research communications

Crystal structures of (*E*)-1-naphthaldehyde oxime and (*E*)-phenanthrene-9-carbaldehyde oxime

Jamal Lasri,[a]* Katherine Chulvi[b]* and Naser Eltaher Eltayeb[a]

[a]Department of Chemistry, Rabigh College of Science and Arts, PO Box 344, King Abdulaziz University, Jeddah, Saudi Arabia, and [b]FB 1.3 Strukturanalytik, Bundesanstalt für Materialforschung und -prüfung (BAM), Berlin, Germany. *Correspondence e-mail: jlasri@kau.edu.sa, katherine.chulvi-iborra@bam.de

The aldoximes $C_{11}H_9NO$ (I) and $C_{15}H_{11}NO$ (II), synthesized in *ca* 90% yield, by treatment of 1-naphthaldehyde or phenanthrene-9-carbaldehyde, respectively, with hydroxylamine hydrochloride and sodium carbonate, have been characterized by IR, ^{1}H, ^{13}C and DEPT-135 NMR spectroscopies, and also by single-crystal X-ray diffraction analysis. The molecules of (I) and (II) are conformationally similar, with the aldoxime substituent groups lying outside the planes of the naphthalene or phenanthrene rings, forming dihedral angles with them of 23.9 (4) and 27.9 (6)°, respectively. The crystal structures of both (I) and (II) are similar with a single intermolecular O—H···N hydrogen-bonding interaction, giving rise to the formation of one-dimensional polymeric chains extending along the 2_1 (*b*) screw axes in each.

1. Chemical context

Oxime compounds have found many applications; for example in the medical field, they are used as antidotes for nerve agents (Kassa, 2002). Oximes are also used as intermediates in the industrial production of caprolactam, a precursor to Nylon 6 (Ritz *et al.*, 2012). Oximes, $HO-N{=}CR^1R^2$, are also valuable and simple reagents containing the $O-N{=}C$ moiety (Kukushkin & Pombeiro, 1999), which easily adds to nitrile ligands, to form a variety of nitrogen-containing products *e.g.* iminoacylated compounds (Kopylovich *et al.*, 2009; Lasri *et al.*, 2007, 2008), amidines (Kopylovich *et al.*, 2001), carboxamides (Kopylovich *et al.*, 2002), phthalocyanines (Kopylovich *et al.*, 2004), or 1,3,5-triazapentadiene species (Kopylovich *et al.*, 2007). In this work, we report the synthesis and crystal structures of two aldoximes, *viz.* (*E*)-1-naphthaldehyde oxime (I) and (*E*)-phenanthrene-9-carbaldehyde oxime (II), by treatment of 1-naphthaldehyde or phenanthrene-9-carbaldehyde, respectively, with hydroxylamine hydrochloride and sodium carbonate.
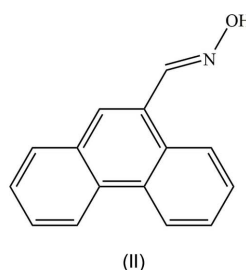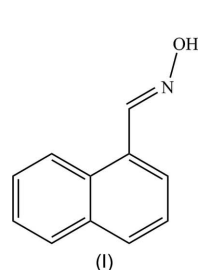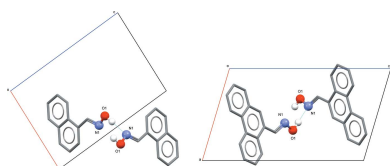
Table 1
Selected torsion angles (°) for the aldoxime groups in (I) and (II).

	Compound (I)	Compound (II)
C1/C9—C11—N1—O1	−175.5 (4)	−175.3 (2)
C2/C10—C1/C9—C11—N1	23.6 (6)	27.6 (4)
C8′—C1—C11—N1	−160.4 (4)	–
C8′—C9—C11—N1	–	−156.1 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1^i	0.90 (6)	1.94 (6)	2.834 (5)	177 (6)

Symmetry code: (i) $-x + 2, y - \frac{1}{2}, -z + 1$.

Table 3
Hydrogen-bond geometry (Å, °) for (II).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H···N1^i	0.88 (3)	1.99 (3)	2.852 (3)	169 (3)

Symmetry code: (i) $-x + 1, y + \frac{1}{2}, -z + 1$.

2. Structural commentary

The title compounds (I) and (II) both crystallize in the non-centrosymmetric monoclinic space group $P2_1$ with $Z = 2$, with similar unit-cell parameters. The asymmetric unit contents for each are shown in Figs. 1 and 2. Compound (I) comprises a naphthalene unit functionalized with an aldoxime group at position 1. The naphthalene unit is, as expected, essentially
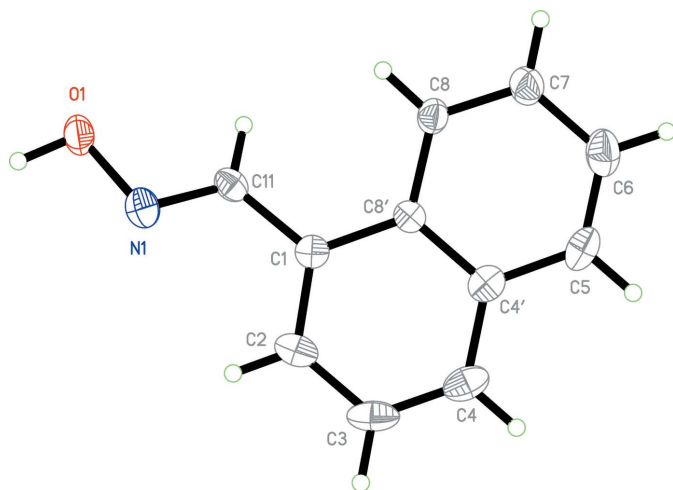

Figure 1
The molecular conformation and atom-numbering scheme for (I), with non-H atoms represented as 30% probability ellipsoids.
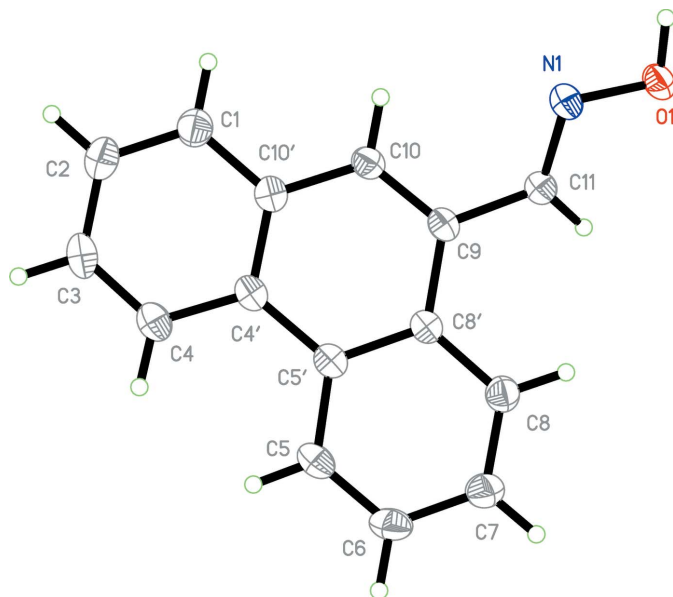

Figure 2
The molecular conformation and atom-numbering scheme for (II), with non-H atoms represented as 30% probability ellipsoids.

planar but the plane containing the aldoxime atoms lies significantly out of the naphthalene plane [torsion angle N1—C11—C1—C2 = 23.6 (6)°] (Table 1). In the case of compound (II), the plane of the aldoxime group lies similarly out-of-plane with the phenanthrene ring system [comparative torsion angle N1—C11—C9—C10 = 27.6 (4)°], corresponding to dihedral angles between the two planes of 23.9 (4) and 27.9 (5)° for (I) and (II), respectively. The aldoxime group shows similar bond lengths for both structures: 1.395 (5) and 1.405 (3) Å for O1—N1, 1.273 (5) and 1.268 (3) Å for N1—C11, 1.461 (6) and 1.466 (4) Å for C1—C11 or C9—C11, for (I) and (II), respectively.

3. Supramolecular features

Similar intermolecular interactions are observed in the crystal structures of both (I) and (II). In each, molecules are linked through a single intermolecular O1—H···N1^i hydrogen-bonding interaction [Tables 2 and 3 for (I) and (II), respectively]. These basic interactions are shown in Fig. 3, defining an oxime $C(3)$ type II motif. It is well known that oximes are able to form different types of hydrogen-bonding motifs (Bruton *et al.*, 2003). In the structures of both (I) and (II), the formation of a one-dimensional polymeric chain arrangement of molecules results, extending along the 2_1 (b) screw axes in each (Fig. 4).
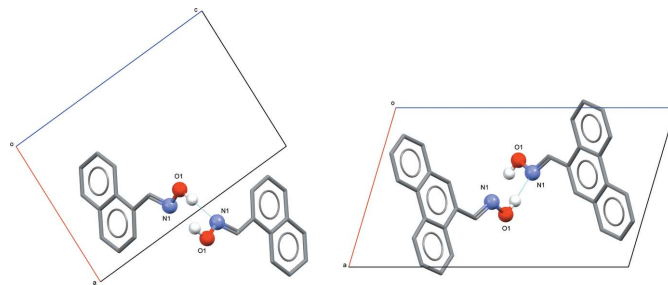

Figure 3
Intermolecular hydrogen-bonding associations for (I) (left) and (II) (right), shown as dashed lines. Non-associative H atoms have been omitted for clarity.
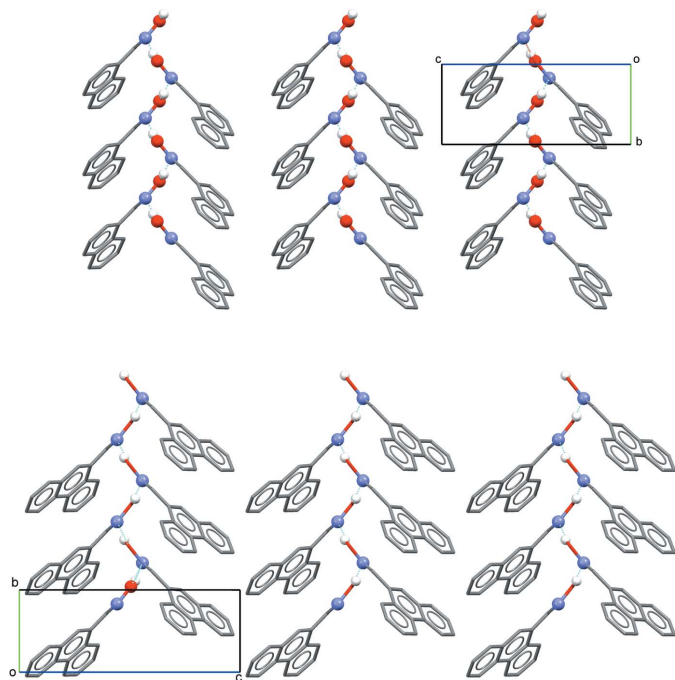
Figure 4
A packing diagram viewed along the *a* axis for (I) (top) and (II) (bottom), showing polymeric chain extensions.

4. Database survey

Many naphthalene-carbaldehyde oxime derivatives are present in the Cambridge Structural Database (Version 5.38; Groom *et al.* 2016) but no one crystal structure containing only an aldoxime group in position 1 of the naphthalene ring system has been reported. The most similar structures that can be found are LIVROY/LIVROY01 (Guo *et al.*, 2008; Tarai & Baruah, 2016) with an additional hydroxyl group in position 2 and TIJPOS (Asaad *et al.*, 2005) with a dimethylamino group in position 9. The most important difference between (I) and LIVROY/LIVROY01 are the two hydrogen bonds: one intramolecular O—H⋯N and another intermolecular O—H⋯O. As a result of the intramolecular hydrogen-bonding interaction, the aldoxime group in the latter compound is coplanar with the central naphthalene ring with a dihedral angle of 1.21° and torsion angles C1—C11—N1—O2 = 179.27, C3—C1—C11—N1 = −179.91 and C4—C1—C11—N1 = −0.76°. However, TIJPOS (Asaad *et al.*, 2005), with just one type of intermolecular hydrogen bond, shows a rotation in the aldoxime group that is more dramatic than in (I) and (II) (Table 1), with a 40.35° deviation from the central naphthalene plane.

No examples of structures of phenanthrene-carbaldehyde oxime derivatives are present in the Cambridge Structural Database.

5. Synthesis and crystallization

The aldoximes (*E*)-1-naphthaldehyde oxime (I) and (*E*)-phenanthrene-9-carbaldehyde oxime (II) were synthesized, in

ca 90% yield, by treatment of 1-naphthaldehyde or phenanthrene-9-carbaldehyde, respectively, with hydroxylamine hydrochloride and sodium carbonate in MeOH at room temperature. To a solution of hydroxylamine hydrochloride (41.6 mg, 0.60 mmol) in MeOH (10 ml) was added sodium carbonate (31.7 mg, 0.30 mmol). The reaction mixture was stirred at room temperature for 5 min. 1-Naphthaldehyde (85.0 mg, 0.54 mmol) or phenanthrene-9-carbaldehyde (112.2 mg, 0.54 mmol) was added and the reaction mixture was stirred at room temperature for 12 h. The precipitate formed was then filtered off and the filtrate was evaporated *in vacuo*. The crude residue was purified by column chromatography on silica (CHCl$_3$ as the eluent, 50 ml), followed by evaporation of the solvent *in vacuo* to give the pure aldoximes [(I), 84 mg, 90% yield and (II), 107 mg, 89% yield] (see reaction scheme).
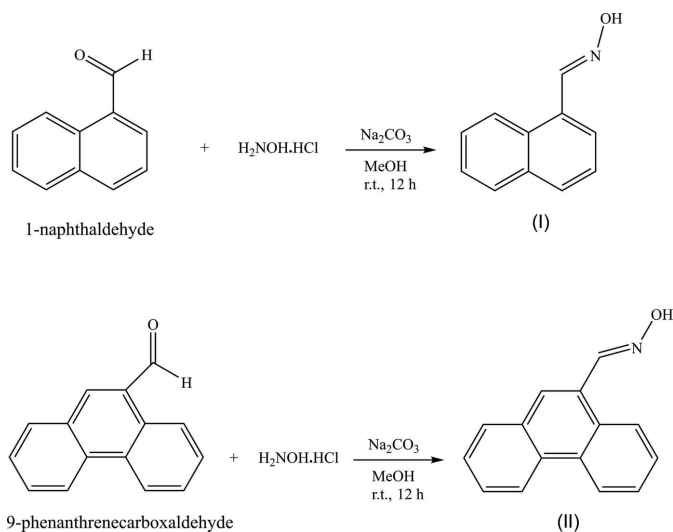
Single crystals of the aldoximes (I) and (II) suitable for X-ray diffraction were obtained by slow evaporation of their 10 ml CHCl$_3$ solutions at room temperature. Compounds (I) and (II) were characterized by IR, ^{1}H, ^{13}C and DEPT-135 NMR spectroscopies and also by single crystal X-ray diffraction analysis.

In the IR spectra of (I) and (II), the characteristic bands at wavenumbers 3389 and 3200 cm^{-1} (O—H), and 1614 and 1607 cm^{-1} (C=N), confirm the formation of the aldoximes (I) and (II), respectively. In the ^{1}H NMR spectra, we observed the absence of the signal of the aldehyde at *ca* 10 ppm and a new signal at *ca* 8.8 ppm due to the imine proton C*H*=N was detected. Moreover, in the ^{13}C and DEPT-135 NMR spectra, the signal of the aldehyde at *ca* 190 ppm was not observed, and a new signal at *ca* 150 ppm due to the oxime carbon C*H*=NOH was detected, confirming the formation of the aldoximes (I) and (II).

(*E*)-1-naphthaldehyde oxime (I)
Yield: 90%. IR (cm^{-1}): 3389 (OH), 1614 (C=N). ^{1}H NMR (CDCl$_3$), δ: 7.53 (*t*, J_{HH} 7.5 Hz, 1H, C*H*$_{aromatic}$), 7.56 (*t*, J_{HH} 7.0 Hz, 1H, C*H*$_{aromatic}$), 7.61 (*t*, J_{HH} 7.0 Hz, 1H, C*H*$_{aromatic}$), 7.82 (*d*, J_{HH} 7.1 Hz, 1H, C*H*$_{aromatic}$), 7.93 (*t*, J_{HH} 8.1 Hz, 2H,

Table 4
Experimental details.

	(I)	(II)
Crystal data		
Chemical formula	$C_{11}H_9NO$	$C_{15}H_{11}NO$
M_r	171.19	221.25
Crystal system, space group	Monoclinic, $P2_1$	Monoclinic, $P2_1$
Temperature (K)	295	295
a, b, c (Å)	7.928 (5), 4.843 (3), 11.444 (7)	8.2397 (8), 4.9728 (5), 13.9332 (14)
β (°)	94.03 (5)	106.680 (7)
V (Å^3)	438.3 (5)	546.88 (10)
Z	2	2
Radiation type	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.08	0.09
Crystal size (mm)	$0.10 \times 0.06 \times 0.02$	$0.16 \times 0.09 \times 0.05$
Data collection		
Diffractometer	Bruker D8 Quest	Bruker D8 Quest
Absorption correction	Multi-scan (*SADABS*; Bruker, 2016)	Multi-scan (*SADABS*; Bruker, 2016)
T_{min}, T_{max}	0.684, 0.745	0.698, 0.745
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	5762, 1570, 957	7330, 1988, 1509
R_{int}	0.100	0.053
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.603	0.602
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.055, 0.098, 1.06	0.040, 0.093, 1.04
No. of reflections	1570	1988
No. of parameters	122	159
No. of restraints	1	1
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å^{-3})	0.15, −0.15	0.15, −0.15

Computer programs: *APEX3* and *SAINT* (Bruker, 2016), *SHELXT2014* (Sheldrick, 2015a) and *SHELXL2014* (Sheldrick, 2015b).

$CH_{aromatic}$), 8.48 (*d*, J_{HH} 8.3 Hz, 1H, $CH_{aromatic}$), 8.87 (*s*, 1H, $CH{=}N$). ^{13}C NMR (CDCl$_3$), δ: 124.2, 125.4, 126.2, 127.0, 127.1 ($CH_{aromatic}$), 128.0 ($C_{aromatic}$), 128.8, 130.6 ($CH_{aromatic}$), 130.8, 133.8 ($C_{aromatic}$), 150.0 ($CH{=}N$). DEPT-135 NMR (CDCl$_3$), δ: 124.2, 125.4, 126.2, 127.0, 127.1, 128.8, 130.6 ($CH_{aromatic}$), 150.0 ($CH{=}N$).

E-phenanthrene-9-carbaldehyde oxime (II)

Yield: 89%. IR (cm^{-1}): 3200 (OH), 1607 (C=N). ^1H NMR (CDCl$_3$), δ: 7.64 (*t*, J_{HH} 7.9 Hz, 1H, $CH_{aromatic}$), 7.68–7.75 (m, 3H, $CH_{aromatic}$), 7.94 (*d*, J_{HH} 7.9 Hz, 1H, $CH_{aromatic}$), 8.04 (*s*, 1H, $CH_{aromatic}$), 8.62 (*d*, J_{HH} 7.9 Hz, 1H, $CH_{aromatic}$), 8.70 (*d*, J_{HH} 8.2 Hz, 1H, $CH_{aromatic}$), 8.77 (*d*, J_{HH} 8.2 Hz, 1H, $CH_{aromatic}$), 8.85 (*s*, 1H, $CH{=}N$). ^{13}C NMR (CDCl$_3$), δ: 122.6, 123.1, 125.4 ($CH_{aromatic}$), 126.8 ($C_{aromatic}$), 126.9, 127.0, 127.2, 127.9, 129.3 ($CH_{aromatic}$), 130.7, 131.0, 131.1 ($C_{aromatic}$), 150.8 ($CH{=}N$). DEPT-135 NMR (CDCl$_3$), δ: 122.6, 123.1, 125.4, 126.9, 127.0, 127.2, 127.9, 129.3 ($CH_{aromatic}$), 150.8 ($CH{=}N$).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 4. All C-bound H atoms were located in difference-Fourier maps but were subsequently treated as riding with C—H = 0.93 Å and with $U_{iso}(H) = 1.2U_{eq}(C)$. The H atoms of the OH groups were positioned with idealized geometry and were refined freely in both structures.

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research communications

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supporting information

Crystal structures of (*E*)-1-naphthaldehyde oxime and (*E*)-phenanthrene-9-carbaldehyde oxime

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Computing details

For both structures, data collection: *APEX3* (Bruker, 2016); cell refinement: *SAINT* (Bruker, 2016); data reduction: *SAINT* (Bruker, 2016); program(s) used to solve structure: SHELXT2014 (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015b); molecular graphics: *SHELXL2014* (Sheldrick, 2015b).

(*E*)-1-Naphthaldehyde oxime (I)

Crystal data

$C_{11}H_9NO$	$F(000) = 180$
$M_r = 171.19$	$D_x = 1.297$ Mg m^{-3}
Monoclinic, $P2_1$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 7.928$ (5) Å	Cell parameters from 1367 reflections
$b = 4.843$ (3) Å	$\theta = 2.6–22.6°$
$c = 11.444$ (7) Å	$\mu = 0.08$ mm^{-1}
$\beta = 94.03$ (5)°	$T = 295$ K
$V = 438.3$ (5) Å^3	Block, colourless
$Z = 2$	$0.10 \times 0.06 \times 0.02$ mm

Data collection

Bruker D8 Quest diffractometer	1570 independent reflections
φ and ω scans	957 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Bruker, 2016)	$R_{int} = 0.100$
	$\theta_{max} = 25.4°$, $\theta_{min} = 2.6°$
$T_{min} = 0.684$, $T_{max} = 0.745$	$h = -9 \rightarrow 9$
5762 measured reflections	$k = -5 \rightarrow 5$
	$l = -13 \rightarrow 13$

Refinement

Refinement on F^2	Hydrogen site location: mixed
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.055$	
$wR(F^2) = 0.098$	$w = 1/[\sigma^2(F_o^2) + (0.0164P)^2 + 0.1408P]$
$S = 1.06$	where $P = (F_o^2 + 2F_c^2)/3$
1570 reflections	$(\Delta/\sigma)_{max} < 0.001$
122 parameters	$\Delta\rho_{max} = 0.15$ e Å^{-3}
1 restraint	$\Delta\rho_{min} = -0.15$ 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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	$U_{iso}*/U_{eq}$
O1	0.8254 (4)	−0.0401 (7)	0.5056 (3)	0.0458 (9)
N1	0.8708 (5)	0.1715 (9)	0.4312 (3)	0.0418 (11)
C1	0.7559 (5)	0.4959 (10)	0.2895 (4)	0.0318 (12)
C2	0.8998 (6)	0.5156 (11)	0.2295 (4)	0.0452 (14)
H2	0.9903	0.3987	0.2494	0.054*
C3	0.9129 (7)	0.7072 (14)	0.1393 (5)	0.0569 (16)
H3	1.0128	0.7207	0.1017	0.068*
C4	0.7801 (7)	0.8737 (11)	0.1066 (4)	0.0499 (15)
H4	0.7888	0.9970	0.0450	0.060*
C4′	0.6296 (6)	0.8624 (10)	0.1645 (4)	0.0377 (12)
C5	0.4924 (6)	1.0383 (10)	0.1338 (4)	0.0468 (15)
H5	0.5009	1.1626	0.0726	0.056*
C6	0.3485 (7)	1.0323 (12)	0.1907 (5)	0.0570 (17)
H6	0.2585	1.1479	0.1677	0.068*
C7	0.3370 (6)	0.8491 (11)	0.2846 (4)	0.0478 (15)
H7	0.2393	0.8461	0.3250	0.057*
C8	0.4663 (5)	0.6760 (11)	0.3176 (4)	0.0387 (13)
H8	0.4557	0.5558	0.3800	0.046*
C8′	0.6172 (5)	0.6760 (10)	0.2582 (4)	0.0285 (11)
C11	0.7413 (6)	0.2826 (9)	0.3785 (4)	0.0350 (12)
H11	0.6343	0.2264	0.3972	0.042*
H1	0.922 (7)	−0.129 (13)	0.528 (5)	0.10 (2)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.041 (2)	0.044 (2)	0.051 (2)	0.008 (2)	−0.0047 (18)	0.008 (2)
N1	0.040 (2)	0.038 (3)	0.046 (3)	0.001 (2)	−0.005 (2)	−0.005 (2)
C1	0.033 (3)	0.029 (3)	0.033 (3)	−0.007 (3)	−0.002 (2)	−0.007 (3)
C2	0.035 (3)	0.054 (4)	0.048 (3)	−0.005 (3)	0.009 (2)	−0.014 (3)
C3	0.051 (3)	0.080 (5)	0.043 (3)	−0.018 (4)	0.021 (3)	−0.012 (4)
C4	0.064 (4)	0.051 (4)	0.036 (3)	−0.017 (3)	0.011 (3)	−0.005 (3)
C4′	0.046 (3)	0.037 (3)	0.029 (3)	−0.008 (3)	−0.003 (2)	−0.007 (3)
C5	0.060 (4)	0.037 (4)	0.041 (3)	−0.009 (3)	−0.011 (3)	0.008 (3)
C6	0.049 (3)	0.049 (4)	0.070 (4)	0.006 (3)	−0.013 (3)	0.008 (4)
C7	0.035 (3)	0.048 (4)	0.060 (4)	0.002 (3)	0.000 (3)	0.009 (3)
C8	0.036 (3)	0.039 (3)	0.040 (3)	0.002 (3)	−0.002 (2)	0.008 (3)
C8′	0.029 (2)	0.027 (3)	0.029 (3)	−0.004 (2)	−0.0016 (19)	−0.008 (3)
C11	0.032 (3)	0.029 (3)	0.044 (3)	0.002 (2)	0.003 (2)	−0.008 (3)

Geometric parameters (Å, º)

O1—N1	1.395 (5)	C4′—C5	1.407 (6)
O1—H1	0.90 (6)	C4′—C8′	1.410 (6)
N1—C11	1.273 (5)	C5—C6	1.353 (6)
C1—C2	1.375 (6)	C5—H5	0.9300
C1—C8′	1.429 (6)	C6—C7	1.402 (7)
C1—C11	1.461 (6)	C6—H6	0.9300
C2—C3	1.397 (7)	C7—C8	1.357 (6)
C2—H2	0.9300	C7—H7	0.9300
C3—C4	1.358 (7)	C8—C8′	1.417 (5)
C3—H3	0.9300	C8—H8	0.9300
C4—C4′	1.406 (6)	C11—H11	0.9300
C4—H4	0.9300		
N1—O1—H1	106 (4)	C6—C5—H5	119.0
C11—N1—O1	111.5 (4)	C4′—C5—H5	119.0
C2—C1—C8′	118.9 (4)	C5—C6—C7	119.0 (5)
C2—C1—C11	120.4 (5)	C5—C6—H6	120.5
C8′—C1—C11	120.6 (4)	C7—C6—H6	120.5
C1—C2—C3	121.5 (5)	C8—C7—C6	121.0 (5)
C1—C2—H2	119.3	C8—C7—H7	119.5
C3—C2—H2	119.3	C6—C7—H7	119.5
C4—C3—C2	120.1 (5)	C7—C8—C8′	120.9 (5)
C4—C3—H3	119.9	C7—C8—H8	119.5
C2—C3—H3	119.9	C8′—C8—H8	119.5
C3—C4—C4′	120.9 (5)	C4′—C8′—C8	118.1 (4)
C3—C4—H4	119.5	C4′—C8′—C1	119.2 (4)
C4′—C4—H4	119.5	C8—C8′—C1	122.7 (4)
C4—C4′—C5	121.7 (5)	N1—C11—C1	121.9 (4)
C4—C4′—C8′	119.3 (5)	N1—C11—H11	119.0
C5—C4′—C8′	118.9 (4)	C1—C11—H11	119.0
C6—C5—C4′	122.0 (5)		
C8′—C1—C2—C3	0.1 (7)	C5—C4′—C8′—C8	−0.3 (6)
C11—C1—C2—C3	176.2 (5)	C4—C4′—C8′—C1	−2.1 (6)
C1—C2—C3—C4	−2.0 (8)	C5—C4′—C8′—C1	179.7 (4)
C2—C3—C4—C4′	1.8 (8)	C7—C8—C8′—C4′	0.5 (7)
C3—C4—C4′—C5	178.3 (5)	C7—C8—C8′—C1	−179.6 (4)
C3—C4—C4′—C8′	0.3 (7)	C2—C1—C8′—C4′	1.9 (6)
C4—C4′—C5—C6	−178.7 (5)	C11—C1—C8′—C4′	−174.2 (4)
C8′—C4′—C5—C6	−0.6 (7)	C2—C1—C8′—C8	−178.0 (4)
C4′—C5—C6—C7	1.3 (8)	C11—C1—C8′—C8	5.9 (6)
C5—C6—C7—C8	−1.2 (8)	O1—N1—C11—C1	−175.5 (4)
C6—C7—C8—C8′	0.2 (7)	C2—C1—C11—N1	23.6 (6)
C4—C4′—C8′—C8	177.8 (4)	C8′—C1—C11—N1	−160.4 (4)

Hydrogen-bond geometry (Å, º)

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O1—H1$\cdots$N1[i]	0.90 (6)	1.94 (6)	2.834 (5)	177 (6)

Symmetry code: (i) $-x+2$, $y-1/2$, $-z+1$.

(*E*)-Phenanthrene-9-carbaldehyde oxime (II)

Crystal data

$C_{15}H_{11}NO$	$F(000) = 232$
$M_r = 221.25$	$D_x = 1.344$ Mg m^{-3}
Monoclinic, $P2_1$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 8.2397$ (8) Å	Cell parameters from 2141 reflections
$b = 4.9728$ (5) Å	$\theta = 2.6$–24.9°
$c = 13.9332$ (14) Å	$\mu = 0.09$ mm^{-1}
$\beta = 106.680$ (7)°	$T = 295$ K
$V = 546.88$ (10) Å^3	Block, colourless
$Z = 2$	$0.16 \times 0.09 \times 0.05$ mm

Data collection

Bruker D8 Quest diffractometer	1988 independent reflections
φ and ω scans	1509 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Bruker, 2016)	$R_{int} = 0.053$
	$\theta_{max} = 25.3°$, $\theta_{min} = 2.6°$
$T_{min} = 0.698$, $T_{max} = 0.745$	$h = -9 \rightarrow 9$
7330 measured reflections	$k = -5 \rightarrow 5$
	$l = -16 \rightarrow 16$

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0478P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.040$	
$wR(F^2) = 0.093$	
$S = 1.04$	$(\Delta/\sigma)_{max} < 0.001$
1988 reflections	$\Delta\rho_{max} = 0.15$ e Å^{-3}
159 parameters	$\Delta\rho_{min} = -0.15$ e Å^{-3}
1 restraint	Extinction correction: SHELXL2016 (Sheldrick, 2015b), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Hydrogen site location: mixed	Extinction coefficient: 0.058 (11)

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.

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

	x	y	z	U_{iso}*/U_{eq}
O1	0.3294 (3)	1.0407 (5)	0.49418 (15)	0.0446 (6)
N1	0.4084 (3)	0.8293 (5)	0.55737 (16)	0.0371 (6)
C11	0.3086 (4)	0.7264 (5)	0.6016 (2)	0.0340 (7)

H11	0.197024	0.785637	0.585614	0.041*
C9	0.3660 (3)	0.5157 (6)	0.67732 (18)	0.0322 (7)
C10	0.5314 (4)	0.5027 (7)	0.7316 (2)	0.0366 (7)
H10	0.607395	0.622514	0.716727	0.044*
C10′	0.5935 (3)	0.3140 (6)	0.81023 (19)	0.0349 (7)
C1	0.7655 (4)	0.3096 (7)	0.8651 (2)	0.0472 (8)
H1	0.840812	0.427685	0.848577	0.057*
C2	0.8232 (4)	0.1335 (8)	0.9426 (2)	0.0510 (9)
H2	0.937339	0.131717	0.978623	0.061*
C3	0.7116 (4)	−0.0423 (7)	0.9676 (2)	0.0481 (9)
H3	0.751583	−0.162219	1.020293	0.058*
C4	0.5438 (4)	−0.0424 (6)	0.9159 (2)	0.0445 (8)
H4	0.470941	−0.162230	0.933886	0.053*
C4′	0.4792 (4)	0.1366 (6)	0.83561 (19)	0.0333 (7)
C5′	0.3013 (4)	0.1442 (6)	0.77844 (19)	0.0334 (7)
C5	0.1811 (4)	−0.0332 (6)	0.7975 (2)	0.0431 (8)
H5	0.215809	−0.156417	0.849626	0.052*
C6	0.0153 (4)	−0.0300 (7)	0.7419 (3)	0.0499 (9)
H6	−0.061247	−0.149898	0.756119	0.060*
C7	−0.0389 (4)	0.1524 (7)	0.6641 (2)	0.0499 (9)
H7	−0.151904	0.154288	0.626056	0.060*
C8	0.0728 (4)	0.3292 (7)	0.6429 (2)	0.0424 (8)
H8	0.034520	0.451063	0.590667	0.051*
C8′	0.2453 (3)	0.3306 (6)	0.69893 (19)	0.0317 (7)
H	0.413 (4)	1.109 (7)	0.475 (2)	0.061 (12)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0474 (13)	0.0426 (14)	0.0448 (12)	0.0035 (11)	0.0145 (10)	0.0153 (11)
N1	0.0446 (14)	0.0344 (14)	0.0343 (13)	0.0040 (13)	0.0142 (11)	0.0020 (12)
C11	0.0375 (16)	0.0301 (17)	0.0356 (15)	0.0031 (14)	0.0127 (13)	−0.0001 (13)
C9	0.0408 (16)	0.0295 (16)	0.0284 (14)	0.0016 (16)	0.0131 (12)	−0.0015 (14)
C10	0.0416 (16)	0.0334 (17)	0.0352 (14)	−0.0037 (15)	0.0116 (12)	0.0008 (15)
C10′	0.0427 (16)	0.0307 (16)	0.0313 (14)	0.0026 (15)	0.0106 (12)	−0.0056 (14)
C1	0.0453 (18)	0.046 (2)	0.0475 (18)	0.0005 (19)	0.0093 (14)	0.0020 (17)
C2	0.0461 (19)	0.053 (2)	0.0475 (19)	0.0078 (18)	0.0024 (15)	−0.0014 (17)
C3	0.060 (2)	0.044 (2)	0.0371 (17)	0.0147 (18)	0.0089 (15)	0.0060 (15)
C4	0.0555 (19)	0.041 (2)	0.0390 (17)	0.0035 (17)	0.0168 (14)	0.0040 (15)
C4′	0.0464 (17)	0.0265 (15)	0.0294 (14)	0.0033 (15)	0.0149 (13)	−0.0035 (13)
C5′	0.0432 (16)	0.0297 (15)	0.0306 (14)	0.0021 (15)	0.0157 (12)	−0.0040 (14)
C5	0.0552 (19)	0.036 (2)	0.0441 (18)	−0.0002 (17)	0.0247 (15)	0.0039 (15)
C6	0.0458 (19)	0.047 (2)	0.065 (2)	−0.0071 (18)	0.0282 (16)	0.0019 (18)
C7	0.0401 (18)	0.051 (2)	0.059 (2)	−0.0031 (18)	0.0148 (15)	0.0046 (19)
C8	0.0440 (18)	0.0402 (18)	0.0413 (17)	0.0011 (17)	0.0096 (14)	0.0018 (16)
C8′	0.0386 (15)	0.0285 (15)	0.0305 (14)	0.0027 (15)	0.0139 (12)	−0.0031 (14)

Geometric parameters (Å, º)

O1—N1	1.405 (3)	C3—C4	1.363 (4)
O1—H	0.88 (3)	C3—H3	0.9300
N1—C11	1.268 (3)	C4—C4′	1.409 (4)
C11—C9	1.466 (4)	C4—H4	0.9300
C11—H11	0.9300	C4′—C5′	1.454 (4)
C9—C10	1.357 (3)	C5′—C5	1.408 (4)
C9—C8′	1.448 (4)	C5′—C8′	1.416 (4)
C10—C10′	1.422 (4)	C5—C6	1.364 (4)
C10—H10	0.9300	C5—H5	0.9300
C10′—C1	1.405 (4)	C6—C7	1.385 (4)
C10′—C4′	1.408 (4)	C6—H6	0.9300
C1—C2	1.365 (4)	C7—C8	1.365 (4)
C1—H1	0.9300	C7—H7	0.9300
C2—C3	1.384 (5)	C8—C8′	1.412 (4)
C2—H2	0.9300	C8—H8	0.9300
N1—O1—H	102 (2)	C3—C4—H4	119.5
C11—N1—O1	110.9 (2)	C4′—C4—H4	119.5
N1—C11—C9	121.2 (3)	C10′—C4′—C4	117.9 (2)
N1—C11—H11	119.4	C10′—C4′—C5′	119.2 (2)
C9—C11—H11	119.4	C4—C4′—C5′	122.9 (3)
C10—C9—C8′	119.6 (2)	C5—C5′—C8′	118.0 (2)
C10—C9—C11	120.0 (3)	C5—C5′—C4′	122.2 (3)
C8′—C9—C11	120.4 (2)	C8′—C5′—C4′	119.8 (2)
C9—C10—C10′	122.9 (3)	C6—C5—C5′	122.0 (3)
C9—C10—H10	118.6	C6—C5—H5	119.0
C10′—C10—H10	118.6	C5′—C5—H5	119.0
C1—C10′—C4′	119.8 (3)	C5—C6—C7	119.9 (3)
C1—C10′—C10	120.9 (3)	C5—C6—H6	120.1
C4′—C10′—C10	119.2 (2)	C7—C6—H6	120.1
C2—C1—C10′	120.6 (3)	C8—C7—C6	120.3 (3)
C2—C1—H1	119.7	C8—C7—H7	119.8
C10′—C1—H1	119.7	C6—C7—H7	119.8
C1—C2—C3	119.9 (3)	C7—C8—C8′	121.2 (3)
C1—C2—H2	120.1	C7—C8—H8	119.4
C3—C2—H2	120.1	C8′—C8—H8	119.4
C4—C3—C2	120.9 (3)	C8—C8′—C5′	118.6 (3)
C4—C3—H3	119.5	C8—C8′—C9	122.0 (3)
C2—C3—H3	119.5	C5′—C8′—C9	119.3 (2)
C3—C4—C4′	120.9 (3)		
O1—N1—C11—C9	−175.3 (2)	C4—C4′—C5′—C5	−1.9 (4)
N1—C11—C9—C10	27.6 (4)	C10′—C4′—C5′—C8′	−0.5 (4)
N1—C11—C9—C8′	−156.1 (2)	C4—C4′—C5′—C8′	179.7 (3)
C8′—C9—C10—C10′	−0.2 (4)	C8′—C5′—C5—C6	0.2 (4)
C11—C9—C10—C10′	176.1 (3)	C4′—C5′—C5—C6	−178.2 (3)

C9—C10—C10′—C1	−179.3 (3)	C5′—C5—C6—C7	−0.1 (5)
C9—C10—C10′—C4′	−1.9 (4)	C5—C6—C7—C8	−0.1 (5)
C4′—C10′—C1—C2	0.4 (5)	C6—C7—C8—C8′	0.3 (5)
C10—C10′—C1—C2	177.9 (3)	C7—C8—C8′—C5′	−0.2 (4)
C10′—C1—C2—C3	0.0 (5)	C7—C8—C8′—C9	179.7 (3)
C1—C2—C3—C4	−0.2 (5)	C5—C5′—C8′—C8	−0.1 (4)
C2—C3—C4—C4′	0.0 (5)	C4′—C5′—C8′—C8	178.4 (3)
C1—C10′—C4′—C4	−0.5 (4)	C5—C5′—C8′—C9	−179.9 (2)
C10—C10′—C4′—C4	−178.1 (3)	C4′—C5′—C8′—C9	−1.5 (4)
C1—C10′—C4′—C5′	179.7 (3)	C10—C9—C8′—C8	−178.0 (3)
C10—C10′—C4′—C5′	2.2 (4)	C11—C9—C8′—C8	5.7 (4)
C3—C4—C4′—C10′	0.3 (4)	C10—C9—C8′—C5′	1.9 (4)
C3—C4—C4′—C5′	−179.9 (3)	C11—C9—C8′—C5′	−174.4 (2)
C10′—C4′—C5′—C5	177.8 (3)		

Hydrogen-bond geometry (Å, º)

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
O1—H⋯N1[i]	0.88 (3)	1.99 (3)	2.852 (3)	169 (3)

Symmetry code: (i) −x+1, y+1/2, −z+1.