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Ethyl 1-phenyl-1,4-dihydroindeno[1,2-*c*]pyrazole-3-carboxylate

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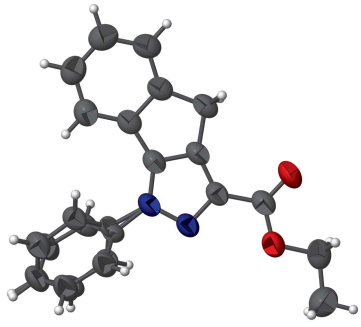
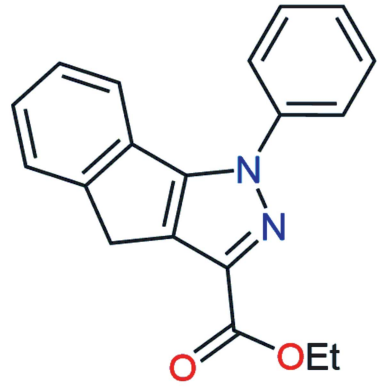
Structural data: full structural data are available from iucrdata.iucr.org

The non-H atoms of the title molecule, $C_{19}H_{16}N_2O_2$, are almost coplanar (r.m.s. deviation = 0.019 Å), apart from the phenyl group, which is disordered with two components of almost equal occupancy: the dihedral angle between them is 78.9 (3)°. In the crystal, weak C—H···N hydrogen bonds link the molecules into [001] chains and aromatic π–π stacking interactions [shortest centroid–centroid separation = 3.747 (2) Å] form columns parallel to the *c*-axis direction.

3D view



Chemical scheme



Structure description

Pyrazole-3-carboylates can be synthesized using various efficient procedures (*e.g.* Khidre *et al.*, 2016; Radwan *et al.*, 2014). As part of our studies in this area, we now describe the synthesis and structure of the title compound.

The title molecule is almost planar (r.m.s. deviation = 0.017 Å) apart from the phenyl ring (Fig. 1), which is disordered with two components of almost equal occupancy [50.5 (4)% and 49.5 (4)%]. The components of the disordered phenyl rings are twisted by 54.6 (2) and 46.9 (2)° away from the least-squares plane of the rest of the molecule and the angle between the disorder components is 78.9 (3)°.

In the crystal, weak C—H···N hydrogen bonds (Table 1) link the molecules into [001] chains and aromatic π–π stacking interactions [shortest centroid–centroid separation = 3.747 (2) Å] generate columns parallel to the *c*-axis direction (Fig. 2).

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

D—H···A	D—H	H···A	D···A	D—H···A
C19—H19···N2^{i}	0.93	2.56	3.489 (5)	178

Symmetry code: (i) $x, y, z-1$.

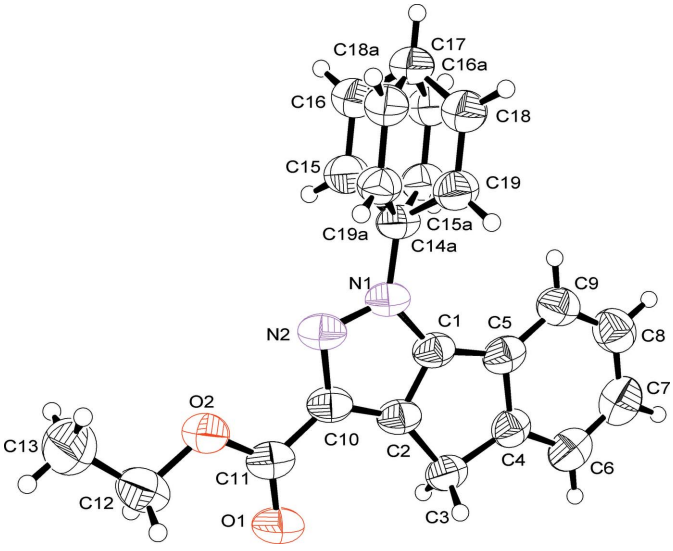


Figure 1
An *ORTEP* representation of the title molecule showing 50% probability displacement ellipsoids.

Synthesis and crystallization

Ethyl 2-oxo-2-(1-oxo-2,3-dihydro-1*H*-inden-2-yl)acetate and phenyl hydrazine hydrochloride were refluxed in ethanol solution for 4 h. The mixture was left to cool and the solid obtained was filtered, washed (ethanol) and dried. Recrystallization from dimethylformamide solution provided pale-yellow crystals.

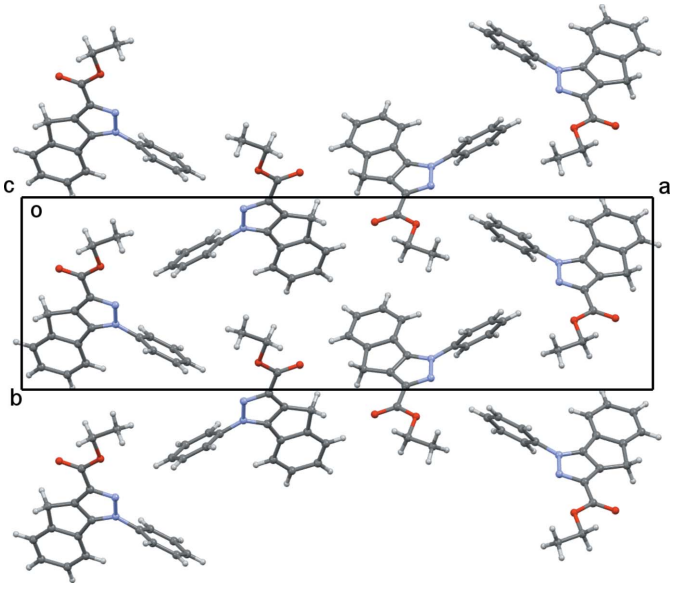


Figure 2
Crystal packing showing one component of the disordered ring in the molecules.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{19}H_{16}N_2O_2$
M_r	304.34
Crystal system, space group	Orthorhombic, $Pna2_1$
Temperature (K)	296
a, b, c (Å)	32.387 (3), 9.8559 (15), 5.0043 (8)
V (Å^3)	1597.4 (4)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.08
Crystal size (mm)	0.38 × 0.13 × 0.05
Data collection	
Diffractometer	Agilent SuperNova, Dual, Cu at zero, Atlas
Absorption correction	Gaussian (*CrysAlis PRO*; Agilent, 2014)
T_{min}, T_{max}	0.991, 0.998
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	10362, 3460, 2661
R_{int}	0.054
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.696
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.055, 0.149, 1.05
No. of reflections	3460
No. of parameters	240
No. of restraints	37
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.18, −0.17

Computer programs: *CrysAlis PRO* (Agilent, 2014), *SHELXS2013* (Sheldrick, 2008), *SHELXL2013* (Sheldrick, 2015), *ORTEP-3 for Windows* and *WinGX* (Farrugia, 2012) and *CHEMDRAW Ultra* (Cambridge Soft, 2001).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The phenyl ring was modelled with two disorder components with occupancies of 49.5 (4) and 50.5 (4)%.

Funding information

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References

Agilent (2014). *CrysAlis PRO*. Agilent Technologies, Yarnton, England.
Cambridge Soft. *CHEMDRAW Ultra*. Cambridge Soft Corporation, Cambridge, Massachusetts, USA.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Khidre, R. E., Abdel-Wahab, B. F., Farahat, A. A. & Mohamed, H. A. (2016). *J. Heterocycl. Chem.* **53**, 13–31.
Radwan, A. A., Ghorab, M. A., Alsaid, M. S. & Alanazi, F. K. (2014). *Acta Pharm.* **64**, 335–344.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.

full crystallographic data

IUCrData (2018). **3**, x171840 [https://doi.org/10.1107/S2414314617018405]

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Crystal data

$C_{19}H_{16}N_2O_2$
M_r = 304.34
Orthorhombic, $Pna2_1$
a = 32.387 (3) Å
b = 9.8559 (15) Å
c = 5.0043 (8) Å
V = 1597.4 (4) Å^3
Z = 4
$F(000)$ = 640
D_x = 1.265 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1394 reflections
θ = 4.1–25.3°
μ = 0.08 mm^{-1}
T = 296 K
Block, pale yellow
0.38 × 0.13 × 0.05 mm

Data collection

Agilent SuperNova, Dual, Cu at zero, Atlas diffractometer
ω scans
Absorption correction: gaussian (CrysAlisPro; Agilent, 2014)
T_{min} = 0.991, T_{max} = 0.998
10362 measured reflections
3460 independent reflections
2661 reflections with $I > 2\sigma(I)$
R_{int} = 0.054
θ_{max} = 29.6°, θ_{min} = 2.2°
h = −44→39
k = −11→12
l = −5→6

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.149
S = 1.05
3460 reflections
240 parameters
37 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0805P)^2 + 0.1345P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

Special details

Experimental. Version 1.171.37.35g (release 09-12-2014 CrysAlis171 .NET) (compiled Dec 9 2014,15:38:47) Numerical absorption correction based on gaussian integration over a multifaceted crystal model Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.
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.

Refinement. All hydrogen atoms were placed in calculated positions (C—H = 0.93–0.97 Å) and using a riding model with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C). The methyl groups were allowed to rotate, but not to tip, to best fit the electron density.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.38927 (8)	0.1784 (3)	0.9703 (6)	0.0522 (6)	
C2	0.41595 (7)	0.0900 (3)	1.0968 (7)	0.0532 (6)	
C3	0.45872 (8)	0.1067 (3)	0.9863 (7)	0.0593 (7)	
H3A	0.4683	0.0241	0.9010	0.071*	
H3B	0.4782	0.1330	1.1245	0.071*	
C4	0.45247 (9)	0.2195 (3)	0.7847 (7)	0.0580 (7)	
C5	0.41075 (8)	0.2621 (3)	0.7730 (7)	0.0552 (6)	
C6	0.48178 (10)	0.2807 (4)	0.6220 (8)	0.0705 (8)	
H6	0.5093	0.2542	0.6295	0.085*	
C7	0.46916 (13)	0.3826 (4)	0.4476 (8)	0.0779 (10)	
H7	0.4885	0.4246	0.3378	0.093*	
C8	0.42798 (12)	0.4225 (4)	0.4353 (8)	0.0748 (9)	
H8	0.4202	0.4904	0.3164	0.090*	
C9	0.39854 (10)	0.3633 (3)	0.5956 (7)	0.0648 (8)	
H9	0.3711	0.3903	0.5857	0.078*	
C10	0.39149 (8)	0.0192 (3)	1.2805 (7)	0.0537 (6)	
C11	0.40527 (8)	−0.0897 (3)	1.4630 (7)	0.0588 (7)	
C12	0.38793 (12)	−0.2480 (4)	1.7995 (8)	0.0749 (9)	
H12A	0.4093	−0.2157	1.9192	0.090*	
H12B	0.3987	−0.3246	1.6995	0.090*	
C13	0.35063 (14)	−0.2894 (5)	1.9549 (11)	0.0985 (13)	
H13A	0.3399	−0.2123	2.0491	0.148*	
H13B	0.3581	−0.3588	2.0807	0.148*	
H13C	0.3300	−0.3237	1.8349	0.148*	
C14	0.31175 (11)	0.2208 (5)	1.0428 (9)	0.050 (3)	0.505 (4)
C15	0.28917 (13)	0.2838 (5)	1.2438 (7)	0.0645 (16)	0.505 (4)
H15	0.2983	0.2801	1.4197	0.077*	0.505 (4)
C16	0.25288 (12)	0.3522 (5)	1.1818 (8)	0.075 (2)	0.505 (4)
H16	0.2378	0.3943	1.3163	0.090*	0.505 (4)
C17	0.23917 (12)	0.3576 (6)	0.9188 (9)	0.074 (4)	0.505 (4)
H17	0.2149	0.4033	0.8774	0.089*	0.505 (4)
C18	0.26174 (13)	0.2946 (6)	0.7178 (7)	0.083 (2)	0.505 (4)
H18	0.2526	0.2982	0.5419	0.099*	0.505 (4)
C19	0.29804 (13)	0.2262 (5)	0.7798 (8)	0.0689 (18)	0.505 (4)
H19	0.3131	0.1840	0.6453	0.083*	0.505 (4)
C14A	0.31445 (9)	0.2308 (4)	1.0024 (12)	0.045 (2)	0.495 (4)
C15A	0.31241 (10)	0.3716 (3)	0.9934 (12)	0.0573 (14)	0.495 (4)
H15A	0.3363	0.4229	1.0129	0.069*	0.495 (4)
C16A	0.27464 (13)	0.4357 (3)	0.9553 (12)	0.0685 (17)	0.495 (4)
H16A	0.2733	0.5299	0.9493	0.082*	0.495 (4)
C17A	0.23890 (10)	0.3590 (4)	0.9262 (12)	0.072 (4)	0.495 (4)

H17A	0.2136	0.4019	0.9007	0.086*	0.495 (4)
C18A	0.24094 (9)	0.2182 (4)	0.9351 (11)	0.0652 (17)	0.495 (4)
H18A	0.2170	0.1669	0.9156	0.078*	0.495 (4)
C19A	0.27872 (12)	0.1541 (3)	0.9732 (11)	0.0565 (14)	0.495 (4)
H19A	0.2801	0.0599	0.9792	0.068*	0.495 (4)
N1	0.35113 (6)	0.1593 (2)	1.0792 (6)	0.0543 (6)	
N2	0.35193 (6)	0.0611 (2)	1.2712 (6)	0.0575 (6)	
O1	0.44040 (7)	−0.1290 (3)	1.4686 (7)	0.0823 (8)	
O2	0.37529 (6)	−0.1400 (2)	1.6176 (5)	0.0663 (6)	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0458 (13)	0.0540 (14)	0.0569 (15)	0.0039 (10)	−0.0074 (12)	−0.0135 (12)
C2	0.0431 (12)	0.0557 (14)	0.0607 (15)	0.0047 (10)	−0.0058 (12)	−0.0127 (13)
C3	0.0469 (13)	0.0653 (16)	0.0658 (17)	0.0072 (11)	−0.0032 (13)	−0.0138 (15)
C4	0.0532 (14)	0.0636 (17)	0.0571 (15)	0.0008 (11)	−0.0005 (13)	−0.0135 (14)
C5	0.0540 (14)	0.0554 (14)	0.0561 (15)	0.0024 (11)	−0.0036 (13)	−0.0156 (13)
C6	0.0594 (16)	0.080 (2)	0.072 (2)	−0.0042 (15)	0.0023 (15)	−0.0102 (18)
C7	0.087 (2)	0.075 (2)	0.072 (2)	−0.0097 (17)	0.0097 (19)	−0.0071 (17)
C8	0.093 (2)	0.0641 (19)	0.068 (2)	−0.0001 (16)	−0.0030 (18)	0.0000 (16)
C9	0.0684 (18)	0.0589 (16)	0.0669 (18)	0.0061 (13)	−0.0076 (16)	−0.0089 (15)
C10	0.0448 (12)	0.0489 (13)	0.0674 (16)	0.0024 (10)	−0.0085 (12)	−0.0085 (13)
C11	0.0498 (14)	0.0550 (15)	0.0717 (19)	0.0048 (11)	−0.0071 (14)	−0.0122 (14)
C12	0.086 (2)	0.0666 (19)	0.072 (2)	0.0158 (16)	−0.0073 (19)	−0.0028 (17)
C13	0.099 (3)	0.100 (3)	0.096 (3)	−0.005 (2)	−0.002 (3)	0.018 (3)
C14	0.039 (4)	0.055 (5)	0.056 (5)	0.003 (3)	0.005 (3)	0.001 (4)
C15	0.058 (3)	0.084 (4)	0.051 (3)	0.015 (3)	0.002 (3)	−0.001 (3)
C16	0.064 (4)	0.099 (5)	0.062 (4)	0.030 (3)	0.007 (3)	−0.001 (3)
C17	0.054 (7)	0.098 (7)	0.069 (7)	0.023 (7)	−0.002 (7)	0.000 (7)
C18	0.062 (4)	0.130 (7)	0.057 (4)	0.025 (4)	−0.010 (3)	0.009 (4)
C19	0.053 (3)	0.091 (5)	0.063 (4)	0.012 (3)	0.000 (3)	−0.019 (4)
C14A	0.040 (4)	0.052 (5)	0.043 (3)	0.004 (3)	−0.005 (3)	−0.001 (3)
C15A	0.052 (3)	0.050 (3)	0.070 (4)	0.003 (2)	0.001 (3)	−0.007 (3)
C16A	0.063 (3)	0.062 (3)	0.081 (4)	0.014 (3)	−0.002 (3)	−0.005 (3)
C17A	0.050 (6)	0.098 (7)	0.067 (7)	0.026 (6)	−0.004 (6)	0.008 (7)
C18A	0.041 (3)	0.093 (5)	0.062 (4)	0.000 (3)	−0.006 (2)	0.000 (3)
C19A	0.049 (3)	0.061 (3)	0.060 (3)	0.000 (2)	−0.005 (3)	−0.001 (3)
N1	0.0425 (10)	0.0509 (12)	0.0696 (15)	0.0043 (8)	−0.0064 (10)	−0.0064 (11)
N2	0.0440 (11)	0.0502 (12)	0.0782 (16)	0.0002 (8)	−0.0075 (12)	−0.0025 (12)
O1	0.0547 (12)	0.0882 (16)	0.104 (2)	0.0218 (10)	−0.0061 (13)	0.0110 (15)
O2	0.0587 (11)	0.0633 (12)	0.0768 (14)	0.0092 (9)	−0.0048 (11)	0.0027 (11)

Geometric parameters (Å, º)

C1—N1	1.363 (4)	C13—H13B	0.9600
C1—C2	1.380 (4)	C13—H13C	0.9600
C1—C5	1.463 (5)	C14—C15	1.3900

C2—C10	1.400 (5)	C14—C19	1.3900
C2—C3	1.501 (4)	C14—N1	1.424 (3)
C3—C4	1.515 (5)	C15—C16	1.3900
C3—H3A	0.9700	C15—H15	0.9300
C3—H3B	0.9700	C16—C17	1.3900
C4—C6	1.388 (5)	C16—H16	0.9300
C4—C5	1.416 (4)	C17—C18	1.3900
C5—C9	1.393 (5)	C17—H17	0.9300
C6—C7	1.392 (6)	C18—C19	1.3900
C6—H6	0.9300	C18—H18	0.9300
C7—C8	1.392 (5)	C19—H19	0.9300
C7—H7	0.9300	C14A—C15A	1.3900
C8—C9	1.376 (5)	C14A—C19A	1.3900
C8—H8	0.9300	C14A—N1	1.434 (4)
C9—H9	0.9300	C15A—C16A	1.3900
C10—N2	1.347 (3)	C15A—H15A	0.9300
C10—C11	1.478 (4)	C16A—C17A	1.3900
C11—O1	1.202 (3)	C16A—H16A	0.9300
C11—O2	1.337 (4)	C17A—C18A	1.3900
C12—O2	1.459 (4)	C17A—H17A	0.9300
C12—C13	1.494 (6)	C18A—C19A	1.3900
C12—H12A	0.9700	C18A—H18A	0.9300
C12—H12B	0.9700	C19A—H19A	0.9300
C13—H13A	0.9600	N1—N2	1.364 (4)

N1—C1—C2	107.3 (3)	H13A—C13—H13C	109.5
N1—C1—C5	141.1 (2)	H13B—C13—H13C	109.5
C2—C1—C5	111.6 (2)	C15—C14—C19	120.0
C1—C2—C10	105.2 (2)	C15—C14—N1	124.7 (3)
C1—C2—C3	109.8 (3)	C19—C14—N1	115.1 (3)
C10—C2—C3	145.0 (3)	C16—C15—C14	120.0
C2—C3—C4	101.7 (2)	C16—C15—H15	120.0
C2—C3—H3A	111.4	C14—C15—H15	120.0
C4—C3—H3A	111.4	C15—C16—C17	120.0
C2—C3—H3B	111.4	C15—C16—H16	120.0
C4—C3—H3B	111.4	C17—C16—H16	120.0
H3A—C3—H3B	109.3	C16—C17—C18	120.0
C6—C4—C5	120.0 (3)	C16—C17—H17	120.0
C6—C4—C3	128.2 (3)	C18—C17—H17	120.0
C5—C4—C3	111.9 (3)	C19—C18—C17	120.0
C9—C5—C4	120.6 (3)	C19—C18—H18	120.0
C9—C5—C1	134.4 (3)	C17—C18—H18	120.0
C4—C5—C1	105.0 (3)	C18—C19—C14	120.0
C4—C6—C7	118.7 (3)	C18—C19—H19	120.0
C4—C6—H6	120.6	C14—C19—H19	120.0
C7—C6—H6	120.6	C15A—C14A—C19A	120.0
C8—C7—C6	120.8 (3)	C15A—C14A—N1	122.6 (2)
C8—C7—H7	119.6	C19A—C14A—N1	116.8 (3)

C6—C7—H7	119.6	C14A—C15A—C16A	120.0
C9—C8—C7	121.2 (3)	C14A—C15A—H15A	120.0
C9—C8—H8	119.4	C16A—C15A—H15A	120.0
C7—C8—H8	119.4	C17A—C16A—C15A	120.0
C8—C9—C5	118.6 (3)	C17A—C16A—H16A	120.0
C8—C9—H9	120.7	C15A—C16A—H16A	120.0
C5—C9—H9	120.7	C16A—C17A—C18A	120.0
N2—C10—C2	111.3 (3)	C16A—C17A—H17A	120.0
N2—C10—C11	122.1 (3)	C18A—C17A—H17A	120.0
C2—C10—C11	126.7 (2)	C19A—C18A—C17A	120.0
O1—C11—O2	123.6 (3)	C19A—C18A—H18A	120.0
O1—C11—C10	122.3 (3)	C17A—C18A—H18A	120.0
O2—C11—C10	114.1 (2)	C18A—C19A—C14A	120.0
O2—C12—C13	107.3 (3)	C18A—C19A—H19A	120.0
O2—C12—H12A	110.3	C14A—C19A—H19A	120.0
C13—C12—H12A	110.3	C1—N1—N2	111.2 (2)
O2—C12—H12B	110.3	C1—N1—C14	134.6 (3)
C13—C12—H12B	110.3	N2—N1—C14	114.1 (3)
H12A—C12—H12B	108.5	C1—N1—C14A	125.2 (3)
C12—C13—H13A	109.5	N2—N1—C14A	123.6 (3)
C12—C13—H13B	109.5	C10—N2—N1	105.1 (2)
H13A—C13—H13B	109.5	C11—O2—C12	115.3 (2)
C12—C13—H13C	109.5		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C19—H19···N2^{i}	0.93	2.56	3.489 (5)	178

Symmetry code: (i) *x*, *y*, *z*−1.