



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

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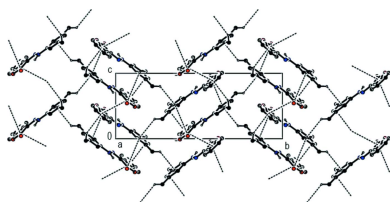
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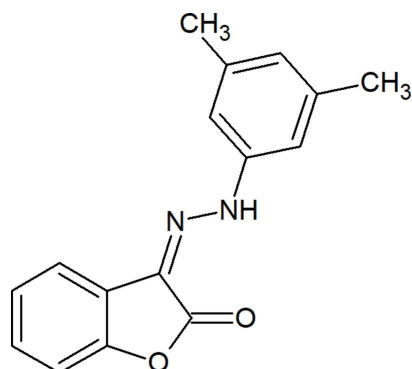
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In the title compound, C₁₆H₁₄N₂O₂, the 2,3-dihydro-1-benzofuran ring system is essentially planar and makes a dihedral angle of 3.69 (7)° with the dimethylphenyl ring. The molecular conformation is stabilized by an intramolecular N—H···O hydrogen bond 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 (11 $\bar{1}$) 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.*, 2017*a,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.*, 2020*a,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(3*H*)-one, which can form intermolecular interactions.





2. Structural commentary

The molecular conformation of the title compound is stabilized by an intramolecular N—H \cdots O hydrogen bond (N2—H2 \cdots 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) $^\circ$ 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) $^\circ$ between them.

3. Supramolecular features

In the crystal, molecules are connected by C—H \cdots π interactions [C15—H15A \cdots Cg3ⁱ and C16—H16F \cdots Cg2ⁱⁱ; symmetry codes as given in Table 1; Fig. 2] and π — π stacking interactions [Cg1 \cdots Cg2ⁱⁱⁱ = 3.6227 (11) Å, slippage = 1.226 Å; Cg1 \cdots Cg3ⁱⁱ = 3.7128 (10) Å, slippage = 1.339 Å; Symmetry

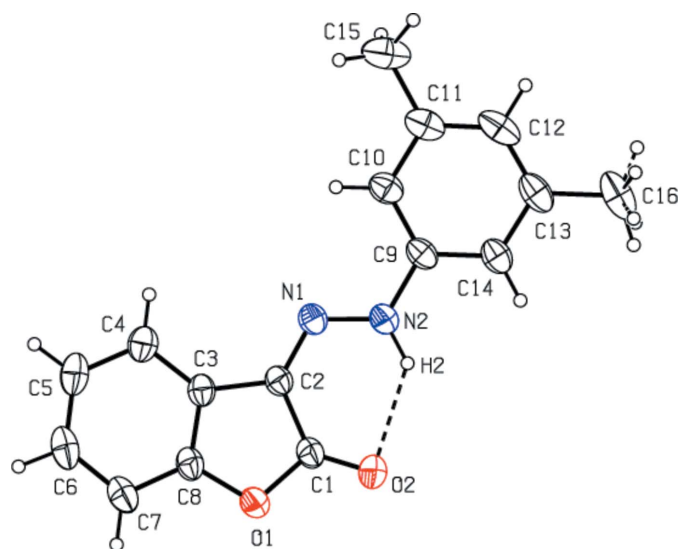


Figure 1

The molecular structure of the title compound with displacement ellipsoids for the non-hydrogen atoms drawn at the 30% probability level.

Table 1

Hydrogen-bond geometry (Å, $^\circ$).

Cg2 and Cg3 are the centroids of the C3—C8 and C9—C14 rings, respectively.

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N2—H2 \cdots O2	0.93 (2)	2.12 (2)	2.840 (2)	133.8 (16)
C15—H15A \cdots Cg3 ⁱ	0.96	2.90	3.591 (3)	130
C16—H16F \cdots Cg2 ⁱⁱ	0.96	2.92	3.715 (3)	141

Symmetry codes: (i) $x, -y - \frac{1}{2}, z - \frac{3}{2}$; (ii) $-x + 1, -y + 1, -z + 1$.

codes: (ii) $-x + 1, -y + 1, -z + 1$; (iii) $-x + 2, -y + 1, -z + 2$], where Cg1 and Cg2 are the centroids of the oxolane O1/C1—C3/C8 and benzene C3—C8 rings, respectively, of the 2,3-di-

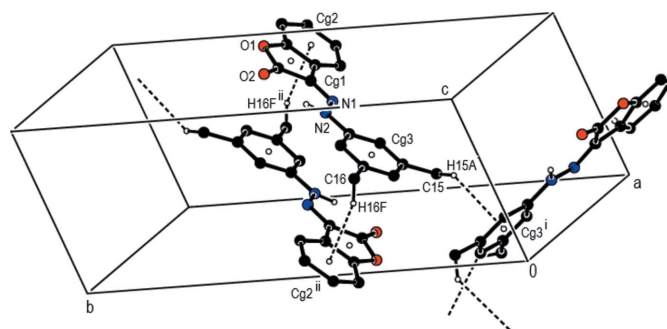


Figure 2

A partial packing view of the title compound, showing the C—H \cdots π interactions (dashed lines). Only H atoms involved in the interactions and N-bound H atoms are shown for clarity [Symmetry codes: (i) $x, -y - \frac{1}{2}, z - \frac{3}{2}$; (ii) $-x + 1, -y + 1, -z + 1$].

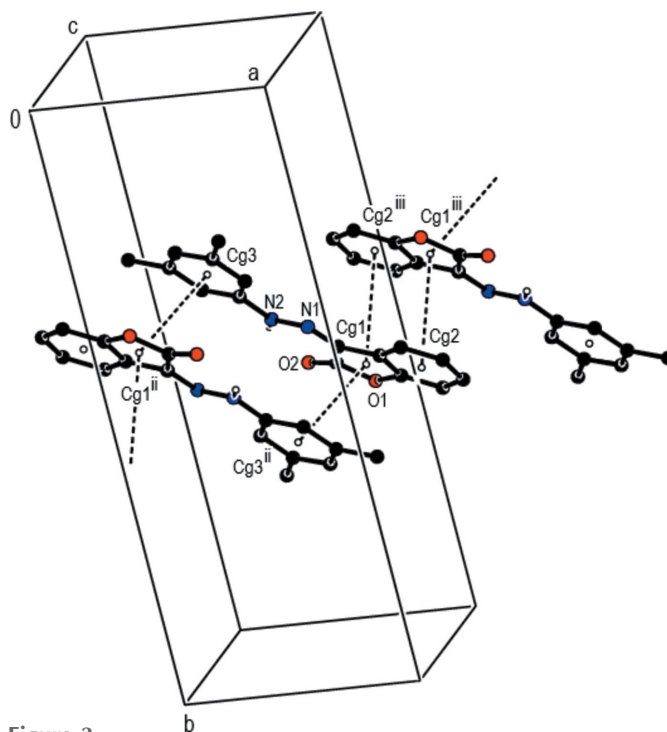


Figure 3

A partial packing view of the title compound, showing the π — π stacking interactions (dashed lines). Only N-bound H atoms are shown for clarity [Symmetry codes: (ii) $-x + 1, -y + 1, -z + 1$; (iii) $2 - x, 1 - y, 2 - z$].

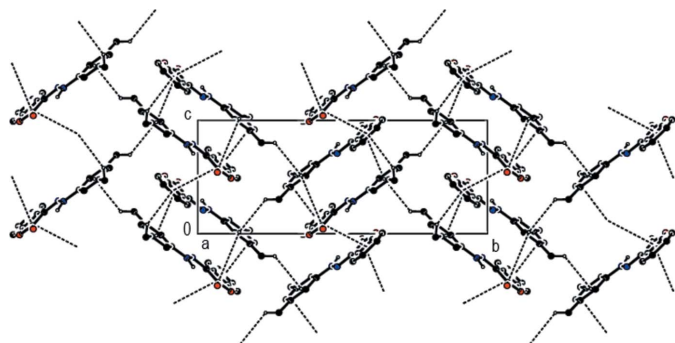


Figure 4
A packing diagram of the title compound viewed along the *a*-axis, showing C–H... π interactions (dashed lines). Only H atoms involved in the interactions and N-bound H atoms are shown for clarity.

hydro-1-benzofuran ring system while *Cg*3 is the centroid of the dimethylphenyl C9–C14 ring (Fig. 3). These interactions link the molecules into a layer structure lying parallel to the (11 $\bar{1}$) plane (Fig. 4).

4. Hirshfeld surface analysis

*Crystal Explorer*17 (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. 5*a*). 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. 5*b* 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. 6*b*). The O...H/H...O contacts contribute 17.9% (Fig. 6*c*) to the Hirshfeld surface, while the C...H/H...C contacts contribute 15.2% (Fig. 6*d*), resulting in a pair of distinctive wings. The C...C interactions account for 8.1% of the Hirshfeld surface. The percentage

Table 2

Percentage contributions of interatomic contacts to the Hirshfeld surface of the title compound.

Contact	Percentage contribution
H...H	51.2
O...H/H...O	17.9
C...H/H...C	15.2
C...C	8.1
N...C/C...N	4.2
N...H/H...N	1.9
O...C/C...O	0.9
N...N	0.6
O...O	0.1

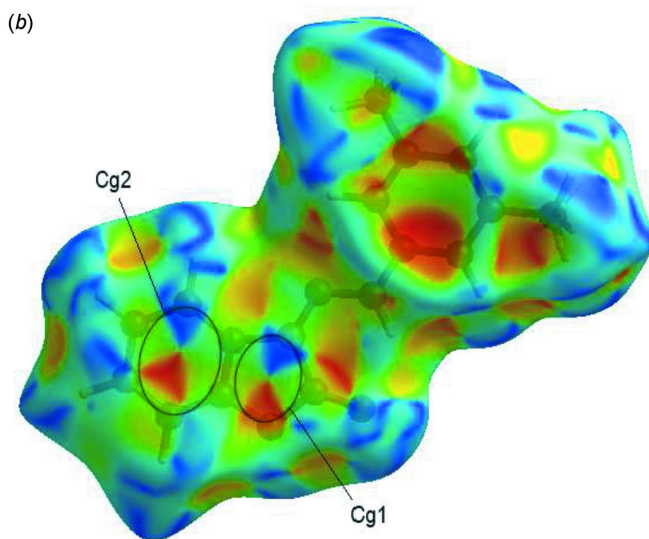
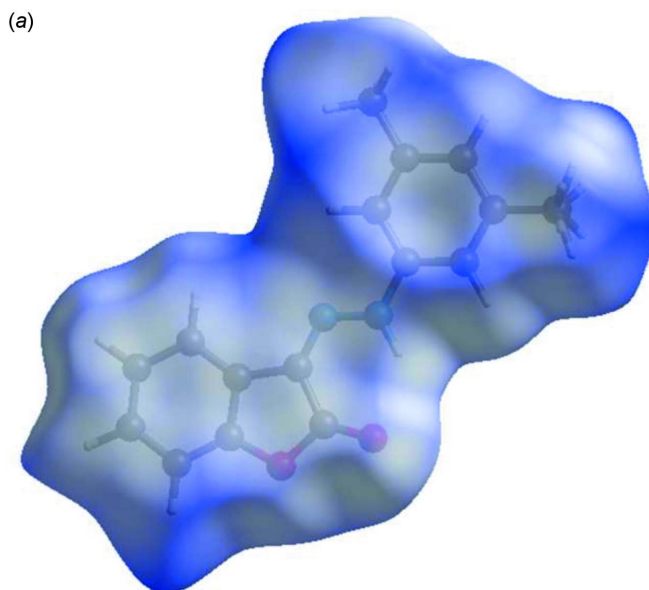
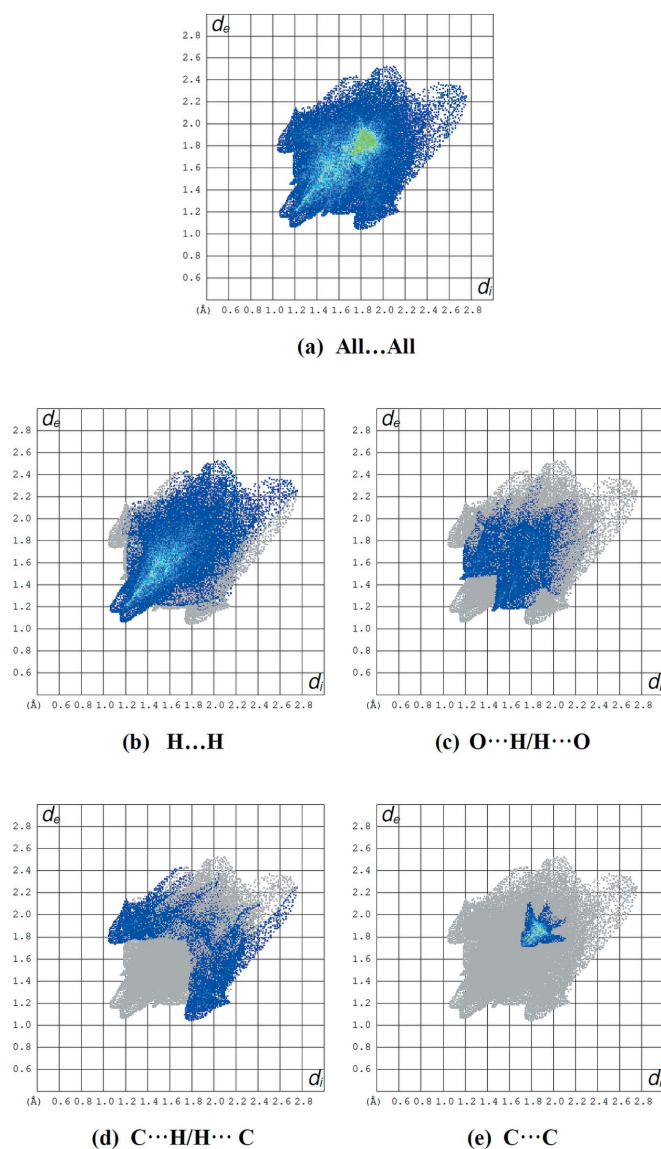


Figure 5
Hirshfeld surfaces of the title molecule, (a) mapped with d_{norm} in the range -0.0001 to 1.5993 a.u. and (b) plotted over shape-index.

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(3*H*)-one unit gave 220 hits. Of these, the compound most similar to the title compound is 7-methoxy-3-(2-phenylhydrazinylidene)-1-benzofuran-2(3*H*)-one, **I** (CSD refcode IBADIC; Atioğlu *et al.*, 2021). Four compounds reported by Oliveira *et al.* (2019) are closely related to the title compound, *viz.* 2-(4-nitro-1*H*-imidazol-1-yl)-*N'*-[1-(pyridin-2-


Figure 6

Fingerprint plots showing (a) all intermolecular interactions and delineated into (b) H...H, (c) O...H/H...O, (d) C...H/H...C and (e) C...C contacts.

yl)ethylidene]acetohydrazide, **II** (TODMEH), 2-(2-nitro-1*H*-imidazol-1-yl)-*N'*-[1-(pyridin-2-yl)ethylidene]acetohydrazide, **III** (TODMIL), 2-(4-nitro-1*H*-imidazol-1-yl)-*N'*-[phenyl(pyridin-2-yl)methylidene]acetohydrazide, **IV** (TODMOR) and 2-(4-nitro-1*H*-imidazol-1-yl)-*N'*-[phenyl(pyridin-2-yl)methylidene]acetohydrazide, **V** (TODMUX). Compound **I** crystallizes in the monoclinic space group $C2/c$ with $Z = 8$. In the crystal of **I**, pairs of molecules are linked into dimers by N—H...O hydrogen bonds, forming an $R_2^2(12)$ ring motif, with the dimers stacked along the a -axis direction. These dimers are connected through π - π stacking interactions between the centroids of the benzene and furan rings of their 2,3-dihydro-1-benzofuran ring systems. Furthermore, there exists a C—H... π interaction that consolidates the crystal packing. Compounds **II** and **IV** crystallize in the monoclinic space group $P2_1/c$ with $Z = 4$. Compound **III** crystallizes in the

monoclinic space group $I2/a$ with $Z = 8$ and **V** crystallizes in the triclinic space group $P\bar{1}$ 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...O interaction. These interactions lead to the formation of dimeric structural arrangements. In the crystal of **IV**, an intermolecular N—H...N 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(3*H*)-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]methyl]phenol (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) δ 12.04 (1H, NH), 6.79–7.69 (7H, Ar), 2.37 (6H, 2CH₃). ¹³C NMR (75 MHz, CDCl₃) δ 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) Å]. All C-bound H atoms were placed at calculated positions using a riding model, with C—H = 0.93 or 0.96 Å, and with $U_{iso}(H) = 1.2$ or $1.5U_{eq}(C)$. 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 ($\bar{5}$ 14 10, 7 13 0, $\bar{11}$ 6 5, $\bar{10}$ 12 4, 11 1 0, $\bar{11}$ 1 1, $\bar{8}$ 19 6, $\bar{8}$ 0 8 and $\bar{10}$ 17 4) were omitted during the final refinement cycle.

Table 3

Experimental details.

Crystal data	
Chemical formula	C ₁₆ H ₁₄ N ₂ O ₂
<i>M_r</i>	266.29
Crystal system, space group	Monoclinic, <i>P</i> ₂ ₁ / <i>c</i>
Temperature (K)	296
<i>a</i> , <i>b</i> , <i>c</i> (Å)	8.8644 (4), 19.9222 (8), 8.1736 (3)
β (°)	107.240 (1)
<i>V</i> (Å ³)	1378.59 (10)
<i>Z</i>	4
Radiation type	Mo <i>K</i> α
μ (mm ⁻¹)	0.09
Crystal size (mm)	0.40 × 0.21 × 0.06
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (<i>SADABS</i> ; Bruker, 2003)
<i>T</i> _{min} , <i>T</i> _{max}	0.684, 0.746
No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections	22343, 4176, 2332
<i>R</i> _{int}	0.058
(sin θ / λ) _{max} (Å ⁻¹)	0.714
Refinement	
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	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
$\Delta\rho_{\text{max}}$, $\Delta\rho_{\text{min}}$ (e Å ⁻³)	0.15, -0.14

Computer programs: *APEX2* (Bruker, 2003), *SAINT* (Bruker, 2003), *SHELXT* (Sheldrick, 2015a), *SHELXL* (Sheldrick, 2015b), *ORTEP-3 for Windows* (Farrugia, 2012), *PLATON* (Spek, 2020).

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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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References

Atioğlu, Z., Akkurt, M., Askerova, U. F., Mukhtarova, S. H., Askerov, R. K. & Mlowe, S. (2021). *Acta Cryst.* **E77**, 907–911.
 Bruker (2003). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin.
 Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
 Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* **B72**, 171–179.

Gurbanov, A. V., Kuznetsov, M. L., Demukhamedova, S. D., Alieva, I. N., Godjaev, N. M., Zubkov, F. I., Mahmudov, K. T. & Pombeiro, A. J. L. (2020a). *CrystEngComm*, **22**, 628–633.
 Gurbanov, A. V., Kuznetsov, M. L., Mahmudov, K. T., Pombeiro, A. J. L. & Resnati, G. (2020b). *Chem. Eur. J.* **26**, 14833–14837.
 Kopylovich, M. N., Mahmudov, K. T., Mizar, A. & Pombeiro, A. J. L. (2011). *Chem. Commun.* **47**, 7248–7250.
 Ma, Z., Gurbanov, A. V., Maharramov, A. M., Guseinov, F. I., Kopylovich, M. N., Zubkov, F. I., Mahmudov, K. T. & Pombeiro, A. J. L. (2017a). *J. Mol. Catal. A Chem.* **426**, 526–533.
 Ma, Z., Gurbanov, A. V., Sutradhar, M., Kopylovich, M. N., Mahmudov, K. T., Maharramov, A. M., Guseinov, F. I., Zubkov, F. I. & Pombeiro, A. J. L. (2017b). *Mol. Catal.* **428**, 17–23.
 Ma, Z., Mahmudov, K. T., Aliyeva, V. A., Gurbanov, A. V., Guedes da Silva, M. F. C. & Pombeiro, A. J. L. (2021). *Coord. Chem. Rev.* **437**, 213859.
 Ma, Z., Mahmudov, K. T., Aliyeva, V. A., Gurbanov, A. V. & Pombeiro, A. J. L. (2020). *Coord. Chem. Rev.* **423**, 213482.
 Maharramov, A. M., Shikhaliyev, N. Q., Suleymanova, G. T., Gurbanov, A. V., Babayeva, G. V., Mammadova, G. Z., Zubkov, F. I., Nenajdenko, V. G., Mahmudov, K. T. & Pombeiro, A. J. L. (2018). *Dyes Pigments*, **159**, 135–141.
 Mahmoudi, G., Afkhami, F. A., Castiñeiras, A., García-Santos, I., Gurbanov, A., Zubkov, F. I., Mitoraj, M. P., Kukulka, M., Sagan, F., Szczepanik, D. W., Konyaeva, I. A. & Safin, D. A. (2018). *Inorg. Chem.* **57**, 4395–4408.
 Mahmoudi, G., Zaręba, J. K., Gurbanov, A. V., Bauzá, A., Zubkov, F. I., Kubicki, M., Stilinović, V., Kinzhybalov, V. & Frontera, A. (2017). *Eur. J. Inorg. Chem.* pp. 4763–4772.
 Mahmudov, K. T., Gurbanov, A. V., Aliyeva, V. A., Resnati, G. & Pombeiro, A. J. L. (2020). *Coord. Chem. Rev.* **418**, 213381.
 Mahmudov, K. T., Kopylovich, M. N., Haukka, M., Mahmudova, G. S., Esmaeila, E. F., Chyragov, F. M. & Pombeiro, A. J. L. (2013). *J. Mol. Struct.* **1048**, 108–112.
 Mizar, A., Guedes da Silva, M. F. C., Kopylovich, M. N., Mukherjee, S., Mahmudov, K. T. & Pombeiro, A. J. L. (2012). *Eur. J. Inorg. Chem.* pp. 2305–2313.
 Oliveira, A. P. A., Ferreira, I. P., Despaigne, A. A. R., da Silva, J. G., Vieira, A. C. S., Santos, M. S., Alexandre-Moreira, M. S., Diniz, R. & Beraldo, H. (2019). *Acta Cryst.* **C75**, 320–328.
 Sheldrick, G. M. (2015a). *Acta Cryst.* **A71**, 3–8.
 Sheldrick, G. M. (2015b). *Acta Cryst.* **C71**, 3–8.
 Shikhaliyev, N. Q., Ahmadova, N. E., Gurbanov, A. V., Maharramov, A. M., Mammadova, G. Z., Nenajdenko, V. G., Zubkov, F. I., Mahmudov, K. T. & Pombeiro, A. J. L. (2018). *Dyes Pigments*, **150**, 377–381.
 Shikhaliyev, N. Q., Kuznetsov, M. L., Maharramov, A. M., Gurbanov, A. V., Ahmadova, N. E., Nenajdenko, V. G., Mahmudov, K. T. & Pombeiro, A. J. L. (2019). *CrystEngComm*, **21**, 5032–5038.
 Shikhaliyev, N. Q., Gurbanov, A. V., Maharramov, A. M., Mahmudov, K. T., Kopylovich, M. N., Martins, L. M. D. R. S., Muzalevskiy, V. M., Nenajdenko, V. G. & Pombeiro, A. J. L. (2014). *New J. Chem.* **38**, 4807–4815.
 Spek, A. L. (2020). *Acta Cryst.* **E76**, 1–11.
 Turner, M. J., McKinnon, J. J., Wolff, S. K., Grimwood, D. J., Spackman, P. R., Jayatilaka, D. & Spackman, M. A. (2017). *CrystalExplorer17*. The University of Western Australia.
 Viswanathan, A., Kute, D., Musa, A., Konda Mani, S., Sipilä, V., Emmert-Streib, F., Zubkov, F. I., Gurbanov, A. V., Yli-Harja, O. & Kandhavelu, M. (2019). *Eur. J. Med. Chem.* **166**, 291–303.

supporting information

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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, 2015*a*); program(s) used to refine structure: *SHELXL* (Sheldrick, 2015*b*); 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(3*H*)-one

Crystal data

$C_{16}H_{14}N_2O_2$

$M_r = 266.29$

Monoclinic, $P2_1/c$

$a = 8.8644$ (4) Å

$b = 19.9222$ (8) Å

$c = 8.1736$ (3) Å

$\beta = 107.240$ (1)°

$V = 1378.59$ (10) Å³

$Z = 4$

$F(000) = 560$

$D_x = 1.283$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 3738 reflections

$\theta = 2.4$ – 30.5 °

$\mu = 0.09$ mm⁻¹

$T = 296$ K

Prism, colourless

$0.40 \times 0.21 \times 0.06$ mm

Data collection

Bruker APEXII CCD
diffractometer

φ and ω scans

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

$T_{\min} = 0.684$, $T_{\max} = 0.746$

22343 measured reflections

4176 independent reflections

2332 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.058$

$\theta_{\max} = 30.5$ °, $\theta_{\min} = 2.4$ °

$h = -12 \rightarrow 12$

$k = -28 \rightarrow 28$

$l = -11 \rightarrow 11$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.062$

$wR(F^2) = 0.146$

$S = 1.03$

4176 reflections

188 parameters

0 restraints

Hydrogen site location: mixed

H atoms treated by a mixture of independent
and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0465P)^2 + 0.2174P]$

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

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

$\Delta\rho_{\max} = 0.15$ e Å⁻³

$\Delta\rho_{\min} = -0.14$ 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
C1	0.7095 (2)	0.56971 (8)	0.9270 (2)	0.0519 (4)	
C2	0.76884 (18)	0.52981 (8)	0.8095 (2)	0.0482 (4)	
C3	0.92693 (18)	0.55352 (8)	0.8274 (2)	0.0493 (4)	
C4	1.0448 (2)	0.53565 (9)	0.7563 (3)	0.0624 (5)	
H4	1.028478	0.502040	0.673914	0.075*	
C5	1.1879 (2)	0.56929 (11)	0.8114 (3)	0.0728 (6)	
H5	1.268324	0.558312	0.764500	0.087*	
C6	1.2129 (2)	0.61884 (11)	0.9347 (3)	0.0749 (6)	
H6	1.310276	0.640405	0.969456	0.090*	
C7	1.0974 (2)	0.63715 (10)	1.0075 (3)	0.0673 (5)	
H7	1.114142	0.670299	1.091089	0.081*	
C8	0.95594 (19)	0.60361 (8)	0.9495 (2)	0.0538 (4)	
C9	0.4768 (2)	0.40926 (8)	0.6127 (2)	0.0530 (4)	
C10	0.5476 (2)	0.37321 (9)	0.5108 (2)	0.0603 (5)	
H10	0.648784	0.384407	0.508401	0.072*	
C11	0.4678 (3)	0.32049 (9)	0.4125 (2)	0.0702 (6)	
C12	0.3181 (3)	0.30497 (10)	0.4188 (3)	0.0779 (6)	
H12	0.263853	0.269826	0.351887	0.093*	
C13	0.2457 (2)	0.33984 (10)	0.5213 (3)	0.0693 (6)	
C14	0.3266 (2)	0.39267 (9)	0.6189 (2)	0.0598 (5)	
H14	0.280284	0.416993	0.688386	0.072*	
C15	0.5458 (3)	0.28029 (12)	0.3040 (3)	0.1039 (9)	
H15A	0.513973	0.234174	0.302284	0.156*	
H15B	0.658448	0.283384	0.351251	0.156*	
H15C	0.514651	0.297710	0.189347	0.156*	
C16	0.0820 (3)	0.32121 (12)	0.5296 (3)	0.0972 (8)	
H16D	0.052299	0.350758	0.607547	0.146*	0.67 (4)
H16E	0.082700	0.275746	0.568674	0.146*	0.67 (4)
H16F	0.007489	0.325362	0.417644	0.146*	0.67 (4)
H16A	0.013993	0.359673	0.500891	0.146*	0.33 (4)
H16B	0.088531	0.306500	0.643291	0.146*	0.33 (4)
H16C	0.039964	0.285692	0.449682	0.146*	0.33 (4)
N1	0.69683 (16)	0.48115 (7)	0.70999 (18)	0.0508 (3)	
N2	0.55439 (17)	0.46316 (7)	0.7133 (2)	0.0545 (4)	
H2	0.513 (2)	0.4843 (10)	0.792 (3)	0.077 (6)*	
O1	0.82605 (14)	0.61480 (6)	1.00954 (16)	0.0609 (3)	
O2	0.58433 (14)	0.56775 (7)	0.95705 (17)	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)		
O2—C1—O1	121.34 (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)
C1—C2—C3—C8	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—C4	178.17 (19)	C12—C13—C14—C9	-0.3 (3)
C8—C3—C4—C5	-0.1 (3)	C16—C13—C14—C9	179.33 (17)
C2—C3—C4—C5	-177.49 (18)	C10—C9—C14—C13	-0.5 (3)
C3—C4—C5—C6	0.5 (3)	N2—C9—C14—C13	179.91 (15)
C4—C5—C6—C7	-0.3 (3)	C3—C2—N1—N2	176.94 (15)
C5—C6—C7—C8	-0.4 (3)	C1—C2—N1—N2	-1.3 (2)
C6—C7—C8—C3	0.9 (3)	C2—N1—N2—C9	-177.48 (14)
C6—C7—C8—O1	179.25 (16)	C10—C9—N2—N1	1.1 (2)

C4—C3—C8—C7	-0.7 (3)	C14—C9—N2—N1	-179.26 (15)
C2—C3—C8—C7	177.41 (16)	O2—C1—O1—C8	178.89 (16)
C4—C3—C8—O1	-179.19 (14)	C2—C1—O1—C8	-0.88 (17)
C2—C3—C8—O1	-1.11 (19)	C7—C8—O1—C1	-177.24 (17)
C14—C9—C10—C11	0.6 (3)	C3—C8—O1—C1	1.27 (19)

Hydrogen-bond geometry (Å, °)

*Cg*2 and *Cg*3 are the centroids of the C3—C8 and C9—C14 rings, respectively.

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N2—H2 \cdots O2	0.93 (2)	2.12 (2)	2.840 (2)	133.8 (16)
C15—H15 <i>A</i> \cdots <i>Cg</i> 3 ⁱ	0.96	2.90	3.591 (3)	130
C16—H16 <i>F</i> \cdots <i>Cg</i> 2 ⁱⁱ	0.96	2.92	3.715 (3)	141

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