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4-(3,5-Dimethyl-1*H*-pyrazol-1-yl)-phthalazin-1(2*H*)-one monohydrate

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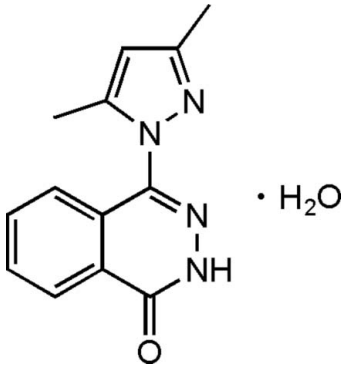
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.048; wR factor = 0.145; data-to-parameter ratio = 13.0.

In the title compound, $C_{13}H_{12}N_4O\cdot H_2O$, N···H—O and O···H—O hydrogen bonds involving the uncoordinated water molecules link the molecules into two-dimensional networks parallel to the (101) planes.

Related literature

For related literature, see: Elguero (1984); Fun *et al.* (1996); Liao *et al.* (2000); Lu *et al.* (1996);



Experimental

Crystal data

$C_{13}H_{12}N_4O\cdot H_2O$
M_r = 258.28
Monoclinic, $P2_1/n$
a = 7.6782 (12) Å
b = 14.844 (2) Å
c = 11.2565 (16) Å
β = 96.348 (2)°
V = 1275.1 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 298 (2) K
0.54 × 0.48 × 0.45 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.951, T_{max} = 0.959
6118 measured reflections
2243 independent reflections
1398 reflections with $I > 2\sigma(I)$
R_{int} = 0.053

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.145
S = 1.02
2243 reflections
172 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H1···O1	0.85	1.93	2.781 (3)	173
O2—H3···N4^{i}	0.85	2.03	2.870 (3)	169
N2—H2···O2^{ii}	0.86	1.94	2.789 (3)	170

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

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; 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: BI2237).

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supplementary materials

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4-(3,5-Dimethyl-1*H*-pyrazol-1-yl)phthalazin-1(2*H*)-one monohydrate

Z. Kai, X.-H. Yin, F. Yu and Z. Jie

Comment

The chemical and pharmacological properties of pyrazoles have been investigated extensively, owing to their chelating ability with metal ions and their potentially beneficial chemical and biological activities (Elguero, 1984; Fun *et al.*, 1996; Liao *et al.*, 2000; Lu *et al.*, 1996). As part of our studies on the synthesis and characterization of these compounds, we report here the synthesis and crystal structure of the title compound.

Experimental

A solution of 4-hydrazinylphthalazin-1(2*H*)-one (10 mmol) in 50 ml toluene was added to a solution of pentane-2,4-dione (10 mmol) in 10 ml toluene. The reaction mixture was refluxed for 1 h with stirring. The resulting pale yellow precipitate was isolated by filtration, washed several times with ethanol and dried *in vacuo* (yield 90%). Single crystals suitable for X-ray diffraction analysis were obtained by slow evaporation of a methanol solution of the compound.

Refinement

H atoms were placed geometrically with O—H = 0.85 Å, N—H = 0.86 Å and C—H in the range 0.93–0.96 Å, and refined using a riding model, with U_{iso}(H) = 1.2 U_{eq}(C/N) or 1.5U_{eq}(*O*/methyl C).

Figures

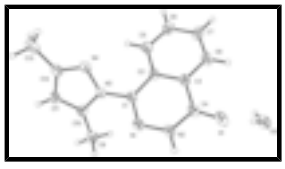

Fig. 1. The molecular structure of the title compound showing displacement ellipsoids at 50% probability for non-H atoms.

4-(3,5-Dimethyl-1*H*-pyrazol-1-yl)phthalazin-1(2*H*)-one monohydrate

Crystal data

$C_{13}H_{12}N_4O·H_2O$	F_{000} = 544
M_r = 258.28	D_x = 1.345 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 1490 reflections
a = 7.6782 (12) Å	θ = 2.3–23.9º
b = 14.844 (2) Å	μ = 0.10 mm^{-1}
c = 11.2565 (16) Å	T = 298 (2) K

$\beta = 96.348\ (2)^{\circ}$	Block, colorless
$V = 1275.1\ (3)\ Å^3$	0.54 × 0.48 × 0.45 mm
$Z = 4$	

Data collection

Bruker SMART CCD diffractometer	2243 independent reflections
Radiation source: fine-focus sealed tube	1398 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.053$
$T = 298(2)$ K	$\theta_{max} = 25.0^{\circ}$
φ and ω scans	$\theta_{min} = 2.3^{\circ}$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -9 \rightarrow 9$
$T_{min} = 0.951$, $T_{max} = 0.959$	$k = -17 \rightarrow 17$
6118 measured reflections	$l = -13 \rightarrow 5$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.048$	H-atom parameters constrained
$wR(F^2) = 0.145$	$w = 1/[\sigma^2(F_o^2) + (0.0598P)^2 + 0.3268P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.02$	$(\Delta/\sigma)_{max} < 0.001$
2243 reflections	$\Delta\rho_{max} = 0.18$ e Å^{-3}
172 parameters	$\Delta\rho_{min} = -0.22$ 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 $F^2 > 2\text{sigma}(F^2)$ 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}
N1	1.0662 (3)	0.86029 (14)	0.20505 (18)	0.0420 (5)
N2	1.0820 (3)	0.89129 (14)	0.31927 (18)	0.0448 (6)

H2	1.1197	0.9455	0.3300	0.054*
N3	0.9777 (3)	0.75338 (13)	0.06555 (17)	0.0379 (5)
N4	1.0434 (3)	0.67089 (13)	0.03713 (18)	0.0431 (6)
O1	1.0755 (2)	0.88111 (14)	0.51770 (16)	0.0598 (6)
O2	0.8248 (3)	0.92707 (12)	0.66750 (16)	0.0593 (6)
H1	0.9076	0.9137	0.6264	0.071*
H3	0.7326	0.9014	0.6351	0.071*
C1	0.9959 (3)	0.78127 (16)	0.1866 (2)	0.0353 (6)
C2	0.9365 (3)	0.72550 (16)	0.2784 (2)	0.0370 (6)
C3	0.9644 (3)	0.75784 (16)	0.3955 (2)	0.0386 (6)
C4	1.0448 (3)	0.84615 (18)	0.4184 (2)	0.0424 (7)
C5	0.8487 (3)	0.64325 (17)	0.2566 (2)	0.0452 (7)
H5	0.8263	0.6214	0.1790	0.054*
C6	0.7964 (4)	0.59558 (19)	0.3501 (3)	0.0533 (7)
H6	0.7379	0.5412	0.3355	0.064*
C7	0.8287 (4)	0.6267 (2)	0.4664 (3)	0.0567 (8)
H7	0.7944	0.5926	0.5291	0.068*
C8	0.9106 (4)	0.70722 (19)	0.4892 (2)	0.0508 (7)
H8	0.9307	0.7284	0.5672	0.061*
C9	0.8321 (4)	0.89067 (17)	−0.0299 (2)	0.0515 (7)
H9A	0.9225	0.9348	−0.0119	0.077*
H9B	0.7535	0.8919	0.0307	0.077*
H9C	0.7685	0.9040	−0.1062	0.077*
C10	0.9124 (3)	0.79988 (16)	−0.0337 (2)	0.0387 (6)
C11	0.9363 (3)	0.74606 (17)	−0.1278 (2)	0.0447 (7)
H11	0.9054	0.7591	−0.2081	0.054*
C12	1.0164 (3)	0.66706 (17)	−0.0810 (2)	0.0434 (7)
C13	1.0716 (4)	0.5861 (2)	−0.1462 (3)	0.0654 (9)
H13A	1.1515	0.5507	−0.0936	0.098*
H13B	1.1284	0.6049	−0.2138	0.098*
H13C	0.9704	0.5505	−0.1730	0.098*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0479 (13)	0.0410 (12)	0.0370 (12)	−0.0034 (10)	0.0043 (10)	−0.0081 (10)
N2	0.0517 (13)	0.0432 (12)	0.0394 (13)	−0.0055 (10)	0.0049 (10)	−0.0105 (10)
N3	0.0479 (12)	0.0353 (11)	0.0315 (11)	0.0020 (10)	0.0090 (9)	−0.0037 (9)
N4	0.0510 (13)	0.0386 (12)	0.0408 (13)	0.0036 (10)	0.0103 (10)	−0.0051 (10)
O1	0.0635 (13)	0.0744 (14)	0.0405 (12)	−0.0013 (10)	0.0016 (9)	−0.0197 (10)
O2	0.0665 (13)	0.0588 (13)	0.0536 (12)	−0.0145 (10)	0.0105 (10)	−0.0130 (10)
C1	0.0377 (13)	0.0373 (14)	0.0314 (13)	0.0034 (11)	0.0063 (11)	−0.0043 (11)
C2	0.0346 (13)	0.0425 (15)	0.0345 (14)	0.0047 (11)	0.0064 (11)	−0.0013 (11)
C3	0.0374 (13)	0.0453 (15)	0.0339 (14)	0.0082 (12)	0.0072 (11)	−0.0008 (12)
C4	0.0379 (15)	0.0506 (16)	0.0388 (16)	0.0057 (12)	0.0046 (12)	−0.0094 (13)
C5	0.0485 (15)	0.0452 (15)	0.0428 (16)	−0.0031 (13)	0.0093 (13)	−0.0055 (13)
C6	0.0597 (18)	0.0487 (16)	0.0545 (18)	−0.0043 (14)	0.0200 (15)	0.0025 (14)
C7	0.0626 (19)	0.0586 (19)	0.0528 (19)	0.0045 (15)	0.0236 (15)	0.0122 (15)

C8	0.0565 (17)	0.0628 (19)	0.0347 (15)	0.0107 (15)	0.0115 (13)	−0.0010 (13)
C9	0.0610 (18)	0.0439 (16)	0.0490 (17)	0.0037 (13)	0.0029 (14)	0.0026 (13)
C10	0.0406 (14)	0.0395 (14)	0.0361 (14)	−0.0040 (11)	0.0055 (11)	0.0030 (12)
C11	0.0539 (16)	0.0500 (16)	0.0306 (14)	−0.0057 (13)	0.0063 (12)	−0.0023 (12)
C12	0.0454 (15)	0.0447 (16)	0.0411 (16)	−0.0047 (12)	0.0097 (13)	−0.0086 (12)
C13	0.073 (2)	0.063 (2)	0.062 (2)	0.0016 (16)	0.0162 (16)	−0.0248 (16)

Geometric parameters (Å, °)

N1—C1	1.298 (3)	C5—H5	0.930
N1—N2	1.358 (3)	C6—C7	1.385 (4)
N2—C4	1.359 (3)	C6—H6	0.930
N2—H2	0.860	C7—C8	1.362 (4)
N3—C10	1.361 (3)	C7—H7	0.930
N3—N4	1.375 (3)	C8—H8	0.930
N3—C1	1.417 (3)	C9—C10	1.484 (3)
N4—C12	1.325 (3)	C9—H9A	0.960
O1—C4	1.231 (3)	C9—H9B	0.960
O2—H1	0.850	C9—H9C	0.960
O2—H3	0.850	C10—C11	1.356 (3)
C1—C2	1.437 (3)	C11—C12	1.400 (4)
C2—C3	1.397 (3)	C11—H11	0.930
C2—C5	1.403 (3)	C12—C13	1.494 (4)
C3—C8	1.394 (3)	C13—H13A	0.960
C3—C4	1.460 (4)	C13—H13B	0.960
C5—C6	1.366 (4)	C13—H13C	0.960
C1—N1—N2	116.7 (2)	C8—C7—C6	120.1 (3)
N1—N2—C4	127.3 (2)	C8—C7—H7	119.9
N1—N2—H2	116.4	C6—C7—H7	119.9
C4—N2—H2	116.4	C7—C8—C3	120.0 (3)
C10—N3—N4	111.84 (19)	C7—C8—H8	120.0
C10—N3—C1	129.0 (2)	C3—C8—H8	120.0
N4—N3—C1	118.97 (19)	C10—C9—H9A	109.5
C12—N4—N3	104.6 (2)	C10—C9—H9B	109.5
H1—O2—H3	107.5	H9A—C9—H9B	109.5
N1—C1—N3	114.4 (2)	C10—C9—H9C	109.5
N1—C1—C2	124.4 (2)	H9A—C9—H9C	109.5
N3—C1—C2	121.2 (2)	H9B—C9—H9C	109.5
C3—C2—C5	119.0 (2)	C11—C10—N3	105.8 (2)
C3—C2—C1	116.9 (2)	C11—C10—C9	130.5 (2)
C5—C2—C1	124.0 (2)	N3—C10—C9	123.6 (2)
C8—C3—C2	120.1 (2)	C10—C11—C12	107.0 (2)
C8—C3—C4	120.4 (2)	C10—C11—H11	126.5
C2—C3—C4	119.5 (2)	C12—C11—H11	126.5
O1—C4—N2	120.1 (2)	N4—C12—C11	110.8 (2)
O1—C4—C3	125.0 (2)	N4—C12—C13	120.4 (2)
N2—C4—C3	114.9 (2)	C11—C12—C13	128.8 (2)
C6—C5—C2	119.5 (2)	C12—C13—H13A	109.5
C6—C5—H5	120.3	C12—C13—H13B	109.5

C2—C5—H5	120.3	H13A—C13—H13B	109.5
C5—C6—C7	121.3 (3)	C12—C13—H13C	109.5
C5—C6—H6	119.4	H13A—C13—H13C	109.5
C7—C6—H6	119.4	H13B—C13—H13C	109.5
C1—N1—N2—C4	5.5 (4)	C2—C3—C4—O1	−179.9 (2)
C10—N3—N4—C12	−0.4 (3)	C8—C3—C4—N2	−176.2 (2)
C1—N3—N4—C12	−176.2 (2)	C2—C3—C4—N2	1.8 (3)
N2—N1—C1—N3	178.17 (19)	C3—C2—C5—C6	−1.6 (4)
N2—N1—C1—C2	−0.6 (3)	C1—C2—C5—C6	−179.5 (2)
C10—N3—C1—N1	−47.5 (3)	C2—C5—C6—C7	−0.2 (4)
N4—N3—C1—N1	127.5 (2)	C5—C6—C7—C8	1.5 (4)
C10—N3—C1—C2	131.3 (3)	C6—C7—C8—C3	−0.9 (4)
N4—N3—C1—C2	−53.7 (3)	C2—C3—C8—C7	−0.9 (4)
N1—C1—C2—C3	−3.0 (4)	C4—C3—C8—C7	177.1 (2)
N3—C1—C2—C3	178.4 (2)	N4—N3—C10—C11	0.1 (3)
N1—C1—C2—C5	174.9 (2)	C1—N3—C10—C11	175.4 (2)
N3—C1—C2—C5	−3.7 (4)	N4—N3—C10—C9	179.6 (2)
C5—C2—C3—C8	2.2 (4)	C1—N3—C10—C9	−5.1 (4)
C1—C2—C3—C8	−179.8 (2)	N3—C10—C11—C12	0.2 (3)
C5—C2—C3—C4	−175.8 (2)	C9—C10—C11—C12	−179.2 (3)
C1—C2—C3—C4	2.2 (3)	N3—N4—C12—C11	0.5 (3)
N1—N2—C4—O1	175.6 (2)	N3—N4—C12—C13	179.9 (2)
N1—N2—C4—C3	−6.0 (3)	C10—C11—C12—N4	−0.5 (3)
C8—C3—C4—O1	2.1 (4)	C10—C11—C12—C13	−179.8 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H1···O1	0.85	1.93	2.781 (3)	173
O2—H3···N4^{i}	0.85	2.03	2.870 (3)	169
N2—H2···O2^{ii}	0.86	1.94	2.789 (3)	170

Symmetry codes: (i) *x*−1/2, −*y*+3/2, *z*+1/2; (ii) −*x*+2, −*y*+2, −*z*+1.

Fig. 1