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(*Z*)-2-(4-Methoxyanilino)-1,4-diphenylbut-2-ene-1,4-dione

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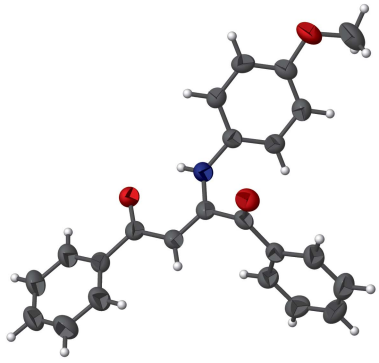
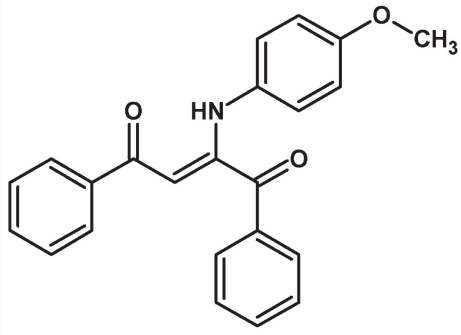
In the molecule of the title compound, $C_{23}H_{19}NO_3$, the mean plane of the methoxyphenyl ring makes dihedral angles of 51.63 (8) and 50.86 (8)° with the terminal phenyl rings. An intramolecular N—H···O hydrogen bond occurs. The crystal structure features C—H···O hydrogen bonds.

3D view



Chemical scheme



Structure description

β-Enaminones containing a conjugated –N—C═C—C═O system can be used to synthesise many heterocycles: for instance, biologically important heterocycles, such as isoxazoles (Lin *et al.*, 1980), pyrroles (Yan *et al.*, 2010), indoles (Würtz *et al.*, 2008) and pyrazoles (Neumann *et al.*, 2010), have been synthesized conveniently from suitably substituted *β*-enaminones. As part of our studies in this area, the title compound was synthesized and its single-crystal structure has been reported here.

In the molecular structure of the title compound (Fig. 1), the mean plane of the methoxyphenyl ring (C6–C11) makes dihedral angles of 51.63 (8) and 50.86 (8)°, respectively, with the C14–C19 and C22–C27 phenyl rings. An intramolecular N—H···O hydrogen bond occurs. The crystal structure features C—H···O hydrogen bonds (Table 1, Fig. 2).

Synthesis and crystallization

(*Z*)-2-Methylsulfanyl-1,4-diphenylbut-2-ene-1,4-dione (2.0 mmol, 1 equivalent), 4-methoxyaniline (2.6 mmol, 1.6 equivalents) and 5 volume of acidic silica with respect to starting substrate was thoroughly ground using a pestle and mortar. The solid reaction

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

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4···O1	0.86	1.96	2.6346 (17)	135
C7—H7···O1^{i}	0.93	2.51	3.334 (2)	148
C26—H26···O2^{ii}	0.93	2.58	3.261 (2)	130

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

mixture was transferred to an oven-dried 20 ml screw-cap reaction vial with magnetic stir-bar followed by the addition of anhydrous $AlCl_3$ (0.03 equivalents). The reaction mixture was stirred vigorously for 6 h. After completion of the reaction (monitored by TLC), the crude reaction mixture was purified through silica-gel column chromatography. Crystals suitable for analysis were obtained from an ethyl acetate–hexane (2:8 *v*/*v*) solution by slow evaporation at room temperature.

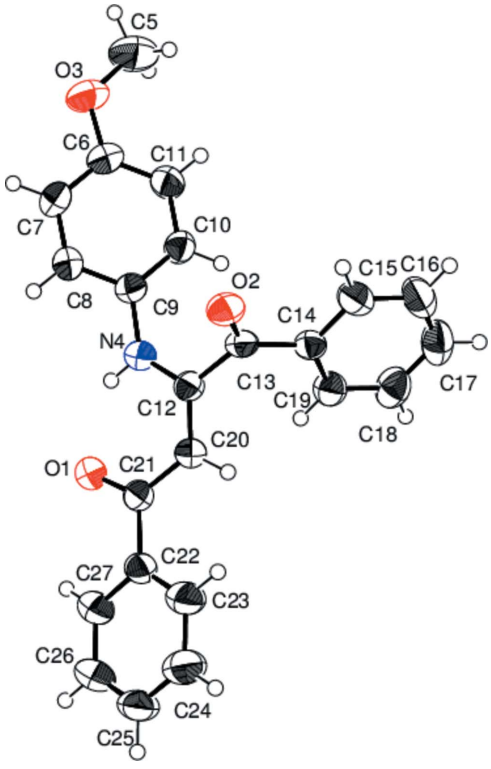


Figure 1
The molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level. H atoms are shown as spheres of arbitrary radius.

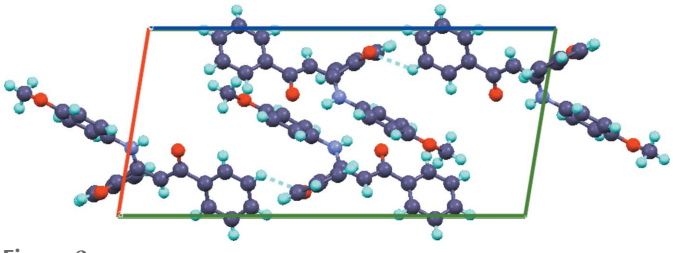


Figure 2
Packing diagram of the molecule viewed parallel to the *b* axis.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{23}H_{19}NO_3$
M_r	357.39
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	296
a, b, c (Å)	10.1567 (5), 8.5396 (4), 21.8019 (10)
β (°)	99.329 (2)
V (Å^3)	1865.96 (15)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	0.68
Crystal size (mm)	0.24 × 0.20 × 0.12
Data collection	
Diffractometer	Bruker SMART CCD area-detector
Absorption correction	multi- scan (*SADABS*; Bruker, 2009)
T_{min}, T_{max}	0.770, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	18393, 3060, 2801
R_{int}	0.048
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.584
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.050, 0.151, 1.06
No. of reflections	3060
No. of parameters	245
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.23, −0.22

Computer programs: *SMART* and *SAINT* (Bruker, 2009), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015) and *ORTEP-3 for Windows* (Farrugia, 2012).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

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References

Bruker (2009). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Lin, Y.-I. & Lang, S. A. (1980). *J. Org. Chem.* **45**, 4857–4860.
Neumann, J. J., Suri, M. & Glorius, F. (2010). *Angew. Chem. Int. Ed.* **49**, 7790–7794.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.
Würtz, S., Rakshit, S., Neumann, J. J., Dröge, T. & Glorius, F. (2008). *Angew. Chem.* **120**, 7340–7343.
Yan, R. L., Luo, J., Wang, C. X., Ma, C. W., Huang, G. S. & Liang, Y. M. (2010). *J. Org. Chem.* **75**, 5395–5397.

full crystallographic data

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Crystal data

$C_{23}H_{19}NO_3$
M_r = 357.39
Monoclinic, $P2_1/c$
a = 10.1567 (5) Å
b = 8.5396 (4) Å
c = 21.8019 (10) Å
β = 99.329 (2)°
V = 1865.96 (15) Å^3
Z = 4
$F(000)$ = 752
D_x = 1.272 Mg m^{-3}
Melting point: 375 K
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 3060 reflections
θ = 4.1–64.3°
μ = 0.68 mm^{-1}
T = 296 K
Prism, colourless
0.24 × 0.20 × 0.12 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scans
Absorption correction: multi-scan (SADABS; Bruker, 2009)
T_{min} = 0.770, T_{max} = 1.000
18393 measured reflections
3060 independent reflections
2801 reflections with $I > 2\sigma(I)$
R_{int} = 0.048
θ_{max} = 64.3°, θ_{min} = 4.1°
h = −11→11
k = −9→8
l = −25→25

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.151
S = 1.06
3060 reflections
245 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1038P)^2 + 0.2139P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}
Extinction correction: SHELXL2014 (Sheldrick 2015), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0053 (10)

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}
O1	0.64804 (12)	0.77341 (13)	0.62059 (5)	0.0584 (3)
O2	0.87120 (11)	0.58731 (12)	0.44792 (5)	0.0543 (3)
O3	0.37864 (16)	0.77684 (17)	0.26306 (6)	0.0781 (4)
N4	0.63382 (13)	0.67640 (14)	0.50499 (6)	0.0478 (3)
H4	0.5974	0.7271	0.5319	0.057*
C5	0.3437 (3)	0.6556 (3)	0.21998 (10)	0.1011 (8)
H5A	0.3013	0.6990	0.1812	0.152*
H5B	0.4226	0.5999	0.2138	0.152*
H5C	0.2834	0.5849	0.2355	0.152*
C6	0.44064 (16)	0.7394 (2)	0.32159 (7)	0.0530 (4)
C7	0.46065 (16)	0.86315 (19)	0.36271 (7)	0.0542 (4)
H7	0.4300	0.9624	0.3500	0.065*
C8	0.52575 (15)	0.84044 (17)	0.42243 (7)	0.0489 (4)
H8	0.5399	0.9248	0.4497	0.059*
C9	0.57053 (14)	0.69229 (17)	0.44240 (7)	0.0427 (4)
C10	0.54586 (14)	0.56800 (17)	0.40179 (7)	0.0488 (4)
H10	0.5718	0.4677	0.4152	0.059*
C11	0.48277 (16)	0.59083 (18)	0.34112 (7)	0.0536 (4)
H11	0.4689	0.5068	0.3137	0.064*
C12	0.74285 (14)	0.59305 (15)	0.52800 (6)	0.0414 (4)
C13	0.81574 (13)	0.50796 (16)	0.48246 (6)	0.0412 (4)
C14	0.81966 (13)	0.33465 (16)	0.48181 (6)	0.0423 (4)
C15	0.87236 (17)	0.2625 (2)	0.43389 (8)	0.0554 (4)
H15	0.9038	0.3232	0.4039	0.066*
C16	0.87819 (19)	0.1019 (2)	0.43071 (9)	0.0671 (5)
H16	0.9121	0.0544	0.3982	0.080*
C17	0.83407 (18)	0.0114 (2)	0.47545 (10)	0.0689 (5)
H17	0.8398	−0.0971	0.4736	0.083*
C18	0.78127 (18)	0.0815 (2)	0.52307 (9)	0.0642 (5)
H18	0.7515	0.0199	0.5532	0.077*
C19	0.77249 (15)	0.24275 (18)	0.52614 (7)	0.0503 (4)
H19	0.7351	0.2896	0.5578	0.060*
C20	0.79907 (15)	0.59469 (16)	0.58952 (6)	0.0455 (4)
H20	0.8718	0.5298	0.6026	0.055*
C21	0.75096 (16)	0.69174 (16)	0.63469 (7)	0.0461 (4)
C22	0.82350 (16)	0.69738 (17)	0.70020 (7)	0.0477 (4)
C23	0.94938 (19)	0.6344 (2)	0.71859 (8)	0.0632 (5)
H23	0.9919	0.5833	0.6896	0.076*
C24	1.0125 (2)	0.6467 (3)	0.77957 (9)	0.0729 (5)
H24	1.0977	0.6056	0.7913	0.088*
C25	0.9490 (2)	0.7201 (2)	0.82313 (8)	0.0651 (5)
H25	0.9910	0.7279	0.8642	0.078*
C26	0.8240 (2)	0.7811 (2)	0.80563 (8)	0.0709 (5)
H26	0.7808	0.8296	0.8350	0.085*
C27	0.7618 (2)	0.7709 (2)	0.74466 (8)	0.0632 (5)

H27	0.6773	0.8140	0.7332	0.076*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0743 (8)	0.0535 (6)	0.0464 (6)	0.0173 (5)	0.0065 (5)	−0.0035 (5)
O2	0.0662 (7)	0.0557 (7)	0.0433 (6)	−0.0025 (5)	0.0162 (5)	0.0092 (5)
O3	0.0962 (10)	0.0822 (9)	0.0477 (7)	0.0060 (7)	−0.0130 (6)	0.0084 (6)
N4	0.0566 (7)	0.0490 (7)	0.0376 (7)	0.0071 (5)	0.0072 (5)	−0.0003 (5)
C5	0.1192 (19)	0.115 (2)	0.0568 (12)	0.0020 (15)	−0.0224 (12)	−0.0074 (12)
C6	0.0529 (8)	0.0607 (10)	0.0437 (8)	0.0016 (7)	0.0023 (7)	0.0077 (7)
C7	0.0605 (9)	0.0458 (8)	0.0551 (9)	0.0084 (7)	0.0059 (7)	0.0102 (7)
C8	0.0550 (8)	0.0419 (8)	0.0490 (9)	0.0034 (6)	0.0065 (6)	−0.0023 (6)
C9	0.0452 (7)	0.0441 (8)	0.0388 (7)	0.0002 (6)	0.0066 (6)	0.0030 (6)
C10	0.0539 (8)	0.0401 (8)	0.0500 (9)	0.0001 (6)	0.0010 (6)	0.0014 (6)
C11	0.0570 (9)	0.0516 (9)	0.0495 (9)	−0.0013 (7)	0.0005 (7)	−0.0077 (7)
C12	0.0502 (8)	0.0363 (7)	0.0379 (7)	−0.0023 (5)	0.0074 (6)	0.0026 (5)
C13	0.0451 (7)	0.0460 (8)	0.0313 (7)	−0.0020 (6)	0.0021 (5)	0.0017 (5)
C14	0.0411 (7)	0.0457 (8)	0.0380 (7)	0.0002 (5)	0.0006 (6)	−0.0029 (6)
C15	0.0570 (9)	0.0612 (10)	0.0483 (9)	0.0011 (7)	0.0098 (7)	−0.0084 (7)
C16	0.0661 (10)	0.0616 (11)	0.0727 (12)	0.0084 (8)	0.0087 (9)	−0.0224 (9)
C17	0.0610 (10)	0.0450 (9)	0.0956 (14)	0.0039 (7)	−0.0023 (9)	−0.0125 (9)
C18	0.0670 (10)	0.0454 (9)	0.0779 (12)	−0.0060 (7)	0.0053 (9)	0.0073 (8)
C19	0.0527 (8)	0.0468 (8)	0.0516 (9)	−0.0031 (6)	0.0088 (7)	0.0001 (6)
C20	0.0556 (8)	0.0439 (8)	0.0368 (7)	0.0047 (6)	0.0067 (6)	0.0005 (6)
C21	0.0606 (9)	0.0379 (7)	0.0402 (8)	0.0014 (6)	0.0091 (6)	0.0021 (6)
C22	0.0640 (9)	0.0410 (8)	0.0385 (8)	−0.0022 (6)	0.0094 (7)	0.0011 (6)
C23	0.0714 (11)	0.0756 (11)	0.0427 (9)	0.0093 (9)	0.0094 (7)	−0.0030 (8)
C24	0.0747 (11)	0.0884 (14)	0.0515 (10)	0.0096 (10)	−0.0023 (8)	0.0019 (9)
C25	0.0878 (13)	0.0673 (11)	0.0378 (8)	−0.0092 (9)	0.0031 (8)	−0.0025 (7)
C26	0.0919 (14)	0.0795 (12)	0.0417 (9)	0.0064 (10)	0.0122 (9)	−0.0135 (8)
C27	0.0756 (11)	0.0681 (11)	0.0458 (9)	0.0108 (8)	0.0095 (8)	−0.0095 (8)

Geometric parameters (Å, º)

O1—C21	1.253 (2)	C14—C15	1.393 (2)
O2—C13	1.2170 (16)	C15—C16	1.374 (3)
O3—C6	1.366 (2)	C15—H15	0.9300
O3—C5	1.405 (3)	C16—C17	1.375 (3)
N4—C12	1.3433 (19)	C16—H16	0.9300
N4—C9	1.4173 (19)	C17—C18	1.380 (3)
N4—H4	0.8600	C17—H17	0.9300
C5—H5A	0.9600	C18—C19	1.383 (2)
C5—H5B	0.9600	C18—H18	0.9300
C5—H5C	0.9600	C19—H19	0.9300
C6—C7	1.380 (2)	C20—C21	1.432 (2)
C6—C11	1.384 (2)	C20—H20	0.9300
C7—C8	1.375 (2)	C21—C22	1.498 (2)

C7—H7	0.9300	C22—C23	1.385 (2)
C8—C9	1.390 (2)	C22—C27	1.387 (2)
C8—H8	0.9300	C23—C24	1.383 (2)
C9—C10	1.379 (2)	C23—H23	0.9300
C10—C11	1.387 (2)	C24—C25	1.382 (3)
C10—H10	0.9300	C24—H24	0.9300
C11—H11	0.9300	C25—C26	1.368 (3)
C12—C20	1.3701 (19)	C25—H25	0.9300
C12—C13	1.5166 (19)	C26—C27	1.379 (3)
C13—C14	1.481 (2)	C26—H26	0.9300
C14—C19	1.389 (2)	C27—H27	0.9300

C6—O3—C5	118.70 (16)	C16—C15—H15	119.9
C12—N4—C9	128.91 (12)	C14—C15—H15	119.9
C12—N4—H4	115.5	C15—C16—C17	120.21 (17)
C9—N4—H4	115.5	C15—C16—H16	119.9
O3—C5—H5A	109.5	C17—C16—H16	119.9
O3—C5—H5B	109.5	C16—C17—C18	120.08 (16)
H5A—C5—H5B	109.5	C16—C17—H17	120.0
O3—C5—H5C	109.5	C18—C17—H17	120.0
H5A—C5—H5C	109.5	C17—C18—C19	120.28 (16)
H5B—C5—H5C	109.5	C17—C18—H18	119.9
O3—C6—C7	115.13 (14)	C19—C18—H18	119.9
O3—C6—C11	125.08 (16)	C18—C19—C14	119.80 (15)
C7—C6—C11	119.79 (14)	C18—C19—H19	120.1
C8—C7—C6	120.32 (14)	C14—C19—H19	120.1
C8—C7—H7	119.8	C12—C20—C21	122.99 (13)
C6—C7—H7	119.8	C12—C20—H20	118.5
C7—C8—C9	120.51 (14)	C21—C20—H20	118.5
C7—C8—H8	119.7	O1—C21—C20	121.16 (14)
C9—C8—H8	119.7	O1—C21—C22	118.56 (13)
C10—C9—C8	118.89 (14)	C20—C21—C22	120.28 (13)
C10—C9—N4	123.46 (13)	C23—C22—C27	118.28 (15)
C8—C9—N4	117.58 (13)	C23—C22—C21	123.49 (14)
C9—C10—C11	120.80 (14)	C27—C22—C21	118.22 (15)
C9—C10—H10	119.6	C24—C23—C22	120.73 (16)
C11—C10—H10	119.6	C24—C23—H23	119.6
C6—C11—C10	119.63 (14)	C22—C23—H23	119.6
C6—C11—H11	120.2	C25—C24—C23	119.99 (18)
C10—C11—H11	120.2	C25—C24—H24	120.0
N4—C12—C20	123.21 (13)	C23—C24—H24	120.0
N4—C12—C13	118.02 (12)	C26—C25—C24	119.77 (16)
C20—C12—C13	118.40 (12)	C26—C25—H25	120.1
O2—C13—C14	122.32 (12)	C24—C25—H25	120.1
O2—C13—C12	117.54 (12)	C25—C26—C27	120.29 (17)
C14—C13—C12	120.13 (11)	C25—C26—H26	119.9
C19—C14—C15	119.33 (14)	C27—C26—H26	119.9
C19—C14—C13	123.03 (13)	C26—C27—C22	120.94 (17)

C15—C14—C13	117.64 (13)	C26—C27—H27	119.5
C16—C15—C14	120.28 (16)	C22—C27—H27	119.5
C5—O3—C6—C7	−174.3 (2)	C19—C14—C15—C16	−0.3 (2)
C5—O3—C6—C11	5.7 (3)	C13—C14—C15—C16	−179.77 (14)
O3—C6—C7—C8	−178.02 (15)	C14—C15—C16—C17	−1.1 (3)
C11—C6—C7—C8	1.9 (2)	C15—C16—C17—C18	1.3 (3)
C6—C7—C8—C9	−0.9 (2)	C16—C17—C18—C19	−0.1 (3)
C7—C8—C9—C10	−1.5 (2)	C17—C18—C19—C14	−1.4 (2)
C7—C8—C9—N4	−178.42 (13)	C15—C14—C19—C18	1.5 (2)
C12—N4—C9—C10	44.7 (2)	C13—C14—C19—C18	−179.03 (14)
C12—N4—C9—C8	−138.48 (15)	N4—C12—C20—C21	−3.3 (2)
C8—C9—C10—C11	2.9 (2)	C13—C12—C20—C21	169.52 (13)
N4—C9—C10—C11	179.62 (13)	C12—C20—C21—O1	4.8 (2)
O3—C6—C11—C10	179.39 (15)	C12—C20—C21—C22	−175.16 (13)
C7—C6—C11—C10	−0.6 (2)	O1—C21—C22—C23	−168.49 (17)
C9—C10—C11—C6	−1.9 (2)	C20—C21—C22—C23	11.5 (2)
C9—N4—C12—C20	176.00 (14)	O1—C21—C22—C27	10.9 (2)
C9—N4—C12—C13	3.1 (2)	C20—C21—C22—C27	−169.14 (15)
N4—C12—C13—O2	66.90 (17)	C27—C22—C23—C24	−0.9 (3)
C20—C12—C13—O2	−106.31 (15)	C21—C22—C23—C24	178.44 (17)
N4—C12—C13—C14	−114.13 (14)	C22—C23—C24—C25	1.1 (3)
C20—C12—C13—C14	72.67 (17)	C23—C24—C25—C26	−0.4 (3)
O2—C13—C14—C19	171.17 (13)	C24—C25—C26—C27	−0.6 (3)
C12—C13—C14—C19	−7.8 (2)	C25—C26—C27—C22	0.8 (3)
O2—C13—C14—C15	−9.4 (2)	C23—C22—C27—C26	−0.1 (3)
C12—C13—C14—C15	171.68 (12)	C21—C22—C27—C26	−179.45 (16)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H4···O1	0.86	1.96	2.6346 (17)	135
C7—H7···O1^{i}	0.93	2.51	3.334 (2)	148
C26—H26···O2^{ii}	0.93	2.58	3.261 (2)	130

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