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4-[(Ethoxyimino)(phenyl)methyl]-5-methyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one

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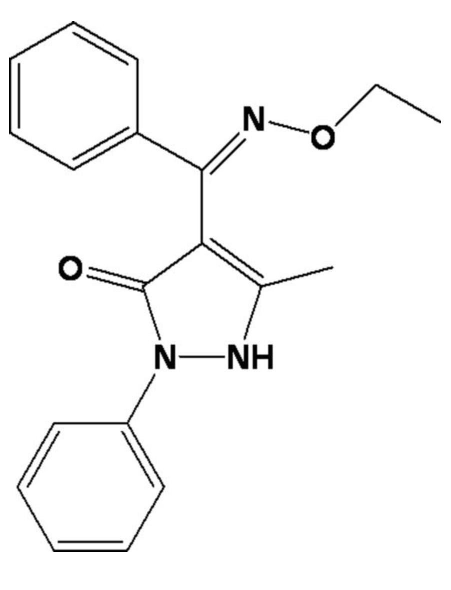
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.044; wR factor = 0.141; data-to-parameter ratio = 13.8.

In the molecule of the title compound, $C_{19}H_{19}N_3O_2$, the central pyrazole ring makes dihedral angles of 9.89 (3) and 66.06 (5)° with the two phenyl rings, and the two phenyl rings form an angle of 74.05 (5)°. An intramolecular C—H···O hydrogen bond forms a six-membered ring, producing an S(6) ring motif. In the crystal structure, intermolecular N—H···O and C—H···O hydrogen bonds link each molecule to two others, forming an infinite one-dimensional supramolecular structure along the c axis.

Related literature

For related literature, see: Beeam *et al.* (1984); Bonati (1980); Dong & Feng (2006); Dong *et al.* (2008*a*,*b*); Duan *et al.* (2007).



Experimental

Crystal data

$C_{19}H_{19}N_3O_2$
M_r = 321.37
Monoclinic, $P2_1/c$
a = 13.0046 (15) Å
b = 11.4657 (11) Å
c = 11.6874 (12) Å
β = 99.4530 (10)°
V = 1719.0 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 298 (2) K
0.50 × 0.18 × 0.16 mm

Data collection

Brucker SMART 1000 CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.960, T_{max} = 0.987
8480 measured reflections
3028 independent reflections
1756 reflections with $I > 2\sigma(I)$
R_{int} = 0.041

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.140
S = 1.02
3028 reflections
219 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3···O2^{i}	0.86	1.80	2.653 (2)	171
C15—H15···O2^{i}	0.93	2.42	3.200 (3)	141
C19—H19···O2	0.93	2.32	2.900 (3)	120

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

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZL2137).

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supporting information

Acta Cryst. (2008). E**64**, o1794 [doi:10.1107/S1600536808026263]

4-[(Ethoxyimino)(phenyl)methyl]-5-methyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one

Jiu-Si Wang, Yan-Ling Jiang, Wen-Kui Dong, Li Xu and Ai-Ping Kong

S1. Comment

The pyrazole ring is a prominent structural motif found in numerous pharmaceutically active compounds. Due to the easy preparation and rich biological activity, the pyrazole framework plays an essential role in biologically active compounds and therefore represents an interesting template for combinatorial as well as medicinal chemistry (Beeam *et al.*, 1984; Bonati *et al.*, 1980). As an extension of our work (Dong *et al.*, 2006; Duan *et al.*, 2007; Dong *et al.*, 2008*a*; Dong *et al.*, 2008*b*) on the structural characterization of oxime compounds, the title compound, (Fig. 1), is reported here.

The single-crystal structure of the title compound is built up by only the $C_{19}H_{19}N_3O_2$ molecules, in which all bond lengths are in normal ranges. In the title compound, the central pyrazole ring makes dihedral angles of 9.89 (3) and 66.06 (5)° with the two outer benzene rings, and the two outer benzene rings form an angle of 74.05 (5)°. An intramolecular C—H···O hydrogen bond forms a six-membered ring, producing an S(6) ring motif. In the crystal structure, intermolecular N—H···O and C—H···O hydrogen bonds link each molecule to two others, forming an infinite one-dimensional supramolecular structure along the *c* axis (Fig. 2).

S2. Experimental

To a solution of 1-phenyl-3-methyl-4-benzoyl-5-pyrazolon (5 mmol) in warm ethanol (5 ml) was added an ethanol (5 ml) solution of ethoxyamine (10 mmol). After stirring the reaction mixture at 338 K for 6 h, the solvent was removed under reduced pressure and the residue was recrystallized from ethanol to give the title compound. Yield, 68%. mp. 444–445 K. Anal. Calc. for $C_{19}H_{19}N_3O_2$: C, 71.01; H, 5.96; N, 13.08. Found: C, 71.32; H, 5.81; N, 13.15.

Colorless prismatic crystals suitable for single-crystal X-ray diffraction were obtained by recrystallization from ethanol at room temperature.

S3. Refinement

Non-H atoms were refined anisotropically. H atoms were treated as riding atoms with distances C—H = 0.96 (CH_3), C—H = 0.97 (CH_2), or 0.93 Å (CH), N—H = 0.86 Å, and U_{iso}(H) = 1.2 U_{eq}(C) and 1.5 U_{eq}(O).

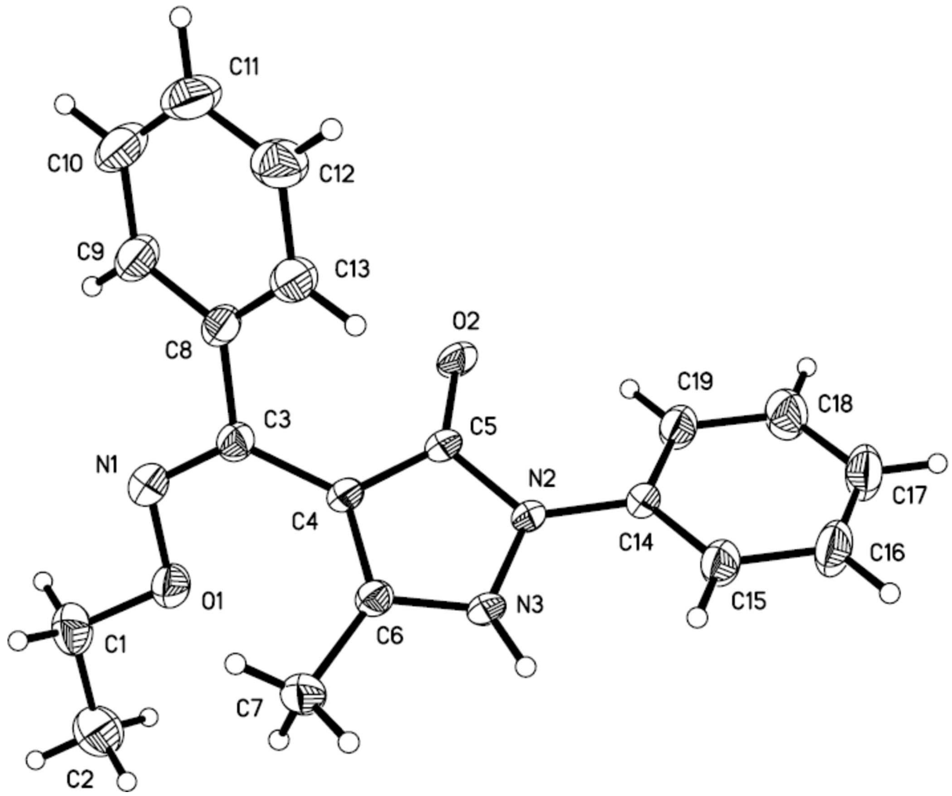

Figure 1

ORTEP representation of the title compound with atom numbering. Displacement ellipsoids for non-hydrogen atoms are drawn at the 30% probability level.

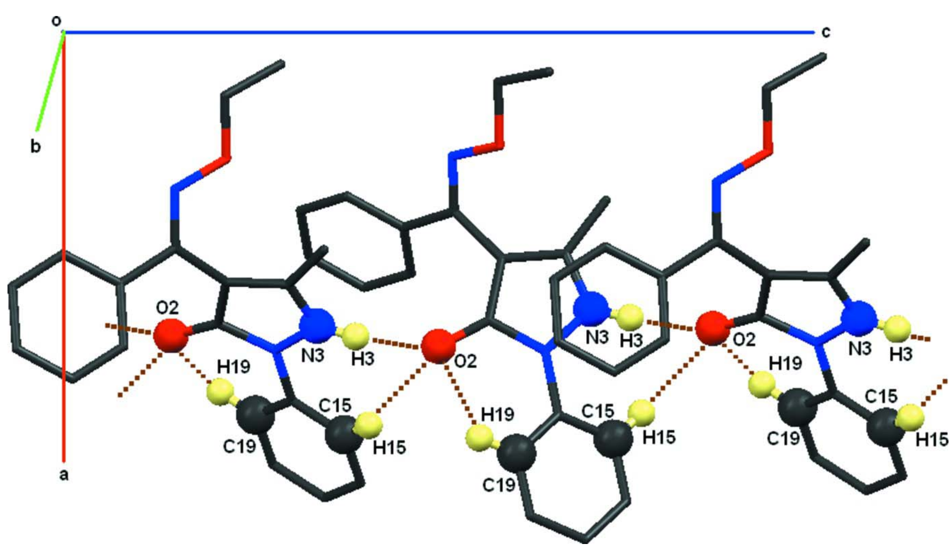

Figure 2

Part of the supramolecular structure of the title compound viewed along the *b* axis. Intra- and intermolecular hydrogen bonds are shown as dashed lines.

4-[(Ethoxyimino)(phenyl)methyl]-5-methyl-2-phenyl-1H-pyrazol-3(2H)-one

Crystal data

$C_{19}H_{19}N_3O_2$
M_r = 321.37
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 13.0046 (15) Å
b = 11.4657 (11) Å
c = 11.6874 (12) Å
β = 99.453 (1)°
V = 1719.0 (3) Å^3
Z = 4
F(000) = 680
D_x = 1.242 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1770 reflections
θ = 2.4–22.5°
μ = 0.08 mm^{-1}
T = 298 K
Prismatic, colorless
0.50 × 0.18 × 0.16 mm

Data collection

Brucker SMART 1000 CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.960, T_{max} = 0.987
8480 measured reflections
3028 independent reflections
1756 reflections with $I > 2\sigma(I)$
R_{int} = 0.041
θ_{max} = 25.0°, θ_{min} = 1.6°
h = −15→15
k = −13→9
l = −13→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.140
S = 1.02
3028 reflections
219 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
$w = 1/[\sigma^2(F_o^2) + (0.0687P)^2 + 0.0591P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ < 0.001
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
N1	0.53222 (14)	0.34824 (17)	−0.06367 (17)	0.0486 (5)
N2	0.84502 (13)	0.23475 (16)	0.16076 (15)	0.0369 (5)
N3	0.78882 (13)	0.29323 (15)	0.23325 (15)	0.0386 (5)
H3	0.8064	0.2996	0.3072	0.046*

O1	0.49758 (11)	0.28139 (15)	0.02320 (14)	0.0546 (5)
O2	0.82634 (12)	0.20296 (15)	−0.03673 (13)	0.0529 (5)
C1	0.38665 (18)	0.2834 (2)	0.0039 (3)	0.0623 (8)
H1A	0.3595	0.2505	−0.0715	0.075*
H1B	0.3617	0.3630	0.0061	0.075*
C2	0.3515 (2)	0.2130 (3)	0.0975 (3)	0.0805 (10)
H2A	0.3776	0.1349	0.0954	0.121*
H2B	0.2767	0.2113	0.0859	0.121*
H2C	0.3774	0.2474	0.1715	0.121*
C3	0.63196 (17)	0.35862 (19)	−0.04638 (19)	0.0395 (6)
C4	0.70377 (15)	0.31546 (19)	0.05486 (18)	0.0363 (5)
C5	0.79385 (17)	0.24753 (19)	0.04860 (19)	0.0371 (5)
C6	0.70309 (16)	0.33834 (19)	0.17079 (19)	0.0360 (5)
C7	0.62708 (17)	0.4013 (2)	0.2301 (2)	0.0496 (7)
H7A	0.5735	0.3484	0.2446	0.074*
H7B	0.5964	0.4640	0.1817	0.074*
H7C	0.6622	0.4323	0.3023	0.074*
C8	0.67571 (18)	0.4228 (2)	−0.1379 (2)	0.0432 (6)
C9	0.6212 (2)	0.4282 (2)	−0.2505 (2)	0.0576 (7)
H9	0.5572	0.3908	−0.2688	0.069*
C10	0.6611 (3)	0.4881 (3)	−0.3347 (3)	0.0755 (9)
H10	0.6238	0.4914	−0.4097	0.091*
C11	0.7555 (3)	0.5432 (3)	−0.3093 (3)	0.0773 (9)
H11	0.7823	0.5832	−0.3671	0.093*
C12	0.8107 (2)	0.5397 (2)	−0.1988 (3)	0.0664 (8)
H12	0.8743	0.5782	−0.1811	0.080*
C13	0.77084 (19)	0.4782 (2)	−0.1138 (2)	0.0516 (7)
H13	0.8089	0.4743	−0.0393	0.062*
C14	0.94564 (15)	0.19033 (19)	0.20265 (19)	0.0358 (5)
C15	0.99788 (18)	0.2239 (2)	0.3093 (2)	0.0517 (7)
H15	0.9678	0.2775	0.3535	0.062*
C16	1.09499 (19)	0.1782 (3)	0.3508 (2)	0.0659 (8)
H16	1.1296	0.2005	0.4235	0.079*
C17	1.1406 (2)	0.1007 (3)	0.2862 (3)	0.0667 (8)
H17	1.2063	0.0706	0.3143	0.080*
C18	1.0891 (2)	0.0678 (2)	0.1801 (3)	0.0647 (8)
H18	1.1203	0.0153	0.1358	0.078*
C19	0.99121 (17)	0.1113 (2)	0.1374 (2)	0.0520 (7)
H19	0.9564	0.0875	0.0652	0.062*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0485 (12)	0.0550 (13)	0.0405 (13)	0.0035 (10)	0.0019 (10)	0.0055 (10)
N2	0.0349 (10)	0.0546 (12)	0.0209 (10)	0.0067 (8)	0.0036 (8)	0.0001 (9)
N3	0.0394 (10)	0.0560 (12)	0.0200 (10)	0.0020 (9)	0.0038 (8)	−0.0052 (9)
O1	0.0391 (9)	0.0719 (12)	0.0507 (11)	−0.0002 (8)	0.0010 (8)	0.0123 (9)
O2	0.0585 (10)	0.0778 (12)	0.0223 (9)	0.0200 (9)	0.0069 (8)	−0.0005 (8)

C1	0.0402 (14)	0.0705 (19)	0.073 (2)	0.0002 (13)	−0.0002 (14)	0.0023 (16)
C2	0.0532 (17)	0.109 (3)	0.080 (2)	−0.0073 (16)	0.0143 (16)	0.007 (2)
C3	0.0398 (13)	0.0446 (14)	0.0328 (13)	0.0031 (10)	0.0020 (10)	−0.0023 (11)
C4	0.0359 (12)	0.0465 (13)	0.0258 (13)	−0.0004 (10)	0.0034 (10)	0.0039 (10)
C5	0.0396 (12)	0.0490 (14)	0.0225 (12)	0.0010 (10)	0.0039 (10)	0.0016 (11)
C6	0.0354 (12)	0.0423 (13)	0.0297 (13)	−0.0027 (10)	0.0036 (10)	0.0004 (10)
C7	0.0479 (14)	0.0618 (16)	0.0400 (15)	0.0050 (12)	0.0098 (12)	−0.0069 (13)
C8	0.0518 (14)	0.0436 (14)	0.0332 (14)	0.0124 (12)	0.0038 (11)	0.0021 (11)
C9	0.0661 (17)	0.0619 (17)	0.0428 (17)	0.0110 (14)	0.0026 (14)	0.0081 (14)
C10	0.096 (2)	0.084 (2)	0.0456 (19)	0.0188 (19)	0.0093 (17)	0.0216 (17)
C11	0.101 (3)	0.076 (2)	0.062 (2)	0.0172 (19)	0.034 (2)	0.0259 (17)
C12	0.0723 (19)	0.0598 (18)	0.072 (2)	0.0014 (15)	0.0254 (17)	0.0119 (16)
C13	0.0570 (16)	0.0532 (16)	0.0447 (16)	0.0036 (13)	0.0088 (13)	0.0033 (13)
C14	0.0325 (12)	0.0446 (13)	0.0293 (13)	0.0011 (10)	0.0025 (10)	0.0048 (11)
C15	0.0437 (14)	0.0661 (17)	0.0423 (16)	0.0060 (12)	−0.0023 (12)	−0.0078 (13)
C16	0.0507 (16)	0.086 (2)	0.0528 (18)	0.0087 (15)	−0.0162 (14)	−0.0103 (16)
C17	0.0473 (15)	0.080 (2)	0.066 (2)	0.0174 (15)	−0.0093 (15)	0.0000 (17)
C18	0.0568 (16)	0.0708 (19)	0.065 (2)	0.0203 (14)	0.0056 (15)	−0.0057 (16)
C19	0.0487 (14)	0.0649 (17)	0.0395 (15)	0.0124 (13)	−0.0011 (12)	−0.0048 (13)

Geometric parameters (Å, º)

N1—C3	1.285 (3)	C8—C13	1.378 (3)
N1—O1	1.404 (2)	C8—C9	1.389 (3)
N2—C5	1.377 (3)	C9—C10	1.371 (4)
N2—N3	1.380 (2)	C9—H9	0.9300
N2—C14	1.415 (3)	C10—C11	1.369 (4)
N3—C6	1.333 (3)	C10—H10	0.9300
N3—H3	0.8600	C11—C12	1.371 (4)
O1—C1	1.423 (3)	C11—H11	0.9300
O2—C5	1.254 (2)	C12—C13	1.386 (3)
C1—C2	1.491 (4)	C12—H12	0.9300
C1—H1A	0.9700	C13—H13	0.9300
C1—H1B	0.9700	C14—C15	1.373 (3)
C2—H2A	0.9600	C14—C19	1.379 (3)
C2—H2B	0.9600	C15—C16	1.380 (3)
C2—H2C	0.9600	C15—H15	0.9300
C3—C4	1.468 (3)	C16—C17	1.363 (3)
C3—C8	1.487 (3)	C16—H16	0.9300
C4—C6	1.382 (3)	C17—C18	1.362 (4)
C4—C5	1.419 (3)	C17—H17	0.9300
C6—C7	1.485 (3)	C18—C19	1.383 (3)
C7—H7A	0.9600	C18—H18	0.9300
C7—H7B	0.9600	C19—H19	0.9300
C7—H7C	0.9600		
C3—N1—O1	111.86 (18)	H7B—C7—H7C	109.5
C5—N2—N3	108.11 (16)	C13—C8—C9	118.3 (2)

C5—N2—C14	130.00 (18)	C13—C8—C3	121.2 (2)
N3—N2—C14	121.03 (17)	C9—C8—C3	120.5 (2)
C6—N3—N2	109.39 (17)	C10—C9—C8	120.5 (3)
C6—N3—H3	125.3	C10—C9—H9	119.7
N2—N3—H3	125.3	C8—C9—H9	119.7
N1—O1—C1	108.28 (17)	C11—C10—C9	120.5 (3)
O1—C1—C2	107.5 (2)	C11—C10—H10	119.7
O1—C1—H1A	110.2	C9—C10—H10	119.7
C2—C1—H1A	110.2	C10—C11—C12	120.2 (3)
O1—C1—H1B	110.2	C10—C11—H11	119.9
C2—C1—H1B	110.2	C12—C11—H11	119.9
H1A—C1—H1B	108.5	C11—C12—C13	119.4 (3)
C1—C2—H2A	109.5	C11—C12—H12	120.3
C1—C2—H2B	109.5	C13—C12—H12	120.3
H2A—C2—H2B	109.5	C8—C13—C12	121.1 (3)
C1—C2—H2C	109.5	C8—C13—H13	119.5
H2A—C2—H2C	109.5	C12—C13—H13	119.5
H2B—C2—H2C	109.5	C15—C14—C19	119.5 (2)
N1—C3—C4	126.0 (2)	C15—C14—N2	120.3 (2)
N1—C3—C8	115.4 (2)	C19—C14—N2	120.1 (2)
C4—C3—C8	118.62 (19)	C14—C15—C16	120.0 (2)
C6—C4—C5	107.05 (19)	C14—C15—H15	120.0
C6—C4—C3	128.4 (2)	C16—C15—H15	120.0
C5—C4—C3	124.40 (19)	C17—C16—C15	120.6 (3)
O2—C5—N2	122.71 (19)	C17—C16—H16	119.7
O2—C5—C4	130.8 (2)	C15—C16—H16	119.7
N2—C5—C4	106.46 (18)	C18—C17—C16	119.4 (2)
N3—C6—C4	108.91 (19)	C18—C17—H17	120.3
N3—C6—C7	119.62 (19)	C16—C17—H17	120.3
C4—C6—C7	131.5 (2)	C17—C18—C19	121.0 (3)
C6—C7—H7A	109.5	C17—C18—H18	119.5
C6—C7—H7B	109.5	C19—C18—H18	119.5
H7A—C7—H7B	109.5	C14—C19—C18	119.4 (2)
C6—C7—H7C	109.5	C14—C19—H19	120.3
H7A—C7—H7C	109.5	C18—C19—H19	120.3

C5—N2—N3—C6	2.1 (2)	N1—C3—C8—C13	155.2 (2)
C14—N2—N3—C6	172.45 (18)	C4—C3—C8—C13	−23.6 (3)
C3—N1—O1—C1	174.5 (2)	N1—C3—C8—C9	−24.8 (3)
N1—O1—C1—C2	−179.5 (2)	C4—C3—C8—C9	156.4 (2)
O1—N1—C3—C4	−4.5 (3)	C13—C8—C9—C10	−0.6 (4)
O1—N1—C3—C8	176.79 (17)	C3—C8—C9—C10	179.4 (2)
N1—C3—C4—C6	−55.2 (4)	C8—C9—C10—C11	0.3 (4)
C8—C3—C4—C6	123.5 (2)	C9—C10—C11—C12	−0.5 (4)
N1—C3—C4—C5	130.1 (2)	C10—C11—C12—C13	1.1 (4)
C8—C3—C4—C5	−51.3 (3)	C9—C8—C13—C12	1.1 (3)
N3—N2—C5—O2	−179.5 (2)	C3—C8—C13—C12	−178.9 (2)
C14—N2—C5—O2	11.3 (4)	C11—C12—C13—C8	−1.4 (4)

N3—N2—C5—C4	−0.3 (2)	C5—N2—C14—C15	155.0 (2)
C14—N2—C5—C4	−169.5 (2)	N3—N2—C14—C15	−13.0 (3)
C6—C4—C5—O2	177.7 (2)	C5—N2—C14—C19	−26.1 (3)
C3—C4—C5—O2	−6.7 (4)	N3—N2—C14—C19	165.9 (2)
C6—C4—C5—N2	−1.4 (2)	C19—C14—C15—C16	−0.4 (4)
C3—C4—C5—N2	174.25 (19)	N2—C14—C15—C16	178.5 (2)
N2—N3—C6—C4	−3.0 (2)	C14—C15—C16—C17	0.9 (4)
N2—N3—C6—C7	177.33 (17)	C15—C16—C17—C18	−0.5 (4)
C5—C4—C6—N3	2.8 (2)	C16—C17—C18—C19	−0.3 (4)
C3—C4—C6—N3	−172.7 (2)	C15—C14—C19—C18	−0.4 (4)
C5—C4—C6—C7	−177.7 (2)	N2—C14—C19—C18	−179.3 (2)
C3—C4—C6—C7	6.9 (4)	C17—C18—C19—C14	0.8 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3···O2^{i}	0.86	1.80	2.653 (2)	171
C15—H15···O2^{i}	0.93	2.42	3.200 (3)	141
C19—H19···O2	0.93	2.32	2.900 (3)	120

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