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Two 3-substituted 5-nitroindoles

J. G. Garcia, D. R. Billodeaux and F. R. Fronczek

Abstract

The structures of 3-(3-bromopropyl)-5-nitro-1*H*-indole, $C_{11}H_{11}N_2O_2Br$, and 3-(3-hydroxypropyl)-5-nitro-1*H*-indole, $C_{11}H_{12}N_2O_3$, have the nitro groups rotated out of the plane of the indole backbone by 6.6 (3)° for the bromo compound and 5.4 (2)° for the OH compound. The propyl subunits adopt the synclinal–synclinal conformation along the internal Csp^3 carbons consistently in both structures. Bond distances to the terminal substituent on the propyl group are: Br—C 1.955 (2) and O—C 1.428 (2) Å.

Comment

The title compounds were prepared as part of a structural study involving 3-substituted nitroindoles and their key synthetic precursors. The propyl group in both structures adopts the synclinal–synclinal conformation (Klyne & Prelog, 1960) with torsion angles about C9—C10 and C10—C11 of 52.6 (2)° and 62.4 (2)° for the Br compound, and 66.8 (2)° and 65.3 (2)° for the OH compound. The nitro groups are twisted slightly out of the indole plane, with the torsion angle C8—C7—N2—O1 having the value −6.6 (3)° in the Br compound and −5.4 (2)° in the OH compound. Geometrical data for the two compounds are in agreement with the accepted literature values (Allen *et al.*, 1987) of Csp^3—Br: 1.966Å [1.955 (2)Å in the Br compound]; Csp^3—OH: 1.426Å [1.428 (2)Å in the OH compound]; and Csp^2—Csp^2: 1.364Å [C1—C2 in the indole subunit 1.361 (3) for the Br compound and 1.359 (2) for the OH compound].

The indole N—H group does not engage in strong hydrogen bonding in either structure, but in the Br compound, it forms a N1—H···O2($x + 1$, y, $z - 1$) contact with an N···O distance 3.426 (3) Å and a 140 (3) ° angle about hydrogen. The OH group forms an intermolecular hydrogen bond with an O···O distance of 2.807 (2)Å to nitro group O atom O1, forming chains along the a direction.

Experimental

The compounds were synthesized according to published procedures (Eur. Patent Appl., 1996) from nitroindole in three steps for the OH compound and four steps for the Br compound. Single crystals of the Br compound were grown by slow and cold evaporation of dichloromethane. Crystals of the OH compound were grown by slow evaporation of a 1:1 ethyl acetate:dichloromethane solution.

Refinement

For the Br compound, C—H and N—H distances range 0.86 (3)–1.08 (2) Å, and U_{iso} values for H atoms range 0.042 (6)–0.12 (2) Å^2. Corresponding values for the OH compound are 0.87 (2)–1.04 (2) Å and 0.059 (5)–0.110 (7) Å^2, respectively.

CIF access

Computing details

Data collection: CAD-4 Operations Manual (Enraf-Nonius, 1977) for (I); *CAD-4 Software* (Enraf-Nonius, 1994) for (II). Cell refinement: CAD-4 Operations Manual for (I); *CAD-4 Software* for (II). For both compounds, data reduction: *PROCESS* in *MolEN* (Fair, 1990). Program(s) used to solve structure: direct methods (*SIR*; Burla *et al.*, 1989) for (I); *DIRDIF* (Beurskens, 1984) for (II). For both compounds, program(s) used to refine structure: *LSFM* in *MolEN*. Molecular graphics: *ORTEPII* (Johnson, 1976) for (I). Software used to prepare material for publication: CIF in *MolEN* for (I); *BTABLE PTABLE* CIF IN in *MolEN* for (II)

3-(3-bromopropyl)-5-nitro-1*H*-indole

Crystal data

$C_{11}H_{11}BrN_2O_2$	$V = 554.9\ (1)\ Å^3$
$M_r = 283.1$	$Z = 2$
Monoclinic, $P2_1$	Cu $K\alpha$
$a = 5.2326\ (3)$ Å	$\mu = 4.98\ mm^{-1}$
$b = 16.766\ (2)$ Å	$T = 298$ K
$c = 6.6130\ (5)$ Å	$0.32 \times 0.22 \times 0.20$ mm
$\beta = 106.983\ (5)^o$	

Data collection

Enraf Nonius CAD-4 diffractometer	2312 reflections with $I > 0$
Absorption correction: ψ scans (North et al., 1968)	$R_{int} = 0.019$
$T_{min} = 0.24$, $T_{max} = 0.37$	3 standard reflections
4694 measured reflections	every 120 min
2314 independent reflections	intensity decay: 0.2%

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.025$	Refined isotropically
$wR(F^2) = 0.036$	$\Delta\rho_{max} = 0.71$ e Å^{-3}
2312 reflections	$\Delta\rho_{min} = -0.63$ e Å^{-3}
189 parameters	Absolute structure: Refinement of the antipodal structure under identical circumstances yielded R = 0.040, wR = 0.055, S = 3.056.

Table 1
Selected geometric parameters (Å, °)

Br—C11	1.955 (2)	N1—C1	1.373 (3)
O1—N2	1.227 (3)	N1—C4	1.365 (3)
O2—N2	1.220 (3)	C1—C2	1.361 (3)
C1—N1—C4	109.1 (2)	O2—N2—C7	119.5 (2)

O1—N2—O2	122.9 (2)	N1—C1—C2	110.6 (2)
O1—N2—C7	117.6 (2)	Br—C11—C10	112.6 (1)
O1—N2—C7—C8	−6.6 (3)	C2—C9—C10—C11	52.6 (2)
C1—C2—C9—C10	−111.5 (2)	C9—C10—C11—Br	62.4 (2)

3-(3-hydroxypropyl)-5-nitro-1*H*-indole

Crystal data

$C_{11}H_{12}N_2O_3$	V = 1058.6 (3) Å^3
M_r = 220.2	Z = 4
Monoclinic, $P2_1/c$	Cu $K\alpha$
a = 10.3272 (7) Å	μ = 0.81 mm^{-1}
b = 13.962 (1) Å	T = 299 K
c = 7.4435 (6) Å	0.35 × 0.25 × 0.15 mm
β = 99.489 (6)°	

Data collection

Enraf Nonius CAD4 diffractometer	1946 reflections with > 1.0σ(I)
Absorption correction: ψ scans (North et al., 1968)	R_{int} = 0.017
T_{min} = 0.73, T_{max} = 0.89	3 standard reflections
2397 measured reflections	every 120 min
2171 independent reflections	intensity decay: 1.1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047	194 parameters
$wR(F^2)$ = 0.056	Refined isotropically
S = 1.85	$\Delta\rho_{max}$ = 0.22 e Å^{-3}
1946 reflections	$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Table 2
Selected geometric parameters (Å, °)

O1—N2	1.228 (2)	N1—C1	1.378 (2)
O2—N2	1.227 (2)	N1—C4	1.358 (2)
O3—C11	1.428 (2)	C1—C2	1.359 (2)
C1—N1—C4	108.9 (1)	O2—N2—C7	119.6 (1)
O1—N2—O2	122.0 (1)	O3—C11—C10	109.1 (1)
O1—N2—C7	118.4 (1)		
O2—N2—C7—C6	−5.1 (2)	C2—C9—C10—C11	66.8 (2)
C1—C2—C9—C10	−101.1 (2)	C9—C10—C11—O3	65.3 (2)

Table 3

CIF access

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3O···O1^{i}	0.87 (2)	1.97 (2)	2.807 (2)	161 (2)

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

Acknowledgements

The purchase of the diffractometer was made possible by a National Science Foundation chemical instrumentation grant, which we gratefully acknowledge. Improvements to the LSU X-ray Crystallography Facility were supported by Grant No. LEQSF(1996–97)-ENH-TR-10, administered by the Louisiana Board of Regents.

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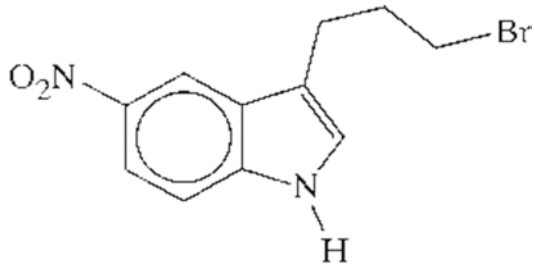
Scheme 1



supplementary materials

3-(3-bromopropyl)-5-nitro-1*H*-indole

Crystal data

$C_{11}H_{11}BrN_2O_2$	F_{000} = 284
M_r = 283.1	D_x = 1.694 Mg m^{-3}
Monoclinic, $P2_1$	Cu $K\alpha$ radiation λ = 1.54184 Å
Hall symbol: P 2y1	Cell parameters from 25 reflections
a = 5.2326 (3) Å	θ = 33–43º
b = 16.766 (2) Å	μ = 4.98 mm^{-1}
c = 6.6130 (5) Å	T = 298 K
β = 106.983 (5)º	Fragment, yellow
V = 554.9 (1) Å^3	0.32 × 0.22 × 0.20 mm
Z = 2	

Data collection

Enraf Nonius CAD-4 diffractometer	2312 reflections with $I > 0$
Radiation source: sealed tube	R_{int} = 0.019
Monochromator: graphite	θ_{max} = 76º
T = 298 K	h = −6→6
ω/2θ scans	k = −21→21
Absorption correction: ψ scans (North et al., 1968)	l = −7→8
T_{min} = 0.24, T_{max} = 0.37	3 standard reflections
4694 measured reflections	every 120 min
2314 independent reflections	intensity decay: 0.2%

Refinement

Refinement on F^2	$w = 4F_o^2/[\sigma^2(F_o^2) + 0.0004F_o^4]$?
$R[F^2 > 2\sigma(F^2)]$ = 0.025	$\Delta\rho_{max}$ = 0.71 e Å^{-3}
$wR(F^2)$ = 0.036	$\Delta\rho_{min}$ = −0.63 e Å^{-3}
2312 reflections	Extinction correction: isotropic (Zachariasen, 1963)
189 parameters	Extinction coefficient: 1.39E-5 (3)
Refined isotropically	Absolute structure: Refinement of the antipodal structure under identical circumstances yielded R = 0.040, wR = 0.055, S = 3.056.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Br	0.90568 (5)	0	−0.08162 (3)	0.0690 (1)
O1	0.4514 (3)	0.2052 (1)	0.8484 (3)	0.0587 (7)
O2	0.3563 (3)	0.3302 (1)	0.8239 (3)	0.0667 (8)
N1	0.9996 (3)	0.2867 (1)	0.1697 (3)	0.0526 (8)

N2	0.4624 (3)	0.2716 (1)	0.7739 (3)	0.0440 (7)
C1	1.1036 (4)	0.2110 (2)	0.1862 (3)	0.0503 (9)
C2	1.0321 (3)	0.1687 (1)	0.3368 (3)	0.0419 (8)
C3	0.8738 (3)	0.2216 (1)	0.4209 (3)	0.0363 (7)
C4	0.8579 (4)	0.2955 (1)	0.3119 (3)	0.0423 (8)
C5	0.7132 (4)	0.3603 (1)	0.3534 (4)	0.0476 (9)
C6	0.5876 (4)	0.3522 (1)	0.5092 (4)	0.0451 (9)
C7	0.6063 (3)	0.2795 (1)	0.6173 (3)	0.0376 (8)
C8	0.7450 (3)	0.2145 (1)	0.5767 (3)	0.0369 (7)
C9	1.1022 (4)	0.0838 (1)	0.3995 (3)	0.0459 (9)
C10	0.8616 (4)	0.0273 (1)	0.3380 (3)	0.0452 (8)
C11	0.6999 (4)	0.0311 (1)	0.1093 (3)	0.0466 (9)
H1N	1.026 (6)	0.318 (2)	0.070 (4)	0.060 (8)*
H1	1.230 (6)	0.196 (2)	0.107 (4)	0.059 (8)*
H5	0.729 (8)	0.413 (3)	0.277 (6)	0.12 (2)*
H6	0.493 (5)	0.396 (2)	0.542 (4)	0.049 (6)*
H8	0.738 (5)	0.166 (1)	0.637 (4)	0.042 (6)*
H9a	1.187 (5)	0.080 (2)	0.569 (4)	0.056 (7)*
H9b	1.245 (5)	0.062 (2)	0.326 (4)	0.048 (6)*
H10a	0.746 (5)	0.040 (2)	0.428 (4)	0.062 (8)*
H10b	0.918 (5)	−0.032 (2)	0.384 (4)	0.059 (8)*
H11a	0.551 (4)	0.005 (3)	0.083 (3)	0.052 (7)*
H11b	0.655 (5)	0.085 (2)	0.069 (4)	0.051 (7)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br	0.0899 (1)	0.0653 (1)	0.0558 (1)	−0.0062 (1)	0.03660 (9)	−0.0128 (1)
O1	0.0612 (6)	0.0578 (9)	0.0572 (7)	0.0040 (7)	0.0331 (6)	0.0076 (8)
O2	0.0723 (7)	0.0603 (9)	0.0683 (9)	0.0171 (7)	0.0348 (6)	−0.0065 (9)
N1	0.0551 (7)	0.058 (1)	0.0454 (8)	−0.0157 (7)	0.0244 (6)	0.0057 (8)
N2	0.0422 (6)	0.0507 (8)	0.0397 (7)	0.0015 (6)	0.0157 (6)	−0.0045 (8)
C1	0.0420 (7)	0.065 (1)	0.0464 (8)	−0.0142 (8)	0.0227 (6)	−0.008 (1)
C2	0.0343 (6)	0.052 (1)	0.0412 (8)	−0.0070 (7)	0.0154 (6)	−0.0068 (8)
C3	0.0324 (6)	0.0393 (9)	0.0377 (8)	−0.0059 (6)	0.0082 (6)	−0.0018 (7)
C4	0.0431 (7)	0.0463 (9)	0.0380 (8)	−0.0111 (7)	0.0117 (7)	0.0006 (8)
C5	0.0549 (9)	0.0384 (9)	0.051 (1)	−0.0079 (8)	0.0110 (8)	0.0039 (9)
C6	0.0490 (8)	0.0382 (9)	0.049 (1)	0.0001 (8)	0.0133 (7)	−0.0015 (9)
C7	0.0377 (7)	0.0410 (9)	0.0344 (7)	−0.0007 (6)	0.0084 (6)	−0.0009 (8)
C8	0.0369 (6)	0.0384 (8)	0.0354 (7)	−0.0031 (7)	0.0117 (6)	−0.0012 (8)
C9	0.0378 (7)	0.053 (1)	0.048 (1)	0.0059 (8)	0.0131 (7)	−0.0058 (9)
C10	0.0496 (7)	0.0437 (9)	0.0425 (8)	0.0039 (7)	0.0228 (6)	0.0011 (8)
C11	0.0459 (7)	0.0438 (9)	0.050 (1)	0.0002 (8)	0.0171 (7)	−0.0031 (9)

Geometric parameters (Å, °)

Br—C11	1.955 (2)	C5—C6	1.382 (4)
O1—N2	1.227 (3)	C5—H5	1.03 (4)
O2—N2	1.220 (3)	C6—C7	1.402 (3)

N1—C1	1.373 (3)	C6—H6	0.95 (3)
N1—C4	1.365 (3)	C7—C8	1.377 (3)
N1—H1N	0.88 (3)	C8—H8	0.92 (3)
N2—C7	1.454 (3)	C9—C10	1.533 (3)
C1—C2	1.361 (3)	C9—H9a	1.08 (2)
C1—H1	0.99 (3)	C9—H9b	1.07 (3)
C2—C3	1.432 (3)	C10—C11	1.503 (3)
C2—C9	1.498 (3)	C10—H10a	0.99 (3)
C3—C4	1.424 (3)	C10—H10b	1.05 (3)
C3—C8	1.391 (3)	C11—H11a	0.86 (3)
C4—C5	1.396 (3)	C11—H11b	0.95 (3)
C1—N1—C4	109.1 (2)	N2—C7—C6	117.9 (2)
C1—N1—H1N	117 (2)	N2—C7—C8	118.6 (2)
C4—N1—H1N	133 (2)	C6—C7—C8	123.4 (2)
O1—N2—O2	122.9 (2)	C3—C8—C7	118.3 (2)
O1—N2—C7	117.6 (2)	C3—C8—H8	119 (1)
O2—N2—C7	119.5 (2)	C7—C8—H8	122 (1)
N1—C1—C2	110.6 (2)	C2—C9—C10	113.5 (1)
N1—C1—H1	120 (1)	C2—C9—H9a	109 (1)
C2—C1—H1	128 (1)	C2—C9—H9b	110 (1)
C1—C2—C3	106.0 (2)	C10—C9—H9a	107 (1)
C1—C2—C9	126.9 (2)	C10—C9—H9b	107 (1)
C3—C2—C9	127.1 (2)	H9a—C9—H9b	108 (1)
C2—C3—C4	107.3 (2)	C9—C10—C11	114.7 (2)
C2—C3—C8	134.1 (2)	C9—C10—H10a	108 (1)
C4—C3—C8	118.7 (2)	C9—C10—H10b	111 (1)
N1—C4—C3	106.9 (2)	C11—C10—H10a	109 (1)
N1—C4—C5	130.9 (2)	C11—C10—H10b	111 (1)
C3—C4—C5	122.2 (2)	H10a—C10—H10b	101 (2)
C4—C5—C6	118.3 (2)	Br—C11—C10	112.6 (1)
C4—C5—H5	116 (2)	Br—C11—H11a	111 (2)
C6—C5—H5	125 (2)	Br—C11—H11b	102 (1)
C5—C6—C7	119.2 (2)	C10—C11—H11a	112 (1)
C5—C6—H6	119 (1)	C10—C11—H11b	110 (1)
C7—C6—H6	121 (1)	H11a—C11—H11b	106 (3)
C4—N1—C1—C2	0.4 (3)	C2—C3—C4—N1	0.2 (2)
C1—N1—C4—C3	−0.3 (2)	C2—C3—C4—C5	178.9 (2)
C1—N1—C4—C5	−178.9 (2)	C8—C3—C4—N1	179.9 (2)
O1—N2—C7—C6	171.0 (2)	C8—C3—C4—C5	−1.4 (3)
O1—N2—C7—C8	−6.6 (3)	C2—C3—C8—C7	−179.9 (2)
O2—N2—C7—C6	−8.3 (3)	C4—C3—C8—C7	0.5 (3)
O2—N2—C7—C8	174.2 (2)	N1—C4—C5—C6	−180.0 (4)
N1—C1—C2—C3	−0.3 (2)	C3—C4—C5—C6	1.6 (3)
N1—C1—C2—C9	179.0 (2)	C4—C5—C6—C7	−0.9 (3)
C1—C2—C3—C4	0.1 (3)	C5—C6—C7—N2	−177.4 (2)
C1—C2—C3—C8	−179.6 (2)	C5—C6—C7—C8	0.1 (4)
C9—C2—C3—C4	−179.2 (2)	N2—C7—C8—C3	177.6 (2)
C9—C2—C3—C8	1.1 (3)	C6—C7—C8—C3	0.2 (3)

C1—C2—C9—C10	−111.5 (2)	C2—C9—C10—C11	52.6 (2)
C3—C2—C9—C10	67.7 (3)	C9—C10—C11—Br	62.4 (2)

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T_{min} = 0.73, T_{max} = 0.89	3 standard reflections
2397 measured reflections	every 120 min
2171 independent reflections	intensity decay: 1.1%
1946 reflections with > 1.0σ(I)	

Refinement

Refinement on F^2	Refined isotropically
Least-squares matrix: full	$w = 4F_o^2/[\sigma^2(F_o^2) + 0.0004F_o^4]$?
$R[F^2 > 2\sigma(F^2)]$ = 0.047	$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$wR(F^2)$ = 0.056	$\Delta\rho_{min}$ = −0.18 e Å^{-3}
S = 1.85	Extinction correction: isotropic (Zachariasen, 1963)
1946 reflections	Extinction coefficient: 3.3E-6 (3)
194 parameters	

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

	x	y	z	U_{iso}*/U_{eq}
O1	0.3130 (1)	0.58090 (8)	0.3046 (2)	0.0647 (7)
O2	0.2369 (1)	0.43943 (9)	0.3313 (2)	0.0682 (7)

O3	1.0723 (1)	0.67467 (8)	0.3020 (2)	0.0629 (7)
N1	0.8025 (1)	0.35725 (9)	0.1770 (2)	0.0550 (7)
N2	0.3261 (1)	0.49344 (9)	0.3067 (2)	0.0485 (6)
C1	0.8608 (2)	0.4421 (1)	0.1397 (2)	0.0552 (8)
C2	0.7794 (1)	0.5174 (1)	0.1519 (2)	0.0438 (7)
C3	0.6625 (1)	0.4768 (1)	0.2019 (2)	0.0392 (6)
C4	0.6812 (1)	0.3763 (1)	0.2159 (2)	0.0443 (7)
C5	0.5851 (2)	0.3147 (1)	0.2618 (2)	0.0514 (8)
C6	0.4691 (1)	0.3542 (1)	0.2925 (2)	0.0506 (8)
C7	0.4507 (1)	0.4538 (1)	0.2773 (2)	0.0427 (7)
C8	0.5443 (1)	0.5157 (1)	0.2331 (2)	0.0394 (6)
C9	0.8042 (1)	0.6213 (1)	0.1224 (2)	0.0485 (7)
C10	0.8415 (1)	0.6785 (1)	0.2977 (2)	0.0469 (7)
C11	0.9713 (2)	0.6509 (1)	0.4044 (2)	0.0542 (9)
H3O	1.138 (2)	0.635 (2)	0.313 (3)	0.110 (7)*
H1N	0.838 (2)	0.299 (1)	0.176 (2)	0.087 (6)*
H1	0.951 (2)	0.444 (1)	0.109 (2)	0.071 (5)*
H5	0.603 (2)	0.249 (1)	0.270 (2)	0.081 (6)*
H6	0.403 (2)	0.314 (1)	0.323 (2)	0.077 (6)*
H8	0.525 (1)	0.585 (1)	0.224 (2)	0.060 (5)*
H9a	0.725 (2)	0.650 (1)	0.051 (2)	0.069 (5)*
H9b	0.875 (1)	0.628 (1)	0.050 (2)	0.059 (5)*
H10a	0.772 (2)	0.671 (1)	0.382 (2)	0.063 (5)*
H10b	0.844 (2)	0.749 (1)	0.265 (2)	0.065 (5)*
H11a	0.987 (2)	0.686 (1)	0.527 (3)	0.099 (7)*
H11b	0.978 (1)	0.582 (1)	0.429 (2)	0.068 (5)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0483 (5)	0.0520 (6)	0.1079 (9)	0.0080 (5)	0.0214 (6)	−0.0062 (6)
O2	0.0490 (5)	0.0728 (8)	0.0892 (8)	−0.0124 (5)	0.0252 (5)	0.0014 (6)
O3	0.0386 (5)	0.0483 (6)	0.134 (1)	0.0019 (5)	0.0176 (6)	0.0104 (7)
N1	0.0485 (6)	0.0416 (6)	0.0823 (9)	0.0104 (5)	0.0134 (6)	−0.0056 (6)
N2	0.0396 (6)	0.0513 (7)	0.0560 (6)	−0.0016 (5)	0.0096 (5)	−0.0035 (6)
C1	0.0444 (7)	0.0531 (9)	0.0714 (9)	0.0041 (7)	0.0177 (7)	−0.0043 (7)
C2	0.0390 (6)	0.0442 (7)	0.0487 (7)	0.0007 (6)	0.0091 (5)	−0.0017 (6)
C3	0.0382 (6)	0.0360 (6)	0.0437 (6)	0.0011 (5)	0.0041 (5)	−0.0021 (5)
C4	0.0415 (7)	0.0367 (7)	0.0572 (8)	0.0044 (6)	0.0038 (6)	−0.0051 (6)
C5	0.0543 (8)	0.0310 (7)	0.081 (1)	−0.0009 (6)	0.0048 (8)	−0.0017 (7)
C6	0.0475 (7)	0.0387 (7)	0.071 (1)	−0.0083 (6)	0.0072 (7)	0.0007 (7)
C7	0.0378 (6)	0.0412 (7)	0.0501 (7)	−0.0011 (6)	0.0063 (6)	−0.0035 (6)
C8	0.0387 (6)	0.0344 (6)	0.0459 (7)	0.0019 (5)	0.0054 (5)	−0.0017 (6)
C9	0.0444 (7)	0.0471 (7)	0.0544 (8)	−0.0019 (6)	0.0111 (6)	0.0064 (7)
C10	0.0413 (6)	0.0401 (7)	0.0623 (8)	0.0008 (6)	0.0155 (6)	0.0011 (6)
C11	0.0533 (8)	0.0432 (8)	0.069 (1)	−0.0039 (7)	0.0022 (8)	0.0013 (7)

Geometric parameters (Å, °)

O1—N2	1.228 (2)	C5—C6	1.372 (2)
O2—N2	1.227 (2)	C5—H5	0.94 (2)
O3—C11	1.428 (2)	C6—C7	1.405 (2)
O3—H3O	0.87 (2)	C6—H6	0.94 (2)
N1—C1	1.378 (2)	C7—C8	1.376 (2)
N1—C4	1.358 (2)	C8—H8	0.99 (2)
N1—H1N	0.89 (2)	C9—C10	1.525 (2)
N2—C7	1.451 (2)	C9—H9a	0.98 (2)
C1—C2	1.359 (2)	C9—H9b	0.98 (2)
C1—H1	0.99 (2)	C10—C11	1.492 (2)
C2—C3	1.438 (2)	C10—H10a	1.04 (2)
C2—C9	1.494 (2)	C10—H10b	1.01 (2)
C3—C4	1.417 (2)	C11—H11a	1.03 (2)
C3—C8	1.390 (2)	C11—H11b	0.98 (2)
C4—C5	1.398 (2)		
C11—O3—H3O	114 (1)	N2—C7—C6	118.5 (1)
C1—N1—C4	108.9 (1)	N2—C7—C8	118.1 (1)
C1—N1—H1N	126 (1)	C6—C7—C8	123.3 (1)
C4—N1—H1N	125 (1)	C3—C8—C7	117.7 (1)
O1—N2—O2	122.0 (1)	C3—C8—H8	123.2 (9)
O1—N2—C7	118.4 (1)	C7—C8—H8	119.1 (9)
O2—N2—C7	119.6 (1)	C2—C9—C10	114.1 (1)
N1—C1—C2	110.9 (1)	C2—C9—H9a	109 (1)
N1—C1—H1	122 (1)	C2—C9—H9b	109.8 (9)
C2—C1—H1	127 (1)	C10—C9—H9a	109 (1)
C1—C2—C3	105.5 (1)	C10—C9—H9b	108.1 (9)
C1—C2—C9	128.2 (1)	H9a—C9—H9b	106 (1)
C3—C2—C9	126.3 (1)	C9—C10—C11	113.5 (1)
C2—C3—C4	107.4 (1)	C9—C10—H10a	111.6 (8)
C2—C3—C8	133.4 (1)	C9—C10—H10b	108.4 (9)
C4—C3—C8	119.2 (1)	C11—C10—H10a	107.8 (8)
N1—C4—C3	107.4 (1)	C11—C10—H10b	108.9 (9)
N1—C4—C5	130.4 (1)	H10a—C10—H10b	106 (1)
C3—C4—C5	122.2 (1)	O3—C11—C10	109.1 (1)
C4—C5—C6	117.9 (1)	O3—C11—H11a	110 (1)
C4—C5—H5	118 (1)	O3—C11—H11b	107 (1)
C6—C5—H5	123 (1)	C10—C11—H11a	110 (1)
C5—C6—C7	119.7 (1)	C10—C11—H11b	112.5 (8)
C5—C6—H6	119 (1)	H11a—C11—H11b	107 (1)
C7—C6—H6	121 (1)		
H3O—O3—C11—C10	−144 (1)	C2—C3—C4—N1	0.1 (2)
C4—N1—C1—C2	−0.5 (2)	C2—C3—C4—C5	−179.7 (1)
C1—N1—C4—C3	0.2 (2)	C8—C3—C4—N1	179.2 (1)
C1—N1—C4—C5	180.0 (3)	C8—C3—C4—C5	−0.6 (2)
O1—N2—C7—C6	175.5 (1)	C2—C3—C8—C7	179.0 (1)
O1—N2—C7—C8	−5.4 (2)	C4—C3—C8—C7	0.3 (2)

O2—N2—C7—C6	−5.1 (2)	N1—C4—C5—C6	−179.3 (2)
O2—N2—C7—C8	174.0 (1)	C3—C4—C5—C6	0.5 (2)
N1—C1—C2—C3	0.6 (2)	C4—C5—C6—C7	−0.0 (3)
N1—C1—C2—C9	179.9 (2)	C5—C6—C7—N2	178.7 (1)
C1—C2—C3—C4	−0.4 (2)	C5—C6—C7—C8	−0.3 (2)
C1—C2—C3—C8	−179.3 (2)	N2—C7—C8—C3	−178.9 (1)
C9—C2—C3—C4	−179.8 (1)	C6—C7—C8—C3	0.2 (2)
C9—C2—C3—C8	1.3 (3)	C2—C9—C10—C11	66.8 (2)
C1—C2—C9—C10	−101.1 (2)	C9—C10—C11—O3	65.3 (2)
C3—C2—C9—C10	78.1 (2)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3O···O1^{i}	0.87 (2)	1.97 (2)	2.807 (2)	161 (2)

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