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(*E*)-4-[3-Ethoxy-4-(2-phenoxyethoxy)benzylideneamino]-1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one

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The title compound, $C_{28}H_{29}N_3O_4$, was prepared by the reaction of 4-amino-1,5-dimethyl-2-phenylpyrazol-3-one and 3-ethoxy-4-(2-phenoxyethoxy)benzaldehyde. The vanillin group makes dihedral angles of 73.08 (8) and 66.54 (6)° with the planes of the two terminal phenyl rings, and an angle of 30.49 (6)° with the pyrazolone ring plane. The crystal structure is stabilized by intermolecular C—H···O hydrogen bonds that form centrosymmetric dimers.

Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.004 Å
R factor = 0.045
wR factor = 0.130
Data-to-parameter ratio = 13.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

Metal complexes based on Schiff bases have received a good deal of attention in the development of coordination chemistry because of their significant biological activity (Kahwa *et al.*, 1986). Consequently, a significant effort has been devoted to the synthesis of Schiff base derivatives to develop protein and enzyme mimics (Santos *et al.*, 2001). Among the large number of compounds, 4-amino-1,5-dimethyl-2-phenylpyrazol-3-one forms a variety of Schiff bases with aldehydes, and the synthesis and crystal structures of some them, such as (*E*)-(4)-(3-ethoxy-4-hydroxybenzylideneamino)-1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one (Han & Zhen, 2005) and (*E*)-4-[4-(4-chlorobenzyloxy)-3-ethoxybenzylideneamino]-1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one (Zhang *et al.*, 2006) have been reported. We report here the synthesis and structure of the title compound, (I)

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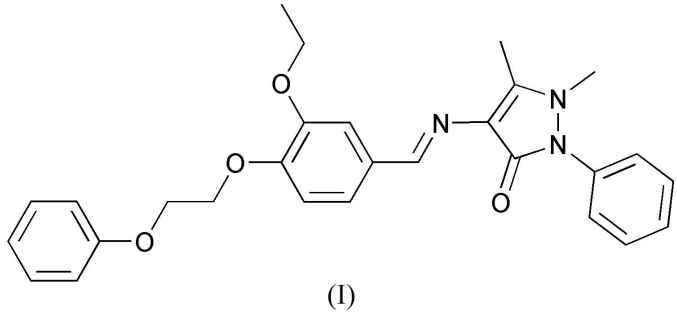

(I)

In the title molecule (Fig. 1), bond lengths and angles are within normal ranges (Allen *et al.*, 1987). The pyrazolone ring (C18–C20/N2/N3/O4) is almost planar, with an r.m.s. deviation for fitted atoms of 0.036 Å. It makes a dihedral angle of 46.35 (7)° with the attached phenyl ring (C23–C28). The vanillin group (C9/C10/C13–C17/O2/O3) is planar, with an r.m.s. deviation for fitted atoms of 0.014 Å. This group makes dihedral angles of 30.49 (6), 73.08 (8) and 66.54 (6)°, respectively, with the pyrazolone ring (C18–C20/N2/N3/O4) and the terminal C1–C6 and C23–C28 phenyl rings, respectively.

The crystal structure is stabilized by weak non-classical intermolecular C—H···O═C hydrogen bonds (Table 1) that form centrosymmetric dimers (Fig. 2).

Experimental

An anhydrous ethanol solution (50 ml) of 3-ethoxy-4-(2-phenoxyethoxy)benzaldehyde (2.86 g, 10 mmol) was added to an anhydrous ethanol solution (50 ml) of 4-amino-1,5-dimethyl-2-phenylpyrazol-3-one (2.03 g, 10 mmol) and the mixture stirred at 350 K for 3 h under N_2, giving a yellow precipitate. The product was isolated, recrystallized from ethanol, and then dried in a vacuum to give pure compound (I) in 84% yield. Yellow single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an ethanol solution.

Crystal data

$C_{28}H_{29}N_3O_4$
M_r = 471.54
Triclinic, $P\overline{1}$
a = 9.804 (6) Å
b = 10.226 (7) Å
c = 13.055 (8) Å
α = 99.985 (11)°
β = 90.178 (11)°
γ = 106.223 (10)°
V = 1235.8 (13) Å^3
Z = 2
D_x = 1.267 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 294 (2) K
Block, yellow
0.30 × 0.24 × 0.18 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.961, T_{max} = 0.985
6294 measured reflections
4325 independent reflections
2786 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.130
S = 1.01
4325 reflections
319 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0597P)^2 + 0.2487P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C17—H17···O4	0.93	2.42	3.049 (3)	125
C27—H27···O4^{i}	0.93	2.56	3.341 (3)	142

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

H atoms were included at calculated positions and refined using a riding model. Constrained bond lengths and U_{iso}(H) parameters: 0.93 Å and $1.2U_{eq}$(C) for aromatic, 0.97 Å and $1.2U_{eq}$(C) for methylene, 0.96 Å and $1.5U_{eq}$(C) for methyl H atoms. The U^{ij} components of atom C12 were restrained to approximate isotropic behaviour.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

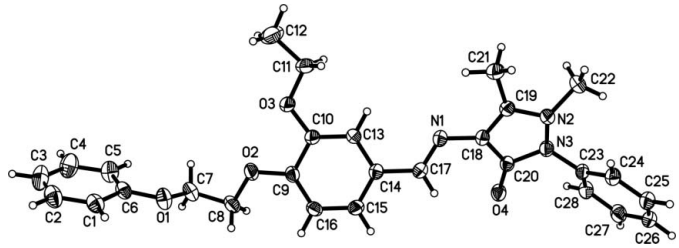

Figure 1
The structure of (I), with displacement ellipsoids for non-H atoms drawn at the 30% probability level.

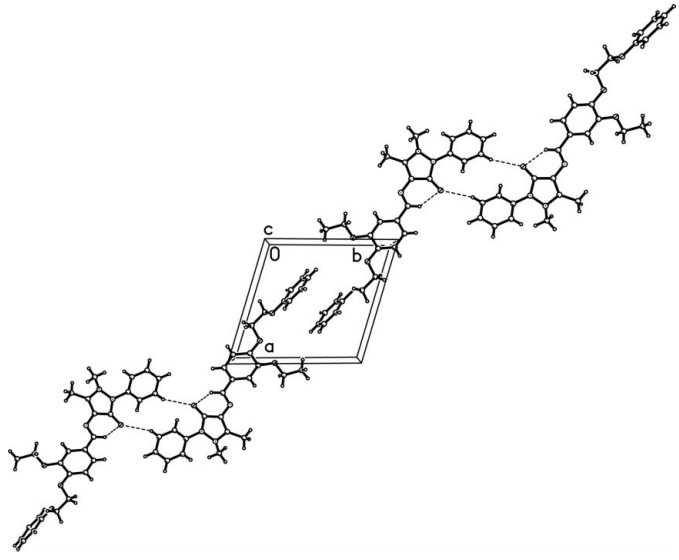

Figure 2
A partial packing diagram for (I), with hydrogen bonds shown as dashed lines.

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