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(*E*)-*N*′-Hydroxy-*N*,*N*-dimethyl-2-(3-nitrophenyl)-acetimidamide

Yao Ruan[a] and Hui Zhao[b]*

[a]National Engineering Research Center for Carbohydrate Synthesis, Jiangxi Normal University, Nanchang 330022, People's Republic of China, and [b]Anhui Engineering Technology Research Center for Extraction and Isolation of Active Components, Anhui Academy of Science and Technology, Hefei 230031, People's Republic of China. *Correspondence e-mail: zh1986@iccas.ac.cn

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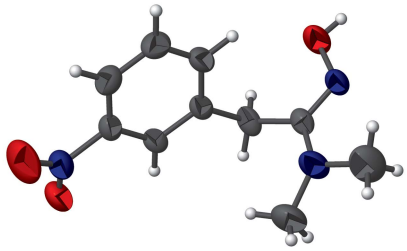
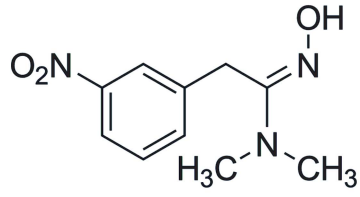
Structural data: full structural data are available from iucrdata.iucr.org

In the crystal of the title compound, $C_{10}H_{13}N_2O_3$, inversion dimers linked by pairs of N—H···O hydrogen bonds generate $R_2^2(6)$ loops. The dimers are linked by weak C—H···O and C—H···π interactions, resulting in a three-dimensional network. A short $NO_2\cdots NO_2$ contact [3.107 (2) Å] is also seen.

3D view



Chemical scheme



Structure description

Amidoxime, also referred to as *N*-hydroxy amidine, is a well-known amphoteric functional group that has been frequently grafted onto various surfaces for the recovery and removal of U^{VI} from aqueous media (*e.g.* sea water) owing to its high sorption capacity and fast sorption rate for uranium and its own low environmental effects (Saeed *et al.*, 2008; Yuan *et al.*, 2016; Zhao *et al.*, 2014). As part of our studies in this area, we now describe the synthesis and structure of the title compound.

The title compound crystallizes in the triclinic $P\overline{1}$ space group with one molecule in the asymmetric unit (Fig. 1). The bond lengths and angles are comparable to its known analogues (*e.g.* Röhrig *et al.*, 2017) and the C8—N2—O3—H3 grouping has an *anti* conformation (torsion angle = −169°).

In the crystal, the molecules are linked into a three-dimensional network by a combination of N—H···O, C—H···O and C—H···π (Table 1, Fig. 2) interactions. Inversion dimers linked by pairwise N—H···O bonds generate a classic $R_2^2(6)$ loop and the weak interactions link the dimers into a three-dimensional network. A short $NO_2\cdots NO_2$ contact [O2···O2($-x$, 2 − y, $-z$) = 3.017 (2) Å] is also observed.

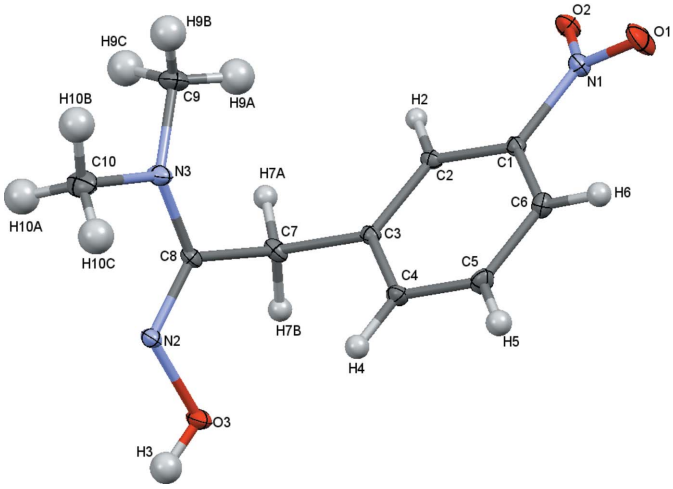


Figure 1
The molecular structure of the title compound, with displacement ellipsoids drawn at the 50% probability level.

Synthesis and crystallization

To a solution of 1-(2,2-difluorovinyl)-3-nitrobenzene (1 mmol) in *N*,*N*-dimethylformamide (DMF, 10 ml) were added hydroxylamine hydrochloride (350 mg, 5 mmol), triethylamine (505 mg, 5 mmol) and powdery 4 Å molecular sieve (Gao *et al.*, 2018). After stirring at room temperature for six h, dimethylamine (1 mmol, 40wt% water solution) was added and the resulting reaction mixture was stirred overnight. The reaction mixture was then added to cold water (50 ml) and the crude product was precipitated out. The crude product was purified by flash column chromatography [silica gel (#100–200), PE:EA = 10:1 to 5:1] to afford the title compound (27 mg, 12%) and colourless blocks were obtained by the slow evaporation of a petroleum ether/ethylacetate (*v*:*v* = 10:1) solution.

Refinement

Crystal data, data collection, and structure refinement details are summarized in Table 2.

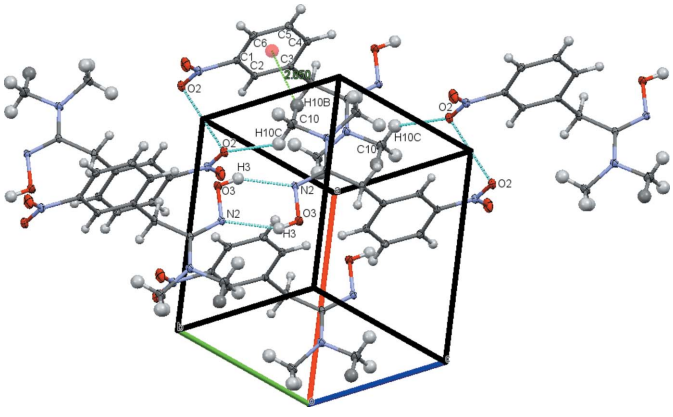


Figure 2
Packing diagram of the title compound viewed along the *c*-axis direction. Hydrogen bonds are drawn as dashed lines.

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

*Cg*1 is the centroid of the C1–C6 benzene ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···N2^{i}	0.82	2.11	2.8292 (19)	146
C10—H10*C*···O2^{ii}	0.96	2.51	3.288 (3)	139
C10—H10*B*···*Cg*1	0.96	2.85	3.718 (3)	151

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{10}H_{13}N_3O_3$
M_r	223.23
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	293
a, *b*, *c* (Å)	7.7740 (6), 8.4772 (5), 9.6599 (7)
α, β, γ (°)	66.595 (6), 77.917 (6), 76.113 (6)
V (Å^3)	562.49 (7)
Z	2
Radiation type	Mo *K*α
μ (mm^{-1})	0.10
Crystal size (mm)	0.25 × 0.22 × 0.12
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2015)
T_{min}, T_{max}	0.976, 0.988
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	6503, 2097, 1722
R_{int}	0.034
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.606
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.059, 0.157, 1.08
No. of reflections	2097
No. of parameters	148
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.30, −0.37

Computer programs: *APEX2* and *SAINT* (Bruker, 2015), *SHELXS97*, *SHELXL97* and *SHELXTL* (Sheldrick, 2008).

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full crystallographic data

IUCrData (2019). **4**, x190607 [https://doi.org/10.1107/S2414314619006072]

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Crystal data

$C_{10}H_{13}N_3O_3$
M_r = 223.23
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 7.7740 (6) Å
b = 8.4772 (5) Å
c = 9.6599 (7) Å
α = 66.595 (6)°
β = 77.917 (6)°
γ = 76.113 (6)°
V = 562.49 (7) Å^3
Z = 2
$F(000)$ = 236
D_x = 1.318 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2839 reflections
θ = 4.1–28.1°
μ = 0.10 mm^{-1}
T = 293 K
Block, colourless
0.25 × 0.22 × 0.12 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2015)
T_{min} = 0.976, T_{max} = 0.988
6503 measured reflections
2097 independent reflections
1722 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 25.5°, θ_{min} = 3.3°
h = −9→9
k = −10→10
l = −11→11

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.157
S = 1.08
2097 reflections
148 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
$w = 1/[\sigma^2(F_o^2) + (0.085P)^2 + 0.0695P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.2028 (2)	1.0701 (2)	−0.00206 (19)	0.0528 (5)
O2	0.1524 (2)	1.07407 (18)	−0.11426 (17)	0.0637 (5)
O1	0.1824 (3)	1.1983 (2)	0.0307 (2)	0.0957 (7)
C2	0.2750 (2)	0.7507 (2)	0.08584 (18)	0.0391 (4)
H2	0.2107	0.7551	0.0129	0.047*
C3	0.3558 (2)	0.5920 (2)	0.18285 (18)	0.0382 (4)
C1	0.2920 (2)	0.9027 (2)	0.09979 (19)	0.0407 (4)
C4	0.4542 (3)	0.5915 (3)	0.2875 (2)	0.0483 (5)
H4	0.5103	0.4856	0.3519	0.058*
C6	0.3873 (3)	0.9029 (3)	0.2038 (2)	0.0504 (5)
H6	0.3958	1.0069	0.2109	0.060*
C5	0.4703 (3)	0.7449 (3)	0.2977 (2)	0.0557 (5)
H5	0.5374	0.7417	0.3683	0.067*
C8	0.2728 (2)	0.2961 (2)	0.32899 (19)	0.0404 (4)
C7	0.3362 (3)	0.4217 (2)	0.17492 (19)	0.0473 (5)
H7A	0.2516	0.4460	0.1051	0.057*
H7B	0.4505	0.3681	0.1357	0.057*
N2	0.3767 (2)	0.16518 (18)	0.41274 (17)	0.0453 (4)
O3	0.55854 (18)	0.15786 (19)	0.34362 (18)	0.0665 (5)
H3	0.6176	0.0640	0.3900	0.100*
N3	0.0994 (2)	0.3282 (2)	0.38795 (19)	0.0583 (5)
C10	0.0425 (4)	0.2126 (4)	0.5410 (3)	0.0842 (8)
H10A	0.0344	0.1025	0.5387	0.126*
H10B	−0.0722	0.2645	0.5782	0.126*
H10C	0.1280	0.1947	0.6070	0.126*
C9	−0.0412 (3)	0.4295 (4)	0.2971 (3)	0.0848 (8)
H9A	0.0017	0.5260	0.2141	0.127*
H9B	−0.1400	0.4726	0.3584	0.127*
H9C	−0.0796	0.3576	0.2579	0.127*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0528 (10)	0.0383 (9)	0.0583 (10)	−0.0045 (7)	−0.0095 (8)	−0.0092 (8)
O2	0.0677 (10)	0.0513 (9)	0.0571 (9)	−0.0024 (7)	−0.0219 (7)	−0.0022 (7)
O1	0.1332 (18)	0.0386 (9)	0.1205 (15)	0.0089 (9)	−0.0488 (13)	−0.0320 (9)

C2	0.0387 (10)	0.0398 (10)	0.0346 (8)	−0.0017 (7)	−0.0061 (7)	−0.0116 (7)
C3	0.0347 (9)	0.0367 (9)	0.0356 (8)	−0.0014 (7)	−0.0007 (7)	−0.0102 (7)
C1	0.0390 (10)	0.0351 (9)	0.0401 (9)	−0.0046 (7)	−0.0014 (7)	−0.0084 (7)
C4	0.0432 (10)	0.0466 (11)	0.0431 (10)	−0.0056 (8)	−0.0117 (8)	−0.0022 (8)
C6	0.0540 (12)	0.0489 (11)	0.0503 (11)	−0.0186 (9)	−0.0032 (9)	−0.0166 (9)
C5	0.0571 (13)	0.0636 (13)	0.0488 (11)	−0.0222 (10)	−0.0172 (9)	−0.0111 (9)
C8	0.0478 (11)	0.0308 (9)	0.0410 (9)	0.0010 (7)	−0.0087 (8)	−0.0144 (7)
C7	0.0591 (12)	0.0357 (10)	0.0402 (9)	0.0032 (8)	−0.0065 (8)	−0.0131 (8)
N2	0.0419 (9)	0.0355 (8)	0.0483 (9)	0.0013 (6)	−0.0071 (7)	−0.0085 (6)
O3	0.0435 (8)	0.0507 (9)	0.0762 (10)	0.0071 (6)	−0.0023 (7)	−0.0045 (7)
N3	0.0421 (10)	0.0573 (10)	0.0576 (10)	0.0055 (8)	−0.0053 (7)	−0.0110 (8)
C10	0.0610 (15)	0.0939 (19)	0.0661 (14)	−0.0061 (13)	0.0136 (12)	−0.0117 (13)
C9	0.0521 (14)	0.0740 (16)	0.108 (2)	0.0076 (12)	−0.0260 (14)	−0.0152 (14)

Geometric parameters (Å, º)

N1—O2	1.213 (2)	C8—N3	1.357 (2)
N1—O1	1.214 (2)	C8—C7	1.509 (2)
N1—C1	1.472 (2)	C7—H7A	0.9700
C2—C3	1.386 (2)	C7—H7B	0.9700
C2—C1	1.387 (2)	N2—O3	1.429 (2)
C2—H2	0.9300	O3—H3	0.8200
C3—C4	1.387 (3)	N3—C9	1.434 (3)
C3—C7	1.521 (2)	N3—C10	1.458 (3)
C1—C6	1.368 (3)	C10—H10A	0.9600
C4—C5	1.379 (3)	C10—H10B	0.9600
C4—H4	0.9300	C10—H10C	0.9600
C6—C5	1.378 (3)	C9—H9A	0.9600
C6—H6	0.9300	C9—H9B	0.9600
C5—H5	0.9300	C9—H9C	0.9600
C8—N2	1.292 (2)		
O2—N1—O1	122.95 (17)	C8—C7—C3	111.66 (14)
O2—N1—C1	118.80 (15)	C8—C7—H7A	109.3
O1—N1—C1	118.24 (18)	C3—C7—H7A	109.3
C3—C2—C1	118.62 (16)	C8—C7—H7B	109.3
C3—C2—H2	120.7	C3—C7—H7B	109.3
C1—C2—H2	120.7	H7A—C7—H7B	107.9
C4—C3—C2	118.85 (16)	C8—N2—O3	111.70 (14)
C4—C3—C7	120.66 (15)	N2—O3—H3	109.5
C2—C3—C7	120.50 (16)	C8—N3—C9	123.55 (18)
C6—C1—C2	122.78 (17)	C8—N3—C10	117.97 (17)
C6—C1—N1	119.16 (16)	C9—N3—C10	115.5 (2)
C2—C1—N1	118.06 (16)	N3—C10—H10A	109.5
C5—C4—C3	121.24 (17)	N3—C10—H10B	109.5
C5—C4—H4	119.4	H10A—C10—H10B	109.5
C3—C4—H4	119.4	N3—C10—H10C	109.5
C1—C6—C5	118.26 (18)	H10A—C10—H10C	109.5

C1—C6—H6	120.9	H10B—C10—H10C	109.5
C5—C6—H6	120.9	N3—C9—H9A	109.5
C6—C5—C4	120.23 (18)	N3—C9—H9B	109.5
C6—C5—H5	119.9	H9A—C9—H9B	109.5
C4—C5—H5	119.9	N3—C9—H9C	109.5
N2—C8—N3	117.67 (16)	H9A—C9—H9C	109.5
N2—C8—C7	123.72 (16)	H9B—C9—H9C	109.5
N3—C8—C7	118.47 (15)		

C1—C2—C3—C4	−1.6 (2)	C1—C6—C5—C4	−1.0 (3)
C1—C2—C3—C7	178.20 (15)	C3—C4—C5—C6	0.4 (3)
C3—C2—C1—C6	0.9 (3)	N2—C8—C7—C3	−101.67 (19)
C3—C2—C1—N1	−178.90 (14)	N3—C8—C7—C3	73.9 (2)
O2—N1—C1—C6	164.10 (17)	C4—C3—C7—C8	51.8 (2)
O1—N1—C1—C6	−16.2 (3)	C2—C3—C7—C8	−128.00 (17)
O2—N1—C1—C2	−16.1 (3)	N3—C8—N2—O3	−173.71 (15)
O1—N1—C1—C2	163.65 (19)	C7—C8—N2—O3	1.9 (2)
C2—C3—C4—C5	1.0 (3)	N2—C8—N3—C9	−159.9 (2)
C7—C3—C4—C5	−178.82 (17)	C7—C8—N3—C9	24.3 (3)
C2—C1—C6—C5	0.4 (3)	N2—C8—N3—C10	−0.2 (3)
N1—C1—C6—C5	−179.78 (16)	C7—C8—N3—C10	−176.0 (2)

Hydrogen-bond geometry (Å, º)

*Cg*1 is the centroid of the C1–C6 benzene ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···N2^{i}	0.82	2.11	2.8292 (19)	146
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Symmetry codes: (i) −*x*+1, −*y*, −*z*+1; (ii) *x*, *y*−1, *z*+1.