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(*E*)-*N*-[(Anthracen-9-yl)methylidene]hydroxylamine

Jihad Sebhaoui,[a]* Youness El Bakri,[a] El Mokhtar Essassi[a] and Joel T. Mague[b]

[a]Laboratoire de Chimie Organique Hétérocyclique, URAC 21, Pôle de Compétence Pharmacochimie, Av Ibn Battouta, BP 1014, Faculté des Sciences, Mohammed V University, Rabat, Morocco, and [b]Department of Chemistry, Tulane University, New Orleans, LA 70118, USA. *Correspondence e-mail: sebhaoui.jihad@gmail.com

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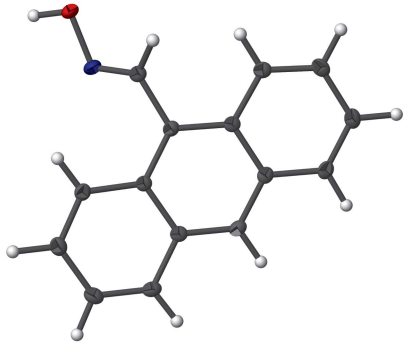
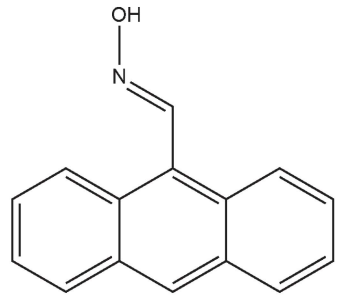
In the title compound, $C_{15}H_{11}NO$, the anthracene unit is slightly bowed. Inversion-related pairs of O—H···N hydrogen bonds form dimers that are stacked along the *b*-axis direction by offset π–π stacking interactions between the anthracene units.

3D view



Chemical scheme



Structure description

Oximes constitute the key structural motif in numerous drug scaffolds and bioactive compounds, including macrolide antibiotics (roxithromycin), antifungal agents (econazole), cytotoxic agents (Park *et al.*, 2005) and anti-inflammatory derivatives (cloximate). They are also intermediates in the production of isoxazole derivatives by 1,3-dipolar cycloaddition (Tribak *et al.*, 2017; Al Houari *et al.*, 2008). It is noteworthy that these compounds are also used in several important synthetic reactions, including oxime-carbapalladacycle-catalysed Suzuki cross-coupling of aryl chlorides in water (Botella & Nájera, 2002), nucleophilic catalysis of oxime ligation (Dirksen *et al.*, 2006) and palladium-catalysed amination of aromatic C—H bonds with oxime esters (Tan & Hartwig, 2010).

In the title compound (Fig. 1), the anthracene moiety is slightly bowed, as indicated by the dihedral angles of 2.77 (4) and 2.13 (4)°, respectively, that the C2–C7 and C9–C14 rings make with the central ring. The hydroxylimino side chain is rotated out of the mean plane of the anthracene unit, as indicated by the C2—C1—C15—N1 torsion angle of −42.5 (1)° and C1—C15—N1—O1 torsion angle of −175.87 (8)°.

In the crystal, inversion-related pairs of O—H···N hydrogen bonds form dimers and generate $R_2^2(6)$ rings (Table 1). These dimers form parallel stacks along the *b*-axis direction through offset π–π-stacking interactions between the anthracene moieties (Figs. 2 and 3). The centroid–centroid distances between equivalent rings of adjacent anthra-

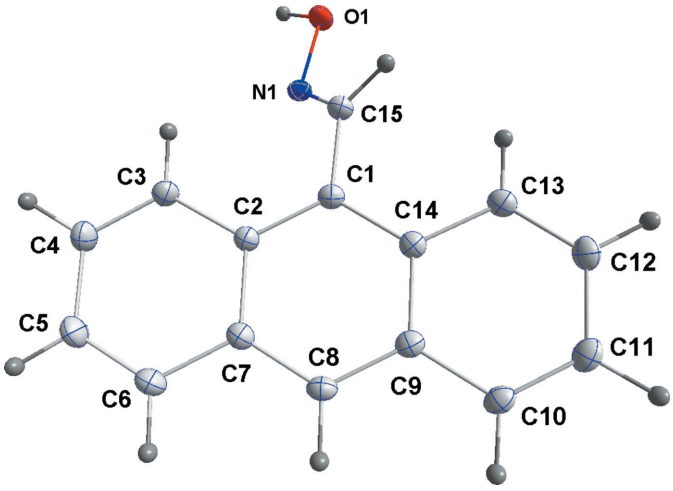


Figure 1
The title molecule with labelling scheme and 50% probability displacement ellipsoids.

cene ring systems are 3.869 (1) Å. The mean planes of the anthracene moieties are inclined by 24.43 (1)° to [010].

Synthesis and crystallization

A solution of 2.00 g (9.70 mmol) 9-anthraldehyde, 1.35 g) (19.39 mmol) hydroxylamine hydrochloride and 775.74 mg) (19.39 mmol) of sodium hydroxide in 30 ml of ethanol and 10 ml of water, was refluxed for 30 min. The mixture reaction was neutralized with a solution of HCl, and extracted with dichloromethane. The solvent was removed under reduced pressure and the residue was recrystallized from ethanol ethanol to afford the title compound as light-yellow plates.

Refinement

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

Acknowledgements

JTM thanks Tulane University for support of the Tulane Crystallography Laboratory.

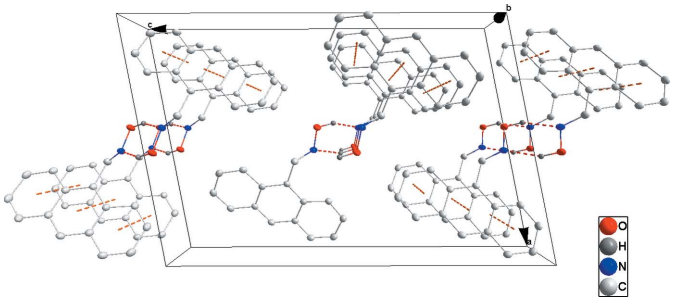


Figure 2
Packing viewed along the *b*-axis direction, with O—H···N hydrogen bonds and offset π-stacking interactions shown, respectively, as red and orange dashed lines.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1^{i}	0.954 (15)	1.937 (15)	2.8175 (12)	152.4 (13)

Symmetry code: (i) $-x+1, -y+1, -z+1$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{15}H_{11}NO$
M_r	221.25
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	100
a, *b*, *c* (Å)	13.463 (3), 3.8694 (8), 20.377 (4)
β (°)	101.023 (3)
V (Å^3)	1041.9 (4)
Z	4
Radiation type	Mo *Kα*
μ (mm^{-1})	0.09
Crystal size (mm)	0.38 × 0.26 × 0.06
Data collection	
Diffractometer	Bruker SMART APEX CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2016)
T_{min}, T_{max}	0.93, 0.99
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	18825, 2790, 2217
R_{int}	0.035
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.685
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.044, 0.125, 1.04
No. of reflections	2790
No. of parameters	198
H-atom treatment	All H-atom parameters refined
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.44, −0.24

Computer programs: *APEX3* and *SAINT* (Bruker, 2016), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*), *DIAMOND* (Brandenburg & Putz, 2012) and *SHELXTL* (Sheldrick, 2008).

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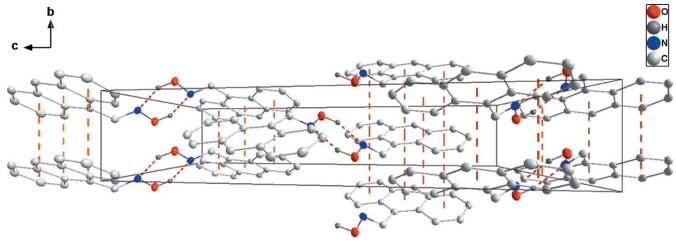

Figure 3
Packing viewed along the *a*-axis direction, with O—H···N hydrogen bonds and offset π-stacking interactions shown, respectively, as red and orange dashed lines.

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full crystallographic data

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Crystal data

$C_{15}H_{11}NO$
$M_r = 221.25$
Monoclinic, $P2_1/c$
$a = 13.463$ (3) Å
$b = 3.8694$ (8) Å
$c = 20.377$ (4) Å
$\beta = 101.023$ (3)°
$V = 1041.9$ (4) Å^3
$Z = 4$
$F(000) = 464$
$D_x = 1.410$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 7044 reflections
$\theta = 2.3$–$29.1°$
$\mu = 0.09$ mm^{-1}
$T = 100$ K
Plate, light yellow
0.38 × 0.26 × 0.06 mm

Data collection

Bruker SMART APEX CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 8.3333 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2016)
$T_{min} = 0.93$, $T_{max} = 0.99$
18825 measured reflections
2790 independent reflections
2217 reflections with $I > 2\sigma(I)$
$R_{int} = 0.035$
$\theta_{max} = 29.1°$, $\theta_{min} = 1.5°$
$h = -18\rightarrow18$
$k = -5\rightarrow5$
$l = -27\rightarrow27$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.125$
$S = 1.04$
2790 reflections
198 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0909P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.44$ e Å^{-3}
$\Delta\rho_{min} = -0.24$ e Å^{-3}

Special details

Experimental. The diffraction data were obtained from 3 sets of 400 frames, each of width 0.5° in ω, colllected at φ = 0.00, 90.00 and 180.00° and 2 sets of 800 frames, each of width 0.45° in φ, collected at ω = –30.00 and 210.00°. The scan time was 15 sec/frame.

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}
O1	0.54777 (5)	0.21769 (19)	0.45469 (4)	0.0199 (2)
H1	0.5653 (11)	0.309 (4)	0.4989 (8)	0.042 (4)*
N1	0.45330 (6)	0.3719 (2)	0.43123 (4)	0.0168 (2)
C1	0.31053 (7)	0.3734 (2)	0.34044 (5)	0.0140 (2)
C2	0.23196 (7)	0.4734 (2)	0.37368 (5)	0.0138 (2)
C3	0.23542 (7)	0.4212 (3)	0.44355 (5)	0.0164 (2)
H3	0.2936 (10)	0.307 (3)	0.4700 (6)	0.024 (3)*
C4	0.15692 (7)	0.5188 (3)	0.47305 (5)	0.0188 (2)
H4	0.1611 (10)	0.475 (4)	0.5242 (7)	0.029 (3)*
C5	0.06890 (8)	0.6760 (3)	0.43518 (5)	0.0194 (2)
H5	0.0123 (11)	0.754 (3)	0.4558 (7)	0.029 (3)*
C6	0.06159 (7)	0.7253 (2)	0.36835 (5)	0.0177 (2)
H6	0.0024 (10)	0.828 (3)	0.3405 (6)	0.023 (3)*
C7	0.14162 (7)	0.6253 (2)	0.33526 (5)	0.0148 (2)
C8	0.13267 (7)	0.6712 (2)	0.26652 (5)	0.0159 (2)
H8	0.0719 (9)	0.784 (3)	0.2404 (6)	0.019 (3)*
C9	0.20768 (7)	0.5601 (2)	0.23283 (5)	0.0149 (2)
C10	0.19705 (7)	0.6009 (3)	0.16186 (5)	0.0168 (2)
H10	0.1347 (11)	0.712 (3)	0.1382 (6)	0.029 (3)*
C11	0.26954 (7)	0.4857 (3)	0.12932 (5)	0.0190 (2)
H11	0.2620 (9)	0.508 (4)	0.0774 (6)	0.028 (3)*
C12	0.35777 (8)	0.3213 (3)	0.16552 (5)	0.0186 (2)
H12	0.4090 (10)	0.228 (3)	0.1414 (6)	0.024 (3)*
C13	0.37252 (8)	0.2879 (3)	0.23316 (5)	0.0166 (2)
H13	0.4333 (10)	0.174 (3)	0.2577 (6)	0.025 (3)*
C14	0.29814 (7)	0.4042 (2)	0.26996 (5)	0.0141 (2)
C15	0.40798 (7)	0.2371 (3)	0.37667 (5)	0.0165 (2)
H15	0.4403 (9)	0.043 (3)	0.3571 (6)	0.023 (3)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0135 (4)	0.0277 (4)	0.0173 (4)	0.0058 (3)	−0.0002 (3)	−0.0001 (3)
N1	0.0120 (4)	0.0192 (4)	0.0184 (4)	0.0016 (3)	0.0008 (3)	0.0028 (3)
C1	0.0129 (4)	0.0131 (4)	0.0154 (5)	−0.0015 (3)	0.0010 (3)	−0.0007 (3)
C2	0.0130 (4)	0.0132 (4)	0.0151 (5)	−0.0013 (3)	0.0022 (3)	−0.0006 (3)
C3	0.0149 (4)	0.0182 (5)	0.0153 (5)	−0.0016 (4)	0.0014 (4)	0.0000 (4)

C4	0.0190 (5)	0.0211 (5)	0.0168 (5)	−0.0025 (4)	0.0045 (4)	−0.0011 (4)
C5	0.0178 (5)	0.0197 (5)	0.0219 (5)	0.0001 (4)	0.0070 (4)	−0.0032 (4)
C6	0.0140 (5)	0.0165 (5)	0.0219 (5)	0.0014 (4)	0.0022 (4)	−0.0013 (4)
C7	0.0138 (4)	0.0128 (4)	0.0176 (5)	−0.0014 (3)	0.0026 (4)	−0.0012 (3)
C8	0.0133 (4)	0.0149 (4)	0.0182 (5)	−0.0003 (4)	0.0001 (4)	0.0006 (4)
C9	0.0144 (4)	0.0136 (4)	0.0159 (5)	−0.0028 (3)	0.0013 (4)	−0.0001 (3)
C10	0.0157 (5)	0.0181 (5)	0.0155 (5)	−0.0038 (4)	0.0002 (4)	0.0012 (4)
C11	0.0206 (5)	0.0207 (5)	0.0157 (5)	−0.0056 (4)	0.0034 (4)	−0.0008 (4)
C12	0.0189 (5)	0.0191 (5)	0.0192 (5)	−0.0037 (4)	0.0074 (4)	−0.0025 (4)
C13	0.0141 (5)	0.0164 (5)	0.0192 (5)	−0.0015 (4)	0.0030 (4)	−0.0006 (4)
C14	0.0131 (4)	0.0129 (4)	0.0161 (5)	−0.0024 (3)	0.0024 (4)	−0.0015 (3)
C15	0.0157 (5)	0.0168 (5)	0.0169 (5)	0.0015 (4)	0.0029 (4)	0.0006 (3)

Geometric parameters (Å, º)

O1—N1	1.4036 (10)	C6—H6	0.971 (13)
O1—H1	0.954 (15)	C7—C8	1.3940 (14)
N1—C15	1.2733 (13)	C8—C9	1.3932 (14)
C1—C2	1.4148 (12)	C8—H8	0.989 (12)
C1—C14	1.4188 (13)	C9—C10	1.4344 (14)
C1—C15	1.4745 (14)	C9—C14	1.4377 (14)
C2—C3	1.4300 (13)	C10—C11	1.3561 (14)
C2—C7	1.4399 (13)	C10—H10	0.984 (14)
C3—C4	1.3652 (13)	C11—C12	1.4228 (15)
C3—H3	0.968 (13)	C11—H11	1.047 (12)
C4—C5	1.4214 (14)	C12—C13	1.3605 (15)
C4—H4	1.047 (13)	C12—H12	0.988 (13)
C5—C6	1.3598 (15)	C13—C14	1.4334 (13)
C5—H5	0.985 (14)	C13—H13	0.980 (13)
C6—C7	1.4294 (13)	C15—H15	0.988 (12)
N1—O1—H1	102.1 (9)	C9—C8—H8	118.3 (7)
C15—N1—O1	112.02 (8)	C7—C8—H8	120.0 (7)
C2—C1—C14	120.51 (8)	C8—C9—C10	121.39 (9)
C2—C1—C15	122.23 (8)	C8—C9—C14	119.40 (9)
C14—C1—C15	117.26 (8)	C10—C9—C14	119.20 (9)
C1—C2—C3	123.58 (8)	C11—C10—C9	120.96 (9)
C1—C2—C7	118.85 (8)	C11—C10—H10	122.1 (7)
C3—C2—C7	117.54 (8)	C9—C10—H10	116.9 (7)
C4—C3—C2	121.25 (9)	C10—C11—C12	120.06 (9)
C4—C3—H3	119.6 (7)	C10—C11—H11	121.8 (7)
C2—C3—H3	119.2 (7)	C12—C11—H11	118.1 (7)
C3—C4—C5	120.96 (9)	C13—C12—C11	120.89 (9)
C3—C4—H4	119.6 (7)	C13—C12—H12	119.2 (8)
C5—C4—H4	119.4 (7)	C11—C12—H12	119.9 (8)
C6—C5—C4	119.80 (9)	C12—C13—C14	121.26 (9)
C6—C5—H5	118.0 (8)	C12—C13—H13	120.4 (7)
C4—C5—H5	122.2 (8)	C14—C13—H13	118.3 (7)

C5—C6—C7	121.18 (9)	C1—C14—C13	122.99 (9)
C5—C6—H6	122.5 (7)	C1—C14—C9	119.46 (8)
C7—C6—H6	116.4 (7)	C13—C14—C9	117.55 (9)
C8—C7—C6	120.76 (9)	N1—C15—C1	121.50 (9)
C8—C7—C2	120.00 (8)	N1—C15—H15	119.2 (7)
C6—C7—C2	119.24 (9)	C1—C15—H15	119.2 (7)
C9—C8—C7	121.62 (9)		

C14—C1—C2—C3	174.19 (8)	C8—C9—C10—C11	−178.66 (9)
C15—C1—C2—C3	−6.34 (14)	C14—C9—C10—C11	1.97 (14)
C14—C1—C2—C7	−3.51 (13)	C9—C10—C11—C12	0.02 (15)
C15—C1—C2—C7	175.96 (8)	C10—C11—C12—C13	−2.31 (15)
C1—C2—C3—C4	−179.17 (9)	C11—C12—C13—C14	2.53 (15)
C7—C2—C3—C4	−1.44 (14)	C2—C1—C14—C13	−176.13 (8)
C2—C3—C4—C5	0.00 (15)	C15—C1—C14—C13	4.38 (13)
C3—C4—C5—C6	1.12 (15)	C2—C1—C14—C9	4.20 (14)
C4—C5—C6—C7	−0.72 (15)	C15—C1—C14—C9	−175.29 (8)
C5—C6—C7—C8	178.83 (9)	C12—C13—C14—C1	179.84 (9)
C5—C6—C7—C2	−0.75 (14)	C12—C13—C14—C9	−0.49 (14)
C1—C2—C7—C8	0.05 (13)	C8—C9—C14—C1	−1.43 (14)
C3—C2—C7—C8	−177.79 (8)	C10—C9—C14—C1	177.96 (8)
C1—C2—C7—C6	179.63 (8)	C8—C9—C14—C13	178.89 (8)
C3—C2—C7—C6	1.79 (13)	C10—C9—C14—C13	−1.72 (13)
C6—C7—C8—C9	−176.82 (8)	O1—N1—C15—C1	−175.87 (8)
C2—C7—C8—C9	2.75 (14)	C2—C1—C15—N1	−42.53 (14)
C7—C8—C9—C10	178.57 (8)	C14—C1—C15—N1	136.96 (10)
C7—C8—C9—C14	−2.05 (14)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1^{i}	0.954 (15)	1.937 (15)	2.8175 (12)	152.4 (13)

Symmetry code: (i) −*x*+1, −*y*+1, −*z*+1.