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Crystal structure of benzo[*h*]quinoline-3-carboxamide

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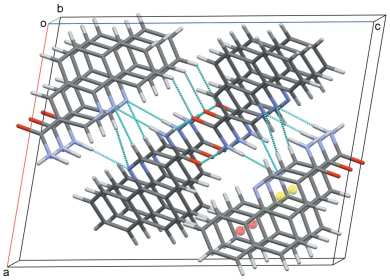
The title compound, $C_{14}H_{10}N_2O$, crystallizes in the monoclinic space group $P2_1/c$ with four molecules in the unit cell. All 17 non-H atoms of one molecule lie essentially in one plane. In the unit cell, two pairs of molecules are exactly coplanar, while the angle between these two orientations is close to perfectly perpendicular at 87.64 (6)°. In the crystal, molecules adopt a 50:50 crisscross arrangement, which is held together by two nonclassical and two classical intermolecular hydrogen bonds. The hydrogen-bonding network together with off-centre π–π stacking interactions between the pyridine and outermost benzene rings, stack the molecules along the *b*-axis direction.



1. Chemical context

Quinoline and benzoquinoline scaffolds are common structural motifs in artificial, as well as natural products, and many of these compounds are of enormous value for pharmacotherapy. Their multifaceted biological efficacy is outstanding and ranges from cardiovascular (Ferlin *et al.*, 2002; Abouzid *et al.*, 2008) and anti-inflammatory effects (Kumar *et al.*, 2009; Hussaini, 2016) to antimicrobial (El Shehry *et al.*, 2018), as well as anticancer activity (Abdelsalam *et al.*, 2019; Haiba *et al.*, 2019; Jafari *et al.*, 2019; Musiol, 2017; Marzaro *et al.*, 2016). In a report on 3-(tetrazol-5-yl)quinolines with antiallergic potential, benzo[*h*]quinoline-3-carboxamide was mentioned as a synthetic intermediate, though its biological activity was not determined in that work (Erickson *et al.*, 1979). In our recent studies on photoswitchable sirtuin inhibitors, we obtained benzo[*h*]quinoline-3-carboxamide as a side product of azastilbene photoisomerization (Grathwol *et al.*, 2019). By UV radiation, (*E*)-5-styrylnicotinamide was transformed to its *Z* isomer as envisioned, but underwent photocyclization and successive oxidation, yielding two isomeric benzoquinoline derivatives; the identity of one of these was determined to be the benzo[*h*]quinoline derivative and its crystal structure is reported here.

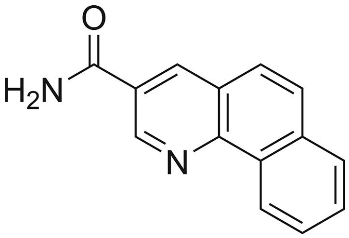


Table 1
Hydrogen-bond geometry (Å, °).

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N1—H1N⋯N2^{i}	0.97 (3)	2.17 (3)	3.133 (3)	173 (2)
N1—H1P⋯O1^{ii}	0.93 (3)	1.96 (3)	2.895 (3)	175 (3)
C3—H3⋯N2^{i}	0.95	2.41	3.361 (3)	174
C7—H7⋯O1^{iii}	0.95	2.45	3.140 (3)	129

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

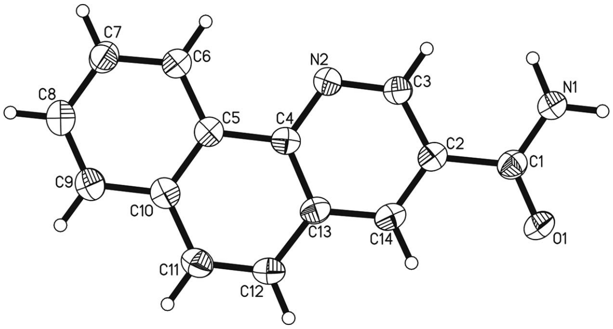


Figure 1
The molecular structure of benzo[*h*]quinoline-3-carboxamide. Displacement ellipsoids are shown at the 50% probability level.

2. Structural commentary

The title compound, benzo[*h*]quinoline-3-carboxamide, crystallizes in the monoclinic space group $P2_1/c$. Four molecules are present in the unit cell ($Z = 4$) and there is one molecule in the asymmetric unit. Benzo[*h*]quinoline-3-carboxamide consists of a nicotinamide unit being fused with a benzo[*h*]quinoline moiety, while the pyridine ring is shared between these two common structural building blocks (Fig. 1). The molecule is essentially flat, with a largest deviation from the plane through all 17 non-H atoms of 0.050 (2) Å (O1) and an r.m.s. deviation of 0.020 (2) Å. In the unit cell, the four molecules are arranged in two perfectly coplanar pairs, with a nearly perpendicular angle between the respective planes of the two pairs of 87.64 (6)° (Fig. 2). A plethora of crystal structures are known for compounds with one or other of the two building blocks that make up this molecule [for the nicotinamide scaffold, *ConQuest* finds over 2000 hits in the Cambridge Structural Database (CSD), while for benzoquinoline, there are over 500; Groom *et al.*, 2016]. However, the specific combination in the title compound is unprecedented. Comparing the title compound to the known structures of unsubstituted nicotinamides, its pronounced planarity is most notable. In the six published structures in the space groups $P2_1/c$ or $P2_1/a$, the angles between the aromatic plane (here C2/C3/N2/C4/C13/C14) and the amide substituent (here O1/N1/C1) range from 22.1 to 23.3° (general CSD refcode NICOAM; Wright & King, 1954; Miwa *et al.*, 1999; Fábián *et al.*, 2011; Jarzembska *et al.*, 2014), *i.e.* this angle is quite consistent. In the only distinct polymorph of a nicotinamide in the space group $P2/a$, four distinct molecules were refined with this angle ranging from 8.1 to 22.4° (Li *et al.*, 2011), *i.e.* they are not very consistent but still considerably larger than the corresponding angle found in the title compound, which is a mere 3.3 (4)°. This points toward an extension of the aromatic resonance systems to include the amide substituent. In the parent nicotinamide scaffolds, this does not occur. Similarly, the comparatively long C1═O1 distance of 1.238 (3) Å (average 1.23 Å) and the comparatively short C1—C2 distance of 1.491 (3) Å (average 1.50 Å in other nicotinamide structures) indicate some involvement of these atoms in resonance effects. In support of this extended resonance, in the nicotinamide structures, the aromatic C—C bonds are much less diverse (range 1.38–1.39 Å, indicating very strong aromaticity in the pyridine ring) than in the structure reported here. In fact, the C—C [range 1.376 (3)–1.414 (3) Å] and C—N [1.321 (3) and 1.360 (3) Å] bond lengths here are much

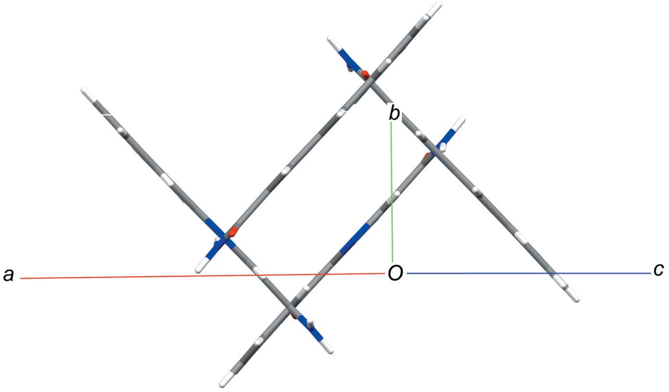


Figure 2
The unit cell of benzo[*h*]quinoline-3-carboxamide in $P2_1/c$, with its four molecules in a coplanar and perpendicular arrangement, viewed along the *ac* diagonal.

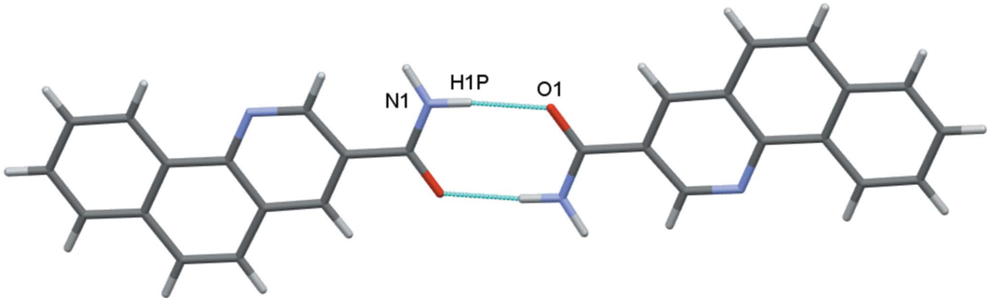


Figure 3
Dimers formed by N—H⋯O hydrogen bonds.

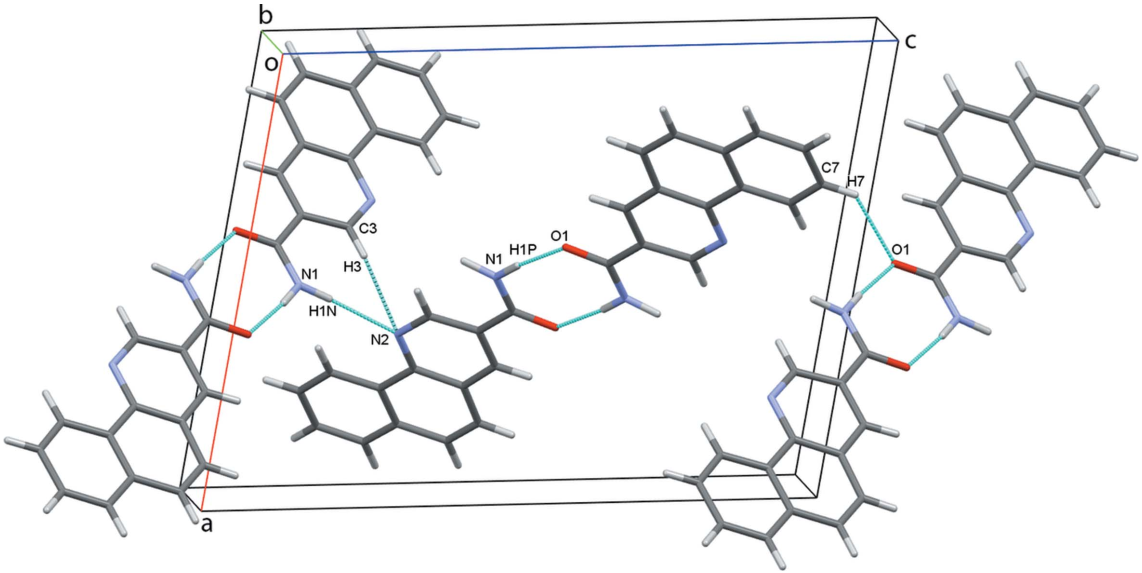


Figure 4
Chains of dimers along the *c*-axis.

more similar to the two known structures of 2-unsubstituted and 3-substituted benzo[*h*]quinolines (refcodes JAFVEU and SUDVES), with ranges of average C—C and C—N bond lengths of 1.38–1.42 and 1.32–1.36 Å, respectively (Martínez *et al.*, 1992; Luo *et al.*, 2015). The benzo[*h*]quinoline structural motif therefore dominates the observed metrical parameters of the molecule reported here, representing a fusion between a nicotinamide and a benzo[*h*]quinoline, with a partial extension of the aromaticity beyond the ring system and extending towards the amide substituent.

3. Supramolecular features

In the crystal, the planar molecules are all arranged in planes in two distinct orientations, which are nearly perpendicular to each other [angle 87.64 (6)°]. This forms a crisscross pattern when viewed along the *ac* diagonal (Fig. 2). Classical inversion-related N1—H1*P*···O1 hydrogen bonds form dimers and generate $R_2^2(8)$ ring motifs (Fig. 3). Each molecule forms two classical (N—H···O and N—H···N) and two nonclassical (C—H···N and C—H···O) hydrogen bonds (Table 1), and these contacts link adjacent dimers into zigzag chains along the *c*-axis direction (Fig. 4). The observed packing is further stabilized by off-centre *π*–*π* stacking between the pyridine and outermost benzene rings of each of the coplanar layers [centroid-to-centroid distance = 3.610 (1) Å] (Fig. 5). These contacts combine to stack the molecules along the *b*-axis direction (Fig. 6).

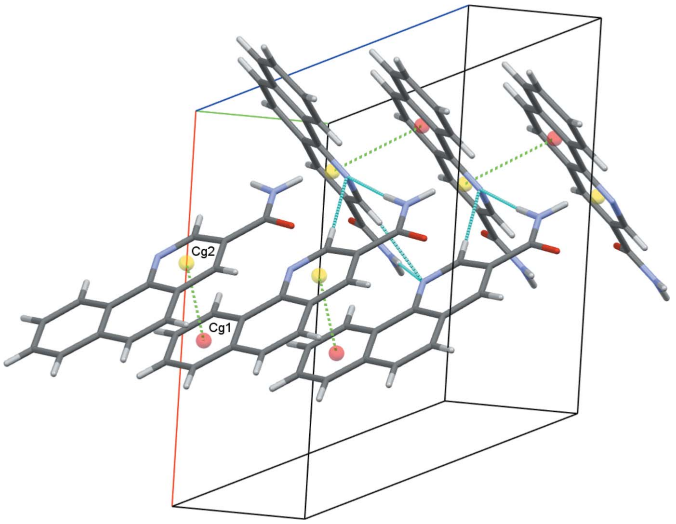


Figure 5
π–*π* stacking interactions, with centroids shown as coloured spheres. *Cg*1 and *Cg*2 are the centroids of the C5–C10 and C2/C3/N2/C4/C13/C14 rings, respectively.

4. Synthesis and crystallization

A solution of (*E*)-5-styrylnicotinamide (673 mg, 3.00 mmol, 1.00 equiv.) in methanol (350 ml) was treated with a solution

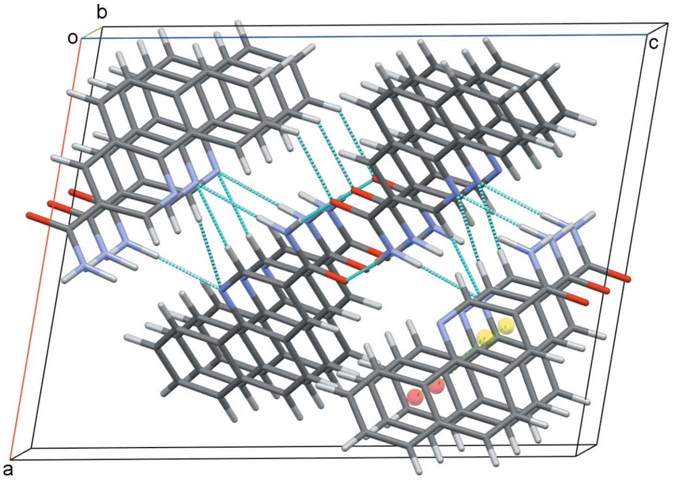


Figure 6
The overall packing of the title compound, viewed along the *b*-axis direction.

of iodine (38 mg, 0.15 mmol, 0.05 equiv.) in methanol (50 ml). A slow stream of compressed air was bubbled through the reaction mixture while it was irradiated with UV light (six Vilber-Lourmat T8-C lamps, 8 W, 254 nm). After complete consumption of the starting material (24 h), the solvent was removed under reduced pressure. Purification of the residue by silica-gel column chromatography (*n*-hexane/THF, 1:1 *v*/*v*) gave pure benzo[*h*]quinoline-3-carboxamide as a colourless solid (yield 80 mg, 0.36 mmol, 12%). Crystallization was accomplished by slow evaporation of a solution in THF (5 mg ml^{-1}) and yielded the title compound as colourless needles: R_F = 0.32 (*n*-hexane/THF, 1:1 *v*/*v*); m.p. 549.8 K (decomposition); ^{1}H NMR, H,H-COSY (400 MHz, DMSO-d_6): δ (ppm) 9.48 (*d*, J = 2.2 Hz, 1H, C3-H), 9.26–9.19 (*m*, 1H, C6-H), 8.89 (*d*, J = 2.1 Hz, 1H, C14-H), 8.36 (*s*, *br*, 1H, N1-H), 8.12–8.07 (*m*, 1H, C9-H), 8.03 (*d*, J = 8.9 Hz, 1H, C11-H), 7.95 (*d*, J = 8.9 Hz, 1H, C12-H), 7.85–7.78 (*m*, 2H, C7-H, C8-H), 7.74 (*s*, *br*, 1H, N1-H); ^{13}C NMR, DEPT135, HSQC, HMBC (101 MHz, DMSO-d_6): δ (ppm) 166.4 (C1), 147.8 (C3), 146.7 (C4), 135.5 (C14), 133.8 (C13), 130.3 (C5), 129.0 (C8), 128.1 (C9/C11), 128.0 (C9/C11), 127.8 (C2), 127.3 (C7), 125.8 (C12), 124.9 (C10), 124.1 (C6); IR (ATR): ν (cm^{-1}) 3336, 3136, 1686, 1482, 1395, 1295, 801, 691, 539, 489; ESI–HRMS calculated for $[C_{14}H_{10}N_2O + H]^+$ 222.0793, found 222.0796; compound purity (220 nm): 100%.

5. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All C-bound H atoms constitute aromatic protons, which were attached in calculated positions and treated as riding with $U_{iso}(H) = 1.2U_{eq}(C)$. The two amine H atoms were found and refined without any constraints or restraints.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{14}H_{10}N_2O$
M_r	222.24
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	170
a, b, c (Å)	12.634 (3), 4.9426 (10), 16.778 (3)
β (°)	100.53 (3)
V (Å^3)	1030.0 (4)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.09
Crystal size (mm)	0.37 × 0.07 × 0.04
Data collection	
Diffractometer	Stoe IPDS-2T
Absorption correction	Numerical face indexed
T_{min}, T_{max}	0.727, 0.997
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	10053, 2551, 1320
R_{int}	0.087
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.667
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.055, 0.167, 0.98
No. of reflections	2551
No. of parameters	163
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.23, −0.27

Computer programs: *X-AREA* (Stoe & Cie, 2010), *SHELXT2018* (Sheldrick, 2015*a*), *SHELXL2018* (Sheldrick, 2015*b*), *XP* in *SHELXTL* (Sheldrick, 2008), *Mercury* (Macrae *et al.*, 2008), *CIFTAB* (Sheldrick, 2015*b*) and *publCIF* (Westrip, 2010).

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Crystal structure of benzo[*h*]quinoline-3-carboxamide

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Computing details

Data collection: *X-AREA* (Stoe & Cie, 2010).; cell refinement: *X-AREA* (Stoe & Cie, 2010).; data reduction: *X-AREA* (Stoe & Cie, 2010).; program(s) used to solve structure: SHELXT2018 (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2018* (Sheldrick, 2015b); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 2008) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: CIFTAB (Sheldrick, 2015b) and *publCIF* (Westrip, 2010).

Benzo[*h*]quinoline-3-carboxamide

Crystal data

$C_{14}H_{10}N_2O$
$M_r = 222.24$
Monoclinic, $P2_1/c$
$a = 12.634$ (3) Å
$b = 4.9426$ (10) Å
$c = 16.778$ (3) Å
$\beta = 100.53$ (3)°
$V = 1030.0$ (4) Å^3
$Z = 4$
$F(000) = 464$
$D_x = 1.433$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 11960 reflections
$\theta = 6.4$–$59.0°$
$\mu = 0.09$ mm^{-1}
$T = 170$ K
Needle, colourless
0.37 × 0.07 × 0.04 mm

Data collection

Stoe IPDS2T diffractometer
Radiation source: fine-focus sealed tube
Detector resolution: 6.67 pixels mm^{-1}
ω scans
Absorption correction: numerical face indexed
$T_{min} = 0.727$, $T_{max} = 0.997$
10053 measured reflections
2551 independent reflections
1320 reflections with $I > 2\sigma(I)$
$R_{int} = 0.087$
$\theta_{max} = 28.3°$, $\theta_{min} = 3.2°$
$h = -16 \rightarrow 16$
$k = -6 \rightarrow 6$
$l = -22 \rightarrow 22$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.055$
$wR(F^2) = 0.167$
$S = 0.98$
2551 reflections
163 parameters
0 restraints
Primary atom site location: dual
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.086P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}

Extinction correction: SHELXL2018 (Sheldrick, 2015b), $Fc^{*}=kFc[1+0.001xFc^{2}\lambda^{3}/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.019 (4)

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*	$U_{\rm iso}$*/$U_{\rm eq}$
O1	0.60103 (14)	−0.2438 (3)	0.51392 (9)	0.0364 (4)
N2	0.63400 (15)	0.2758 (4)	0.28016 (11)	0.0304 (5)
N1	0.48278 (17)	−0.3250 (4)	0.39872 (12)	0.0331 (5)
C1	0.56558 (19)	−0.1907 (4)	0.44191 (13)	0.0312 (5)
C2	0.61839 (18)	0.0252 (4)	0.40098 (12)	0.0287 (5)
C3	0.58725 (19)	0.0909 (4)	0.31880 (13)	0.0311 (5)
H3	0.528010	−0.004871	0.288573	0.037*
C4	0.71867 (17)	0.4149 (4)	0.32278 (12)	0.0274 (5)
C5	0.77008 (18)	0.6182 (4)	0.28123 (13)	0.0292 (5)
C6	0.7362 (2)	0.6735 (5)	0.19826 (13)	0.0332 (5)
H6	0.678537	0.573337	0.167687	0.040*
C7	0.7858 (2)	0.8702 (5)	0.16151 (14)	0.0360 (6)
H7	0.762331	0.905703	0.105414	0.043*
C8	0.8707 (2)	1.0200 (5)	0.20521 (15)	0.0375 (6)
H8	0.904089	1.157365	0.178843	0.045*
C9	0.90571 (19)	0.9702 (5)	0.28530 (14)	0.0346 (6)
H9	0.963477	1.073076	0.314661	0.041*
C10	0.85689 (18)	0.7665 (4)	0.32529 (13)	0.0305 (5)
C11	0.8935 (2)	0.7074 (5)	0.40961 (14)	0.0350 (6)
H11	0.952662	0.805664	0.438992	0.042*
C12	0.84594 (19)	0.5161 (5)	0.44794 (13)	0.0329 (5)
H12	0.872211	0.481242	0.503736	0.039*
C13	0.75657 (17)	0.3646 (4)	0.40605 (12)	0.0291 (5)
C14	0.70365 (19)	0.1668 (4)	0.44428 (13)	0.0307 (5)
H14	0.726853	0.130640	0.500358	0.037*
H1N	0.449 (2)	−0.279 (6)	0.344 (2)	0.059 (9)*
H1P	0.452 (2)	−0.464 (6)	0.4246 (16)	0.054 (8)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0445 (10)	0.0389 (9)	0.0247 (8)	−0.0018 (8)	0.0036 (7)	0.0056 (7)
N2	0.0311 (11)	0.0302 (10)	0.0293 (9)	−0.0018 (8)	0.0037 (8)	0.0005 (8)
N1	0.0373 (12)	0.0331 (11)	0.0280 (10)	−0.0018 (9)	0.0036 (9)	0.0020 (8)
C1	0.0351 (13)	0.0302 (11)	0.0285 (11)	0.0031 (10)	0.0064 (10)	0.0001 (9)

C2	0.0310 (12)	0.0281 (11)	0.0275 (10)	0.0048 (9)	0.0068 (9)	0.0004 (9)
C3	0.0333 (12)	0.0306 (11)	0.0286 (11)	−0.0012 (10)	0.0033 (9)	0.0002 (9)
C4	0.0278 (11)	0.0261 (11)	0.0279 (11)	0.0029 (9)	0.0036 (9)	−0.0021 (9)
C5	0.0299 (12)	0.0276 (11)	0.0307 (11)	0.0034 (10)	0.0068 (9)	−0.0007 (9)
C6	0.0345 (13)	0.0355 (13)	0.0295 (11)	−0.0024 (10)	0.0058 (10)	0.0009 (9)
C7	0.0372 (14)	0.0387 (13)	0.0327 (11)	−0.0005 (11)	0.0082 (10)	0.0042 (10)
C8	0.0367 (13)	0.0348 (13)	0.0428 (13)	−0.0015 (11)	0.0124 (11)	0.0025 (11)
C9	0.0325 (13)	0.0324 (12)	0.0393 (13)	0.0003 (10)	0.0079 (10)	−0.0006 (10)
C10	0.0291 (12)	0.0280 (11)	0.0351 (11)	0.0030 (9)	0.0079 (9)	−0.0021 (9)
C11	0.0319 (13)	0.0379 (13)	0.0342 (12)	−0.0024 (10)	0.0037 (10)	−0.0063 (10)
C12	0.0324 (12)	0.0372 (12)	0.0274 (11)	0.0009 (10)	0.0009 (9)	−0.0027 (9)
C13	0.0303 (12)	0.0302 (11)	0.0260 (10)	0.0045 (10)	0.0033 (9)	0.0006 (9)
C14	0.0352 (13)	0.0312 (12)	0.0248 (10)	0.0053 (10)	0.0034 (9)	0.0009 (9)

Geometric parameters (Å, º)

O1—C1	1.238 (3)	C6—H6	0.9500
N2—C3	1.321 (3)	C7—C8	1.396 (3)
N2—C4	1.360 (3)	C7—H7	0.9500
N1—C1	1.335 (3)	C8—C9	1.358 (3)
N1—H1N	0.97 (3)	C8—H8	0.9500
N1—H1P	0.93 (3)	C9—C10	1.413 (3)
C1—C2	1.491 (3)	C9—H9	0.9500
C2—C14	1.376 (3)	C10—C11	1.436 (3)
C2—C3	1.401 (3)	C11—C12	1.346 (3)
C3—H3	0.9500	C11—H11	0.9500
C4—C13	1.414 (3)	C12—C13	1.428 (3)
C4—C5	1.443 (3)	C12—H12	0.9500
C5—C6	1.406 (3)	C13—C14	1.404 (3)
C5—C10	1.411 (3)	C14—H14	0.9500
C6—C7	1.364 (3)		

C3—N2—C4	118.11 (19)	C6—C7—H7	119.6
C1—N1—H1N	124.6 (17)	C8—C7—H7	119.6
C1—N1—H1P	117.5 (17)	C9—C8—C7	120.2 (2)
H1N—N1—H1P	118 (2)	C9—C8—H8	119.9
O1—C1—N1	122.2 (2)	C7—C8—H8	119.9
O1—C1—C2	119.3 (2)	C8—C9—C10	120.5 (2)
N1—C1—C2	118.55 (19)	C8—C9—H9	119.7
C14—C2—C3	117.0 (2)	C10—C9—H9	119.7
C14—C2—C1	119.62 (19)	C5—C10—C9	119.1 (2)
C3—C2—C1	123.4 (2)	C5—C10—C11	119.4 (2)
N2—C3—C2	125.0 (2)	C9—C10—C11	121.5 (2)
N2—C3—H3	117.5	C12—C11—C10	121.5 (2)
C2—C3—H3	117.5	C12—C11—H11	119.3
N2—C4—C13	121.5 (2)	C10—C11—H11	119.3
N2—C4—C5	118.57 (19)	C11—C12—C13	121.0 (2)
C13—C4—C5	119.92 (19)	C11—C12—H12	119.5

C6—C5—C10	119.0 (2)	C13—C12—H12	119.5
C6—C5—C4	122.1 (2)	C14—C13—C4	118.1 (2)
C10—C5—C4	118.95 (19)	C14—C13—C12	122.68 (19)
C7—C6—C5	120.3 (2)	C4—C13—C12	119.3 (2)
C7—C6—H6	119.9	C2—C14—C13	120.37 (19)
C5—C6—H6	119.9	C2—C14—H14	119.8
C6—C7—C8	120.9 (2)	C13—C14—H14	119.8

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···N2^{i}	0.97 (3)	2.17 (3)	3.133 (3)	173 (2)
N1—H1*P*···O1^{ii}	0.93 (3)	1.96 (3)	2.895 (3)	175 (3)
C3—H3···N2^{i}	0.95	2.41	3.361 (3)	174
C7—H7···O1^{iii}	0.95	2.45	3.140 (3)	129

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