Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry

A concise approach to the spiroiminal fragment of marineosins

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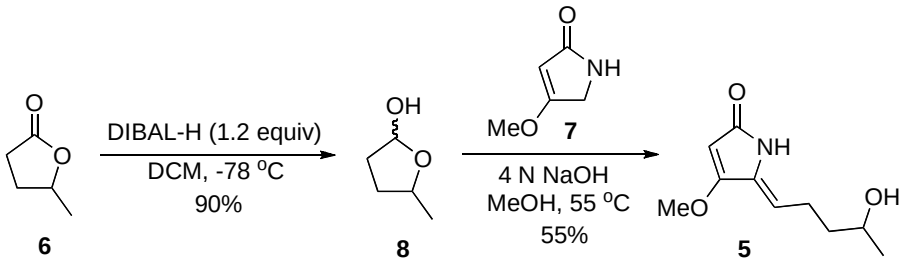
Supporting Information

General Procedures. All commercially available reagents were used without further purification unless otherwise stated. All dry solvents were freshly distilled under nitrogen from appropriate drying agents. CH_2Cl_2 was distilled from CaH_2 and pyrrole was distilled under N_2. Column chromatography was performed on silica gel (200-300 mesh). ^{1}H NMR spectra were recorded on a 400 MHz NMR spectrometer and ^{13}C NMR spectra were recorded on a 100 MHz NMR spectrometer. IR spectra were recorded on a FT-IR spectrometer. Melting points were uncorrected.

Synthesis of Compound 5



To a solution of γ-valerolactone **(6)** (7.51 g, 75.0 mmol) in dry DCM (200 mL) at -78 °C under N_2 was added dropwise a solution of diisobutylaluminum hydride (69.2 mL, 90.0 mmol, 1.3 M in hexanes) via syringe pump over 2 h. Upon stirring at -78 °C for 1 h, the reaction mixture was quenched with MeOH (20 mL) and a saturated solution of Rochelle's salt (200 mL) at -78 °C, stirred at rt for 2 h, and filtered through a Celite pad. The filtrate was extracted with DCM (3 x 300 mL), washed with brine (3 x 100 mL), and concentrated to give compound **8** as a light yellow oil (6.92 g, 90%), which was used directly without further purification.

L. A. Paquette, J. C. Lanter and H-L. Wang, *J. Org. Chem.,* 1996, **61**, 1119.

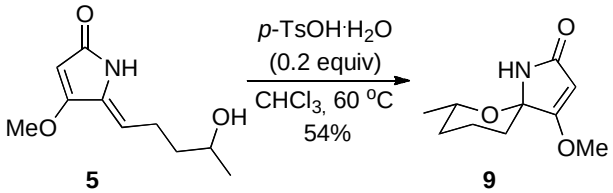
To a solution of compound **7** (8.40 g, 74.4 mmol) in 4 N NaOH (110 mL) at 55 °C was added dropwise a solution of **8** (6.90 g, 67.6 mol) in MeOH (110 mL). The resulting mixture was stirred at 55 °C overnight, cooled to rt, concentrated, and stored in a refrigerator (0 °C) for 24 h. The resulting precipitate was filtered and washed with water to give compound **5** as a white solid (7.28 g, 55 %). R_f = 0.24 (silica gel, EtOAc); mp. 84-86 °C;

IR (film): 3355, 1664, 1598 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz) δ 9.64 (s, 1H), 5.52 (t, J = 8.8 Hz, 1H), 5.11 (s, 1H), 3.81 (s, 3H), 3.84-3.73 (m, 1H), 3.47 (s, 1H), 2.46-2.30 (m, 2H), 1.65-1.53 (m, 2H), 1.18 (d, J = 6.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.3, 166.5, 134.2, 111.5, 92.9, 66.7, 58.2, 38.5, 23.8, 23.4; HRMS Calcd for $C_{10}H_{15}NO_3$ (M^+): 197.1052; Found: 197.1054.

(*a*) T. M. Visp, US4983743., 1991.

(*b*) M. Bänziger, J. F McGarrity, and T. Meul, *J. Org. Chem.*, 1993, **58**, 4010.

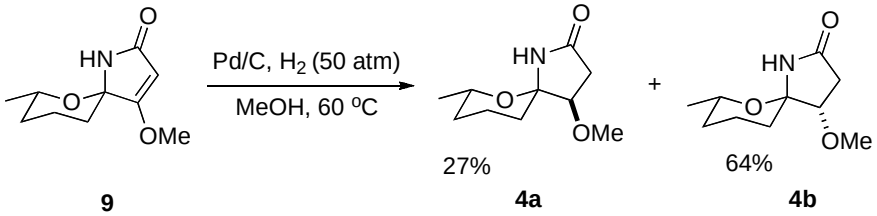
Synthesis of Compound 9



5 **9**

A solution of compound **5** (0.197 g, 1.00 mmol) and *p*-TsOH·H_2O (0.038 g. 0.20 mmol) in $CHCl_3$ (16 mL) was stirred at 60 °C for 24 h, cooled to rt, quenched with NEt_3 (0.5 mL), concentrated, and purified by flash column chromatography (silica gel, eluent: EtOAc) to give compound **9** as a white solid (0.106 g, 54%). R_f = 0.36 (silica gel, EtOAc); mp. 177-180 °C; IR (film): 3232, 1676, 1597 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz) δ 7.70 (br, 1H), 4.96 (s, 1H), 3.85 (s, 3H), 3.83-3.74 (m, 1H), 2.04-1.87 (m, 2H), 1.83-1.66 (m, 2H), 1.60-1.53 (m, 1H), 1.40-1.28 (m, 1H), 1.21 (d, J = 6.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.8, 174.0, 92.6, 88.3, 70.7, 58.8, 32.1, 30.9, 22.2, 20.0; HRMS Calcd for $C_{10}H_{15}NO_3$ (M^+): 197.1052; Found: 197.1055.

Synthesis of Compound 4a and 4b



9 **4a** **4b**

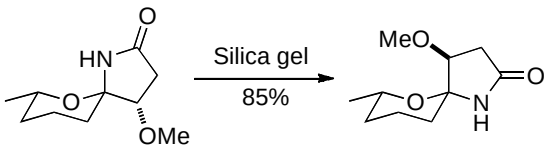
A mixture of compound **9** (0.099 g, 0.50 mmol) and 10% Pd/C (50% H_2O) (0.02 g) in

MeOH (3 mL) was stirred under H_2 atmosphere (50 atm) at 60 ^oC for 24 h, cooled to rt, filtered through a basic alumina pad, concentrated, and purified by flash column chromatography (silica gel, eluent: PE/EtOAc/NEt_3 = 2/1/0.01 to 1/1/0.01) to give compound **4a** (0.0271 g, 27%) and **4b** (0.0635 g, 64%).

4a: white solid; R_f = 0.43 (silica gel, PE/EtOAc = 1/2); mp. 146-149 ^oC; IR (film): 3351, 3183, 1710 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz) δ 7.69 (br, 1H), 3.71-3.61 (m, 2H), 3.36 (s, 3H), 2.74 (dd, *J* = 17.2, 5.6 Hz, 1H), 2.30 (dd, *J* = 17.2, 2.0 Hz, 1H), 1.89-1.80 (m, 2H), 1.73-1.51 (m, 3H), 1.31-1.19 (m, 1H), 1.12 (d, *J* = 6.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 177.5, 92.5, 84.0, 68.0, 57.7, 36.1, 32.9, 29.1, 22.2, 19.8; HRMS Calcd for $C_{10}H_{17}NO_3$ (M^+): 199.1208; Found: 199.1211.

4b: white solid; R_f = 0.17 (silica gel, PE/EtOAc = 1/2); mp. 148-152 ^oC; IR (film): 3355, 3184, 1681 cm^{-1}; ^1H NMR (MeOD, 400 MHz) δ 3.81-3.68 (m, 2H), 3.45 (s, 3H), 2.58 (dd, *J* = 16.0, 7.2 Hz, 1H), 2.44 (dd, *J* = 16.0, 9.2 Hz, 1H), 1.97-1.87 (m, 1H), 1.86-1.72 (m, 2H), 1.66-1.56 (m, 2H), 1.33-1.20 (m, 1H), 1.16 (d, *J* = 6.4 Hz, 3H); ^{13}C NMR (100 MHz, MeOD) δ 177.1, 90.2, 83.1, 69.1, 58.6, 36.0, 33.4, 33.0, 22.1, 20.8; HRMS Calcd for $C_{10}H_{17}NO_3$ (M^+): 199.1208; Found: 199.1210.

Synthesis of Compound 4c

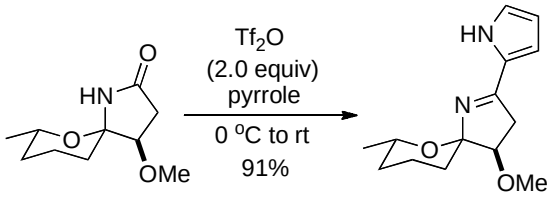


4b **4c**

Compound **4b** (0.030 g, 0.15 mmol) was loaded onto a silica gel column and eluted slowly with PE/EtOAc = 1/1 to give compound **4c** as a white solid (0.0255 g, 85%). R_f = 0.4 (silica gel, EtOAc); mp. 163-167 ^oC; IR (film): 3205, 2933, 1709 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz) δ 5.78 (s, 1H), 3.98 (d, *J* = 5.6 Hz, 1H), 3.78-3.68 (m, 1H), 3.34 (s, 3H), 2.69 (dd, *J* = 17.2, 5.6 Hz, 1H), 2.36-2.29 (m, 1H), 2.27-2.19 (m, 1H), 1.82-1.73 (m, 1H), 1.65-1.50 (m, 3H), 1.39-1.28 (m, 1H), 1.17 (d, *J* = 6.4 Hz, 3H); ^{13}C NMR (100 MHz,

$CDCl_3$) δ 176.0, 92.1, 77.3, 70.6, 56.9, 35.3, 32.1, 28.9, 22.3, 20.7; HRMS Calcd for $C_{10}H_{18}NO_3$ (M+H): 200.1281; Found: 200.1283.

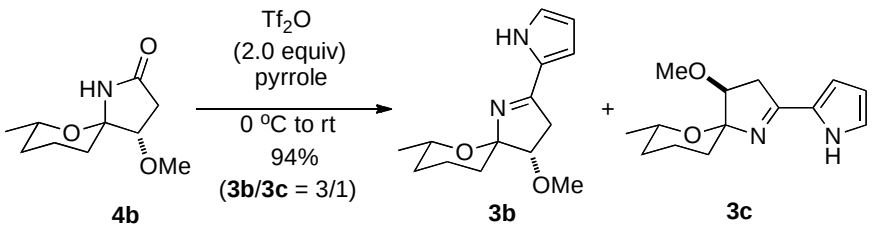
Synthesis of Compound 3a



4a **3a**

To a solution of compound **4a** (0.0200 g, 0.10 mmol) in freshly distilled pyrrole (1 mL) under N_2 at 0 °C was added dropwise Tf_2O (0.0336 mL, 0.0564 g, 0.20 mmol). Upon stirring at rt for 3 h, the reaction mixture was quenched with 1 N NaOH (1 mL), extracted with DCM (3 x 10 mL), washed with water (3 x 5 mL) and brine (3 x 5 mL), dried over Na_2SO_4, filtered, concentrated, and purified by flash column chromatography (silica gel, eluent: PE/EtOAc = 4/1) to give compound **3a** as a light yellow solid (0.0226 g, 91%). R_f = 0.2 (silica gel, PE/EtOAc = 4/1); mp. 68-70 °C; IR (film): 3133, 1606 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz) δ 6.89 (dd, J = 2.4, 1.2 Hz, 1H), 6.55 (dd, J = 3.6, 1.6 Hz, 1H), 6.21 (dd, J = 3.6, 2.8 Hz, 1H), 4.27-4.18 (m, 1H), 3.83 (dd, J = 7.2, 6.4 Hz, 1H), 3.44 (s, 3H), 3.20 (dd, J = 16.4, 6.8 Hz, 1H), 2.74 (dd, J = 16.4, 5.6 Hz, 1H), 2.07-1.92 (m, 1H), 1.83-1.68 (m, 2H), 1.68-1.60 (m, 1H), 1.60-1.52 (m, 1H), 1.38-1.23 (m, 1H), 1.10 (d, J = 6.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.4, 128.0, 122.5, 113.9, 109.9, 103.7, 87.2, 68.7, 58.3, 38.8, 33.7, 29.0, 22.6, 20.0; HRMS Calcd for $C_{14}H_{20}N_2O_2$ (M^+): 248.1525; Found: 248.1528.

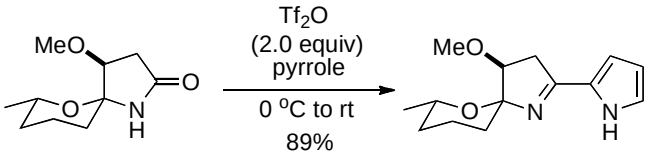
Synthesis of Compound 3b and 3c



4b **3b** **3c**

To a solution of compound **4b** (0.0200 g, 0.10 mmol) in freshly distilled pyrrole (1 mL) under N_2 at 0 ^oC was added dropwise Tf_2O (0.0336 mL, 0.0564 g, 0.20 mmol). Upon stirring at rt for 3 h, the reaction mixture was quenched with 2 N NaOH (2 mL), extracted with DCM (3 x 10 mL), washed with water (3 x 5 mL) and brine (3 x 5 mL), dried over Na_2SO_4, filtered, concentrated to give a mixture of **3b** and **3c** (3:1 as judged by ^1H NMR), which was purified by repeated flash column chromatography (silica gel, eluent: PE/EtOAc/NEt_3 = 4/1/0.01) to give compound **3b** (0.0167 g, 67%) and **3c** (0.0027 g, 11%) along with a mixture of **3b** and **3c** (0.0039 g, 16%) (94% combined yield).

Synthesis of Compound 3c



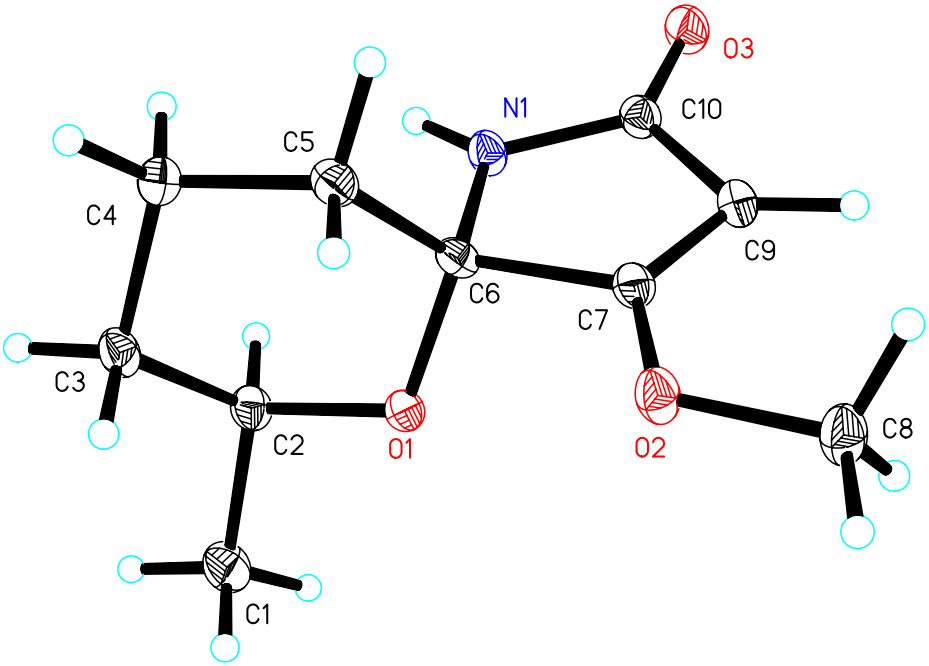
4c **3c**

To a solution of compound **4c** (0.0150 g, 0.075 mmol) in freshly distilled pyrrole (0.75 mL) under N_2 at 0 ^oC was added dropwise Tf_2O (0.0255 mL, 0.0425 g, 0.15 mmol). Upon stirring at rt for 3 h, the reaction mixture was quenched with 1 N NaOH (2 mL), extracted with DCM (3 x 20 mL), washed with water (3 x 10 mL) and brine (3 x 10 mL), dried over Na_2SO_4, filtered, concentrated, and purified by flash column chromatography (silica gel, eluent: PE/EtOAc = 4/1) to give compound **3c** (0.0166 g, 89%).

3b: light yellow solid; R_f = 0.2 (silica gel, PE/EtOAc = 1/2); mp. 120-123 ^oC; IR (film): 3261, 1609 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz) δ 6.92 (dd, *J* = 2.4, 1.2 Hz, 1H), 6.55 (dd, *J* = 3.6, 1.2 Hz, 1H), 6.23 (dd, *J* = 3.6, 2.8 Hz, 1H), 4.33-4.24 (m, 1H), 3.70 (dd, *J* = 6.0, 4.4 Hz, 1H), 3.49 (s, 3H), 3.03 (dd, *J* = 16.4, 6.0 Hz, 1H), 2.96 (dd, *J* = 16.4, 4.4 Hz, 1H), 2.11-1.96 (m, 1H), 1.82-1.63 (m, 3H), 1.50-1.29 (m, 2H), 1.19 (d, *J* = 6.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.5, 128.1, 122.2, 113.7, 110.1, 101.1, 85.6, 68.6, 59.0, 39.0, 34.6, 33.4, 22.5, 20.6; HRMS Calcd for $C_{14}H_{20}N_2O_2$ (M^+): 248.1525; Found: 248.1528.

3c: light yellow solid; R_f = 0.3 (silica gel, PE/EtOAc = 1/2); mp. 149-152 ^{o}C; IR (film): 3125, 1612 cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 6.93 (dd, *J* = 2.4, 1.2 Hz, 1H), 6.56 (dd, *J* = 3.6, 1.2 Hz, 1H), 6.22 (dd, *J* = 3.2, 2.4 Hz, 1H), 4.07 (dd, *J* = 6.4, 3.2 Hz, 1H), 3.82-3.72 (m, 1H), 3.38 (s, 3H), 3.25 (dd, *J* = 17.2, 6.4 Hz, 1H), 2.86 (dd, *J* = 17.2, 3.6 Hz, 1H), 2.06-2.00 (m, 1H), 1.82-1.69 (m, 3H), 1.64-1.57 (m, 1H), 1.40-1.29 (m, 1H), 1.21 (d, *J* = 6.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.7, 127.4, 122.8, 114.3, 109.9, 105.3, 83.3, 70.3, 57.7, 39.7, 32.6, 29.4, 22.6, 21.0; HRMS Calcd for $C_{14}H_{20}N_2O_2$ (M^+): 248.1525; Found: 248.1528.

The X-ray structure of compound 9



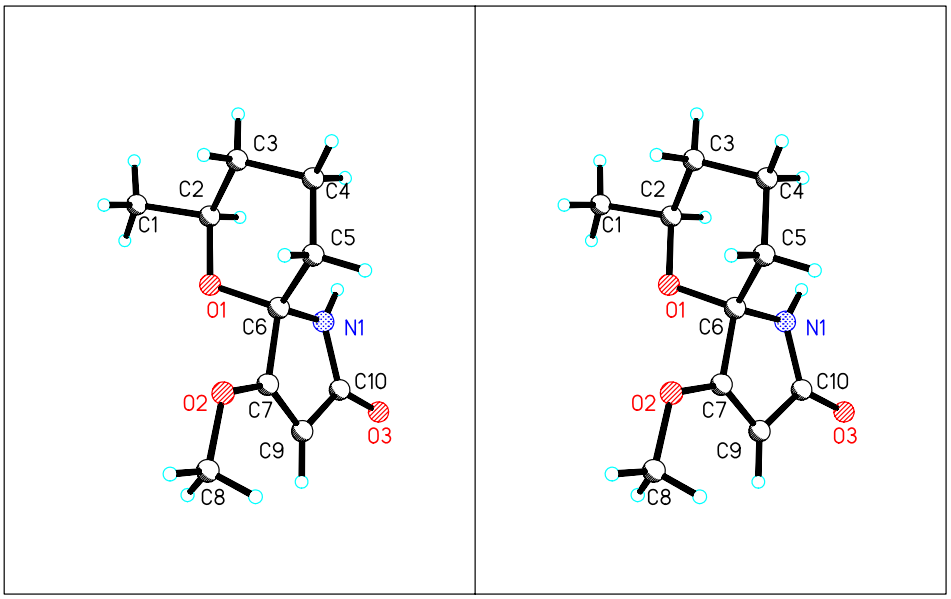


Table 1. Crystal data and structure refinement for A.

Identification code	a
Empirical formula	C10 H15 N O3
Formula weight	197.23
Temperature	173(2) K
Wavelength	0.71073 A
Crystal system, space group	Monoclinic, P2(1)/n
Unit cell dimensions	a = 7.6770(15) A alpha = 90 deg.
	b = 14.890(3) A beta = 105.14(3) deg.
	c = 8.9195(18) A gamma = 90 deg.
Volume	984.2(3) A^3
Z, Calculated density	4, 1.331 Mg/m^3
Absorption coefficient	0.098 mm^-1
F(000)	424
Crystal size	0.26 x 0.15 x 0.12 mm
Theta range for data collection	2.73 to 27.50 deg.
Limiting indices	-9<=h<=9, -19<=k<=19, -11<=l<=11
Reflections collected / unique	6514 / 2255 [R(int) = 0.0471]
Completeness to theta = 27.50	99.6 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	1.0000 and 0.7155
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2255 / 0 / 129
Goodness-of-fit on F^2	1.166
Final R indices [I>2sigma(I)]	R1 = 0.0615, wR2 = 0.1283
R indices (all data)	R1 = 0.0695, wR2 = 0.1326
Largest diff. peak and hole	0.359 and -0.331 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for A.
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
O(1)	9791(2)	8851(1)	2477(1)	23(1)
O(2)	8764(2)	8632(1)	5256(2)	28(1)
O(3)	5103(2)	10697(1)	1766(2)	31(1)
N(1)	6853(2)	9485(1)	1483(2)	24(1)
C(1)	12175(3)	8717(2)	1280(3)	35(1)
C(2)	10170(3)	8587(1)	1037(2)	24(1)
C(3)	9542(3)	7628(1)	635(2)	26(1)
C(4)	7546(3)	7533(1)	543(2)	27(1)
C(5)	7206(3)	7853(1)	2064(2)	25(1)
C(6)	7934(2)	8800(1)	2465(2)	21(1)
C(7)	7855(2)	9122(1)	4061(2)	22(1)
C(8)	8778(3)	8985(1)	6761(2)	30(1)
C(9)	6884(3)	9875(1)	3977(2)	24(1)
C(10)	6172(3)	10091(1)	2325(2)	24(1)

Table 3. Bond lengths [A] and angles [deg] for A.

O(1)-C(6)	1.425(2)
O(1)-C(2)	1.443(2)
O(2)-C(7)	1.330(2)
O(2)-C(8)	1.439(2)
O(3)-C(10)	1.234(2)
N(1)-C(10)	1.363(2)
N(1)-C(6)	1.455(2)
N(1)-H(1)	0.8800
C(1)-C(2)	1.510(3)
C(1)-H(1A)	0.9800
C(1)-H(1C)	0.9800
C(1)-H(1B)	0.9800
C(2)-C(3)	1.519(3)
C(2)-H(2)	1.0000
C(3)-C(4)	1.520(3)
C(3)-H(3B)	0.9900
C(3)-H(3A)	0.9900
C(4)-C(5)	1.524(3)
C(4)-H(4A)	0.9900
C(4)-H(4B)	0.9900
C(5)-C(6)	1.524(3)
C(5)-H(5B)	0.9900
C(5)-H(5A)	0.9900
C(6)-C(7)	1.518(2)
C(7)-C(9)	1.337(3)
C(8)-H(8B)	0.9800
C(8)-H(8A)	0.9800
C(8)-H(8C)	0.9800
C(9)-C(10)	1.467(3)
C(9)-H(9)	0.9500
C(6)-O(1)-C(2)	114.35(14)
C(7)-O(2)-C(8)	114.91(15)
C(10)-N(1)-C(6)	111.91(15)
C(10)-N(1)-H(1)	124.0
C(6)-N(1)-H(1)	124.0
C(2)-C(1)-H(1A)	109.5
C(2)-C(1)-H(1C)	109.5
H(1A)-C(1)-H(1C)	109.5
C(2)-C(1)-H(1B)	109.5

H(1A)-C(1)-H(1B)	109.5
H(1C)-C(1)-H(1B)	109.5
O(1)-C(2)-C(1)	105.78(15)
O(1)-C(2)-C(3)	109.95(15)
C(1)-C(2)-C(3)	113.78(17)
O(1)-C(2)-H(2)	109.1
C(1)-C(2)-H(2)	109.1
C(3)-C(2)-H(2)	109.1
C(2)-C(3)-C(4)	110.72(16)
C(2)-C(3)-H(3B)	109.5
C(4)-C(3)-H(3B)	109.5
C(2)-C(3)-H(3A)	109.5
C(4)-C(3)-H(3A)	109.5
H(3B)-C(3)-H(3A)	108.1
C(3)-C(4)-C(5)	109.13(16)
C(3)-C(4)-H(4A)	109.9
C(5)-C(4)-H(4A)	109.9
C(3)-C(4)-H(4B)	109.9
C(5)-C(4)-H(4B)	109.9
H(4A)-C(4)-H(4B)	108.3
C(4)-C(5)-C(6)	111.18(16)
C(4)-C(5)-H(5B)	109.4
C(6)-C(5)-H(5B)	109.4
C(4)-C(5)-H(5A)	109.4
C(6)-C(5)-H(5A)	109.4
H(5B)-C(5)-H(5A)	108.0
O(1)-C(6)-N(1)	112.28(15)
O(1)-C(6)-C(7)	105.13(14)
N(1)-C(6)-C(7)	100.75(14)
O(1)-C(6)-C(5)	110.87(14)
N(1)-C(6)-C(5)	112.97(15)
C(7)-C(6)-C(5)	114.24(15)
O(2)-C(7)-C(9)	132.34(18)
O(2)-C(7)-C(6)	115.87(15)
C(9)-C(7)-C(6)	111.77(16)
O(2)-C(8)-H(8B)	109.5
O(2)-C(8)-H(8A)	109.5
H(8B)-C(8)-H(8A)	109.5
O(2)-C(8)-H(8C)	109.5
H(8B)-C(8)-H(8C)	109.5
H(8A)-C(8)-H(8C)	109.5
C(7)-C(9)-C(10)	107.22(17)
C(7)-C(9)-H(9)	126.4
C(10)-C(9)-H(9)	126.4

O(3)-C(10)-N(1)	124.90(17)
O(3)-C(10)-C(9)	126.89(18)
N(1)-C(10)-C(9)	108.18(16)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for A.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
O(1)	20(1)	29(1)	20(1)	-3(1)	8(1)	0(1)
O(2)	35(1)	29(1)	20(1)	3(1)	10(1)	8(1)
O(3)	33(1)	28(1)	30(1)	0(1)	5(1)	11(1)
N(1)	26(1)	26(1)	19(1)	2(1)	7(1)	6(1)
C(1)	29(1)	44(1)	39(1)	-6(1)	18(1)	-2(1)
C(2)	25(1)	30(1)	21(1)	0(1)	11(1)	1(1)
C(3)	28(1)	29(1)	24(1)	-2(1)	12(1)	4(1)
C(4)	27(1)	24(1)	30(1)	-5(1)	7(1)	0(1)
C(5)	23(1)	25(1)	30(1)	-1(1)	10(1)	1(1)
C(6)	18(1)	25(1)	23(1)	2(1)	8(1)	3(1)
C(7)	22(1)	25(1)	21(1)	1(1)	9(1)	0(1)
C(8)	39(1)	31(1)	22(1)	0(1)	11(1)	4(1)
C(9)	26(1)	26(1)	24(1)	-2(1)	10(1)	2(1)
C(10)	24(1)	21(1)	28(1)	-2(1)	8(1)	0(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for A.

	x	y	z	U(eq)
H(1)	6662	9505	467	28
H(1A)	12830	8335	2137	53
H(1C)	12484	9348	1533	53
H(1B)	12511	8554	329	53
H(2)	9509	8996	189	29
H(3B)	10247	7215	1436	31
H(3A)	9759	7461	-375	31
H(4A)	7177	6897	358	33
H(4B)	6825	7895	-331	33
H(5B)	7796	7438	2910	30
H(5A)	5893	7846	1974	30
H(8B)	9525	8600	7569	45
H(8A)	7543	8999	6875	45
H(8C)	9275	9594	6863	45
H(9)	6691	10208	4828	29

Table 6. Torsion angles [deg] for A.

C(6)-O(1)-C(2)-C(1)	178.27(15)
C(6)-O(1)-C(2)-C(3)	-58.48(19)
O(1)-C(2)-C(3)-C(4)	56.8(2)
C(1)-C(2)-C(3)-C(4)	175.21(16)
C(2)-C(3)-C(4)-C(5)	-55.3(2)
C(3)-C(4)-C(5)-C(6)	53.8(2)
C(2)-O(1)-C(6)-N(1)	-70.23(19)
C(2)-O(1)-C(6)-C(7)	-178.87(14)
C(2)-O(1)-C(6)-C(5)	57.19(19)
C(10)-N(1)-C(6)-O(1)	-113.76(17)
C(10)-N(1)-C(6)-C(7)	-2.35(19)
C(10)-N(1)-C(6)-C(5)	119.95(18)
C(4)-C(5)-C(6)-O(1)	-54.2(2)
C(4)-C(5)-C(6)-N(1)	72.8(2)
C(4)-C(5)-C(6)-C(7)	-172.80(15)
C(8)-O(2)-C(7)-C(9)	-2.7(3)
C(8)-O(2)-C(7)-C(6)	175.73(16)
O(1)-C(6)-C(7)-O(2)	-62.19(19)
N(1)-C(6)-C(7)-O(2)	-179.01(15)
C(5)-C(6)-C(7)-O(2)	59.6(2)
O(1)-C(6)-C(7)-C(9)	116.54(17)
N(1)-C(6)-C(7)-C(9)	-0.3(2)
C(5)-C(6)-C(7)-C(9)	-121.69(18)
O(2)-C(7)-C(9)-C(10)	-178.99(19)
C(6)-C(7)-C(9)-C(10)	2.6(2)
C(6)-N(1)-C(10)-O(3)	-174.22(18)
C(6)-N(1)-C(10)-C(9)	4.0(2)
C(7)-C(9)-C(10)-O(3)	174.14(19)
C(7)-C(9)-C(10)-N(1)	-4.0(2)

Symmetry transformations used to generate equivalent atoms:

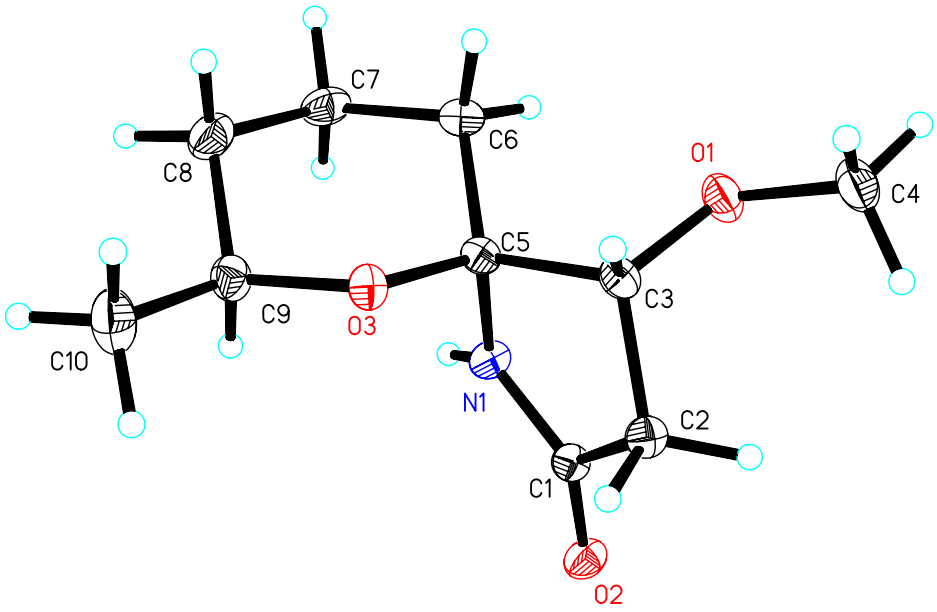
Table 7. Hydrogen bonds for A [A and deg.].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(1)-H(1)...O(3)#1	0.88	2.12	2.908(2)	149.0

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,-y+2,-z

The X-ray structure of compound 4a



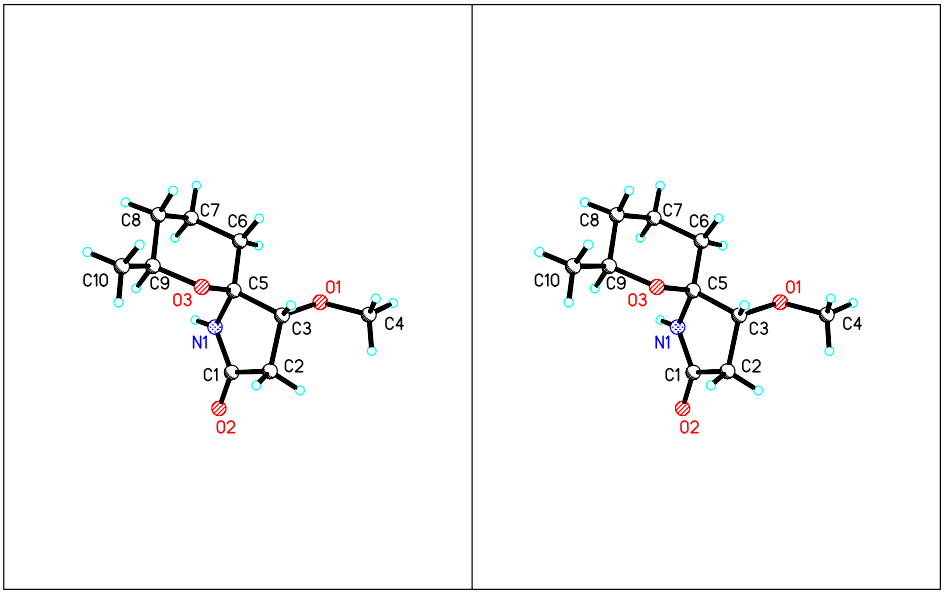


Table 1. Crystal data and structure refinement for A.

Identification code	a
Empirical formula	C10 H17 N O3
Formula weight	199.25
Temperature	173(2) K
Wavelength	0.71073 A
Crystal system, space group	Orthorhombic, Pbca
Unit cell dimensions	a = 13.826(3) A alpha = 90 deg.
	b = 9.5792(19) A beta = 90 deg.
	c = 16.063(3) A gamma = 90 deg.
Volume	2127.4(7) A^3
Z, Calculated density	8, 1.244 Mg/m^3
Absorption coefficient	0.091 mm^-1
F(000)	864
Crystal size	0.16 x 0.14 x 0.10 mm
Theta range for data collection	2.54 to 27.48 deg.
Limiting indices	-17<=h<=17, -12<=k<=9, -20<=l<=20
Reflections collected / unique	12526 / 2428 [R(int) = 0.0598]
Completeness to theta = 27.48	99.8 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	1.0000 and 0.6676
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2428 / 0 / 129
Goodness-of-fit on F^2	1.191
Final R indices [I>2sigma(I)]	R1 = 0.0610, wR2 = 0.1202
R indices (all data)	R1 = 0.0722, wR2 = 0.1253
Largest diff. peak and hole	0.227 and -0.168 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for A.
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
O(1)	-2114(1)	8186(1)	5951(1)	35(1)
O(2)	-488(1)	6154(1)	4161(1)	32(1)
O(3)	428(1)	8485(1)	6197(1)	26(1)
N(1)	-332(1)	6536(1)	5563(1)	25(1)
C(1)	-576(1)	6897(2)	4783(1)	25(1)
C(2)	-979(1)	8358(2)	4798(1)	26(1)
C(3)	-1174(1)	8626(2)	5721(1)	25(1)
C(4)	-2836(2)	9168(3)	5719(1)	46(1)
C(5)	-440(1)	7671(2)	6160(1)	23(1)
C(6)	-724(1)	7162(2)	7019(1)	30(1)
C(7)	107(2)	6389(2)	7440(1)	36(1)
C(8)	1004(2)	7300(2)	7436(1)	38(1)
C(9)	1244(1)	7771(2)	6561(1)	30(1)
C(10)	2081(2)	8782(2)	6517(2)	47(1)

Table 3. Bond lengths [A] and angles [deg] for A.

O(1)-C(3)	1.416(2)
O(1)-C(4)	1.421(2)
O(2)-C(1)	1.233(2)
O(3)-C(5)	1.432(2)
O(3)-C(9)	1.443(2)
N(1)-C(1)	1.342(2)
N(1)-C(5)	1.458(2)
N(1)-H(1)	0.8800
C(1)-C(2)	1.507(2)
C(2)-C(3)	1.528(2)
C(2)-H(2A)	0.9900
C(2)-H(2B)	0.9900
C(3)-C(5)	1.538(2)
C(3)-H(3)	1.0000
C(4)-H(4A)	0.9800
C(4)-H(4B)	0.9800
C(4)-H(4C)	0.9800
C(5)-C(6)	1.515(2)
C(6)-C(7)	1.525(3)
C(6)-H(6A)	0.9900
C(6)-H(6B)	0.9900
C(7)-C(8)	1.517(3)
C(7)-H(7B)	0.9900
C(7)-H(7A)	0.9900
C(8)-C(9)	1.513(3)
C(8)-H(8A)	0.9900
C(8)-H(8B)	0.9900
C(9)-C(10)	1.510(3)
C(9)-H(9)	1.0000
C(10)-H(10A)	0.9800
C(10)-H(10C)	0.9800
C(10)-H(10B)	0.9800
C(3)-O(1)-C(4)	112.32(15)
C(5)-O(3)-C(9)	114.43(13)
C(1)-N(1)-C(5)	113.37(14)
C(1)-N(1)-H(1)	123.3
C(5)-N(1)-H(1)	123.3
O(2)-C(1)-N(1)	125.64(16)
O(2)-C(1)-C(2)	125.86(16)

N(1)-C(1)-C(2)	108.50(14)
C(1)-C(2)-C(3)	103.70(14)
C(1)-C(2)-H(2A)	111.0
C(3)-C(2)-H(2A)	111.0
C(1)-C(2)-H(2B)	111.0
C(3)-C(2)-H(2B)	111.0
H(2A)-C(2)-H(2B)	109.0
O(1)-C(3)-C(2)	111.40(14)
O(1)-C(3)-C(5)	108.01(13)
C(2)-C(3)-C(5)	103.24(13)
O(1)-C(3)-H(3)	111.3
C(2)-C(3)-H(3)	111.3
C(5)-C(3)-H(3)	111.3
O(1)-C(4)-H(4A)	109.5
O(1)-C(4)-H(4B)	109.5
H(4A)-C(4)-H(4B)	109.5
O(1)-C(4)-H(4C)	109.5
H(4A)-C(4)-H(4C)	109.5
H(4B)-C(4)-H(4C)	109.5
O(3)-C(5)-N(1)	110.32(13)
O(3)-C(5)-C(6)	110.80(14)
N(1)-C(5)-C(6)	112.74(14)
O(3)-C(5)-C(3)	104.35(13)
N(1)-C(5)-C(3)	102.04(13)
C(6)-C(5)-C(3)	115.98(14)
C(5)-C(6)-C(7)	111.37(15)
C(5)-C(6)-H(6A)	109.4
C(7)-C(6)-H(6A)	109.4
C(5)-C(6)-H(6B)	109.4
C(7)-C(6)-H(6B)	109.4
H(6A)-C(6)-H(6B)	108.0
C(8)-C(7)-C(6)	109.53(15)
C(8)-C(7)-H(7B)	109.8
C(6)-C(7)-H(7B)	109.8
C(8)-C(7)-H(7A)	109.8
C(6)-C(7)-H(7A)	109.8
H(7B)-C(7)-H(7A)	108.2
C(9)-C(8)-C(7)	110.80(16)
C(9)-C(8)-H(8A)	109.5
C(7)-C(8)-H(8A)	109.5
C(9)-C(8)-H(8B)	109.5
C(7)-C(8)-H(8B)	109.5
H(8A)-C(8)-H(8B)	108.1
O(3)-C(9)-C(10)	106.04(15)

O(3)-C(9)-C(8)	110.29(15)
C(10)-C(9)-C(8)	113.75(17)
O(3)-C(9)-H(9)	108.9
C(10)-C(9)-H(9)	108.9
C(8)-C(9)-H(9)	108.9
C(9)-C(10)-H(10A)	109.5
C(9)-C(10)-H(10C)	109.5
H(10A)-C(10)-H(10C)	109.5
C(9)-C(10)-H(10B)	109.5
H(10A)-C(10)-H(10B)	109.5
H(10C)-C(10)-H(10B)	109.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for A.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
O(1)	24(1)	41(1)	41(1)	9(1)	4(1)	3(1)
O(2)	40(1)	29(1)	26(1)	-4(1)	-3(1)	8(1)
O(3)	25(1)	23(1)	31(1)	0(1)	-2(1)	0(1)
N(1)	34(1)	17(1)	24(1)	0(1)	-1(1)	4(1)
C(1)	26(1)	23(1)	25(1)	1(1)	1(1)	1(1)
C(2)	30(1)	21(1)	28(1)	2(1)	0(1)	3(1)
C(3)	24(1)	22(1)	30(1)	0(1)	3(1)	1(1)
C(4)	30(1)	60(2)	47(1)	12(1)	6(1)	16(1)
C(5)	25(1)	19(1)	26(1)	-2(1)	2(1)	1(1)
C(6)	35(1)	29(1)	26(1)	-1(1)	5(1)	-1(1)
C(7)	48(1)	34(1)	24(1)	4(1)	1(1)	5(1)
C(8)	42(1)	41(1)	30(1)	0(1)	-8(1)	7(1)
C(9)	27(1)	29(1)	34(1)	1(1)	-3(1)	6(1)
C(10)	32(1)	51(1)	60(1)	9(1)	-9(1)	0(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for A.

	x	y	z	U(eq)
H(1)	-127	5697	5699	30
H(2A)	-1583	8419	4469	31
H(2B)	-505	9036	4574	31
H(3)	-1065	9628	5867	30
H(4A)	-2685	10078	5965	69
H(4B)	-3468	8849	5921	69
H(4C)	-2854	9252	5111	69
H(6A)	-1289	6532	6973	36
H(6B)	-915	7970	7367	36
H(7B)	-72	6156	8020	43
H(7A)	239	5506	7140	43
H(8A)	896	8127	7793	45
H(8B)	1557	6769	7667	45
H(9)	1400	6933	6215	36
H(10A)	2658	8348	6758	71
H(10C)	1920	9629	6829	71
H(10B)	2207	9024	5934	71

Table 6. Torsion angles [deg] for A.

C(5)-N(1)-C(1)-O(2)	175.02(17)
C(5)-N(1)-C(1)-C(2)	-5.3(2)
O(2)-C(1)-C(2)-C(3)	165.30(17)
N(1)-C(1)-C(2)-C(3)	-14.38(19)
C(4)-O(1)-C(3)-C(2)	-79.45(19)
C(4)-O(1)-C(3)-C(5)	167.84(15)
C(1)-C(2)-C(3)-O(1)	-88.80(16)
C(1)-C(2)-C(3)-C(5)	26.89(17)
C(9)-O(3)-C(5)-N(1)	-68.72(17)
C(9)-O(3)-C(5)-C(6)	56.84(18)
C(9)-O(3)-C(5)-C(3)	-177.65(13)
C(1)-N(1)-C(5)-O(3)	-88.06(17)
C(1)-N(1)-C(5)-C(6)	147.48(15)
C(1)-N(1)-C(5)-C(3)	22.38(18)
O(1)-C(3)-C(5)-O(3)	-156.38(13)
C(2)-C(3)-C(5)-O(3)	85.54(15)
O(1)-C(3)-C(5)-N(1)	88.72(15)
C(2)-C(3)-C(5)-N(1)	-29.36(16)
O(1)-C(3)-C(5)-C(6)	-34.2(2)
C(2)-C(3)-C(5)-C(6)	-152.28(15)
O(3)-C(5)-C(6)-C(7)	-53.83(19)
N(1)-C(5)-C(6)-C(7)	70.37(19)
C(3)-C(5)-C(6)-C(7)	-172.52(15)
C(5)-C(6)-C(7)-C(8)	53.4(2)
C(6)-C(7)-C(8)-C(9)	-54.6(2)
C(5)-O(3)-C(9)-C(10)	178.34(15)
C(5)-O(3)-C(9)-C(8)	-58.08(19)
C(7)-C(8)-C(9)-O(3)	56.2(2)
C(7)-C(8)-C(9)-C(10)	175.14(16)

Symmetry transformations used to generate equivalent atoms:

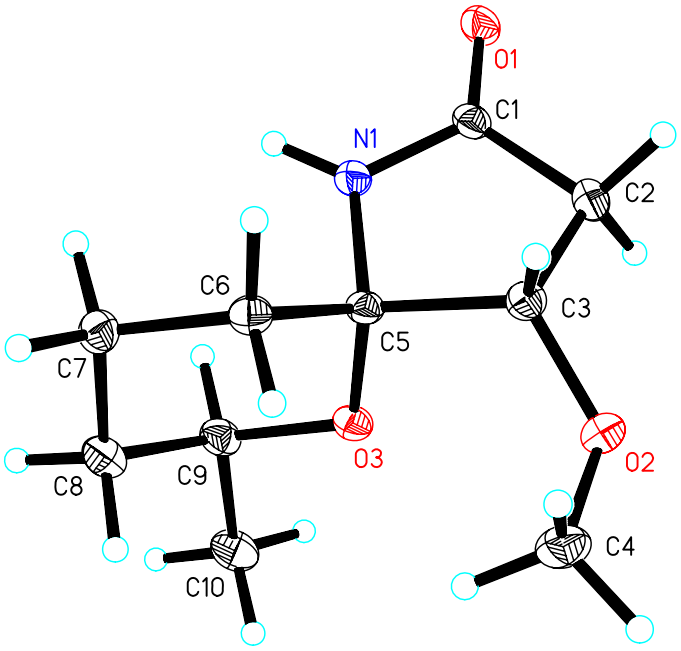
Table 7. Hydrogen bonds for A [A and deg.].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(1)-H(1)...O(2)#1	0.88	1.98	2.8495(19)	170.0

Symmetry transformations used to generate equivalent atoms:

#1 -x,-y+1,-z+1

The X-ray structure of compound 4b



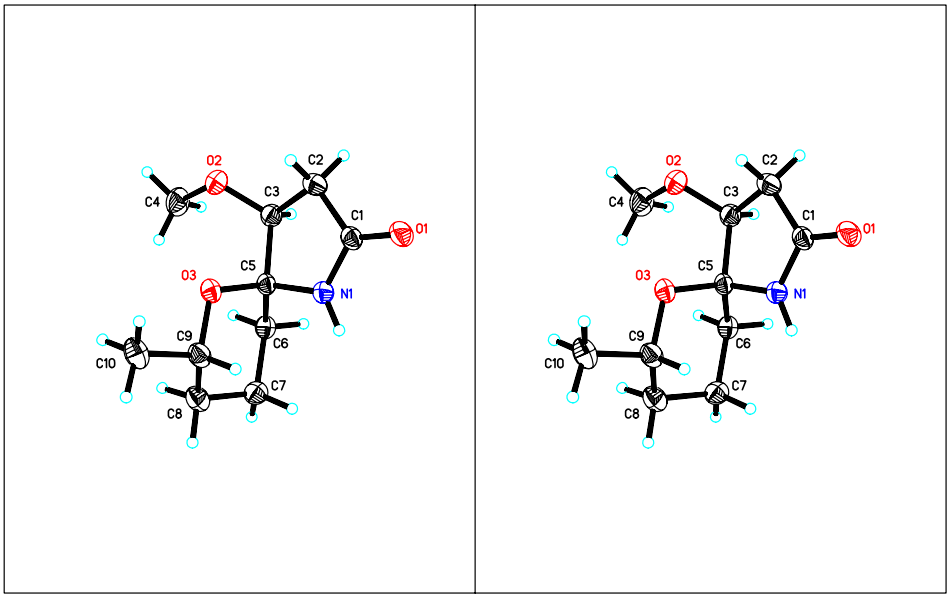


Table 1. Crystal data and structure refinement for A.

Identification code	a
Empirical formula	C10 H17 N O3
Formula weight	199.25
Temperature	173(2) K
Wavelength	0.71073 A
Crystal system, space group	Monoclinic, C2/c
Unit cell dimensions	a = 25.951(5) A alpha = 90 deg.
	b = 5.6442(11) A beta = 113.62(3) deg.
	c = 15.203(3) A gamma = 90 deg.
Volume	2040.3(7) A^3
Z, Calculated density	8, 1.297 Mg/m^3
Absorption coefficient	0.095 mm^-1
F(000)	864
Crystal size	0.23 x 0.13 x 0.09 mm
Theta range for data collection	2.73 to 27.50 deg.
Limiting indices	-26<=h<=33, -7<=k<=7, -19<=l<=19
Reflections collected / unique	7381 / 2343 [R(int) = 0.0595]
Completeness to theta = 27.50	99.9 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	1.0000 and 0.6611
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2343 / 0 / 129
Goodness-of-fit on F^2	1.233
Final R indices [I>2sigma(I)]	R1 = 0.0833, wR2 = 0.1619
R indices (all data)	R1 = 0.0947, wR2 = 0.1678
Largest diff. peak and hole	0.275 and -0.230 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for A.
U(eq) is defined as one third of the trace of the orthogonalized
Uij tensor.

	x	y	z	U(eq)
O(1)	2418(1)	12839(3)	1120(1)	30(1)
O(2)	3922(1)	8093(3)	3098(1)	32(1)
O(3)	4009(1)	9858(3)	1531(1)	24(1)
N(1)	3015(1)	10312(4)	814(1)	24(1)
C(1)	2793(1)	11335(4)	1382(2)	24(1)
C(2)	3080(1)	10342(5)	2375(2)	28(1)
C(3)	3418(1)	8242(5)	2267(2)	25(1)
C(4)	4251(1)	6044(5)	3141(2)	37(1)
C(5)	3483(1)	8705(4)	1302(2)	21(1)
C(6)	3438(1)	6479(4)	708(2)	25(1)
C(7)	3544(1)	7007(5)	-190(2)	28(1)
C(8)	4109(1)	8284(5)	104(2)	31(1)
C(9)	4124(1)	10459(5)	698(2)	27(1)
C(10)	4691(1)	11685(5)	1093(2)	37(1)

Table 3. Bond lengths [A] and angles [deg] for A.

O(1)-C(1)	1.232(3)
O(2)-C(3)	1.410(3)
O(2)-C(4)	1.424(3)
O(3)-C(5)	1.424(3)
O(3)-C(9)	1.452(3)
N(1)-C(1)	1.344(3)
N(1)-C(5)	1.458(3)
N(1)-H(1)	0.8800
C(1)-C(2)	1.498(3)
C(2)-C(3)	1.523(3)
C(2)-H(2A)	0.9900
C(2)-H(2B)	0.9900
C(3)-C(5)	1.565(3)
C(3)-H(3)	1.0000
C(4)-H(4B)	0.9800
C(4)-H(4A)	0.9800
C(4)-H(4C)	0.9800
C(5)-C(6)	1.525(3)
C(6)-C(7)	1.526(3)
C(6)-H(6A)	0.9900
C(6)-H(6B)	0.9900
C(7)-C(8)	1.529(4)
C(7)-H(7A)	0.9900
C(7)-H(7B)	0.9900
C(8)-C(9)	1.514(4)
C(8)-H(8A)	0.9900
C(8)-H(8B)	0.9900
C(9)-C(10)	1.516(4)
C(9)-H(9)	1.0000
C(10)-H(10B)	0.9800
C(10)-H(10C)	0.9800
C(10)-H(10A)	0.9800
C(3)-O(2)-C(4)	114.4(2)
C(5)-O(3)-C(9)	113.92(18)
C(1)-N(1)-C(5)	115.1(2)
C(1)-N(1)-H(1)	122.4
C(5)-N(1)-H(1)	122.4
O(1)-C(1)-N(1)	125.1(2)
O(1)-C(1)-C(2)	126.3(2)

N(1)-C(1)-C(2)	108.6(2)
C(1)-C(2)-C(3)	104.9(2)
C(1)-C(2)-H(2A)	110.8
C(3)-C(2)-H(2A)	110.8
C(1)-C(2)-H(2B)	110.8
C(3)-C(2)-H(2B)	110.8
H(2A)-C(2)-H(2B)	108.8
O(2)-C(3)-C(2)	108.5(2)
O(2)-C(3)-C(5)	116.17(19)
C(2)-C(3)-C(5)	104.71(19)
O(2)-C(3)-H(3)	109.1
C(2)-C(3)-H(3)	109.1
C(5)-C(3)-H(3)	109.1
O(2)-C(4)-H(4B)	109.5
O(2)-C(4)-H(4A)	109.5
H(4B)-C(4)-H(4A)	109.5
O(2)-C(4)-H(4C)	109.5
H(4B)-C(4)-H(4C)	109.5
H(4A)-C(4)-H(4C)	109.5
O(3)-C(5)-N(1)	111.22(19)
O(3)-C(5)-C(6)	111.09(18)
N(1)-C(5)-C(6)	111.1(2)
O(3)-C(5)-C(3)	107.49(18)
N(1)-C(5)-C(3)	101.54(18)
C(6)-C(5)-C(3)	114.0(2)
C(5)-C(6)-C(7)	111.7(2)
C(5)-C(6)-H(6A)	109.3
C(7)-C(6)-H(6A)	109.3
C(5)-C(6)-H(6B)	109.3
C(7)-C(6)-H(6B)	109.3
H(6A)-C(6)-H(6B)	107.9
C(6)-C(7)-C(8)	109.1(2)
C(6)-C(7)-H(7A)	109.9
C(8)-C(7)-H(7A)	109.9
C(6)-C(7)-H(7B)	109.9
C(8)-C(7)-H(7B)	109.9
H(7A)-C(7)-H(7B)	108.3
C(9)-C(8)-C(7)	110.5(2)
C(9)-C(8)-H(8A)	109.6
C(7)-C(8)-H(8A)	109.6
C(9)-C(8)-H(8B)	109.6
C(7)-C(8)-H(8B)	109.6
H(8A)-C(8)-H(8B)	108.1
O(3)-C(9)-C(8)	111.4(2)

O(3)-C(9)-C(10)	105.5(2)
C(8)-C(9)-C(10)	113.3(2)
O(3)-C(9)-H(9)	108.8
C(8)-C(9)-H(9)	108.8
C(10)-C(9)-H(9)	108.8
C(9)-C(10)-H(10B)	109.5
C(9)-C(10)-H(10C)	109.5
H(10B)-C(10)-H(10C)	109.5
C(9)-C(10)-H(10A)	109.5
H(10B)-C(10)-H(10A)	109.5
H(10C)-C(10)-H(10A)	109.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for A. The anisotropic displacement factor exponent takes the form: -2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
O(1)	29(1)	34(1)	30(1)	4(1)	15(1)	11(1)
O(2)	26(1)	41(1)	25(1)	4(1)	6(1)	5(1)
O(3)	20(1)	26(1)	28(1)	-1(1)	10(1)	-2(1)
N(1)	22(1)	27(1)	23(1)	2(1)	9(1)	3(1)
C(1)	20(1)	26(1)	27(1)	-2(1)	12(1)	-3(1)
C(2)	29(1)	33(1)	27(1)	1(1)	15(1)	1(1)
C(3)	22(1)	28(1)	24(1)	3(1)	9(1)	-2(1)
C(4)	30(1)	40(2)	37(2)	11(1)	9(1)	11(1)
C(5)	17(1)	24(1)	23(1)	1(1)	8(1)	-2(1)
C(6)	22(1)	25(1)	27(1)	0(1)	9(1)	-1(1)
C(7)	30(1)	26(1)	26(1)	-4(1)	10(1)	3(1)
C(8)	29(1)	31(1)	36(1)	2(1)	18(1)	4(1)
C(9)	25(1)	29(1)	31(1)	5(1)	14(1)	1(1)
C(10)	28(1)	39(2)	47(2)	1(1)	20(1)	-5(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for A.

	x	y	z	U(eq)
H(1)	2887	10593	194	28
H(2A)	2800	9823	2626	34
H(2B)	3330	11539	2817	34
H(3)	3196	6757	2205	30
H(4B)	4433	6222	2690	56
H(4A)	4008	4642	2968	56
H(4C)	4540	5859	3794	56
H(6A)	3057	5788	516	30
H(6B)	3715	5294	1105	30
H(7A)	3239	8014	-634	33
H(7B)	3551	5510	-524	33
H(8A)	4417	7197	482	37
H(8B)	4165	8760	-478	37
H(9)	3833	11607	290	32
H(10B)	4677	13055	1478	55
H(10C)	4784	12218	560	55
H(10A)	4980	10576	1496	55

Table 6. Torsion angles [deg] for A.

C(5)-N(1)-C(1)-O(1)	175.8(2)
C(5)-N(1)-C(1)-C(2)	-4.1(3)
O(1)-C(1)-C(2)-C(3)	169.2(2)
N(1)-C(1)-C(2)-C(3)	-11.0(3)
C(4)-O(2)-C(3)-C(2)	173.4(2)
C(4)-O(2)-C(3)-C(5)	-69.1(3)
C(1)-C(2)-C(3)-O(2)	145.0(2)
C(1)-C(2)-C(3)-C(5)	20.4(2)
C(9)-O(3)-C(5)-N(1)	-68.3(2)
C(9)-O(3)-C(5)-C(6)	56.1(3)
C(9)-O(3)-C(5)-C(3)	-178.61(19)
C(1)-N(1)-C(5)-O(3)	-97.4(2)
C(1)-N(1)-C(5)-C(6)	138.2(2)
C(1)-N(1)-C(5)-C(3)	16.7(3)
O(2)-C(3)-C(5)-O(3)	-24.7(3)
C(2)-C(3)-C(5)-O(3)	95.0(2)
O(2)-C(3)-C(5)-N(1)	-141.5(2)
C(2)-C(3)-C(5)-N(1)	-21.9(2)
O(2)-C(3)-C(5)-C(6)	98.9(2)
C(2)-C(3)-C(5)-C(6)	-141.5(2)
O(3)-C(5)-C(6)-C(7)	-54.4(3)
N(1)-C(5)-C(6)-C(7)	70.0(2)
C(3)-C(5)-C(6)-C(7)	-176.05(19)
C(5)-C(6)-C(7)-C(8)	53.7(3)
C(6)-C(7)-C(8)-C(9)	-54.2(3)
C(5)-O(3)-C(9)-C(8)	-57.5(3)
C(5)-O(3)-C(9)-C(10)	179.1(2)
C(7)-C(8)-C(9)-O(3)	55.8(3)
C(7)-C(8)-C(9)-C(10)	174.6(2)

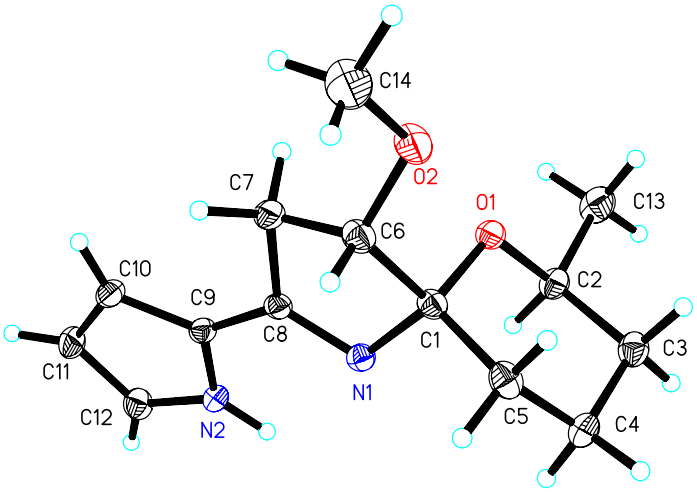
Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for A [A and deg.].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(1)-H(1)...O(1)#1	0.88	2.03	2.889(3)	164.3

Symmetry transformations used to generate equivalent atoms:
#1 -x+1/2,-y+5/2,-z

The X-ray structure of compound 3b



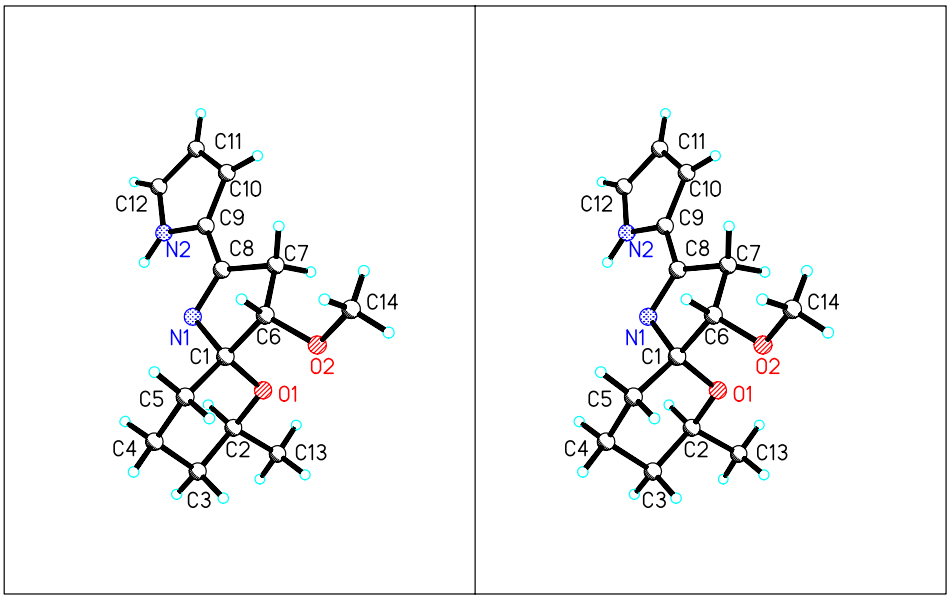


Table 1. Crystal data and structure refinement for A.

Identification code	a
Empirical formula	C14 H20 N2 O2
Formula weight	248.32
Temperature	173(2) K
Wavelength	0.71073 A
Crystal system, space group	Orthorhombic, Pbcn
Unit cell dimensions	a = 15.663(3) A alpha = 90 deg.
b = 8.2891(17) A	beta = 90 deg.
c = 21.056(4) A	gamma = 90 deg.
Volume	2733.7(10) A^3
Z, Calculated density	8, 1.207 Mg/m^3
Absorption coefficient	0.081 mm^-1
F(000)	1072
Crystal size	0.20 x 0.20 x 0.17 mm
Theta range for data collection	1.93 to 24.99 deg.
Limiting indices	-18<=h<=18, -8<=k<=9, -25<=l<=16
Reflections collected / unique	9483 / 2396 [R(int) = 0.0665]
Completeness to theta = 24.99	99.9 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9863 and 0.9839
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2396 / 0 / 165
Goodness-of-fit on F^2	1.245
Final R indices [I>2sigma(I)]	R1 = 0.0731, wR2 = 0.1230
R indices (all data)	R1 = 0.0889, wR2 = 0.1285
Largest diff. peak and hole	0.186 and -0.196 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for A. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
O(1)	5964(1)	4975(2)	3735(1)	28(1)
O(2)	6924(1)	6293(2)	4634(1)	43(1)
N(1)	5662(1)	7524(2)	3282(1)	24(1)
N(2)	5990(1)	9061(2)	2076(1)	27(1)
C(1)	5751(2)	6610(3)	3884(1)	26(1)
C(2)	5294(2)	4103(3)	3415(1)	29(1)
C(3)	4500(2)	4032(3)	3827(1)	33(1)
C(4)	4215(2)	5722(3)	4013(1)	35(1)
C(5)	4952(2)	6674(3)	4289(1)	33(1)
C(6)	6540(2)	7360(3)	4198(1)	31(1)
C(7)	7084(2)	7870(3)	3637(1)	32(1)
C(8)	6404(2)	8129(3)	3144(1)	24(1)
C(9)	6571(2)	8975(3)	2562(1)	25(1)
C(10)	7280(2)	9849(3)	2378(1)	31(1)
C(11)	7116(2)	10485(3)	1773(1)	35(1)
C(12)	6317(2)	9964(3)	1601(1)	33(1)
C(13)	5653(2)	2459(3)	3261(2)	42(1)
C(14)	7541(2)	7080(4)	5018(2)	61(1)

Table 3. Bond lengths [A] and angles [deg] for A.

O(1)-C(1)	1.431(3)
O(1)-C(2)	1.442(3)
O(2)-C(6)	1.410(3)
O(2)-C(14)	1.419(3)
N(1)-C(8)	1.298(3)
N(1)-C(1)	1.483(3)
N(2)-C(12)	1.349(3)
N(2)-C(9)	1.371(3)
N(2)-H(2)	0.8800
C(1)-C(5)	1.516(4)
C(1)-C(6)	1.534(3)
C(2)-C(13)	1.510(4)
C(2)-C(3)	1.515(4)
C(2)-H(2A)	1.0000
C(3)-C(4)	1.522(4)
C(3)-H(3B)	0.9900
C(3)-H(3A)	0.9900
C(4)-C(5)	1.515(4)
C(4)-H(4A)	0.9900
C(4)-H(4B)	0.9900
C(5)-H(5B)	0.9900
C(5)-H(5A)	0.9900
C(6)-C(7)	1.516(4)
C(6)-H(6)	1.0000
C(7)-C(8)	1.504(3)
C(7)-H(7B)	0.9900
C(7)-H(7A)	0.9900
C(8)-C(9)	1.436(4)
C(9)-C(10)	1.381(3)
C(10)-C(11)	1.403(4)
C(10)-H(10)	0.9500
C(11)-C(12)	1.372(4)
C(11)-H(11)	0.9500
C(12)-H(12)	0.9500
C(13)-H(13C)	0.9800
C(13)-H(13B)	0.9800
C(13)-H(13A)	0.9800
C(14)-H(14C)	0.9800
C(14)-H(14A)	0.9800
C(14)-H(14B)	0.9800

C(1)-O(1)-C(2)	114.02(18)
C(6)-O(2)-C(14)	111.9(2)
C(8)-N(1)-C(1)	107.8(2)
C(12)-N(2)-C(9)	109.2(2)
C(12)-N(2)-H(2)	125.4
C(9)-N(2)-H(2)	125.4
O(1)-C(1)-N(1)	108.6(2)
O(1)-C(1)-C(5)	110.5(2)
N(1)-C(1)-C(5)	112.7(2)
O(1)-C(1)-C(6)	106.8(2)
N(1)-C(1)-C(6)	103.7(2)
C(5)-C(1)-C(6)	114.1(2)
O(1)-C(2)-C(13)	106.3(2)
O(1)-C(2)-C(3)	110.5(2)
C(13)-C(2)-C(3)	113.2(2)
O(1)-C(2)-H(2A)	108.9
C(13)-C(2)-H(2A)	108.9
C(3)-C(2)-H(2A)	108.9
C(2)-C(3)-C(4)	110.6(2)
C(2)-C(3)-H(3B)	109.5
C(4)-C(3)-H(3B)	109.5
C(2)-C(3)-H(3A)	109.5
C(4)-C(3)-H(3A)	109.5
H(3B)-C(3)-H(3A)	108.1
C(5)-C(4)-C(3)	110.8(2)
C(5)-C(4)-H(4A)	109.5
C(3)-C(4)-H(4A)	109.5
C(5)-C(4)-H(4B)	109.5
C(3)-C(4)-H(4B)	109.5
H(4A)-C(4)-H(4B)	108.1
C(4)-C(5)-C(1)	113.2(2)
C(4)-C(5)-H(5B)	108.9
C(1)-C(5)-H(5B)	108.9
C(4)-C(5)-H(5A)	108.9
C(1)-C(5)-H(5A)	108.9
H(5B)-C(5)-H(5A)	107.7
O(2)-C(6)-C(7)	116.3(2)
O(2)-C(6)-C(1)	111.7(2)
C(7)-C(6)-C(1)	103.3(2)
O(2)-C(6)-H(6)	108.4
C(7)-C(6)-H(6)	108.4
C(1)-C(6)-H(6)	108.4
C(8)-C(7)-C(6)	100.4(2)
C(8)-C(7)-H(7B)	111.7

C(6)-C(7)-H(7B)	111.7
C(8)-C(7)-H(7A)	111.7
C(6)-C(7)-H(7A)	111.7
H(7B)-C(7)-H(7A)	109.5
N(1)-C(8)-C(9)	122.9(2)
N(1)-C(8)-C(7)	115.1(2)
C(9)-C(8)-C(7)	122.0(2)
N(2)-C(9)-C(10)	107.3(2)
N(2)-C(9)-C(8)	122.8(2)
C(10)-C(9)-C(8)	129.9(2)
C(9)-C(10)-C(11)	107.7(2)
C(9)-C(10)-H(10)	126.1
C(11)-C(10)-H(10)	126.1
C(12)-C(11)-C(10)	106.7(2)
C(12)-C(11)-H(11)	126.6
C(10)-C(11)-H(11)	126.6
N(2)-C(12)-C(11)	109.0(3)
N(2)-C(12)-H(12)	125.5
C(11)-C(12)-H(12)	125.5
C(2)-C(13)-H(13C)	109.5
C(2)-C(13)-H(13B)	109.5
H(13C)-C(13)-H(13B)	109.5
C(2)-C(13)-H(13A)	109.5
H(13C)-C(13)-H(13A)	109.5
H(13B)-C(13)-H(13A)	109.5
O(2)-C(14)-H(14C)	109.5
O(2)-C(14)-H(14A)	109.5
H(14C)-C(14)-H(14A)	109.5
O(2)-C(14)-H(14B)	109.5
H(14C)-C(14)-H(14B)	109.5
H(14A)-C(14)-H(14B)	109.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for A.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
O(1)	25(1)	24(1)	33(1)	-2(1)	-6(1)	-2(1)
O(2)	49(1)	38(1)	41(1)	8(1)	-25(1)	-11(1)
N(1)	23(1)	24(1)	25(1)	-2(1)	-1(1)	1(1)
N(2)	22(1)	28(1)	31(1)	2(1)	2(1)	-2(1)
C(1)	28(1)	23(1)	25(2)	0(1)	-5(1)	0(1)
C(2)	27(1)	31(1)	29(2)	-4(1)	-5(1)	-5(1)
C(3)	33(2)	34(2)	32(2)	1(1)	-3(1)	-7(1)
C(4)	28(2)	39(2)	38(2)	5(1)	9(1)	-2(1)
C(5)	43(2)	29(1)	27(2)	1(1)	2(1)	1(1)
C(6)	35(2)	27(1)	30(2)	1(1)	-8(1)	-1(1)
C(7)	26(1)	26(1)	43(2)	-2(1)	-9(1)	-2(1)
C(8)	23(1)	19(1)	30(2)	-4(1)	-3(1)	2(1)
C(9)	21(1)	23(1)	31(2)	-3(1)	0(1)	1(1)
C(10)	21(1)	30(1)	42(2)	-5(1)	0(1)	-1(1)
C(11)	30(2)	34(2)	41(2)	5(1)	10(1)	-4(1)
C(12)	36(2)	35(2)	28(2)	5(1)	5(1)	-1(1)
C(13)	38(2)	36(2)	54(2)	-14(2)	1(2)	-6(1)
C(14)	68(2)	58(2)	57(2)	8(2)	-37(2)	-18(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for A.

	x	y	z	U(eq)
H(2)	5484	8601	2074	32
H(2A)	5152	4671	3010	35
H(3B)	4622	3396	4214	39
H(3A)	4036	3485	3591	39
H(4A)	3990	6288	3634	42
H(4B)	3749	5649	4329	42
H(5B)	5086	6246	4717	39
H(5A)	4774	7814	4339	39
H(6)	6358	8351	4432	37
H(7B)	7403	8876	3727	38
H(7A)	7489	7012	3510	38
H(10)	7787	9995	2618	37
H(11)	7486	11148	1529	42
H(12)	6038	10201	1212	39
H(13C)	5835	1926	3654	63
H(13B)	6144	2578	2976	63
H(13A)	5213	1805	3052	63
H(14C)	7785	6306	5319	91
H(14A)	7269	7963	5252	91
H(14B)	7996	7514	4747	91

Table 6. Torsion angles [deg] for A.

C(2)-O(1)-C(1)-N(1)	-66.9(2)
C(2)-O(1)-C(1)-C(5)	57.2(3)
C(2)-O(1)-C(1)-C(6)	-178.1(2)
C(8)-N(1)-C(1)-O(1)	-90.7(2)
C(8)-N(1)-C(1)-C(5)	146.6(2)
C(8)-N(1)-C(1)-C(6)	22.7(3)
C(1)-O(1)-C(2)-C(13)	176.4(2)
C(1)-O(1)-C(2)-C(3)	-60.4(3)
O(1)-C(2)-C(3)-C(4)	55.9(3)
C(13)-C(2)-C(3)-C(4)	175.1(2)
C(2)-C(3)-C(4)-C(5)	-51.0(3)
C(3)-C(4)-C(5)-C(1)	49.4(3)
O(1)-C(1)-C(5)-C(4)	-51.3(3)
N(1)-C(1)-C(5)-C(4)	70.3(3)
C(6)-C(1)-C(5)-C(4)	-171.7(2)
C(14)-O(2)-C(6)-C(7)	73.7(3)
C(14)-O(2)-C(6)-C(1)	-168.0(3)
O(1)-C(1)-C(6)-O(2)	-42.2(3)
N(1)-C(1)-C(6)-O(2)	-156.8(2)
C(5)-C(1)-C(6)-O(2)	80.2(3)
O(1)-C(1)-C(6)-C(7)	83.6(2)
N(1)-C(1)-C(6)-C(7)	-31.0(2)
C(5)-C(1)-C(6)-C(7)	-154.0(2)
O(2)-C(6)-C(7)-C(8)	149.8(2)
C(1)-C(6)-C(7)-C(8)	27.0(2)
C(1)-N(1)-C(8)-C(9)	174.5(2)
C(1)-N(1)-C(8)-C(7)	-5.1(3)
C(6)-C(7)-C(8)-N(1)	-14.8(3)
C(6)-C(7)-C(8)-C(9)	165.6(2)
C(12)-N(2)-C(9)-C(10)	-0.2(3)
C(12)-N(2)-C(9)-C(8)	178.5(2)
N(1)-C(8)-C(9)-N(2)	-7.3(4)
C(7)-C(8)-C(9)-N(2)	172.3(2)
N(1)-C(8)-C(9)-C(10)	171.1(3)
C(7)-C(8)-C(9)-C(10)	-9.4(4)
N(2)-C(9)-C(10)-C(11)	0.6(3)
C(8)-C(9)-C(10)-C(11)	-178.0(2)
C(9)-C(10)-C(11)-C(12)	-0.7(3)
C(9)-N(2)-C(12)-C(11)	-0.2(3)
C(10)-C(11)-C(12)-N(2)	0.6(3)

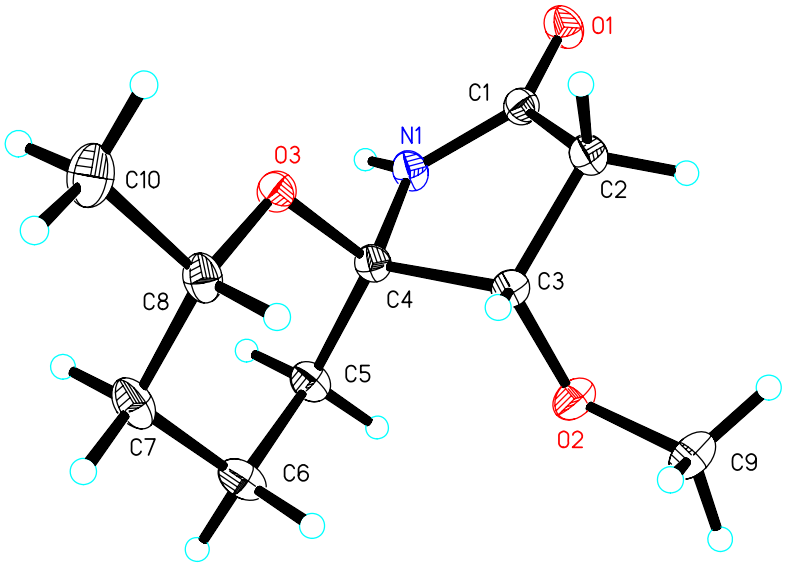
Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for A [A and deg.].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(2)-H(2)...N(1)	0.88	2.71	2.887(3)	92.6

Symmetry transformations used to generate equivalent atoms:

The X-ray structure of compound 4c



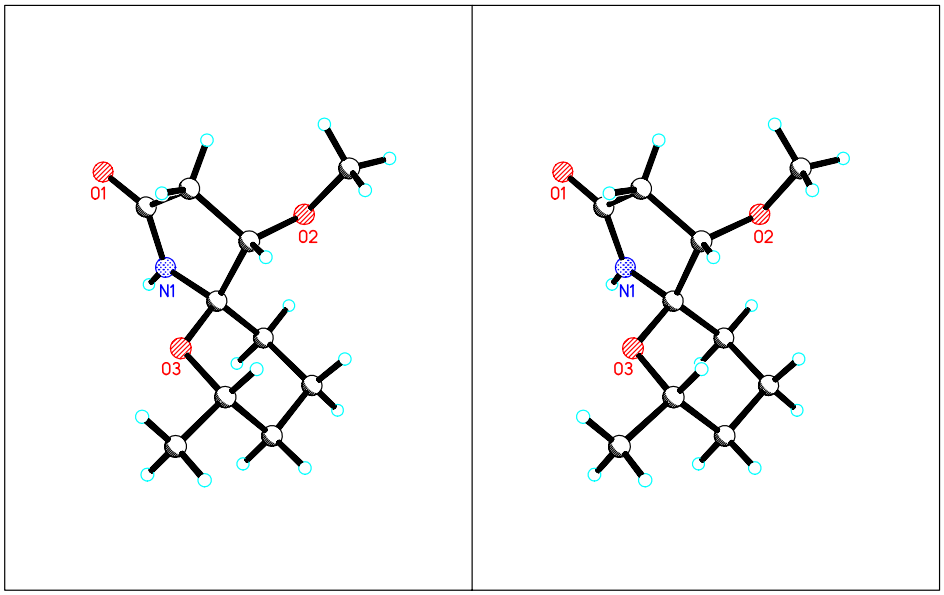


Table 1. Crystal data and structure refinement for sa1523.

Identification code	sa1523
Empirical formula	C10 H17 N O3
Formula weight	199.25
Temperature	173(2) K
Wavelength	0.71073 A
Crystal system, space group	Monoclinic, P2(1)/n
Unit cell dimensions	a = 11.044(3) A alpha = 90 deg. b = 8.0060(19) A beta = 98.346(4) deg. c = 11.730(3) A gamma = 90 deg.
Volume	1026.1(4) A^3
Z, Calculated density	4, 1.290 Mg/m^3
Absorption coefficient	0.095 mm^-1
F(000)	432
Crystal size	0.39 x 0.34 x 0.28 mm
Theta range for data collection	3.09 to 27.48 deg.
Limiting indices	-14<=h<=12, -8<=k<=10, -15<=l<=15
Reflections collected / unique	5182 / 2344 [R(int) = 0.0296]
Completeness to theta = 27.48	99.4 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9740 and 0.9640
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2344 / 0 / 129
Goodness-of-fit on F^2	1.112
Final R indices [I>2sigma(I)]	R1 = 0.0502, wR2 = 0.1136
R indices (all data)	R1 = 0.0555, wR2 = 0.1176
Largest diff. peak and hole	0.249 and -0.189 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for sa1523. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
O(1)	9739(1)	4798(2)	3373(1)	33(1)
O(2)	6005(1)	4087(1)	3455(1)	30(1)
O(3)	8050(1)	1059(1)	5028(1)	26(1)
N(1)	8617(1)	3761(2)	4711(1)	26(1)
C(1)	8868(1)	3997(2)	3636(1)	25(1)
C(2)	7891(1)	3125(2)	2810(1)	26(1)
C(3)	6874(1)	2767(2)	3538(1)	23(1)
C(4)	7569(1)	2718(2)	4789(1)	23(1)
C(5)	6865(1)	3323(2)	5732(1)	28(1)
C(6)	5900(2)	2062(2)	5967(1)	33(1)
C(7)	6496(2)	376(2)	6236(1)	33(1)
C(8)	7164(1)	-181(2)	5259(1)	28(1)
C(9)	5138(2)	3990(2)	2431(2)	37(1)
C(10)	7855(2)	-1806(2)	5493(2)	40(1)

Table 3. Bond lengths [A] and angles [deg] for sa1523.

O(1)-C(1)	1.2318(17)
O(2)-C(3)	1.4209(17)
O(2)-C(9)	1.4248(18)
O(3)-C(4)	1.4431(18)
O(3)-C(8)	1.4464(17)
N(1)-C(1)	1.3435(18)
N(1)-C(4)	1.4410(18)
N(1)-H(1)	0.8800
C(1)-C(2)	1.512(2)
C(2)-C(3)	1.5328(19)
C(2)-H(2B)	0.9900
C(2)-H(2A)	0.9900
C(3)-C(4)	1.5553(19)
C(3)-H(3)	1.0000
C(4)-C(5)	1.520(2)
C(5)-C(6)	1.523(2)
C(5)-H(5A)	0.9900
C(5)-H(5B)	0.9900
C(6)-C(7)	1.515(2)
C(6)-H(6B)	0.9900
C(6)-H(6A)	0.9900
C(7)-C(8)	1.518(2)
C(7)-H(7A)	0.9900
C(7)-H(7B)	0.9900
C(8)-C(10)	1.513(2)
C(8)-H(8)	1.0000
C(9)-H(9C)	0.9800
C(9)-H(9B)	0.9800
C(9)-H(9A)	0.9800
C(10)-H(10C)	0.9800
C(10)-H(10B)	0.9800
C(10)-H(10A)	0.9800
C(3)-O(2)-C(9)	112.43(11)
C(4)-O(3)-C(8)	115.32(11)
C(1)-N(1)-C(4)	114.73(12)
C(1)-N(1)-H(1)	122.6
C(4)-N(1)-H(1)	122.6
O(1)-C(1)-N(1)	125.71(13)
O(1)-C(1)-C(2)	126.16(13)
N(1)-C(1)-C(2)	108.12(12)
C(1)-C(2)-C(3)	104.00(11)
C(1)-C(2)-H(2B)	111.0

C(3)-C(2)-H(2B)	111.0
C(1)-C(2)-H(2A)	111.0
C(3)-C(2)-H(2A)	111.0
H(2B)-C(2)-H(2A)	109.0
O(2)-C(3)-C(2)	111.39(12)
O(2)-C(3)-C(4)	108.61(11)
C(2)-C(3)-C(4)	103.26(11)
O(2)-C(3)-H(3)	111.1
C(2)-C(3)-H(3)	111.1
C(4)-C(3)-H(3)	111.1
N(1)-C(4)-O(3)	105.60(11)
N(1)-C(4)-C(5)	111.18(12)
O(3)-C(4)-C(5)	111.43(12)
N(1)-C(4)-C(3)	102.48(11)
O(3)-C(4)-C(3)	108.90(11)
C(5)-C(4)-C(3)	116.39(12)
C(4)-C(5)-C(6)	111.46(12)
C(4)-C(5)-H(5A)	109.3
C(6)-C(5)-H(5A)	109.3
C(4)-C(5)-H(5B)	109.3
C(6)-C(5)-H(5B)	109.3
H(5A)-C(5)-H(5B)	108.0
C(7)-C(6)-C(5)	109.51(13)
C(7)-C(6)-H(6B)	109.8
C(5)-C(6)-H(6B)	109.8
C(7)-C(6)-H(6A)	109.8
C(5)-C(6)-H(6A)	109.8
H(6B)-C(6)-H(6A)	108.2
C(6)-C(7)-C(8)	110.42(13)
C(6)-C(7)-H(7A)	109.6
C(8)-C(7)-H(7A)	109.6
C(6)-C(7)-H(7B)	109.6
C(8)-C(7)-H(7B)	109.6
H(7A)-C(7)-H(7B)	108.1
O(3)-C(8)-C(10)	106.68(13)
O(3)-C(8)-C(7)	110.88(12)
C(10)-C(8)-C(7)	113.79(14)
O(3)-C(8)-H(8)	108.4
C(10)-C(8)-H(8)	108.4
C(7)-C(8)-H(8)	108.4
O(2)-C(9)-H(9C)	109.5
O(2)-C(9)-H(9B)	109.5
H(9C)-C(9)-H(9B)	109.5
O(2)-C(9)-H(9A)	109.5

H(9C)-C(9)-H(9A)	109.5
H(9B)-C(9)-H(9A)	109.5
C(8)-C(10)-H(10C)	109.5
C(8)-C(10)-H(10B)	109.5
H(10C)-C(10)-H(10B)	109.5
C(8)-C(10)-H(10A)	109.5
H(10C)-C(10)-H(10A)	109.5
H(10B)-C(10)-H(10A)	109.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for sa1523.

The anisotropic displacement factor exponent takes the form:

-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
O(1)	30(1)	39(1)	31(1)	4(1)	7(1)	-11(1)
O(2)	29(1)	30(1)	31(1)	-3(1)	-2(1)	7(1)
O(3)	24(1)	24(1)	29(1)	3(1)	3(1)	-3(1)
N(1)	25(1)	28(1)	23(1)	0(1)	1(1)	-10(1)
C(1)	24(1)	24(1)	26(1)	3(1)	4(1)	-1(1)
C(2)	28(1)	29(1)	23(1)	0(1)	5(1)	-3(1)
C(3)	22(1)	21(1)	24(1)	-2(1)	2(1)	-1(1)
C(4)	23(1)	22(1)	23(1)	0(1)	3(1)	-4(1)
C(5)	30(1)	29(1)	24(1)	-5(1)	6(1)	-6(1)
C(6)	32(1)	39(1)	30(1)	-6(1)	12(1)	-10(1)
C(7)	39(1)	35(1)	28(1)	1(1)	8(1)	-15(1)
C(8)	33(1)	24(1)	25(1)	2(1)	2(1)	-8(1)
C(9)	31(1)	36(1)	39(1)	0(1)	-6(1)	7(1)
C(10)	52(1)	26(1)	40(1)	5(1)	0(1)	-3(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for sa1523.

	x	y	z	U(eq)
H(1)	9057	4208	5320	31
H(2B)	8208	2075	2519	32
H(2A)	7590	3854	2148	32
H(3)	6467	1674	3320	27
H(5A)	6464	4401	5497	33
H(5B)	7444	3514	6447	33
H(6B)	5504	2442	6627	39
H(6A)	5261	1970	5284	39
H(7A)	5862	-458	6350	40
H(7B)	7081	449	6958	40
H(8)	6551	-322	4549	33
H(9C)	5544	4247	1763	55
H(9B)	4479	4796	2475	55
H(9A)	4795	2860	2352	55
H(10C)	8433	-1706	6207	60
H(10B)	8305	-2054	4851	60
H(10A)	7275	-2711	5571	60

Table 6. Torsion angles [deg] for sa1523.

C(4)-N(1)-C(1)-O(1)	-177.67(14)
C(4)-N(1)-C(1)-C(2)	2.90(17)
O(1)-C(1)-C(2)-C(3)	-164.74(15)
N(1)-C(1)-C(2)-C(3)	14.70(15)
C(9)-O(2)-C(3)-C(2)	80.59(15)
C(9)-O(2)-C(3)-C(4)	-166.34(12)
C(1)-C(2)-C(3)-O(2)	91.53(13)
C(1)-C(2)-C(3)-C(4)	-24.86(14)
C(1)-N(1)-C(4)-O(3)	95.15(14)
C(1)-N(1)-C(4)-C(5)	-143.85(13)
C(1)-N(1)-C(4)-C(3)	-18.82(16)
C(8)-O(3)-C(4)-N(1)	174.00(10)
C(8)-O(3)-C(4)-C(5)	53.16(15)
C(8)-O(3)-C(4)-C(3)	-76.57(14)
O(2)-C(3)-C(4)-N(1)	-92.24(13)
C(2)-C(3)-C(4)-N(1)	26.10(14)
O(2)-C(3)-C(4)-O(3)	156.24(11)
C(2)-C(3)-C(4)-O(3)	-85.42(13)
O(2)-C(3)-C(4)-C(5)	29.29(16)
C(2)-C(3)-C(4)-C(5)	147.63(13)
N(1)-C(4)-C(5)-C(6)	-169.72(12)
O(3)-C(4)-C(5)-C(6)	-52.21(16)
C(3)-C(4)-C(5)-C(6)	73.47(17)
C(4)-C(5)-C(6)-C(7)	54.65(16)
C(5)-C(6)-C(7)-C(8)	-56.36(16)
C(4)-O(3)-C(8)-C(10)	-179.58(12)
C(4)-O(3)-C(8)-C(7)	-55.17(16)
C(6)-C(7)-C(8)-O(3)	56.00(16)
C(6)-C(7)-C(8)-C(10)	176.25(13)

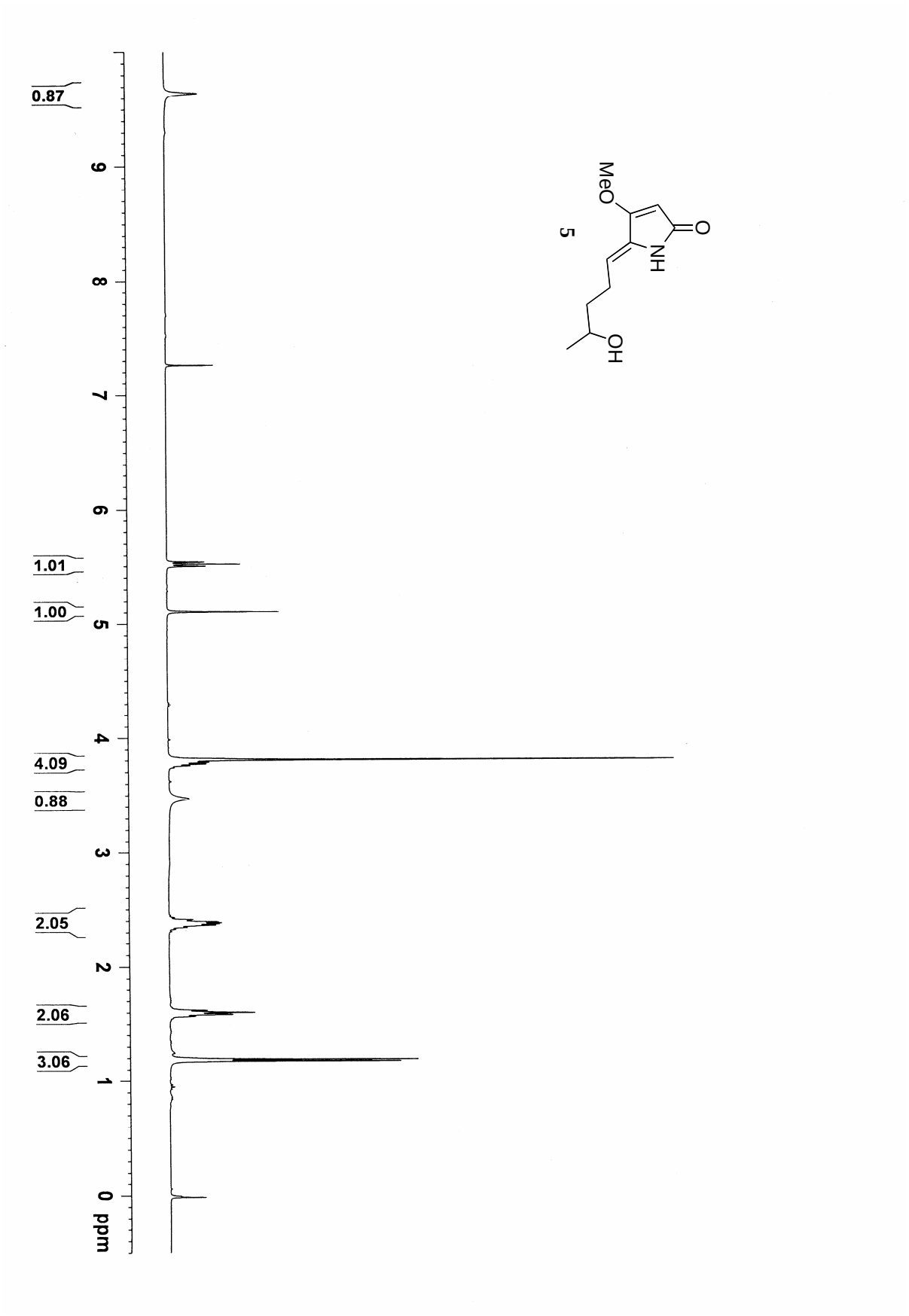
Symmetry transformations used to generate equivalent atoms:

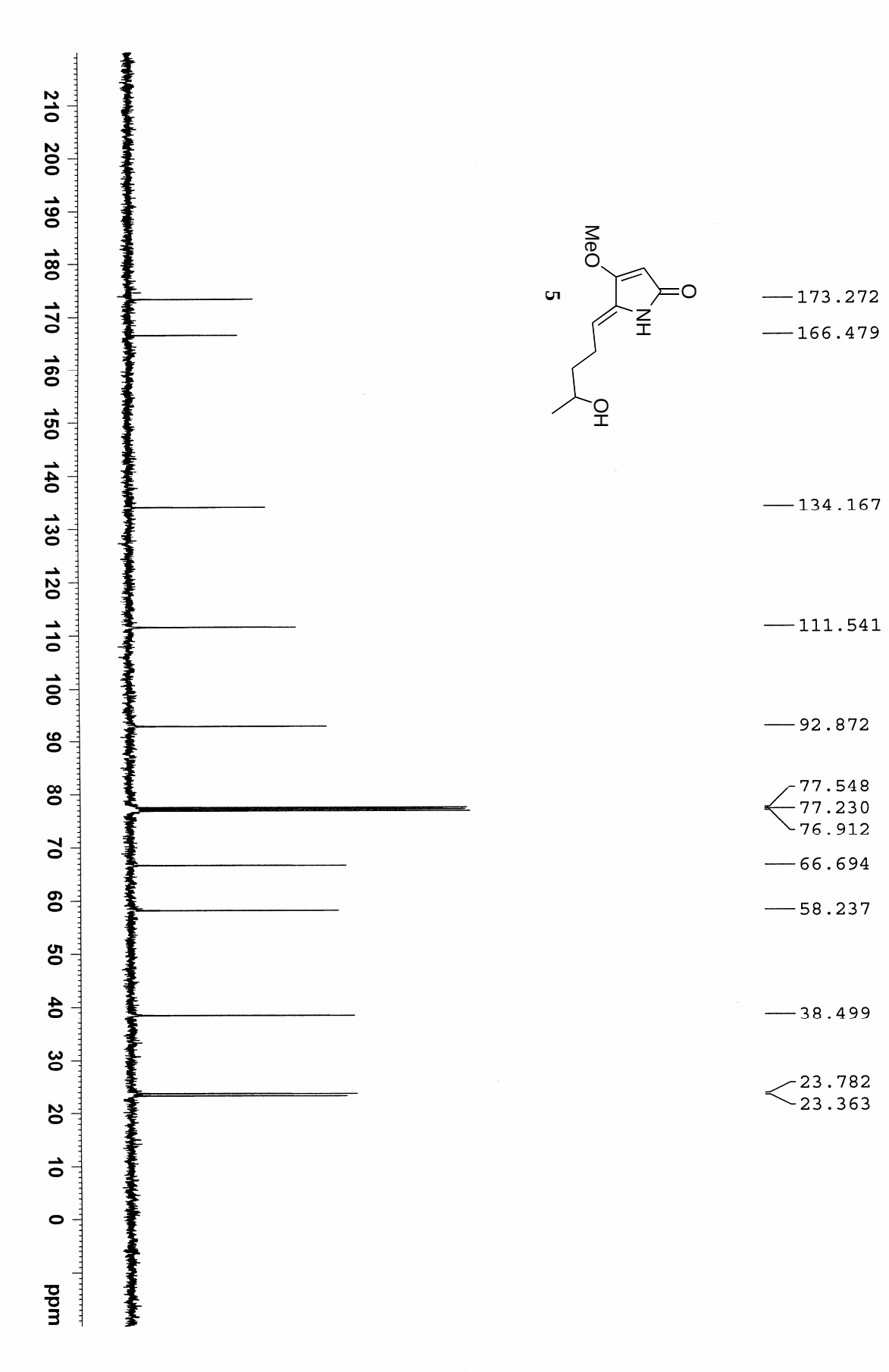
Table 7. Hydrogen bonds for sa1523 [A and deg.].

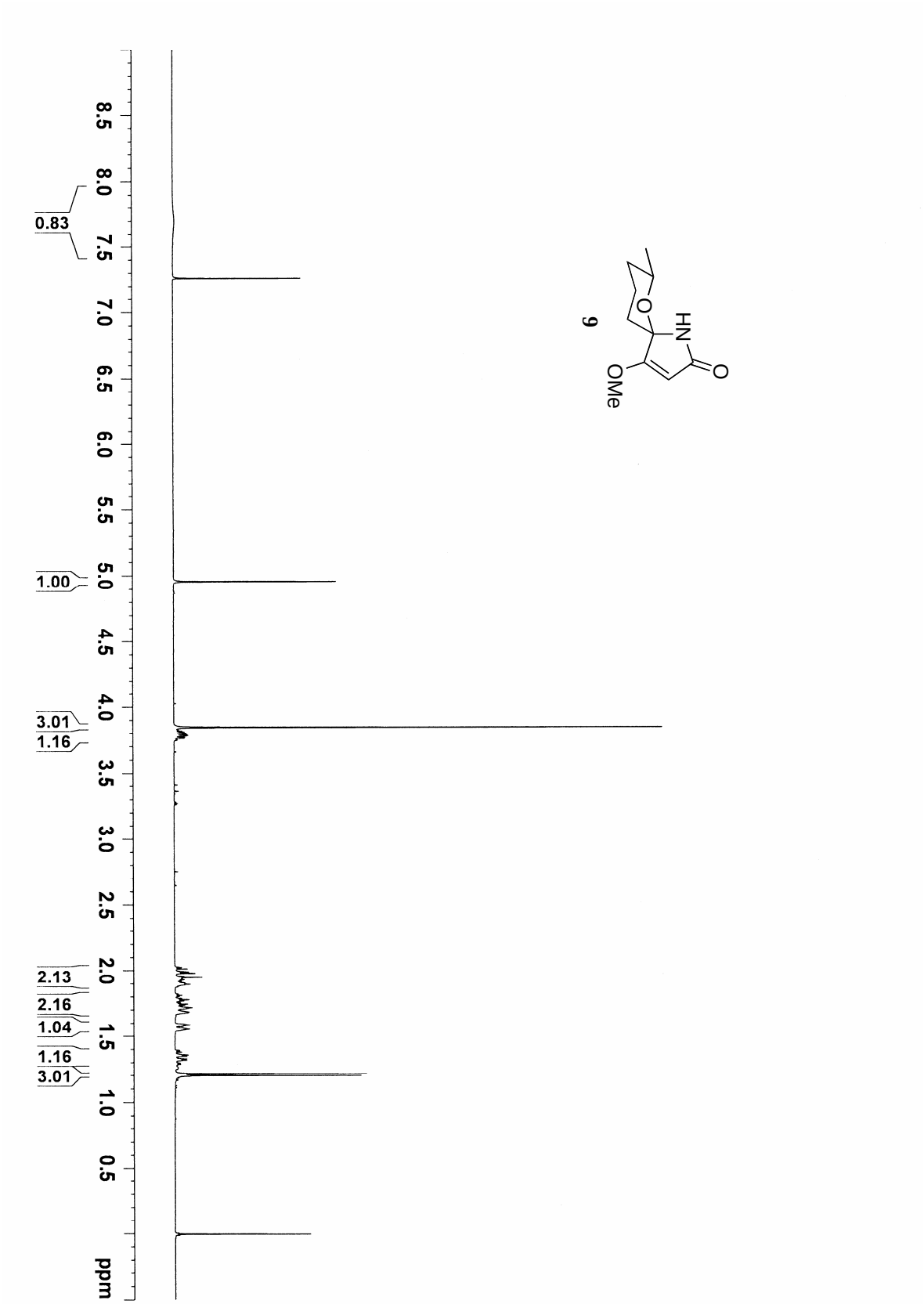
D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(1)-H(1)...O(1)#1	0.88	2.04	2.9146(17)	172.9

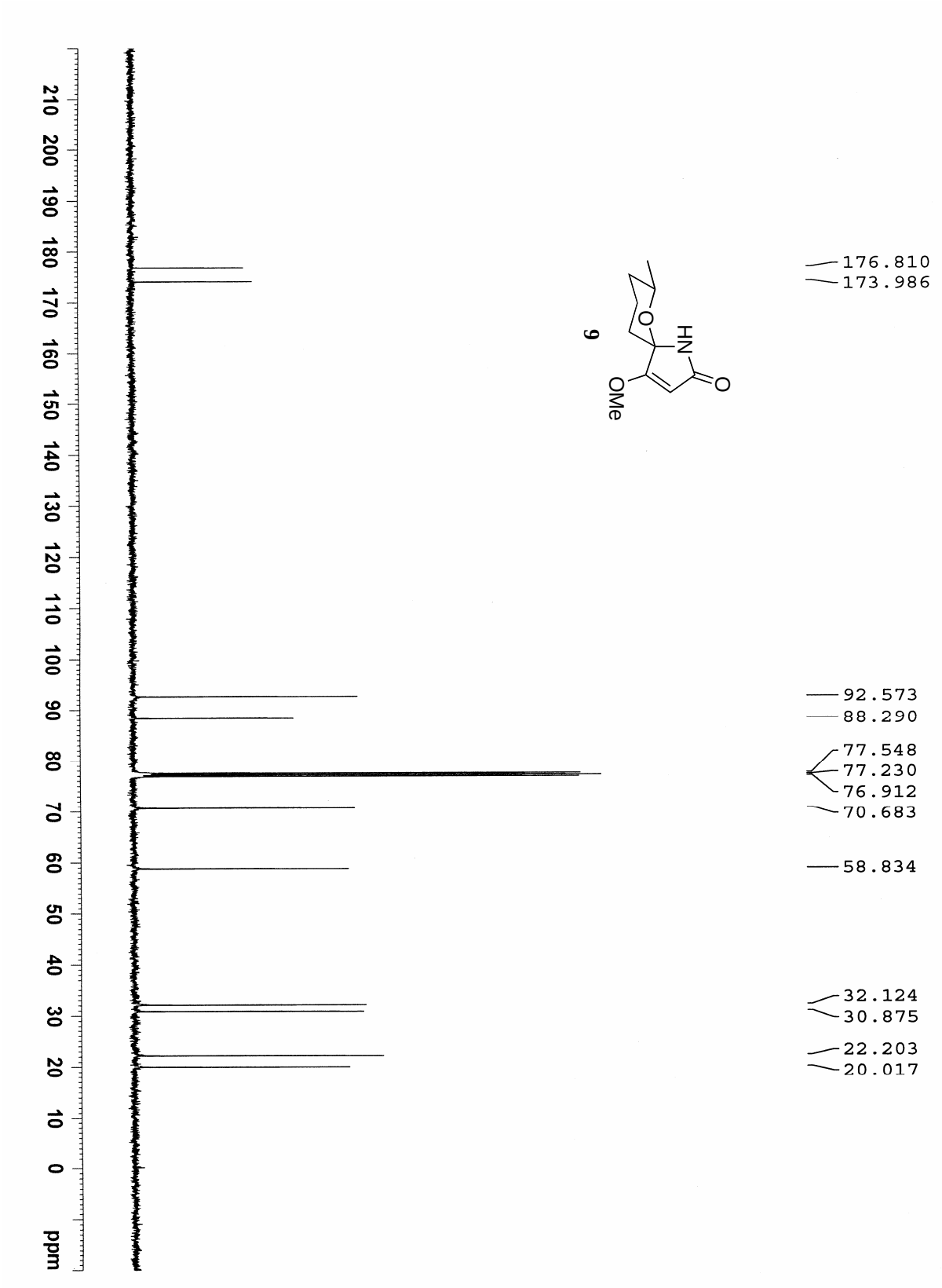
Symmetry transformations used to generate equivalent atoms:

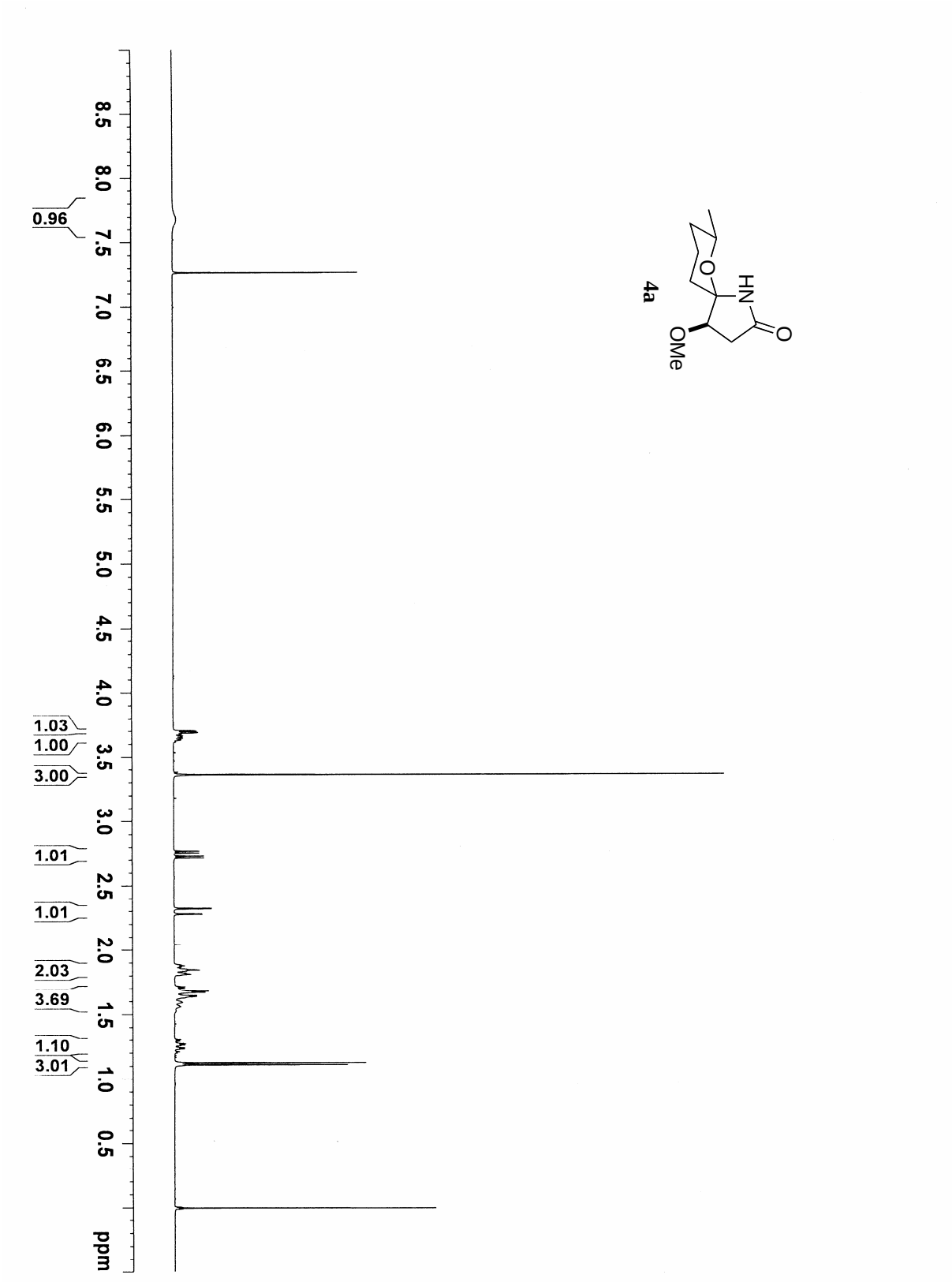
#1 -x+2,-y+1,-z+1

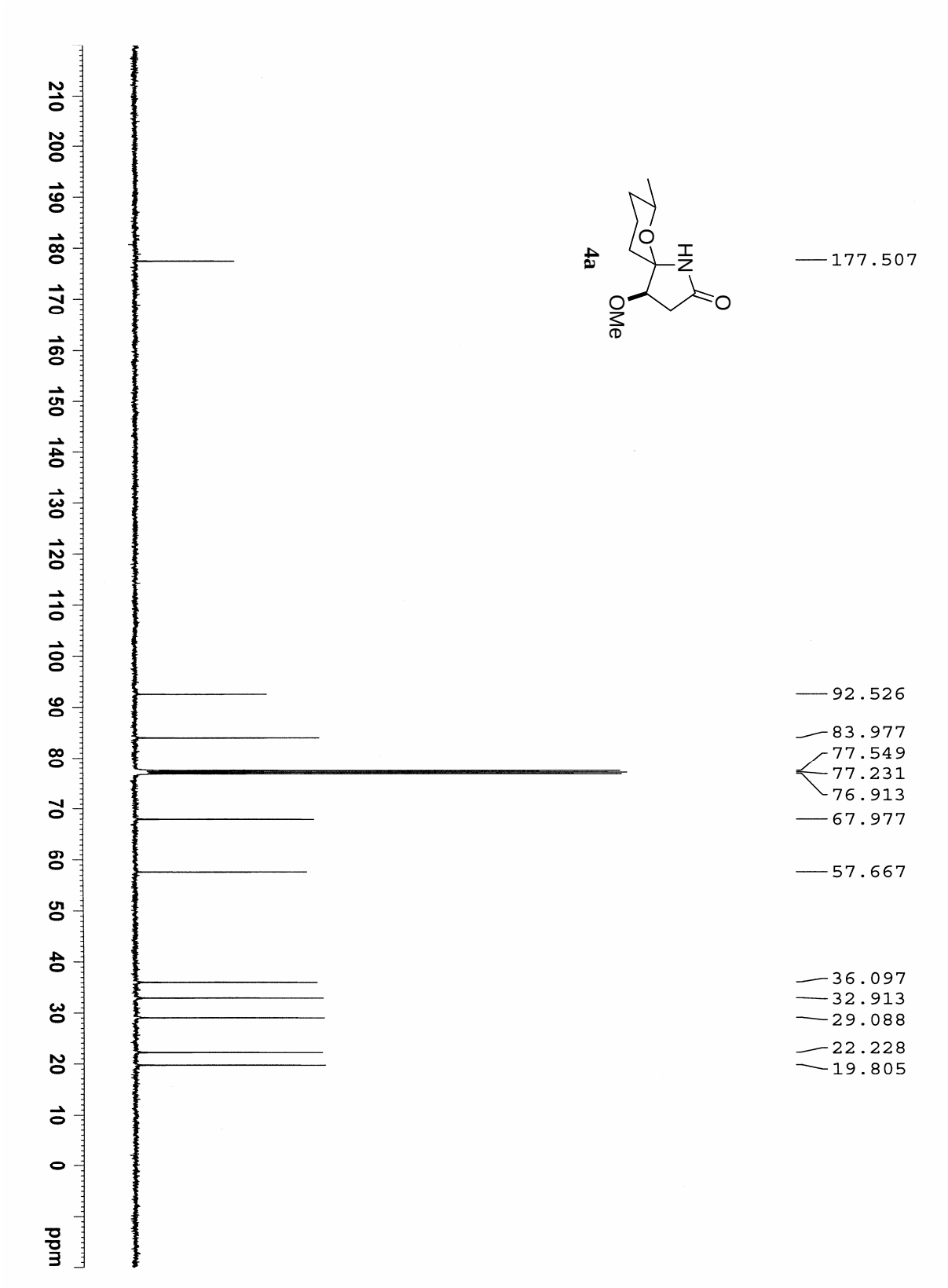


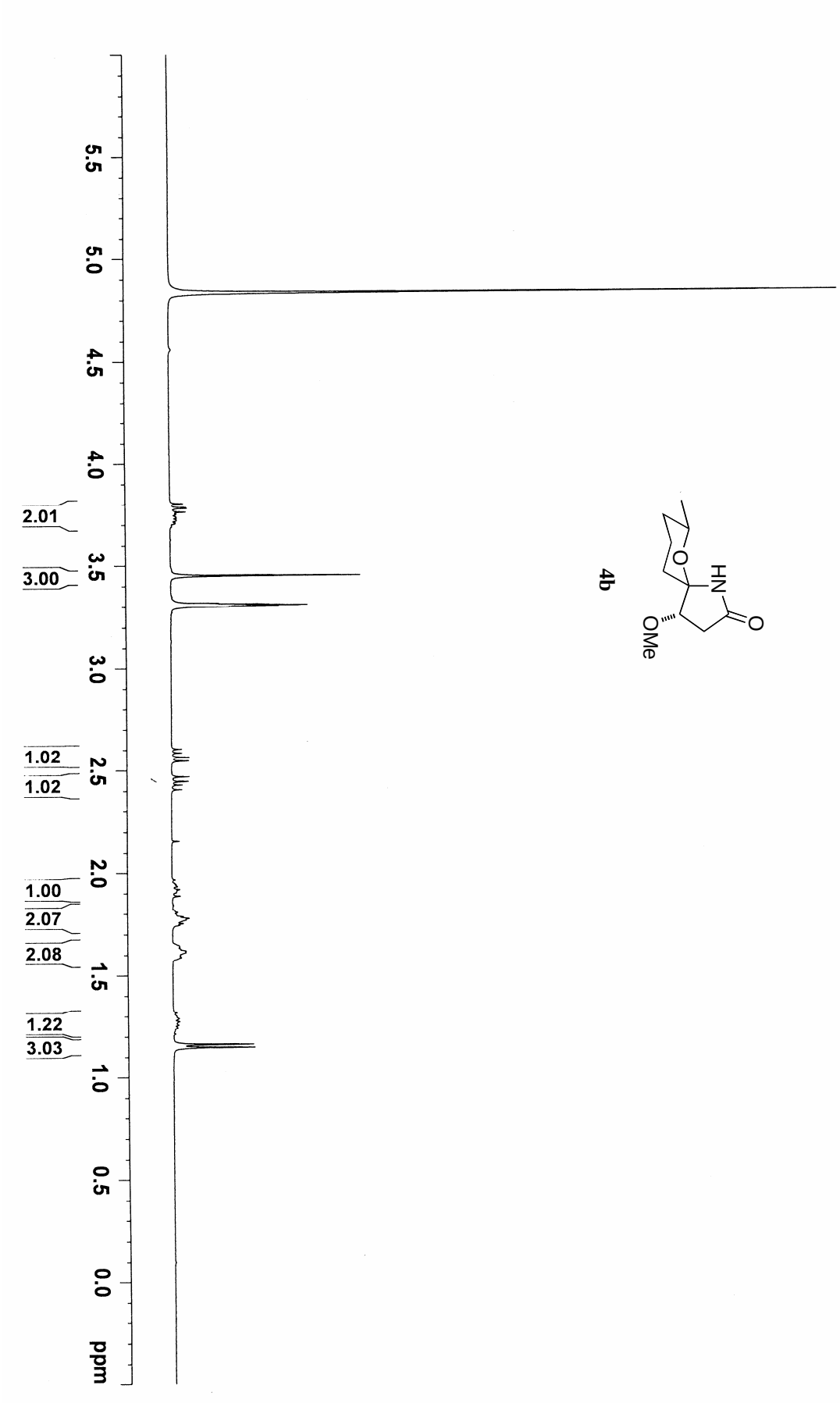


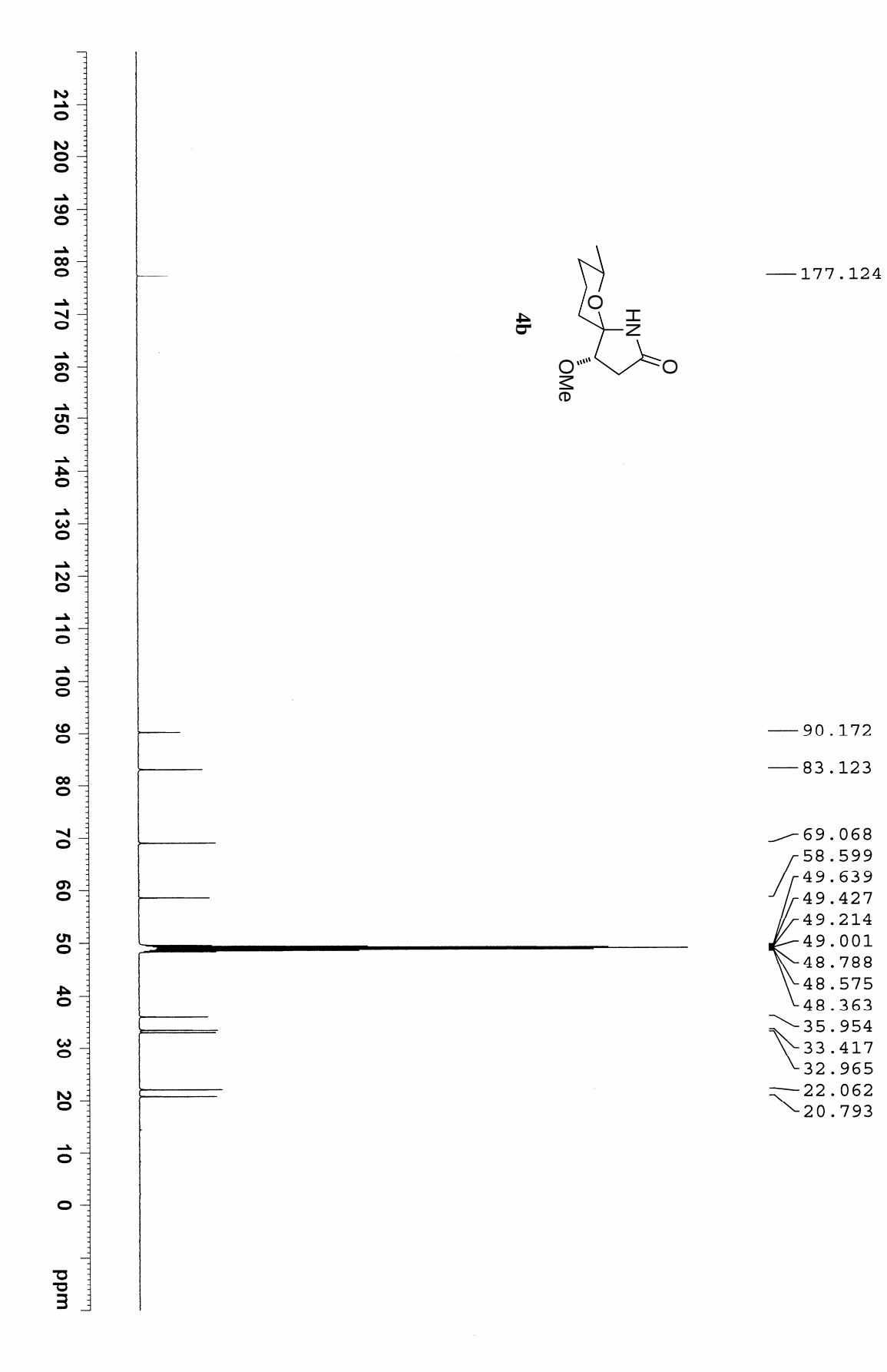


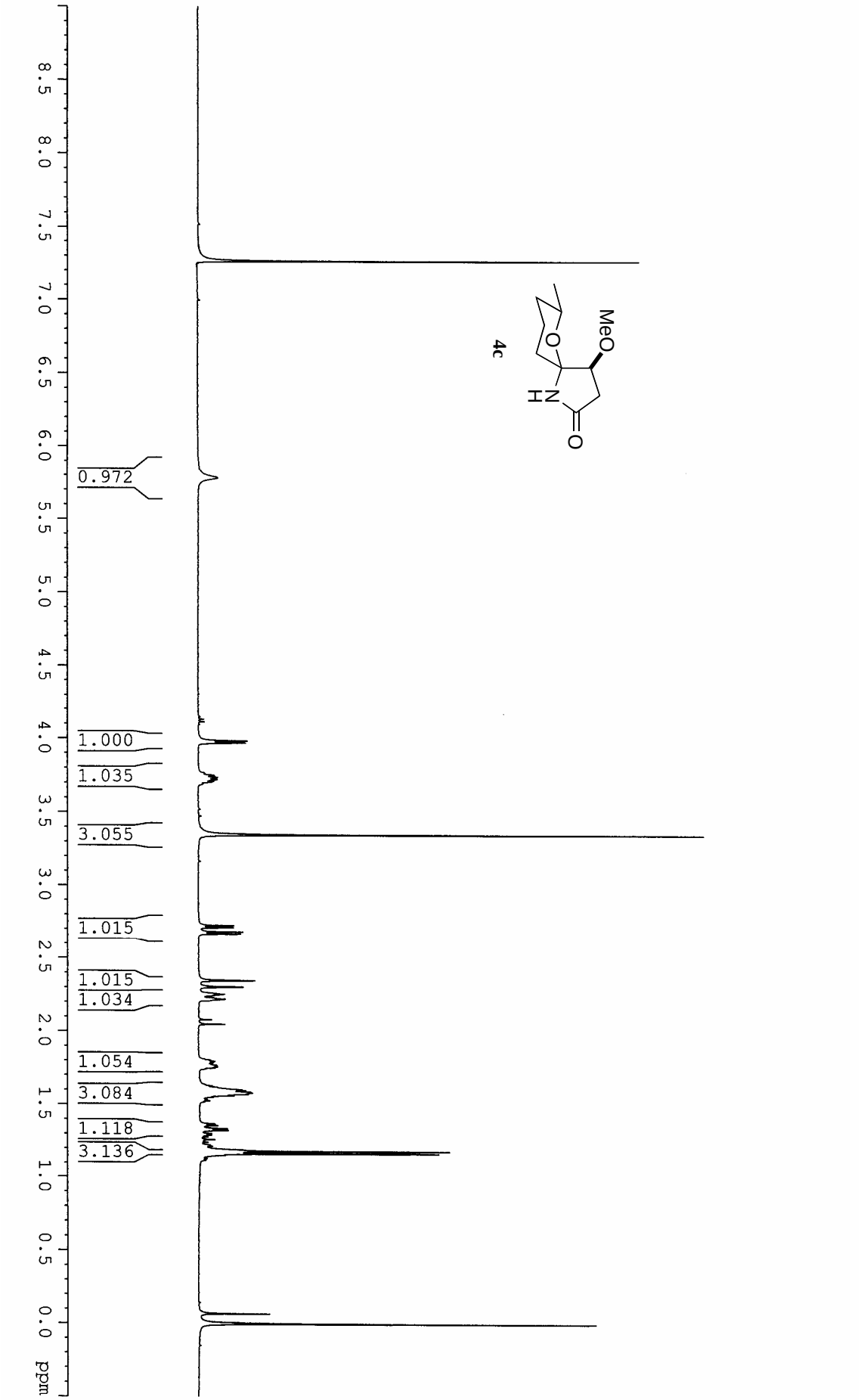


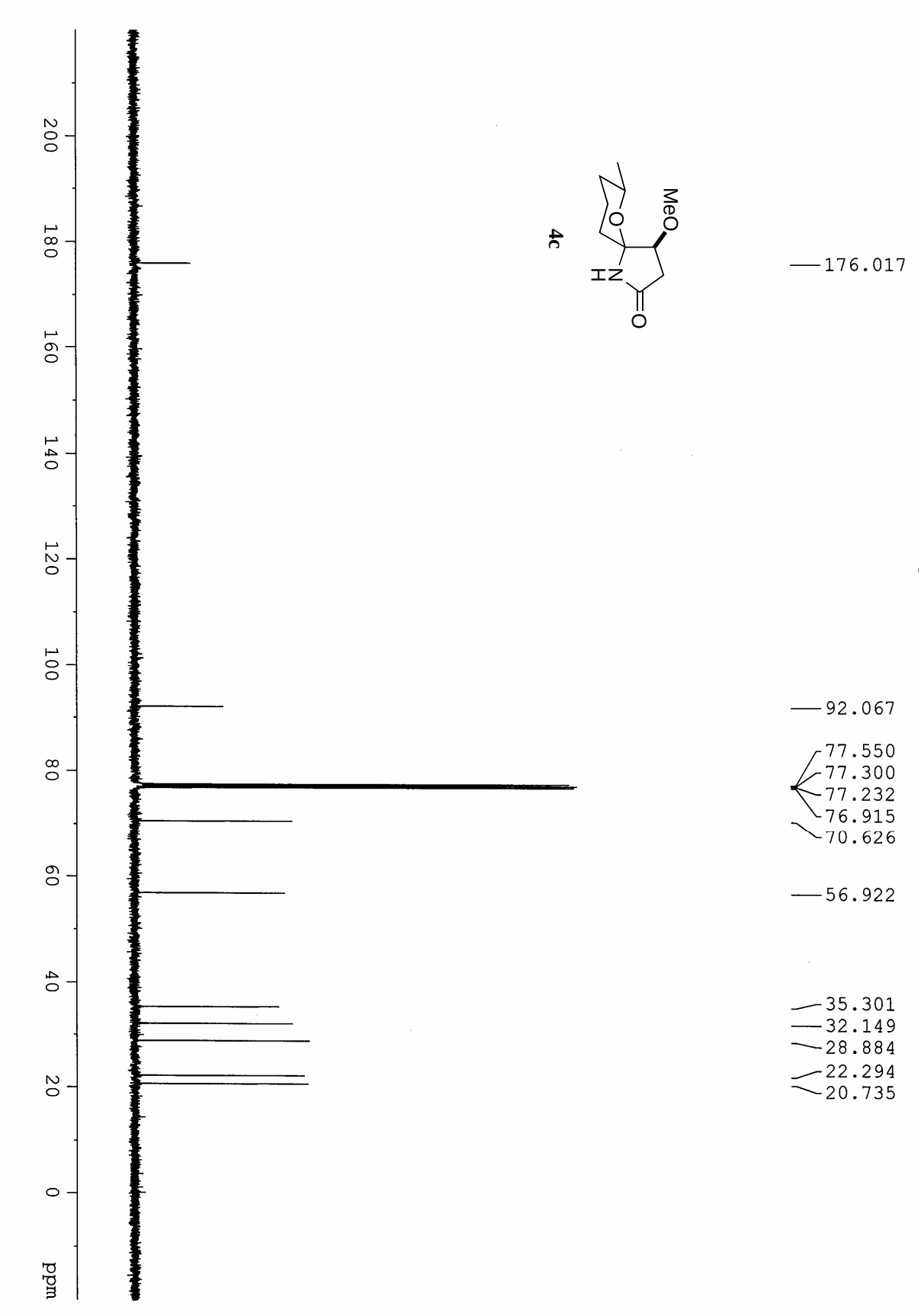


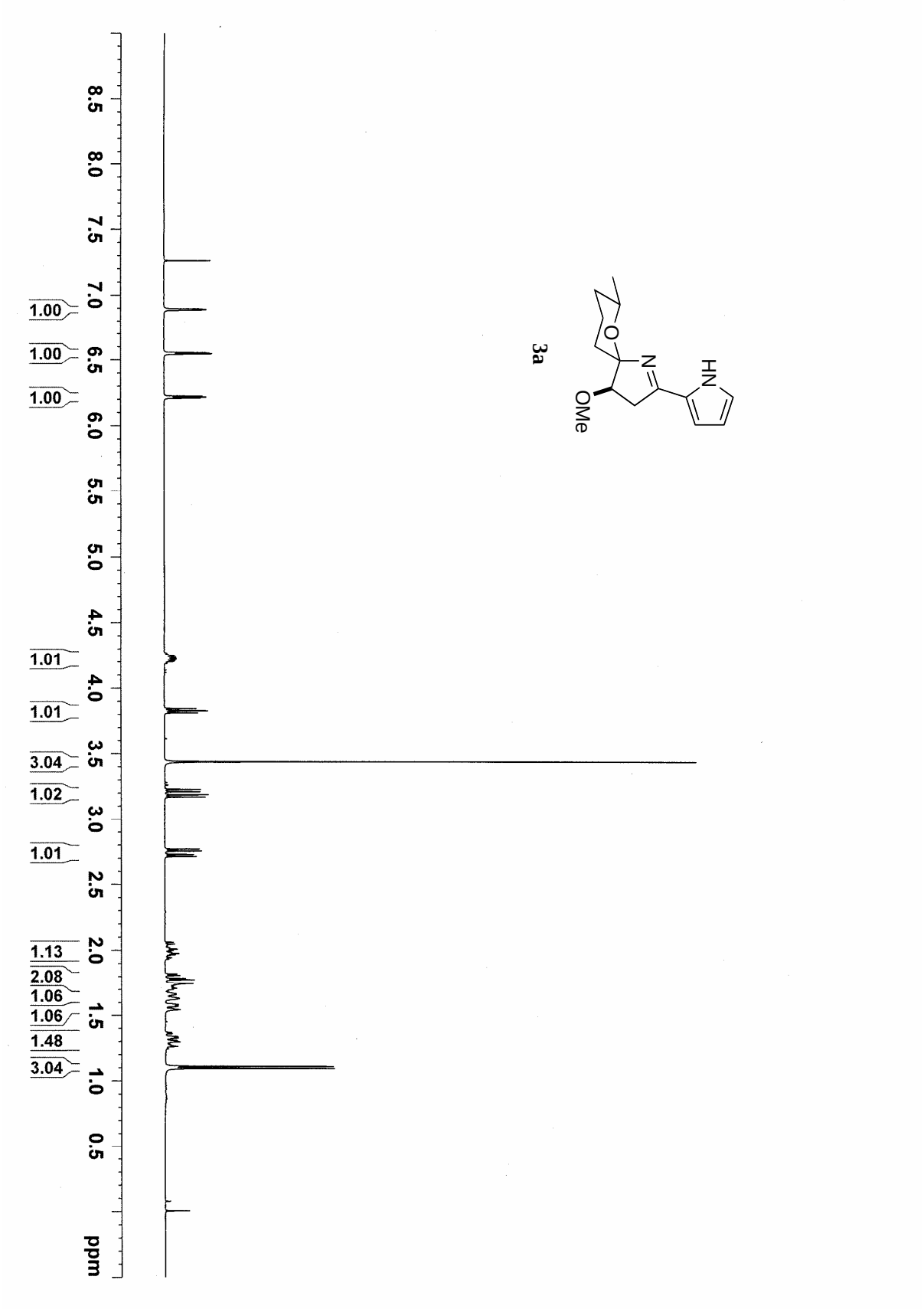












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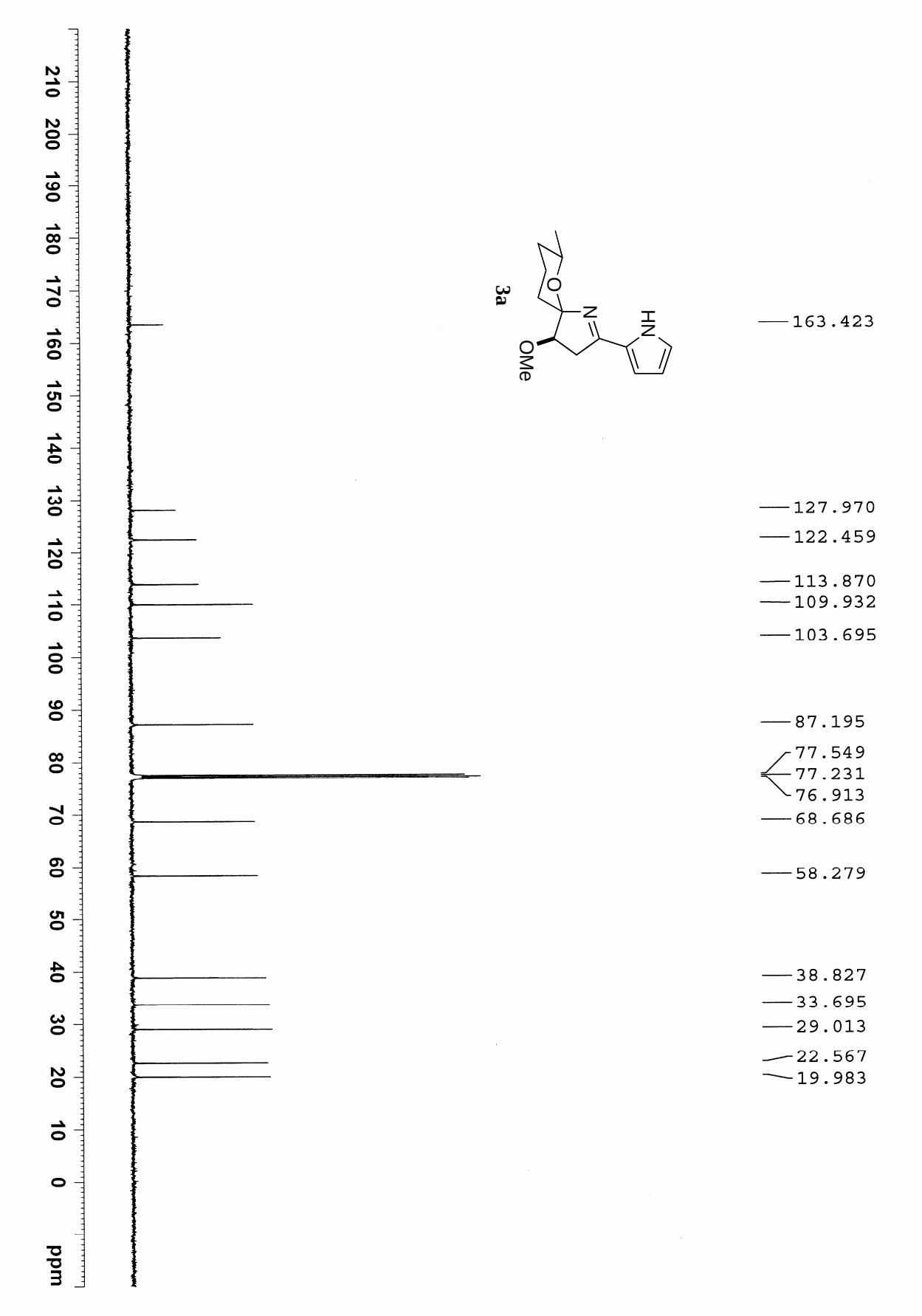

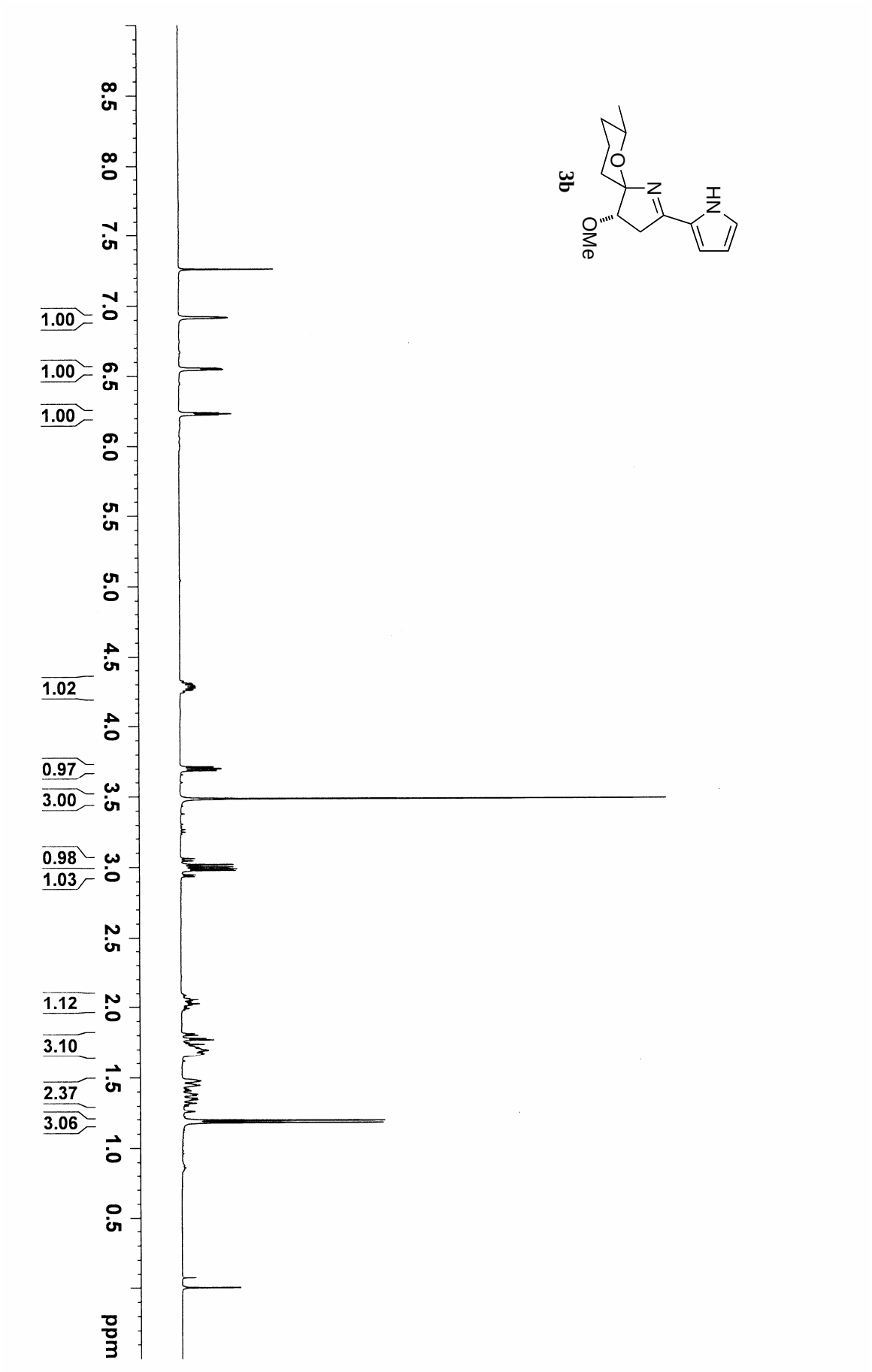

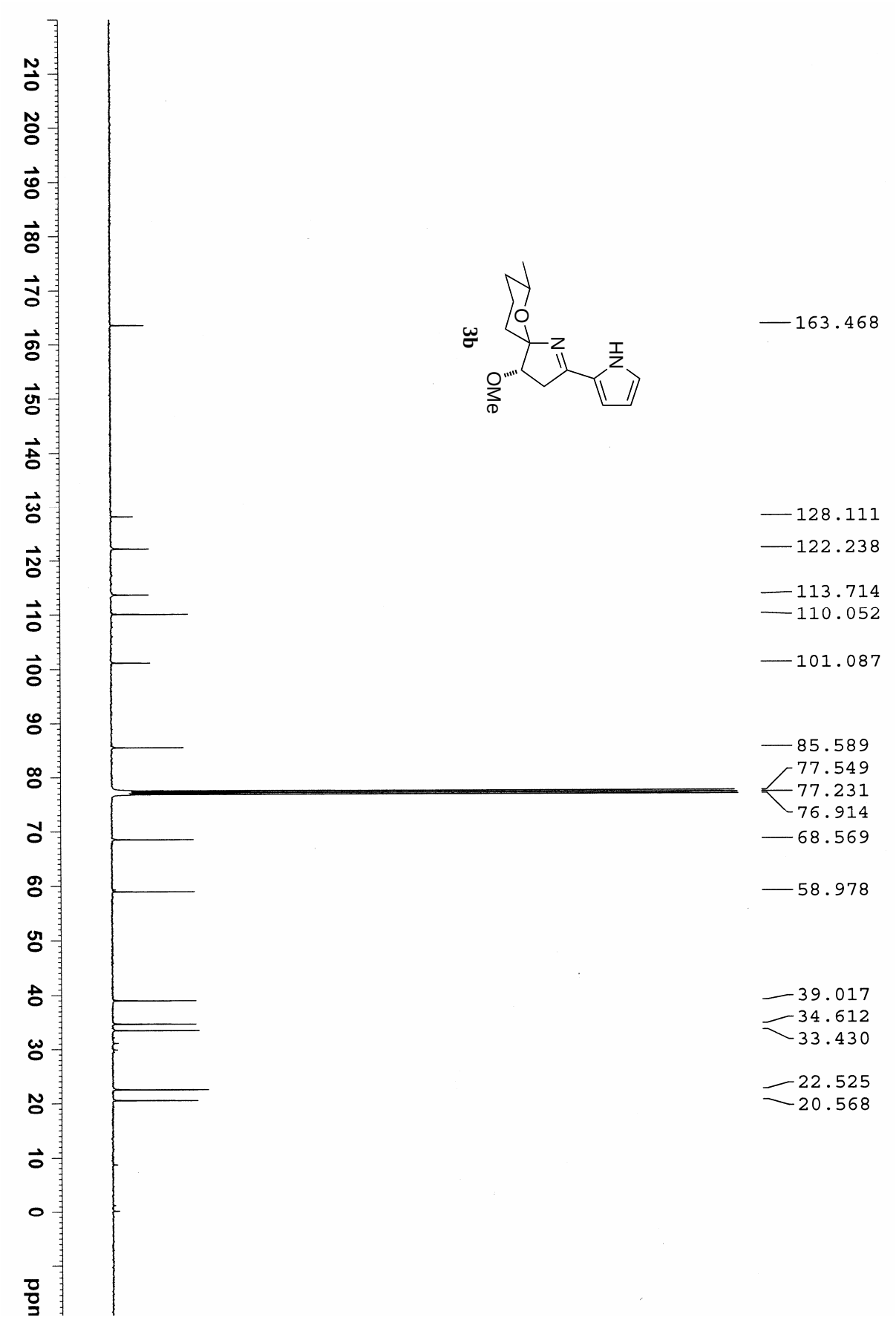

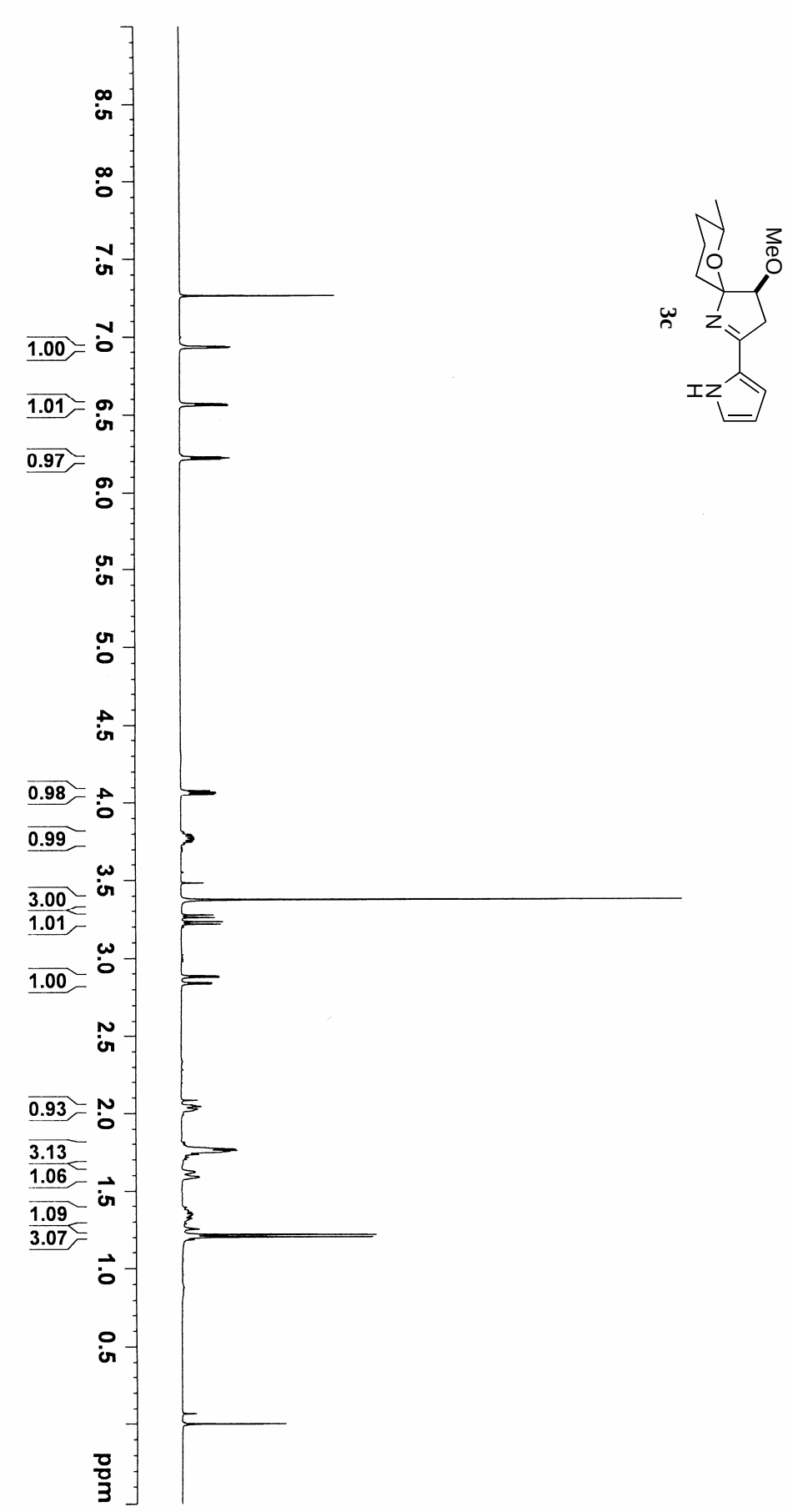

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