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Crystal structure and Hirshfeld surface analysis of (2*Z*)-4-oxo-4-{phenyl[(2*E*)-3-phenylprop-2-en-1-yl]-amino}but-2-enoic acid

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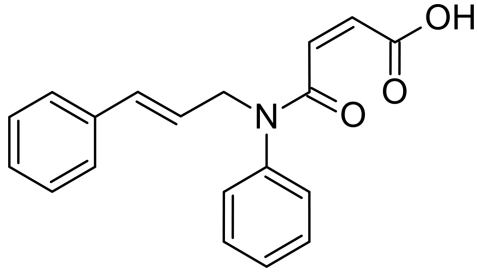
In the crystal structure of the title compound, $C_{19}H_{17}NO_3$, C—H···O hydrogen bonds connect molecular pairs to produce dimers with an $R_2^2(16)$ ring motif. Additionally, C—H···π interactions form ribbons along the [101] direction. Van der Waals interactions between the ribbons help to consolidate the molecular packing. Hirshfeld surface analysis shows that H···H (45.5%), C···H/H···C (30.4%), and O···H/H···O (19.3%) interactions are the main contributors to the crystal packing.

1. Chemical context

The intramolecular Diels–Alder (IMDA) reaction provides an efficient and versatile approach for the one-step construction of condensed carbo- and heterocyclic systems (Krishna *et al.*, 2022). However, successful implementation of this approach requires the presence of both diene and dienophile fragments within the same molecule, which is not always easily achievable. The starting compounds suitable for the intramolecular Diels–Alder reaction often possess complex molecular architectures and are obtained through multistep synthetic sequences (Patre *et al.*, 2007; Hu *et al.*, 2018).



The title compound can be synthesized in a single, straightforward step from amine, which, in turn, is readily prepared by the condensation of cinnamaldehyde with aniline followed by reduction of the resulting C=N bond. In the title compound, the vinyl arene fragment acts as a diene, while the maleimide fragment serves as a dienophile, making it a promising substrate for investigation of the intramolecular Diels–Alder reaction. It should be noted here, that the heterocyclic products derived from the title compound and its substituted analogues have demonstrated notable antiviral

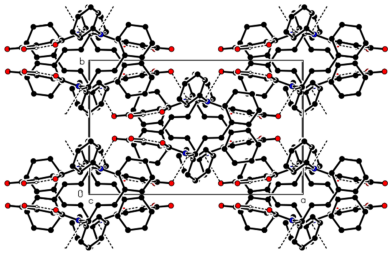


Table 1
Hydrogen-bond geometry (Å, °).

*Cg*1 and *Cg*2 are the centroids of the C8–C13 and C14–C19 phenyl rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5*B*···O1^{i}	0.99	2.51	3.2193 (19)	128
O2—H2*O*···O3	0.99 (2)	1.54 (2)	2.5341 (19)	173 (2)
C2—H2···*Cg*1^{ii}	0.95	2.76	3.597 (2)	147
C9—H9···*Cg*2^{ii}	0.95	2.91	3.6850 (19)	140
C12—H12···*Cg*2^{iii}	0.95	2.92	3.746 (2)	146

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

activity against the H1N1 influenza virus (Voronov *et al.*, 2018). Moreover, the carboxylic group in the title compound can be used in the synthesis of metal complex catalysts (Aliyeva *et al.*, 2024; Huseynov *et al.*, 2018, 2021), or act as the hydrogen-bond donor/acceptor in the synthesis of new supramolecular compounds (Burkin *et al.*, 2024; Maharramov *et al.*, 2011).

2. Structural commentary

The title compound (Fig. 1) exhibits a *Z* configuration about the C2═C3 double bond and *E* configuration about the C6═C7 double bond. The molecular conformation is consolidated by an intramolecular O—H···O hydrogen bond forming an *S*(7) motif (Table 1, Fig. 1; Bernstein *et al.*, 1995). The C—N bond lengths are: C4—N1 = 1.3544 (19), C5—N1 = 1.488 (2), and C14—N1 = 1.447 (2) Å. The sum of the angles around the N-atom [C4—N1—C14 = 123.00 (13), C4—N1—C5 = 118.99 (13), and C14—N1—C5 = 117.96 (12)°] is 359.95 (13)°. The molecular conformation is roughly planar [maximum deviations: −1.611 (2) Å for C5, 1.316 (2) Å for C10, −1.026 (2) Å for C7, and 1.189 (2) Å for C16]. The angle between the phenyl rings is 68.01 (8)°. The torsion angles C6—C7—C8—C9, C5—C6—C7—C8, N1—C5—C6—C7, C5—N1—C14—C15, C5—N1—C4—O3, C5—N1—C4—C3, N1—C4—C3—C2, C4—C3—C2—C1, C3—C2—C1—O1, and C3—C2—C1—O2 are −3.2 (3), −178.36 (14), −130.63 (16), −73.02 (17), −0.4 (2), 176.76 (12), 170.26 (15), −1.5 (3), −173.25 (16) and 7.5 (3)°, respectively.

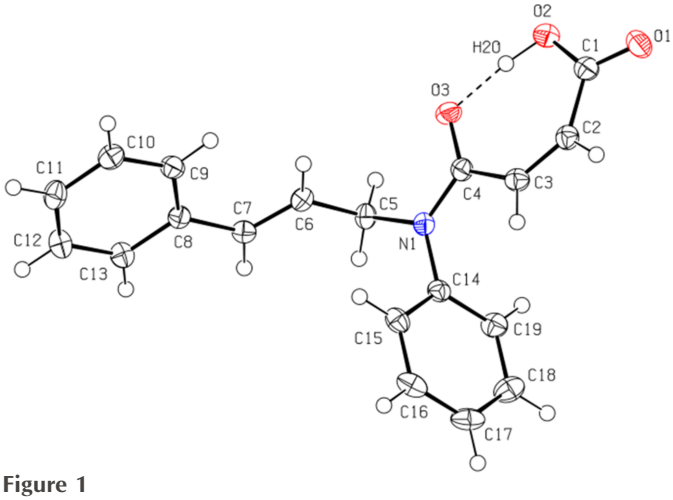


Figure 1
The molecular structure of the title compound, showing the atom labelling and displacement ellipsoids drawn at the 50% probability level. The intramolecular O—H···O hydrogen bond (dashed line) forms an *S*(7) motif between the hydroxyl hydrogen and the carbonyl oxygen atom.

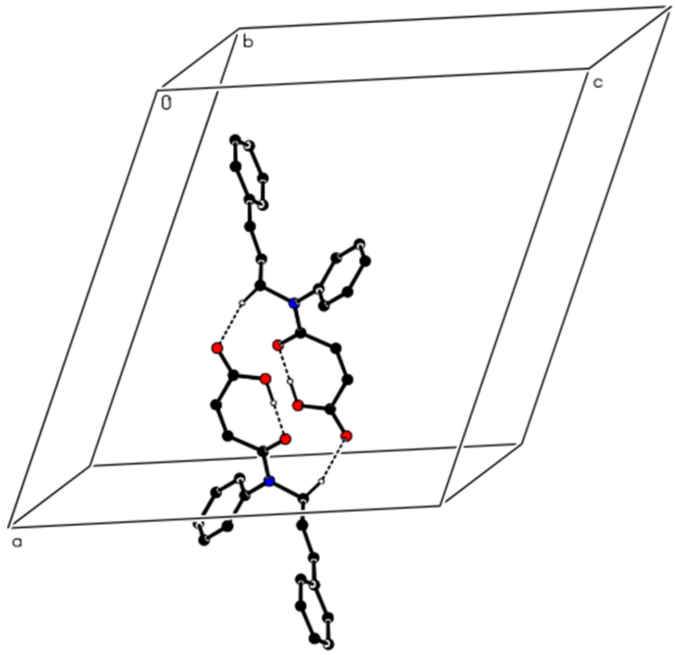


Figure 2
A partial view of the intramolecular O—H···O hydrogen bonds forming an *S*(7) motif and the intermolecular C—H···O hydrogen bonds between molecular pairs forming an $R_2^2(16)$ motif. Hydrogen atoms not involved in hydrogen bonding have been omitted for clarity.

3. Supramolecular features and Hirshfeld surface analysis

In the crystal, molecular pairs are linked by intermolecular C—H···O hydrogen bonds, forming dimers with an $R_2^2(16)$ ring motif (Table 1, Figs. 2 and 3). Additionally, C—H···π

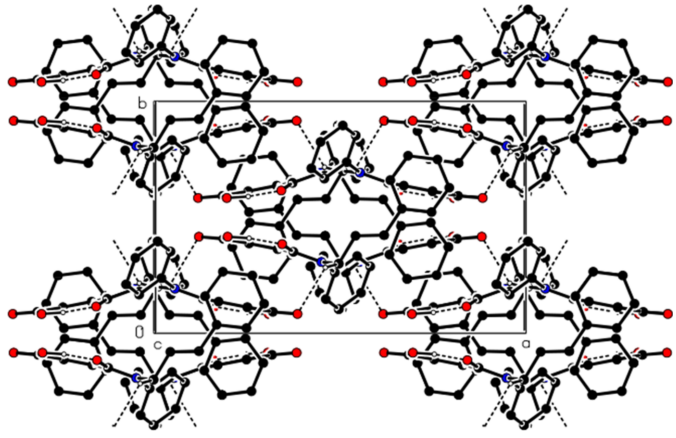


Figure 3
View of the molecular packing of the title compound along the *c* axis. The intramolecular O—H···O and intermolecular C—H···O hydrogen bonds are shown with dashed lines. Hydrogen atoms not involved in hydrogen bonding have been omitted for clarity.

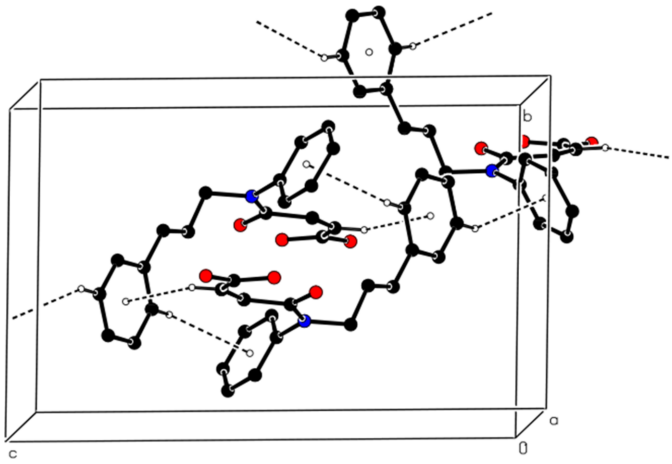


Figure 4
A partial view of the C—H···π interactions of the title compound in the unit cell. Hydrogen atoms not involved in hydrogen bonding have been omitted for clarity.

interactions connect the molecules into ribbons along the [101] direction (Table 1, Figs. 4 and 5). van der Waals interactions between the ribbons consolidate the molecular packing.

In order to visualize the intermolecular interactions (Tables 1 and 2) in the crystal, a Hirshfeld surface analysis was carried out using *Crystal Explorer 17.5* (Spackman *et al.*, 2021). Fig. 6 shows the Hirshfeld surface plotted over d_{norm} in the range −0.1614 to 1.4060 a.u. The red spots on the Hirshfeld surface represent O—H···O and C—H···O contacts.

Table 2
Summary of short interatomic contacts (Å).

Contact	Distance	Symmetry operation
O1···H17	2.62	$\frac{1}{2}+x,\frac{1}{2}+y,z$
H5*B*···O1	2.51	$\frac{3}{2}-x,\frac{1}{2}-y,1-z$
H5*B*···H5*B*	2.56	$1-x,y,\frac{1}{2}-z$
H3···C9	2.66	$1-x,1-y,1-z$
H10···H17	2.56	$x,1-y,-\frac{1}{2}+z$
C15···H11	2.97	$\frac{1}{2}-x,-\frac{1}{2}+y,\frac{1}{2}-z$
H19···C18	3.07	$1-x,-y,1-z$

Fig. 7 shows the full two-dimensional fingerprint plot and those delineated into the major contacts: H···H (Fig. 7*b*), C···H/H···C (Fig. 7*c*) and O···H/H···O (Fig. 7*d*) contacts contribute 45.5%, 30.4% and 19.3%, respectively, to the Hirshfeld surface. Specifically, the fingerprint plots reveal the presence of the C···H and O···H contacts appearing as pairs of spikes with the tips at $d_e + d_i$ = 2.53 Å and $d_e + d_i$ = 2.40 Å, respectively. The other remaining weak interactions (contribution percentages) are O···C/C···O (3.8%), O···O (0.5%), O···N/N···O (0.2%), N···H/H···N (0.2%) and C···C (0.1%).

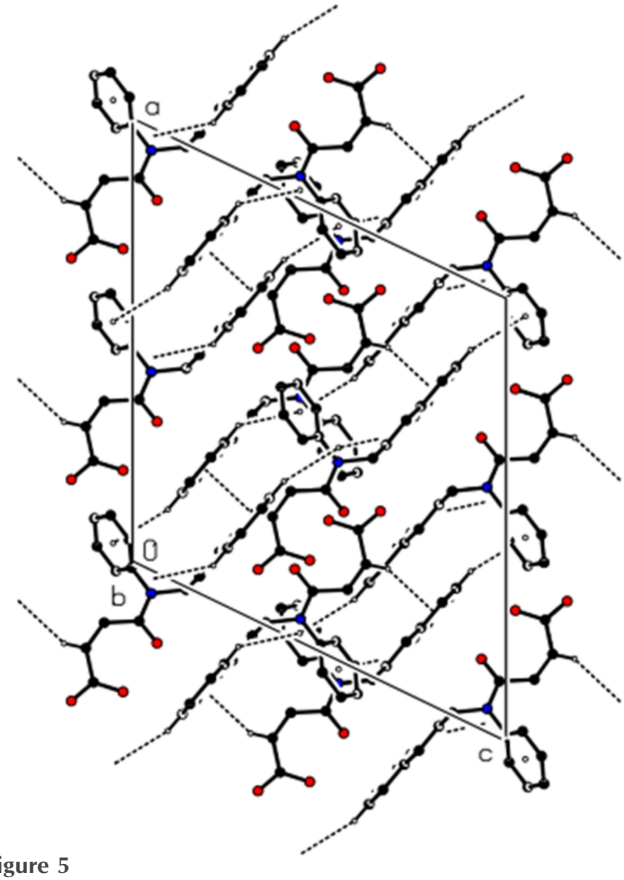


Figure 5
A view of the C—H···π interactions (dashed lines) of the title compound along the *b* axis. Hydrogen atoms not involved in hydrogen bonding have been omitted for clarity.

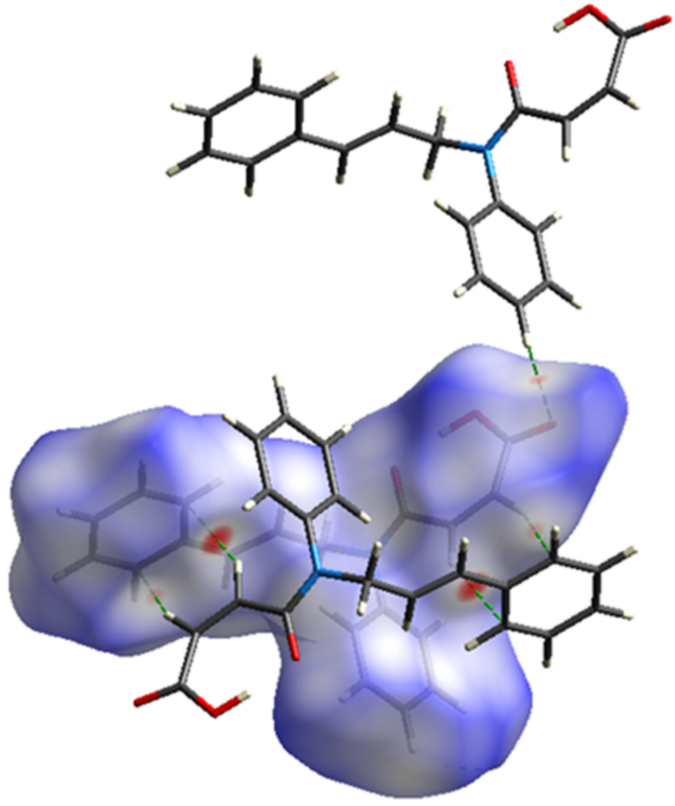


Figure 6
View of the three-dimensional Hirshfeld surface of the title compound plotted over d_{norm}. The intermolecular hydrogen bonds are shown with dashed lines.

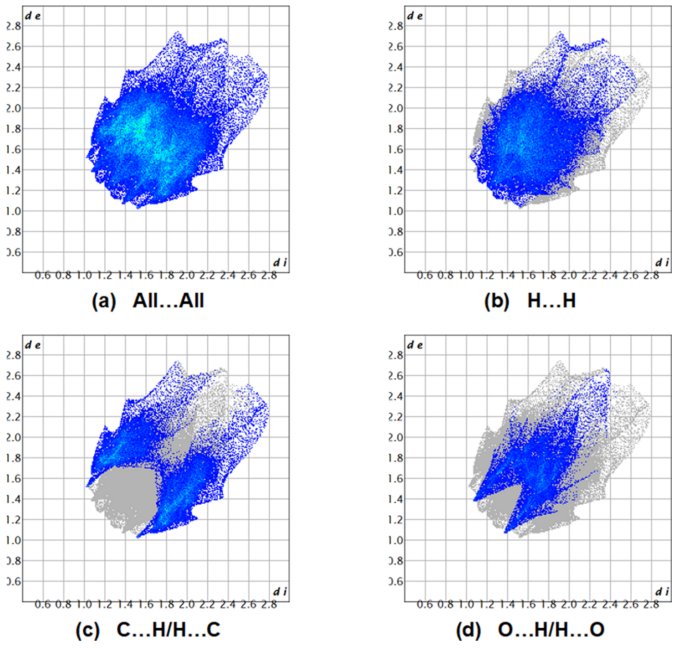


Figure 7
The full two-dimensional fingerprint plots for the title compound, showing (*a*) all interactions, and those delineated into (*b*) H···H, (*c*) C···H/H···C and (*d*) O···H/H···O interactions. The d_i and d_e values are the closest internal and external distances (in Å) from given points on the Hirshfeld surface.

4. Database survey

A search of the Cambridge Structural Database (CSD, Version 6.00, update of August 2025; Groom *et al.*, 2016) for the fragment N—C(═O)—C═C—COOH (4-amino-4-oxobut-2-enoic acid) gave in 97 hits. The closely related compounds are CSD refcodes UCOHON (Tahir *et al.*, 2023), AFIMUA (Dugarte-Dugarte *et al.*, 2019), IKECUX (Shah *et al.*, 2011), ANSMAL01 (Gowda *et al.*, 2010*a*), QUYYOZ (Gowda *et al.*, 2010*b*), LOSJUZ (Lo & Ng, 2009) and BIHXIA (Parvez *et al.*, 2004).

UCOHON, QUYYOZ and LOSJUZ crystallize in the monoclinic space group $P2_1/c$, with Z = 4 for UCOHON and QUYYOZ (Z = 8 for LOSJUZ). AFIMUA crystallizes in the monoclinic space group $P2_1/m$, with Z = 2. IKECUX crystallizes in the orthorhombic space group $Pna2_1$, with Z = 4. ANSMAL01 and BIHXIA crystallize in the triclinic space group $P\overline{1}$, with Z = 4.

The torsion angles of the central C—C═C—C group in the N—C(═O)—C═C—COOH fragment are 2.4° for UCOHON, 0.0° for AFIMUA, 1.3° for IKECUX, 0.6° and 3.5° for ANSMAL01 (two molecules in the asymmetric unit), 0.0° for QUYYOZ, 3.7° and 5.2° for LOSJUZ (two molecules in the asymmetric unit), and 1.0° and 0.5° for BIHXIA (two molecules in the asymmetric unit). As can be seen, the torsion angles are smaller than 5.2°, meaning that the functional groups at the ends of the C—C═C—C group point in the same direction and the structures therefore have the same Z configuration.

In UCOHON, molecules are connected as $R_2^1(6)$ dimers *via* N—H···O and C—H···O hydrogen bonds. Intermolecular bonding produces a monoperiodic infinite chain of molecules with a base vector [201]. Furthermore, π-π- stacking enhances the cohesion of the packing. In AFIMUA, molecules are connected by C—H···O and N—H···O hydrogen bonds, forming layers parallel to the (020) plane. The crystal cohesion is provided by van der Waals interactions between the layers. In IKECUX, intermolecular N—H···O bonds lead to the formation of polymer chains propagating along [011]. In ANSMAL01, intermolecular N—H···O hydrogen bonds link the molecules into zigzag chains extending along [1$\overline{1}$0]. Weak intermolecular C—H···O hydrogen bonds also occur. In QUYYOZ, intermolecular N—H···O hydrogen bonds link the molecules into *C*(7) chains running [010]. In LOSJUZ, adjacent molecules are linked by N—H···O hydrogen bonds into a flat ribbon that runs along the [100] direction. In the BIHXIA, the strong intermolecular N—H···O hydrogen bonds create a hydrophobic area in the centre of the unit cell.

5. Synthesis and crystallization

The synthesis of the title compound was described earlier (Voronov *et al.*, 2018). *N*-[(2*E*)-3-Phenylprop-2-en-1-yl]aniline (0.84 g, 4.00 mmol) was dissolved in diethyl ether (5 mL), and maleic anhydride (0.39 g, 4.00 mmol) was added. Hexane (~4 mL) was then added dropwise until the solution became turbid, after 3–5 drops of diethyl ether were added to restore clarity. The reaction mixture was allowed to stand for 2 h at room temperature. The resulting crystalline precipitate was collected by filtration and dried to afford the target amide as yellowish crystals (1.17 g, 3.80 mmol, 95%, m.p. 362–363 K). The single crystal suitable for XRD analysis was selected from the reaction mixture.

^{1}H NMR (600 MHz, $CDCl_3$, 294 K) (*J*, Hz): δ 7.50–7.20 (*m*, 10 H, H-Ph), 6.48 (*d*, *J* = 16.0, 1 H, H-3-allyl), 6.27 (*dt*, *J* = 6.6, 16.0, 1 H, H-2-allyl), 6.20 (*d*, *J* = 13.2, 1 H, H-maleic), 6.16 (*d*, *J* = 13.2, 1 H, H-maleic), 4.55 (*d*, *J* = 6.6, 2 H, H-CH_2) ppm. ^{13}C{^{1}H} NMR (151 MHz, $CDCl_3$, 294 K) δ 165.6, 165.0, 140.0, 135.8 (2C), 135.6, 135.0, 130.1 (2C), 129.2, 128.6, 128.5 (2C), 128.0, 127.4, 126.4 (2C), 121.4, 52.8 ppm. MS (ESI$^+$): *m*/*z* (%) = 308.1 [*M* + H]$^+$. IR (KBr), ν (cm^{-1}) 3420, 3026, 1714, 1625. Analysis calculated for $C_{19}H_{17}NO_3$: C, 74.25; H, 5.58; N, 4.56. Found: C, 74.19; H, 5.32; N, 4.68.

6. Refinement

The SC X-ray diffraction data for title compound were collected at the Belok/XSA beamline at the Kurchatov Synchrotron Radiation Source (National Research Center 'Kurchatov Institute', Moscow, Russia) at 100 K (Svetogorov *et al.*, 2020) using a 1-axis MarDTB goniometer equipped with a Rayonix SX165 two-dimensional CCD position-sensitive detector (λ = 0.96990 Å) in direct geometry with the detector plane perpendicular to the photon beam.Crystal data, data collection and structure refinement details are summarized in Table 3. The OH hydrogen was located in a difference-Fourier

map and refined with $U_{iso}(H) = 1.5U_{eq}(O)$. The C-bound H-atom positions were calculated geometrically at distances of 0.95 (for aromatic CH) and 0.99 (for CH_2), and refined using a riding model by applying the constraint $U_{iso}(H) = 1.2U_{eq}(C)$. Owing to poor agreement between the observed and calculated intensities, eighteen outliers (0 8 14, −5 5 18, −14 0 20, −15 1 20, 5 5 14, −4 6 17, −13 1 19, −22 2 5, 2 4 16, 1 5 16, −3 9 13, 4 2 15, 1 3 17, −5 9 15, 4 2 16, −2 6 17, 3 7 14 and −12 2 19) were omitted in the final cycles of refinement.

Acknowledgements

The authors' contributions are as follows. Conceptualization, MA and GMM; synthesis, KAA and AGK; spectral analysis AAZ and PJJ; X-ray analysis AAZ; writing (review and editing of the manuscript) KAA and MA; funding acquisition, KIH; supervision, MA and GMM.

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Table 3
Experimental details.

Crystal data	
Chemical formula	$C_{19}H_{17}NO_3$
M_r	307.33
Crystal system, space group	Monoclinic, *C*2/*c*
Temperature (K)	100
a, *b*, *c* (Å)	18.745 (4), 10.550 (2), 17.540 (4)
β (°)	115.74 (3)
V (Å^3)	3124.5 (13)
Z	8
Radiation type	Synchrotron, λ = 0.96990 Å
μ (mm^{-1})	0.20
Crystal size (mm)	0.15 × 0.15 × 0.15
Data collection	
Diffractometer	MAR CCD
Absorption correction	Multi-scan (*SCALA*; Evans, 2006)
T_{min}, T_{max}	0.960, 0.960
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	25658, 3213, 2779
R_{int}	0.064
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.637
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.042, 0.132, 1.15
No. of reflections	3213
No. of parameters	212
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.25, −0.27

Computer programs: *Automar* (Doyle, 2011), *iMosflm* (Battye *et al.*, 2011), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2018* (Sheldrick, 2015*b*), *ORTEP-3 for Windows* (Farrugia, 2012) and *PLATON* (Spek, 2020).

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supporting information

Acta Cryst. (2026). E**82**, 56-60 [https://doi.org/10.1107/S2056989025010746]

Crystal structure and Hirshfeld surface analysis of (2*Z*)-4-oxo-4-{phenyl[(2*E*)-3-phenylprop-2-en-1-yl]amino}but-2-enoic acid

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Computing details

(2*Z*)-4-Oxo-4-{phenyl[(2*E*)-3-phenylprop-2-en-1-yl]amino}but-2-enoic acid

Crystal data

$C_{19}H_{17}NO_3$
M_r = 307.33
Monoclinic, *C*2/*c*
a = 18.745 (4) Å
b = 10.550 (2) Å
c = 17.540 (4) Å
β = 115.74 (3)°
V = 3124.5 (13) Å^3
Z = 8
F(000) = 1296
D_x = 1.307 Mg m^{-3}
Synchrotron radiation, *λ* = 0.96990 Å
Cell parameters from 600 reflections
θ = 3.6–36.0°
μ = 0.20 mm^{-1}
T = 100 K
Prism, colourless
0.15 × 0.15 × 0.15 mm

Data collection

MAR CCD diffractometer
/f scan
Absorption correction: multi-scan (Scala; Evans, 2006)
T_{min} = 0.960, T_{max} = 0.960
25658 measured reflections
3213 independent reflections
2779 reflections with $I > 2\sigma(I)$
R_{int} = 0.064
θ_{max} = 38.2°, θ_{min} = 3.5°
h = −23→23
k = −13→13
l = −20→20

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.132
S = 1.15
3213 reflections
212 parameters
0 restraints
Primary atom site location: difference Fourier map
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0541P)^2 + 3.0894P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}
Extinction correction: SHELXL2019/2 (Sheldrick, 2015b), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0077 (6)

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*	U_{iso}*/U_{eq}
C1	0.81635 (9)	0.40744 (13)	0.60505 (11)	0.0235 (4)
C2	0.74709 (9)	0.38736 (14)	0.62488 (11)	0.0232 (4)
H2	0.760172	0.392749	0.683533	0.028*
C3	0.66985 (9)	0.36316 (14)	0.57599 (10)	0.0228 (3)
H3	0.637932	0.352217	0.605290	0.027*
C4	0.62796 (9)	0.35132 (13)	0.48221 (10)	0.0207 (3)
C5	0.50610 (9)	0.30015 (14)	0.35641 (10)	0.0219 (3)
H5A	0.468419	0.228464	0.342232	0.026*
H5B	0.542462	0.283513	0.330163	0.026*
C6	0.46102 (9)	0.42042 (14)	0.32047 (10)	0.0215 (3)
H6	0.488691	0.498638	0.336577	0.026*
C7	0.38409 (9)	0.42312 (14)	0.26705 (10)	0.0211 (3)
H7	0.357350	0.343931	0.253055	0.025*
C8	0.33638 (9)	0.53712 (14)	0.22753 (10)	0.0204 (3)
C9	0.36739 (9)	0.66059 (14)	0.24580 (10)	0.0236 (4)
H9	0.421666	0.672530	0.283325	0.028*
C10	0.31969 (10)	0.76554 (15)	0.20969 (10)	0.0260 (4)
H10	0.341277	0.848460	0.223502	0.031*
C11	0.24038 (10)	0.74943 (15)	0.15334 (11)	0.0284 (4)
H11	0.207920	0.821191	0.128587	0.034*
C12	0.20879 (10)	0.62771 (16)	0.13338 (11)	0.0283 (4)
H12	0.154942	0.616287	0.094289	0.034*
C13	0.25630 (9)	0.52277 (15)	0.17083 (10)	0.0241 (4)
H13	0.234092	0.440158	0.157767	0.029*
C14	0.51582 (8)	0.26228 (14)	0.50220 (10)	0.0205 (3)
C15	0.45572 (9)	0.33305 (15)	0.50853 (10)	0.0238 (4)
H15	0.439150	0.411006	0.478917	0.029*
C16	0.42017 (9)	0.28861 (16)	0.55858 (11)	0.0288 (4)
H16	0.379095	0.336094	0.563066	0.035*
C17	0.44493 (10)	0.17476 (16)	0.60189 (12)	0.0312 (4)
H17	0.420875	0.144754	0.636241	0.037*
C18	0.50497 (11)	0.10416 (15)	0.59522 (12)	0.0309 (4)
H18	0.521832	0.026592	0.625254	0.037*
C19	0.54025 (9)	0.14736 (14)	0.54451 (11)	0.0254 (4)
H19	0.580441	0.098896	0.538949	0.031*
N1	0.55259 (7)	0.30759 (11)	0.44989 (8)	0.0202 (3)
O1	0.88229 (6)	0.41635 (11)	0.66379 (8)	0.0308 (3)
O2	0.80530 (7)	0.41655 (11)	0.52536 (8)	0.0287 (3)
H2O	0.7476 (14)	0.410 (2)	0.4880 (14)	0.043*

O3	0.65846 (6)	0.38441 (11)	0.43448 (7)	0.0257 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0237 (8)	0.0162 (7)	0.0309 (10)	0.0003 (5)	0.0121 (7)	−0.0005 (6)
C2	0.0239 (8)	0.0209 (7)	0.0233 (9)	0.0002 (6)	0.0087 (6)	0.0000 (6)
C3	0.0238 (7)	0.0219 (7)	0.0248 (9)	−0.0006 (6)	0.0124 (6)	−0.0007 (6)
C4	0.0207 (7)	0.0174 (7)	0.0236 (8)	0.0018 (5)	0.0093 (6)	0.0008 (6)
C5	0.0224 (7)	0.0227 (7)	0.0185 (8)	0.0027 (6)	0.0070 (6)	−0.0005 (6)
C6	0.0249 (7)	0.0195 (7)	0.0201 (8)	0.0008 (5)	0.0100 (6)	0.0004 (6)
C7	0.0225 (7)	0.0204 (7)	0.0226 (8)	−0.0003 (5)	0.0118 (6)	−0.0021 (6)
C8	0.0219 (7)	0.0216 (7)	0.0194 (8)	0.0019 (5)	0.0106 (6)	0.0001 (6)
C9	0.0214 (7)	0.0230 (7)	0.0270 (9)	−0.0004 (6)	0.0111 (6)	0.0002 (6)
C10	0.0308 (8)	0.0204 (7)	0.0281 (9)	0.0004 (6)	0.0141 (7)	0.0000 (6)
C11	0.0296 (8)	0.0242 (8)	0.0297 (9)	0.0084 (6)	0.0113 (7)	0.0043 (7)
C12	0.0233 (8)	0.0301 (8)	0.0273 (9)	0.0038 (6)	0.0071 (7)	−0.0006 (7)
C13	0.0237 (7)	0.0230 (7)	0.0240 (9)	0.0000 (6)	0.0089 (6)	−0.0025 (6)
C14	0.0191 (7)	0.0202 (7)	0.0206 (8)	−0.0035 (5)	0.0072 (6)	−0.0025 (6)
C15	0.0196 (7)	0.0226 (7)	0.0262 (9)	−0.0014 (6)	0.0074 (6)	−0.0052 (6)
C16	0.0233 (8)	0.0313 (8)	0.0344 (10)	−0.0063 (6)	0.0149 (7)	−0.0102 (7)
C17	0.0353 (9)	0.0302 (8)	0.0340 (10)	−0.0152 (7)	0.0205 (8)	−0.0093 (7)
C18	0.0416 (10)	0.0208 (7)	0.0324 (10)	−0.0064 (6)	0.0179 (8)	−0.0022 (7)
C19	0.0269 (8)	0.0193 (7)	0.0313 (9)	−0.0009 (6)	0.0138 (7)	−0.0022 (6)
N1	0.0195 (6)	0.0199 (6)	0.0200 (7)	0.0011 (5)	0.0074 (5)	0.0005 (5)
O1	0.0208 (6)	0.0285 (6)	0.0381 (8)	−0.0001 (4)	0.0082 (5)	−0.0003 (5)
O2	0.0250 (6)	0.0324 (6)	0.0319 (7)	0.0007 (5)	0.0153 (5)	0.0043 (5)
O3	0.0274 (6)	0.0291 (6)	0.0233 (6)	−0.0027 (4)	0.0135 (5)	0.0027 (5)

Geometric parameters (Å, º)

C1—O1	1.221 (2)	C10—C11	1.393 (2)
C1—O2	1.325 (2)	C10—H10	0.9500
C1—C2	1.498 (2)	C11—C12	1.394 (2)
C2—C3	1.348 (2)	C11—H11	0.9500
C2—H2	0.9500	C12—C13	1.394 (2)
C3—C4	1.489 (2)	C12—H12	0.9500
C3—H3	0.9500	C13—H13	0.9500
C4—O3	1.2511 (19)	C14—C19	1.391 (2)
C4—N1	1.3544 (19)	C14—C15	1.395 (2)
C5—N1	1.488 (2)	C14—N1	1.447 (2)
C5—C6	1.503 (2)	C15—C16	1.394 (2)
C5—H5A	0.9900	C15—H15	0.9500
C5—H5B	0.9900	C16—C17	1.389 (3)
C6—C7	1.336 (2)	C16—H16	0.9500
C6—H6	0.9500	C17—C18	1.396 (3)
C7—C8	1.479 (2)	C17—H17	0.9500
C7—H7	0.9500	C18—C19	1.395 (2)

C8—C13	1.403 (2)	C18—H18	0.9500
C8—C9	1.406 (2)	C19—H19	0.9500
C9—C10	1.391 (2)	O2—H2O	0.99 (2)
C9—H9	0.9500		

O1—C1—O2	121.44 (15)	C11—C10—H10	119.9
O1—C1—C2	118.47 (15)	C10—C11—C12	119.79 (14)
O2—C1—C2	120.09 (14)	C10—C11—H11	120.1
C3—C2—C1	132.74 (16)	C12—C11—H11	120.1
C3—C2—H2	113.6	C13—C12—C11	119.92 (15)
C1—C2—H2	113.6	C13—C12—H12	120.0
C2—C3—C4	128.61 (15)	C11—C12—H12	120.0
C2—C3—H3	115.7	C12—C13—C8	121.08 (14)
C4—C3—H3	115.7	C12—C13—H13	119.5
O3—C4—N1	120.78 (15)	C8—C13—H13	119.5
O3—C4—C3	122.75 (14)	C19—C14—C15	120.92 (15)
N1—C4—C3	116.41 (14)	C19—C14—N1	119.27 (13)
N1—C5—C6	111.80 (12)	C15—C14—N1	119.80 (13)
N1—C5—H5A	109.3	C16—C15—C14	119.57 (15)
C6—C5—H5A	109.3	C16—C15—H15	120.2
N1—C5—H5B	109.3	C14—C15—H15	120.2
C6—C5—H5B	109.3	C17—C16—C15	119.85 (15)
H5A—C5—H5B	107.9	C17—C16—H16	120.1
C7—C6—C5	123.45 (14)	C15—C16—H16	120.1
C7—C6—H6	118.3	C16—C17—C18	120.37 (16)
C5—C6—H6	118.3	C16—C17—H17	119.8
C6—C7—C8	126.49 (14)	C18—C17—H17	119.8
C6—C7—H7	116.8	C19—C18—C17	120.10 (16)
C8—C7—H7	116.8	C19—C18—H18	119.9
C13—C8—C9	118.09 (13)	C17—C18—H18	119.9
C13—C8—C7	119.17 (13)	C14—C19—C18	119.17 (15)
C9—C8—C7	122.73 (13)	C14—C19—H19	120.4
C10—C9—C8	120.90 (14)	C18—C19—H19	120.4
C10—C9—H9	119.6	C4—N1—C14	123.00 (13)
C8—C9—H9	119.6	C4—N1—C5	118.99 (13)
C9—C10—C11	120.20 (15)	C14—N1—C5	117.96 (12)
C9—C10—H10	119.9	C1—O2—H2O	108.4 (13)

O1—C1—C2—C3	−173.25 (16)	N1—C14—C15—C16	179.61 (14)
O2—C1—C2—C3	7.5 (3)	C14—C15—C16—C17	0.2 (2)
C1—C2—C3—C4	−1.5 (3)	C15—C16—C17—C18	−0.3 (2)
C2—C3—C4—O3	−12.6 (2)	C16—C17—C18—C19	−0.3 (3)
C2—C3—C4—N1	170.26 (15)	C15—C14—C19—C18	−1.3 (2)
N1—C5—C6—C7	−130.63 (16)	N1—C14—C19—C18	179.70 (14)
C5—C6—C7—C8	−178.36 (14)	C17—C18—C19—C14	1.2 (2)
C6—C7—C8—C13	178.36 (16)	O3—C4—N1—C14	176.87 (13)
C6—C7—C8—C9	−3.2 (3)	C3—C4—N1—C14	−6.0 (2)
C13—C8—C9—C10	1.0 (2)	O3—C4—N1—C5	−0.4 (2)

C7—C8—C9—C10	−177.52 (15)	C3—C4—N1—C5	176.76 (12)
C8—C9—C10—C11	−1.2 (3)	C19—C14—N1—C4	−71.30 (19)
C9—C10—C11—C12	0.2 (3)	C15—C14—N1—C4	109.69 (16)
C10—C11—C12—C13	1.0 (3)	C19—C14—N1—C5	105.99 (16)
C11—C12—C13—C8	−1.2 (3)	C15—C14—N1—C5	−73.02 (17)
C9—C8—C13—C12	0.2 (2)	C6—C5—N1—C4	−90.16 (16)
C7—C8—C13—C12	178.78 (15)	C6—C5—N1—C14	92.43 (15)
C19—C14—C15—C16	0.6 (2)		

Hydrogen-bond geometry (Å, º)

*Cg*1 and *Cg*2 are the centroids of the C8–C13 and C14–C19 phenyl rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5*B*···O1^{i}	0.99	2.51	3.2193 (19)	128
O2—H2*O*···O3	0.99 (2)	1.54 (2)	2.5341 (19)	173 (2)
C2—H2···*Cg*1^{ii}	0.95	2.76	3.597 (2)	147
C9—H9···*Cg*2^{ii}	0.95	2.91	3.6850 (19)	140
C12—H12···*Cg*2^{iii}	0.95	2.92	3.746 (2)	146

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