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2-[(4-Methylbenzoyl)hydrazono]-propionic acid monohydrate

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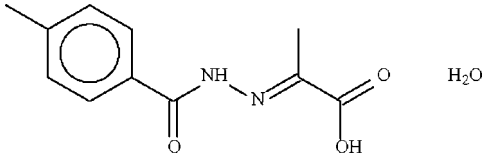
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; R factor = 0.030; wR factor = 0.083; data-to-parameter ratio = 7.8.

In the title compound, $C_{11}H_{12}N_2O_3\cdot H_2O$, the water molecule is a hydrogen-bond donor to the double-bond amide and the carbonyl O atoms of two acid molecules; it is also a hydrogen-bond acceptor to the acid –OH and amide –NH– groups. These hydrogen-bonding interactions give rise to a layer structure, with the layers parallel to the *ab* plane.

Related literature

The deprotonated anion of 2-aroylhydrazonopropionic acid furnishes a number of metal complexes; see, for example: Wu, Chen *et al.* (2006); Liu *et al.* (2007); Wu & Zeng (2007); Wu *et al.* (2006*a*,*b*); Yang *et al.* (2004); Yin & Chen (2006); Zhai *et al.* (2007).



Experimental

Crystal data

$C_{11}H_{12}N_2O_3\cdot H_2O$
M_r = 238.24
Monoclinic, $P2_1$
a = 6.8464 (1) Å
b = 11.9753 (2) Å
c = 7.0005 (1) Å
β = 102.169 (1)°
V = 561.06 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 100 (2) K
0.20 × 0.10 × 0.10 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: none
5272 measured reflections
1335 independent reflections
1211 reflections with $I > 2\sigma(I)$
R_{int} = 0.029

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.083
S = 1.02
1335 reflections
172 parameters
5 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}
Absolute structure: 1126 Friedel pairs were merged

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O1W	0.83 (2)	2.03 (2)	2.777 (2)	149 (3)
O1W—H11···O3	0.84 (2)	1.97 (2)	2.794 (2)	165 (4)
O1W—H12···O2^{i}	0.84 (2)	2.00 (1)	2.829 (2)	168 (3)
N2—H2···O1W^{ii}	0.87 (2)	2.35 (1)	3.210 (2)	168 (3)

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2007); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2513).

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supplementary materials

Acta Cryst. (2009). E**65**, o419 [**doi:10.1107/S1600536809003067**]

2-[(4-Methylbenzoyl)hydrazono]propionic acid monohydrate

H. W. Wong, K. M. Lo and S. W. Ng

Experimental

4-Toluihydrazide (1 g, 0.007 mol) and pyruvic acid (0.6 g, 0.007 mol) were dissolved in methanol (30 ml). The solution was heated for 3 h; slow evaporation of the solvent gave colorless crystals.

Refinement

Carbon-bound H atoms were placed in calculated positions (C—H 0.93–0.99 Å) and were included in the refinement in the riding model approximation, with U(H) set to 1.2 to 1.5U(C). The methyl H atoms were rotated to fit the electron density.

The oxygen- and nitrogen-bound H atoms were located in a difference Fourier map, and were refined with distance restraints [N—H 0.88 (2) and O—H 0.84 (2) Å]; their temperature factors were freely refined.

Figures

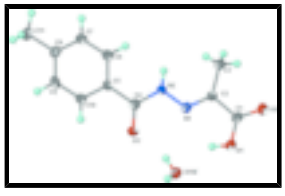

Fig. 1. Displacement ellipsoids plot (Barbour, 2001) of the title compound at the 70% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius.

2-[(4-Methylbenzoyl)hydrazono]propionic acid monohydrate

Crystal data

$C_{11}H_{12}N_2O_3·H_2O$	F_{000} = 252
M_r = 238.24	D_x = 1.410 Mg m^{-3}
Monoclinic, $P2_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2yb	Cell parameters from 1743 reflections
a = 6.8464 (1) Å	θ = 3.0–26.9º
b = 11.9753 (2) Å	μ = 0.11 mm^{-1}
c = 7.0005 (1) Å	T = 100 (2) K
β = 102.169 (1)º	Irregular block, colourless
V = 561.06 (2) Å^3	0.20 × 0.10 × 0.10 mm
Z = 2	

Data collection

Bruker SMART APEX	1211 reflections with $I > 2\sigma(I)$

diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

T = 100(2) K

ω scans

Absorption correction: none

5272 measured reflections

1335 independent reflections

R_{int} = 0.029

θ_{max} = 27.5º

θ_{min} = 3.0º

h = −8→8

k = −14→15

l = −9→9

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)]$ = 0.030

$wR(F^2)$ = 0.083

S = 1.02

1335 reflections

172 parameters

5 restraints

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0559P)^2 + 0.0248P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max}$ = 0.001

$\Delta\rho_{max}$ = 0.19 e Å^{-3}

$\Delta\rho_{min}$ = −0.16 e Å^{-3}

Extinction correction: none

Absolute structure: 1126 Friedel pairs were merged

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
O1	1.4450 (2)	0.99988 (13)	0.8798 (3)	0.0255 (4)
O2	1.2160 (2)	1.13156 (13)	0.8463 (2)	0.0269 (4)
O3	1.2831 (2)	0.64501 (13)	0.7200 (2)	0.0260 (4)
O1W	1.6032 (2)	0.78718 (13)	0.8663 (3)	0.0251 (4)
N1	1.1624 (2)	0.84748 (15)	0.7821 (3)	0.0192 (4)
N2	1.0279 (3)	0.76060 (15)	0.7458 (3)	0.0202 (4)
C1	1.2584 (3)	1.03349 (18)	0.8395 (3)	0.0204 (5)
C2	1.0961 (3)	0.94692 (18)	0.7861 (3)	0.0183 (4)
C3	0.8847 (3)	0.98625 (18)	0.7447 (3)	0.0232 (5)
H3A	0.8131	0.9412	0.6686	0.035*
H3B	0.8400	0.9951	0.8651	0.035*
H3C	0.8754	1.0568	0.6777	0.035*
C4	1.1067 (3)	0.65752 (18)	0.7236 (3)	0.0198 (4)

C5	0.9684 (3)	0.56054 (18)	0.7054 (3)	0.0178 (4)
C6	0.7608 (3)	0.5709 (2)	0.6510 (3)	0.0218 (4)
H6	0.7018	0.6422	0.6192	0.026*
C7	0.6408 (3)	0.47675 (19)	0.6437 (3)	0.0231 (5)
H7	0.4998	0.4845	0.6067	0.028*
C8	0.7226 (3)	0.37199 (18)	0.6891 (3)	0.0214 (5)
C9	0.9315 (3)	0.3617 (2)	0.7379 (3)	0.0221 (5)
H9A	0.9904	0.2901	0.7659	0.026*
C10	1.0524 (3)	0.45480 (19)	0.7457 (3)	0.0202 (4)
H10	1.1936	0.4467	0.7787	0.024*
C11	0.5927 (3)	0.27036 (19)	0.6870 (4)	0.0288 (5)
H11A	0.6157	0.2411	0.7985	0.043*
H11B	0.4542	0.2924	0.6689	0.043*
H11C	0.6071	0.2211	0.5815	0.043*
H11	1.496 (3)	0.755 (3)	0.813 (5)	0.059 (10)*
H12	1.653 (4)	0.748 (2)	0.963 (3)	0.052 (10)*
H1	1.445 (5)	0.9307 (9)	0.867 (5)	0.056 (11)*
H2	0.906 (2)	0.773 (2)	0.761 (4)	0.031 (7)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0193 (8)	0.0198 (9)	0.0371 (10)	−0.0029 (6)	0.0049 (7)	−0.0031 (7)
O2	0.0234 (7)	0.0182 (8)	0.0384 (9)	−0.0001 (6)	0.0047 (7)	−0.0028 (7)
O3	0.0171 (7)	0.0206 (8)	0.0416 (9)	−0.0005 (6)	0.0094 (6)	−0.0039 (7)
O1W	0.0205 (7)	0.0208 (8)	0.0331 (10)	−0.0018 (6)	0.0036 (7)	0.0028 (7)
N1	0.0186 (9)	0.0171 (9)	0.0218 (9)	−0.0017 (7)	0.0042 (7)	0.0008 (7)
N2	0.0139 (8)	0.0165 (9)	0.0301 (10)	−0.0001 (7)	0.0048 (7)	−0.0012 (7)
C1	0.0198 (10)	0.0212 (11)	0.0202 (11)	−0.0033 (8)	0.0043 (8)	−0.0013 (8)
C2	0.0167 (9)	0.0179 (10)	0.0209 (10)	−0.0012 (8)	0.0054 (8)	−0.0004 (8)
C3	0.0174 (10)	0.0189 (11)	0.0329 (13)	0.0007 (8)	0.0044 (9)	0.0033 (9)
C4	0.0200 (10)	0.0186 (10)	0.0207 (10)	−0.0004 (8)	0.0041 (8)	0.0004 (9)
C5	0.0170 (10)	0.0169 (10)	0.0201 (10)	0.0010 (8)	0.0049 (8)	−0.0013 (8)
C6	0.0205 (10)	0.0204 (10)	0.0240 (11)	0.0024 (9)	0.0036 (8)	−0.0003 (9)
C7	0.0159 (10)	0.0238 (11)	0.0289 (12)	0.0017 (9)	0.0034 (8)	−0.0049 (9)
C8	0.0225 (11)	0.0216 (11)	0.0211 (11)	−0.0046 (9)	0.0068 (8)	−0.0057 (9)
C9	0.0260 (11)	0.0154 (10)	0.0254 (11)	0.0042 (9)	0.0067 (9)	0.0005 (8)
C10	0.0160 (9)	0.0214 (11)	0.0226 (11)	0.0021 (8)	0.0030 (8)	−0.0004 (9)
C11	0.0282 (11)	0.0228 (12)	0.0370 (14)	−0.0050 (10)	0.0110 (10)	−0.0044 (10)

Geometric parameters (Å, °)

O1—C1	1.312 (3)	C4—C5	1.487 (3)
O1—H1	0.83 (2)	C5—C10	1.395 (3)
O2—C1	1.213 (3)	C5—C6	1.397 (3)
O3—C4	1.222 (2)	C6—C7	1.390 (3)
O1W—H11	0.84 (2)	C6—H6	0.9500
O1W—H12	0.82 (2)	C7—C8	1.383 (3)
N1—C2	1.277 (3)	C7—H7	0.9500

N1—N2	1.377 (2)	C8—C9	1.404 (3)
N2—C4	1.369 (3)	C8—C11	1.506 (3)
N2—H2	0.87 (2)	C9—C10	1.383 (3)
C1—C2	1.508 (3)	C9—H9A	0.9500
C2—C3	1.491 (3)	C10—H10	0.9500
C3—H3A	0.8400	C11—H11A	0.8400
C3—H3B	0.9620	C11—H11B	0.9663
C3—H3C	0.9620	C11—H11C	0.9662
C1—O1—H1	108 (2)	C6—C5—C4	123.14 (19)
H11—O1W—H12	106 (3)	C7—C6—C5	119.8 (2)
C2—N1—N2	118.80 (17)	C7—C6—H6	120.1
C4—N2—N1	116.00 (17)	C5—C6—H6	120.1
C4—N2—H2	124.9 (19)	C8—C7—C6	121.34 (19)
N1—N2—H2	118.1 (19)	C8—C7—H7	119.3
O2—C1—O1	121.2 (2)	C6—C7—H7	119.3
O2—C1—C2	120.35 (19)	C7—C8—C9	118.5 (2)
O1—C1—C2	118.40 (18)	C7—C8—C11	121.41 (19)
N1—C2—C3	128.72 (19)	C9—C8—C11	120.1 (2)
N1—C2—C1	113.55 (17)	C10—C9—C8	120.6 (2)
C3—C2—C1	117.73 (19)	C10—C9—H9A	119.7
C2—C3—H3A	109.5	C8—C9—H9A	119.7
C2—C3—H3B	109.9	C9—C10—C5	120.40 (18)
H3A—C3—H3B	112.0	C9—C10—H10	119.8
C2—C3—H3C	109.5	C5—C10—H10	119.8
H3A—C3—H3C	106.6	C8—C11—H11A	109.5
H3B—C3—H3C	109.3	C8—C11—H11B	110.0
O3—C4—N2	121.82 (19)	H11A—C11—H11B	102.8
O3—C4—C5	121.12 (19)	C8—C11—H11C	110.2
N2—C4—C5	117.06 (17)	H11A—C11—H11C	115.2
C10—C5—C6	119.3 (2)	H11B—C11—H11C	108.8
C10—C5—C4	117.61 (17)		
C2—N1—N2—C4	173.5 (2)	N2—C4—C5—C6	20.0 (3)
N2—N1—C2—C3	−3.9 (3)	C10—C5—C6—C7	2.0 (3)
N2—N1—C2—C1	176.43 (17)	C4—C5—C6—C7	−177.78 (19)
O2—C1—C2—N1	179.1 (2)	C5—C6—C7—C8	−0.1 (3)
O1—C1—C2—N1	−1.5 (3)	C6—C7—C8—C9	−1.8 (3)
O2—C1—C2—C3	−0.6 (3)	C6—C7—C8—C11	178.2 (2)
O1—C1—C2—C3	178.78 (19)	C7—C8—C9—C10	1.8 (3)
N1—N2—C4—O3	−5.9 (3)	C11—C8—C9—C10	−178.2 (2)
N1—N2—C4—C5	173.67 (17)	C8—C9—C10—C5	0.1 (3)
O3—C4—C5—C10	19.8 (3)	C6—C5—C10—C9	−2.0 (3)
N2—C4—C5—C10	−159.73 (19)	C4—C5—C10—C9	177.77 (19)
O3—C4—C5—C6	−160.4 (2)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O1W	0.83 (2)	2.03 (2)	2.777 (2)	149 (3)
O1W—H11···O3	0.84 (2)	1.97 (2)	2.794 (2)	165 (4)

O1W—H12···O2^{i}	0.84 (2)	2.00 (1)	2.829 (2)	168 (3)
N2—H2···O1W^{ii}	0.87 (2)	2.35 (1)	3.210 (2)	168 (3)

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

Fig. 1