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5-Carboxylato-5-hydroxy-4-(4-methoxybenzoyl)-3-(4-methoxyphenyl)-1,1-dimethyl-4,5-dihydro-1*H*-pyrazol-1-ium monohydrate

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.003 Å
R factor = 0.047
wR factor = 0.130
Data-to-parameter ratio = 14.6

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

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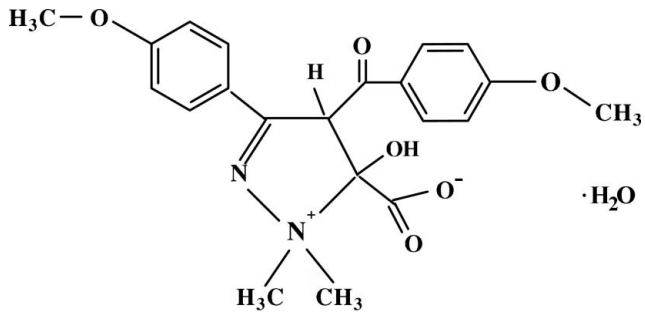
In the title compound, $C_{21}H_{22}N_2O_6\cdot H_2O$, the pyrazolium ring is in an envelope conformation. In the crystal structure, intermolecular O—H···O hydrogen bonds form molecular tapes along [001]. In addition, weak C—H···O and a C—H···π interaction link molecules into a three-dimensional network.

Comment

Pyrazoles are one of the important classes of biologically active compounds. Some pyrazoles have been reported to possess significant antimicrobial (Mahajan *et al.*, 1991), antiviral (Baraldi *et al.*, 1998), antifungal (Chen & Li, 1998), pesticidal (Londershausen, 1996), antihistaminic (Mishra *et al.*, 1998) and antidepressant activities (Bailley *et al.*, 1985). In view of these important properties, we have undertaken the X-ray diffaction study of the title compound, (I).



(I)

One benzene ring (C8–C13) and the attached methoxy group (C14 and O5) are essentionally in the same plane (r.m.s. deviation = 0.099 Å) are are rotated by 7.63 (3)° from the adjacent carbonyl group plane, while the methoxy substituent (O6 and C21) of the other methoxyphenyl group is rotated by 6.2 (3) Å from the benzene ring (C15–C20). The two benzene rings in the molecule form a dihedral angle of 82.5 (6) Å. The pyrazole ring in (I) is in an envelope conformation, with atoms N1, N2, C2 and C3 coplanar (r.m.s. deviation = 0.002 Å), and with atom C1 forming the flap, 0.421 (3) Å from this plane. The bond lengths and angles in the five-membered ring are normal (Allen *et al.*, 1987).

In the crystal structure, intermolecular O—H···O hydrogen bonds form centrosymmetric dimers which are linked by further O—H···O hydrogen bonds involving water molecules to form molecular tapes along [001] (Fig. 2). In addition, several weak C—H···O interactions and a C—H···π interaction link molecules into a three-dimensional network (Table 2).

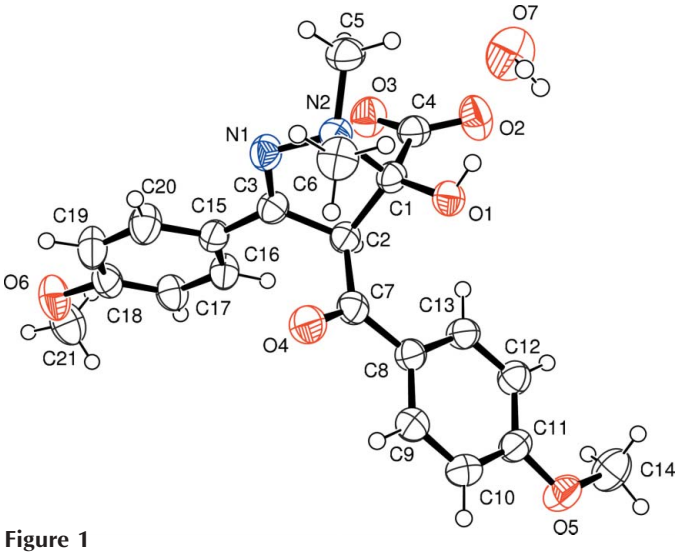


Figure 1
An *ORTEP-3* (Farrugia, 1997) drawing of the title compound, (I), showing the atomic numbering scheme. Displacement ellipsoids of non-H atoms are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

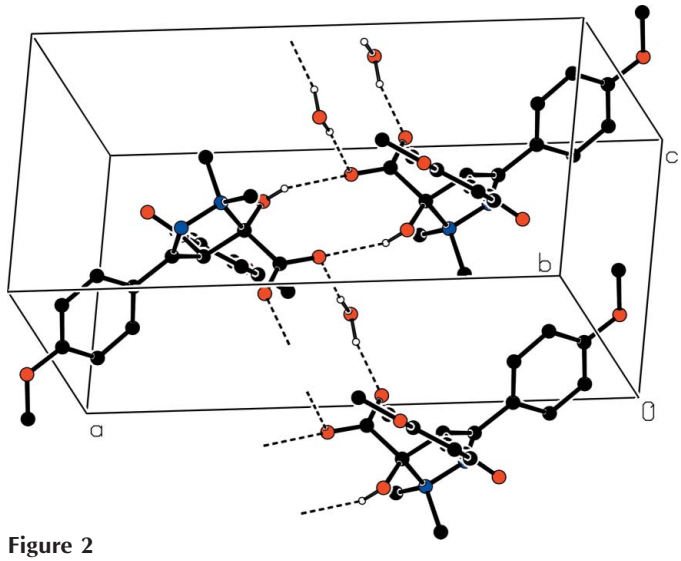


Figure 2
Packing diagram (Spek, 2003), showing intermolecular O—H···O hydrogen bonds as dashed lines.

Experimental

An equimolar mixture of 4-(4-methoxybenzoyl)-5-(4-methoxyphenyl)-2,3-dihydrofuran-2,3-dione (0.60 g) [easily obtained from oxalyl dichloride and *p,p′*-dimethoxydibenzoylmethane (Hökelek *et al.*, 2002), and also described by Ziegler *et al.* (1967)] and *N,N*-dimethylhydrazine (0.14 ml) was stirred in benzene (30 ml) for 24 h at room temperature. The white precipitate which formed was filtered off and recrystallized from 2-propanol (yield 0.70 g, 98%; m.p. 402–403 K). Solvents were dried by refluxing with the appropriate drying agents and distilled before use. All other reagents were purchased from Merck, Fluka, Aldrich and Acros Chemical Co., and used without further purification.

Crystal data

$C_{21}H_{22}N_2O_6\cdot H_2O$
M_r = 416.42
Monoclinic, $P2_1/c$
a = 16.5325 (8) Å
b = 16.0283 (11) Å
c = 7.8717 (4) Å
β = 94.896 (4)°
V = 2078.3 (2) Å^3
Z = 4
D_x = 1.331 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 18802 reflections
θ = 1.8–28.0°
μ = 0.10 mm^{-1}
T = 296 K
Plate, colorless
0.49 × 0.37 × 0.18 mm

Data collection

Stoe IPDS-2 diffractometer
ω scans
19824 measured reflections
4084 independent reflections
2753 reflections with $I > 2\sigma(I)$
R_{int} = 0.059
θ_{max} = 26.0°
$h = -20 \rightarrow 20$
$k = -19 \rightarrow 19$
$l = -9 \rightarrow 9$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.130
S = 1.02
4084 reflections
280 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0745P)^2 + 0.0106P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.71 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0073 (16)

Table 1
Selected geometric parameters (Å, °).

O2—C4	1.236 (3)	N1—N2	1.446 (2)
O3—C4	1.235 (3)	N2—C1	1.585 (3)
C11—O5—C14	116.86 (19)	O3—C4—O2	129.3 (2)
C18—O6—C21	117.72 (19)	C8—C7—C2	119.50 (18)
C3—C2—C7	111.63 (16)	O5—C11—C12	124.7 (2)
C7—C2—C1	114.51 (15)	C16—C15—C3	121.11 (18)
N1—C3—C15	120.87 (18)	O6—C18—C17	124.6 (2)
C4—C1—C2—C7	146.42 (17)	O5—C11—C12—C13	179.2 (2)
N2—N1—C3—C15	−179.58 (17)	C7—C8—C13—C12	180.0 (2)
O1—C1—C4—O3	173.61 (18)	N1—C3—C15—C20	−23.5 (3)
O4—C7—C8—C13	−172.4 (2)	C3—C15—C16—C17	−175.7 (2)
C2—C7—C8—C9	−170.85 (19)	C16—C17—C18—O6	178.6 (2)
C14—O5—C11—C10	178.7 (2)	C3—C15—C20—C19	177.2 (2)
C9—C10—C11—O5	179.2 (2)		

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

D—H···A	D—H	H···A	D···A	D—H···A
C6—H6A···O4	0.96	2.46	3.316 (3)	148
C5—H5A···O3	0.96	2.45	3.101 (3)	125
C16—H16···O5^{i}	0.93	2.54	3.465 (3)	172
O7—H7B···O2^{ii}	0.93 (2)	2.01 (2)	2.923 (3)	169 (3)
O7—H7A···O3^{iii}	0.93 (2)	1.90 (2)	2.809 (3)	167 (4)
C5—H5C···O3^{iv}	0.96	2.59	3.339 (3)	136
C21—H21C···O4^{v}	0.96	2.59	3.321 (3)	134
O1—H1···O2^{ii}	0.82	2.21	2.788 (2)	128
C14—H14C···O1^{i}	0.96	2.49	3.142 (3)	125
C21—H21B···$Cg1^{vi}$	0.96	3.01	3.9016	154

Symmetry codes: (i) $x, -y+\frac{3}{2}, z+\frac{1}{2}$; (ii) $-x+1, -y+1, -z+1$; (iii) $x, y, z-1$; (iv) $x, -y+\frac{1}{2}, z-\frac{1}{2}$; (v) $x, y, z+1$; (vi) $-x, -y+1, -z+2$.

The H atoms of the water molecule were refined independently with isotropic displacement parameters. Other H atoms were positioned geometrically and treated using a riding-model approximation, with C—H = 0.93–0.98 Å and O—H = 0.82 Å. The displacement parameters of the H atoms were constrained to U_{iso}(H) = 1.2U_{eq}(C,O) or 1.2U_{eq}(methyl C).

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 2003).

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