Crystal structure and Hirshfeld surface analysis of 3-[2-(3,5-dimethylphenyl)hydrazinylidene]benzofuran-2(3H)-one

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In the title compound, C_{16}H_{14}N_{2}O_{2}, the 2,3-dihydro-1-benzofuran ring system is essentially planar and makes a dihedral angle of 3.69 (7)° with the dimethylphenyl ring. The molecular conformation is stabilized by an intramolecular N—H···O hydrogen bond with an S(6) ring motif. In the crystal, molecules are connected by C—H···π and π—π stacking interactions, forming a layer lying parallel to the (11̅1) plane. One methyl group is disordered over two orientations, with occupancies of 0.67 (4) and 0.33 (4). Hirshfeld surface analysis indicates that the most important contributions to the crystal packing are from H···H (51.2%), O···H/H···O (17.9%), C···H/H···C (15.2%) and C···C (8.1%) contacts.

1. Chemical context

Hydrazones have many applications in diverse areas, such as in optical data storage, as molecular switches and antimicrobial agents, in non-linear optics, molecular recognition, dye-sensitized solar cells, color-changing materials, catalysis, liquid crystals, etc., mainly because of the azo-to-hydrazo tautomerism/isomerism and the optical properties of –N=N– unit (Maharramov et al., 2018; Ma et al., 2020, 2021; Viswanathan et al., 2019). Not only E/Z isomerization, but also azo-hydrazo tautomerism is important in organic and the coordination chemistry of hydrazone dyes (Ma et al., 2017a,b; Mahmoudi et al., 2017, 2018). The design of hydrazone dyes with electron donor or acceptor substituents has led to multidentate ligands, the corresponding coordination compounds of which have been applied effectively as catalysts in oxidation and C—C coupling reactions (Mahmudov et al., 2013; Mizar et al., 2012). Moreover, the functional properties of hydrazones or their metal complexes can be regulated by attaching functional groups to the =N—NH— unit (Gurbanov et al., 2020a,b; Kopylovich et al., 2011; Mahmudov et al., 2020; Shikhaliyev et al., 2014). Thus, we have attached C=O groups and furan and aryl rings to the =N—NH— moiety, leading to a new hydrazone compound, (Z)-3-[2-(3,5-dimethylphenyl)hydrazinylidene]benzofuran-2(3H)-one, which can form intermolecular interactions.
2. Structural commentary

The molecular conformation of the title compound is stabilized by an intramolecular N—H⋯O hydrogen bond (N2—H2⋯O2; Table 1) with an S(6) ring motif (Fig. 1). The 2,3-dihydro-1-benzofuran ring system (O1/C1–C8) of the title compound is essentially planar [maximum deviations = 0.031 (2) Å for C3 and 0.026 (2) Å for C6] and makes a dihedral angle of 3.69 (7)° with the dimethylphenyl C9–C14 ring. In the molecule, the aromatic C9–C14 ring and the C≡N—NH– unit are almost coplanar with a dihedral angle of 4.8 (8)° between them.

3. Supramolecular features

In the crystal, molecules are connected by C—H⋯π interactions \([C15—H15A⋯Cg3^i]\) and \(C16—H16F⋯Cg2^ii\); symmetry codes as given in Table 1; Fig. 2] and π⋯π stacking interactions \([Cg1⋯Cg2^{iii} = 3.6227\ (11) \text{ Å}, \text{slippage} = 1.226 \text{ Å}; Cg1⋯Cg3^{ii} = 3.7128\ (10) \text{ Å}, \text{slippage} = 1.339 \text{ Å}]; \) Symmetry codes: (i) \(x,-y,-z\); (ii) \(-x+1,-y+1,-z+1\); (iii) \(-x+2,-y+1,-z+2\).

Table 1
Hydrogen-bond geometry (Å, °).

| D—H⋯A   | D—H  | H⋯A  | D⋯A  | D⋯H⋯A |
|---------|-------|------|------|--------|
| N2—H2⋯O2 | 0.93 (2) | 2.12 (2) | 2.840 (2) | 133.8 (16) |
| C15—H15A⋯Cg3^i | 0.96 | 2.90 | 3.591 (3) | 130 |
| C16—H16F⋯Cg2^ii | 0.96 | 2.92 | 3.715 (3) | 141 |

Symmetry codes: (i) \(x,-y,-z\); (ii) \(-x+1,-y+1,-z+1\); (iii) \(-x+2,-y+1,-z+2\).
hydro-1-benzofuran ring system while Cg3 is the centroid of the dimethylphenyl C9–C14 ring (Fig. 3). These interactions link the molecules into a layer structure lying parallel to the (111) plane (Fig. 4).

4. Hirshfeld surface analysis

Crystal Explorer17 (Turner et al., 2017) was used to perform a Hirshfeld surface analysis and generate the associated two-dimensional fingerprint plots, with a standard resolution of the three-dimensional $d_{norm}$ surfaces plotted over a fixed color scale of −0.0001 (red) to 1.5993 (blue) a.u. (Fig. 5a). All of the disordered H atoms of the C16 methyl group were taken into account together. The shape-index of the Hirshfeld surface is a tool to visualize the 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. 5b clearly indicates that there are π–π interactions in the title compound.

Two-dimensional fingerprint plots for the H–H, O–H/H–O, C⋅⋅⋅H/H–C and C⋅⋅⋅C contacts are presented in Fig. 6. H–H interactions, which are located in the middle region of the fingerprint plot, contribute the most to overall crystal packing, with 51.2% (Fig. 6b). The O⋅⋅⋅H/H⋅⋅⋅O contacts contribute 17.9% (Fig. 6c) to the Hirshfeld surface, while the C⋅⋅⋅H/H⋅⋅⋅C contacts contribute 15.2% (Fig. 6d), resulting in a pair of distinctive wings. The C⋅⋅⋅C interactions account for 8.1% of the Hirshfeld surface. The percentage contributions to the Hirshfeld surface including other minor ones are summarized in Table 2.

5. Database survey

A search of the Cambridge Crystallographic Database (CSD version 5.42, updated September 2021; Groom et al., 2016) for the 1-benzofuran-2(3H)-one unit gave 220 hits. Of these, the compound most similar to the title compound is 7-methoxy-3-(2-phenylhydrazinylidene)-1-benzofuran-2(3H)-one, I (CSD refcode IBADIC; Atioglu et al., 2021). Four compounds reported by Oliveira et al. (2019) are closely related to the title compound, viz. 2-(4-nitro-1H-imidazol-1-yl)-N'-[1-(pyridin-2-
monoclinic space group \( P2_1/\alpha \) with \( Z = 8 \) and \( V \) crystallizes in the triclinic space group \( \overline{P}\overline{T} \) with \( Z = 2 \). Compound \( VI \) crystallizes in the monoclinic space group \( P2_1/c \) with \( Z = 4 \). The \( E \) conformation in \( II, III \) and \( V \) is stabilized by a strong intermolecular N–H ⋅⋅⋅N hydrogen bond interaction. These interactions lead to the formation of dimeric structural arrangements. In the crystal of IV, an intermolecular N–H ⋅⋅⋅O hydrogen bond results in a helical chain structure along the \( b \)-axis direction. Non-classical intermolecular C–H ⋅⋅⋅N and C–H ⋅⋅⋅O interactions are also observed in the crystals of \( II, III, IV \) and \( V \).

6. Synthesis and crystallization

\((Z)-3-[2-(3,5-Dimethylphenyl)hydrazinylidene]benzofuran-2(3H)-one\) was synthesized according to the reported method (Shikhaliyev et al., 2018, 2019). A 20 mL screw-neck vial was charged with DMSO (10 mL), (E)-2-[2-(3,5-dimethylphenyl)hydrazinylidene]methylphenol (240 mg, 1 mmol), tetramethylethylenediamine (TMEDA) (295 mg, 2.5 mmol), CuCl (2 mg, 0.02 mmol) and CCl4 (20 mmol, 10 equiv). After 1–3 h (until TLC analysis showed complete consumption of the corresponding Schiff base), the reaction mixture was poured into a 0.01 \( M \) solution of HCl (100 mL, pH 2–3), and extracted with dichloromethane (3 × 20 mL). The combined organic phase was washed with water (3 × 20 mL), brine (30 mL), dried over anhydrous Na2SO4 and concentrated in vacuo using a rotary evaporator. The residue was purified by column chromatography on silica gel using appropriate mixtures of hexane and dichloromethane (3/1–1/1). Then the substance was refluxed in methanol for 30 min, and left for evaporation. After three days, single crystals of the title compound suitable for X-ray analysis were obtained. Colorless solid (65%); m.p. 475 K. Analysis calculated for C16H14N2O2 (\( M = 266.30 \)): C 72.17, H 5.30, N 10.52; found: C 72.13, H 5.26, N 10.48%. 1H NMR (300 MHz, CDCl3) \( \delta \) 7.22 (2H, CH), 7.20 (2H, CH), 6.83 (2H, CH), 6.79–7.69 (7H, Ar), 2.37 (6H, 2CH3). 13C NMR (75 MHz, CDCl3) \( \delta \) 160.47, 157.77, 134.91, 125.14, 124.12, 121.77, 121.56, 119.86, 118.21, 114.55, 108.16, 106.59, 16.85 and 16.52. ESI–MS: \( m/z \): 267.23 [\( M + H \)]+. 7. Refinement details

Crystal data, data collection and structure refinement details are summarized in Table 3. The amine H atom was located in a difference-Fourier map and refined freely [N2–H2 = 0.93 (2) \( \AA \)]. All C-bound H atoms were placed at calculated positions using a riding model, with C–H = 0.93 or 0.96 \( \AA \), and with \( U_{iso}(H) = 1.2 \) or 1.5\( U_{eq}(C) \). The methyl group with the C16 atom attached to the atom C13 is disordered over two orientations, with occupancies of 0.67 (4) and 0.33 (4). Owing to poor agreement, nine reflections (5 14 10, 7 13 0, 10 6 5, T0 12 4, 11 1 0, T1 1 1, S 19 6, S 0 8 and T0 17 4) were omitted during the final refinement cycle.

Figure 6

Fingerprint plots showing (a) all intermolecular interactions and delineated into (b) H ⋅⋅⋅H, (c) O ⋅⋅⋅H ⋅⋅⋅O, (d) C ⋅⋅⋅H ⋅⋅⋅C and (e) C ⋅⋅⋅C contacts.
Table 3
Experimental details.

| Crystal data |  |
|--------------|---|
| Chemical formula | C_16H_{14}N_2O_2 |
| M, | 266.29 |
| Crystal system, space group | Monoclinic, P2_1/c |
| Temperature (K) | 296 |
| No. of measured, independent and observed | 4176 |
| No. of reflections | 2332 |
| R(min), max | 0.09, 0.746 |
| Radiation type | Mo Kα |
| μ (mm⁻¹) | 0.40 × 0.21 × 0.06 |
| Crystal size (mm) | 0.40 × 0.21 × 0.06 |

Data collection

| Diffractometer | Bruker APEXII CCD |
|----------------|------------------|
| T_{min}, T_{max} | 0.684, 0.746 |
| No. of measured, independent and observed [I > 2σ(I)] reflections | 22343, 4176, 2332 |
| R(int) | 0.058 |
| (sinθ/λ)_{max} (Å⁻¹) | 0.714 |
| Refinement | |
| R[F² > 2σ(F²)], wR(F²), S | 0.062, 0.146, 1.04 |
| No. of reflections | 4176 |
| No. of parameters | 188 |
| H-atom treatment | H atoms treated by a mixture of independent and constrained refinement |
| Δρ_{max}, Δρ_{min} (e Å⁻³) | 0.15, −0.14 |

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The author’s contributions are as follows. Conceptualization, NQS, MA and AB; synthesis, UFA and SHM; X-ray analysis, ZA, RKA and MA; writing (review and editing of the manuscript) ZA, MA and AB; funding acquisition, NQS, UFA, SHM and RKA; supervision, NQS, MA and AB.

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Crystal structure and Hirshfeld surface analysis of 3-[2-(3,5-dimethylphenyl)hydrazinylidene]benzofuran-2(3H)-one

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Computing details
Data collection: APEX2 (Bruker, 2003); cell refinement: SAINT (Bruker, 2003); data reduction: SAINT (Bruker, 2003); program(s) used to solve structure: SHELXT (Sheldrick, 2015a); program(s) used to refine structure: SHELXL (Sheldrick, 2015b); molecular graphics: ORTEP-3 for Windows (Farrugia, 2012); software used to prepare material for publication: PLATON (Spek, 2020).

3-[2-(3,5-Dimethylphenyl)hydrazinylidene]benzofuran-2(3H)-one

Crystal data
C16H14N2O2

Mr = 266.29

Monoclinic, P21/c

a = 8.8644 (4) Å
b = 19.9222 (8) Å

c = 8.1736 (3) Å

β = 107.240 (1)°

V = 1378.59 (10) Å3

Z = 4

F(000) = 560

Dx = 1.283 Mg m−3

Mo Kα radiation, λ = 0.71073 Å

Cell parameters from 3738 reflections

θ = 2.4–30.5°

µ = 0.09 mm−1

T = 296 K

Prism, colourless

0.40 × 0.21 × 0.06 mm

Data collection
Bruker APEXII CCD
diffractometer
φ and ω scans

Absorption correction: multi-scan
(SADABS; Bruker, 2003)

Tmin = 0.684, Tmax = 0.746

22343 measured reflections

4176 independent reflections

2332 reflections with I > 2σ(I)

Rint = 0.058

θmax = 30.5°, θmin = 2.4°

h = −12→12

k = −28→28

l = −11→11

Refinement

Refinement on F2
Least-squares matrix: full

R[F2 > 2σ(F2)] = 0.062

wR(F2) = 0.146

S = 1.03

4176 reflections

188 parameters

0 restraints

Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement

w = 1/[σ2(Fo2) + (0.0465P)2 + 0.2174P]

where P = (Fo2 + 2Fc2)/3

(Δρ)max < 0.001

Δρmax = 0.15 e Å−3

Δρmin = −0.14 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)**

| Atom | x     | y     | z     | U(eq) | Occ. (<1) |
|------|-------|-------|-------|-------|-----------|
| C1   | 0.7095 (2) | 0.56971 (8) | 0.9270 (2) | 0.0519 (4) | 0.0519 (4) |
| C2   | 0.76884 (18) | 0.52981 (8) | 0.8095 (2) | 0.0482 (4) | 0.0482 (4) |
| C3   | 0.92693 (18) | 0.55352 (8) | 0.8274 (2) | 0.0493 (4) | 0.0493 (4) |
| C4   | 1.0448 (2) | 0.53565 (9) | 0.7563 (3) | 0.0624 (5) | 0.0624 (5) |
| H4   | 1.028478 | 0.502040 | 0.673914 | 0.075* | 0.075* |
| C5   | 1.1879 (2) | 0.56929 (11) | 0.8114 (3) | 0.0728 (6) | 0.0728 (6) |
| H5   | 1.268324 | 0.558312 | 0.764500 | 0.087* | 0.087* |
| C6   | 1.2129 (2) | 0.61884 (11) | 0.9347 (3) | 0.0749 (6) | 0.0749 (6) |
| H6   | 1.310276 | 0.640405 | 0.969456 | 0.090* | 0.090* |
| C7   | 1.0974 (2) | 0.63715 (10) | 1.0075 (3) | 0.0673 (5) | 0.0673 (5) |
| H7   | 1.114142 | 0.670299 | 1.091089 | 0.081* | 0.081* |
| C8   | 0.95594 (19) | 0.60361 (8) | 0.9495 (2) | 0.0538 (4) | 0.0538 (4) |
| C9   | 0.4768 (2) | 0.40926 (8) | 0.6127 (2) | 0.0530 (4) | 0.0530 (4) |
| C10  | 0.5476 (2) | 0.37321 (9) | 0.5108 (2) | 0.0603 (5) | 0.0603 (5) |
| H10  | 0.648784 | 0.384407 | 0.508401 | 0.072* | 0.072* |
| C11  | 0.4678 (3) | 0.32049 (9) | 0.4125 (2) | 0.0702 (6) | 0.0702 (6) |
| C12  | 0.3181 (3) | 0.30497 (10) | 0.4188 (3) | 0.0779 (6) | 0.0779 (6) |
| H12  | 0.263853 | 0.269826 | 0.351887 | 0.093* | 0.093* |
| C13  | 0.2457 (2) | 0.33984 (10) | 0.5213 (3) | 0.0693 (6) | 0.0693 (6) |
| C14  | 0.3266 (2) | 0.39267 (9) | 0.6189 (2) | 0.0598 (5) | 0.0598 (5) |
| H14  | 0.280284 | 0.416993 | 0.688386 | 0.072* | 0.072* |
| C15  | 0.5458 (3) | 0.28029 (12) | 0.3040 (3) | 0.1039 (9) | 0.1039 (9) |
| H15A | 0.513973 | 0.234174 | 0.302284 | 0.156* | 0.156* |
| H15B | 0.658448 | 0.283384 | 0.351251 | 0.156* | 0.156* |
| H15C | 0.514651 | 0.297710 | 0.189347 | 0.156* | 0.156* |
| C16  | 0.0820 (3) | 0.32121 (12) | 0.5296 (3) | 0.0972 (8) | 0.0972 (8) |
| H16D | 0.052299 | 0.350758 | 0.607547 | 0.146* | 0.146* |
| H16E | 0.082700 | 0.275746 | 0.568674 | 0.146* | 0.146* |
| H16F | 0.007489 | 0.325362 | 0.417644 | 0.146* | 0.146* |
| H16A | 0.013993 | 0.359673 | 0.500891 | 0.146* | 0.146* |
| H16B | 0.088531 | 0.306500 | 0.643291 | 0.146* | 0.146* |
| H16C | 0.039964 | 0.285692 | 0.449682 | 0.146* | 0.146* |
| N1   | 0.69683 (16) | 0.48115 (7) | 0.70999 (18) | 0.0508 (3) | 0.0508 (3) |
| N2   | 0.55439 (17) | 0.46316 (7) | 0.7133 (2) | 0.0545 (4) | 0.0545 (4) |
| H2   | 0.513 (2) | 0.4843 (10) | 0.792 (3) | 0.077 (6)* | 0.077 (6)* |
| O1   | 0.82605 (14) | 0.61480 (6) | 1.00954 (16) | 0.0609 (3) | 0.0609 (3) |
| O2   | 0.58433 (14) | 0.56775 (7) | 0.95705 (17) | 0.0666 (4) | 0.0666 (4) |
### Atomic displacement parameters (Å²)

|     | \(U_{11}\)       | \(U_{22}\)       | \(U_{33}\)       | \(U_{12}\)       | \(U_{13}\)       | \(U_{23}\)       |
|-----|------------------|------------------|------------------|------------------|------------------|------------------|
| C1  | 0.0428 (9)       | 0.0531 (9)       | 0.0561 (10)      | −0.0041 (7)      | 0.0089 (8)       | −0.0012 (8)      |
| C2  | 0.0422 (9)       | 0.0501 (9)       | 0.0482 (9)       | −0.0004 (7)      | 0.0073 (7)       | 0.0016 (7)       |
| C3  | 0.0403 (8)       | 0.0503 (9)       | 0.0546 (10)      | 0.0026 (7)       | 0.0100 (7)       | 0.0083 (7)       |
| C4  | 0.0525 (11)      | 0.0673 (11)      | 0.0680 (12)      | 0.0056 (9)       | 0.0186 (9)       | 0.0090 (9)       |
| C5  | 0.0459 (11)      | 0.0842 (14)      | 0.0898 (16)      | 0.0066 (10)      | 0.0223 (11)      | 0.0224 (12)      |
| C6  | 0.0445 (11)      | 0.0770 (13)      | 0.0958 (16)      | −0.0094 (9)      | 0.0092 (11)      | 0.0179 (12)      |
| C7  | 0.0512 (11)      | 0.0607 (11)      | 0.0808 (14)      | −0.0092 (9)      | 0.0053 (10)      | 0.0015 (10)      |
| C8  | 0.0409 (9)       | 0.0531 (9)       | 0.0630 (11)      | −0.0004 (7)      | 0.0088 (8)       | 0.0055 (8)       |
| C9  | 0.0536 (10)      | 0.0501 (9)       | 0.0461 (9)       | −0.0056 (8)      | 0.0005 (8)       | 0.0030 (7)       |
| C10 | 0.0700 (12)      | 0.0553 (10)      | 0.0511 (10)      | −0.0063 (9)      | 0.0109 (9)       | 0.0003 (8)       |
| C11 | 0.0950 (16)      | 0.0537 (11)      | 0.0542 (11)      | −0.0090 (10)     | 0.0102 (11)      | −0.0011 (9)      |
| C12 | 0.0988 (17)      | 0.0575 (12)      | 0.0572 (12)      | −0.0205 (11)     | −0.0078 (12)     | −0.0005 (10)     |
| C13 | 0.0649 (12)      | 0.0635 (12)      | 0.0621 (12)      | −0.0153 (9)      | −0.0080 (10)     | 0.0156 (10)      |
| C14 | 0.0534 (10)      | 0.0616 (11)      | 0.0553 (10)      | −0.0066 (8)      | 0.0020 (8)       | 0.0065 (8)       |
| C15 | 0.150 (2)        | 0.0746 (15)      | 0.0865 (17)      | −0.0083 (15)     | 0.0348 (17)      | −0.0243 (13)     |
| C16 | 0.0724 (15)      | 0.0949 (17)      | 0.1019 (18)      | −0.0327 (12)     | −0.0085 (13)     | 0.0192 (14)      |
| N1  | 0.0466 (8)       | 0.0525 (8)       | 0.0497 (8)       | −0.0017 (6)      | 0.0086 (6)       | 0.0028 (6)       |
| N2  | 0.0475 (8)       | 0.0574 (9)       | 0.0553 (9)       | −0.0059 (7)      | 0.0100 (7)       | −0.0065 (7)      |
| O1  | 0.0496 (7)       | 0.0610 (7)       | 0.0690 (8)       | −0.0061 (6)      | 0.0127 (6)       | −0.0130 (6)      |
| O2  | 0.0473 (7)       | 0.0790 (9)       | 0.0757 (9)       | −0.0041 (6)      | 0.0213 (6)       | −0.0101 (7)      |

### Geometric parameters (Å, °)

- C1—O2 1.2064 (19)  C10—H10 0.9300
- C1—O1 1.3842 (19)  C11—C12 1.378 (3)
- C1—C2 1.459 (2)    C11—C15 1.506 (3)
- C2—N1 1.304 (2)    C12—C13 1.384 (3)
- C2—C3 1.445 (2)    C12—H12 0.9300
- C3—C8 1.380 (2)    C13—C14 1.385 (2)
- C3—C4 1.385 (2)    C13—C16 1.519 (3)
- C4—C5 1.386 (3)    C14—H14 0.9300
- C4—H4 0.9300       C15—H15A 0.9600
- C5—C6 1.381 (3)    C15—H15B 0.9600
- C5—H5 0.9300       C15—H15C 0.9600
- C6—C7 1.377 (3)    C16—H16D 0.9600
- C6—H6 0.9300       C16—H16E 0.9600
- C7—C8 1.375 (2)    C16—H16F 0.9600
- C7—H7 0.9300       C16—H16A 0.9600
- C8—O1 1.397 (2)    C16—H16B 0.9600
- C9—C10 1.383 (2)   C16—H16C 0.9600
- C9—C14 1.387 (2)   N1—N2 1.3206 (19)
- C9—N2 1.402 (2)    N2—H2 0.93 (2)
- C10—C11 1.383 (2)  N1—O1 1.2134 (16)  C11—C12—C13 122.29 (18)
O2—C1—C2 130.48 (16) C11—C12—H12 118.9
O1—C1—C2 108.18 (14) C13—C12—H12 118.9
N1—C2—C3 125.97 (16) C12—C13—C14 118.4 (2)
N1—C2—C1 127.59 (15) C12—C13—C16 121.7 (2)
C3—C2—C1 106.43 (14) C14—C13—C16 119.9 (2)
C8—C3—C4 119.17 (16) C13—C14—C9 119.93 (19)
C8—C3—C2 106.10 (15) C13—C14—H14 120.0
C4—C3—C2 134.70 (17) C9—C14—H14 120.0
C3—C4—C5 118.10 (19) C11—C15—H15A 109.5
C3—C4—H4 121.0 C11—C15—H15B 109.5
C5—C4—H4 121.0 H15A—C15—H15B 109.5
C6—C5—C4 121.07 (19) C11—C15—H15C 109.5
C6—C5—H5 119.5 H15A—C15—H15C 109.5
C4—C5—H5 119.5 H15B—C15—H15C 109.5
C7—C6—C5 121.71 (19) C13—C16—H16D 109.5
C7—C6—H6 119.1 C13—C16—H16E 109.5
C5—C6—H6 119.1 H16D—C16—H16E 109.5
C8—C7—C6 116.17 (19) C13—C16—H16F 109.5
C8—C7—H7 121.9 H16D—C16—H16F 109.5
C6—C7—H7 121.9 H16E—C16—H16F 109.5
C7—C8—C3 123.77 (18) C13—C16—H16A 109.5
C7—C8—O1 124.32 (17) C13—C16—H16B 109.5
C3—C8—O1 111.89 (14) H16A—C16—H16B 109.5
C10—C9—C14 120.63 (16) C13—C16—H16C 109.5
C10—C9—N2 121.31 (16) H16A—C16—H16C 109.5
C14—C9—N2 118.05 (17) H16B—C16—H16C 109.5
C11—C10—C9 119.94 (19) C2—N1—N2 118.89 (15)
C11—C10—H10 120.0 N1—N2—C9 120.08 (15)
C9—C10—H10 120.0 N1—N2—H2 117.9 (13)
C12—C11—C10 118.8 (2) C9—N2—H2 121.7 (13)
C12—C11—C15 121.18 (19) C1—O1—C8 107.39 (13)
C10—C11—C15 120.0 (2)

O2—C1—C2—N1 −1.0 (3) N2—C9—C10—C11 −179.74 (16)
O1—C1—C2—N1 178.73 (15) C9—C10—C11—C12 −0.1 (3)
O2—C1—C2—C3 −179.52 (18) C9—C10—C11—C15 −178.83 (18)
O1—C1—C2—C3 0.23 (17) C10—C11—C12—C13 −0.7 (3)
N1—C2—C3—C8 −178.02 (16) C15—C11—C12—C13 178.06 (19)
N1—C2—C3—C4 0.52 (17) C11—C12—C13—C14 0.8 (3)
N1—C2—C3—C4 −0.4 (3) C11—C12—C13—C16 −178.75 (19)
C1—C2—C3—C8 −178.17 (19) C12—C13—C14—C9 −0.3 (3)
C1—C2—C3—C4 0.5 (3) C16—C13—C14—C9 179.33 (17)
C8—C3—C4—C5 −0.1 (3) C10—C9—C14—C13 −0.5 (3)
C2—C3—C4—C5 −177.49 (18) C9—C14—C13—C16 179.91 (15)
C3—C4—C5—C6 0.5 (3) N2—C9—C14—C13 176.94 (15)
C4—C5—C6—C7 −0.3 (3) C3—C2—N1—N2 −1.3 (2)
C5—C6—C7—C8 −0.4 (3) C2—N1—N2—C9 −177.48 (14)
C6—C7—C8—C3 0.9 (3) C10—C9—N2—N1 1.1 (2)
C6—C7—C8—O1 179.25 (16) C10—C9—N2—N1 1.1 (2)
C4—C3—C8—C7  
C2—C3—C8—C7  
C4—C3—C8—O1  
C2—C3—C8—O1  
C14—C9—C10—C11

C14—C9—N2—N1  
O2—C1—O1—C8  
C2—C1—O1—C8  
C7—C8—O1—C1  
C3—C8—O1—C1

Hydrogen-bond geometry (Å, °)
Cg2 and Cg3 are the centroids of the C3–C8 and C9–C14 rings, respectively.

| D—H···A       | D—H  | H···A | D···A | D—H···A |
|---------------|------|------|------|---------|
| N2—H2···O2    | 0.93 (2) | 2.12 (2) | 2.840 (2) | 133.8 (16) |
| C15—H15A···Cg3i | 0.96 | 2.90 | 3.591 (3) | 130 |
| C16—H16F···Cg2ii | 0.96 | 2.92 | 3.715 (3) | 141 |

Symmetry codes: (i) x, −y−1/2, z−3/2; (ii) −x+1, −y+1, −z+1.