.2 (6)  C23—C26—C25—C24  18.5 (4)
N7—C22—C23—C26  101.0 (5)  C25—C24—C23—C22  18.4 (4)
O1—C1—C6—C5  3.0 (6)  C35—C36—C37—C38  19.6 (5)
C2—C1—C6—C5  0.2 (7)  C39—C38—C37—C36  19.3 (5)
O1—C1—C6—C7  3.0 (6)  C36—C37—C38—C39  19.6 (5)
C2—C1—C6—C7  177.8 (4)  C36—C39—C38—C37  19.7 (5)
N1—C7—C6—C5  175.3 (5)  C9—C10—C11—C12  139.3 (7)
N1—C7—C6—C1  2.2 (7)  C13—C10—C11—C12  18.5 (8)
C8—N4—C9—N3  0.6 (6)  C10—C11—C12—C13  18.9 (8)
C8—N4—C9—C10  174.3 (6)  C11—C12—C13—C10  18.7 (8)
N2—N3—C9—N4  0.8 (6)  C9—C10—C13—C12  137.3 (7)
N2—N3—C9—C10  174.7 (5)  C11—C10—C13—C12  18.0 (7)
C18—C19—C14—O2  179.7 (4)

Hydrogen-bond geometry (Å, °)

| D—H···A | D—H | H···A | D···A | D—H···A |
|---------|-----|-------|-------|---------|
| N7—H7D···N2 | 0.86 | 1.98  | 2.836 (5) | 174 |
| N11—H11···N6  | 0.86 | 1.99  | 2.817 (5) | 161 |
| O1—H1···N1   | 0.82 | 1.89  | 2.615 (5) | 146 |
| O2—H2···N5   | 0.82 | 1.90  | 2.619 (4) | 146 |
| O3—H3···N9   | 0.82 | 1.87  | 2.588 (5) | 146 |
| C10—H10···O3  | 0.98 | 2.52  | 3.411 (6) | 151 |
| N3—H3D···N10  | 0.86 | 2.07  | 2.874 (5) | 155 |

Symmetry codes: (i) x−2, y, z; (ii) x−1, y, z.