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(*E*)-Benzoyl[1-(2-hydroxyethyl)-imidazolidin-2-ylidene]acetonitrile

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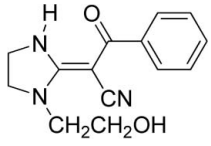
Key indicators: single-crystal X-ray study; T = 113 K; mean σ(C–C) = 0.002 Å; R factor = 0.041; wR factor = 0.103; data-to-parameter ratio = 16.6.

In the title compound, $C_{14}H_{15}N_3O_2$, the C═C(H)—C═O grouping and the imidazolidine ring are coplanar as a result of an intramolecular N—H···O hydrogen bond and extended electronic conjugation. Intermolecular N—H···O and O—H···N hydrogen bonds help to establish the packing.

Related literature

For related literature, see: Wang & Huang (1996).



Experimental

Crystal data

$C_{14}H_{15}N_3O_2$
M_r = 257.29
Monoclinic, $P2_1/c$
a = 8.3748 (17) Å
b = 14.633 (3) Å
c = 10.784 (2) Å
β = 107.33 (3)°
V = 1261.6 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 113 (2) K
0.10 × 0.08 × 0.06 mm

Data collection

Rigaku Saturn diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.982, T_{max} = 0.994
9551 measured reflections
2994 independent reflections
2534 reflections with $I > 2\sigma(I)$
R_{int} = 0.036

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.103
S = 1.10
2994 reflections
180 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Table 1
Selected torsion angles (°).

C5—N1—C3—C4	−12.82 (13)	C4—N2—C5—N1	11.16 (14)
C5—N2—C4—C3	−18.34 (13)	C3—N1—C5—N2	1.74 (14)
N1—C3—C4—N2	17.71 (12)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O2^{i}	0.881 (16)	2.318 (16)	2.9557 (15)	129.3 (13)
N2—H2···O2	0.881 (16)	1.953 (16)	2.6252 (15)	132.0 (14)
O1—H1···N3^{ii}	0.87 (2)	2.04 (2)	2.8794 (16)	160.9 (17)

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

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

We thank Haibin Song at Nankai University for the X-ray crystallographic determination.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2622).

References

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Wang, L.-B. & Huang, Z.-T. (1996). *Synth. Commun.* **26**, 459–473.

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supporting information

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(*E*)-Benzoyl[1-(2-hydroxyethyl)imidazolidin-2-ylidene]acetonitrile

Lin Li, Yan-Hong Tian, Chu-Yi Yu and Li-Ben Wang

S1. Comment

Heterocyclic ketene aminals (HKAs) are versatile synthons for heterocyclic synthesis. The title compound, (I), (Fig. 1), which possesses a *β*-hydroxyethyl group on the nitrogen atom of the imidazolidine ring, is a member of this family (Wang & Huang, 1996).

Due to the extended conjugation in the molecule, some abnormal geometrical parameters occur. For example, O2—C8 = 1.2487 (14) Å, which is longer than a normal double bond; the length of N1—C5 [1.3435 (16) Å] and N2—C5 [1.3366 (16) Å] are shorter than those of normal C—N single bonds; the length of C5—C6 [1.4348 (17) Å] double bond is longer than that of a normal C=C bond. The atoms of imidazoline ring in this compound (I) are approximately coplanar, in which, the torsion angle of C3—N1—C5—N2 is 1.74 (17)°, the torsion angle of C4—N2—C5—N1 is 11.16 (14)°, and the torsion angle of C5—N1—C3—C4 is -12.82 (13)°.

The molecules are linked by intermolecular N—H···O hydrogen bonds and O—H···N bonds (Table 1). There is also an intramolecular hydrogen bond involving the O2 and amide N2 atoms.

S2. Experimental

The title compound was prepared according to the procedure of Wang & Huang (1996) and recrystallized from methanol in 86% yield to yield colourless prisms of (I) (m.p. 449–450 K). IR: *v*= 3400 (OH), 3240 (NH), 2180 (CN), 1580 (CO), 1560, 1545 $cm^{-1.}$ ^{1}H-NMR (DMSO-d_6): *δ*= 9.83 (1*H*, s), 7.34–7.62 (5*H*, m), 4.44 (1*H*, s), 3.56–3.84 p.p.m. (8*H*, m), ^{13}C-NMR (DMSO-d_6): *δ*= 189.8, 163.6, 140.6, 129.8, 127.6, 127.4, 121.6, 64.5, 59.6, 50.4, 48.9, 41.6 p.p.m.. MS: *m/z* = 257 (*M*+, 29),226 (7), 212 (6), 160 (14), 105 (100). Anal. Calcd. for $C_{14}H_{15}N_3O_2$: C 65.35, H 5.88, N 16.33; found: C 65.39, H 5.77, N 16.42.

S3. Refinement

The N– and O-bound H atoms were located in a difference map and freely refined. The C-bound H atoms were placed in geometrically idealized positions (C—H = 0.95–1.00 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C).

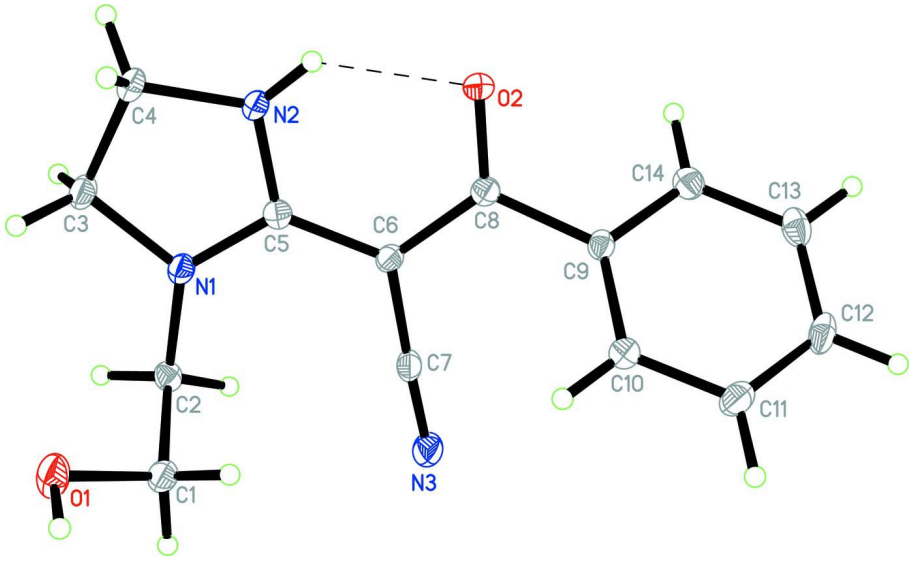

Figure 1

The molecular structure of (I). Ellipsoids are drawn at the 40% probability level (H atoms represented by arbitrary spheres). The intramolecular hydrogen bond is indicated by a dashed line.

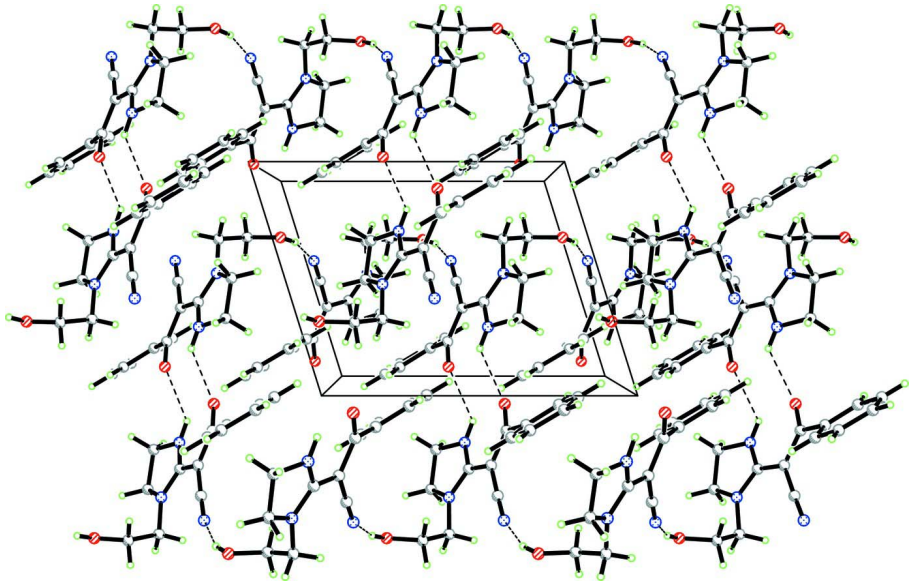

Figure 2

Packing diagram for (I), viewed down the *b* axis with hydrogen bonds indicated by dashed lines.

(*E*)-Benzoyl[1-(2-hydroxyethyl)imidazolidin-2-ylidene]acetonitrile

Crystal data

$C_{14}H_{15}N_3O_2$
$M_r = 257.29$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 8.3748\ (17)$ Å
$b = 14.633\ (3)$ Å
$c = 10.784\ (2)$ Å
$\beta = 107.33\ (3)°$
$V = 1261.6\ (4)$ Å^3
$Z = 4$
$F(000) = 544$
$D_x = 1.355$ Mg m^{-3}

Melting point = 449–450 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4329 reflections
θ = 2.3–22.5°
μ = 0.09 mm^{-1}
T = 113 K
Prism, colourless
0.10 × 0.08 × 0.06 mm

Data collection

Rigaku Saturn diffractometer
Radiation source: rotating anode
Confocal monochromator
Detector resolution: 7.31 pixels mm^{-1}
ω and φ scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.982, T_{max} = 0.994
9551 measured reflections
2994 independent reflections
2534 reflections with $I > 2\sigma(I)$
R_{int} = 0.036
θ_{max} = 27.9°, θ_{min} = 2.4°
h = −11→11
k = −19→18
l = −14→11

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.103
S = 1.10
2994 reflections
180 parameters
0 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
$w = 1/[\sigma^2(F_o^2) + (0.0503P)^2 + 0.2093P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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 $F^2 > \sigma(F^2)$ 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}
O1	0.30219 (13)	0.59907 (7)	0.03041 (9)	0.0280 (2)
H1	0.323 (2)	0.6459 (13)	−0.0116 (18)	0.044 (5)*
O2	0.91862 (10)	0.56888 (6)	0.57744 (8)	0.0186 (2)
N1	0.47423 (12)	0.51059 (7)	0.28326 (10)	0.0171 (2)
N2	0.72478 (13)	0.46439 (7)	0.39657 (10)	0.0170 (2)
N3	0.41182 (14)	0.73000 (8)	0.45144 (11)	0.0259 (3)
C1	0.30005 (16)	0.63405 (9)	0.15308 (12)	0.0195 (3)
H1A	0.1954	0.6687	0.1432	0.023*
H1B	0.3957	0.6760	0.1876	0.023*
C2	0.31137 (15)	0.55527 (9)	0.24601 (11)	0.0169 (3)
H2A	0.2878	0.5781	0.3252	0.020*

H2B	0.2244	0.5097	0.2049	0.020*
C3	0.49585 (16)	0.42196 (9)	0.22523 (12)	0.0201 (3)
H3A	0.4665	0.4264	0.1295	0.024*
H3B	0.4264	0.3740	0.2483	0.024*
C4	0.68186 (16)	0.40238 (9)	0.28553 (12)	0.0210 (3)
H4A	0.7016	0.3378	0.3135	0.025*
H4B	0.7461	0.4167	0.2244	0.025*
C5	0.61006 (14)	0.53014 (8)	0.38307 (11)	0.0150 (2)
C6	0.63996 (14)	0.60969 (8)	0.46455 (11)	0.0154 (2)
C7	0.51223 (15)	0.67519 (9)	0.45570 (11)	0.0178 (3)
C8	0.80273 (14)	0.62602 (8)	0.55445 (11)	0.0151 (2)
C10	0.79435 (15)	0.79899 (9)	0.56137 (12)	0.0194 (3)
H10	0.7338	0.7999	0.4716	0.023*
C11	0.83750 (16)	0.88063 (9)	0.62852 (13)	0.0236 (3)
H11	0.8066	0.9372	0.5848	0.028*
C12	0.92588 (16)	0.87938 (10)	0.75974 (13)	0.0238 (3)
H12	0.9541	0.9352	0.8062	0.029*
C13	0.97302 (16)	0.79694 (10)	0.82305 (12)	0.0225 (3)
H13	1.0342	0.7964	0.9127	0.027*
C9	0.83921 (14)	0.71581 (8)	0.62480 (11)	0.0154 (2)
C14	0.93138 (15)	0.71500 (9)	0.75621 (12)	0.0194 (3)
H14	0.9654	0.6586	0.7997	0.023*
H2	0.827 (2)	0.4743 (11)	0.4482 (16)	0.031 (4)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0475 (6)	0.0210 (5)	0.0173 (5)	−0.0073 (4)	0.0125 (4)	−0.0009 (4)
O2	0.0197 (4)	0.0155 (5)	0.0179 (4)	0.0038 (3)	0.0016 (3)	0.0001 (3)
N1	0.0172 (5)	0.0146 (5)	0.0186 (5)	−0.0013 (4)	0.0041 (4)	−0.0033 (4)
N2	0.0173 (5)	0.0137 (5)	0.0188 (5)	0.0001 (4)	0.0033 (4)	−0.0040 (4)
N3	0.0213 (6)	0.0246 (6)	0.0289 (6)	0.0036 (5)	0.0030 (4)	−0.0091 (5)
C1	0.0230 (6)	0.0166 (6)	0.0188 (6)	−0.0009 (5)	0.0063 (5)	−0.0014 (5)
C2	0.0148 (6)	0.0179 (6)	0.0175 (6)	−0.0019 (5)	0.0038 (4)	−0.0002 (5)
C3	0.0223 (6)	0.0159 (6)	0.0215 (6)	−0.0022 (5)	0.0057 (5)	−0.0054 (5)
C4	0.0232 (6)	0.0161 (6)	0.0223 (6)	0.0000 (5)	0.0049 (5)	−0.0064 (5)
C5	0.0166 (6)	0.0148 (6)	0.0142 (5)	−0.0014 (4)	0.0053 (4)	0.0016 (4)
C6	0.0172 (6)	0.0129 (6)	0.0160 (6)	0.0005 (4)	0.0050 (4)	−0.0008 (4)
C7	0.0179 (6)	0.0182 (6)	0.0160 (6)	−0.0020 (5)	0.0031 (4)	−0.0047 (5)
C8	0.0191 (6)	0.0139 (6)	0.0129 (5)	0.0001 (4)	0.0055 (4)	0.0016 (4)
C10	0.0188 (6)	0.0172 (6)	0.0207 (6)	0.0008 (5)	0.0034 (5)	−0.0015 (5)
C11	0.0222 (6)	0.0159 (7)	0.0331 (7)	0.0005 (5)	0.0087 (5)	−0.0024 (5)
C12	0.0190 (6)	0.0216 (7)	0.0326 (7)	−0.0043 (5)	0.0102 (5)	−0.0144 (5)
C13	0.0188 (6)	0.0300 (8)	0.0185 (6)	−0.0027 (5)	0.0055 (5)	−0.0079 (5)
C9	0.0144 (6)	0.0155 (6)	0.0166 (6)	−0.0007 (4)	0.0052 (4)	−0.0029 (4)
C14	0.0181 (6)	0.0219 (7)	0.0181 (6)	0.0003 (5)	0.0054 (5)	−0.0014 (5)

Geometric parameters (Å, º)

O1—C1	1.4235 (15)	C4—H4A	0.9900
O1—H1	0.87 (2)	C4—H4B	0.9900
O2—C8	1.2487 (14)	C5—C6	1.4348 (17)
N1—C5	1.3435 (16)	C6—C7	1.4182 (17)
N1—C2	1.4569 (16)	C6—C8	1.4381 (17)
N1—C3	1.4744 (16)	C8—C9	1.5022 (16)
N2—C5	1.3366 (16)	C10—C11	1.3879 (18)
N2—C4	1.4595 (16)	C10—C9	1.3920 (17)
N2—H2	0.881 (16)	C10—H10	0.9500
N3—C7	1.1530 (16)	C11—C12	1.388 (2)
C1—C2	1.5119 (17)	C11—H11	0.9500
C1—H1A	0.9900	C12—C13	1.384 (2)
C1—H1B	0.9900	C12—H12	0.9500
C2—H2A	0.9900	C13—C14	1.3889 (18)
C2—H2B	0.9900	C13—H13	0.9500
C3—C4	1.5258 (18)	C9—C14	1.3974 (17)
C3—H3A	0.9900	C14—H14	0.9500
C3—H3B	0.9900		
C1—O1—H1	105.2 (12)	H4A—C4—H4B	109.3
C5—N1—C2	128.69 (10)	N2—C5—N1	110.10 (11)
C5—N1—C3	110.22 (10)	N2—C5—C6	121.91 (11)
C2—N1—C3	120.06 (10)	N1—C5—C6	127.95 (11)
C5—N2—C4	111.39 (10)	C7—C6—C5	121.11 (11)
C5—N2—H2	118.7 (11)	C7—C6—C8	118.52 (11)
C4—N2—H2	125.1 (11)	C5—C6—C8	120.35 (11)
O1—C1—C2	109.04 (10)	N3—C7—C6	177.90 (13)
O1—C1—H1A	109.9	O2—C8—C6	123.17 (11)
C2—C1—H1A	109.9	O2—C8—C9	117.15 (10)
O1—C1—H1B	109.9	C6—C8—C9	119.68 (10)
C2—C1—H1B	109.9	C11—C10—C9	120.40 (12)
H1A—C1—H1B	108.3	C11—C10—H10	119.8
N1—C2—C1	113.19 (10)	C9—C10—H10	119.8
N1—C2—H2A	108.9	C10—C11—C12	119.83 (13)
C1—C2—H2A	108.9	C10—C11—H11	120.1
N1—C2—H2B	108.9	C12—C11—H11	120.1
C1—C2—H2B	108.9	C13—C12—C11	120.10 (12)
H2A—C2—H2B	107.8	C13—C12—H12	120.0
N1—C3—C4	102.90 (10)	C11—C12—H12	120.0
N1—C3—H3A	111.2	C12—C13—C14	120.37 (12)
C4—C3—H3A	111.2	C12—C13—H13	119.8
N1—C3—H3B	111.2	C14—C13—H13	119.8
C4—C3—H3B	111.2	C10—C9—C14	119.50 (11)
H3A—C3—H3B	109.1	C10—C9—C8	122.15 (11)
N2—C4—C3	101.74 (10)	C14—C9—C8	118.20 (11)
N2—C4—H4A	111.4	C13—C14—C9	119.77 (12)

C3—C4—H4A	111.4	C13—C14—H14	120.1
N2—C4—H4B	111.4	C9—C14—H14	120.1
C3—C4—H4B	111.4		

C5—N1—C2—C1	89.49 (14)	C7—C6—C8—O2	173.51 (11)
C3—N1—C2—C1	−103.33 (13)	C5—C6—C8—O2	−8.42 (18)
O1—C1—C2—N1	69.56 (13)	C7—C6—C8—C9	−6.98 (16)
C5—N1—C3—C4	−12.82 (13)	C5—C6—C8—C9	171.09 (10)
C2—N1—C3—C4	177.82 (10)	C9—C10—C11—C12	−0.02 (19)
C5—N2—C4—C3	−18.34 (13)	C10—C11—C12—C13	0.94 (19)
N1—C3—C4—N2	17.71 (12)	C11—C12—C13—C14	−0.45 (19)
C4—N2—C5—N1	11.16 (14)	C11—C10—C9—C14	−1.39 (18)
C4—N2—C5—C6	−166.78 (11)	C11—C10—C9—C8	−176.84 (11)
C2—N1—C5—N2	169.93 (11)	O2—C8—C9—C10	133.69 (12)
C3—N1—C5—N2	1.74 (14)	C6—C8—C9—C10	−45.85 (16)
C2—N1—C5—C6	−12.3 (2)	O2—C8—C9—C14	−41.82 (15)
C3—N1—C5—C6	179.52 (11)	C6—C8—C9—C14	138.64 (12)
N2—C5—C6—C7	−174.96 (11)	C12—C13—C14—C9	−0.96 (18)
N1—C5—C6—C7	7.49 (19)	C10—C9—C14—C13	1.87 (18)
N2—C5—C6—C8	7.02 (17)	C8—C9—C14—C13	177.50 (10)
N1—C5—C6—C8	−170.52 (11)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O2^{i}	0.881 (16)	2.318 (16)	2.9557 (15)	129.3 (13)
N2—H2···O2	0.881 (16)	1.953 (16)	2.6252 (15)	132.0 (14)
O1—H1···N3^{ii}	0.87 (2)	2.04 (2)	2.8794 (16)	160.9 (17)

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