Crystal structure, Hirshfeld surface analysis and DFT calculations of (E)-3-[1-(2-hydroxyphenyl-anilino)ethylidene]-6-methylpyran-2,4-dione

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The asymmetric unit of the title compound, C14H13NO4, contains three independent molecules, which differ slightly in conformation. Each contains an intramolecular N—H⋯C1/C1/O hydrogen bond. In the crystal, O—H⋯C1/C1/O hydrogen bonds form chains of molecules, which are linked into corrugated sheets parallel to (103) plane by C—H⋯C1/C1/O hydrogen bonds together with H/C25 interactions between the carbonyl groups and the 2-hydroxyphenyl rings. The layers are linked by further C—H⋯C1/C1/O hydrogen bonds. The Hirshfeld surface analysis of the crystal structure indicates that the most important contributions for the crystal packing are from H/C1/C1/O (49.0%), H/C1/C1/O/C1/C1/H (28.3%) and H/C1/C1/C/C (10.9%) interactions. van der Waals interactions are the dominant interactions in the crystal packing. Moreover, density functional theory (DFT) optimized structures at the B3LYP/6–311G(d,p) level are compared with the experimentally determined molecular structure in the solid state. The HOMO–LUMO behavior was elucidated to determine the energy gap of 4.53 eV.

1. Chemical context

Heterocyclic molecules play a very important role in life processes and are of major interest in the industrial development of dyes, pharmaceuticals, pesticides, and natural products (Saber et al., 2020; El Ghayati et al., 2021; Patra & Saxena, 2010). Therefore, scientists have devoted considerable effort to finding efficient synthetic methods for a wide variety of heterocyclic compounds (Yeh et al., 2014; Liaw et al., 2015). Among these molecules, pyrone derivatives constitute an important class in the heterocycle family since the pyrone structural unit is found in a wide variety of natural bioactive compounds (McGlacken & Fairlamb, 2005; Beckert et al., 1997) and also in a wide range of synthetic products with demonstrated efficacy in various fields such as the pharmaceutical and therapeutic field as cytotoxic (Calderón-Montaño et al., 2013), antitumor (Suzuki et al., 1997; Kondoh et al., 1998) and antimicrobial agents (Fairlamb et al., 2004). Another representative example of the pyrone class of compounds, kavalactones, possess many biological activities such as antituberculosis, local anesthetic, anticonvulsant, analgesic, anti-
malarial, and sedative activities (Altomare et al., 1997; Scherer, 1998; Bilia et al., 2002; Ernst, 2007). In this work, we report the synthesis of (E)-3-[1-(2-hydroxyphenylanilino)-ethyldiene]-6-methylpyran-2,4-dione, (I) (Fig. 1) in good yield by the condensation of 2-aminophenol and dehydroacetic acid along with its crystal and molecular structures as well as the Hirshfeld surface analysis and the density functional theory (DFT) computational calculations carried out at the B3LYP/6–311G(d,p) levels.

2. Structural commentary

The asymmetric unit of the title compound comprises three independent molecules, two of which (those containing O5 and O9) differ modestly in the orientations of the methyl groups while the third differs more in conformation from the other two (Fig. 1). In each molecule, the conformation is partially determined by an intramolecular N—H···O hydrogen bond (Fig. 1 and Table 1), which can be described as a resonance-assisted hydrogen bond (RAHB). With reference to the scheme below, in the three independent molecules the bonds designated a are the same within experimental error. The same is true for each of the bonds labeled b–f and the average values are a = 1.323 (3) Å, b = 1.431 (3) Å, c = 1.447 (3) Å, d = 1.433 (3) Å, e = 1.226 (3) Å and f = 1.254 (3) Å. These compare quite favorably with those found in molecules with R = Me (Gilli et al., 2000) and 4-XC6H4 (X = F, Cl, Br; Boulemche et al., 2019) and accompanied by in depth discussions of the RAHB.

3. Supramolecular features

In the crystal, chains containing all three independent molecules are formed by O1—H1B···O7, O5—H5B···O11 and O9—H9B···O3 hydrogen bonds repeating in that order (Table 1 and Fig. 2). The chains are linked into corrugated layers parallel to the (010) plane by C8—H8C···O8, C33—H33C···O3 and C36—H36B···O12 hydrogen bonds together.

Table 1

| D—H···A       | D—H | H···A | D···A | D—H···A |
|---------------|-----|-------|-------|---------|
| O1—H1B···O7   | 0.87| 1.79  | 2.662 | 2 (177) |
| N1—H1A···O2   | 0.91| 1.72  | 2.538 | (3) 148 |
| C8—H8C···O8   | 0.98| 2.48  | 3.441 | (3) 167 |
| C11—H11···O6  | 0.95| 2.57  | 3.253 | (3) 129 |
| O5—H5B···O11  | 0.87| 1.83  | 2.689 | (2) 170 |
| N2—H2A···O6   | 0.91| 1.71  | 2.539 | (3) 151 |
| O9—H9B···O3   | 0.87| 1.82  | 2.691 | (2) 129 |
| N3—H3A···O10  | 0.91| 1.71  | 2.532 | (3) 148 |
| C33—H33···O3  | 0.95| 2.53  | 3.225 | (3) 130 |
| C36—H36B···O12| 0.98| 2.56  | 3.531 | (3) 173 |

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

Figure 1

The asymmetric unit with the atom-labeling scheme and 50% probability ellipsoids. The intramolecular hydrogen bonds are depicted by dashed lines.

Figure 2

A portion of one layer viewed along the b-axis direction (left) and along the c-axis direction (right) with O···H···O and C···H···O hydrogen bonds depicted, respectively, by red and black dashed lines. Non-interacting H atoms are omitted for clarity.
with π interactions (Fig. 3) between the carbonyl groups and the 2-hydroxyphenyl rings [O2 ··Cg2 = 3.4827 (18) Å, C10 ··Cg2 = 3.731 (2) Å, C10=O2 ··Cg2 = 91.41 (13)° (Cg2 is the centroid of the C1–C6 ring at x + 3/2, y + 1/2, z + 1/2); O6 ··Cg6 = 3.451 (2) Å, C24 ··Cg6 = 3.694 (2) Å, C24=O6 ··Cg6 = 91.12 (14)° (Cg6 is the centroid of the C29-C34 ring at x, y, z); O10 ··Cg4 = 3.4110 (18) Å, C38 ··Cg4 = 3.656 (2) Å, C38=O10 ··Cg4 = 91.00 (13)° (Cg4 is the centroid of the C15−C20 ring at x, y − 1, z)]. The layers are held together by C11−H11 ··O6 hydrogen bonds (Table 1 and Fig. 3).

4. Hirshfeld surface analysis

In order to visualize the intermolecular interactions, a Hirshfeld surface (HS) analysis (Hirshfeld, 1977) was carried out using Crystal Explorer 17.5 (Turner et al., 2017). In the HS plotted over \(d_{\text{norm}}\) (Fig. 4), the white surface indicates contacts with distances equal to the sum of van der Waals radii, and the red and blue colors indicate distances shorter (in close contact) or longer (distinct contact) than the sum of the van der Waals radii, respectively (Venkatesan et al., 2016). The shape-index of the HS is a tool to visualize π−π stacking by the presence of adjacent red and blue triangles; if there are no adjacent red and/or blue triangles, then there are no π−π interactions. Fig. 5 clearly suggests that there are π−π interactions in (I). The overall two-dimensional fingerprint plot, Fig. 6a, and those delineated into H·-H, H··O/O··H,
H - C/C - H, C - C, C - O/O - O - C, O - O, N - O/O - N, H - N/N - H, N - N and C - N/N - C contacts (McKinnon et al., 2007) are illustrated in Fig. 6 b–k, respectively, together with their relative contributions to the Hirshfeld surface. The most important interaction is H - H contributing 49.0% to the overall crystal packing, which is reflected in Fig. 6b as widely scattered points of high density due to the large hydrogen content of the molecule with the tip at $d_e = d_i = 1.09\,\text{Å}$. The pair of spikes in the fingerprint plot delineated into H - O/O - H contacts with a 28.3% contribution to the HS, Fig. 6c, has a symmetric distribution of points with the tips at $d_e + d_i = 1.69\,\text{Å}$. In the presence of C - H - π interactions, the pair of characteristic wings in the fingerprint plot delineated into H - C/C - C - H contacts, Fig. 6d, with a 10.9% contribution to the HS has the tips at $d_e + d_i = 2.67\,\text{Å}$. The C - C, C - H, Fig. 6e, with a 6.2% contribution to the HS have a bullet-shaped distribution of points and the tip at $d_e = d_i = 1.64\,\text{Å}$. The symmetric distribution of points for the C - O/O - O - C contacts, Fig. 6f, with 3.8% contribution to the HS has a pair of the scattered points of spikes with the tips at $d_e + d_i = 3.11\,\text{Å}$. Finally, the contributions of the remaining O - O, N - O/O - O - N, H - N/N - H, N - N and C - N/N - C contacts (Fig. 6g-k) are smaller than 1.0% with low densities of points.

The Hirshfeld surface representations with the function $d_{norm}$ plotted onto the surface for the H - H, H - O/O - H and H - C/C - H interactions in Fig. 7a–c, respectively. The Hirshfeld surface analysis confirms the importance of H-atom contacts in establishing the packing. The large number of H - H, H - O/O - H and H - C/C - H interactions suggest that van der Waals interactions play the major role in the crystal packing (Hathwar et al., 2015).

5. DFT calculations

The optimized structure of the title compound in the gas phase was generated theoretically via density functional theory (DFT) using the standard B3LYP functional and 6-311 G(d,p) basis-set calculations (Becke, 1993) as implemented in GAUSSIAN 09 (Frisch et al., 2009). The theoretical and experimental results are in good agreement (Table 2). The highest-occupied molecular orbital (HOMO), acting as an electron donor, and the lowest-unoccupied molecular orbital (LUMO), acting as an electron acceptor, are very important parameters for quantum chemistry. When the energy gap is small, the molecule is highly polarizable and has high chemical reactivity. The DFT calculations provide some important information on the reactivity and site selectivity of the molecular framework. $E_{\text{HOMO}}$ and $E_{\text{LUMO}}$, which clarify the inevitable charge-exchange collaboration inside the molecule, electronegativity (χ), hardness (η), potential (µ), electrophilicity (ω) and softness (σ) are recorded in Table 3. The significance of η and σ is to evaluate both the reactivity and stability. The electron transition from the HOMO to the LUMO energy level is shown in Fig. 8. The HOMO and LUMO are localized in the plane extending from the whole (E)-3-[1-(2-hydroxyphenylamino)ethylidene]-6-methyl-3H-pyran-2,4-dione ring. The energy band gap [$ΔE = E_{\text{LUMO}} - E_{\text{HOMO}}$] of the molecule is 4.54 eV, and the frontier molecular orbital energies, $E_{\text{HOMO}}$ and $E_{\text{LUMO}}$ are −6.12 and −1.58 eV, respectively.

6. Molecular electrostatic (MEP)

Molecular electrostatic potential (MEP) was used to broadly predict reactive sites for electrophilic and nucleophilic attack.
in the title compound by B3LYP/6-31G optimized geometries using Gaussview software (Frisch et al., 2009). The total electron density onto which the electrostatic potential surface has been mapped is shown in Fig. 9. This figure gives a visual representation of the chemically active sites and comparative reactivity of atoms where red regions denote the most negative electrostatic potential, blue represents regions of the most positive electrostatic potential, and green represents the region of zero potential. The distribution favors the existence of the intra and intermolecular C—H···O and N—H···O hydrogen bonding.

| Molecular energy | Compound (I) |
|------------------|--------------|
| Total energy, TE (eV) | -24399.73 |
| $E_{\text{HOMO}}$ (eV) | -6.12 |
| $E_{\text{LUMO}}$ (eV) | -1.58 |
| Gap, $\Delta E_{\text{HOMO-LUMO}}$ (eV) | 4.53 |
| Dipole moment, $\mu$ (Debye) | 4.1895 |
| Ionization potential, $I$ (eV) | 6.12 |
| Electron affinity, $A$ | 1.58 |
| Electronegativity, $\chi$ | 3.85 |
| Hardness, $\eta$ | 2.27 |
| Electrophilicity index, $\omega$ | 3.27 |
| Softness, $\sigma$ | 0.44 |
| Fraction of electron transferred, $\Delta N$ | 0.69 |

7. Database survey

A search of the Cambridge Structural Database (CSD, version 5.43, updated to March 2022; Groom et al., 2016) for the fragment $A$ (allowing $R$ to be any substituent) yielded 66 hits of which 15 were deemed most similar to the title molecule. These include molecules with $R = \text{Me}$ (FOTQOW; Kwocz et al., 2015), $p$-anis (GOWYOG; Gilli et al., 2000), 4-ClC$_6$H$_4$ (GOXLOU, GOXLOU02; Boulemche et al., 2019), 4-BrC$_6$H$_4$ (VOPLOC01; Boulemche et al., 2019), Et (HABNED; Xiao et al., 1993), H (HIVTUD; Seijas et al. 2014), Ph (PAEXPY; Gilli et al., 2000), 4-H$_2$NC$_6$H$_4$ (QADRIY; Užarević et al. 2010), 4-EtOC$_6$H$_4$ (QEOQEL; Djedouani et al., 2018), 4-MeOC$_6$H$_4$CH$_2$ (XECGEV; Wang et al., 2022), PhCH(Me) (XECGOF; Wang et al., 2022) and 2-CH$_2$C$_5$H$_4$N (XECHEW; Wang et al., 2022). Although not all of these reports discuss the intramolecular N—H···O hydrogen bonds in detail, it is clear that all have very similar metrical parameters to one another and to those in the title molecule.

8. Synthesis and crystallization

To a solution of 2-aminophenol (2.5 mmol) in 30 mL of ethanol, 2.5 mmol of dehydroacetic acid were added. The mixture was refluxed for 1 h. After cooling, the precipitate that formed was recrystallized from ethanol solution to give yellow crystals in 88% yield.
9. Refinement

Crystal, data collection and refinement details are presented in Table 4. Hydrogen atoms were included as riding contributions in idealized positions (O—H = 0.87 Å, N—H = 0.91 Å, C—H = 0.95–0.98 Å) with $U_{iso}(H) = 1.2U_{eq}(C,N)$ or $1.5U_{eq}(O,C,methyl)$.

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Crystal structure, Hirshfeld surface analysis and DFT calculations of (E)-3-[1-(2-hydroxyphenylanilino)ethylidene]-6-methylpyran-2,4-dione

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Computing details
Data collection: APEX3 (Bruker, 2016); cell refinement: SAINT (Bruker, 2016); data reduction: SAINT (Bruker, 2016); program(s) used to solve structure: SHELXT5 (Sheldrick, 2015a); program(s) used to refine structure: SHELXL2018/3 (Sheldrick, 2015b); molecular graphics: DIAMOND (Brandenburg & Putz, 2012); software used to prepare material for publication: SHELXTL (Sheldrick, 2008).

(E)-3-[1-(2-Hydroxyphenylanilino)ethylidene]-6-methylpyran-2,4-dione

Crystal data

\[C_{14}H_{13}NO_{4}\]

\(M_r = 259.25\)

Monoclinic, \(P2_1/n\)

\(a = 11.6407 (4) \text{ Å}\)

\(b = 7.4412 (2) \text{ Å}\)

\(c = 42.2828 (12) \text{ Å}\)

\(\beta = 93.038 (2) ^\circ\)

\(V = 3657.42 (19) \text{ Å}^3\)

\(Z = 12\)

\(F(000) = 1632\)

\(D_x = 1.412 \text{ Mg m}^{-3}\)

Cu \(K\alpha\) radiation, \(\lambda = 1.54178 \text{ Å}\)

Cell parameters from 9858 reflections

\(\theta = 4.0-72.3^\circ\)

\(\mu = 0.87 \text{ mm}^{-1}\)

\(T = 150 \text{ K}\)

Plate, colourless

\(0.27 \times 0.07 \times 0.07 \text{ mm}\)

Data collection

Bruker D8 VENTURE PHOTON 100 CMOS diffractometer

Radiation source: INCOATEC \(\mu\)S micro-focus source

Mirror monochromator

Detector resolution: 10.4167 pixels mm\(^{-1}\)

\(\omega\) scans

Absorption correction: multi-scan

\(\text{(SADABS; Krause et al., 2015)}\)

7135 independent reflections

5024 reflections with \(I > 2\sigma(I)\)

\(R_{	ext{int}} = 0.070\)

\(\theta_{\text{max}} = 72.4^\circ, \theta_{\text{min}} = 3.9^\circ\)

\(h = -13\rightarrow14\)

\(k = -8\rightarrow9\)

\(l = -52\rightarrow51\)

Refinement

Refinement on \(F^2\)

Least-squares matrix: full

\(R[F^2 > 2\sigma(F^2)] = 0.056\)

\(wR(F^2) = 0.146\)

\(S = 1.04\)

7135 reflections

520 parameters

0 restraints

Primary atom site location: dual

Secondary atom site location: difference Fourier map

Hydrogen site location: mixed

H-atom parameters constrained
w = 1/\left[\sigma^2(F_o^2) + (0.0569P)^2 + 2.375P\right]

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

\((\Delta\sigma)_{\text{max}} < 0.001 \)

\( \Delta \rho_{\text{max}} = 0.49 \ \text{e} \ \text{Å}^{-3} \)

\( \Delta \rho_{\text{min}} = -0.43 \ \text{e} \ \text{Å}^{-3} \)

**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.** Refinement of \( F^2 \) against ALL reflections. The weighted R-factor \( wR \) and goodness of fit \( S \) are based on \( F^2 \), conventional R-factors \( R \) are based on \( F \), with \( F \) set to zero for negative \( F^2 \). The threshold expression of \( F^2 > 2\sigma(F^2) \) is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on \( F^2 \) are statistically about twice as large as those based on \( F \), and R-factors based on ALL data will be even larger. H-atoms attached to carbon were placed in calculated positions (C—H = 0.95 - 0.98 Å) while those attached to nitrogen and to oxygen were placed in locations derived from a difference map and their parameters adjusted to give N—H = 0.91 and O—H = 0.87 Å. All were included as riding contributions with isotropic displacementparameters 1.2 - 1.5 times those of the attached atoms.

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

|     | x     | y     | z     | U(eq) |
|-----|-------|-------|-------|-------|
| O   | 0.61999 (14) | 0.3312 (2) | 0.21067 (4) | 0.0333 (4) |
| H1B | 0.582256 | 0.290388 | 0.193862 | 0.050* |
| O2  | 0.58889 (14) | 0.5447 (2) | 0.29280 (4) | 0.0341 (4) |
| O3  | 0.92249 (14) | 0.2912 (2) | 0.34495 (4) | 0.0357 (4) |
| O4  | 0.77660 (15) | 0.3859 (2) | 0.37143 (4) | 0.0383 (4) |
| N1  | 0.75437 (16) | 0.4599 (3) | 0.25901 (5) | 0.0266 (4) |
| H1A | 0.682525 | 0.495459 | 0.263795 | 0.032* |
| C1  | 0.7928 (2) | 0.4732 (3) | 0.22776 (6) | 0.0265 (5) |
| C2  | 0.8949 (2) | 0.5605 (3) | 0.22159 (6) | 0.0297 (5) |
| H2  | 0.941384 | 0.610471 | 0.238523 | 0.036* |
| C3  | 0.9286 (2) | 0.5746 (3) | 0.19105 (6) | 0.0352 (6) |
| H3  | 0.998909 | 0.632341 | 0.186842 | 0.042* |
| C4  | 0.8590 (2) | 0.5037 (4) | 0.16627 (6) | 0.0361 (6) |
| H4  | 0.883123 | 0.511350 | 0.145199 | 0.043* |
| C5  | 0.7553 (2) | 0.4223 (3) | 0.17201 (6) | 0.0329 (6) |
| H5  | 0.707586 | 0.377192 | 0.154888 | 0.040* |
| C6  | 0.7211 (2) | 0.4068 (3) | 0.20290 (6) | 0.0279 (5) |
| C7  | 0.81395 (19) | 0.3983 (3) | 0.28431 (6) | 0.0254 (5) |
| C8  | 0.92598 (19) | 0.3054 (3) | 0.28010 (6) | 0.0300 (5) |
| H8A | 0.935580 | 0.284869 | 0.257501 | 0.045* |
| H8B | 0.926635 | 0.189818 | 0.291232 | 0.045* |
| H8C | 0.989161 | 0.380487 | 0.288817 | 0.045* |
| C9  | 0.76450 (19) | 0.4190 (3) | 0.31430 (6) | 0.0269 (5) |
| C10 | 0.6510 (2) | 0.4963 (3) | 0.31640 (6) | 0.0285 (5) |
| C11 | 0.6080 (2) | 0.5161 (3) | 0.34727 (6) | 0.0291 (5) |
| H11 | 0.534842 | 0.570016 | 0.349396 | 0.035* |
| C12 | 0.6685 (2) | 0.4605 (3) | 0.37321 (6) | 0.0334 (6) |
| C13 | 0.8268 (2) | 0.3612 (3) | 0.34269 (6) | 0.0299 (5) |
| C14 | 0.6307 (3) | 0.4684 (5) | 0.40622 (7) | 0.0560 (8) |
| Atom  | x     | y     | z     | Ueq  |
|-------|-------|-------|-------|------|
| H14A  | 0.551287 | 0.512407 | 0.406083 | 0.084* |
| H14B  | 0.681053 | 0.550015 | 0.418746 | 0.084* |
| H14C  | 0.634724 | 0.347979 | 0.415605 | 0.084* |
| O5    | 0.38904 (14) | 0.6512 (2) | 0.45296 (4) | 0.0318 (4) |
| H5B   | 0.442098 | 0.689109 | 0.466620 | 0.048* |
| O6    | 0.35346 (14) | 0.4346 (3) | 0.37210 (4) | 0.0377 (4) |
| O7    | −0.00724 (15) | 0.6953 (2) | 0.34030 (4) | 0.0374 (4) |
| O8    | 0.12052 (17) | 0.6280 (2) | 0.30612 (4) | 0.0418 (5) |
| N2    | 0.21857 (16) | 0.5135 (3) | 0.41527 (5) | 0.0271 (4) |
| H2A   | 0.282524 | 0.476791 | 0.405602 | 0.033* |
| C15   | 0.21047 (19) | 0.5018 (3) | 0.44869 (6) | 0.0264 (5) |
| C16   | 0.1203 (2) | 0.4113 (3) | 0.46191 (6) | 0.0298 (5) |
| N16   | 0.061519 | 0.357723 | 0.448597 | 0.036* |
| C17   | 0.1160 (2) | 0.3992 (3) | 0.49450 (6) | 0.0331 (6) |
| H17   | 0.054670 | 0.337409 | 0.503716 | 0.040* |
| C18   | 0.2028 (2) | 0.4786 (3) | 0.51356 (6) | 0.0350 (6) |
| H18   | 0.198635 | 0.474495 | 0.535929 | 0.042* |
| C19   | 0.2955 (2) | 0.5638 (3) | 0.50054 (6) | 0.0318 (5) |
| H19   | 0.355280 | 0.614281 | 0.513917 | 0.038* |
| C20   | 0.30016 (19) | 0.5747 (3) | 0.46783 (6) | 0.0272 (5) |
| C21   | 0.14059 (19) | 0.5724 (3) | 0.39396 (6) | 0.0261 (5) |
| C22   | 0.0313 (2) | 0.6523 (3) | 0.40471 (6) | 0.0321 (5) |
| H22A  | 0.037677 | 0.669862 | 0.427702 | 0.048* |
| H22B  | −0.032943 | 0.571023 | 0.399231 | 0.048* |
| H22C  | 0.017599 | 0.768387 | 0.394222 | 0.048* |
| C23   | 0.1680 (2) | 0.5631 (3) | 0.36137 (6) | 0.0283 (5) |
| C24   | 0.2774 (2) | 0.4919 (3) | 0.35259 (6) | 0.0320 (5) |
| C25   | 0.3014 (2) | 0.4926 (3) | 0.31974 (6) | 0.0346 (6) |
| H25   | 0.371930 | 0.444072 | 0.313268 | 0.042* |
| C26   | 0.2256 (3) | 0.5610 (3) | 0.29800 (7) | 0.0407 (6) |
| C27   | 0.0887 (2) | 0.6317 (3) | 0.33735 (6) | 0.0321 (5) |
| C28   | 0.2375 (4) | 0.5756 (5) | 0.26316 (7) | 0.0691 (11) |
| H28A  | 0.316636 | 0.546088 | 0.258193 | 0.104* |
| H28B  | 0.219602 | 0.698620 | 0.256236 | 0.104* |
| H28C  | 0.184278 | 0.491755 | 0.252138 | 0.104* |
| O9    | 0.06273 (13) | 0.1503 (2) | 0.39114 (4) | 0.0304 (4) |
| H9B   | 0.017440 | 0.197100 | 0.376319 | 0.046* |
| O10   | 0.10567 (13) | −0.0837 (2) | 0.47274 (4) | 0.0321 (4) |
| O11   | 0.46131 (14) | 0.1935 (2) | 0.50481 (4) | 0.0326 (4) |
| O12   | 0.33818 (15) | 0.1056 (2) | 0.53955 (4) | 0.0347 (4) |
| N3    | 0.23507 (16) | 0.0148 (3) | 0.42968 (5) | 0.0263 (4) |
| H3A   | 0.171281 | −0.027001 | 0.438829 | 0.032* |
| C29   | 0.24495 (19) | 0.0095 (3) | 0.39632 (6) | 0.0260 (5) |
| C30   | 0.3378 (2) | −0.0727 (3) | 0.38300 (6) | 0.0304 (5) |
| H30   | 0.396873 | −0.125418 | 0.396305 | 0.036* |
| C31   | 0.3448 (2) | −0.0780 (3) | 0.35050 (6) | 0.0341 (6) |
| H31   | 0.408973 | −0.132571 | 0.341388 | 0.041* |
| C32   | 0.2569 (2) | −0.0025 (3) | 0.33125 (6) | 0.0356 (6) |
### Atomic displacement parameters (Å²)

|     | \(U^{11}\) | \(U^{22}\) | \(U^{33}\) | \(U^{12}\) | \(U^{13}\) | \(U^{23}\) |
|-----|-------------|-------------|-------------|-------------|-------------|-------------|
| O1  | 0.0348 (9)  | 0.0391 (10) | 0.0249 (9)  | -0.0058 (7) | -0.0089 (7) | -0.0014 (7) |
| O2  | 0.0313 (9)  | 0.0448 (10) | 0.0255 (9)  | 0.0051 (7)  | -0.0051 (7) | 0.0043 (7)  |
| O3  | 0.0324 (9)  | 0.0441 (10) | 0.0293 (10) | 0.0025 (8)  | -0.0107 (7) | 0.0057 (8)  |
| O4  | 0.0406 (10) | 0.0476 (11) | 0.0258 (9)  | -0.0066 (8) | -0.0063 (8) | 0.0060 (8)  |
| N1  | 0.0270 (10) | 0.0296 (10) | 0.0227 (10) | 0.0011 (8)  | -0.0041 (8) | -0.0006 (8) |
| C1  | 0.0312 (12) | 0.0259 (12) | 0.0218 (12) | 0.0054 (9)  | -0.0043 (9) | -0.0002 (9) |
| C2  | 0.0303 (12) | 0.0316 (13) | 0.0270 (13) | 0.0023 (9)  | -0.0004 (10)| -0.0009 (10)|
| C3  | 0.0352 (13) | 0.0358 (14) | 0.0349 (15) | 0.0053 (10) | 0.0039 (11) | 0.0033 (11) |
| C4  | 0.0443 (14) | 0.0404 (14) | 0.0242 (13) | 0.0105 (11) | 0.0056 (11) | 0.0008 (11) |
| C5  | 0.0425 (14) | 0.0345 (13) | 0.0210 (12) | 0.0063 (10) | -0.0051 (11)| -0.0045 (10)|
| C6  | 0.0336 (12) | 0.0263 (12) | 0.0233 (12) | 0.0025 (9)  | -0.0039 (10)| 0.0002 (9)  |
| C7  | 0.0246 (11) | 0.0260 (11) | 0.0247 (12) | -0.0041 (9) | -0.0067 (9) | 0.0025 (9)  |
| C8  | 0.0283 (12) | 0.0327 (13) | 0.0280 (13) | 0.0007 (9)  | -0.0063 (10)| 0.0017 (10) |
| C9  | 0.0266 (11) | 0.0300 (12) | 0.0232 (12) | -0.0029 (9) | -0.0068 (9) | 0.0012 (9)  |
| C10 | 0.0306 (12) | 0.0284 (12) | 0.0259 (13) | -0.0046 (9) | -0.0057 (10)| 0.0024 (10) |
| C11 | 0.0266 (11) | 0.0381 (13) | 0.0224 (12) | -0.0026 (10)| 0.0001 (9)  | -0.0011 (10)|
| C12 | 0.0345 (13) | 0.0413 (14) | 0.0241 (13) | -0.0081 (11)| -0.0009 (10)| 0.0002 (10) |
| C13 | 0.0341 (13) | 0.0309 (12) | 0.0238 (12) | -0.0073 (10)| -0.0056 (10)| 0.0016 (10) |
| C14 | 0.066 (2)   | 0.073 (2)   | 0.0297 (16) | -0.0211 (17)| 0.0054 (15) | -0.0052 (15)|
| O5  | 0.0311 (9)  | 0.0378 (9)  | 0.0255 (9)  | -0.0043 (7) | -0.0069 (7) | 0.0001 (7)  |
| O6  | 0.0313 (9)  | 0.0514 (11) | 0.0301 (10) | 0.0049 (8)  | -0.0010 (8) | 0.0032 (8)  |
| O7  | 0.0354 (10) | 0.0425 (10) | 0.0328 (10) | 0.0009 (8)  | -0.0125 (8) | 0.0065 (8)  |
| O8  | 0.0585 (12) | 0.0394 (10) | 0.0262 (10) | -0.0038 (9) | -0.0102 (9) | 0.0012 (8)  |
| N2  | 0.0243 (9)  | 0.0356 (11) | 0.0206 (10) | -0.0007 (8) | -0.0055 (8) | 0.0013 (8)  |
| C15 | 0.0291 (12) | 0.0282 (12) | 0.0214 (12) | 0.0063 (9)  | -0.0036 (9) | 0.0008 (9)  |

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### Geometric parameters (Å, °)

| Bond/Angle | Distances (Å) | Angles (°) |
|------------|---------------|------------|

**Bond Distances**

- O1—C6 1.361 (3)
- O1—H1B 0.8700
- O2—C10 1.253 (3)
- O3—C13 1.253 (3)
- O4—C12 1.381 (3)
- O4—C13 1.389 (3)
- N1—C7 1.326 (3)
- N1—C1 1.420 (3)
- N1—H1A 0.9101
- C1—C2 1.392 (3)
- C1—C6 1.397 (3)
- C2—C3 1.373 (4)
- C2—H2 0.9500

**Angle Distances**

- C19—C20 1.389 (3)
- C19—H19 0.9500
- C21—C22 1.497 (3)
- C22—H22A 0.9500
- C22—H22B 0.9500
- C22—H22C 0.9500
- C23—C24 1.432 (3)
- C23—C25 1.432 (4)
- C24—C25 1.432 (4)
- C25—H25 0.9500
- C26—C28 1.491 (4)
### Supporting Information

| Bond          | Distance (Å) | Distance (Å) | Angle (°) | Angle (°) |
|---------------|--------------|--------------|-----------|-----------|
| C3—C4         | 1.393 (4)    | C28—H28A     | 0.980     |           |
| C3—H3         | 0.9500       | C28—H28B     | 0.980     |           |
| C4—C5         | 1.384 (4)    | C28—H28C     | 0.980     |           |
| C4—H4         | 0.9500       | O9—C34       | 1.360 (3) |           |
| C5—C6         | 1.390 (3)    | O9—H9B       | 0.8701    |           |
| C5—H5         | 0.9500       | O10—C38      | 1.257 (3) |           |
| C7—C9         | 1.428 (3)    | O11—C41      | 1.225 (3) |           |
| C7—C8         | 1.495 (3)    | O12—C40      | 1.377 (3) |           |
| C8—H8A        | 0.9800       | O12—C41      | 1.400 (3) |           |
| C8—H8B        | 0.9800       | N3—C35       | 1.322 (3) |           |
| C8—H8C        | 0.9800       | N3—C29       | 1.422 (3) |           |
| C9—C13        | 1.435 (3)    | N3—H3A       | 0.9101    |           |
| C9—C10        | 1.448 (3)    | C29—C30      | 1.390 (3) |           |
| C10—C11       | 1.430 (3)    | C29—C34      | 1.398 (3) |           |
| C11—C12       | 1.337 (3)    | C30—C31      | 1.382 (4) |           |
| C11—H11       | 0.9500       | C30—H30      | 0.9500    |           |
| C12—C14       | 1.487 (4)    | C31—C32      | 1.392 (4) |           |
| C14—H14A      | 0.9800       | C31—H31      | 0.9500    |           |
| C14—H14B      | 0.9800       | C32—C33      | 1.382 (4) |           |
| C14—H14C      | 0.9800       | C32—H32      | 0.9500    |           |
| O5—C20        | 1.363 (3)    | C33—C34      | 1.389 (3) |           |
| O5—H5B        | 0.8700       | C33—H33      | 0.9500    |           |
| O6—C24        | 1.253 (3)    | C35—C36      | 1.433 (3) |           |
| O7—C27        | 1.225 (3)    | C35—C37      | 1.500 (3) |           |
| O8—C26        | 1.381 (4)    | C36—H36A     | 0.9800    |           |
| O8—C27        | 1.391 (3)    | C36—H36B     | 0.9800    |           |
| N2—C21        | 1.320 (3)    | C36—H36C     | 0.9800    |           |
| N2—C15        | 1.424 (3)    | C37—C41      | 1.434 (3) |           |
| N2—H2A        | 0.9101       | C37—C38      | 1.448 (3) |           |
| C15—C16       | 1.390 (3)    | C38—C39      | 1.422 (3) |           |
| C15—C20       | 1.396 (3)    | C39—C40      | 1.338 (4) |           |
| C16—C17       | 1.384 (4)    | C39—H39      | 0.9500    |           |
| C16—H16       | 0.9500       | C40—C42      | 1.488 (4) |           |
| C17—C18       | 1.390 (4)    | C42—H42A     | 0.9800    |           |
| C17—H17       | 0.9500       | C42—H42B     | 0.9800    |           |
| C18—C19       | 1.391 (4)    | C42—H42C     | 0.9800    |           |
| C18—H18       | 0.9500       |               |           |           |
| C6—O1—H1B     | 110.7        | C21—C22—H22B | 109.5     |           |
| C12—O4—C13    | 121.9 (2)    | H22A—C22—H22B | 109.5  |           |
| C7—N1—C1      | 126.7 (2)    | C21—C22—H22C | 109.5     |           |
| C7—N1—H1A     | 111.6        | H22A—C22—H22C | 109.5    |           |
| C1—N1—H1A     | 121.7        | H22B—C22—H22C | 109.5     |           |
| C2—C1—C6      | 120.4 (2)    | C27—C23—C21  | 119.8 (2) |           |
| C2—C1—N1      | 121.4 (2)    | C27—C23—C24  | 119.5 (2) |           |
| C6—C1—N1      | 118.1 (2)    | C21—C23—C24  | 120.6 (2) |           |
| C3—C2—C1      | 120.1 (2)    | O6—C24—C25   | 118.2 (2) |           |
| C3—C2—H2      | 120.0        | O6—C24—C23   | 123.9 (2) |           |

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| Bond          | Angle (°) | Bond          | Angle (°) |
|--------------|-----------|--------------|-----------|
| C1—C2—H2    | 120.0     | C25—C24—C23  | 117.9 (2) |
| C2—C3—C4    | 119.7 (2) | C26—C25—C24  | 120.7 (3) |
| C2—C3—H3    | 120.2     | C26—C25—H25  | 119.6     |
| C4—C3—H3    | 120.2     | C24—C25—H25  | 119.6     |
| C5—C4—C3    | 120.8 (2) | C25—C26—O8   | 121.8 (2) |
| C5—C4—H4    | 119.6     | C25—C26—C28  | 127.8 (3) |
| C3—C4—H4    | 119.6     | O8—C26—C28   | 110.5 (3) |
| C4—C5—C6    | 119.8 (2) | O7—C27—O8    | 113.2 (2) |
| C4—C5—H5    | 120.1     | O7—C27—C23   | 128.6 (2) |
| C6—C5—H5    | 120.1     | O8—C27—C23   | 118.2 (2) |
| O1—C6—C5    | 123.7 (2) | C26—C28—H28A | 109.5     |
| O1—C6—C1    | 117.0 (2) | C26—C28—H28B | 109.5     |
| C5—C6—C1    | 119.3 (2) | H28A—C28—H28B | 109.5     |
| N1—C7—C9    | 117.5 (2) | C26—C28—H28C | 109.5     |
| N1—C7—C8    | 119.1 (2) | H28A—C28—H28C | 109.5     |
| C9—C7—C8    | 123.4 (2) | H28B—C28—H28C | 109.5     |
| C7—C8—H8A   | 109.5     | C34—O9—H9B   | 107.2     |
| C7—C8—H8B   | 109.5     | C40—O12—C41  | 121.86 (19)|
| H8A—C8—H8B  | 109.5     | C5—N3—C29    | 127.1 (2) |
| C7—C8—H8C   | 109.5     | C35—N3—H3A   | 111.5     |
| H8A—C8—H8C  | 109.5     | C29—N3—H3A   | 121.4     |
| H8B—C8—H8C  | 109.5     | C31—C30—C34  | 120.2 (2) |
| C7—C9—C13   | 120.0 (2) | C30—C29—N3   | 121.2 (2) |
| C7—C9—C10   | 120.6 (2) | C34—C29—N3   | 118.5 (2) |
| C13—C9—C10  | 119.3 (2) | C31—C30—C29  | 120.3 (2) |
| O2—C10—C11  | 118.8 (2) | C31—C30—H30  | 119.9     |
| O2—C10—C9   | 123.7 (2) | C29—C30—H30  | 119.9     |
| C11—C10—C9  | 117.5 (2) | C30—C31—C32  | 119.4 (2) |
| C12—C11—C10 | 121.5 (2) | C30—C31—H31  | 120.3     |
| C12—C11—H11 | 119.2     | C32—C31—H31  | 120.3     |
| C10—C11—H11 | 119.2     | C33—C32—C31  | 120.8 (2) |
| C11—C12—O4  | 121.4 (2) | C33—C32—H32  | 119.6     |
| C11—C12—C14 | 126.1 (3) | C31—C32—H32  | 119.6     |
| O4—C12—C14  | 112.5 (2) | C32—C33—C34  | 120.1 (2) |
| O3—C13—O4   | 114.2 (2) | C32—C33—H33  | 120.0     |
| O3—C13—C9   | 127.4 (2) | C34—C33—H33  | 120.0     |
| O4—C13—C9   | 118.3 (2) | O9—C34—C33   | 123.0 (2) |
| C12—C14—H14A| 109.5     | O9—C34—C29   | 117.8 (2) |
| C12—C14—H14B| 109.5     | C33—C34—C29  | 119.2 (2) |
| H14A—C14—H14B| 109.5   | N3—C35—C37   | 117.7 (2) |
| C12—C14—H14C| 109.5     | N3—C35—C36   | 119.1 (2) |
| H14A—C14—H14C| 109.5     | C37—C35—C36  | 123.2 (2) |
| H14B—C14—H14C| 109.5    | C35—C36—H36A | 109.5     |
| C20—O5—H5B  | 111.0     | C35—C36—H36B | 109.5     |
| C26—O8—C27  | 121.8 (2) | H36A—C36—H36B | 109.5     |
| C21—N2—C15  | 128.2 (2) | C35—C36—H36C | 109.5     |
| C21—N2—H2A  | 110.0     | H36A—C36—H36C | 109.5     |
| C15—N2—H2A  | 121.7     | H36B—C36—H36C | 109.5     |
| Bond Lengths (Å) and Angles (°) |
|--------------------------------|
| C16—C15—C20                        120.8 (2) | C35—C37—C41                        120.0 (2) |
| C16—C15—N2                          121.3 (2) | C35—C37—C38                        120.2 (2) |
| C20—C15—N2                          117.7 (2) | C41—C37—C38                        119.7 (2) |
| C17—C16—C15                         120.0 (2) | O10—C38—C39                        118.8 (2) |
| C17—C16—H16                         120.0              | O10—C38—C37                        123.8 (2) |
| C15—C16—H16                         120.0              | C39—C38—C37                        117.4 (2) |
| C16—C17—C18                         119.1 (2) | C41—C37—C38                        121.9 (2) |
| C16—C17—H17                         120.5              | C40—C39—C38                        119.1 |
| C18—C17—H17                         120.5              | C38—C39—H39                        119.1 |
| C17—C18—C19                         121.3 (2) | C39—C40—O12                        121.3 (2) |
| C17—C18—H18                         119.3              | C39—C40—C42                        127.3 (3) |
| C19—C18—H18                         119.3              | O12—C40—C42                        111.4 (2) |
| C20—C19—C18                         119.5 (2) | O11—C41—C37                        128.0 (2) |
| C20—C19—H19                         120.3              | O12—C41—O12                        117.8 (2) |
| O5—C20—C19                           123.6 (2) | C40—C42—H42A                       109.5 |
| O5—C20—C15                           117.2 (2) | C40—C42—H42B                       109.5 |
| C19—C20—C15                          119.2 (2) | H42A—C42—H42B                      109.5 |
| N2—C21—C23                           117.4 (2) | C40—C42—H42C                       109.5 |
| N2—C21—C22                           119.3 (2) | H42A—C42—H42C                      109.5 |
| C23—C21—C22                          123.2 (2) | H42B—C42—H42C                      109.5 |
| C21—C22—H22A                         109.5 |
| C7—N1—C1—C2                          −52.8 (3) | C22—C21—C23—C24                    177.1 (2) |
| C7—N1—C1—C6                          131.6 (2) | C27—C23—C24—O6                     177.6 (2) |
| C6—C1—C2—C3                          −3.1 (4) | C21—C23—C24—O6                     0.2 (4) |
| N1—C1—C2—C3                          −178.6 (2) | C27—C23—C24—C25                    −0.3 (3) |
| C1—C2—C3—C4                          1.1 (4)  | C21—C23—C24—C25                    −177.6 (2) |
| C2—C3—C4—C5                          1.3 (4)  | O6—C24—C25—C26                     −176.5 (2) |
| C3—C4—C5—C6                          −1.6 (4) | C23—C24—C25—C26                    1.5 (4) |
| C4—C5—C6—O1                          179.0 (2) | C24—C25—C26—O8                     −2.0 (4) |
| C4—C5—C6—C1                          −0.3 (4) | C24—C25—C26—C28                    178.8 (3) |
| C2—C1—C6—O1                          −176.7 (2) | C27—O8—C26—C25                     1.4 (4) |
| N1—C1—C6—O1                          −1.1 (3)  | C27—O8—C26—C28                     −179.3 (2) |
| C2—C1—C6—C5                          2.7 (3)   | C26—O8—C27—O7                      −180.0 (2) |
| N1—C1—C6—C5                          178.3 (2) | C26—O8—C27—C23                     −0.2 (3) |
| C1—N1—C7—C9                          171.8 (2) | C21—C23—C27—O7                     −3.3 (4) |
| C1—N1—C7—C8                          −10.5 (3) | C24—C23—C27—O7                     179.4 (2) |
| N1—C7—C9—C13                         −177.6 (2) | C21—C23—C27—O8                     177.0 (2) |
| C8—C7—C9—C13                         4.8 (3)   | C24—C23—C27—O8                     −0.3 (3) |
| N1—C7—C9—C10                         3.0 (3)   | C35—N3—C29—C30                     53.3 (3) |
| C8—C7—C9—C10                         −174.5 (2) | C35—N3—C29—C34                     −130.3 (2) |
| C7—C9—C10—O2                         1.9 (4)   | C34—C29—C30—C31                    3.0 (4) |
| C13—C9—C10—O2                        −177.5 (2) | N3—C29—C30—C31                     179.4 (2) |
| C7—C9—C10—C11                        −178.8 (2) | C29—C30—C31—C32                    −0.9 (4) |
| C13—C9—C10—C11                       1.9 (3)   | C30—C31—C32—C33                    −1.4 (4) |
| O2—C10—C11—C12                       177.4 (2) | C31—C32—C33—C34                    1.5 (4) |
| C9—C10—C11—C12                       −2.0 (4)  | C32—C33—C34—O9                     −178.8 (2) |
C10—C11—C12—O4  1.9 (4)    C32—C33—C34—C29  0.7 (4)
C10—C11—C12—C14 −177.7 (3) C30—C29—C34—O9  176.6 (2)
C13—O4—C12—C11 −1.5 (4)    N3—C29—C34—O9  0.2 (3)
C13—O4—C12—C14  178.1 (2) C30—C29—C34—C33 −2.9 (3)
C12—O4—C13—C9  1.3 (3)     C29—N3—C35—C37 −175.2 (2)
C12—O4—C13—O3 −179.1 (2) C30—C29—C34—O9  6.7 (3)
C7—C9—C13—O3 −0.4 (4)      C30—C29—C34—C33 −177.8 (2)
C10—C9—C13—O3  178.9 (2) C36—C35—C37—C41 0.3 (3)
C7—C9—C13—O4  179.1 (2) C30—C29—C34—O9  0.3 (3)
C10—C9—C13—O4 −1.6 (3)      N3—C35—C37—C41 −179.3 (2)
C21—N2—C15—C16  54.1 (3)   C36—C35—C37—C38 −2.9 (3)
C21—N2—C15—C20 −129.8 (2) C35—C37—C38—O10  0.6 (3)
C20—C15—C16—C17  2.8 (3)   C30—C29—C34—O9  177.4 (2)
N2—C15—C16—C17  178.7 (2) C29—N3—C35—C37 −179.2 (2)
C15—C16—C17—C18 0.1 (3)    C36—C35—C37—C38 −1.3 (3)
C16—C17—C18—C19 −2.4 (4)   O10—C38—C39—C40 −176.6 (2)
C17—C18—C19—C20 1.9 (4)    C37—C38—C39—C40 −2.3 (4)
C18—C19—C20—C15 −178.1 (2) C35—C37—C38—O10 −179.6 (2)
C16—C15—C20—C19 175.9 (2) C30—C29—C34—O9  177.1 (2)
C15—C16—C17—C18 0.1 (3)    C30—C29—C34—C33 −178.2 (2)
N2—C15—C16—C17 −0.2 (3)    C35—C37—C38—O10 −1.3 (3)
C16—C17—C18—C19 −178.7 (2) C40—O12—C41—O11 −179.2 (2)
C17—C18—C19—C20 −3.2 (3)   C30—C29—C34—O9  179.9 (2)
C18—C19—C20—O5  179.9 (2) C30—C29—C34—O9  179.9 (2)
C18—C19—C20—C15 179.3 (2) C40—O12—C41—O11 −179.9 (2)
C16—C15—C20—O5  0.3 (3)    C35—C37—C38—O10 0.4 (3)
C15—C16—C17—C18 −2.3 (4)   O10—C38—C39—C40 −177.1 (2)
N2—C15—C16—C17 −179.3 (2) C30—C29—C34—O9  177.30 (19)
C15—C16—C17—C18 −177.3 (2) C30—C29—C34—O9  177.30 (19)
C16—C15—C20—C19 5.3 (4)    C35—C37—C38—O10 0.4 (3)
C15—C16—C17—C18 −3.1 (4)   C30—C29—C34—O9  177.30 (19)
N2—C15—C20—C19 −0.2 (3)    C30—C29—C34—C33 −178.2 (2)
C15—N2—C21—C23  5.3 (4)    C35—C37—C38—O10 0.4 (3)
C15—N2—C21—C22 −177.5 (2) C40—O12—C41—O11 −179.9 (2)
N2—C21—C23—C27 −0.2 (3)    C35—C37—C38—O10 0.4 (3)
C22—C21—C23—C27 −0.2 (3)   C35—C37—C38—O10 0.4 (3)
N2—C21—C23—C24 −0.2 (3)

Hydrogen-bond geometry (Å, °)

| D—H···A  | D—H  | H···A | D···A  | D—H···A |
|---------|-------|-------|-------|---------|
| O1—H1B···O7i | 0.87  | 1.79  | 2.662 (2) | 177    |
| N1—H1A···O2 | 0.91  | 1.72  | 2.538 (3) | 148    |
| C8—H8C···O8ii | 0.98  | 2.48  | 3.441 (3) | 167    |
| C11—H11···O6 | 0.95  | 2.57  | 3.253 (3) | 129    |
| O5—H5B···O11iii | 0.87  | 1.83  | 2.689 (2) | 170    |
| N2—H2A···O6 | 0.91  | 1.71  | 2.539 (3) | 151    |
| O9—H9B···O3iv | 0.87  | 1.82  | 2.691 (2) | 179    |
| N3—H3A···O10 | 0.91  | 1.71  | 2.532 (3) | 148    |
| C33—H33···O3v | 0.95  | 2.53  | 3.225 (3) | 130    |
| C36—H36B···O12vi | 0.98 | 2.56  | 3.531 (3) | 173    |

Symmetry codes: (i) −x+1/2, y−1/2, −z+1/2; (ii) x+1, y, z; (iii) −x+1, −y+1, −z+1; (iv) x−1, y, z; (v) −x+1, −y, −z+1.