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(*E*)-4-{2-[2-(2,4-Dinitrophenoxy)ethoxy]-3-methoxybenzylideneamino}-1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one

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In the title compound, $C_{27}H_{25}N_5O_8$, the *o*-vanillin (2-hydroxy-3-methoxybenzaldehyde) group makes dihedral angles of 10.42 (9), 33.90 (9) and 61.62 (8)° with the pyrazolone ring, the terminal dinitrobenzene ring and the terminal phenyl ring, respectively. The crystal packing is stabilized by an intermolecular C—H···O=C hydrogen bond that forms a centrosymmetric dimer, together with an intermolecular C—H···O=N hydrogen bond.

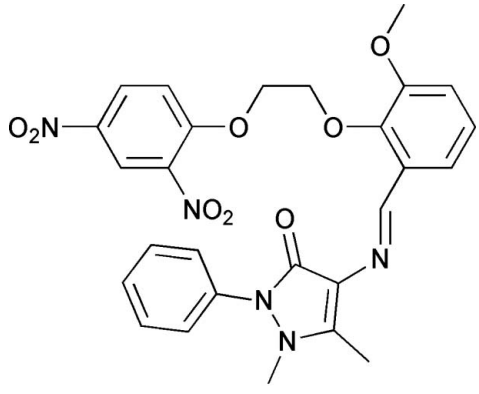
Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.004 Å
R factor = 0.045
wR factor = 0.110
Data-to-parameter ratio = 12.4

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

Comment

There has been steady growth of interest in the structure and reactivity of Schiff bases owing to their potential biological activities such as antibacterial and antitumor (Klayman *et al.*, 1979). 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 of them, such as 4-[(2-hydroxy-3-methoxybenzylidene)amino]-1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one (Diao *et al.*, 2005) and (*E*)-4-[3-(4-chlorobenzyloxy)-4-methoxybenzylideneamino]-1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one (Duan *et al.*, 2006), have been reported. We report here the synthesis and structure of the title compound, (I) (Fig. 1).



(I)

The bond lengths and angles in (I) are within normal ranges (Allen *et al.*, 1987). The pyrazolone ring (C17–C19/N3/N4/N5/O8) is almost planar, with an r.m.s. deviation for fitted atoms of 0.0205 Å. It makes a dihedral angle of 54.15 (8)° with the attached phenyl ring (C22–C27). The *o*-vanillin unit (C9–C14/C16/O6/O7) is planar, with an r.m.s. deviation for fitted atoms of 0.0334 Å, and makes dihedral angles of 10.42 (9), 33.90 (9) and 61.62 (8)° with the pyrazolone ring, the terminal C1–C6 benzene ring and the terminal phenyl ring, respectively. The

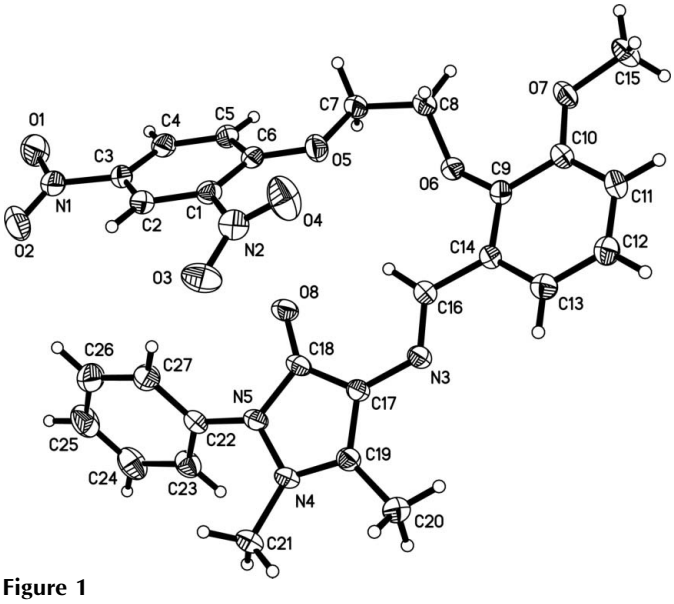


Figure 1
The molecular structure of (I), with displacement ellipsoids for non-H atoms drawn at the 30% probability level.

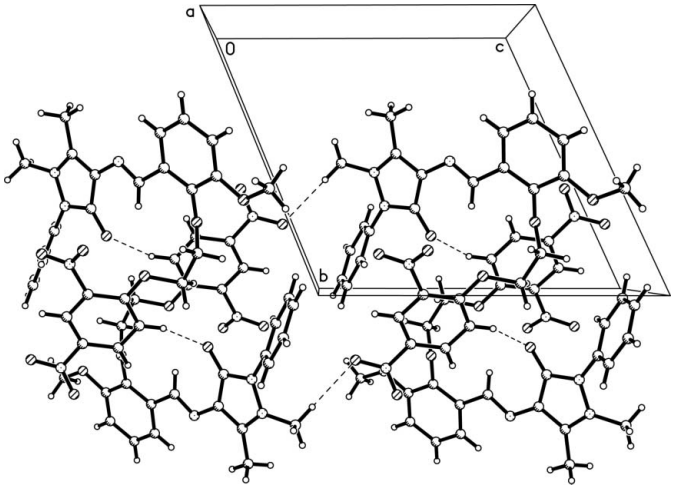


Figure 2
Partial packing diagram of (I), viewed along the *a* axis, showing intermolecular C—H···O hydrogen bonds (dashed lines).

crystal packing is stabilized by a weak non-classical intermolecular C5—H5···O8^{ii}═C18^{ii} hydrogen bond that forms a centrosymmetric dimer (Fig. 2), together with an intermolecular C21—H21*C*···O2^{i}═N1^{i} hydrogen bond (symmetry codes as given in Table 1).

Experimental

An anhydrous ethanol solution (30 ml) of 2-[2-(2,4-dinitrophenoxy)ethoxy]-3-methoxybenzaldehyde (3.62 g, 10 mmol) was added to an anhydrous ethanol solution (30 ml) of 4-amino-1,5-dimethyl-2-phenylpyrazol-3-one (2.03 g, 10 mmol) and the mixture was stirred at 350 K for 5 h under nitrogen, giving a yellow precipitate. The product was isolated and recrystallized from acetonitrile, and then dried in a vacuum to give the pure compound (I) in 85% yield. Yellow single crystals suitable for X-ray analysis were obtained by slow evaporation of an acetonitrile solution.

Crystal data

$C_{27}H_{25}N_5O_8$
M_r = 547.52
Triclinic, $P\overline{1}$
a = 9.307 (7) Å
b = 12.130 (10) Å
c = 12.718 (10) Å
α = 67.920 (13)°
β = 89.065 (13)°
γ = 77.497 (12)°
V = 1295.6 (18) Å^3
Z = 2
D_x = 1.404 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 294 (2) K
Block, yellow
0.24 × 0.20 × 0.16 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.958, T_{max} = 0.983
6619 measured reflections
4537 independent reflections
2634 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.110
S = 1.07
4537 reflections
365 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0418P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0196 (16)

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

D—H···A	D—H	H···A	D···A	D—H···A
C21—H21*C*···O2^{i}	0.96	2.50	3.463 (4)	179
C5—H5···O8^{ii}	0.93	2.28	3.093 (3)	146

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

H atoms were included in calculated positions (C—H = 0.93-0.97 Å) and refined as riding, with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C).

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*.

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