Crystal structure and Hirshfeld surface analysis of dimethyl 3,3’-[(1E,2E)-ethane-1,2-diylidene]bis-(azanylylidene)bis(4-methylbenzoate)

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The title Schiff base compound, C20H20N2O4, synthesized by the condensation reaction of methyl 3-amino-4-methylbenzoate and glyoxal in ethanol, crystallizes in the monoclinic space group P21/n. The molecule is Z-shaped with the C—N—C—C torsion angle being 47.58 (18)°. In the crystal, pairs of molecules are linked via C—H···N hydrogen bonds, forming centrosymmetric dimers with an R22(8) ring motif; this connectivity leads to the formation of columns running along the a-axis direction. Hirshfeld surface analysis and two-dimensional fingerprint plots were used to explore the intermolecular interactions and revealed that the most significant contributions to the crystal packing are from H···C (94.9%), H···O/O···H (19.0%) and H···C/C···H (17.5%) contacts. Energy frameworks were constructed through different intermolecular interaction energies to investigate the stability of the compound. The net interaction energies for the title compound were found to be electrostatic (Eele = −48.4 kJ mol−1), polarization (Epolar = −9.7 kJ mol−1), dispersion (Edisp = −186.9 kJ mol−1) and repulsion (Erep = 94.9 kJ mol−1) with a total interaction energy, Etot, of −162.4 kJ mol−1.

1. Chemical context

In this study, the title Schiff base compound was synthesized by the condensation reaction of methyl 3-amino-4-methylbenzoate and glyoxal in ethanol. Schiff bases are studied widely because of their synthetic flexibility, selectivity and sensitivity towards the central metal atom, structural similarities with natural biological compounds and because of the presence of an azomethine group (–N==CH–), which is important for elucidating the mechanism of the transformation and racemization reaction biologically (Sharghi et al., 2003). Schiff bases having chelation with oxygen and nitrogen donors and their complexes have been used as drugs and are reported to possess a wide variety of biological activities against bacteria, fungi and certain types of tumors; in addition, they have many biochemical, clinical and pharmacological properties (Przybylski et al., 2009; Barbosa et al., 2020). In recent years, these molecules, which belong to a large family of click reactions, have attracted a lot of interest for their role in the development of self-healing hydrogels (Xu et al., 2019). Over the past few years, some metal complexes of Schiff bases have attracted great interest in many fields. The binding interactions of metal complexes with DNA have been studied (Shahabadi et al., 2010). Schiff bases have different applica-
tions in many research areas including organic, inorganic, biological and materials chemistry (Fan et al., 2020) and as dyes for the textile and related industries. These compounds also have unique characteristics that make them promising candidates for photovoltaic and photonic materials applications (Abdel-Shakour et al., 2019; Imer et al., 2018). We report herein XRD data and Hirshfeld surface analysis of a new Schiff base compound, dimethyl 3,3'-[(1E,2E)-ethane-1,2-diylidene]bis(azanylylidene)bis(4-methylbenzoate), for which energy frameworks of the crystal packing were calculated.

2. Structural commentary

The molecular structure of the title complex is illustrated in Fig. 1. The molecule is located in a special position related to the inversion centre $8i$ ($mm2$) at the middle of the $C10$—$C10^i$ bond [symmetry code: (i) $x+1$, $y$, $z$; (ii) $x-1$, $y+1$, $z$; (iii) $-x+2$, $-y+1$, $-z$; (iv) $x$, $y+1$, $z$. The molecule is Z-shaped with the $C10$—$N1$—$C7$—$C8$ torsion angle being 47.58 (18)$^\circ$. The benzene rings are located in planes parallel to each other. The values of the $C1$—$O2$, $O2$—$C2$ and $C2$—$O1$ bond lengths and the $O1$—$C2$—$O2$, $C2$—$O2$—$C1$ bond angles are close to those reported for similar complexes (see Database survey). Some selected geometric parameters of the molecule are given in Table 1. The azomethine $C\equiv N$ bond length is 1.2713 (17) Å, which is quite close to the corresponding values reported by Gumus et al. (2021) and Kansiz et al. (2021) [1.276 (6) and 1.287 (6) Å and 1.287 (5) Å, respectively].

3. Supramolecular features

Although no classical hydrogen bonds are found in the crystal structure, weak hydrogen bonds are present (Table 2, Fig. 2). The role of hydrogen bonds in the formation of the crystal lattice is shown in Fig. 2a. Pairs of molecules form inversion dimers with an $R_2^2(8)$ ring motif via $C10$—$H10$···$N1$ hydrogen

![Figure 1](image1.png)

**Figure 1**
The molecular structure of the title compound, showing the atom labelling. Displacement ellipsoids are drawn at the 40% probability level.

![Figure 2](image2.png)

**Figure 2**
A view of the crystal packing of the title compound.
bonds, leading to the formation of columns running along the $a$-axis direction. A weak C9—H9A···C1 contact is also present (Table 2), which reinforces the crystal structure and plays a major role in the supramolecular framework stabilization, see Fig. 2b.

4. Database survey

A search of the Cambridge Structural Database (CSD, version 5.40, update of August 2020; Groom et al., 2016) found a structure that is very similar to the title compound, viz. 2-(4-carbomethoxy-2-nitrobenzyl)-1,3,5-trimethylbenzene (CBYMBZ; van der Heijden et al., 1975). In CBYMBZ, the bond lengths and bond angles for the methyl formate are: C8—O4 = 1.448 (4) Å, O4—C7 = 1.326 (3) Å, C7—O3 = 1.193 (3) Å, C8—O4—C7 = 116.2 (3)° and O4—C7—O3 = 123.9 (2)°.

5. Hirshfeld surface analysis

The intermolecular interactions present in the crystal structure were visualized by drawing contact and shape descriptors using Crystal Explorer17.5 (Turner et al., 2017). The Hirshfeld surfaces mapped over $d_{norm}$, curvedness, shape-index and electrostatic potential are shown in Fig. 3. The molecular Hirshfeld surfaces were calculated using a standard (high) surface resolution and with the three-dimensional $d_{norm}$ surfaces mapped over a fixed colour scale from $-0.083$ (red) to $1.171$ (blue) a.u. Red spots in Fig. 3e correspond to the near-type H···O contacts resulting from C—H···O and N—H···O hydrogen bonds. The shape-index surface (Fig. 3b) shows red concave regions with ‘bow-tie’ patterns, indicating the presence of aromatic stacking interactions (C—H···π). In Fig. 3c, the curvedness plots show flat surface patches characteristic of planar stacking. The molecular properties can be described by mapping the molecular electrostatic potential ($-0.067$ to $0.025$ a.u.), which plays a key role in identifying reactive positions on the molecular surface. The Fig. 3d map is useful for predicting the position of nucleophile and electrophile attacks. The blue and red regions observed on the surface around the different atoms correspond to positive and negative electrostatic potentials, respectively. It shows clearly that the electron-rich sites are mainly localized around the oxygen atoms.

Intermolecular contacts and the location of electron-rich regions provide an indication of the stacking in the crystal. To understand this stacking, the crystal voids [calculated with Crystal Explorer17.5 (Turner et al., 2017)] were visualized (Fig. 4). The void parameters of the title compound give a void volume of 76.77 Å³, an area of 340.15 Å², a globularity of 0.257 and asphericity value of 0.807. Fig. 5a shows the two-dimensional fingerprint plot of the sum of all the contacts contributing to the Hirshfeld surface represented in normal mode. The H···H contacts make the largest contribution to the overall crystal packing at 49.4%. This contribution arises as widely scattered points of high density due to the large hydrogen content of the molecule with the two tips at $d_e + d_i = 2.43$ Å (Fig. 5b). Scattered points of the H···O/O···H interactions contribution (19.0%) have a tip at $d_e + d_i = 2.68$ Å (Fig. 5c). The pair of characteristic wings in Fig. 5d arise from H···C/C···H contacts (17.5%) and pairs of spikes are observed with the tips at $d_e + d_i = 2.75$ Å and 2.80 Å. The H···N/N···H contacts, contributing 6.3% to the Hirshfeld

Figure 3
The Hirshfeld surface of the title compound mapped over (a) $d_{norm}$, (b) shape-index, (c) curvedness and (d) electrostatic potential.

Figure 4
A view of the crystal voids.
surface, are also represented by a pair of sharp spikes at $d_e + d_i = 2.76$ Å, Fig. 5e. As seen in Fig. 5f, the C⋯C contacts (4.9%) have an arrow-shaped distribution of points with its tip at $d_e = d_i = 3.59$ Å. The contribution of the C⋯O⋯O⋯C contacts to the Hirshfeld surface (2.9%) is negligible, Fig. 5g.

6. Interaction energies

Interaction energies for the title compound were calculated using the CE-B3LYP/6-31G(d,p) quantum level of theory, as available in CrystalExplorer (Turner et al., 2017). The total intermolecular interaction energy ($E_{\text{tot}}$) is the sum of four energy terms: electrostatic ($E_{\text{ele}}$), polarization ($E_{\text{pol}}$), dispersion ($E_{\text{disp}}$) and exchange-repulsion ($E_{\text{rep}}$) with scale factors of 1.057, 0.740, 0.871 and 0.618, respectively. The relative strengths of the interaction energies in individual directions are represented by cylinder-shaped energy frameworks. The energy-framework calculations were analysed to understand the topologies of the pair-wise intermolecular interaction energies. The energy framework is constructed to compare the different energy components, i.e. repulsion ($E_{\text{rep}}$), electrostatic ($E_{\text{ele}}$), dispersion ($E_{\text{disp}}$), polarization ($E_{\text{pol}}$) and total ($E_{\text{tot}}$) energy (Mackenzie et al., 2017). The energies between molecular pairs are indicated as cylinders joining the centroids of pairs of molecules with the thickness of the cylinder radius being directly proportional to the amount of interaction.

Figure 5
The two-dimensional fingerprint plots for (a) all interactions and those delineated into (b) H⋯H, (c) H⋯O⋯O⋯H, (d) H⋯C/C⋯H, (e) H⋯N/N⋯H, (f) C⋯C and (g) C⋯O/O⋯C contacts.
energy between the pair of molecules (Wu et al., 2020). As seen in Fig. 6, the red molecule with symmetry \((x, y, z)\) located at a distance of 4.60 Å from the centroid of the selected molecule has shown the highest total interaction energy of \(-63.7\ \text{kJ mol}^{-1}\), whereas the purple molecule at the symmetry position \((-x + 1/2, y + 1/2, -z + 1/2)\) located at a distance of 15.88 Å from the centroid of the selected molecule has the lowest total interaction energy of \(-13.4\ \text{kJ mol}^{-1}\). The net interaction energies for the title compound are electrostatic \((E_{\text{ele}}) = -48.4\ \text{kJ mol}^{-1}\), polarization \((E_{\text{pol}}) = -9.7\ \text{kJ mol}^{-1}\), dispersion \((E_{\text{disp}}) = -186.9\ \text{kJ mol}^{-1}\), repulsion \((E_{\text{rep}}) = 94.9\ \text{kJ mol}^{-1}\) and total interaction energy \((E_{\text{tot}}) = -162.4\ \text{kJ mol}^{-1}\). The dispersion energy is dominant.

### 7. Synthesis and Crystallization

27.3 mg (0.165 mmol) of 2-amino-3-methylphenol were dissolved in 20 ml of ethanol. To this was added 11.98 mg (0.083 mmol) of glyoxal (40 wt % in H₂O) dissolved in 20 ml of ethanol and the mixture was refluxed for 12 h. At the end of the reaction, the solution was allowed to cool. The orange product obtained was washed with hexane and crystallized from isopropyl alcohol at room temperature (m.p. = 427–430 K, yield 84%).

![Chemical structure](image)

8. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. H atoms were positioned geometrically and refined using a riding model: C–H = 0.93–0.97 Å with \(U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})\).

### Acknowledgements

Author contributions are as follows: Conceptualization, EBÇ, ES and ND; synthesis, EA and SY; writing EBÇ and SY; formal analysis, EBÇ and ND; validation, ND; project administration, ND, EA and ES.

### Table 3

| Crystal data | Chemical formula | Chemical formula | Chemical formula |
|--------------|------------------|------------------|------------------|
| C₂₀H₂₀N₂O₄   | M₉₈ | Monoclinic, \(P_2_1/n\) | Monoclinic, \(P_2_1/n\) |
| Temperature (K) | 296 | 4.6003 (5), 6.2969 (5), 30.726 (4) | 90.886 (9) |
| \(a, b, c\) (Å) | 9.7 kJ mol⁻¹ | 889.94 (16) | 0.09 |
| \(\beta\) (°) | 2 | | |
| \(\mu\) (mm⁻¹) | Mo Kα | | |
| Crystal size (mm) | 0.38 \(\times\) 0.25 \(\times\) 0.12 | | |

| Data collection | Diffractionometer | Stoe IPDS 2 |
|-----------------|------------------|-------------|
| Absorption correction | Integration (X-RED32; Stoe & Cie, 2002) | |
| \(R_{\text{int}}\) | 0.036 | 0.041, 0.126, 1.06 |
| \((\sin \theta/\lambda)_{\text{max}}\) (Å⁻¹) | 0.647 | 0.129 |
| Reﬁnement | \(R[F^2 > 2\sigma(F^2)]\), \(wR(F^2)\), \(S\) | |
| No. of reﬂections | 6876, 2002, 1490 | 2002 |
| No. of parameters | 129 | 120 |
| H-atom treatment | H-atom parameters constrained \(\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}\) (e Å⁻³) | 0.12, –0.12 |

Computer programs: X-AREA and X-RED32 (Stoe & Cie, 2002), SHELXTL2018/3 (Sheldrick, 2015a), SHELXL2018/3 (Sheldrick, 2015b), OLEX2 (Dolomanov et al., 2009), Mercury (Macrae et al., 2020), WinGX (Farrugia, 2012), PLATON (Spek, 2020) and pubCIF (Westrip, 2010).

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Crystal structure and Hirshfeld surface analysis of dimethyl 3,3′-[[(1E,2E)-ethane-1,2-diylidene]bis(azanylylidene)]bis(4-methylbenzoate)

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Computing details
Data collection: X-AREA (Stoe & Cie, 2002); cell refinement: X-AREA (Stoe & Cie, 2002); data reduction: X-RED32 (Stoe & Cie, 2002); program(s) used to solve structure: SHELXT2018/3 (Sheldrick, 2015a); program(s) used to refine structure: SHELXL2018/3 (Sheldrick, 2015b); molecular graphics: OLEX2 (Dolomanov et al., 2009) and Mercury (Macrae et al., 2020); software used to prepare material for publication: WinGX (Farrugia, 2012), SHELXL2018/3 (Sheldrick, 2015b), PLATON (Spek, 2020) and publCIF (Westrip, 2010).

3,3′-[[(1E,2E)-Ethane-1,2-diylidene]bis(azanylylidene)]bis(4-methylbenzoate)

Crystal data
C_{20}H_{20}N_{2}O_{4}  
M_r = 352.38
Monoclinic, P2_{1}/n
a = 4.6003 (5) Å
b = 6.2969 (5) Å
c = 30.726 (4) Å
β = 90.886 (9)°
V = 889.94 (16) Å³
Z = 2

F(000) = 372
D_x = 1.315 Mg m⁻³
Mo Kα radiation, λ = 0.71073 Å
Cell parameters from 7667 reflections
θ = 1.3–27.9°
μ = 0.09 mm⁻¹
T = 296 K
Plate, colorless
0.38 × 0.25 × 0.12 mm

Data collection
Stoe IPDS 2
diffraclometer
Radiation source: sealed X-ray tube, 12 x 0.4 mm long-fine focus
Plane graphite monochromator
Detector resolution: 6.67 pixels mm⁻¹
rotation method scans
Absorption correction: integration
(X-RED32; Stoe & Cie, 2002)

T_{min} = 0.971, T_{max} = 0.990
6876 measured reflections
2002 independent reflections
1490 reflections with I > 2σ(I)
R_{int} = 0.036
θ_{max} = 27.4°, θ_{min} = 1.3°
h = −5→5
k = −8→8
l = −39→39

Refinement
Refinement on F²
Least-squares matrix: full
R[F² > 2σ(F²)] = 0.041
wR(F²) = 0.126
S = 1.06
2002 reflections
120 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
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) + (0.0582P)^2 + 0.0582P
where \( P = (F_o^2 + 2F_c^2)/3 \)
\((\Delta/\sigma)_{\text{max}} < 0.001
\Delta \rho_{\text{max}} = 0.12 \text{ e Å}^{-3}
\Delta \rho_{\text{min}} = -0.12 \text{ e 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.

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

|    | x     | y     | z     | Uiso*/Ueq |
|----|-------|-------|-------|-----------|
| O2 | 0.9388(2) | 0.49454(16) | 0.32206(3) | 0.0649(3) |
| N1 | 0.3480(2) | 0.65258(18) | 0.45753(3) | 0.0535(3) |
| O1 | 0.9663(3) | 0.80450(19) | 0.28689(4) | 0.0830(4) |
| C7 | 0.4336(3) | 0.7641(2) | 0.41938(4) | 0.0491(3) |
| C3 | 0.6914(3) | 0.7890(2) | 0.35160(4) | 0.0515(3) |
| C10 | 0.5415(3) | 0.5584(2) | 0.48034(4) | 0.0517(3) |
| H10 | 0.7352 | 0.5633 | 0.4722 | 0.062* |
| C8 | 0.6168(3) | 0.6742(2) | 0.38878(4) | 0.0506(3) |
| H8 | 0.6894 | 0.5379 | 0.3931 | 0.061* |
| C6 | 0.3168(3) | 0.9680(2) | 0.41297(4) | 0.0514(3) |
| C2 | 0.8798(3) | 0.7010(2) | 0.31694(4) | 0.0562(3) |
| C5 | 0.3996(3) | 1.0803(2) | 0.37606(4) | 0.0578(4) |
| H5 | 0.3290 | 1.2172 | 0.3717 | 0.069* |
| C4 | 0.5835(3) | 0.9933(2) | 0.34590(4) | 0.0585(4) |
| H4 | 0.6356 | 1.0717 | 0.3216 | 0.070* |
| C9 | 0.1143(3) | 1.0662(3) | 0.44519(5) | 0.0653(4) |
| H9A | −0.0500 | 0.9745 | 0.4491 | 0.098* |
| H9B | 0.0488 | 1.0214 | 0.4345 | 0.098* |
| H9C | 0.2146 | 1.0852 | 0.4725 | 0.098* |
| C1 | 1.1124(4) | 0.3998(3) | 0.28799(5) | 0.0732(5) |
| H1A | 1.0235 | 0.4286 | 0.2601 | 0.110* |
| H1B | 1.1237 | 0.2491 | 0.2924 | 0.110* |
| H1C | 1.3046 | 0.4592 | 0.2890 | 0.110* |

Atomic displacement parameters (Å²)

|     | U11 | U22 | U33 | U12 | U13 | U23 |
|-----|-----|-----|-----|-----|-----|-----|
| O2  | 0.0768(7) | 0.0665(7) | 0.0519(5) | 0.0086(5) | 0.0164(5) | 0.0044(5) |
| N1  | 0.0585(6) | 0.0587(7) | 0.0434(6) | −0.0063(5) | 0.0068(5) | 0.0059(5) |
| O1  | 0.1034(9) | 0.0811(8) | 0.0656(7) | 0.0040(6) | 0.0373(6) | 0.0176(6) |
| C7  | 0.0512(7) | 0.0560(7) | 0.0402(6) | −0.0093(5) | 0.0021(5) | 0.0041(5) |
| C3  | 0.0528(7) | 0.0577(8) | 0.0441(7) | −0.0039(6) | 0.0038(5) | 0.0044(6) |
| C10 | 0.0575(7) | 0.0561(7) | 0.0446(6) | −0.0072(6) | 0.0067(5) | 0.0021(6) |
| C8  | 0.0535(7) | 0.0528(7) | 0.0455(6) | −0.0027(6) | 0.0029(5) | 0.0049(5) |
| C6  | 0.0523(7) | 0.0554(7) | 0.0464(6) | −0.0050(6) | 0.0004(5) | −0.0012(6) |
| C2  | 0.0578(8) | 0.0647(8) | 0.0463(7) | −0.0023(6) | 0.0063(6) | 0.0063(6) |
### Geometric parameters (Å, °)

|          |                |                |                |                |                |      |                |                |                |                |                |
|----------|----------------|----------------|----------------|----------------|----------------|------|----------------|----------------|----------------|----------------|----------------|
| O2—C2   | 1.3370 (18)    |                |                |                |                |      |                |                |                |                |                |
| O2—C1   | 1.4544 (17)    |                |                |                |                |      |                |                |                |                |                |
| N1—C10  | 1.2713 (17)    |                |                |                |                |      |                |                |                |                |                |
| N1—C7   | 1.4272 (16)    |                |                |                |                |      |                |                |                |                |                |
| O1—C2   | 1.2027 (16)    |                |                |                |                |      |                |                |                |                |                |
| C7—C8   | 1.3925 (18)    |                |                |                |                |      |                |                |                |                |                |
| C7—C6   | 1.4044 (19)    |                |                |                |                |      |                |                |                |                |                |
| C3—C4   | 1.389 (2)      |                |                |                |                |      |                |                |                |                |                |
| C3—C8   | 1.3991 (17)    |                |                |                |                |      |                |                |                |                |                |
| C3—C2   | 1.4903 (19)    |                |                |                |                |      |                |                |                |                |                |
| C10—C10^i | 1.469 (2)   |                |                |                |                |      |                |                |                |                |                |
| C10—H10 | 0.9300         |                |                |                |                |      |                |                |                |                |                |
| C2—O2—C1| 115.27 (11)    |                |                |                |                |      |                |                |                |                |                |
| C10—N1—C7| 118.87 (11) |                |                |                |                |      |                |                |                |                |                |
| C8—C7—C6| 120.74 (11)    |                |                |                |                |      |                |                |                |                |                |
| C8—C7—N1| 122.13 (12)    |                |                |                |                |      |                |                |                |                |                |
| C6—C7—N1| 117.09 (12)    |                |                |                |                |      |                |                |                |                |                |
| C4—C3—C8| 119.33 (13)    |                |                |                |                |      |                |                |                |                |                |
| C4—C3—C2| 117.70 (12)    |                |                |                |                |      |                |                |                |                |                |
| C8—C3—C2| 122.96 (13)    |                |                |                |                |      |                |                |                |                |                |
| N1—C10—C10^i | 119.86 (16) |                |                |                |                |      |                |                |                |                |                |
| N1—C10—H10| 120.1         |                |                |                |                |      |                |                |                |                |                |
| C10—C10—H10 | 120.1     |                |                |                |                |      |                |                |                |                |                |
| C7—C8—C3| 119.99 (13)    |                |                |                |                |      |                |                |                |                |                |
| C7—C8—H8| 120.0          |                |                |                |                |      |                |                |                |                |                |
| C3—C8—H8| 120.0          |                |                |                |                |      |                |                |                |                |                |
| C5—C6—C7| 117.97 (12)    |                |                |                |                |      |                |                |                |                |                |
| C5—C6—C9| 120.47 (13)    |                |                |                |                |      |                |                |                |                |                |
| C7—C6—C9| 121.54 (12)    |                |                |                |                |      |                |                |                |                |                |
| O1—C2—O2| 123.25 (13)    |                |                |                |                |      |                |                |                |                |                |
| O1—C2—C3| 123.36 (14)    |                |                |                |                |      |                |                |                |                |                |
| C10—N1—C7—C8 | 47.58 (18) |                |                |                |                |      |                |                |                |                |                |
| C10—N1—C7—C6 | −134.55 (13) |                |                |                |                |      |                |                |                |                |                |
| C7—N1—C10—C10^i | −179.71 (14) |                |                |                |                |      |                |                |                |                |                |
| C6—C7—C8—C3 | 1.39 (19)   |                |                |                |                |      |                |                |                |                |                |
| N1—C7—C8—C3 | 179.18 (11) |                |                |                |                |      |                |                |                |                |                |
| C4—C3—C8—C7 | 0.6 (2)     |                |                |                |                |      |                |                |                |                |                |
| C2—C3—C8—C7 | −178.35 (12) |                |                |                |                |      |                |                |                |                |                |
| C8—C7—C6—C5 | −2.60 (19)  |                |                |                |                |      |                |                |                |                |                |
### Supporting Information

| Bond          | Angle (°)   | Bond          | Angle (°)   |
|---------------|-------------|---------------|-------------|
| N1—C7—C6—C5  | 179.50 (12) | C6—C5—C4—C3  | 0.1 (2)     |
| C8—C7—C6—C9  | 178.82 (13) | C8—C3—C4—C5  | -1.4 (2)    |
| N1—C7—C6—C9  | 0.93 (18)   | C2—C3—C4—C5  | 177.63 (13) |

Symmetry code: (i) $-x+1, -y+1, -z+1$.

### Hydrogen-bond geometry (Å, °)

$Cg1$ is the centroid of the C3–C8 ring.

|       | D—H     | H···A     | D···A   | D—H···A |
|-------|---------|-----------|---------|---------|
| C10—H10···N1$^{ii}$ | 0.93    | 2.92      | 3.833 (2) | 169     |
| C5—H5···O2$^{iii}$  | 0.93    | 2.92      | 3.734 (2) | 147     |
| C1—H1A···O1$^{iv}$ | 0.96    | 2.77      | 3.543 (2) | 138     |
| C1—H1B···O1$^{v}$  | 0.96    | 2.90      | 3.808 (2) | 159     |
| C9—H9A···Cg1$^{ii}$ | 0.96    | 2.93      | 3.572 (2) | 125     |

Symmetry codes: (ii) $x+1, y, z$; (iii) $x-1, y+1, z$; (iv) $-x+3/2, y-1/2, -z+1/2$; (v) $x, y-1, z$. 

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