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5-(4-Chlorophenoxy)-3-methyl-1-phenyl-1*H*-pyrazole-4-carbaldehyde

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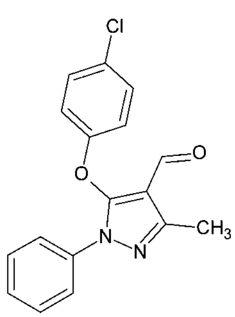
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.039; wR factor = 0.105; data-to-parameter ratio = 12.5.

In the title compound, $C_{17}H_{13}ClN_2O_2$, the phenyl and chlorobenzene rings are inclined to the central pyrazole ring at 40.84 (9) and 65.30 (9)°, respectively. In the crystal, pairs of C—H⋯π interactions link the molecules into inversion dimers and C—H⋯O hydrogen bonds link these dimers into columns extended in [010]. The crystal packing exhibits short intermolecular O⋯Cl contacts of 3.0913 (16) Å.

Related literature

For biological properties and pharmacological applications of aryloxy pyrazole derivatives, see: Rai *et al.* (2008); Girisha *et al.* (2010); Isloor *et al.* (2009, 2010); Shobhitha *et al.* (2013). For related structures, see: Shahani, Fun, Ragavan *et al.* (2011); Shahani, Fun, Shetty *et al.* (2011); Prasath *et al.* (2011).



Experimental

Crystal data

$C_{17}H_{13}ClN_2O_2$
M_r = 312.74
Monoclinic, $P2_1/c$
a = 9.1016 (7) Å
b = 7.5298 (6) Å
c = 22.1242 (16) Å
β = 93.908 (3)°
V = 1512.7 (2) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 2.31 mm^{-1}
T = 296 K
0.23 × 0.22 × 0.21 mm

Data collection

Bruker X8 Proteum diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2013)
T_{min} = 0.619, T_{max} = 0.643
9744 measured reflections
2501 independent reflections
2314 reflections with $I > 2\sigma(I)$
R_{int} = 0.041

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.105
S = 1.03
2501 reflections
200 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

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

Cg is the centroid of the C11–C16 ring.

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C6—H6⋯O2^{i}	0.93	2.58	3.503 (2)	171
C2—H2⋯Cg^{ii}	0.93	2.63	3.476 (2)	152

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

Data collection: *APEX2* (Bruker, 2013); cell refinement: *SAINT* (Bruker, 2013); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *PLATON* (Spek, 2009).

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Supporting information for this paper is available from the IUCr electronic archives (Reference: CV5447).

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supplementary materials

Acta Cryst. (2014). E**70**, o560 [doi:10.1107/S1600536814007879]

5-(4-Chlorophenoxy)-3-methyl-1-phenyl-1*H*-pyrazole-4-carbaldehyde

N. Vinutha, S. Madan Kumar, S. Shobhitha, B. Kalluraya, N. K. Lokanath and D. Revannasiddaiah

1. Comment

Aryloxy pyrazoles and their derivatives possess a significant pharmcological activities such as antimicrobial (Rai *et al.* 2008; Girisha *et al.*, 2010), anti-inflammatory (Isloor *et al.*, 2009) and analgesic activities (Shobhitha *et al.*, 2013). The title compound can serve as an intermediate in the synthesis of various pyrazole derivatives with significant pharmacological activities (Isloor *et al.*, 2010).

In the title compound (Fig.1), all bond lengths and angles are normal and correspond well to those observed in the related compounds (Shahani, Fun, Ragavan *et al.*, 2011; Shahani, Fun, Shetty *et al.*, 2011; Prasath *et al.*, 2011). The pyrazole ring makes dihedral angles of 65.30 (9)° with chlorobenzene ring and 40.84 (9)° with benzene ring. The dihedral angle between the chlorobenzene ring and benzene ring is 76.23 (9)°.

In the crystal, C–H···π interactions (Table 1) link the molecules into inversion dimers, and intermolecular C–H···O hydrogen bonds (Table 1) link these dimers into columns extended in [010]. The crystal packing exhibits short intermolecular O···Cl contacts of 3.0913 (16) Å.

2. Experimental

The title compound was prepared by refluxing a mixture of 5-chloro-3-methyl-1-phenyl-1*H*-pyrazol-4-carboxaldehyde (0.1 mol) and 4-chloro phenol (0.1 mol) in 10 ml of dimethyl sulfoxide. To this solution, 0.1 mol of potassium hydroxide was added. The reaction mixture was refluxed for 3 hrs and then it was cooled to room temperature and poured to crushed ice. The solid product that separated was filtered and dried. It was then recrystallized from ethanol. Crystals suitable for X-ray analysis were obtained from slow evaporation of ethanol.

3. Refinement

All the H atoms were fixed geometrically (C—H= 0.93–0.96 Å) and allowed to ride on their parent atoms with U_{iso}(H) = 1.5U_{eq}(*C*-methyl) and = 1.2U_{eq}(C) for other H atoms.

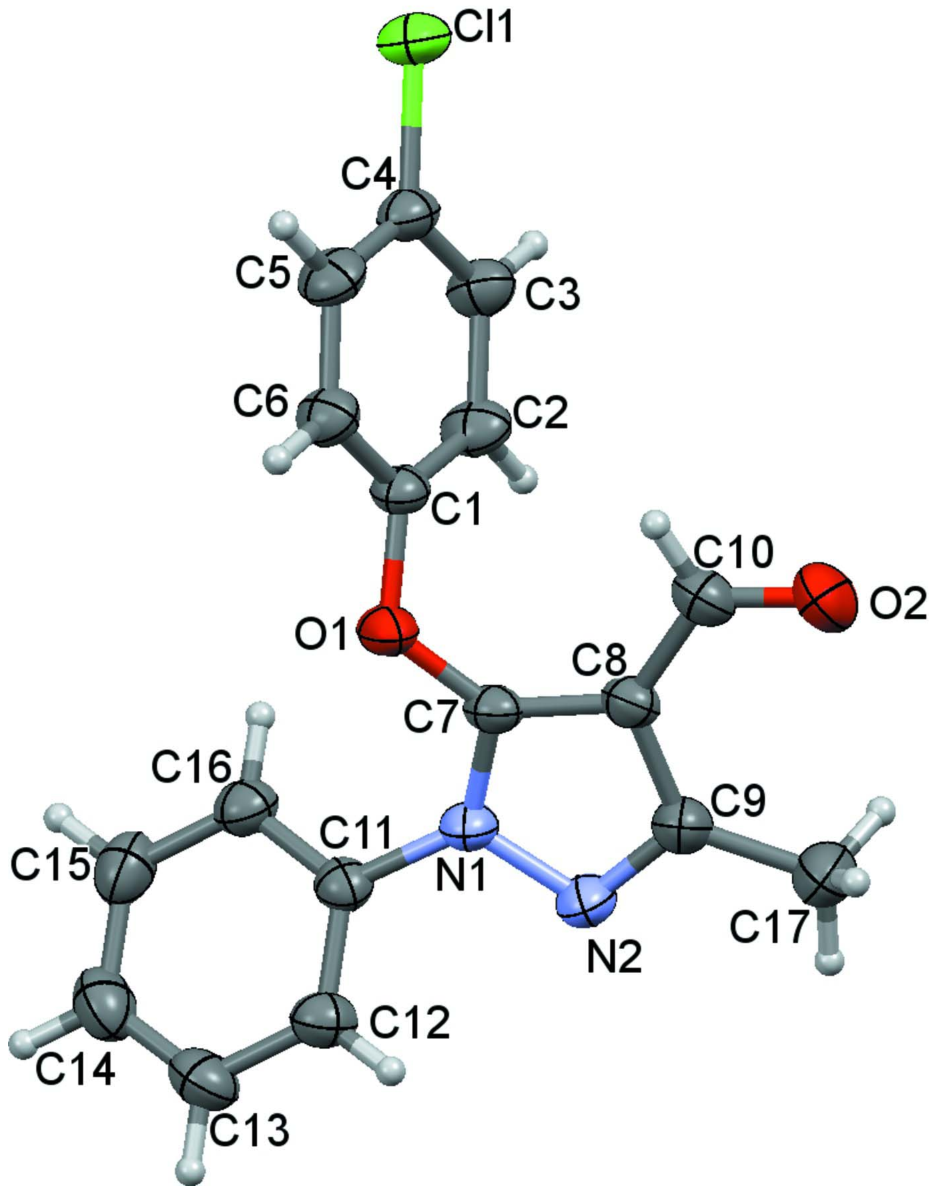

Figure 1

The molecular structure of the title compound showing the atomic numbering and 50% probability displacement ellipsoids.

5-(4-Chlorophenoxy)-3-methyl-1-phenyl-1*H*-pyrazole-4-carbaldehyde

Crystal data

$C_{17}H_{13}ClN_2O_2$	V = 1512.7 (2) Å^3
M_r = 312.74	Z = 4
Monoclinic, $P2_1/c$	F(000) = 648
Hall symbol: -P 2ybc	D_x = 1.373 Mg m^{-3}
a = 9.1016 (7) Å	Cu $K\alpha$ radiation, λ = 1.54178 Å
b = 7.5298 (6) Å	Cell parameters from 2501 reflections
c = 22.1242 (16) Å	θ = 4.0–64.4°
β = 93.908 (3)°	μ = 2.31 mm^{-1}

T = 296 K
Block, brown
0.23 × 0.22 × 0.21 mm

Data collection

Bruker X8 Proteum diffractometer
Radiation source: Bruker MicroStar microfocus rotating anode
Helios multilayer optics monochromator
Detector resolution: 18.4 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2013)
T_{min} = 0.619, T_{max} = 0.643
9744 measured reflections
2501 independent reflections
2314 reflections with $I > 2\sigma(I)$
R_{int} = 0.041
θ_{max} = 64.5°, θ_{min} = 4.0°
$h = -10 \rightarrow 10$
$k = -3 \rightarrow 8$
$l = -25 \rightarrow 25$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.105
S = 1.03
2501 reflections
200 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
$w = 1/[\sigma^2(F_o^2) + (0.0555P)^2 + 0.5076P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $FC^*=KFC[1+0.001XFC^2\Lambda^3/SIN(2\Theta)]^{-1/4}$
Extinction coefficient: 0.0171 (10)

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted R-factors wR and all goodnesses 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 observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating -R-factor-obs *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}
Cl1	0.33674 (5)	0.67384 (7)	0.29951 (2)	0.0525 (2)
O1	0.96938 (13)	0.67414 (15)	0.37256 (6)	0.0417 (4)
O2	0.96678 (16)	0.16557 (19)	0.27222 (7)	0.0579 (5)
N1	1.15356 (14)	0.51714 (19)	0.42604 (6)	0.0354 (4)
N2	1.23196 (16)	0.3599 (2)	0.42482 (7)	0.0420 (5)
C1	0.81860 (18)	0.6650 (2)	0.35492 (7)	0.0335 (5)
C2	0.72891 (19)	0.5340 (3)	0.37544 (8)	0.0419 (5)
C3	0.58011 (19)	0.5360 (3)	0.35785 (8)	0.0417 (5)
C4	0.52407 (19)	0.6712 (2)	0.32108 (7)	0.0378 (5)
C5	0.6139 (2)	0.8024 (2)	0.30129 (8)	0.0438 (6)
C6	0.7637 (2)	0.8000 (2)	0.31799 (8)	0.0403 (6)
C7	1.04684 (16)	0.5223 (2)	0.38079 (7)	0.0332 (5)

C8	1.05021 (18)	0.3640 (2)	0.34914 (7)	0.0354 (5)
C9	1.16849 (19)	0.2680 (2)	0.37941 (8)	0.0398 (5)
C10	0.9588 (2)	0.3101 (3)	0.29611 (8)	0.0407 (6)
C11	1.20153 (18)	0.6520 (2)	0.46814 (7)	0.0336 (5)
C12	1.3510 (2)	0.6767 (2)	0.47974 (8)	0.0407 (6)
C13	1.4008 (2)	0.8051 (3)	0.52092 (9)	0.0488 (6)
C14	1.3028 (2)	0.9086 (3)	0.54945 (9)	0.0533 (7)
C15	1.1529 (2)	0.8822 (3)	0.53819 (8)	0.0495 (6)
C16	1.10095 (19)	0.7530 (2)	0.49751 (7)	0.0404 (5)
C17	1.2243 (2)	0.0881 (3)	0.36473 (11)	0.0592 (7)
H02A	1.30480	0.05740	0.39310	0.0890*
H2	0.76800	0.44510	0.40090	0.0500*
H02B	1.25740	0.08840	0.32440	0.0890*
H3	0.51850	0.44700	0.37070	0.0500*
H02C	1.14670	0.00270	0.36720	0.0890*
H5	0.57430	0.89290	0.27670	0.0530*
H6	0.82560	0.88770	0.30450	0.0480*
H10	0.89020	0.39080	0.27940	0.0490*
H12	1.41770	0.60760	0.46010	0.0490*
H13	1.50150	0.82140	0.52930	0.0590*
H14	1.33690	0.99660	0.57640	0.0640*
H15	1.08660	0.95150	0.55800	0.0590*
H16	1.00030	0.73440	0.49010	0.0480*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0314 (3)	0.0597 (4)	0.0649 (3)	0.0089 (2)	−0.0083 (2)	−0.0045 (2)
O1	0.0335 (6)	0.0355 (7)	0.0536 (7)	0.0015 (5)	−0.0141 (5)	0.0023 (5)
O2	0.0543 (9)	0.0558 (9)	0.0619 (9)	−0.0033 (6)	−0.0090 (7)	−0.0195 (7)
N1	0.0304 (7)	0.0367 (8)	0.0378 (7)	0.0046 (6)	−0.0069 (5)	−0.0024 (6)
N2	0.0365 (8)	0.0382 (8)	0.0497 (8)	0.0084 (6)	−0.0094 (6)	−0.0035 (6)
C1	0.0294 (8)	0.0379 (9)	0.0321 (8)	0.0039 (6)	−0.0057 (6)	−0.0005 (6)
C2	0.0382 (9)	0.0447 (10)	0.0418 (9)	0.0044 (8)	−0.0038 (7)	0.0129 (7)
C3	0.0342 (9)	0.0454 (10)	0.0456 (9)	0.0018 (7)	0.0038 (7)	0.0068 (8)
C4	0.0316 (9)	0.0442 (10)	0.0368 (8)	0.0072 (7)	−0.0034 (7)	−0.0030 (7)
C5	0.0423 (10)	0.0432 (10)	0.0445 (9)	0.0091 (8)	−0.0071 (8)	0.0099 (8)
C6	0.0390 (10)	0.0379 (10)	0.0435 (9)	0.0020 (7)	−0.0014 (7)	0.0076 (7)
C7	0.0258 (7)	0.0365 (9)	0.0364 (8)	0.0000 (6)	−0.0044 (6)	0.0037 (7)
C8	0.0302 (8)	0.0366 (9)	0.0386 (8)	−0.0034 (7)	−0.0030 (6)	0.0003 (7)
C9	0.0340 (9)	0.0393 (10)	0.0453 (9)	0.0013 (7)	−0.0021 (7)	−0.0040 (7)
C10	0.0360 (9)	0.0442 (11)	0.0409 (9)	−0.0062 (7)	−0.0037 (7)	−0.0020 (8)
C11	0.0350 (9)	0.0340 (9)	0.0308 (8)	0.0012 (6)	−0.0042 (6)	0.0031 (6)
C12	0.0340 (9)	0.0490 (11)	0.0386 (9)	0.0006 (7)	−0.0020 (7)	−0.0009 (7)
C13	0.0440 (10)	0.0539 (12)	0.0468 (10)	−0.0090 (9)	−0.0097 (8)	−0.0019 (8)
C14	0.0658 (13)	0.0454 (12)	0.0467 (10)	−0.0040 (9)	−0.0104 (9)	−0.0069 (8)
C15	0.0623 (12)	0.0430 (11)	0.0429 (9)	0.0136 (9)	0.0021 (8)	−0.0035 (8)
C16	0.0360 (9)	0.0432 (10)	0.0412 (9)	0.0062 (7)	−0.0023 (7)	0.0021 (8)
C17	0.0530 (12)	0.0482 (12)	0.0741 (13)	0.0116 (10)	−0.0122 (10)	−0.0135 (10)

Geometric parameters (Å, º)

Cl1—C4	1.7390 (18)	C11—C16	1.386 (2)
O1—C1	1.403 (2)	C12—C13	1.384 (3)
O1—C7	1.3490 (19)	C13—C14	1.370 (3)
O2—C10	1.214 (3)	C14—C15	1.384 (3)
N1—N2	1.384 (2)	C15—C16	1.386 (3)
N1—C7	1.347 (2)	C2—H2	0.9300
N1—C11	1.426 (2)	C3—H3	0.9300
N2—C9	1.320 (2)	C5—H5	0.9300
C1—C2	1.377 (3)	C6—H6	0.9300
C1—C6	1.377 (2)	C10—H10	0.9300
C2—C3	1.384 (2)	C12—H12	0.9300
C3—C4	1.379 (3)	C13—H13	0.9300
C4—C5	1.373 (2)	C14—H14	0.9300
C5—C6	1.388 (3)	C15—H15	0.9300
C7—C8	1.384 (2)	C16—H16	0.9300
C8—C9	1.425 (2)	C17—H02A	0.9600
C8—C10	1.449 (2)	C17—H02B	0.9600
C9—C17	1.490 (3)	C17—H02C	0.9600
C11—C12	1.380 (2)		
C1—O1—C7	119.23 (12)	C13—C14—C15	119.99 (19)
N2—N1—C7	110.91 (13)	C14—C15—C16	120.39 (18)
N2—N1—C11	119.12 (13)	C11—C16—C15	118.86 (16)
C7—N1—C11	129.64 (14)	C1—C2—H2	120.00
N1—N2—C9	105.30 (13)	C3—C2—H2	120.00
O1—C1—C2	122.29 (14)	C2—C3—H3	120.00
O1—C1—C6	115.95 (14)	C4—C3—H3	120.00
C2—C1—C6	121.69 (16)	C4—C5—H5	120.00
C1—C2—C3	119.37 (18)	C6—C5—H5	120.00
C2—C3—C4	119.31 (18)	C1—C6—H6	121.00
Cl1—C4—C3	119.12 (13)	C5—C6—H6	121.00
Cl1—C4—C5	119.89 (13)	O2—C10—H10	118.00
C3—C4—C5	121.00 (16)	C8—C10—H10	118.00
C4—C5—C6	120.08 (15)	C11—C12—H12	120.00
C1—C6—C5	118.54 (15)	C13—C12—H12	120.00
O1—C7—N1	117.94 (14)	C12—C13—H13	120.00
O1—C7—C8	133.72 (14)	C14—C13—H13	120.00
N1—C7—C8	108.15 (13)	C13—C14—H14	120.00
C7—C8—C9	103.96 (14)	C15—C14—H14	120.00
C7—C8—C10	128.28 (16)	C14—C15—H15	120.00
C9—C8—C10	127.72 (15)	C16—C15—H15	120.00
N2—C9—C8	111.66 (14)	C11—C16—H16	121.00
N2—C9—C17	120.29 (16)	C15—C16—H16	121.00
C8—C9—C17	128.05 (16)	C9—C17—H02A	109.00
O2—C10—C8	123.73 (18)	C9—C17—H02B	109.00
N1—C11—C12	118.14 (14)	C9—C17—H02C	109.00
N1—C11—C16	120.96 (14)	H02A—C17—H02B	110.00
C12—C11—C16	120.89 (15)	H02A—C17—H02C	109.00

C11—C12—C13	119.43 (16)	H02B—C17—H02C	109.00
C12—C13—C14	120.43 (17)		
C7—O1—C1—C2	36.1 (2)	C2—C3—C4—C5	0.6 (3)
C7—O1—C1—C6	−146.92 (15)	Cl1—C4—C5—C6	−179.47 (13)
C1—O1—C7—N1	−143.39 (14)	C3—C4—C5—C6	0.3 (3)
C1—O1—C7—C8	42.4 (2)	C4—C5—C6—C1	−0.6 (2)
C7—N1—N2—C9	−1.81 (18)	O1—C7—C8—C9	174.01 (17)
C11—N1—N2—C9	−175.88 (14)	O1—C7—C8—C10	−3.9 (3)
N2—N1—C7—O1	−174.08 (13)	N1—C7—C8—C9	−0.60 (17)
N2—N1—C7—C8	1.51 (18)	N1—C7—C8—C10	−178.50 (16)
C11—N1—C7—O1	−0.8 (2)	C7—C8—C9—N2	−0.55 (19)
C11—N1—C7—C8	174.78 (15)	C7—C8—C9—C17	−179.41 (17)
N2—N1—C11—C12	37.2 (2)	C10—C8—C9—N2	177.37 (17)
N2—N1—C11—C16	−141.54 (15)	C10—C8—C9—C17	−1.5 (3)
C7—N1—C11—C12	−135.63 (17)	C7—C8—C10—O2	−177.79 (18)
C7—N1—C11—C16	45.7 (2)	C9—C8—C10—O2	4.8 (3)
N1—N2—C9—C8	1.42 (19)	N1—C11—C12—C13	−179.23 (16)
N1—N2—C9—C17	−179.62 (16)	C16—C11—C12—C13	−0.5 (2)
O1—C1—C2—C3	177.88 (16)	N1—C11—C16—C15	179.80 (15)
C6—C1—C2—C3	1.0 (3)	C12—C11—C16—C15	1.1 (2)
O1—C1—C6—C5	−177.13 (15)	C11—C12—C13—C14	−0.8 (3)
C2—C1—C6—C5	−0.1 (3)	C12—C13—C14—C15	1.5 (3)
C1—C2—C3—C4	−1.3 (3)	C13—C14—C15—C16	−0.9 (3)
C2—C3—C4—Cl1	−179.59 (14)	C14—C15—C16—C11	−0.4 (3)

Hydrogen-bond geometry (Å, º)

Cg is the centroid of the C11–C16 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6···O2^{i}	0.93	2.58	3.503 (2)	171
C2—H2···*Cg*^{ii}	0.93	2.63	3.476 (2)	152

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