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1,5-Dimethyl-4-[(*E*)-3-phenoxybenzylideneamino]-2-phenyl-1*H*-pyrazol-3(2*H*)-one

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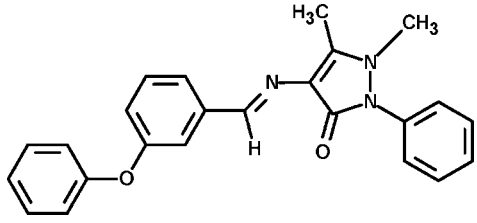
Key indicators: single-crystal X-ray study; T = 273 K; mean σ(C–C) = 0.003 Å; R factor = 0.043; wR factor = 0.118; data-to-parameter ratio = 13.3.

The title Schiff base, $C_{24}H_{21}N_3O_2$, adopts an *E* configuration with respect to the central C=N bond. The pyrazole ring and the central benzene ring attached to the imino group are almost coplanar. The phenyl ring attached to the pyrazole unit is twisted by 39.3 (2)° with respect to the pyrazole ring plane. The phenoxy benzene ring makes a dihedral angle of 79.8 (2)° with the central benzene ring.

Related literature

For related crystal structures, see: Sun *et al.* (2007*a*,*b*,*c*).



Experimental

Crystal data

$C_{24}H_{21}N_3O_2$
M_r = 383.44
Triclinic, $P\overline{1}$
a = 7.6640 (12) Å
b = 8.3593 (14) Å
c = 16.731 (3) Å
α = 77.396 (3)°
β = 77.587 (2)°
γ = 76.240 (3)°
V = 1000.9 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 273 (2) K
0.18 × 0.16 × 0.12 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.985, T_{max} = 0.990
5300 measured reflections
3505 independent reflections
2584 reflections with $I > 2\sigma(I)$
R_{int} = 0.015

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.117
S = 1.04
3505 reflections
264 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); 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: FJ2143).

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supplementary materials

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1,5-Dimethyl-4-[(*E*)-3-phenoxybenzylideneamino]-2-phenyl-1*H*-pyrazol-3(2*H*)-one

Y.-F. Sun, F.-Y. Zhang, Y. Liu, Z.-Y. Wang and X.-L. Cheng

Comment

Antipyrine (2,3-dimethyl-1-phenylpyrazol-5-one) and its derivatives have been long known for their wide spectrum of biological activities. As part of our ongoing studies of antipyrine derivatives, the title compound, (I), has been prepared and its crystal structure is reported here (Fig. 1).

The molecule adopts an E configuration with respect to the central C=N double bond (Fig. 1).The pyrazole ring (N1/N2/C7—C9), the C13—C18 phenyl ring and the imino group are almost coplanar which allows conjugation. But the C1—C6 phenyl ring is twisted with respect to the central pyrazole ring plane by 39.3 (2)°. In addition, the mean planes of the C13—C18 and C19—C24 phenyl rings make a dihedral angle of 79.8 (2)°. Therefore the molecule is not planar. The bond distances and angles agree with the corresponding values found in similar compounds (Sun *et al.*, 2007*a*,b,c).

Experimental

A mixture of 4-aminoantipyrine (1 mmol) and 3-phenoxybenzaldehyde (1 mmol) in anhydrous ethanol (20 ml) was refluxed for 3 hr, and then cooled to room temperature. After cooling, the solvent was removed under reduced pressure and the solid residue was recrystallized from ethanol to yield the pure product(66% yield). m.p. 425–427 K. A single-crystal suitable for an X-ray structural analysis was obtained by slowly evaporating a ethanolic solution at room temperature.

Refinement

All H atoms were initially located in a difference Fourier map. The methyl H atoms were then constrained to an ideal geometry with C—H distances of 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C). All other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms with C—H distances 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C).

Figures

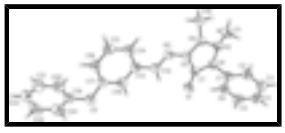


Fig. 1. View of the molecule of (I) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are shown as small spheres of arbitrary radii.

1,5-Dimethyl-4-[(E)-3-phenoxybenzylideneamino]-2-phenyl-1*H*- pyrazol-3(2*H*)-one

Crystal data

$C_{24}H_{21}N_3O_2$	$Z = 2$
$M_r = 383.44$	$F_{000} = 404$
Triclinic, $P\overline{1}$	$D_x = 1.272$ Mg m^{-3}

Hall symbol: -P 1	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 7.6640 (12) Å	Cell parameters from 1817 reflections
b = 8.3593 (14) Å	θ = 2–25.1º
c = 16.731 (3) Å	μ = 0.08 mm^{-1}
α = 77.396 (3)º	T = 273 (2) K
β = 77.587 (2)º	Block, colorless
γ = 76.240 (3)º	0.18 × 0.16 × 0.12 mm
V = 1000.9 (3) Å^3	

Data collection

Bruker SMART CCD area-detector diffractometer	3505 independent reflections
Radiation source: fine-focus sealed tube	2584 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.016
T = 273(2) K	θ_{max} = 25.1º
φ and ω scans	θ_{min} = 2.5º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -9 \rightarrow 9$
T_{min} = 0.985, T_{max} = 0.990	$k = -9 \rightarrow 8$
5300 measured reflections	$l = -19 \rightarrow 19$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)]$ = 0.043	H-atom parameters constrained
$wR(F^2)$ = 0.117	$w = 1/[\sigma^2(F_o^2) + (0.0553P)^2 + 0.1379P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.04	$(\Delta/\sigma)_{max}$ = 0.001
3505 reflections	$\Delta\rho_{max}$ = 0.17 e Å^{-3}
264 parameters	$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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	−0.09698 (15)	0.55049 (16)	1.14179 (8)	0.0559 (3)
O2	−0.41493 (16)	1.0621 (2)	0.79039 (8)	0.0727 (4)
N1	0.17090 (17)	0.46344 (17)	1.19591 (8)	0.0451 (4)
N2	0.34291 (17)	0.50949 (17)	1.17773 (9)	0.0436 (3)
N3	0.15472 (18)	0.75399 (18)	1.00126 (8)	0.0463 (4)
C1	0.1017 (2)	0.3971 (2)	1.27873 (10)	0.0436 (4)
C2	0.1611 (2)	0.4293 (2)	1.34520 (11)	0.0534 (5)
H2	0.2455	0.4978	1.3359	0.064*
C3	0.0942 (3)	0.3590 (3)	1.42535 (12)	0.0644 (6)
H3	0.1348	0.3798	1.4699	0.077*
C4	−0.0311 (3)	0.2590 (3)	1.43974 (13)	0.0696 (6)
H4	−0.0752	0.2118	1.4938	0.084*
C5	−0.0915 (3)	0.2288 (3)	1.37361 (13)	0.0641 (5)
H5	−0.1778	0.1620	1.3833	0.077*
C6	−0.0253 (2)	0.2967 (2)	1.29318 (11)	0.0507 (4)
H6	−0.0659	0.2749	1.2488	0.061*
C7	0.0644 (2)	0.5536 (2)	1.13636 (10)	0.0430 (4)
C8	0.1860 (2)	0.6450 (2)	1.07513 (10)	0.0412 (4)
C9	0.3485 (2)	0.6120 (2)	1.10112 (10)	0.0431 (4)
C10	0.5167 (2)	0.6735 (3)	1.05804 (11)	0.0573 (5)
H10A	0.6070	0.5828	1.0386	0.086*
H10B	0.5625	0.7175	1.0960	0.086*
H10C	0.4894	0.7600	1.0117	0.086*
C11	0.4972 (2)	0.3701 (2)	1.19262 (13)	0.0607 (5)
H11A	0.5038	0.2889	1.1588	0.091*
H11B	0.4801	0.3187	1.2502	0.091*
H11C	0.6084	0.4116	1.1786	0.091*
C12	0.0019 (2)	0.7838 (2)	0.97743 (10)	0.0477 (4)
H12	−0.0906	0.7318	1.0099	0.057*
C13	−0.0321 (2)	0.8980 (2)	0.90021 (10)	0.0442 (4)
C14	0.1005 (2)	0.9784 (2)	0.84859 (11)	0.0502 (4)
H14	0.2152	0.9607	0.8628	0.060*
C15	0.0622 (2)	1.0844 (2)	0.77623 (12)	0.0548 (5)
H15	0.1519	1.1379	0.7420	0.066*
C16	−0.1063 (2)	1.1126 (2)	0.75369 (11)	0.0493 (4)
H16	−0.1304	1.1832	0.7044	0.059*
C17	−0.2385 (2)	1.0347 (2)	0.80524 (11)	0.0474 (4)
C18	−0.2022 (2)	0.9280 (2)	0.87751 (11)	0.0492 (4)
H18	−0.2927	0.8753	0.9115	0.059*
C19	−0.4469 (2)	1.1295 (2)	0.71019 (11)	0.0482 (4)
C20	−0.3803 (3)	1.0387 (3)	0.64795 (13)	0.0618 (5)
H20	−0.3083	0.9324	0.6586	0.074*
C21	−0.4186 (3)	1.1025 (3)	0.57065 (14)	0.0781 (7)
H21	−0.3719	1.0402	0.5283	0.094*
C22	−0.5239 (3)	1.2556 (4)	0.55469 (15)	0.0885 (8)

H22	−0.5492	1.2985	0.5013	0.106*
C23	−0.5939 (3)	1.3483 (3)	0.61630 (18)	0.0836 (7)
H23	−0.6673	1.4536	0.6049	0.100*
C24	−0.5555 (3)	1.2854 (2)	0.69626 (14)	0.0621 (5)
H24	−0.6020	1.3472	0.7388	0.075*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0386 (7)	0.0680 (9)	0.0613 (8)	−0.0141 (6)	−0.0158 (6)	−0.0009 (6)
O2	0.0417 (7)	0.1136 (12)	0.0539 (8)	−0.0163 (7)	−0.0152 (6)	0.0112 (8)
N1	0.0369 (7)	0.0496 (9)	0.0479 (9)	−0.0084 (6)	−0.0113 (6)	−0.0030 (7)
N2	0.0319 (7)	0.0491 (8)	0.0488 (8)	−0.0050 (6)	−0.0119 (6)	−0.0051 (7)
N3	0.0424 (8)	0.0543 (9)	0.0427 (8)	−0.0071 (7)	−0.0114 (6)	−0.0085 (7)
C1	0.0398 (9)	0.0379 (9)	0.0476 (10)	0.0016 (7)	−0.0093 (7)	−0.0046 (7)
C2	0.0522 (10)	0.0498 (11)	0.0575 (12)	−0.0003 (9)	−0.0169 (9)	−0.0114 (9)
C3	0.0641 (12)	0.0728 (14)	0.0502 (12)	0.0075 (11)	−0.0153 (10)	−0.0160 (10)
C4	0.0636 (13)	0.0783 (15)	0.0484 (12)	0.0006 (12)	−0.0003 (10)	0.0024 (10)
C5	0.0561 (11)	0.0622 (13)	0.0640 (14)	−0.0119 (10)	0.0010 (10)	−0.0012 (10)
C6	0.0458 (10)	0.0500 (11)	0.0527 (11)	−0.0052 (8)	−0.0071 (8)	−0.0078 (8)
C7	0.0376 (9)	0.0453 (10)	0.0469 (10)	−0.0037 (7)	−0.0125 (7)	−0.0097 (8)
C8	0.0369 (8)	0.0476 (10)	0.0400 (9)	−0.0061 (7)	−0.0095 (7)	−0.0092 (7)
C9	0.0386 (9)	0.0492 (10)	0.0419 (9)	−0.0071 (7)	−0.0074 (7)	−0.0103 (8)
C10	0.0409 (10)	0.0769 (14)	0.0537 (11)	−0.0155 (9)	−0.0075 (8)	−0.0070 (10)
C11	0.0441 (10)	0.0598 (12)	0.0742 (13)	0.0031 (9)	−0.0205 (9)	−0.0080 (10)
C12	0.0426 (9)	0.0555 (11)	0.0445 (10)	−0.0105 (8)	−0.0102 (8)	−0.0044 (8)
C13	0.0422 (9)	0.0477 (10)	0.0437 (10)	−0.0073 (8)	−0.0109 (7)	−0.0089 (8)
C14	0.0398 (9)	0.0562 (11)	0.0568 (11)	−0.0123 (8)	−0.0131 (8)	−0.0070 (9)
C15	0.0497 (10)	0.0565 (12)	0.0590 (12)	−0.0211 (9)	−0.0095 (9)	−0.0004 (9)
C16	0.0508 (10)	0.0451 (10)	0.0497 (10)	−0.0113 (8)	−0.0128 (8)	0.0020 (8)
C17	0.0378 (9)	0.0568 (11)	0.0470 (10)	−0.0085 (8)	−0.0112 (8)	−0.0050 (8)
C18	0.0429 (9)	0.0589 (11)	0.0449 (10)	−0.0142 (8)	−0.0078 (8)	−0.0024 (8)
C19	0.0363 (9)	0.0573 (11)	0.0488 (11)	−0.0105 (8)	−0.0137 (8)	0.0028 (8)
C20	0.0598 (12)	0.0541 (12)	0.0644 (13)	−0.0029 (9)	−0.0083 (10)	−0.0070 (10)
C21	0.0711 (14)	0.1030 (19)	0.0636 (15)	−0.0168 (14)	−0.0149 (12)	−0.0192 (13)
C22	0.0640 (14)	0.129 (2)	0.0611 (15)	−0.0159 (15)	−0.0246 (12)	0.0161 (15)
C23	0.0583 (13)	0.0630 (14)	0.108 (2)	0.0043 (11)	−0.0239 (13)	0.0230 (14)
C24	0.0521 (11)	0.0516 (12)	0.0806 (15)	−0.0063 (9)	−0.0097 (10)	−0.0134 (10)

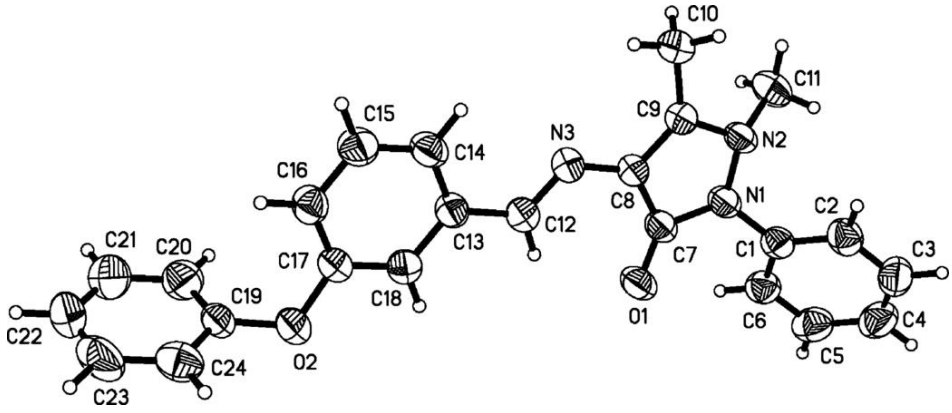
Geometric parameters (Å, °)

O1—C7	1.2264 (19)	C11—H11A	0.9600
O2—C17	1.383 (2)	C11—H11B	0.9600
O2—C19	1.387 (2)	C11—H11C	0.9600
N1—C7	1.399 (2)	C12—C13	1.462 (2)
N1—N2	1.4130 (18)	C12—H12	0.9300
N1—C1	1.413 (2)	C13—C18	1.386 (2)
N2—C9	1.376 (2)	C13—C14	1.387 (2)
N2—C11	1.471 (2)	C14—C15	1.378 (2)

N3—C12	1.269 (2)	C14—H14	0.9300
N3—C8	1.395 (2)	C15—C16	1.376 (2)
C1—C6	1.382 (2)	C15—H15	0.9300
C1—C2	1.386 (2)	C16—C17	1.374 (2)
C2—C3	1.382 (3)	C16—H16	0.9300
C2—H2	0.9300	C17—C18	1.375 (2)
C3—C4	1.369 (3)	C18—H18	0.9300
C3—H3	0.9300	C19—C20	1.362 (3)
C4—C5	1.379 (3)	C19—C24	1.371 (3)
C4—H4	0.9300	C20—C21	1.353 (3)
C5—C6	1.379 (3)	C20—H20	0.9300
C5—H5	0.9300	C21—C22	1.346 (4)
C6—H6	0.9300	C21—H21	0.9300
C7—C8	1.445 (2)	C22—C23	1.366 (4)
C8—C9	1.354 (2)	C22—H22	0.9300
C9—C10	1.483 (2)	C23—C24	1.393 (3)
C10—H10A	0.9600	C23—H23	0.9300
C10—H10B	0.9600	C24—H24	0.9300
C10—H10C	0.9600		
C17—O2—C19	118.13 (13)	H11A—C11—H11B	109.5
C7—N1—N2	110.26 (13)	N2—C11—H11C	109.5
C7—N1—C1	125.01 (13)	H11A—C11—H11C	109.5
N2—N1—C1	119.24 (13)	H11B—C11—H11C	109.5
C9—N2—N1	105.45 (12)	N3—C12—C13	121.58 (17)
C9—N2—C11	119.28 (14)	N3—C12—H12	119.2
N1—N2—C11	114.71 (14)	C13—C12—H12	119.2
C12—N3—C8	121.20 (15)	C18—C13—C14	118.58 (16)
C6—C1—C2	119.78 (17)	C18—C13—C12	119.30 (16)
C6—C1—N1	119.10 (15)	C14—C13—C12	122.11 (15)
C2—C1—N1	121.11 (16)	C15—C14—C13	120.02 (16)
C3—C2—C1	119.65 (19)	C15—C14—H14	120.0
C3—C2—H2	120.2	C13—C14—H14	120.0
C1—C2—H2	120.2	C16—C15—C14	121.13 (17)
C4—C3—C2	120.64 (19)	C16—C15—H15	119.4
C4—C3—H3	119.7	C14—C15—H15	119.4
C2—C3—H3	119.7	C17—C16—C15	118.93 (17)
C3—C4—C5	119.6 (2)	C17—C16—H16	120.5
C3—C4—H4	120.2	C15—C16—H16	120.5
C5—C4—H4	120.2	C16—C17—C18	120.61 (16)
C6—C5—C4	120.6 (2)	C16—C17—O2	123.25 (16)
C6—C5—H5	119.7	C18—C17—O2	116.10 (16)
C4—C5—H5	119.7	C17—C18—C13	120.72 (17)
C5—C6—C1	119.70 (18)	C17—C18—H18	119.6
C5—C6—H6	120.1	C13—C18—H18	119.6
C1—C6—H6	120.1	C20—C19—C24	120.94 (18)
O1—C7—N1	123.73 (16)	C20—C19—O2	120.53 (17)
O1—C7—C8	131.93 (16)	C24—C19—O2	118.42 (17)
N1—C7—C8	104.32 (14)	C21—C20—C19	120.2 (2)
C9—C8—N3	121.71 (15)	C21—C20—H20	119.9

C9—C8—C7	108.38 (15)	C19—C20—H20	119.9
N3—C8—C7	129.89 (14)	C22—C21—C20	120.4 (2)
C8—C9—N2	111.07 (15)	C22—C21—H21	119.8
C8—C9—C10	128.06 (16)	C20—C21—H21	119.8
N2—C9—C10	120.86 (14)	C21—C22—C23	120.5 (2)
C9—C10—H10A	109.5	C21—C22—H22	119.8
C9—C10—H10B	109.5	C23—C22—H22	119.8
H10A—C10—H10B	109.5	C22—C23—C24	120.1 (2)
C9—C10—H10C	109.5	C22—C23—H23	120.0
H10A—C10—H10C	109.5	C24—C23—H23	120.0
H10B—C10—H10C	109.5	C19—C24—C23	117.9 (2)
N2—C11—H11A	109.5	C19—C24—H24	121.1
N2—C11—H11B	109.5	C23—C24—H24	121.1

Fig. 1


C10
C11
C9
N2
C15
C14
N3
C16
C8
N1
C2
C21
C20
C17
C13
C12
C7
C1
C3
C19
C18
O1
C22
C6
C4
O2
C24
C5
C23