Acta Cryst E CRYSTALLOGRAPHIC COMMUNICATIONS

ISSN 2056-9890

Syntheses and structures of two norpsilocin derivatives: *N*-ethyl-4-hydroxytryptamine (4-HO-NET) and 4-hydroxy-*N*-propyltryptamine (4-HO-NPT)

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Received 16 August 2026
Accepted 18 August 2026

Edited by W. T. A. Harrison, University of Aberdeen, United Kingdom

Keywords: crystal structure; tryptamines; indoles; hydrogen bonds.

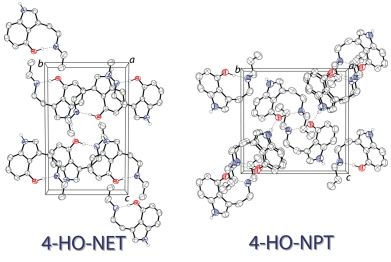
CCDC references: 2581888; 2581887

Supporting information: this article has supporting information at journals.iucr.org/e

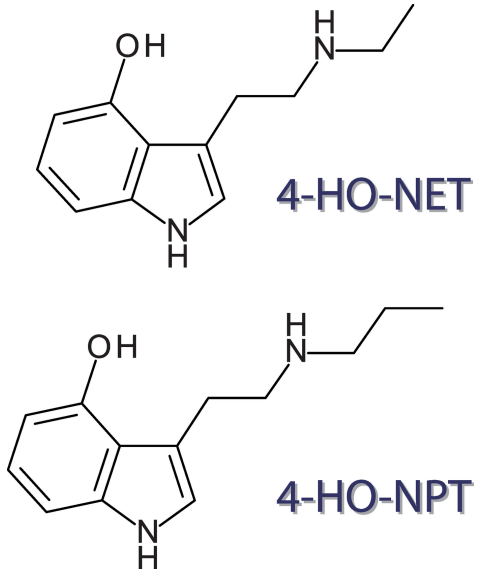
The solid-state structures of *N*-ethyl-4-hydroxytryptamine (4-HO-NET) {systematic name: 3-[2-(ethylamino)ethyl]-1*H*-indol-4-ol}, $C_{12}H_{16}N_2O$, and 4-hydroxy-*N*-propyltryptamine (4-HO-NPT) {systematic name: 3-[2-(propylamino)ethyl]-1*H*-indol-4-ol}, $C_{13}H_{18}N_2O$, are reported. Both compounds possess a single tryptamine molecule in the asymmetric unit that exhibits an internal O—H···N hydrogen bond. In the extended structures, the molecules are linked by N—H···O hydrogen bonds to form infinite chains along [001] for 4-HO-NET and along [101] for 4-HO-NPT.

1. Chemical context

Traditional psychoactive compounds including psilocybin (4-phosphoryloxy-*N*,*N*-dimethyltryptamine; $C_{12}H_{17}N_2O_4P$), psilocin (4-hydroxy-*N*,*N*-dimethyltryptamine; $C_{12}H_{16}N_2O$), DMT (*N*,*N*-dimethyltryptamine; $C_{12}H_{16}N_2$), and 5-MeO-DMT (5-methoxy-*N*,*N*-dimethyltryptamine; $C_{13}H_{18}N_2O$) are all based upon an *N*,*N*-dialkyltryptamine backbone. Lesser known naturally occurring compounds such as baeocystin (4-phosphoryloxy-*N*-methyltryptamine; $C_{11}H_{15}N_2O_4P$) and norpsilocin (4-hydroxy-*N*-methyltryptamine; $C_{11}H_{14}N_2O$) have a suggested biological relevance but an underdeveloped chemistry. We have previously reported the crystal structures of both of these *N*-monoalkyltryptamine compounds (Naeem *et al.*, 2022; Chadeayne *et al.*, 2020*b*), as well as explored their pharmacological properties (Glatfelter, Pottie *et al.*, 2022). The removal of a single methyl group from the traditional *N*,*N*-dimethyltryptamines can significantly alter lipophilicity, central nervous system exposure, metabolism, receptor activity and transporter pharmacology. The *N*-monoalkyltryptamines are a group of serotonergic chemicals that could present pharmacology not accessible through the class of *N*,*N*-dialkyltryptamines. In early 2023, we reported the synthesis and structure of 4-hydroxy-*N*-isopropyltryptamine (4-HO-NiPT) as the first direct *N*-alkyl analogue of norpsilocin (Laban *et al.*, 2023). Sherwood and co-workers later reported a number of other *N*-alkyl analogues, exploring their pharmacology. Of note, derivatives of norpsilocin where the steric bulk of the alkyl group was increased (*i.e.* 4-HO-NiPT) showed psychoactive effects in animal models (Sherwood *et al.*, 2024). We recently reported the 4-methoxy variant of norpsilocin, as well as other 4-methoxy mono-*N*-alkyl derivatives, which were shown to be potent 5-HT_{2A} agonists with reduced psychedelic-like effects in animal models (Glatfelter, Schalk *et al.*, 2026). Herein, we report the syntheses and



structures of two norpsilocin analogues, *N*-ethyl-4-hydroxytryptamine (4-HO-NET; $C_{12}H_{16}N_2O$) and 4-hydroxy-*N*-propyltryptamine (4-HO-NPT; $C_{13}H_{18}N_2O$).



2. Structural commentary

The molecular structures of 4-HO-NET and 4-HO-NPT are shown in Fig. 1. The asymmetric unit of each structure contains a single tryptamine molecule. The side chain in 4-HO-NET displays an extended conformation, as shown by the C9—C10—N2—C11 and C10—N2—C11—C12 torsion angles of −174.88 (12) and −174.34 (14)°, respectively. The longer side chain in 4-HO-NPT shows a more twisted conformation with C9—C10—N2—C11 = −101.41 (15), C10—N1—C11—C12 = 174.61 (13) and N2—C11—C12—C13 = 169.17 (13)°.

Both molecules possess an internal O—H···N hydrogen bond between the phenol oxygen atom (O1) and the acyclic amine nitrogen atom (N2), which generates an *S*(8) ring in each case. As expected, both C1–C8/N1 indole ring systems are near planar, showing a r.m.s. deviation of 0.009Å for 4-HO-NET and 0.011Å for 4-HO-NPT.

Table 1
Hydrogen-bond geometry (Å, °) for 4-HO-NET.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O1^{i}	0.87 (1)	2.06 (1)	2.8789 (15)	158 (2)
O1—H1···N2	1.00 (1)	1.58 (1)	2.5764 (16)	173 (2)

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

Table 2
Hydrogen-bond geometry (Å, °) for 4-HO-NPT.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N2	1.01 (1)	1.59 (1)	2.5905 (15)	175 (2)
N1—H1*A*···O1^{i}	0.87 (1)	2.06 (1)	2.9347 (16)	177 (2)
N2—H2···O1^{ii}	0.90 (1)	2.62 (1)	3.4957 (17)	166 (2)

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

3. Supramolecular features

In the solid-state structure of 4-HO-NET, the molecules are linked by N1—H1*A*···O1 hydrogen bonds between the indole N—H grouping and the hydroxide group. These hydrogen bonds link the molecules into infinite chains propagating along the [001] direction (Table 1). The crystal packing of 4-HO-NET is shown on the left in Fig. 2.

In the extended structure of 4-HO-NPT, two molecules are connected through N2—H2···O1 hydrogen bonds. These intermolecular hydrogen bonds combine with the O1—H1···N2 intramolecular hydrogen bonds to form rings demonstrating a graph set notation of $R_2^2(16)$ (Etter *et al.*, 1990). These dimers are then joined together through the indole N1—H1A···O1 hydrogen bond to the next dimer, forming infinite chains propagating along the [101] direction (Table 2). The crystal packing of 4-HO-NPT is shown on the right in Fig. 2.

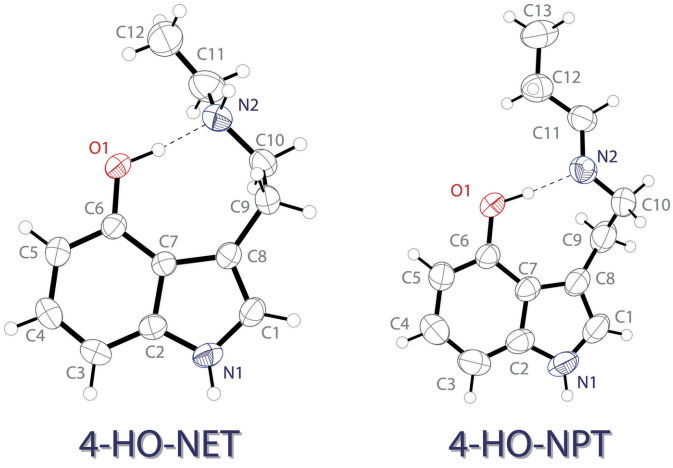


Figure 1
The molecular structures of 4-HO-NET (left) and 4-HO-NPT (right) with displacement ellipsoids drawn at the 50% probability level. Hydrogen bonds are shown as dashed lines.

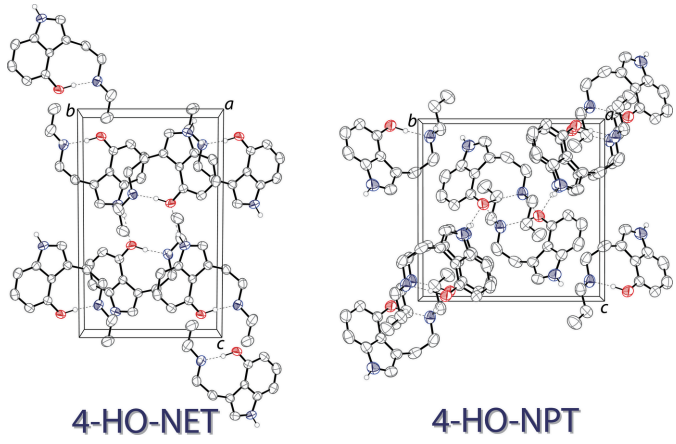


Figure 2
The crystal packing of 4-HO-NET (left) and 4-HO-NPT (right), both shown along the *a*-axis direction. Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding are omitted for clarity.

4. Database survey

The structures of monoalkyltryptamines are not common in the Cambridge Structural Database, with only ten crystal structures previously reported, all in the past few years. These include the natural product baeocystin (Naeem *et al.*, 2022; FETBAB), its hydrolysis product norpsilocin as its freebase and fumarate salt (Chadeayne *et al.*, 2020*b*; MULXAV, MULXEZ) and the synthetic prodrug of norpsilocin, 4-acetoxy-*N*-methyltryptamine (Glatfelter, Pottie *et al.*, 2022). The others are *N*-methylserotonin as its hydrogen oxalate salt (Naeem, Anas *et al.*, 2023), 4-benzyl-*N*-isopropyltryptamine, 4-hydroxy-*N*-isopropyltryptamine (Laban *et al.*, 20233; YEYZIP, YEYZOV), and the freebase, bromide and fumarate salts of *N*-cyclohexyltryptamine (Naeem, Le *et al.*, 2023; YITWAD, YITWEH, YITWIL).

There are eight 4-hydroxy-*N*,*N*-dialkyltryptamine crystal structures reported in the literature, which are 4-hydroxy-*N*-methyl-*N*-isopropyltryptamine as its fumarate (Chadeayne *et al.*, 2020*a*; TUFQAP) and hydrofumarate (Chadeayne *et al.*, 2019*a*; RONSUL), 4-hydroxy-*N*,*N*-dipropyltryptamine as its fumarate (Chadeayne *et al.*, 2019*b*; WUCGAF) and chloride (Sammeta *et al.*, 2020; WAMGEA) salts, 4-hydroxy-*N*,*N*-diisopropyltryptamine as its hydrofumarate salt (Naeem *et al.*, 2025; BOWSOF), and the natural product psilocin as its freebase (Petcher & Weber, 1974; PSILIN; Zeller *et al.*, 2024; PSILIN03), its tatrate salt (Barrow *et al.*, 2025; XOWXIU), and as a co-crystal with the natural product psilocybin (Silverstone, 2024; MIMKOM). There are also four 4-hydroxy-*N*,*N*,*N*-trialkyltryptamine crystal structures reported including 4-hydroxy-*N*,*N*,*N*-trimethyltryptamine (Chadeayne *et al.*, 2020*c*; XUXFAA), 4-hydroxy-*N*,*N*-dimethyl-*N*-ethyltryptamine, 4-hydroxy-*N*,*N*-dimethyl-*N*-propyltryptamine, and 4-hydroxy-*N*,*N*-dimethyl-*N*-isopropyltryptamine (Glatfelter, Pham *et al.*, 2022; EDOYIJ, EDOYUV, EDOZEG).

5. Synthesis and crystallization

4-HO-NET: To an ice-bath-cooled diethyl ether (60 ml) solution of 4-benzyloxy-1*H*-indole (3.0 g) was added oxalylchloride (3.4 g) dropwise. The mixture was stirred in the ice bath for 6 h and then added dropwise to an ice-bath-cooled solution of 70% aqueous ethylamine (5.8 g). The mixture was warmed to room temperature and stirred overnight. The resulting suspension was concentrated under reduced pressure and the residue was purified by silica gel chromatography (methylene chloride/methanol) to afford 2-(4-benzyloxy)-1*H*-indol-3-yl)-*N*-ethyl-2-oxoacetamide as a yellow oil (3.2 g). The yellow oil was dissolved in tetrahydrofuran (50 ml), cooled in an ice bath, and 30 ml of 1 *M* borane-tetrahydrofuran was added dropwise. The mixture was heated at reflux overnight. The resulting yellow solution was quenched with 2 *M* hydrochloric acid and heated at reflux for 2 h. The mixture was cooled and ammonium hydroxide was added until the solution was basic. The resulting mixture was extracted with methylene chloride; the organic layer was washed with water and brine, and then dried over sodium sulfate. Solvent was removed *in vacuo* and the resulting residue was purified by silica gel chromatography [methylene chloride/methanol (3.5 *M* ammonia)] to afford 4-benzyloxy-*N*-ethyltryptamine (4-BnO-NET) as a yellow solid (1.6 g, 54% yield). To a solution of 4-BnO-NET (600 mg) in methanol (12 ml) was added Pd/C (120 mg) and $Pd(OH)_2$/C (120 mg). The mixture was stirred for 3 h under a hydrogen atmosphere. The resulting black suspension was filtered, the solid was washed with methanol, and the combined filtrates were concentrated under reduced pressure. The resulting residue was purified by silica gel chromatography [methylene chloride/methanol (3.5 *M* ammonia)] to afford 4-hydroxy-*N*-ethyltryptamine (4-HO-NET) as a white solid (162 mg, 39% yield). Single crystals suitable for X-ray diffraction were grown from the slow evaporation of an acetone solution.

4-HO-NPT: To an ice bath-cooled diethyl ether (40 ml) solution of 4-benzyloxy-1*H*-indole (2.0 g) was added oxalylchloride (2.3 g) dropwise. The mixture was stirred for 6 h under cooling, and then added dropwise into an ice-bath cooled vessel containing propan-1-amine (5.3 g). The mixture was warmed to room temperature and stirred overnight. The solvent was removed *in vacuo* and the resulting residue was purified by silica gel chromatography (methylene chloride/methanol) to afford 2-(4-benzyloxy-1*H*-indol-3-yl)-2-oxo-*N*-propylacetamide as a yellow oil (1 g, 33% yield). This yellow oil was dissolved in tetrahydrofuran (15 ml) and cooled in an ice bath, then 9 ml of 1.0 *M* borane-tetrahydrofuran in tetrahydrofuran was added dropwise. The mixture was heated at reflux for 2 h, cooled to room temperature, and then ammonium hydroxide was added until it was basic. The mixture was extracted with methylene chloride and the organic phase was washed with water and brine. It was dried over sodium sulfate, then solvent was removed *in vacuo*, and purified by silica gel chromatography to yield 4-benzyloxy-*N*-propyltryptamine (4-BnO-NPT) as a yellow solid (192 mg, 21% yield). To a solution of 4-BnO-NPT (190 mg) in methanol (4 ml) was added Pd/C (40 mg) and $Pd(OH)_2$/C (40 mg). The mixture was stirred for 3 h under a hydrogen atmosphere. The resulting black suspension was filtered, the solid was washed with methanol, and the combined filtrates were concentrated under reduced pressure. The resulting residue was purified by silica gel chromatography [methylene chloride/methanol (3.5 *M* ammonia)] to afford 4-hydroxy-*N*-propyltryptamine (4-HO-NPT) as a white solid (75 mg, 55% yield). Single crystals suitable for X-ray diffraction studies were grown from the slow evaporation of an acetone solution.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. N- and O-bound H atoms were refined with restrained distances. C-bound H atoms were positioned geometrically (0.93–0.97 Å) and refined as riding with $U_{iso}(H) = 1.2–1.5U_{eq}(C)$.

Table 3
Experimental details.

	4-HO-NET	4-HO-NPT
Crystal data		
Chemical formula	$C_{12}H_{16}N_2O$	$C_{13}H_{18}N_2O$
M_r	204.27	218.29
Crystal system, space group	Monoclinic, $P2_1/c$	Monoclinic, $P2_1/n$
Temperature (K)	300	300
a, b, c (Å)	8.3887 (5), 9.0776 (5), 14.5657 (9)	9.0478 (6), 11.6680 (8), 11.3885 (7)
β (°)	100.132 (2)	91.523 (2)
V (Å^3)	1091.87 (11)	1201.86 (14)
Z	4	4
$F(000)$	440	472
D_x (Mg m^{-3})	1.243	1.206
Radiation type	Mo $K\alpha$	Mo $K\alpha$
No. of reflections for cell measurement	9957	8762
θ range (°) for cell measurement	2.7–26.3	2.8–26.4
μ (mm^{-1})	0.08	0.08
Crystal shape	Block	Block
Colour	Yellow	Colourless
Crystal size (mm)	0.27 × 0.21 × 0.20	0.24 × 0.22 × 0.21
Data collection		
Diffractometer	Bruker D8 Venture CMOS	Bruker D8 Venture CMOS
Scan method	φ and ω scans	φ and ω scans
Absorption correction	Multi-scan (*SADABS*; Krause *et al.*, 2015)	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.704, 0.745	0.705, 0.745
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	23801, 2243, 1953	34791, 2472, 1941
R_{int}	0.028	0.040
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.625	0.626
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.040, 0.112, 1.05	0.038, 0.107, 1.01
No. of reflections	2243	2472
No. of parameters	150	159
No. of restraints	3	3
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})	0.25, −0.13	0.14, −0.11

Computer programs: *APEX3* and *SAINT* (Bruker, 2021), *SHELXT2014* (Sheldrick, 2015*a*), *SHELXL2018* (Sheldrick, 2015*b*), *OLEX2* (Dolomanov *et al.*, 2009) and *publCIF* (Westrip, 2010).

Acknowledgements

Financial statements and conflict of interest: This study was funded by CaaMTech, Inc. ARC reports an ownership interest in CaaMTech, Inc., which owns US and worldwide patent applications, covering new tryptamine compounds, compositions, formulations, novel crystalline forms, and methods of making and using the same.

Funding information

Funding for this research was provided by: National Science Foundation, Directorate for Mathematical and Physical Sciences (grant No. CHE-1429086).

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supporting information

Acta Cryst. (2026). E**82**, 1082-1086 [https://doi.org/10.1107/S2056989026008571]

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Marilyn Naeem, Andrew R. Chadeayne, James A. Golen and David R. Manke

Computing details

3-[2-(Ethylamino)ethyl]-1*H*-indol-4-ol (4-HO-NET)

Crystal data

$C_{12}H_{16}N_2O$
M_r = 204.27
Monoclinic, $P2_1/c$
a = 8.3887 (5) Å
b = 9.0776 (5) Å
c = 14.5657 (9) Å
β = 100.132 (2)°
V = 1091.87 (11) Å^3
Z = 4
$F(000)$ = 440
D_x = 1.243 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9957 reflections
θ = 2.7–26.3°
μ = 0.08 mm^{-1}
T = 300 K
Block, yellow
0.27 × 0.21 × 0.20 mm

Data collection

Bruker D8 Venture CMOS diffractometer
φ and ω scans
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
T_{min} = 0.704, T_{max} = 0.745
23801 measured reflections
2243 independent reflections
1953 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 26.4°, θ_{min} = 2.7°
h = −10→10
k = −11→11
l = −18→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.112
S = 1.05
2243 reflections
150 parameters
3 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0518P)^2 + 0.3161P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}
Extinction correction: SHELXL2018 (Sheldrick, 2015b), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.007 (2)

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	0.26670 (16)	0.64120 (12)	0.59539 (6)	0.0558 (3)
N1	0.24109 (16)	0.75788 (14)	0.90599 (8)	0.0457 (3)
N2	0.35265 (16)	0.37322 (13)	0.63603 (9)	0.0457 (3)
C1	0.32861 (17)	0.63081 (16)	0.90504 (9)	0.0425 (3)
H1B	0.373600	0.578230	0.958099	0.051*
C2	0.19401 (15)	0.80509 (14)	0.81596 (9)	0.0365 (3)
C3	0.10539 (17)	0.93092 (16)	0.78357 (10)	0.0444 (3)
H3	0.068680	0.996395	0.824333	0.053*
C4	0.07518 (17)	0.95333 (16)	0.68926 (11)	0.0462 (4)
H4	0.017317	1.036282	0.665300	0.055*
C5	0.12934 (17)	0.85438 (15)	0.62827 (10)	0.0439 (3)
H5	0.105785	0.872798	0.564516	0.053*
C6	0.21698 (16)	0.72971 (14)	0.65966 (9)	0.0362 (3)
C7	0.25324 (14)	0.70272 (13)	0.75676 (8)	0.0313 (3)
C8	0.34133 (15)	0.59139 (14)	0.81676 (9)	0.0354 (3)
C9	0.43485 (16)	0.45785 (15)	0.79633 (10)	0.0422 (3)
H9A	0.480105	0.411568	0.855178	0.051*
H9B	0.524860	0.490499	0.767807	0.051*
C10	0.34164 (18)	0.34175 (15)	0.73357 (10)	0.0451 (3)
H10A	0.386353	0.245039	0.750729	0.054*
H10B	0.229064	0.342004	0.741166	0.054*
C11	0.2549 (2)	0.2752 (2)	0.56823 (13)	0.0634 (5)
H11A	0.140925	0.290158	0.569964	0.076*
H11B	0.281328	0.173480	0.584668	0.076*
C12	0.2861 (3)	0.3053 (2)	0.47154 (13)	0.0805 (6)
H12A	0.219642	0.241769	0.427983	0.121*
H12B	0.398139	0.287178	0.469347	0.121*
H12C	0.260572	0.406187	0.455488	0.121*
H1A	0.229 (2)	0.8046 (19)	0.9562 (9)	0.067 (5)*
H1	0.301 (2)	0.5388 (13)	0.6159 (14)	0.086 (6)*
H2	0.4583 (12)	0.3621 (19)	0.6293 (12)	0.062 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0953 (9)	0.0449 (6)	0.0303 (5)	0.0192 (6)	0.0194 (5)	0.0045 (4)
N1	0.0593 (7)	0.0476 (7)	0.0307 (6)	0.0011 (6)	0.0096 (5)	−0.0077 (5)
N2	0.0504 (7)	0.0407 (6)	0.0468 (7)	0.0037 (5)	0.0102 (5)	−0.0051 (5)
C1	0.0493 (8)	0.0449 (7)	0.0316 (6)	−0.0017 (6)	0.0026 (5)	0.0025 (5)

C2	0.0374 (6)	0.0373 (7)	0.0355 (6)	−0.0048 (5)	0.0084 (5)	−0.0039 (5)
C3	0.0433 (7)	0.0399 (7)	0.0518 (8)	0.0043 (6)	0.0126 (6)	−0.0084 (6)
C4	0.0419 (7)	0.0383 (7)	0.0576 (9)	0.0073 (6)	0.0067 (6)	0.0052 (6)
C5	0.0491 (8)	0.0437 (7)	0.0377 (7)	0.0039 (6)	0.0046 (6)	0.0064 (6)
C6	0.0426 (7)	0.0344 (6)	0.0324 (6)	−0.0008 (5)	0.0088 (5)	0.0010 (5)
C7	0.0319 (6)	0.0308 (6)	0.0320 (6)	−0.0038 (5)	0.0074 (5)	0.0000 (5)
C8	0.0374 (6)	0.0358 (6)	0.0331 (6)	−0.0028 (5)	0.0063 (5)	0.0021 (5)
C9	0.0438 (7)	0.0401 (7)	0.0424 (7)	0.0062 (6)	0.0065 (6)	0.0060 (6)
C10	0.0524 (8)	0.0341 (7)	0.0512 (8)	0.0051 (6)	0.0159 (6)	0.0025 (6)
C11	0.0675 (11)	0.0538 (9)	0.0665 (11)	0.0006 (8)	0.0049 (8)	−0.0181 (8)
C12	0.1077 (17)	0.0721 (13)	0.0539 (10)	0.0148 (12)	−0.0069 (10)	−0.0130 (9)

Geometric parameters (Å, º)

O1—C6	1.3539 (16)	C5—H5	0.9300
O1—H1	1.003 (9)	C5—C6	1.3827 (19)
N1—C1	1.3687 (19)	C6—C7	1.4147 (17)
N1—C2	1.3700 (17)	C7—C8	1.4506 (17)
N1—H1A	0.867 (9)	C8—C9	1.5018 (18)
N2—C10	1.4679 (19)	C9—H9A	0.9700
N2—C11	1.468 (2)	C9—H9B	0.9700
N2—H2	0.914 (9)	C9—C10	1.519 (2)
C1—H1B	0.9300	C10—H10A	0.9700
C1—C8	1.3569 (18)	C10—H10B	0.9700
C2—C3	1.3988 (19)	C11—H11A	0.9700
C2—C7	1.4159 (17)	C11—H11B	0.9700
C3—H3	0.9300	C11—C12	1.502 (3)
C3—C4	1.368 (2)	C12—H12A	0.9600
C4—H4	0.9300	C12—H12B	0.9600
C4—C5	1.394 (2)	C12—H12C	0.9600
C6—O1—H1	116.7 (12)	C1—C8—C7	105.70 (11)
C1—N1—C2	108.59 (11)	C1—C8—C9	122.11 (12)
C1—N1—H1A	124.3 (13)	C7—C8—C9	132.18 (11)
C2—N1—H1A	126.7 (13)	C8—C9—H9A	108.1
C10—N2—H2	108.0 (11)	C8—C9—H9B	108.1
C11—N2—C10	114.23 (13)	C8—C9—C10	116.60 (11)
C11—N2—H2	107.4 (11)	H9A—C9—H9B	107.3
N1—C1—H1B	124.3	C10—C9—H9A	108.1
C8—C1—N1	111.32 (12)	C10—C9—H9B	108.1
C8—C1—H1B	124.3	N2—C10—C9	109.52 (11)
N1—C2—C3	128.68 (12)	N2—C10—H10A	109.8
N1—C2—C7	107.74 (11)	N2—C10—H10B	109.8
C3—C2—C7	123.58 (12)	C9—C10—H10A	109.8
C2—C3—H3	121.5	C9—C10—H10B	109.8
C4—C3—C2	116.99 (12)	H10A—C10—H10B	108.2
C4—C3—H3	121.5	N2—C11—H11A	109.5
C3—C4—H4	119.3	N2—C11—H11B	109.5

C3—C4—C5	121.37 (13)	N2—C11—C12	110.58 (16)
C5—C4—H4	119.3	H11A—C11—H11B	108.1
C4—C5—H5	119.0	C12—C11—H11A	109.5
C6—C5—C4	122.04 (13)	C12—C11—H11B	109.5
C6—C5—H5	119.0	C11—C12—H12A	109.5
O1—C6—C5	117.88 (12)	C11—C12—H12B	109.5
O1—C6—C7	123.38 (11)	C11—C12—H12C	109.5
C5—C6—C7	118.72 (12)	H12A—C12—H12B	109.5
C2—C7—C8	106.65 (11)	H12A—C12—H12C	109.5
C6—C7—C2	117.29 (11)	H12B—C12—H12C	109.5
C6—C7—C8	136.06 (11)		

O1—C6—C7—C2	179.68 (12)	C3—C2—C7—C6	−1.21 (19)
O1—C6—C7—C8	0.0 (2)	C3—C2—C7—C8	178.54 (12)
N1—C1—C8—C7	−0.41 (15)	C3—C4—C5—C6	−0.5 (2)
N1—C1—C8—C9	178.50 (12)	C4—C5—C6—O1	−178.97 (13)
N1—C2—C3—C4	179.56 (14)	C4—C5—C6—C7	−0.4 (2)
N1—C2—C7—C6	179.46 (11)	C5—C6—C7—C2	1.16 (18)
N1—C2—C7—C8	−0.79 (14)	C5—C6—C7—C8	−178.49 (13)
C1—N1—C2—C3	−178.73 (14)	C6—C7—C8—C1	−179.60 (14)
C1—N1—C2—C7	0.55 (15)	C6—C7—C8—C9	1.7 (2)
C1—C8—C9—C10	121.65 (15)	C7—C2—C3—C4	0.4 (2)
C2—N1—C1—C8	−0.08 (16)	C7—C8—C9—C10	−59.77 (19)
C2—C3—C4—C5	0.5 (2)	C8—C9—C10—N2	90.62 (14)
C2—C7—C8—C1	0.73 (14)	C10—N2—C11—C12	−174.34 (14)
C2—C7—C8—C9	−178.02 (13)	C11—N2—C10—C9	−174.88 (12)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O1^{i}	0.87 (1)	2.06 (1)	2.8789 (15)	158 (2)
O1—H1···N2	1.00 (1)	1.58 (1)	2.5764 (16)	173 (2)

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

3-[2-(Propylamino)ethyl]-1*H*-indol-4-ol (4-HO-NPT)

Crystal data

$C_{13}H_{18}N_2O$
M_r = 218.29
Monoclinic, $P2_1/n$
a = 9.0478 (6) Å
b = 11.6680 (8) Å
c = 11.3885 (7) Å
β = 91.523 (2)°
V = 1201.86 (14) Å^3
Z = 4

$F(000)$ = 472
D_x = 1.206 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 8762 reflections
θ = 2.8–26.4°
μ = 0.08 mm^{-1}
T = 300 K
Block, colourless
0.24 × 0.22 × 0.21 mm

Data collection

Bruker D8 Venture CMOS diffractometer
φ and ω scans
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
T_{min} = 0.705, T_{max} = 0.745
34791 measured reflections
2472 independent reflections
1941 reflections with $I > 2\sigma(I)$
R_{int} = 0.040
θ_{max} = 26.4°, θ_{min} = 2.8°
h = −11→11
k = −14→14
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.107
S = 1.01
2472 reflections
159 parameters
3 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0476P)^2 + 0.2229P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.11 e Å^{-3}
Extinction correction: SHELXL2018 (Sheldrick, 2015b), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.010 (2)

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	0.67289 (11)	0.65319 (8)	0.46631 (9)	0.0625 (3)
N1	0.37615 (16)	0.73704 (13)	0.13702 (12)	0.0744 (4)
N2	0.65373 (13)	0.43935 (10)	0.40664 (11)	0.0581 (3)
C1	0.4626 (2)	0.64513 (15)	0.11322 (13)	0.0727 (5)
H1B	0.459562	0.605365	0.042428	0.087*
C2	0.41003 (15)	0.77475 (12)	0.24805 (12)	0.0555 (4)
C3	0.35237 (17)	0.86658 (14)	0.30944 (15)	0.0664 (4)
H3	0.279287	0.913394	0.276289	0.080*
C4	0.40760 (18)	0.88524 (13)	0.42067 (15)	0.0683 (4)
H4	0.372585	0.946906	0.463557	0.082*
C5	0.51543 (17)	0.81379 (12)	0.47138 (13)	0.0610 (4)
H5	0.549839	0.828481	0.547572	0.073*
C6	0.57192 (14)	0.72214 (11)	0.41115 (11)	0.0474 (3)
C7	0.52103 (13)	0.70145 (11)	0.29519 (11)	0.0459 (3)
C8	0.55428 (15)	0.61854 (12)	0.20607 (11)	0.0549 (3)
C9	0.66469 (18)	0.52272 (14)	0.20690 (13)	0.0671 (4)
H9A	0.759008	0.552462	0.235646	0.081*
H9B	0.677518	0.497638	0.126609	0.081*
C10	0.62556 (19)	0.41950 (13)	0.28017 (15)	0.0703 (4)
H10A	0.521883	0.401214	0.266863	0.084*

H10B	0.683237	0.354207	0.255279	0.084*
C11	0.79192 (15)	0.38631 (13)	0.45205 (14)	0.0606 (4)
H11A	0.873537	0.411109	0.404706	0.073*
H11B	0.784125	0.303640	0.445430	0.073*
C12	0.82328 (17)	0.41798 (14)	0.57805 (14)	0.0652 (4)
H12A	0.733916	0.407145	0.621928	0.078*
H12B	0.848746	0.498669	0.581958	0.078*
C13	0.9464 (2)	0.34963 (17)	0.63607 (16)	0.0825 (5)
H13A	0.960198	0.374269	0.716039	0.124*
H13B	1.036130	0.361400	0.594605	0.124*
H13C	0.921194	0.269711	0.634508	0.124*
H1	0.664 (2)	0.5715 (10)	0.4388 (17)	0.109 (7)*
H1A	0.3140 (18)	0.7706 (16)	0.0882 (14)	0.097 (6)*
H2	0.5807 (16)	0.4070 (15)	0.4476 (15)	0.089 (6)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0740 (6)	0.0542 (6)	0.0577 (6)	0.0054 (5)	−0.0287 (5)	−0.0068 (4)
N1	0.0834 (9)	0.0706 (9)	0.0672 (8)	−0.0035 (7)	−0.0373 (7)	0.0081 (7)
N2	0.0528 (7)	0.0504 (7)	0.0708 (8)	0.0000 (5)	−0.0054 (6)	−0.0020 (6)
C1	0.0961 (12)	0.0687 (10)	0.0517 (8)	−0.0093 (9)	−0.0259 (8)	−0.0037 (7)
C2	0.0519 (7)	0.0557 (8)	0.0581 (8)	−0.0098 (6)	−0.0138 (6)	0.0105 (6)
C3	0.0568 (8)	0.0571 (9)	0.0849 (11)	0.0028 (7)	−0.0074 (7)	0.0147 (8)
C4	0.0762 (10)	0.0531 (8)	0.0759 (10)	0.0061 (7)	0.0057 (8)	−0.0005 (7)
C5	0.0782 (10)	0.0525 (8)	0.0519 (8)	−0.0015 (7)	−0.0044 (7)	−0.0035 (6)
C6	0.0495 (7)	0.0453 (7)	0.0469 (7)	−0.0073 (5)	−0.0082 (5)	0.0020 (5)
C7	0.0434 (6)	0.0478 (7)	0.0460 (7)	−0.0104 (5)	−0.0061 (5)	0.0038 (5)
C8	0.0598 (8)	0.0582 (8)	0.0462 (7)	−0.0103 (6)	−0.0092 (6)	−0.0018 (6)
C9	0.0679 (9)	0.0782 (11)	0.0548 (8)	0.0017 (8)	−0.0053 (7)	−0.0182 (7)
C10	0.0737 (10)	0.0549 (9)	0.0810 (11)	0.0047 (7)	−0.0234 (8)	−0.0175 (8)
C11	0.0556 (8)	0.0516 (8)	0.0741 (10)	0.0037 (6)	−0.0053 (7)	0.0035 (7)
C12	0.0639 (9)	0.0655 (9)	0.0661 (9)	−0.0032 (7)	−0.0010 (7)	0.0121 (7)
C13	0.0751 (11)	0.0896 (13)	0.0819 (11)	−0.0025 (9)	−0.0155 (9)	0.0224 (10)

Geometric parameters (Å, º)

O1—C6	1.3587 (15)	C6—C7	1.4078 (16)
O1—H1	1.006 (9)	C7—C8	1.4397 (19)
N1—C1	1.359 (2)	C8—C9	1.499 (2)
N1—C2	1.3659 (19)	C9—H9A	0.9700
N1—H1A	0.873 (9)	C9—H9B	0.9700
N2—C10	1.474 (2)	C9—C10	1.513 (2)
N2—C11	1.4762 (18)	C10—H10A	0.9700
N2—H2	0.902 (9)	C10—H10B	0.9700
C1—H1B	0.9300	C11—H11A	0.9700
C1—C8	1.3623 (19)	C11—H11B	0.9700
C2—C3	1.389 (2)	C11—C12	1.501 (2)

C2—C7	1.4142 (18)	C12—H12A	0.9700
C3—H3	0.9300	C12—H12B	0.9700
C3—C4	1.367 (2)	C12—C13	1.508 (2)
C4—H4	0.9300	C13—H13A	0.9600
C4—C5	1.397 (2)	C13—H13B	0.9600
C5—H5	0.9300	C13—H13C	0.9600
C5—C6	1.3763 (19)		

C6—O1—H1	111.6 (12)	C8—C9—H9A	108.4
C1—N1—C2	108.83 (12)	C8—C9—H9B	108.4
C1—N1—H1A	126.3 (13)	C8—C9—C10	115.57 (13)
C2—N1—H1A	124.7 (13)	H9A—C9—H9B	107.4
C10—N2—C11	113.53 (12)	C10—C9—H9A	108.4
C10—N2—H2	109.3 (12)	C10—C9—H9B	108.4
C11—N2—H2	105.6 (12)	N2—C10—C9	112.14 (12)
N1—C1—H1B	124.2	N2—C10—H10A	109.2
N1—C1—C8	111.52 (14)	N2—C10—H10B	109.2
C8—C1—H1B	124.2	C9—C10—H10A	109.2
N1—C2—C3	129.54 (13)	C9—C10—H10B	109.2
N1—C2—C7	107.23 (13)	H10A—C10—H10B	107.9
C3—C2—C7	123.23 (13)	N2—C11—H11A	109.3
C2—C3—H3	121.4	N2—C11—H11B	109.3
C4—C3—C2	117.17 (14)	N2—C11—C12	111.62 (13)
C4—C3—H3	121.4	H11A—C11—H11B	108.0
C3—C4—H4	119.3	C12—C11—H11A	109.3
C3—C4—C5	121.49 (15)	C12—C11—H11B	109.3
C5—C4—H4	119.3	C11—C12—H12A	108.7
C4—C5—H5	119.3	C11—C12—H12B	108.7
C6—C5—C4	121.49 (13)	C11—C12—C13	114.03 (14)
C6—C5—H5	119.3	H12A—C12—H12B	107.6
O1—C6—C5	118.93 (11)	C13—C12—H12A	108.7
O1—C6—C7	122.09 (12)	C13—C12—H12B	108.7
C5—C6—C7	118.96 (12)	C12—C13—H13A	109.5
C2—C7—C8	107.36 (11)	C12—C13—H13B	109.5
C6—C7—C2	117.62 (12)	C12—C13—H13C	109.5
C6—C7—C8	135.02 (12)	H13A—C13—H13B	109.5
C1—C8—C7	105.06 (13)	H13A—C13—H13C	109.5
C1—C8—C9	124.52 (14)	H13B—C13—H13C	109.5
C7—C8—C9	130.41 (12)		

O1—C6—C7—C2	176.68 (11)	C3—C2—C7—C6	1.52 (19)
O1—C6—C7—C8	−2.7 (2)	C3—C2—C7—C8	−178.94 (13)
N1—C1—C8—C7	0.13 (18)	C3—C4—C5—C6	0.7 (2)
N1—C1—C8—C9	−179.06 (14)	C4—C5—C6—O1	−177.74 (13)
N1—C2—C3—C4	−179.79 (15)	C4—C5—C6—C7	1.0 (2)
N1—C2—C7—C6	−178.60 (12)	C5—C6—C7—C2	−2.00 (18)
N1—C2—C7—C8	0.94 (15)	C5—C6—C7—C8	178.62 (14)
N2—C11—C12—C13	169.17 (13)	C6—C7—C8—C1	178.77 (15)

C1—N1—C2—C3	179.00 (15)	C6—C7—C8—C9	−2.1 (3)
C1—N1—C2—C7	−0.87 (17)	C7—C2—C3—C4	0.1 (2)
C1—C8—C9—C10	−107.09 (17)	C7—C8—C9—C10	73.94 (19)
C2—N1—C1—C8	0.5 (2)	C8—C9—C10—N2	−77.85 (16)
C2—C3—C4—C5	−1.2 (2)	C10—N2—C11—C12	174.61 (13)
C2—C7—C8—C1	−0.65 (15)	C11—N2—C10—C9	−101.41 (15)
C2—C7—C8—C9	178.47 (15)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N2	1.01 (1)	1.59 (1)	2.5905 (15)	175 (2)
N1—H1*A*···O1^{i}	0.87 (1)	2.06 (1)	2.9347 (16)	177 (2)
N2—H2···O1^{ii}	0.90 (1)	2.62 (1)	3.4957 (17)	166 (2)

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