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

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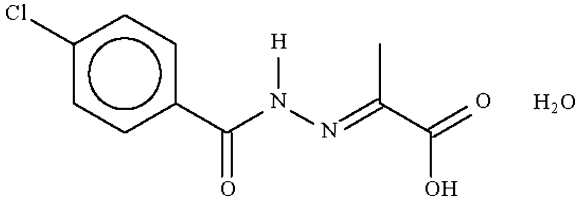
Key indicators: single-crystal X-ray study; T = 118 K; mean σ(C–C) = 0.003 Å; R factor = 0.026; wR factor = 0.070; data-to-parameter ratio = 11.5.

In the title compound, $C_{10}H_9ClN_2O_3\cdot H_2O$, the water molecule is a hydrogen-bond donor to the amide and carbonyl O atoms of two acid molecules; it is also a hydrogen-bond acceptor to the acid OH group and the amide H atom. The hydrogen-bonding interactions give rise to a two-dimensional array.

Related literature

For the structure of 2-[(4-methylbenzoyl)hydrazono]propionic acid monohydrate, see: Wong *et al.* (2009).



Experimental

Crystal data

$C_{10}H_9ClN_2O_3\cdot H_2O$
M_r = 258.66
Triclinic, $P\overline{1}$
a = 6.6516 (1) Å
b = 6.9345 (1) Å
c = 7.0988 (1) Å
α = 73.833 (1)°
β = 80.182 (1)°
γ = 61.613 (1)°
V = 276.39 (1) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 0.35 mm^{-1}
T = 118 K
0.45 × 0.35 × 0.15 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.858, T_{max} = 0.949
2247 measured reflections
1965 independent reflections
1952 reflections with $I > 2\sigma(I)$
R_{int} = 0.011

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.070
S = 1.00
1965 reflections
171 parameters
7 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}
Absolute structure: Flack (1983), 733 Friedel pairs
Flack parameter: 0.02 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1O···O1W	0.83 (1)	1.92 (3)	2.659 (2)	147 (4)
O1W—H11···O2^{i}	0.84 (1)	1.96 (1)	2.784 (2)	165 (2)
O1W—H12···O3	0.84 (1)	1.98 (1)	2.809 (2)	172 (2)
N1—H1N···O1W^{ii}	0.88 (1)	2.48 (2)	3.3596 (18)	177 (2)

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

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; 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: *pubCIF* (Westrip, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2398).

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Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
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supplementary materials

Acta Cryst. (2009). E**65**, o816 [**doi:10.1107/S1600536809009544**]

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

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

Comment

(type here to add)

Experimental

4-Chlorobenzoylhydrazide (0.85 g, 0.005 mol) and pyruvic acid (0.43 g, 0.005 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 0.95-0.98 Å) and were included in the refinement in the riding model approximation, with *U*(H) set to 1.2-1.5*U*(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±0.01 and O–H 0.84±0.01 Å]; their U_{iso} values were freely refined.

Figures

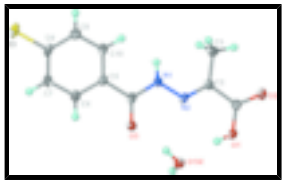

Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of $C_{10}H_9N_2O_3{\cdot}H_2O$ at the 70% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius.

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

Crystal data

$C_{10}H_9ClN_2O_3{\cdot}H_2O$	$Z = 1$
$M_r = 258.66$	$F_{000} = 134$
Triclinic, *P*1	$D_x = 1.554$ Mg m^{-3}
Hall symbol: P 1	Mo *K*α radiation λ = 0.71073 Å
a = 6.6516 (1) Å	Cell parameters from 2199 reflections
b = 6.9345 (1) Å	θ = 3.0–28.2°
c = 7.0988 (1) Å	μ = 0.35 mm^{-1}
α = 73.833 (1)°	*T* = 118 K
β = 80.182 (1)°	Irregular block, colorless

$\gamma = 61.613\ (1)^{\circ}$	0.45 × 0.35 × 0.15 mm
$V = 276.39\ (1)\ Å^3$	

Data collection

Bruker SMART APEX diffractometer	1965 independent reflections
Radiation source: fine-focus sealed tube	1952 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.011$
$T = 118$ K	$\theta_{max} = 27.5^{\circ}$
ω scans	$\theta_{min} = 3.0^{\circ}$
Absorption correction: Multi-scan (SADABS; Sheldrick, 1996)	$h = -8 \rightarrow 8$
$T_{min} = 0.858$, $T_{max} = 0.949$	$k = -8 \rightarrow 8$
2247 measured reflections	$l = -9 \rightarrow 9$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.026$	$w = 1/[\sigma^2(F_o^2) + (0.0628P)^2 + 0.0013P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.070$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.00$	$\Delta\rho_{max} = 0.20$ e Å^{-3}
1965 reflections	$\Delta\rho_{min} = -0.33$ e Å^{-3}
171 parameters	Extinction correction: none
7 restraints	Absolute structure: Flack (1983), 733 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.02 (3)
Secondary atom site location: difference Fourier map	

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.50015 (5)	0.49992 (5)	0.49993 (4)	0.02415 (12)
O1	2.0776 (2)	0.41172 (18)	−0.23906 (16)	0.0184 (2)
O2	1.9975 (2)	0.76916 (19)	−0.31287 (18)	0.0221 (3)
O3	1.5700 (2)	0.18182 (19)	0.11932 (19)	0.0228 (3)
O1W	1.9830 (2)	0.06405 (18)	−0.10712 (18)	0.0216 (2)
N1	1.4403 (2)	0.5584 (2)	−0.0003 (2)	0.0168 (3)
N2	1.6543 (2)	0.5201 (2)	−0.07703 (19)	0.0154 (3)
C1	1.9322 (3)	0.6250 (3)	−0.2491 (2)	0.0161 (3)
C2	1.6887 (3)	0.6861 (2)	−0.1806 (2)	0.0165 (3)
C3	1.5134 (3)	0.9273 (3)	−0.2314 (3)	0.0279 (4)
H3A	1.3862	0.9390	−0.2937	0.042*
H3B	1.5830	1.0164	−0.3222	0.042*

H3C	1.4562	0.9845	−0.1115	0.042*
C4	1.4131 (3)	0.3718 (3)	0.1048 (2)	0.0169 (3)
C5	1.1830 (3)	0.4130 (3)	0.2013 (2)	0.0158 (3)
C6	1.1356 (3)	0.2284 (3)	0.2676 (2)	0.0197 (3)
H6	1.2483	0.0845	0.2492	0.024*
C7	0.9270 (3)	0.2526 (3)	0.3596 (2)	0.0200 (3)
H7	0.8955	0.1268	0.4045	0.024*
C8	0.7637 (3)	0.4649 (3)	0.3852 (2)	0.0187 (3)
C9	0.8072 (3)	0.6496 (3)	0.3209 (2)	0.0194 (3)
H9	0.6934	0.7934	0.3385	0.023*
C10	1.0168 (3)	0.6238 (2)	0.2310 (2)	0.0184 (3)
H10	1.0483	0.7497	0.1891	0.022*
H1O	2.032 (8)	0.321 (6)	−0.244 (7)	0.115 (18)*
H11	1.970 (4)	−0.004 (3)	−0.182 (3)	0.029 (5)*
H12	1.855 (2)	0.111 (4)	−0.048 (3)	0.028 (6)*
H1N	1.318 (3)	0.689 (2)	−0.024 (3)	0.013 (4)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0181 (2)	0.0375 (2)	0.02137 (18)	−0.01711 (17)	0.00484 (13)	−0.00835 (14)
O1	0.0136 (6)	0.0172 (5)	0.0225 (6)	−0.0068 (4)	0.0026 (4)	−0.0041 (4)
O2	0.0191 (6)	0.0202 (5)	0.0283 (6)	−0.0120 (5)	0.0068 (5)	−0.0066 (4)
O3	0.0183 (6)	0.0153 (5)	0.0309 (6)	−0.0077 (4)	0.0049 (5)	−0.0028 (4)
O1W	0.0183 (6)	0.0182 (5)	0.0274 (6)	−0.0087 (5)	0.0051 (5)	−0.0067 (4)
N1	0.0140 (7)	0.0163 (6)	0.0188 (6)	−0.0074 (5)	0.0021 (5)	−0.0027 (5)
N2	0.0120 (7)	0.0196 (6)	0.0160 (6)	−0.0082 (5)	0.0032 (5)	−0.0064 (5)
C1	0.0167 (8)	0.0171 (6)	0.0156 (6)	−0.0087 (6)	0.0011 (6)	−0.0044 (5)
C2	0.0165 (8)	0.0158 (7)	0.0176 (7)	−0.0080 (6)	0.0008 (6)	−0.0038 (5)
C3	0.0177 (8)	0.0165 (7)	0.0415 (10)	−0.0060 (6)	0.0068 (7)	−0.0028 (6)
C4	0.0154 (8)	0.0191 (7)	0.0173 (7)	−0.0093 (6)	0.0014 (6)	−0.0044 (5)
C5	0.0137 (8)	0.0178 (7)	0.0153 (7)	−0.0081 (6)	0.0014 (6)	−0.0026 (5)
C6	0.0197 (8)	0.0195 (7)	0.0206 (7)	−0.0105 (6)	0.0010 (6)	−0.0036 (5)
C7	0.0213 (9)	0.0222 (7)	0.0212 (7)	−0.0147 (7)	0.0018 (6)	−0.0044 (6)
C8	0.0143 (8)	0.0279 (8)	0.0151 (7)	−0.0123 (7)	0.0019 (6)	−0.0030 (6)
C9	0.0168 (8)	0.0191 (7)	0.0182 (7)	−0.0064 (6)	−0.0003 (6)	−0.0016 (5)
C10	0.0176 (8)	0.0187 (7)	0.0185 (7)	−0.0098 (6)	0.0018 (6)	−0.0023 (5)

Geometric parameters (Å, °)

Cl1—C8	1.7363 (18)	C3—H3B	0.9800
O1—C1	1.3161 (19)	C3—H3C	0.9800
O1—H1O	0.829 (10)	C4—C5	1.493 (2)
O2—C1	1.219 (2)	C5—C6	1.399 (2)
O3—C4	1.220 (2)	C5—C10	1.400 (2)
O1W—H11	0.842 (9)	C6—C7	1.382 (2)
O1W—H12	0.836 (10)	C6—H6	0.9500
N1—N2	1.360 (2)	C7—C8	1.393 (2)
N1—C4	1.379 (2)	C7—H7	0.9500

N1—H1N	0.878 (9)	C8—C9	1.383 (2)
N2—C2	1.281 (2)	C9—C10	1.380 (3)
C1—C2	1.495 (2)	C9—H9	0.9500
C2—C3	1.497 (2)	C10—H10	0.9500
C3—H3A	0.9800		
C1—O1—H1O	120 (3)	N1—C4—C5	116.93 (13)
H11—O1W—H12	103 (2)	C6—C5—C10	119.14 (15)
N2—N1—C4	116.41 (12)	C6—C5—C4	117.42 (14)
N2—N1—H1N	124.6 (14)	C10—C5—C4	123.44 (14)
C4—N1—H1N	118.6 (14)	C7—C6—C5	120.85 (14)
C2—N2—N1	119.24 (13)	C7—C6—H6	119.6
O2—C1—O1	119.90 (16)	C5—C6—H6	119.6
O2—C1—C2	121.04 (14)	C6—C7—C8	118.77 (14)
O1—C1—C2	119.06 (13)	C6—C7—H7	120.6
N2—C2—C1	114.38 (13)	C8—C7—H7	120.6
N2—C2—C3	126.56 (16)	C9—C8—C7	121.30 (17)
C1—C2—C3	119.03 (14)	C9—C8—Cl1	119.04 (13)
C2—C3—H3A	109.5	C7—C8—Cl1	119.67 (13)
C2—C3—H3B	109.5	C10—C9—C8	119.64 (15)
H3A—C3—H3B	109.5	C10—C9—H9	120.2
C2—C3—H3C	109.5	C8—C9—H9	120.2
H3A—C3—H3C	109.5	C9—C10—C5	120.28 (14)
H3B—C3—H3C	109.5	C9—C10—H10	119.9
O3—C4—N1	121.63 (16)	C5—C10—H10	119.9
O3—C4—C5	121.44 (15)		
C4—N1—N2—C2	178.11 (13)	N1—C4—C5—C10	−16.0 (2)
N1—N2—C2—C1	177.31 (11)	C10—C5—C6—C7	0.7 (2)
N1—N2—C2—C3	−0.7 (2)	C4—C5—C6—C7	179.58 (13)
O2—C1—C2—N2	−164.68 (14)	C5—C6—C7—C8	0.0 (2)
O1—C1—C2—N2	15.00 (19)	C6—C7—C8—C9	−0.1 (2)
O2—C1—C2—C3	13.5 (2)	C6—C7—C8—Cl1	179.90 (11)
O1—C1—C2—C3	−166.78 (14)	C7—C8—C9—C10	−0.6 (2)
N2—N1—C4—O3	−3.7 (2)	Cl1—C8—C9—C10	179.43 (11)
N2—N1—C4—C5	175.94 (11)	C8—C9—C10—C5	1.3 (2)
O3—C4—C5—C6	−15.2 (2)	C6—C5—C10—C9	−1.3 (2)
N1—C4—C5—C6	165.12 (13)	C4—C5—C10—C9	179.82 (13)
O3—C4—C5—C10	163.61 (15)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1O···O1W	0.83 (1)	1.92 (3)	2.659 (2)	147 (4)
O1W—H11···O2^{i}	0.84 (1)	1.96 (1)	2.784 (2)	165 (2)
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N1—H1N···O1W^{ii}	0.88 (1)	2.48 (2)	3.3596 (18)	177 (2)

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

Fig. 1