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(*E*)-2-[2-(2,3-Dimethyl-5-oxo-1-phenyl-2,5-dihydro-1*H*-pyrazol-4-yliminomethyl)-phenoxy]acetic acid monohydrate

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The title compound, $C_{20}H_{19}N_3O_4\cdot H_2O$, has been synthesized by the reaction of 2-(2-formylphenoxy)acetic acid with 4-aminoantipyrine in ethanol. It comprises a 4-aminoantipyrine Schiff base and a water molecule. The dihedral angles made by the pyrazoline ring with the substituted and unsubstituted phenyl rings are 14.8 (2) and 58.1 (2)°, respectively. In the crystal structure, the molecules are held together by intermolecular O—H···O hydrogen bonds and weak C—H···O, intermolecular hydrogen bonds, as well as π–π stacking interactions.

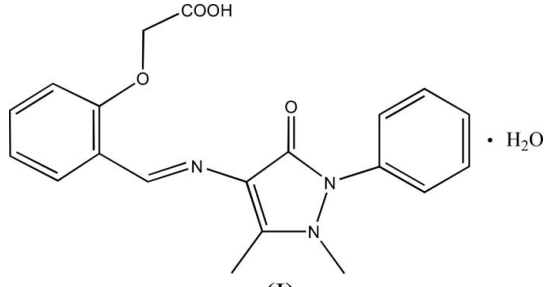
Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.004 Å
R factor = 0.052
wR factor = 0.126
Data-to-parameter ratio = 12.2

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

Comment

4-Aminoantipyrine and its derivatives are important compounds in pharmacology and biochemistry. They are especially used as anti-inflammatory drugs (Hökelek *et al.*, 2002). Aryloxyacetic acids are widely used as herbicides, and some of them have acute toxicity (Zhang & Chen, 2001). Recently, the crystal structures of two 4-aminoantipyrine Schiff bases with 2-(2-formylphenoxy)acetic acid, (II) (You *et al.*, 2003) and (III) (You *et al.*, 2004), have been reported. We report here the third structure of the 4-aminoantipyrine Schiff base with 2-(2-formylphenoxy)acetic acid, (I).



(I)

Compound (I) crystallizes as a monohydrate, (II) as the nitrate salt and (III) as a methanol solvate. All three compounds adopt an (*E*)-configuration about the C=N bond. The C=O bond of the pyrazoline ring and the carboxymethyl group lie on the same side of the molecule in (I) and (III), but on opposite sides in (II). In (I), all the non-H atoms of the 2-(2-formylphenoxy)acetic acid group and atoms C1 and N3 are nearly coplanar. The C=N bond length in (I) is 1.280 (3) Å, which is in accord with (III) [1.2805 (4) Å] and shorter than in (II) [1.304 (5) Å], consistent with the protonation of the imino N atom in (II). The pyrazoline ring in (I) is nearly planar, with a mean deviation from the plane of 0.030 (2) Å. Atoms C13, C14 and C15 deviate from the pyrazoline mean plane by 0.13 (3), 0.571 (3) and −0.563 (3) Å, respectively. The dihedral

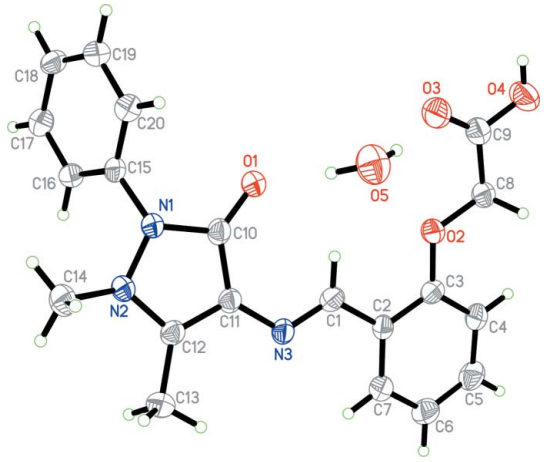


Figure 1
The asymmetric unit of (I), showing 30% probability displacement ellipsoids.

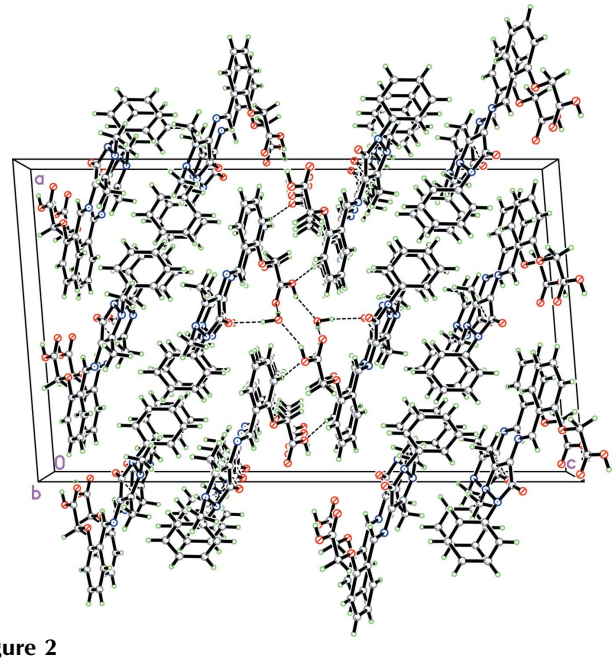


Figure 2
The packing of (I), viewed along the *b* axis; hydrogen bonds are shown as dashed lines.

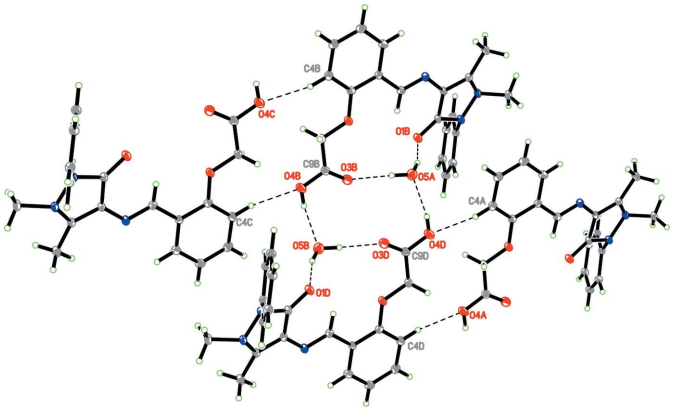


Figure 3
Hydrogen bonds in the packing of (I), viewed along the *b* axis; Hydrogen bonds are shown as dashed lines.

angles between the pyrazoline ring and the substituted and unsubstituted phenyl rings are 14.8 (2) and 58.1 (2)°, respectively. The crystal structure is stabilized by π–π stacking interactions (Fig. 2) (the distance of the offset face-to-face π–π stacking interactions is about 3.8 Å, and about 3.6 Å for the edge-to-face interactions), and by O—H···O intermolecular hydrogen bonds and weak intermolecular C—H···O hydrogen bonds (Table 2 and Fig. 3).

Experimental

2-(2-Formylphenoxy)acetic acid (1 mmol, 0.180 g) and 4-aminoantipyrine (1 mmol, 0.203 g) were dissolved in ethanol (25 ml) and heated for 2 h under reflux. When the solution was cooled, a yellow solid was precipitated with a yield of 84% (m.p. 466.2–466.9 K). The crude solid was washed with ethanol and light petroleum, and then dissolved in 95% ethanol. After keeping the resulting solution in air for a week, single crystals suitable for X-ray diffraction analysis precipitated.

Crystal data

$C_{20}H_{19}N_3O_4\cdot H_2O$
M_r = 383.40
Monoclinic, $C2/c$
a = 18.0213 (15) Å
b = 6.8755 (6) Å
c = 30.352 (3) Å
β = 94.518 (2)°
V = 3749.1 (6) Å^3
Z = 8
D_x = 1.359 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 296 (2) K
Block, yellow
0.20 × 0.14 × 0.08 mm

Data collection

Bruker APEX-II area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
T_{min} = 0.98, T_{max} = 0.99
10093 measured reflections
3231 independent reflections
1522 reflections with $I > 2\sigma(I)$
R_{int} = 0.068
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.127
S = 1.01
3231 reflections
265 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0499P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Extinction correction: *APEX2*
Extinction coefficient: 0.0009 (2)

Table 1
Selected geometric parameters (Å, °).

O1—C10	1.238 (3)	N2—C14	1.457 (3)
N1—N2	1.403 (3)	N3—C1	1.280 (3)
N1—C10	1.405 (3)	N3—C11	1.395 (3)
N1—C15	1.432 (3)	C1—C2	1.468 (4)
N2—C12	1.364 (3)	C11—C12	1.370 (4)
N2—N1—C10	109.0 (2)	N1—N2—C14	116.5 (2)
N2—N1—C15	119.4 (2)	C1—N3—C11	119.9 (3)
C10—N1—C15	121.4 (2)	N3—C1—C2	122.0 (3)
C12—N2—N1	107.3 (2)	O1—C10—N1	122.1 (3)
C12—N2—C14	124.5 (3)	O1—C10—C11	132.6 (3)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4A···O5^{i}	0.82	1.94	2.688 (4)	151
O5—H5B···O1	0.89 (2)	1.96 (3)	2.831 (4)	166 (4)
O5—H5A···O3	0.88 (2)	1.95 (3)	2.823 (4)	173 (4)
C4—H4···O4^{ii}	0.93	2.48	3.383 (4)	163
C13—H13C···O1^{iii}	0.96	2.51	3.459 (4)	172

Symmetry codes: (i) $-x, -y+2, -z+2$; (ii) $-x+\frac{1}{2}, -y+\frac{5}{2}, -z+2$; (iii) $x, y-1, z$.

H atoms attached to water O atoms were found in difference density maps and refined with restraints to make plausible H···O hydrogen bonds [H···O = 0.82(2) Å and H···H = 1.45(2) Å], whilst maintaining the H—O—H bond angle of 107.5°. All other H atoms were positioned in idealized locations and refined as riding on their carrier atoms, with C—H distances of 0.93 (aromatic), 0.97 (methylene) and 0.96 (methyl) Å, O—H = 0.82 Å (carboxyl), and U_{iso}(H) = $1.2U_{eq}$(C) for aromatic and methylene H atoms and $1.5U_{eq}$(C,O) for other H atoms.

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2*; data reduction: *APEX2*; program(s) used to solve structure: *APEX2*; program(s) used to refine structure: *APEX2*; molecular graphics: *APEX2*; software used to prepare material for publication: *APEX2*.

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References

Bruker (2004). *APEX2* (Version 1.22) and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Hökelek, T., Kilic, Z., Isiklan, M. & Hayvali, M. (2002). *Anal. Sci.* **18**, 215–216.
You, Z.-L., Zhu, H.-L. & Liu, W.-S. (2004). *Acta Cryst.* E**60**, o801–o803.
You, Z.-L., Zhu, H.-L., Liu, W.-S. & Tan, M.-Y. (2003). *Acta Cryst.* E**59**, o1920–o1922.
Zhang, L.-X. & Chen, Y.-W. (2001). *J. Instrum. Anal.* **20**, 35–38.