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1-Benzoyl-3,5-diphenyl-4,5-dihydro-1*H*-pyrazole

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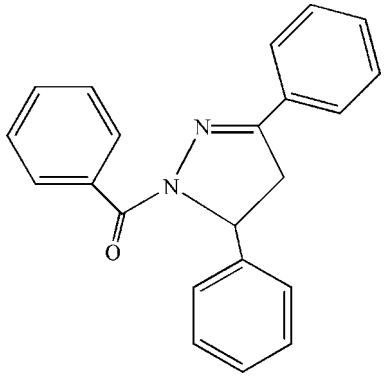
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.035; wR factor = 0.081; data-to-parameter ratio = 7.1.

In the title compound, $C_{22}H_{18}N_2O$, the pyrazole ring is almost planar (r.m.s. deviation = 0.0098 Å) and its mean plane makes dihedral angles of 62.2 (2), 87.2 (2) and 8.0 (2)° with the phenyl and benzoyl rings, respectively. The crystal packing is stabilized by π–π stacking interactions [centroid–centroid distance = 3.658 (2) Å] and weak intermolecular C—H···O hydrogen bonds.

Related literature

For the coordination properties of aroylhydrazones, see: Egli *et al.* (2006); Ge (2006); Chopra *et al.* (2006). For related structures, see: Seebacher *et al.* (2003); Ge (2006); Jian & Wang (2006); Fun *et al.* (2010). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{22}H_{18}N_2O$ M_r = 326.38
Orthorhombic, $Pca2_1$
a = 20.276 (6) Å
b = 5.7859 (17) Å
c = 14.786 (4) Å
V = 1734.5 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 298 K
0.18 × 0.16 × 0.12 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2006)
T_{min} = 0.986, T_{max} = 0.991
8497 measured reflections
1601 independent reflections
1100 reflections with $I > 2\sigma(I)$
R_{int} = 0.050

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.081
S = 1.09
1601 reflections
227 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.13 e Å^{-3}
$\Delta\rho_{min}$ = −0.10 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C21—H21···O1^{i}	0.93	2.72	3.399 (5)	131
C22—H22···O1^{i}	0.93	3.00	3.540 (4)	119
C10—H10···O1^{ii}	0.93	2.87	3.793 (5)	174

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

Data collection: *SMART* (Bruker, 1996); cell refinement: *SAINT* (Bruker, 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: ZQ2084).

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supporting information

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1-Benzoyl-3,5-diphenyl-4,5-dihydro-1*H*-pyrazole

Chang-Zheng Zheng, Liang Wang and Juan Liu

S1. Comment

The chemistry of aroylhydrazones continues to attract much attention due to their coordination ability to metal ions (Egli *et al.*, 2006; Ge, 2006) and their biological activity (Egli *et al.*, 2006; Chopra *et al.*, 2006). As an extension of work on the structural characterization of aroylhydrazone derivatives, the title compound,$C_{22}H_{18}N_2O$, was successfully synthesized and its crystal structure is reported here.

In the title complex, $C_{22}H_{18}N_2O$, all bond lengths and angles are normal (Allen *et al.*, 1987). The pyrazole ring is planar (rms deviation = 0.0098 Å) and its mean plane makes dihedral angles of 62.2 (1), 87.2 (1) and 8.0 (2)° with the benzene rings C2-C7, C9-C14 and C17-C22, respectively (Fig. 1). The crystal packing is stabilized by π-π stacking interactions between the pyrazole ring and one benzene ring with a centroid-centroid separation of 3.658 (2) Å and by weak intermolecular C—H···O hydrogen bonds (Fig.2; Table 1).

S2. Experimental

A methanol solution (10 ml) of *N*′-(*E*)-(benzylidene acetophenone phenmethyl acylhydrazone) (0.25 mmol,0.082 g) was mixed with a DMF solution (5 ml). The mixture was stirred at 298 K for 2 h. and then filtered. A colorless precipitate was produced after about 20 days. A DMF amount (5 ml) was used to dissolve the precipitate at 330 K. Colorless block-shaped crystals of the title complex were obtained after one month (yield 30%).

S3. Refinement

H atoms were placed in calculated positions and refined as riding with the following constraints: C-H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C) for aromatic H atoms, C-H = 0.97 Å and U_{iso}(H) = 1.2U_{eq}(C) for methylene H atoms, and C-H = 0.98 Å and U_{iso}(H) = 1.2U_{eq}(C) for methine H atoms. As the structure has no anomalous scatterer, the Friedel-pair reflections were merged.

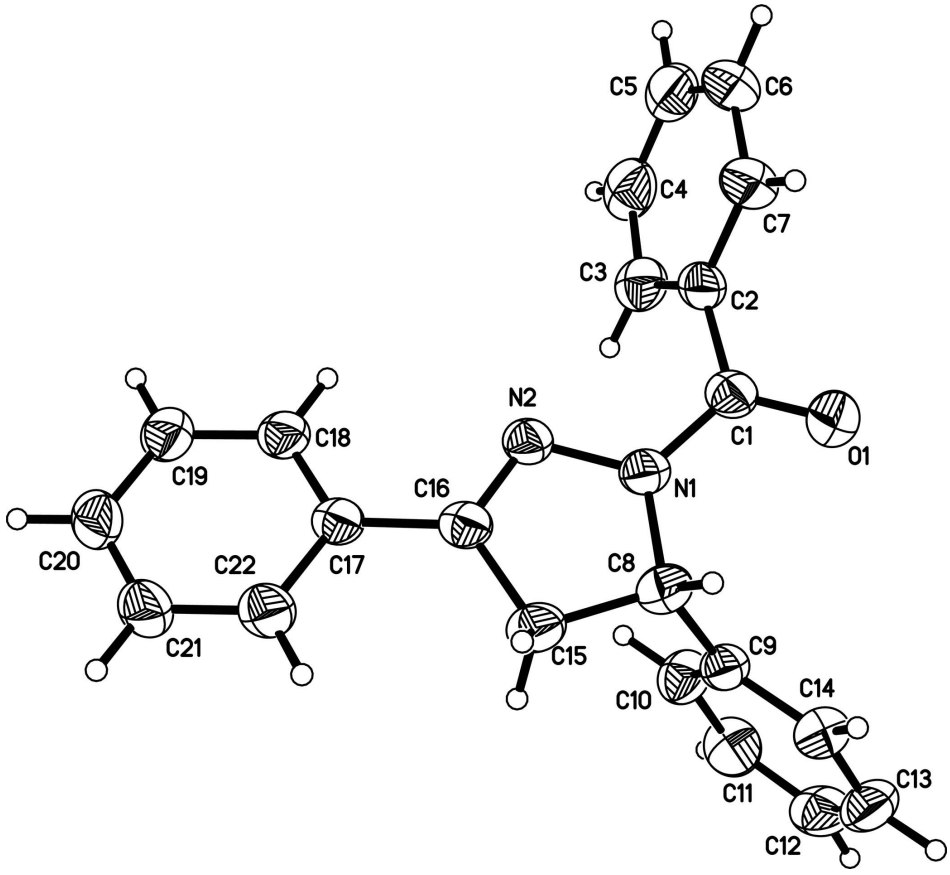

Figure 1

The molecular structure of the title compound, showing displacement ellipsoids drawn at the 30% probability level. H atoms have been omitted for clarity.

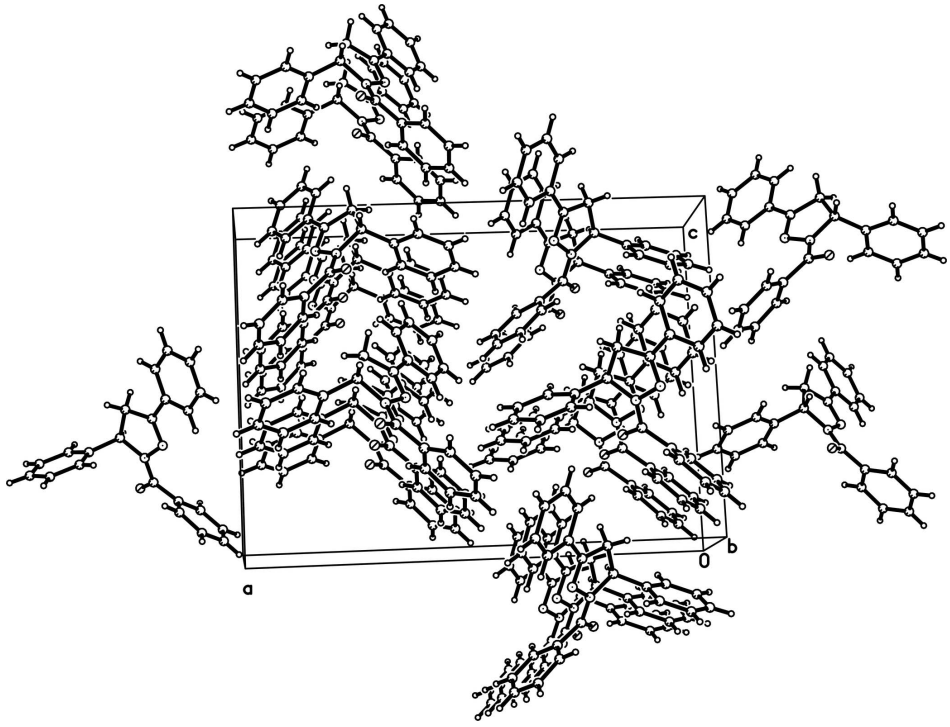

Figure 2

The crystal packing of the title compound.

1-Benzoyl-3,5-diphenyl-4,5-dihydro-1*H*-pyrazole

Crystal data

$C_{22}H_{18}N_2O$
$M_r = 326.38$
Orthorhombic, $Pca2_1$
Hall symbol: P 2c -2ac
$a = 20.276$ (6) Å
$b = 5.7859$ (17) Å
$c = 14.786$ (4) Å
$V = 1734.5$ (9) Å^3
$Z = 4$
$F(000) = 688$
$D_x = 1.250$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1072 reflections
$\theta = 2.4$–17.6°
$\mu = 0.08$ mm^{-1}
$T = 298$ K
Block, colorless
0.18 × 0.16 × 0.12 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2006)
$T_{min} = 0.986$, $T_{max} = 0.991$
8497 measured reflections
1601 independent reflections
1100 reflections with $I > 2\sigma(I)$
$R_{int} = 0.050$
$\theta_{max} = 25.1°$, $\theta_{min} = 2.0°$
$h = -24 \rightarrow 23$
$k = -6 \rightarrow 6$
$l = -17 \rightarrow 13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.081$
$S = 1.09$
1601 reflections
227 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
$w = 1/[\sigma^2(F_o^2) + (0.0335P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.13$ e Å^{-3}
$\Delta\rho_{min} = -0.10$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008)
Extinction coefficient: 0.0113 (15)

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}
O1	0.27196 (11)	0.5466 (4)	0.77340 (17)	0.0769 (7)
N1	0.28823 (12)	0.8795 (5)	0.84788 (17)	0.0617 (7)
N2	0.33000 (12)	1.0610 (4)	0.87296 (18)	0.0587 (7)
C1	0.30374 (16)	0.7269 (6)	0.7810 (2)	0.0607 (8)
C2	0.35797 (16)	0.7877 (6)	0.7181 (2)	0.0602 (8)
C3	0.36177 (18)	0.9982 (7)	0.6747 (3)	0.0742 (10)
H3	0.3325	1.1160	0.6895	0.089*
C4	0.4093 (2)	1.0336 (9)	0.6092 (3)	0.0904 (12)
H4	0.4105	1.1731	0.5780	0.109*
C5	0.4545 (2)	0.8659 (11)	0.5897 (3)	0.1057 (16)
H5	0.4870	0.8929	0.5465	0.127*
C6	0.4521 (2)	0.6593 (10)	0.6336 (3)	0.1046 (16)
H6	0.4833	0.5458	0.6209	0.125*
C7	0.40374 (18)	0.6183 (7)	0.6965 (3)	0.0840 (11)
H7	0.4016	0.4752	0.7250	0.101*
C8	0.23482 (15)	0.8362 (6)	0.9137 (2)	0.0630 (9)
H8	0.2392	0.6799	0.9386	0.076*
C9	0.16756 (15)	0.8651 (6)	0.8719 (2)	0.0566 (8)
C10	0.15150 (17)	1.0621 (6)	0.8242 (3)	0.0719 (10)
H10	0.1833	1.1755	0.8150	0.086*
C11	0.0887 (2)	1.0930 (7)	0.7897 (3)	0.0838 (11)
H11	0.0788	1.2254	0.7568	0.101*
C12	0.04134 (19)	0.9293 (9)	0.8040 (3)	0.0874 (12)

H12	−0.0014	0.9531	0.7830	0.105*
C13	0.05668 (19)	0.7314 (8)	0.8490 (3)	0.0860 (12)
H13	0.0248	0.6176	0.8568	0.103*
C14	0.11969 (17)	0.6987 (6)	0.8833 (3)	0.0726 (10)
H14	0.1297	0.5632	0.9142	0.087*
C15	0.25078 (16)	1.0154 (6)	0.9873 (2)	0.0690 (9)
H15A	0.2141	1.1206	0.9964	0.083*
H15B	0.2612	0.9407	1.0443	0.083*
C16	0.30982 (15)	1.1402 (5)	0.9498 (2)	0.0572 (8)
C17	0.34201 (15)	1.3367 (6)	0.9937 (2)	0.0578 (8)
C18	0.39084 (15)	1.4622 (6)	0.9502 (3)	0.0655 (9)
H18	0.4046	1.4170	0.8929	0.079*
C19	0.41933 (17)	1.6525 (6)	0.9903 (3)	0.0745 (10)
H19	0.4518	1.7353	0.9598	0.089*
C20	0.39988 (19)	1.7206 (7)	1.0755 (3)	0.0785 (11)
H20	0.4191	1.8490	1.1027	0.094*
C21	0.3520 (2)	1.5976 (7)	1.1199 (3)	0.0802 (11)
H21	0.3391	1.6421	1.1777	0.096*
C22	0.32291 (17)	1.4088 (6)	1.0798 (2)	0.0718 (10)
H22	0.2901	1.3283	1.1104	0.086*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0671 (16)	0.0745 (15)	0.0890 (18)	−0.0040 (13)	−0.0021 (13)	−0.0112 (14)
N1	0.0474 (15)	0.0756 (18)	0.0622 (18)	0.0008 (14)	0.0018 (13)	−0.0107 (15)
N2	0.0485 (14)	0.0682 (18)	0.0594 (17)	0.0074 (14)	−0.0021 (13)	−0.0077 (15)
C1	0.049 (2)	0.070 (2)	0.062 (2)	0.0125 (18)	−0.0095 (17)	−0.008 (2)
C2	0.056 (2)	0.074 (2)	0.051 (2)	0.0028 (18)	−0.0054 (16)	−0.0150 (19)
C3	0.066 (2)	0.094 (3)	0.063 (2)	−0.002 (2)	−0.0115 (19)	−0.007 (2)
C4	0.094 (3)	0.116 (3)	0.062 (2)	−0.027 (3)	−0.002 (2)	−0.010 (2)
C5	0.090 (3)	0.149 (4)	0.077 (3)	−0.039 (4)	0.024 (3)	−0.051 (3)
C6	0.084 (3)	0.121 (4)	0.109 (4)	−0.002 (3)	0.027 (3)	−0.055 (3)
C7	0.073 (3)	0.095 (3)	0.083 (3)	0.004 (2)	0.012 (2)	−0.024 (2)
C8	0.052 (2)	0.073 (2)	0.064 (2)	0.0006 (17)	0.0000 (16)	0.0051 (18)
C9	0.0491 (17)	0.064 (2)	0.057 (2)	−0.0009 (16)	0.0016 (15)	−0.0036 (18)
C10	0.062 (2)	0.075 (2)	0.079 (2)	−0.0017 (19)	−0.0084 (18)	0.001 (2)
C11	0.078 (3)	0.091 (3)	0.083 (3)	0.015 (2)	−0.020 (2)	−0.002 (2)
C12	0.053 (2)	0.123 (3)	0.086 (3)	0.012 (3)	−0.007 (2)	−0.015 (3)
C13	0.057 (2)	0.116 (4)	0.085 (3)	−0.022 (2)	0.006 (2)	−0.013 (3)
C14	0.063 (2)	0.078 (3)	0.077 (2)	−0.007 (2)	0.0091 (19)	−0.002 (2)
C15	0.0513 (18)	0.100 (2)	0.056 (2)	−0.0009 (19)	0.0007 (16)	−0.003 (2)
C16	0.0480 (18)	0.075 (2)	0.0483 (19)	0.0102 (16)	−0.0053 (15)	−0.0023 (18)
C17	0.0468 (18)	0.078 (2)	0.0482 (19)	0.0109 (17)	−0.0077 (16)	−0.0046 (17)
C18	0.052 (2)	0.089 (2)	0.0554 (19)	0.0029 (18)	−0.0022 (18)	−0.008 (2)
C19	0.063 (2)	0.091 (3)	0.070 (3)	−0.0057 (19)	−0.0032 (19)	−0.007 (2)
C20	0.071 (3)	0.090 (3)	0.075 (3)	0.008 (2)	−0.018 (2)	−0.020 (2)
C21	0.077 (3)	0.103 (3)	0.060 (2)	0.004 (2)	−0.005 (2)	−0.019 (2)

C22	0.068 (2)	0.098 (3)	0.049 (2)	0.0001 (19)	−0.0013 (17)	−0.006 (2)

Geometric parameters (Å, º)

O1—C1	1.231 (4)	C10—H10	0.9300
N1—C1	1.362 (4)	C11—C12	1.365 (5)
N1—N2	1.399 (3)	C11—H11	0.9300
N1—C8	1.478 (4)	C12—C13	1.360 (5)
N2—C16	1.292 (4)	C12—H12	0.9300
C1—C2	1.482 (4)	C13—C14	1.387 (5)
C2—C3	1.379 (5)	C13—H13	0.9300
C2—C7	1.387 (4)	C14—H14	0.9300
C3—C4	1.381 (5)	C15—C16	1.504 (5)
C3—H3	0.9300	C15—H15A	0.9700
C4—C5	1.366 (6)	C15—H15B	0.9700
C4—H4	0.9300	C16—C17	1.463 (4)
C5—C6	1.361 (6)	C17—C18	1.386 (4)
C5—H5	0.9300	C17—C22	1.394 (4)
C6—C7	1.373 (6)	C18—C19	1.378 (5)
C6—H6	0.9300	C18—H18	0.9300
C7—H7	0.9300	C19—C20	1.378 (5)
C8—C9	1.507 (4)	C19—H19	0.9300
C8—C15	1.537 (4)	C20—C21	1.371 (5)
C8—H8	0.9800	C20—H20	0.9300
C9—C14	1.377 (4)	C21—C22	1.377 (5)
C9—C10	1.380 (4)	C21—H21	0.9300
C10—C11	1.383 (5)	C22—H22	0.9300
C1—N1—N2	122.6 (3)	C12—C11—H11	120.0
C1—N1—C8	122.5 (3)	C10—C11—H11	120.0
N2—N1—C8	113.3 (3)	C13—C12—C11	119.9 (4)
C16—N2—N1	107.9 (3)	C13—C12—H12	120.0
O1—C1—N1	119.6 (3)	C11—C12—H12	120.0
O1—C1—C2	122.1 (3)	C12—C13—C14	120.3 (4)
N1—C1—C2	118.2 (3)	C12—C13—H13	119.9
C3—C2—C7	118.7 (3)	C14—C13—H13	119.9
C3—C2—C1	122.9 (3)	C9—C14—C13	120.6 (4)
C7—C2—C1	118.2 (3)	C9—C14—H14	119.7
C2—C3—C4	119.7 (4)	C13—C14—H14	119.7
C2—C3—H3	120.1	C16—C15—C8	103.3 (3)
C4—C3—H3	120.1	C16—C15—H15A	111.1
C5—C4—C3	120.7 (4)	C8—C15—H15A	111.1
C5—C4—H4	119.6	C16—C15—H15B	111.1
C3—C4—H4	119.6	C8—C15—H15B	111.1
C6—C5—C4	120.0 (4)	H15A—C15—H15B	109.1
C6—C5—H5	120.0	N2—C16—C17	121.6 (3)
C4—C5—H5	120.0	N2—C16—C15	114.0 (3)
C5—C6—C7	120.0 (4)	C17—C16—C15	124.4 (3)

C5—C6—H6	120.0	C18—C17—C22	117.7 (3)
C7—C6—H6	120.0	C18—C17—C16	121.4 (3)
C6—C7—C2	120.8 (4)	C22—C17—C16	120.9 (3)
C6—C7—H7	119.6	C19—C18—C17	121.3 (3)
C2—C7—H7	119.6	C19—C18—H18	119.4
N1—C8—C9	112.0 (2)	C17—C18—H18	119.4
N1—C8—C15	101.4 (3)	C18—C19—C20	120.1 (4)
C9—C8—C15	114.0 (3)	C18—C19—H19	119.9
N1—C8—H8	109.7	C20—C19—H19	119.9
C9—C8—H8	109.7	C21—C20—C19	119.5 (4)
C15—C8—H8	109.7	C21—C20—H20	120.2
C14—C9—C10	118.2 (3)	C19—C20—H20	120.2
C14—C9—C8	120.7 (3)	C20—C21—C22	120.6 (4)
C10—C9—C8	121.0 (3)	C20—C21—H21	119.7
C9—C10—C11	120.8 (4)	C22—C21—H21	119.7
C9—C10—H10	119.6	C21—C22—C17	120.8 (4)
C11—C10—H10	119.6	C21—C22—H22	119.6
C12—C11—C10	120.1 (4)	C17—C22—H22	119.6

C1—N1—N2—C16	−165.4 (3)	C14—C9—C10—C11	0.9 (5)
C8—N1—N2—C16	0.8 (3)	C8—C9—C10—C11	−176.8 (3)
N2—N1—C1—O1	166.0 (3)	C9—C10—C11—C12	1.1 (6)
C8—N1—C1—O1	1.0 (4)	C10—C11—C12—C13	−2.7 (6)
N2—N1—C1—C2	−15.4 (4)	C11—C12—C13—C14	2.3 (6)
C8—N1—C1—C2	179.6 (3)	C10—C9—C14—C13	−1.3 (5)
O1—C1—C2—C3	128.9 (3)	C8—C9—C14—C13	176.4 (3)
N1—C1—C2—C3	−49.7 (4)	C12—C13—C14—C9	−0.3 (6)
O1—C1—C2—C7	−45.7 (4)	N1—C8—C15—C16	2.1 (3)
N1—C1—C2—C7	135.7 (3)	C9—C8—C15—C16	−118.4 (3)
C7—C2—C3—C4	2.0 (5)	N1—N2—C16—C17	−178.2 (2)
C1—C2—C3—C4	−172.5 (3)	N1—N2—C16—C15	0.8 (3)
C2—C3—C4—C5	−3.0 (5)	C8—C15—C16—N2	−1.9 (4)
C3—C4—C5—C6	1.5 (6)	C8—C15—C16—C17	177.0 (3)
C4—C5—C6—C7	0.8 (7)	N2—C16—C17—C18	7.1 (4)
C5—C6—C7—C2	−1.8 (6)	C15—C16—C17—C18	−171.7 (3)
C3—C2—C7—C6	0.3 (5)	N2—C16—C17—C22	−175.0 (3)
C1—C2—C7—C6	175.2 (3)	C15—C16—C17—C22	6.2 (4)
C1—N1—C8—C9	−73.8 (4)	C22—C17—C18—C19	−0.4 (5)
N2—N1—C8—C9	120.0 (3)	C16—C17—C18—C19	177.5 (3)
C1—N1—C8—C15	164.3 (3)	C17—C18—C19—C20	0.6 (5)
N2—N1—C8—C15	−1.9 (3)	C18—C19—C20—C21	0.0 (5)
N1—C8—C9—C14	131.1 (3)	C19—C20—C21—C22	−0.7 (5)
C15—C8—C9—C14	−114.5 (3)	C20—C21—C22—C17	0.9 (5)
N1—C8—C9—C10	−51.3 (4)	C18—C17—C22—C21	−0.3 (5)
C15—C8—C9—C10	63.1 (4)	C16—C17—C22—C21	−178.2 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C21—H21···O1^{i}	0.93	2.72	3.399 (5)	131
C22—H22···O1^{i}	0.93	3.00	3.540 (4)	119
C10—H10···O1^{ii}	0.93	2.87	3.793 (5)	174

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