Supplementary Section

Synthesis and immunological effects of heroin vaccines containing haptens with improved stability

Fuying Li,[a] Kejun Cheng,[a] Joshua F. G. Antoline,[a] Malliga R. Iyer,[a] Gary R. Matyas,[b] Oscar Torres,[b,c] Rashmi Jalah,[b,c] Zoltan Beck,[b,c] Carl R. Alving,[b] Damon A. Parrish,[d] Jeffrey R. Deschamps,[d] Arthur E. Jacobson,[a] Kenner C. Rice[a]*

[a] *Drug Design and Synthesis Section, Chemical Biology Research Branch, National Institute on Drug Abuse and the National Institute on Alcohol Abuse and Alcoholism, National Institutes of Health, Department of Health and Human Services, 9800 Medical Center Drive, Bethesda, MD 20892, USA,*

[b] *Laboratory of Adjuvant and Antigen Research, U.S. Military HIV Research Program, Walter Reed Army Institute of Research, 503 Robert Grant Avenue, Silver Spring, MD 20910, USA,*

[c] *U.S. Military HIV Research Program, Henry M. Jackson Foundation for the Advancement of Military Medicine, 6720A Rockledge Drive, Bethesda, MD 20817 USA*

[d] *Center for Biomolecular Science and Engineering, Naval Research Laboratory, Washington DC 20375, USA*

Single-crystal X-ray diffraction data on compound 8b

Table 1. Crystal data and structure refinement for **8b (knih99)**.

Identification code	KNIH099	
Empirical formula	$C_{17}H_{17}ClN_2O_4$	
Formula weight	348.78	
Temperature	150(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	$P2_12_12_1$	
Unit cell dimensions	a = 7.3581(12) Å	α= 90°.
	b = 9.1360(16) Å	β= 90°.
	c = 23.005(4) Å	γ = 90°.
Volume	1546.5(5) $Å^3$	
Z	4	
Density (calculated)	1.498 Mg/m^3	
Absorption coefficient	0.272 mm^{-1}	
F(000)	728	
Crystal size	0.51 x 0.43 x 0.01 mm^3	
Theta range for data collection	1.77 to 26.48°.	
Index ranges	-8<=h<=9, -11<=k<=11, -28<=l<=28	
Reflections collected	13822	
Independent reflections	3198 [R_{int} = 0.0355]	
Completeness to theta = 26.48°	99.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9973 and 0.8736	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3198 / 0 / 218	
Goodness-of-fit on F^2	1.045	
Final R indices [I>2sigma(I)]	R_1 = 0.0284, wR_2 = 0.0680	
R indices (all data)	R_1 = 0.0329, wR_2 = 0.0702	
Absolute structure parameter	-0.03(5)	
Largest diff. peak and hole	0.218 and -0.177 e.$Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **8b (knih 99)**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	7316(2)	2923(2)	8954(1)	25(1)
Cl(1A)	6685(1)	1451(1)	8518(1)	40(1)
C(2)	6919(2)	2874(2)	9539(1)	26(1)
C(3)	7489(2)	4007(2)	9906(1)	24(1)
N(3A)	7084(2)	3911(2)	10523(1)	28(1)
O(3A)	6447(2)	2769(2)	10709(1)	48(1)
O(3B)	7394(2)	4995(2)	10824(1)	38(1)
C(4)	8462(2)	5166(2)	9670(1)	20(1)
C(5)	10589(2)	6881(2)	9521(1)	20(1)
O(5A)	9286(2)	6311(1)	9945(1)	22(1)
C(6)	12394(2)	6068(2)	9598(1)	23(1)
O(6A)	12744(2)	5396(1)	10036(1)	29(1)
C(7)	13621(2)	6098(2)	9078(1)	27(1)
C(8)	12626(2)	5374(2)	8564(1)	27(1)
C(9)	9781(2)	5674(2)	7913(1)	28(1)
C(10)	8844(2)	4206(2)	8077(1)	27(1)
C(11)	8251(2)	4101(2)	8700(1)	21(1)
C(12)	8737(2)	5202(2)	9076(1)	18(1)
C(13)	9724(2)	6589(2)	8919(1)	19(1)
C(14)	10991(2)	6299(2)	8400(1)	23(1)
C(15)	8365(2)	7788(2)	8738(1)	24(1)
C(16)	7327(3)	7333(2)	8194(1)	29(1)
N(17)	8536(2)	6844(2)	7730(1)	30(1)
C(17A)	7494(3)	6457(3)	7211(1)	45(1)

Table 3. Bond lengths [Å] and angles [°] for **8b (knih 99)**.

C(1)-C(2)	1.377(2)	C(1)-C(11)	1.405(2)
C(1)-Cl(1A)	1.7415(17)	C(2)-C(3)	1.399(2)
C(2)-H(2)	0.9500	C(3)-C(4)	1.388(2)
C(3)-N(3A)	1.454(2)	N(3A)-O(3A)	1.2218(19)
N(3A)-O(3B)	1.230(2)	C(4)-O(5A)	1.3640(19)
C(4)-C(12)	1.382(2)	C(5)-O(5A)	1.4643(18)
C(5)-C(6)	1.532(2)	C(5)-C(13)	1.546(2)
C(5)-H(5)	1.0000	C(6)-O(6A)	1.2072(19)
C(6)-C(7)	1.500(2)	C(7)-C(8)	1.540(2)
C(7)-H(7A)	0.9900	C(7)-H(7B)	0.9900
C(8)-C(14)	1.518(2)	C(8)-H(8A)	0.9900
C(8)-H(8B)	0.9900	C(9)-N(17)	1.469(2)
C(9)-C(14)	1.540(2)	C(9)-C(10)	1.554(2)
C(9)-H(9)	1.0000	C(10)-C(11)	1.502(2)
C(10)-H(10A)	0.9900	C(10)-H(10B)	0.9900
C(11)-C(12)	1.374(2)	C(12)-C(13)	1.504(2)
C(13)-C(14)	1.539(2)	C(13)-C(15)	1.541(2)
C(14)-H(14)	1.0000	C(15)-C(16)	1.524(2)
C(15)-H(15A)	0.9900	C(15)-H(15B)	0.9900
C(16)-N(17)	1.460(2)	C(16)-H(16A)	0.9900
C(16)-H(16B)	0.9900	N(17)-C(17A)	1.464(2)
C(17A)-H(17A)	0.9800	C(17A)-H(17B)	0.9800
C(17A)-H(17C)	0.9800		

C(2)-C(1)-C(11)	122.41(15)	C(2)-C(1)-Cl(1A)	118.77(13)
C(11)-C(1)-Cl(1A)	118.81(13)	C(1)-C(2)-C(3)	120.04(15)
C(1)-C(2)-H(2)	120.0	C(3)-C(2)-H(2)	120.0
C(4)-C(3)-C(2)	118.96(14)	C(4)-C(3)-N(3A)	122.16(15)
C(2)-C(3)-N(3A)	118.86(15)	O(3A)-N(3A)-O(3B)	124.14(14)
O(3A)-N(3A)-C(3)	118.24(15)	O(3B)-N(3A)-C(3)	117.62(13)
O(5A)-C(4)-C(12)	112.01(13)	O(5A)-C(4)-C(3)	129.33(14)
C(12)-C(4)-C(3)	118.62(14)	O(5A)-C(5)-C(6)	108.54(12)
O(5A)-C(5)-C(13)	105.39(12)	C(6)-C(5)-C(13)	112.18(12)
O(5A)-C(5)-H(5)	110.2	C(6)-C(5)-H(5)	110.2
C(13)-C(5)-H(5)	110.2	C(4)-O(5A)-C(5)	104.78(11)
O(6A)-C(6)-C(7)	123.17(16)	O(6A)-C(6)-C(5)	121.90(15)
C(7)-C(6)-C(5)	114.83(13)	C(6)-C(7)-C(8)	108.60(13)
C(6)-C(7)-H(7A)	110.0	C(8)-C(7)-H(7A)	110.0
C(6)-C(7)-H(7B)	110.0	C(8)-C(7)-H(7B)	110.0
H(7A)-C(7)-H(7B)	108.4	C(14)-C(8)-C(7)	109.19(13)
C(14)-C(8)-H(8A)	109.8	C(7)-C(8)-H(8A)	109.8
C(14)-C(8)-H(8B)	109.8	C(7)-C(8)-H(8B)	109.8
H(8A)-C(8)-H(8B)	108.3	N(17)-C(9)-C(14)	107.34(14)

Table 3. Continued

N(17)-C(9)-C(10)	114.91(14)	C(14)-C(9)-C(10)	113.60(13)

N(17)-C(9)-H(9)	106.8	C(14)-C(9)-H(9)	106.8
C(10)-C(9)-H(9)	106.8	C(11)-C(10)-C(9)	114.50(13)
C(11)-C(10)-H(10A)	108.6	C(9)-C(10)-H(10A)	108.6
C(11)-C(10)-H(10B)	108.6	C(9)-C(10)-H(10B)	108.6
H(10A)-C(10)-H(10B)	107.6	C(12)-C(11)-C(1)	115.18(14)
C(12)-C(11)-C(10)	118.65(14)	C(1)-C(11)-C(10)	126.06(14)
C(11)-C(12)-C(4)	124.57(14)	C(11)-C(12)-C(13)	126.21(14)
C(4)-C(12)-C(13)	109.15(13)	C(12)-C(13)-C(14)	109.51(13)
C(12)-C(13)-C(15)	110.49(13)	C(14)-C(13)-C(15)	107.78(12)
C(12)-C(13)-C(5)	97.44(11)	C(14)-C(13)-C(5)	118.40(13)
C(15)-C(13)-C(5)	112.74(13)	C(8)-C(14)-C(13)	112.49(13)
C(8)-C(14)-C(9)	115.62(14)	C(13)-C(14)-C(9)	106.18(13)
C(8)-C(14)-H(14)	107.4	C(13)-C(14)-H(14)	107.4
C(9)-C(14)-H(14)	107.4	C(16)-C(15)-C(13)	110.70(13)
C(16)-C(15)-H(15A)	109.5	C(13)-C(15)-H(15A)	109.5
C(16)-C(15)-H(15B)	109.5	C(13)-C(15)-H(15B)	109.5
H(15A)-C(15)-H(15B)	108.1	N(17)-C(16)-C(15)	112.26(15)
N(17)-C(16)-H(16A)	109.2	C(15)-C(16)-H(16A)	109.2
N(17)-C(16)-H(16B)	109.2	C(15)-C(16)-H(16B)	109.2
H(16A)-C(16)-H(16B)	107.9	C(16)-N(17)-C(17A)	110.63(16)
C(16)-N(17)-C(9)	113.15(13)	C(17A)-N(17)-C(9)	112.58(15)
N(17)-C(17A)-H(17A)	109.5	N(17)-C(17A)-H(17B)	109.5
H(17A)-C(17A)-H(17B)	109.5	N(17)-C(17A)-H(17C)	109.5
H(17A)-C(17A)-H(17C)	109.5	H(17B)-C(17A)-H(17C)	109.5

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **8b (knih 99)**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	21(1)	20(1)	35(1)	-1(1)	-6(1)	0(1)
Cl(1A)	43(1)	25(1)	52(1)	-5(1)	-12(1)	-9(1)
C(2)	18(1)	23(1)	38(1)	10(1)	0(1)	-2(1)
C(3)	19(1)	26(1)	26(1)	9(1)	1(1)	4(1)
N(3A)	23(1)	33(1)	29(1)	13(1)	6(1)	3(1)
O(3A)	56(1)	45(1)	42(1)	19(1)	10(1)	-12(1)
O(3B)	48(1)	40(1)	27(1)	5(1)	8(1)	2(1)
C(4)	14(1)	21(1)	23(1)	1(1)	1(1)	3(1)
C(5)	20(1)	21(1)	21(1)	-1(1)	4(1)	-2(1)
O(5A)	22(1)	24(1)	20(1)	-1(1)	4(1)	-2(1)
C(6)	20(1)	21(1)	27(1)	-4(1)	-4(1)	-4(1)
O(6A)	30(1)	29(1)	28(1)	2(1)	-6(1)	0(1)
C(7)	15(1)	35(1)	30(1)	-3(1)	-1(1)	-1(1)
C(8)	20(1)	34(1)	27(1)	-7(1)	6(1)	-1(1)
C(9)	24(1)	38(1)	20(1)	-2(1)	3(1)	-6(1)
C(10)	26(1)	30(1)	26(1)	-8(1)	0(1)	-4(1)
C(11)	16(1)	22(1)	26(1)	-2(1)	-2(1)	2(1)
C(12)	12(1)	20(1)	23(1)	3(1)	2(1)	2(1)
C(13)	17(1)	20(1)	19(1)	1(1)	0(1)	-1(1)
C(14)	22(1)	27(1)	21(1)	-1(1)	4(1)	-5(1)
C(15)	22(1)	22(1)	28(1)	5(1)	0(1)	1(1)
C(16)	26(1)	32(1)	30(1)	7(1)	-4(1)	0(1)
N(17)	30(1)	39(1)	21(1)	5(1)	-5(1)	-8(1)
C(17A)	50(1)	57(1)	27(1)	7(1)	-13(1)	-15(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **8b (knih 99)**.

	x	y	z	U(eq)
H(2)	6258	2070	9694	31
H(5)	10769	7956	9580	24
H(7A)	14759	5561	9163	32
H(7B)	13937	7122	8979	32
H(8A)	13458	5290	8227	32
H(8B)	12225	4378	8674	32
H(9)	10595	5465	7575	33
H(10A)	7767	4071	7824	33
H(10B)	9697	3393	7994	33
H(14)	11457	7268	8263	28
H(15A)	7497	7966	9059	29
H(15B)	9028	8711	8662	29
H(16A)	6477	6531	8294	35
H(16B)	6599	8173	8054	35
H(17A)	6867	7327	7063	67
H(17B)	6598	5703	7309	67
H(17C)	8320	6081	6912	67

Table 6. Torsion angles [°] for **8b (knih 99)**.

C(11)-C(1)-C(2)-C(3)	-1.5(3)
Cl(1A)-C(1)-C(2)-C(3)	176.84(13)
C(1)-C(2)-C(3)-C(4)	-0.4(2)
C(1)-C(2)-C(3)-N(3A)	-178.70(15)
C(4)-C(3)-N(3A)-O(3A)	-170.04(16)
C(2)-C(3)-N(3A)-O(3A)	8.2(2)
C(4)-C(3)-N(3A)-O(3B)	10.5(2)
C(2)-C(3)-N(3A)-O(3B)	-171.18(15)
C(2)-C(3)-C(4)-O(5A)	-173.58(15)
N(3A)-C(3)-C(4)-O(5A)	4.7(3)
C(2)-C(3)-C(4)-C(12)	3.9(2)
N(3A)-C(3)-C(4)-C(12)	-177.86(14)
C(12)-C(4)-O(5A)-C(5)	-16.56(17)
C(3)-C(4)-O(5A)-C(5)	161.02(16)
C(6)-C(5)-O(5A)-C(4)	-89.15(14)
C(13)-C(5)-O(5A)-C(4)	31.19(15)
O(5A)-C(5)-C(6)-O(6A)	-18.1(2)
C(13)-C(5)-C(6)-O(6A)	-134.09(15)
O(5A)-C(5)-C(6)-C(7)	158.31(13)
C(13)-C(5)-C(6)-C(7)	42.27(18)
O(6A)-C(6)-C(7)-C(8)	116.09(17)
C(5)-C(6)-C(7)-C(8)	-60.22(18)
C(6)-C(7)-C(8)-C(14)	65.36(18)
N(17)-C(9)-C(10)-C(11)	-87.47(18)
C(14)-C(9)-C(10)-C(11)	36.7(2)
C(2)-C(1)-C(11)-C(12)	-0.2(2)
Cl(1A)-C(1)-C(11)-C(12)	-178.54(12)
C(2)-C(1)-C(11)-C(10)	175.90(17)
Cl(1A)-C(1)-C(11)-C(10)	-2.5(2)
C(9)-C(10)-C(11)-C(12)	-6.6(2)
C(9)-C(10)-C(11)-C(1)	177.43(15)
C(1)-C(11)-C(12)-C(4)	4.0(2)
C(10)-C(11)-C(12)-C(4)	-172.38(15)
C(1)-C(11)-C(12)-C(13)	-179.30(14)
C(10)-C(11)-C(12)-C(13)	4.3(2)
O(5A)-C(4)-C(12)-C(11)	171.92(14)
C(3)-C(4)-C(12)-C(11)	-5.9(2)
O(5A)-C(4)-C(12)-C(13)	-5.27(18)
C(3)-C(4)-C(12)-C(13)	176.87(14)
C(11)-C(12)-C(13)-C(14)	-30.5(2)
C(4)-C(12)-C(13)-C(14)	146.62(13)
C(11)-C(12)-C(13)-C(15)	88.06(18)
C(4)-C(12)-C(13)-C(15)	-94.81(15)

Table 6. Continued

C(11)-C(12)-C(13)-C(5)	-154.23(15)
C(4)-C(12)-C(13)-C(5)	22.90(15)
O(5A)-C(5)-C(13)-C(12)	-32.07(14)
C(6)-C(5)-C(13)-C(12)	85.85(14)
O(5A)-C(5)-C(13)-C(14)	-149.04(13)
C(6)-C(5)-C(13)-C(14)	-31.12(19)
O(5A)-C(5)-C(13)-C(15)	83.87(15)
C(6)-C(5)-C(13)-C(15)	-158.20(13)
C(7)-C(8)-C(14)-C(13)	-54.37(18)
C(7)-C(8)-C(14)-C(9)	-176.58(13)
C(12)-C(13)-C(14)-C(8)	-71.70(16)
C(15)-C(13)-C(14)-C(8)	168.05(13)
C(5)-C(13)-C(14)-C(8)	38.64(19)
C(12)-C(13)-C(14)-C(9)	55.70(16)
C(15)-C(13)-C(14)-C(9)	-64.55(16)
C(5)-C(13)-C(14)-C(9)	166.04(13)
N(17)-C(9)-C(14)-C(8)	-167.84(13)
C(10)-C(9)-C(14)-C(8)	63.99(18)
N(17)-C(9)-C(14)-C(13)	66.65(16)
C(10)-C(9)-C(14)-C(13)	-61.52(18)
C(12)-C(13)-C(15)-C(16)	-62.96(17)
C(14)-C(13)-C(15)-C(16)	56.67(17)
C(5)-C(13)-C(15)-C(16)	-170.80(13)
C(13)-C(15)-C(16)-N(17)	-50.63(19)
C(15)-C(16)-N(17)-C(17A)	-178.40(15)
C(15)-C(16)-N(17)-C(9)	54.21(19)
C(14)-C(9)-N(17)-C(16)	-62.14(17)
C(10)-C(9)-N(17)-C(16)	65.27(18)
C(14)-C(9)-N(17)-C(17A)	171.50(14)
C(10)-C(9)-N(17)-C(17A)	-61.09(19)

Single-crystal X-ray diffraction data on compound 13a

Table 1. Crystal data and structure refinement for **13a (knih86)**.

Empirical formula	$C_{18}H_{23}NO_2$	
Formula weight	285.37	
Temperature	175(2) K	
Wavelength	1.54178 Å	
Crystal system	Orthorhombic	
Space group	P $2_12_12_1$	
Unit cell dimensions	a = 6.8411(3) Å	α= 90°
	b = 12.9417(6) Å	β= 90°
	c = 16.7006(8) Å	γ = 90°
Volume	1478.60(12) $Å^3$	
Z	4	
Density (calculated)	1.282 Mg/m^3	
Absorption coefficient	0.653 mm^{-1}	
F(000)	616	
Crystal size	0.432 x 0.307 x 0.207 mm^3	
θ range for data collection	4.32 to 67.85°	
Index ranges	-8<=h<=5, -13<=k<=13, -13<=l<=15	
Reflections collected	4592	
Independent reflections	1649 [R(int) = 0.1088]	
Completeness to θ = 67.85°	74.1 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.8767 and 0.7656	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	1649 / 0 / 192	
Goodness-of-fit on F^2	1.106	
Final R indices [I>2σ(I)]	R1 = 0.0540, wR2 = 0.1320	
R indices (all data)	R1 = 0.0541, wR2 = 0.1320	
Absolute structure parameter	0.9(4)	
Largest diff. peak and hole	0.370 and -0.433 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for **13a (knih86)**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	10737(4)	-2028(2)	9255(2)	34(1)
C(2)	9394(4)	-2278(2)	8665(2)	36(1)
C(3)	7840(4)	-1632(2)	8451(2)	36(1)
C(4)	7651(4)	-734(2)	8891(2)	30(1)
O(5)	6396(2)	77(2)	8734(1)	35(1)
C(5)	7045(4)	964(2)	9230(2)	27(1)
C(6)	8044(3)	1783(2)	8711(2)	25(1)
C(6A)	6841(4)	2092(2)	7983(2)	29(1)
O(6)	4966(2)	2483(2)	8191(1)	30(1)
C(7)	10102(3)	1429(2)	8473(2)	30(1)
C(8)	11453(4)	1247(2)	9193(2)	29(1)
C(9)	11568(3)	421(2)	10582(2)	27(1)
C(10)	11997(4)	-691(2)	10269(2)	31(1)
C(11)	10493(4)	-1114(2)	9697(2)	28(1)
C(12)	8876(3)	-523(2)	9528(2)	24(1)
C(13)	8409(3)	517(2)	9878(2)	24(1)
C(14)	10370(3)	1073(2)	9988(2)	25(1)
C(15)	7369(3)	441(2)	10691(2)	27(1)
C(16)	8644(3)	-49(2)	11335(2)	28(1)
N(17)	10566(3)	457(2)	11367(1)	27(1)
C(17)	11743(4)	36(2)	12024(2)	36(1)

Table 3. Bond lengths [Å] and angles [°] for **13a (knih86)**.

C(1)-C(2)	1.385(4)	C(1)-C(11)	1.404(4)
C(1)-H(1A)	0.9500	C(2)-C(3)	1.399(4)
C(2)-H(2A)	0.9500	C(3)-C(4)	1.381(4)
C(3)-H(3A)	0.9500	C(4)-C(12)	1.382(4)
C(4)-O(5)	1.381(3)	O(5)-C(5)	1.484(3)
C(5)-C(6)	1.530(4)	C(5)-C(13)	1.541(3)
C(5)-H(5A)	1.0000	C(6)-C(6A)	1.522(4)
C(6)-C(7)	1.533(3)	C(6)-H(6A)	1.0000
C(6A)-O(6)	1.422(3)	C(6A)-H(6AA)	0.9900
C(6A)-H(6AB)	0.9900	O(6)-H(6B)	0.8400
C(7)-C(8)	1.535(4)	C(7)-H(7A)	0.9900
C(7)-H(7B)	0.9900	C(8)-C(14)	1.536(4)
C(8)-H(8A)	0.9900	C(8)-H(8B)	0.9900
C(9)-N(17)	1.480(4)	C(9)-C(14)	1.540(4)
C(9)-C(10)	1.559(4)	C(9)-H(9A)	1.0000
C(10)-C(11)	1.507(4)	C(10)-H(10A)	0.9900
C(10)-H(10B)	0.9900	C(11)-C(12)	1.374(4)
C(12)-C(13)	1.501(4)	C(13)-C(15)	1.537(4)
C(13)-C(14)	1.534(3)	C(14)-H(14A)	1.0000
C(15)-C(16)	1.524(4)	C(15)-H(15A)	0.9900
C(15)-H(15B)	0.9900	C(16)-N(17)	1.469(3)
C(16)-H(16A)	0.9900	C(16)-H(16B)	0.9900
N(17)-C(17)	1.465(3)	C(17)-H(17A)	0.9800
C(17)-H(17B)	0.9800	C(17)-H(17C)	0.9800
C(2)-C(1)-C(11)	119.5(3)	C(2)-C(1)-H(1A)	120.3
C(11)-C(1)-H(1A)	120.3	C(1)-C(2)-C(3)	123.0(3)
C(1)-C(2)-H(2A)	118.5	C(3)-C(2)-H(2A)	118.5
C(4)-C(3)-C(2)	116.0(3)	C(4)-C(3)-H(3A)	122.0
C(2)-C(3)-H(3A)	122.0	C(12)-C(4)-C(3)	121.2(3)
C(12)-C(4)-O(5)	111.9(3)	C(3)-C(4)-O(5)	126.6(3)
C(4)-O(5)-C(5)	107.17(19)	O(5)-C(5)-C(6)	110.7(2)
O(5)-C(5)-C(13)	106.4(2)	C(6)-C(5)-C(13)	112.8(2)
O(5)-C(5)-H(5A)	109.0	C(6)-C(5)-H(5A)	109.0
C(13)-C(5)-H(5A)	109.0	C(6A)-C(6)-C(5)	113.2(2)
C(6A)-C(6)-C(7)	111.6(2)	C(5)-C(6)-C(7)	110.5(2)
C(6A)-C(6)-H(6A)	107.1	C(5)-C(6)-H(6A)	107.1
C(7)-C(6)-H(6A)	107.1	O(6)-C(6A)-C(6)	112.7(2)
O(6)-C(6A)-H(6AA)	109.0	C(6)-C(6A)-H(6AA)	109.0
O(6)-C(6A)-H(6AB)	109.0	C(6)-C(6A)-H(6AB)	109.0
H(6AA)-C(6A)-H(6AB)	107.8	C(6A)-O(6)-H(6B)	109.5
C(6)-C(7)-C(8)	113.3(2)	C(6)-C(7)-H(7A)	108.9
C(8)-C(7)-H(7A)	108.9	C(6)-C(7)-H(7B)	108.9
C(8)-C(7)-H(7B)	108.9	H(7A)-C(7)-H(7B)	107.7
C(14)-C(8)-C(7)	114.1(2)	C(14)-C(8)-H(8A)	108.7
C(7)-C(8)-H(8A)	108.7	C(14)-C(8)-H(8B)	108.7
C(7)-C(8)-H(8B)	108.7	H(8A)-C(8)-H(8B)	107.6
N(17)-C(9)-C(14)	107.91(19)	N(17)-C(9)-C(10)	114.4(2)
C(14)-C(9)-C(10)	112.9(2)	N(17)-C(9)-H(9A)	107.0
C(14)-C(9)-H(9A)	107.0	C(10)-C(9)-H(9A)	107.0
C(11)-C(10)-C(9)	114.8(2)	C(11)-C(10)-H(10A)	108.6
C(9)-C(10)-H(10A)	108.6	C(11)-C(10)-H(10B)	108.6
C(9)-C(10)-H(10B)	108.6	H(10A)-C(10)-H(10B)	107.5

Table 3. (continued)

C(12)-C(11)-C(1)	117.2(3)	C(12)-C(11)-C(10)	118.5(2)
C(1)-C(11)-C(10)	124.0(2)	C(11)-C(12)-C(4)	122.4(3)
C(11)-C(12)-C(13)	126.2(2)	C(4)-C(12)-C(13)	110.3(2)
C(12)-C(13)-C(15)	112.7(2)	C(12)-C(13)-C(14)	106.33(19)
C(15)-C(13)-C(14)	109.2(2)	C(12)-C(13)-C(5)	101.1(2)
C(15)-C(13)-C(5)	111.33(19)	C(14)-C(13)-C(5)	115.9(2)
C(13)-C(14)-C(8)	112.8(2)	C(13)-C(14)-C(9)	106.6(2)
C(8)-C(14)-C(9)	112.4(2)	C(13)-C(14)-H(14A)	108.3
C(8)-C(14)-H(14A)	108.3	C(9)-C(14)-H(14A)	108.3
C(16)-C(15)-C(13)	112.7(2)	C(16)-C(15)-H(15A)	109.1
C(13)-C(15)-H(15A)	109.1	C(16)-C(15)-H(15B)	109.1
C(13)-C(15)-H(15B)	109.1	H(15A)-C(15)-H(15B)	107.8
N(17)-C(16)-C(15)	110.7(2)	N(17)-C(16)-H(16A)	109.5
C(15)-C(16)-H(16A)	109.5	N(17)-C(16)-H(16B)	109.5
C(15)-C(16)-H(16B)	109.5	H(16A)-C(16)-H(16B)	108.1
C(17)-N(17)-C(16)	110.7(2)	C(17)-N(17)-C(9)	113.4(2)
C(16)-N(17)-C(9)	111.6(2)	N(17)-C(17)-H(17A)	109.5
N(17)-C(17)-H(17B)	109.5	H(17A)-C(17)-H(17B)	109.5
N(17)-C(17)-H(17C)	109.5	H(17A)-C(17)-H(17C)	109.5
H(17B)-C(17)-H(17C)	109.5		

Table 4. Anisotropic displacement parameters ($Å^2$ x 10^3) for **13a (knih86)**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	29(1)	30(2)	43(2)	-2(1)	5(1)	0(1)
C(2)	40(1)	34(2)	34(2)	-7(1)	5(1)	-10(1)
C(3)	39(2)	39(2)	30(2)	0(1)	-5(1)	-12(1)
C(4)	24(1)	35(2)	30(2)	4(1)	-3(1)	-6(1)
O(5)	26(1)	38(1)	42(2)	1(1)	-14(1)	-6(1)
C(5)	14(1)	36(2)	30(2)	-2(1)	-2(1)	-1(1)
C(6)	17(1)	31(1)	27(2)	-3(1)	-1(1)	2(1)
C(6A)	28(1)	35(2)	26(2)	-1(1)	-1(1)	6(1)
O(6)	19(1)	42(1)	30(1)	4(1)	-6(1)	2(1)
C(7)	19(1)	37(2)	34(2)	2(1)	7(1)	1(1)
C(8)	14(1)	37(2)	36(2)	2(1)	2(1)	-3(1)
C(9)	14(1)	34(2)	31(2)	-2(1)	0(1)	-1(1)
C(10)	18(1)	37(2)	37(2)	-2(1)	-3(1)	5(1)
C(11)	21(1)	32(2)	31(2)	0(1)	0(1)	-1(1)
C(12)	19(1)	30(2)	24(2)	2(1)	0(1)	-4(1)
C(13)	11(1)	31(1)	29(2)	2(1)	0(1)	-3(1)
C(14)	12(1)	31(2)	31(2)	1(1)	0(1)	-2(1)
C(15)	14(1)	36(2)	30(2)	2(1)	1(1)	-2(1)
C(16)	19(1)	33(2)	33(2)	2(1)	-1(1)	-3(1)
N(17)	17(1)	38(1)	27(2)	2(1)	-5(1)	0(1)
C(17)	30(1)	50(2)	27(2)	5(1)	-11(1)	7(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for **13a (knih86)**.

	x	y	z	U(eq)
H(1A)	11815	-2470	9358	41
H(2A)	9534	-2919	8394	43
H(3A)	6968	-1801	8029	43
H(5A)	5879	1279	9496	32
H(6A)	8198	2415	9050	30
H(6AA)	7561	2626	7677	35
H(6AB)	6677	1483	7631	35
H(6B)	4379	2673	7774	45
H(7A)	10696	1959	8121	36
H(7B)	9999	780	8162	36
H(8A)	12283	637	9083	35
H(8B)	12328	1852	9253	35
H(9A)	12858	773	10651	32
H(10A)	13284	-688	9997	37
H(10B)	12094	-1163	10734	37
H(14A)	10112	1762	10237	30
H(15A)	6982	1143	10866	32
H(15B)	6163	27	10628	32
H(16A)	7992	16	11862	34
H(16B)	8816	-793	11219	34
H(17A)	11094	178	12535	53
H(17B)	13038	361	12021	53
H(17C)	11886	-712	11955	53

Table 6. Torsion angles [°] for **13a (knih86)**.

C(11)-C(1)-C(2)-C(3)	3.8(4)	C(1)-C(2)-C(3)-C(4)	-3.0(4)
C(2)-C(3)-C(4)-C(12)	-3.1(4)	C(2)-C(3)-C(4)-O(5)	171.6(3)
C(12)-C(4)-O(5)-C(5)	9.1(3)	C(3)-C(4)-O(5)-C(5)	-166.0(3)
C(4)-O(5)-C(5)-C(6)	106.4(2)	C(4)-O(5)-C(5)-C(13)	-16.5(3)
O(5)-C(5)-C(6)-C(6A)	50.8(3)	C(13)-C(5)-C(6)-C(6A)	169.9(2)
O(5)-C(5)-C(6)-C(7)	-75.2(3)	C(13)-C(5)-C(6)-C(7)	43.9(3)
C(5)-C(6)-C(6A)-O(6)	58.2(3)	C(7)-C(6)-C(6A)-O(6)	-176.4(2)
C(6A)-C(6)-C(7)-C(8)	172.4(2)	C(5)-C(6)-C(7)-C(8)	-60.8(3)
C(6)-C(7)-C(8)-C(14)	20.5(3)	N(17)-C(9)-C(10)-C(11)	95.7(3)
C(14)-C(9)-C(10)-C(11)	-28.3(3)	C(2)-C(1)-C(11)-C(12)	1.5(4)
C(2)-C(1)-C(11)-C(10)	-171.2(3)	C(9)-C(10)-C(11)-C(12)	-2.5(4)
C(9)-C(10)-C(11)-C(1)	170.1(3)	C(1)-C(11)-C(12)-C(4)	-7.6(4)
C(10)-C(11)-C(12)-C(4)	165.5(3)	C(1)-C(11)-C(12)-C(13)	-174.8(3)
C(10)-C(11)-C(12)-C(13)	-1.7(4)	C(3)-C(4)-C(12)-C(11)	8.6(4)
O(5)-C(4)-C(12)-C(11)	-166.8(2)	C(3)-C(4)-C(12)-C(13)	177.7(2)
O(5)-C(4)-C(12)-C(13)	2.3(3)	C(11)-C(12)-C(13)-C(15)	-84.4(3)
C(4)-C(12)-C(13)-C(15)	107.0(2)	C(11)-C(12)-C(13)-C(14)	35.1(4)
C(4)-C(12)-C(13)-C(14)	-133.4(2)	C(11)-C(12)-C(13)-C(5)	156.6(3)
C(4)-C(12)-C(13)-C(5)	-11.9(3)	O(5)-C(5)-C(13)-C(12)	16.7(2)
C(6)-C(5)-C(13)-C(12)	-104.8(2)	O(5)-C(5)-C(13)-C(15)	-103.2(2)
C(6)-C(5)-C(13)-C(15)	135.3(2)	O(5)-C(5)-C(13)-C(14)	131.2(2)
C(6)-C(5)-C(13)-C(14)	9.7(3)	C(12)-C(13)-C(14)-C(8)	61.9(3)
C(15)-C(13)-C(14)-C(8)	-176.2(2)	C(5)-C(13)-C(14)-C(8)	-49.5(3)
C(12)-C(13)-C(14)-C(9)	-61.8(3)	C(15)-C(13)-C(14)-C(9)	60.0(3)
C(5)-C(13)-C(14)-C(9)	-173.3(2)	C(7)-C(8)-C(14)-C(13)	32.9(3)
C(7)-C(8)-C(14)-C(9)	153.3(2)	N(17)-C(9)-C(14)-C(13)	-65.9(3)
C(10)-C(9)-C(14)-C(13)	61.6(3)	N(17)-C(9)-C(14)-C(8)	170.0(2)
C(10)-C(9)-C(14)-C(8)	-62.4(3)	C(12)-C(13)-C(15)-C(16)	64.3(3)
C(14)-C(13)-C(15)-C(16)	-53.6(3)	C(5)-C(13)-C(15)-C(16)	177.1(2)
C(13)-C(15)-C(16)-N(17)	50.7(3)	C(15)-C(16)-N(17)-C(17)	176.0(2)
C(15)-C(16)-N(17)-C(9)	-56.7(3)	C(14)-C(9)-N(17)-C(17)	-168.8(2)
C(10)-C(9)-N(17)-C(17)	64.6(3)	C(14)-C(9)-N(17)-C(16)	65.4(3)
C(10)-C(9)-N(17)-C(16)	-61.3(3)		

Table 7. Hydrogen bonds for **13a (knih86)** [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O6…N17[1]	0.840	1.973	2.809	173.5

Symmetry transformations used to generate equivalent atoms: [1] x+1/2, -y+3/2, -z

Single-crystal X-ray diffraction data on compound 27

Table 1. Crystal data and structure refinement for **27 (knih94)**.

Empirical formula	$C_{17.90}H_{27.58}Br_2N_2O_{1.90}$	
Formula weight	460.86	
Temperature	150(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 2_1	
Unit cell dimensions	a = 8.1091(2) Å	α= 90°
	b = 11.2247(3) Å	β= 106.1160(10)°
	c = 11.0425(4) Å	γ = 90°
Volume	965.61(5) $Å^3$	
Z	2	
Density (calculated)	1.585 Mg/m^3	
Absorption coefficient	4.210 mm^{-1}	
F(000)	468.2	
Crystal size	0.263 x 0.221 x 0.167 mm^3	
θ range for data collection	1.92 to 29.14°	
Index ranges	-8<=h<=11, -15<=k<=15, -15<=l<=15	
Reflections collected	11213	
Independent reflections	5047 [R(int) = 0.0662]	
Completeness to θ = 29.14°	98.4 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.5399 and 0.4039	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5047 / 9 / 222	
Goodness-of-fit on F^2	1.016	
Final R indices [I>2σ(I)]	R1 = 0.0393, wR2 = 0.1040	
R indices (all data)	R1 = 0.0412, wR2 = 0.1051	
Absolute structure parameter	-0.002(8)	
Largest diff. peak and hole	1.622 and -0.749 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for **27 (knih94).** U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Br(1)	-5012(1)	5883(1)	4725(1)	23(1)
Br(2)	-5690(1)	9265(1)	8528(1)	32(1)
C(1)	-12113(5)	11143(3)	1995(3)	23(1)
C(2)	-10489(5)	11575(3)	2044(3)	29(1)
C(3)	-9007(4)	10854(3)	2404(3)	26(1)
C(4)	-9277(4)	9675(3)	2671(3)	19(1)
O(5)	-8026(3)	8837(2)	3179(2)	20(1)
C(5)	-8877(4)	7858(3)	3656(3)	16(1)
C(6)	-8532(4)	7986(3)	5093(3)	17(1)
N(6)	-6672(3)	8265(2)	5643(3)	19(1)
C(7)	-9640(4)	8943(3)	5454(3)	20(1)
C(8)	-11553(4)	8614(3)	5004(3)	21(1)
C(9)	-12042(4)	7830(3)	3797(3)	17(1)
C(10)	-13850(4)	8164(3)	2953(3)	17(1)
C(11)	-14011(4)	9433(3)	2379(3)	19(1)
C(12)	-12344(4)	9947(3)	2265(3)	19(1)
C(13)	-10904(4)	9230(3)	2543(2)	16(1)
C(14)	-10803(4)	7964(3)	2968(3)	14(1)
C(15)	-11322(4)	7074(3)	1865(3)	17(1)
C(16)	-13178(4)	7233(3)	1091(3)	19(1)
N(17)	-14346(3)	7214(2)	1939(2)	18(1)
C(17)	-16177(4)	7264(3)	1189(3)	23(1)
O(1S)	-8538(3)	4699(5)	2072(4)	67(2)
C(1S)	-9197(2)	4503(2)	875(3)	44(1)

Table 3. Bond lengths [Å] and angles [°] for **27 (knih94)**.

C(1)-C(2)	1.390(5)	C(1)-C(12)	1.399(4)
C(1)-H(1A)	0.9500	C(2)-C(3)	1.412(5)
C(2)-H(2A)	0.9500	C(3)-C(4)	1.387(5)
C(3)-H(3A)	0.9500	C(4)-C(13)	1.381(4)
C(4)-O(5)	1.383(4)	O(5)-C(5)	1.470(4)
C(5)-C(6)	1.539(4)	C(5)-C(14)	1.541(4)
C(5)-H(5A)	1.0000	C(6)-N(6)	1.495(4)
C(6)-C(7)	1.523(4)	C(6)-H(6A)	1.0000
N(6)-H(6B)	0.9100	N(6)-H(6C)	0.9100
N(6)-H(6D)	0.9100	C(7)-C(8)	1.537(5)
C(7)-H(7A)	0.9900	C(7)-H(7B)	0.9900
C(8)-C(9)	1.554(4)	C(8)-H(8A)	0.9900
C(8)-H(8B)	0.9900	C(9)-C(14)	1.543(4)
C(9)-C(10)	1.549(4)	C(9)-H(9A)	1.0000
C(10)-N(17)	1.517(4)	C(10)-C(11)	1.551(4)
C(10)-H(10A)	1.0000	C(11)-C(12)	1.506(5)
C(11)-H(11A)	0.9900	C(11)-H(11B)	0.9900
C(12)-C(13)	1.381(4)	C(13)-C(14)	1.492(4)
C(14)-C(15)	1.541(4)	C(15)-C(16)	1.521(4)
C(15)-H(15A)	0.9900	C(15)-H(15B)	0.9900
C(16)-N(17)	1.506(4)	C(16)-H(16A)	0.9900
C(16)-H(16B)	0.9900	N(17)-C(17)	1.487(4)
N(17)-H(17D)	0.9300	C(17)-H(17A)	0.9800
C(17)-H(17B)	0.9800	C(17)-H(17C)	0.9800
O(1S)-C(1S)	1.299(5)	O(1S)-H(1S)	0.8741(10)
C(1S)-H(1SA)	0.9599(10)	C(1S)-H(1SB)	0.9599(10)
C(1S)-H(1SC)	0.9601(10)	C(2)-C(1)-C(12)	120.5(3)
C(2)-C(1)-H(1A)	119.7	C(12)-C(1)-H(1A)	119.7
C(1)-C(2)-C(3)	122.3(3)	C(1)-C(2)-H(2A)	118.9
C(3)-C(2)-H(2A)	118.9	C(4)-C(3)-C(2)	115.8(3)
C(4)-C(3)-H(3A)	122.1	C(2)-C(3)-H(3A)	122.1
C(13)-C(4)-O(5)	111.6(3)	C(13)-C(4)-C(3)	121.7(3)
O(5)-C(4)-C(3)	126.5(3)	C(4)-O(5)-C(5)	106.8(2)
O(5)-C(5)-C(6)	109.3(2)	O(5)-C(5)-C(14)	106.1(2)
C(6)-C(5)-C(14)	111.7(2)	O(5)-C(5)-H(5A)	109.9
C(6)-C(5)-H(5A)	109.9	C(14)-C(5)-H(5A)	109.9
N(6)-C(6)-C(7)	110.4(3)	N(6)-C(6)-C(5)	108.2(3)
C(7)-C(6)-C(5)	112.6(2)	N(6)-C(6)-H(6A)	108.5
C(7)-C(6)-H(6A)	108.5	C(5)-C(6)-H(6A)	108.5
C(6)-N(6)-H(6B)	109.5	C(6)-N(6)-H(6C)	109.5
H(6B)-N(6)-H(6C)	109.5	C(6)-N(6)-H(6D)	109.5
H(6B)-N(6)-H(6D)	109.5	H(6C)-N(6)-H(6D)	109.5
C(6)-C(7)-C(8)	111.2(3)	C(6)-C(7)-H(7A)	109.4
C(8)-C(7)-H(7A)	109.4	C(6)-C(7)-H(7B)	109.4
C(8)-C(7)-H(7B)	109.4	H(7A)-C(7)-H(7B)	108.0
C(7)-C(8)-C(9)	114.1(3)	C(7)-C(8)-H(8A)	108.7
C(9)-C(8)-H(8A)	108.7	C(7)-C(8)-H(8B)	108.7
C(9)-C(8)-H(8B)	108.7	H(8A)-C(8)-H(8B)	107.6
C(14)-C(9)-C(10)	106.5(2)	C(14)-C(9)-C(8)	113.6(3)
C(10)-C(9)-C(8)	110.6(2)	C(14)-C(9)-H(9A)	108.7
C(10)-C(9)-H(9A)	108.7	C(8)-C(9)-H(9A)	108.7
N(17)-C(10)-C(9)	106.7(2)	N(17)-C(10)-C(11)	111.7(2)
C(9)-C(10)-C(11)	115.2(2)	N(17)-C(10)-H(10A)	107.7

Table 3. (continued)

C(9)-C(10)-H(10A)	107.7	C(11)-C(10)-H(10A)	107.7
C(12)-C(11)-C(10)	114.2(2)	C(12)-C(11)-H(11A)	108.7
C(10)-C(11)-H(11A)	108.7	C(12)-C(11)-H(11B)	108.7
C(10)-C(11)-H(11B)	108.7	H(11A)-C(11)-H(11B)	107.6
C(13)-C(12)-C(1)	116.8(3)	C(13)-C(12)-C(11)	118.8(3)
C(1)-C(12)-C(11)	124.2(3)	C(12)-C(13)-C(4)	122.5(3)
C(12)-C(13)-C(14)	126.5(3)	C(4)-C(13)-C(14)	110.2(3)
C(13)-C(14)-C(15)	112.9(2)	C(13)-C(14)-C(5)	100.9(2)
C(15)-C(14)-C(5)	110.9(2)	C(13)-C(14)-C(9)	106.9(2)
C(15)-C(14)-C(9)	109.0(2)	C(5)-C(14)-C(9)	116.0(2)
C(16)-C(15)-C(14)	112.3(2)	C(16)-C(15)-H(15A)	109.2
C(14)-C(15)-H(15A)	109.2	C(16)-C(15)-H(15B)	109.2
C(14)-C(15)-H(15B)	109.2	H(15A)-C(15)-H(15B)	107.9
N(17)-C(16)-C(15)	110.1(2)	N(17)-C(16)-H(16A)	109.6
C(15)-C(16)-H(16A)	109.6	N(17)-C(16)-H(16B)	109.6
C(15)-C(16)-H(16B)	109.6	H(16A)-C(16)-H(16B)	108.1
C(17)-N(17)-C(16)	110.9(2)	C(17)-N(17)-C(10)	113.8(2)
C(16)-N(17)-C(10)	111.5(2)	C(17)-N(17)-H(17D)	106.7
C(16)-N(17)-H(17D)	106.7	C(10)-N(17)-H(17D)	106.7
N(17)-C(17)-H(17A)	109.5	N(17)-C(17)-H(17B)	109.5
H(17A)-C(17)-H(17B)	109.5	N(17)-C(17)-H(17C)	109.5
H(17A)-C(17)-H(17C)	109.5	H(17B)-C(17)-H(17C)	109.5
C(1S)-O(1S)-H(1S)	118.2(5)	O(1S)-C(1S)-H(1SA)	127.2(5)
O(1S)-C(1S)-H(1SB)	118.6(6)	H(1SA)-C(1S)-H(1SB)	106.7(2)
O(1S)-C(1S)-H(1SC)	85.8(6)	H(1SA)-C(1S)-H(1SC)	106.7(2)
H(1SB)-C(1S)-H(1SC)	106.7(2)		

Table 4. Anisotropic displacement parameters ($Å^2$ x 10^3) for **27 (knih94)**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Br(1)	23(1)	16(1)	33(1)	4(1)	12(1)	3(1)
Br(2)	51(1)	18(1)	19(1)	2(1)	-5(1)	-6(1)
C(1)	32(2)	17(2)	20(1)	1(1)	5(1)	6(1)
C(2)	44(2)	17(1)	25(1)	5(1)	9(1)	-6(2)
C(3)	33(2)	21(1)	24(1)	2(1)	9(1)	-5(2)
C(4)	22(1)	21(1)	15(1)	1(1)	6(1)	0(1)
O(5)	18(1)	21(1)	22(1)	3(1)	8(1)	-2(1)
C(5)	16(1)	15(1)	17(1)	1(1)	6(1)	2(1)
C(6)	17(1)	17(1)	15(1)	0(1)	4(1)	2(1)
C(7)	20(1)	25(2)	17(1)	-4(1)	6(1)	3(1)
C(8)	16(1)	32(2)	16(1)	-1(1)	5(1)	4(1)
C(9)	17(1)	18(1)	14(1)	2(1)	4(1)	2(1)
C(10)	14(1)	21(1)	15(1)	0(1)	5(1)	3(1)
C(11)	20(1)	17(1)	20(1)	-1(1)	4(1)	8(1)
C(12)	23(2)	17(1)	14(1)	-1(1)	3(1)	5(1)
C(13)	22(1)	14(1)	13(1)	1(1)	5(1)	2(1)
C(14)	16(1)	13(1)	14(1)	-2(1)	4(1)	2(1)
C(15)	18(1)	15(1)	16(1)	-1(1)	4(1)	4(1)
C(16)	20(1)	20(1)	16(1)	-1(1)	4(1)	1(1)
C(17)	16(1)	29(2)	22(1)	-1(1)	1(1)	0(1)
O(1S)	40(2)	102(4)	56(3)	16(2)	9(2)	7(2)
C(1S)	55(3)	31(2)	59(3)	7(2)	35(2)	-3(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for **27 (knih94)**.

	x	y	z	U(eq)
H(1A)	-13073	11664	1776	28
H(2A)	-10373	12384	1827	34
H(3A)	-7895	11158	2460	31
H(5A)	-8427	7075	3453	19
H(6A)	-8784	7207	5442	20
H(6B)	-6441	8337	6494	29
H(6C)	-6417	8962	5313	29
H(6D)	-6025	7668	5455	29
H(7A)	-9453	9712	5071	24
H(7B)	-9294	9043	6381	24
H(8A)	-12238	9356	4837	25
H(8B)	-11867	8183	5690	25
H(9A)	-12054	6976	4053	20
H(10A)	-14668	8107	3483	20
H(11A)	-14841	9411	1532	23
H(11B)	-14484	9971	2911	23
H(15A)	-10560	7183	1311	20
H(15B)	-11159	6252	2202	20
H(16A)	-13306	8000	632	22
H(16B)	-13501	6583	463	22
H(17D)	-14185	6482	2350	21
H(17A)	-16454	6557	650	35
H(17B)	-16373	7981	663	35
H(17C)	-16911	7285	1761	35
H(1S)	-7468(7)	4498(17)	2413(5)	100
H(1SA)	-8930(7)	4909(5)	188(5)	53
H(1SB)	-10380(3)	4270(6)	583(8)	53
H(1SC)	-8534(7)	3782(4)	1014(9)	53

Table 6. Torsion angles [°] for **27 (knih94)**.

C(12)-C(1)-C(2)-C(3)	-2.2(5)	C(1)-C(2)-C(3)-C(4)	1.9(5)
C(2)-C(3)-C(4)-C(13)	2.4(4)	C(2)-C(3)-C(4)-O(5)	-172.0(3)
C(13)-C(4)-O(5)-C(5)	-12.2(3)	C(3)-C(4)-O(5)-C(5)	162.7(3)
C(4)-O(5)-C(5)-C(6)	-100.3(3)	C(4)-O(5)-C(5)-C(14)	20.3(3)
O(5)-C(5)-C(6)-N(6)	-44.9(3)	C(14)-C(5)-C(6)-N(6)	-162.1(2)
O(5)-C(5)-C(6)-C(7)	77.3(3)	C(14)-C(5)-C(6)-C(7)	-39.8(3)
N(6)-C(6)-C(7)-C(8)	-174.6(2)	C(5)-C(6)-C(7)-C(8)	64.4(3)
C(6)-C(7)-C(8)-C(9)	-29.3(4)	C(7)-C(8)-C(9)-C(14)	-24.7(4)
C(7)-C(8)-C(9)-C(10)	-144.3(3)	C(14)-C(9)-C(10)-N(17)	66.4(3)
C(8)-C(9)-C(10)-N(17)	-169.7(3)	C(14)-C(9)-C(10)-C(11)	-58.1(3)
C(8)-C(9)-C(10)-C(11)	65.7(3)	N(17)-C(10)-C(11)-C(12)	-97.9(3)
C(9)-C(10)-C(11)-C(12)	24.0(4)	C(2)-C(1)-C(12)-C(13)	-1.7(4)
C(2)-C(1)-C(12)-C(11)	172.8(3)	C(10)-C(11)-C(12)-C(13)	6.5(4)
C(10)-C(11)-C(12)-C(1)	-167.9(3)	C(1)-C(12)-C(13)-C(4)	6.0(4)
C(11)-C(12)-C(13)-C(4)	-168.8(3)	C(1)-C(12)-C(13)-C(14)	174.3(3)
C(11)-C(12)-C(13)-C(14)	-0.6(4)	O(5)-C(4)-C(13)-C(12)	168.6(3)
C(3)-C(4)-C(13)-C(12)	-6.6(4)	O(5)-C(4)-C(13)-C(14)	-1.4(3)
C(3)-C(4)-C(13)-C(14)	-176.5(3)	C(12)-C(13)-C(14)-C(15)	85.5(3)
C(4)-C(13)-C(14)-C(15)	-105.0(3)	C(12)-C(13)-C(14)-C(5)	-156.1(3)
C(4)-C(13)-C(14)-C(5)	13.4(3)	C(12)-C(13)-C(14)-C(9)	-34.4(4)
C(4)-C(13)-C(14)-C(9)	135.0(2)	O(5)-C(5)-C(14)-C(13)	-20.0(3)
C(6)-C(5)-C(14)-C(13)	99.1(3)	O(5)-C(5)-C(14)-C(15)	99.9(3)
C(6)-C(5)-C(14)-C(15)	-141.1(3)	O(5)-C(5)-C(14)-C(9)	-135.0(2)
C(6)-C(5)-C(14)-C(9)	-16.0(3)	C(10)-C(9)-C(14)-C(13)	59.7(3)
C(8)-C(9)-C(14)-C(13)	-62.3(3)	C(10)-C(9)-C(14)-C(15)	-62.7(3)
C(8)-C(9)-C(14)-C(15)	175.3(3)	C(10)-C(9)-C(14)-C(5)	171.3(2)
C(8)-C(9)-C(14)-C(5)	49.3(3)	C(13)-C(14)-C(15)-C(16)	-62.4(3)
C(5)-C(14)-C(15)-C(16)	-174.8(2)	C(9)-C(14)-C(15)-C(16)	56.3(3)
C(14)-C(15)-C(16)-N(17)	-52.2(3)	C(15)-C(16)-N(17)-C(17)	-175.1(3)
C(15)-C(16)-N(17)-C(10)	56.9(3)	C(9)-C(10)-N(17)-C(17)	169.0(2)
C(11)-C(10)-N(17)-C(17)	-64.3(3)	C(9)-C(10)-N(17)-C(16)	-64.7(3)
C(11)-C(10)-N(17)-C(16)	62.0(3)		

Table 7. Hydrogen bonds for **27 (knih94)** [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(6)-H(6B)...Br(2)	0.91	2.40	3.261(3)	158.9
N(6)-H(6C)...Br(1)#1	0.91	2.45	3.312(3)	157.3
N(6)-H(6D)...Br(1)	0.91	2.39	3.278(3)	165.7
N(17)-H(17D)...Br(2)#2	0.93	2.66	3.352(3)	131.5
N(17)-H(17D)...Br(1)#3	0.93	2.96	3.594(3)	126.7
O(1S)-H(1S)...Br(1)	0.8741(10)	3.175(10)	3.725(4)	123.1(10)
O(1S)-H(1S)...Br(2)#4	0.8741(10)	3.030(7)	3.700(3)	135.0(8)

Symmetry transformations used to generate equivalent atoms:
#1 -x-1,y+1/2,-z+1 #2 -x-2,y-1/2,-z+1 #3 x-1,y,z
#4 -x-1,y-1/2,-z+1

Single-crystal X-ray diffraction data on compound 32 (knih98)

Table 1. Crystal data and structure refinement for **32** (**knih98)**

Empirical formula	$C_{21}H_{27}N_3O_3$	
Formula weight	369.46	
Temperature	150(2) K	
Wavelength	1.54178 Å	
Crystal system	Orthorhombic	
Space group	P $2_12_12_1$	
Unit cell dimensions	a = 9.1670(2) Å	α= 90°
	b = 13.2568(3) Å	β= 90°
	c = 15.2701(4) Å	γ = 90°
Volume	1855.70(8) $Å^3$	
Z	4	
Density (calculated)	1.322 Mg/m^3	
Absorption coefficient	0.719 mm^{-1}	
F(000)	792	
Crystal size	0.408 x 0.311 x 0.218 mm^3	
θ range for data collection	4.42 to 67.99°	
Index ranges	-9<=h<=10, -14<=k<=14, -17<=l<=17	
Reflections collected	10711	
Independent reflections	3010 [R(int) = 0.1150]	
Completeness to θ = 67.99°	94.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.8590 and 0.7579	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3010 / 0 / 244	
Goodness-of-fit on F^2	1.042	
Final R indices [I>2σ(I)]	R1 = 0.0449, wR2 = 0.1232	
R indices (all data)	R1 = 0.0450, wR2 = 0.1236	
Absolute structure parameter	-0.1(2)	
Largest diff. peak and hole	0.297 and -0.262 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for **32** (**knih98)**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	7521(2)	1437(2)	922(1)	30(1)
C(2)	6613(2)	2146(2)	1306(1)	31(1)
C(3)	7081(2)	2746(2)	2010(1)	27(1)
C(4)	8502(2)	2598(1)	2280(1)	26(1)
O(5)	9127(2)	3015(1)	3019(1)	27(1)
C(5)	10477(2)	2447(1)	3183(1)	26(1)
C(6)	10231(2)	1709(2)	3940(1)	28(1)
C(7)	9358(2)	797(2)	3636(1)	29(1)
C(8)	10195(2)	185(2)	2941(1)	31(1)
C(9)	11446(2)	414(2)	1427(1)	30(1)
C(10)	9982(2)	471(2)	904(1)	30(1)
C(11)	8958(2)	1303(1)	1196(1)	27(1)
C(12)	9426(2)	1939(1)	1857(1)	26(1)
C(13)	10870(2)	1915(1)	2314(1)	26(1)
C(14)	11302(2)	800(1)	2385(1)	28(1)
C(15)	12044(2)	2500(2)	1817(1)	29(1)
C(16)	12345(2)	2041(2)	924(1)	31(1)
N(17)	12671(2)	968(1)	1019(1)	32(1)
C(17)	13140(3)	528(2)	197(2)	39(1)
N(18)	6162(2)	3404(1)	2493(1)	32(1)
C(19)	5186(2)	4062(2)	2147(1)	32(1)
O(19)	4957(2)	4139(1)	1361(1)	43(1)
C(20)	4395(3)	4692(2)	2821(2)	38(1)
N(21)	9550(2)	2257(1)	4660(1)	31(1)
C(22)	10264(2)	2497(2)	5398(1)	28(1)
O(22)	11479(2)	2146(1)	5579(1)	34(1)
C(23)	9524(3)	3241(2)	5992(1)	39(1)

Table 3. Bond lengths [Å] and angles [°] for **32** (**knih98)**

C(1)-C(2)	1.386(3)	C(1)-C(11)	1.394(3)
C(1)-H(1A)	0.9300	C(2)-C(3)	1.404(3)
C(2)-H(2A)	0.9300	C(3)-C(4)	1.381(3)
C(3)-N(18)	1.419(2)	C(4)-C(12)	1.378(3)
C(4)-O(5)	1.381(2)	O(5)-C(5)	1.470(2)
C(5)-C(6)	1.531(3)	C(5)-C(13)	1.545(3)
C(5)-H(5A)	0.9800	C(6)-N(21)	1.458(2)
C(6)-C(7)	1.522(3)	C(6)-H(6A)	0.9800
C(7)-C(8)	1.541(3)	C(7)-H(7A)	0.9700
C(7)-H(7B)	0.9700	C(8)-C(14)	1.553(3)
C(8)-H(8A)	0.9700	C(8)-H(8B)	0.9700
C(9)-N(17)	1.480(3)	C(9)-C(14)	1.556(3)
C(9)-C(10)	1.564(3)	C(9)-H(9A)	0.9800
C(10)-C(11)	1.514(3)	C(10)-H(10A)	0.9700
C(10)-H(10B)	0.9700	C(11)-C(12)	1.383(3)
C(12)-C(13)	1.497(3)	C(13)-C(15)	1.529(3)
C(13)-C(14)	1.534(3)	C(14)-H(14A)	0.9800
C(15)-C(16)	1.519(3)	C(15)-H(15A)	0.9700
C(15)-H(15B)	0.9700	C(16)-N(17)	1.460(3)
C(16)-H(16A)	0.9700	C(16)-H(16B)	0.9700
N(17)-C(17)	1.450(3)	C(17)-H(17A)	0.9600
C(17)-H(17B)	0.9600	C(17)-H(17C)	0.9600
N(18)-C(19)	1.357(3)	N(18)-H(18A)	0.8600
C(19)-O(19)	1.223(3)	C(19)-C(20)	1.510(3)
C(20)-H(20A)	0.9600	C(20)-H(20B)	0.9600
C(20)-H(20C)	0.9600	N(21)-C(22)	1.342(2)
N(21)-H(21A)	0.8600	C(22)-O(22)	1.238(3)
C(22)-C(23)	1.502(3)	C(23)-H(23D)	0.9600
C(23)-H(23A)	0.9600	C(23)-H(23B)	0.9600
C(2)-C(1)-C(11)	121.81(18)	C(2)-C(1)-H(1A)	119.1
C(11)-C(1)-H(1A)	119.1	C(1)-C(2)-C(3)	121.66(19)
C(1)-C(2)-H(2A)	119.2	C(3)-C(2)-H(2A)	119.2
C(4)-C(3)-C(2)	115.87(19)	C(4)-C(3)-N(18)	119.49(18)
C(2)-C(3)-N(18)	124.37(18)	C(12)-C(4)-O(5)	112.43(17)
C(12)-C(4)-C(3)	122.03(18)	O(5)-C(4)-C(3)	125.38(18)
C(4)-O(5)-C(5)	106.49(13)	O(5)-C(5)-C(6)	109.40(15)
O(5)-C(5)-C(13)	106.49(15)	C(6)-C(5)-C(13)	113.03(15)
O(5)-C(5)-H(5A)	109.3	C(6)-C(5)-H(5A)	109.3
C(13)-C(5)-H(5A)	109.3	N(21)-C(6)-C(7)	113.64(16)
N(21)-C(6)-C(5)	108.27(16)	C(7)-C(6)-C(5)	110.76(15)
N(21)-C(6)-H(6A)	108.0	C(7)-C(6)-H(6A)	108.0
C(5)-C(6)-H(6A)	108.0	C(6)-C(7)-C(8)	111.46(16)
C(6)-C(7)-H(7A)	109.3	C(8)-C(7)-H(7A)	109.3
C(6)-C(7)-H(7B)	109.3	C(8)-C(7)-H(7B)	109.3
H(7A)-C(7)-H(7B)	108.0	C(7)-C(8)-C(14)	115.20(16)
C(7)-C(8)-H(8A)	108.5	C(14)-C(8)-H(8A)	108.5
C(7)-C(8)-H(8B)	108.5	C(14)-C(8)-H(8B)	108.5
H(8A)-C(8)-H(8B)	107.5	N(17)-C(9)-C(14)	107.28(16)
N(17)-C(9)-C(10)	114.34(16)	C(14)-C(9)-C(10)	112.97(16)
N(17)-C(9)-H(9A)	107.3	C(14)-C(9)-H(9A)	107.3
C(10)-C(9)-H(9A)	107.3	C(11)-C(10)-C(9)	114.70(17)
C(11)-C(10)-H(10A)	108.6	C(9)-C(10)-H(10A)	108.6

Table 3. (continued)

C(11)-C(10)-H(10B)	108.6	C(9)-C(10)-H(10B)	108.6
H(10A)-C(10)-H(10B)	107.6	C(12)-C(11)-C(1)	115.74(18)
C(12)-C(11)-C(10)	117.76(18)	C(1)-C(11)-C(10)	126.19(18)
C(4)-C(12)-C(11)	122.55(19)	C(4)-C(12)-C(13)	109.80(17)
C(11)-C(12)-C(13)	126.98(18)	C(12)-C(13)-C(15)	112.37(15)
C(12)-C(13)-C(14)	106.35(15)	C(15)-C(13)-C(14)	110.06(16)
C(12)-C(13)-C(5)	100.60(16)	C(15)-C(13)-C(5)	111.04(16)
C(14)-C(13)-C(5)	116.04(15)	C(13)-C(14)-C(8)	112.08(16)
C(13)-C(14)-C(9)	105.82(15)	C(8)-C(14)-C(9)	113.36(16)
C(13)-C(14)-H(14A)	108.5	C(8)-C(14)-H(14A)	108.5
C(9)-C(14)-H(14A)	108.5	C(16)-C(15)-C(13)	111.72(16)
C(16)-C(15)-H(15A)	109.3	C(13)-C(15)-H(15A)	109.3
C(16)-C(15)-H(15B)	109.3	C(13)-C(15)-H(15B)	109.3
H(15A)-C(15)-H(15B)	107.9	N(17)-C(16)-C(15)	109.77(16)
N(17)-C(16)-H(16A)	109.7	C(15)-C(16)-H(16A)	109.7
N(17)-C(16)-H(16B)	109.7	C(15)-C(16)-H(16B)	109.7
H(16A)-C(16)-H(16B)	108.2	C(17)-N(17)-C(16)	111.50(17)
C(17)-N(17)-C(9)	112.92(17)	C(16)-N(17)-C(9)	111.76(16)
N(17)-C(17)-H(17A)	109.5	N(17)-C(17)-H(17B)	109.5
H(17A)-C(17)-H(17B)	109.5	N(17)-C(17)-H(17C)	109.5
H(17A)-C(17)-H(17C)	109.5	H(17B)-C(17)-H(17C)	109.5
C(19)-N(18)-C(3)	125.78(16)	C(19)-N(18)-H(18A)	117.1
C(3)-N(18)-H(18A)	117.1	O(19)-C(19)-N(18)	123.27(19)
O(19)-C(19)-C(20)	122.76(19)	N(18)-C(19)-C(20)	113.97(18)
C(19)-C(20)-H(20A)	109.5	C(19)-C(20)-H(20B)	109.5
H(20A)-C(20)-H(20B)	109.5	C(19)-C(20)-H(20C)	109.5
H(20A)-C(20)-H(20C)	109.5	H(20B)-C(20)-H(20C)	109.5
C(22)-N(21)-C(6)	122.85(17)	C(22)-N(21)-H(21A)	118.6
C(6)-N(21)-H(21A)	118.6	O(22)-C(22)-N(21)	122.57(18)
O(22)-C(22)-C(23)	121.16(18)	N(21)-C(22)-C(23)	116.25(19)
C(22)-C(23)-H(23D)	109.5	C(22)-C(23)-H(23A)	109.5
H(23D)-C(23)-H(23A)	109.5	C(22)-C(23)-H(23B)	109.5
H(23D)-C(23)-H(23B)	109.5	H(23A)-C(23)-H(23B)	109.5

Table 4. Anisotropic displacement parameters ($Å^2$ x 10^3) for **32** (**knih98)**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	36(1)	33(1)	21(1)	0(1)	-2(1)	-4(1)
C(2)	31(1)	36(1)	25(1)	4(1)	-2(1)	0(1)
C(3)	28(1)	30(1)	24(1)	4(1)	2(1)	2(1)
C(4)	32(1)	27(1)	19(1)	3(1)	0(1)	-3(1)
O(5)	30(1)	28(1)	22(1)	-4(1)	-2(1)	4(1)
C(5)	26(1)	29(1)	25(1)	-4(1)	-1(1)	0(1)
C(6)	29(1)	35(1)	20(1)	-3(1)	-1(1)	4(1)
C(7)	32(1)	35(1)	21(1)	4(1)	0(1)	0(1)
C(8)	42(1)	27(1)	23(1)	1(1)	0(1)	2(1)
C(9)	38(1)	31(1)	21(1)	-1(1)	1(1)	5(1)
C(10)	36(1)	32(1)	23(1)	-3(1)	1(1)	1(1)
C(11)	32(1)	29(1)	20(1)	2(1)	2(1)	1(1)
C(12)	30(1)	27(1)	20(1)	4(1)	1(1)	-1(1)
C(13)	27(1)	30(1)	19(1)	0(1)	-1(1)	-1(1)
C(14)	32(1)	31(1)	21(1)	-1(1)	-1(1)	5(1)
C(15)	29(1)	29(1)	28(1)	1(1)	1(1)	1(1)
C(16)	30(1)	37(1)	25(1)	3(1)	1(1)	0(1)
N(17)	32(1)	37(1)	26(1)	-1(1)	3(1)	6(1)
C(17)	39(1)	49(1)	29(1)	-4(1)	7(1)	8(1)
N(18)	32(1)	42(1)	21(1)	-1(1)	1(1)	4(1)
C(19)	30(1)	33(1)	32(1)	3(1)	1(1)	0(1)
O(19)	51(1)	49(1)	29(1)	7(1)	0(1)	13(1)
C(20)	35(1)	41(1)	38(1)	-4(1)	0(1)	5(1)
N(21)	29(1)	43(1)	22(1)	-4(1)	-2(1)	7(1)
C(22)	28(1)	37(1)	21(1)	0(1)	2(1)	-3(1)
O(22)	29(1)	48(1)	26(1)	-5(1)	-2(1)	2(1)
C(23)	38(1)	53(1)	27(1)	-8(1)	0(1)	8(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for **32** (**knih98)**.

	x	y	z	U(eq)
H(1A)	7161	1040	469	36
H(2A)	5670	2227	1093	37
H(5A)	11258	2918	3340	32
H(6A)	11186	1470	4139	33
H(7A)	8436	1022	3392	35
H(7B)	9147	367	4134	35
H(8A)	9493	-123	2548	37
H(8B)	10717	-355	3233	37
H(9A)	11729	-299	1456	36
H(10A)	10205	569	289	37
H(10B)	9481	-170	960	37
H(14A)	12261	758	2667	34
H(15A)	11730	3194	1744	34
H(15B)	12937	2504	2158	34
H(16A)	11500	2127	549	37
H(16B)	13165	2381	653	37
H(17A)	13933	915	-39	58
H(17B)	12342	530	-210	58
H(17C)	13456	-153	295	58
H(18A)	6230	3384	3054	38
H(20A)	3726	5138	2532	57
H(20B)	5090	5080	3149	57
H(20C)	3867	4257	3211	57
H(21A)	8652	2435	4609	37
H(23D)	10123	3358	6497	59
H(23A)	8598	2975	6174	59
H(23B)	9377	3864	5685	59

Table 6. Torsion angles [°] for **32** (**knih98)**

C(11)-C(1)-C(2)-C(3)	-1.9(3)	C(1)-C(2)-C(3)-C(4)	1.2(3)
C(1)-C(2)-C(3)-N(18)	-172.83(17)	C(2)-C(3)-C(4)-C(12)	3.1(3)
N(18)-C(3)-C(4)-C(12)	177.47(17)	C(2)-C(3)-C(4)-O(5)	-171.84(17)
N(18)-C(3)-C(4)-O(5)	2.5(3)	C(12)-C(4)-O(5)-C(5)	-10.76(19)
C(3)-C(4)-O(5)-C(5)	164.59(18)	C(4)-O(5)-C(5)-C(6)	-103.15(16)
C(4)-O(5)-C(5)-C(13)	19.31(18)	O(5)-C(5)-C(6)-N(21)	-48.7(2)
C(13)-C(5)-C(6)-N(21)	-167.16(16)	O(5)-C(5)-C(6)-C(7)	76.53(18)
C(13)-C(5)-C(6)-C(7)	-41.9(2)	N(21)-C(6)-C(7)-C(8)	-174.27(15)
C(5)-C(6)-C(7)-C(8)	63.6(2)	C(6)-C(7)-C(8)-C(14)	-27.2(2)
N(17)-C(9)-C(10)-C(11)	-94.1(2)	C(14)-C(9)-C(10)-C(11)	28.9(2)
C(2)-C(1)-C(11)-C(12)	-1.6(3)	C(2)-C(1)-C(11)-C(10)	171.87(18)
C(9)-C(10)-C(11)-C(12)	2.3(3)	C(9)-C(10)-C(11)-C(1)	-171.11(18)
O(5)-C(4)-C(12)-C(11)	168.49(16)	C(3)-C(4)-C(12)-C(11)	-7.0(3)
O(5)-C(4)-C(12)-C(13)	-2.7(2)	C(3)-C(4)-C(12)-C(13)	-178.20(16)
C(1)-C(11)-C(12)-C(4)	6.0(3)	C(10)-C(11)-C(12)-C(4)	-168.05(17)
C(1)-C(11)-C(12)-C(13)	175.61(17)	C(10)-C(11)-C(12)-C(13)	1.5(3)
C(4)-C(12)-C(13)-C(15)	-104.26(18)	C(11)-C(12)-C(13)-C(15)	85.1(2)
C(4)-C(12)-C(13)-C(14)	135.25(16)	C(11)-C(12)-C(13)-C(14)	-35.4(2)
C(4)-C(12)-C(13)-C(5)	13.9(2)	C(11)-C(12)-C(13)-C(5)	-156.77(18)
O(5)-C(5)-C(13)-C(12)	-19.76(18)	C(6)-C(5)-C(13)-C(12)	100.39(18)
O(5)-C(5)-C(13)-C(15)	99.37(18)	C(6)-C(5)-C(13)-C(15)	-140.48(17)
O(5)-C(5)-C(13)-C(14)	-133.97(16)	C(6)-C(5)-C(13)-C(14)	-13.8(2)
C(12)-C(13)-C(14)-C(8)	-61.97(19)	C(15)-C(13)-C(14)-C(8)	176.07(16)
C(5)-C(13)-C(14)-C(8)	48.9(2)	C(12)-C(13)-C(14)-C(9)	62.06(19)
C(15)-C(13)-C(14)-C(9)	-59.9(2)	C(5)-C(13)-C(14)-C(9)	172.96(16)
C(7)-C(8)-C(14)-C(13)	-27.1(2)	C(7)-C(8)-C(14)-C(9)	-146.77(17)
N(17)-C(9)-C(14)-C(13)	64.6(2)	C(10)-C(9)-C(14)-C(13)	-62.3(2)
N(17)-C(9)-C(14)-C(8)	-172.19(16)	C(10)-C(9)-C(14)-C(8)	60.9(2)
C(12)-C(13)-C(15)-C(16)	-63.0(2)	C(14)-C(13)-C(15)-C(16)	55.3(2)
C(5)-C(13)-C(15)-C(16)	-174.80(16)	C(13)-C(15)-C(16)-N(17)	-53.1(2)
C(15)-C(16)-N(17)-C(17)	-172.94(17)	C(15)-C(16)-N(17)-C(9)	59.6(2)
C(14)-C(9)-N(17)-C(17)	166.89(16)	C(10)-C(9)-N(17)-C(17)	-67.0(2)
C(14)-C(9)-N(17)-C(16)	-66.4(2)	C(10)-C(9)-N(17)-C(16)	59.7(2)
C(4)-C(3)-N(18)-C(19)	140.1(2)	C(2)-C(3)-N(18)-C(19)	-46.0(3)
C(3)-N(18)-C(19)-O(19)	2.3(3)	C(3)-N(18)-C(19)-C(20)	-178.04(19)
C(7)-C(6)-N(21)-C(22)	128.89(19)	C(5)-C(6)-N(21)-C(22)	-107.6(2)
C(6)-N(21)-C(22)-O(22)	-10.9(3)	C(6)-N(21)-C(22)-C(23)	167.7(2)

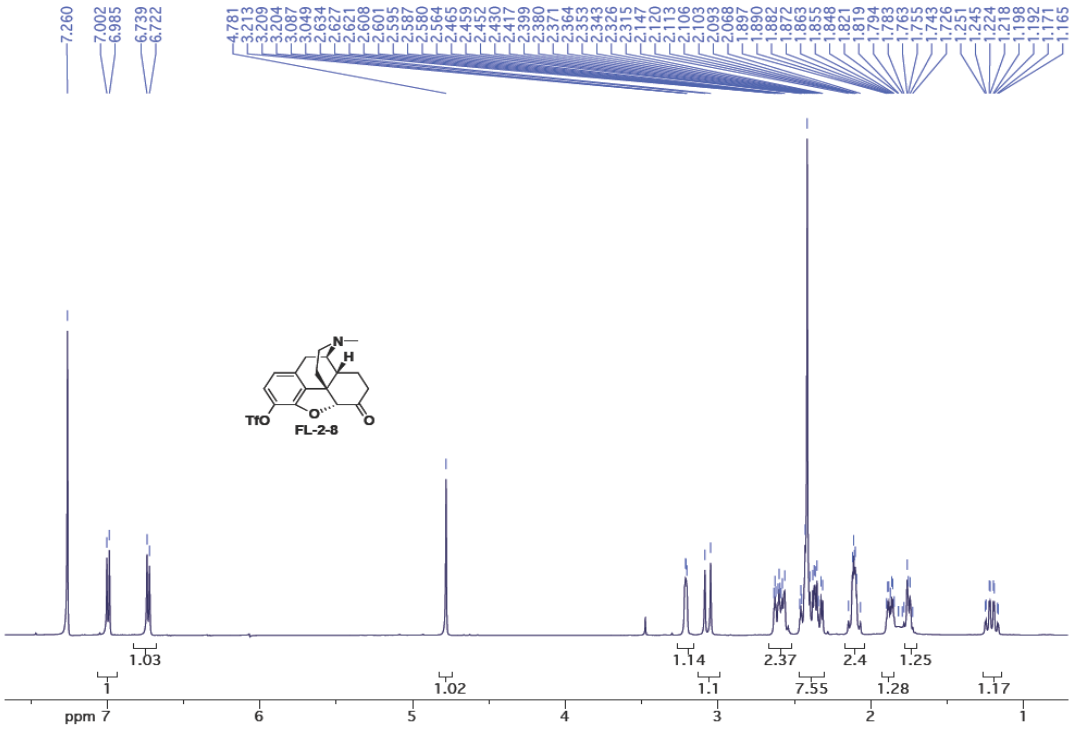
Table 7. Hydrogen bonds for **k32** (**knih98)** [Å and °].

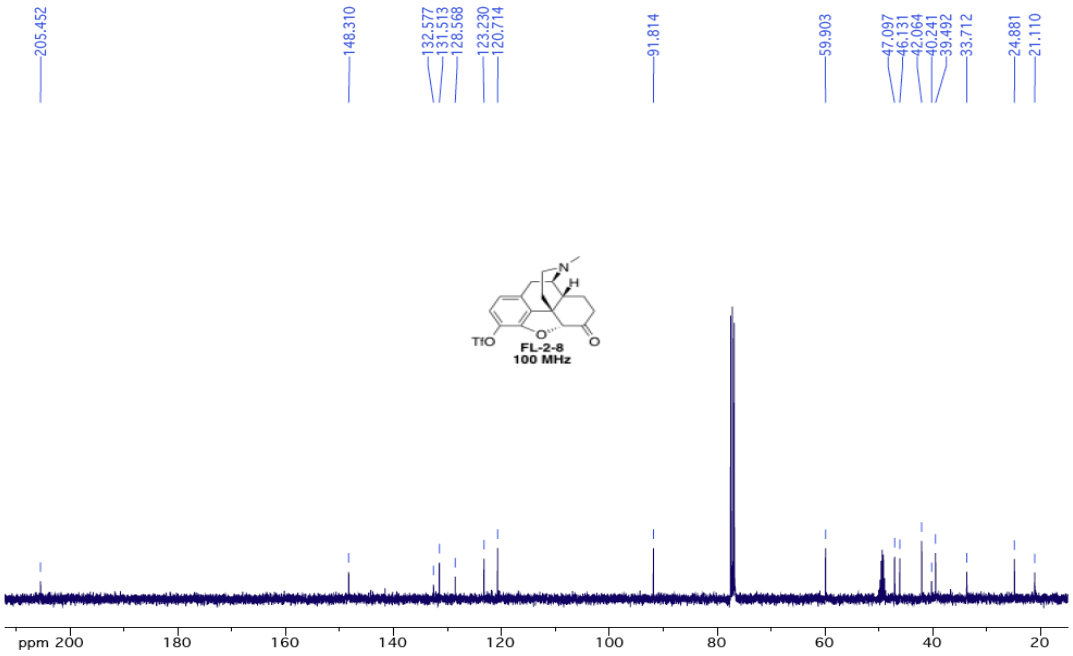
D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(18)-H(18A)...O(22)#1	0.86	2.21	3.047(2)	163.1
N(21)-H(21A)...O(22)#1	0.86	2.09	2.947(2)	177.2

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

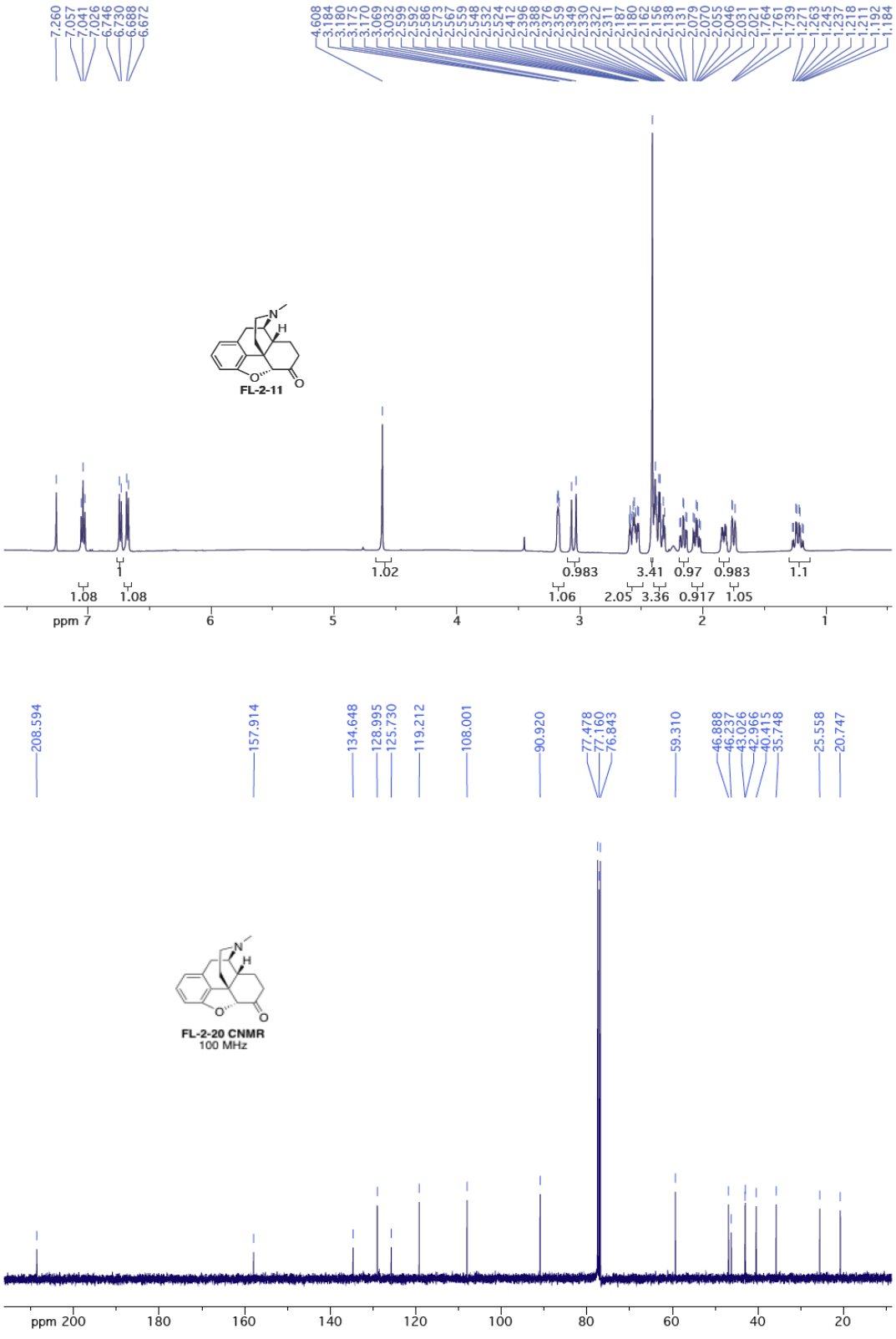
Copies of NMR spectra of all new compounds

Compound 5

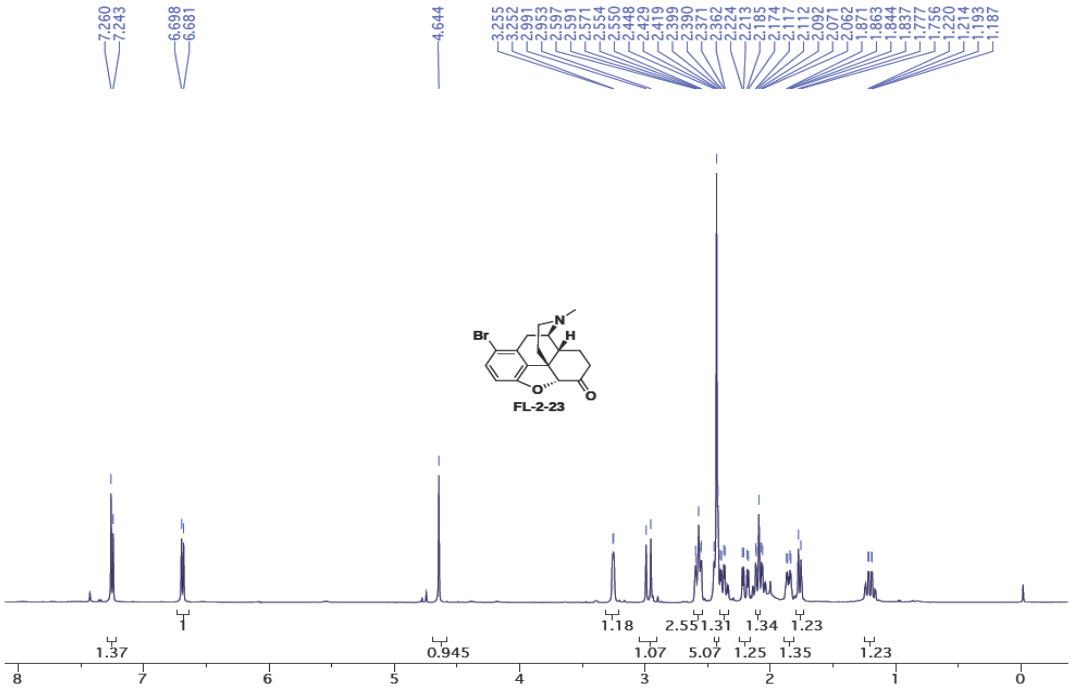


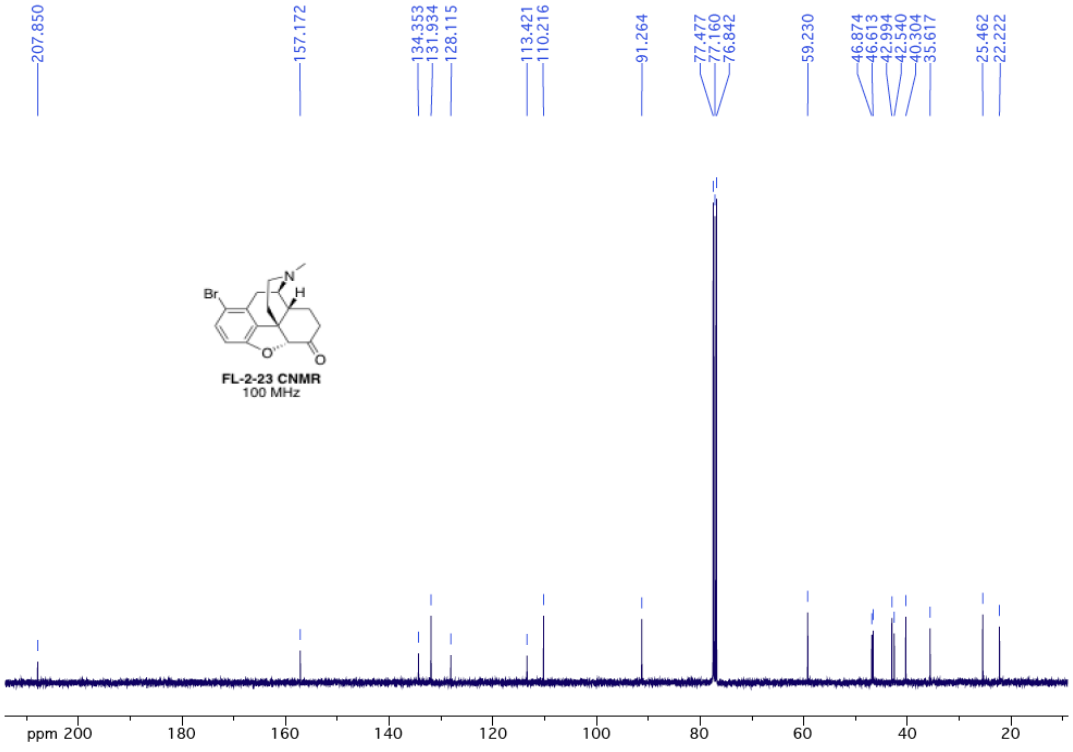


Compound 6

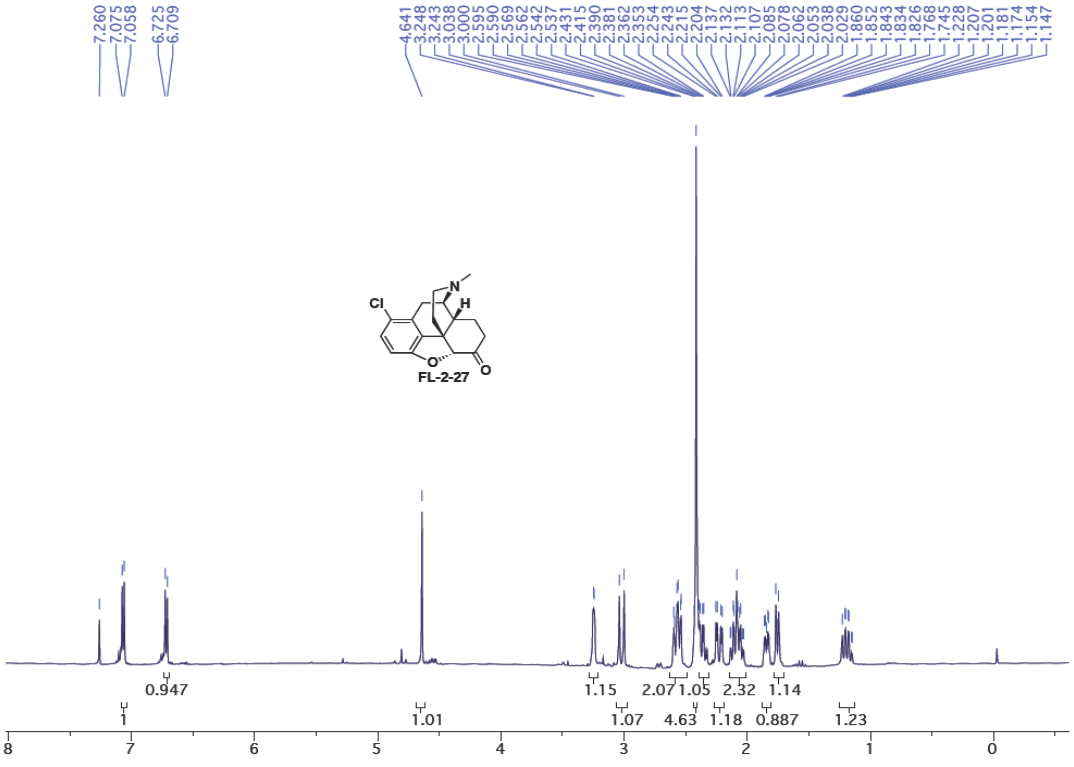

7.260
7.057
7.041
7.026
6.746
6.730
6.688
6.672
4.608
3.184
3.180
3.175
3.170
3.069
3.032
2.599
2.592
2.586
2.573
2.567
2.559
2.548
2.532
2.524
2.412
2.396
2.388
2.376
2.359
2.339
2.349
2.330
2.322
2.311
2.187
2.180
2.162
2.156
2.138
2.131
2.079
2.070
2.055
2.046
2.031
2.021
1.764
1.761
1.739
1.271
1.263
1.245
1.237
1.218
1.211
1.192
1.184
FL-2-11
1
1.02
0.983
3.41
0.97
0.983
1.1
1.08
1.08
1.06
2.05
3.36
0.917
1.05
ppm 7
6
5
4
3
2
1
208.594
157.914
134.648
128.995
125.730
119.212
108.001
90.920
77.478
77.160
76.843
59.310
46.888
46.237
43.026
42.966
40.415
35.748
25.558
20.747
FL-2-20 CNMR
100 MHz
ppm 200
180
160
140
120
100
80
60
40
20

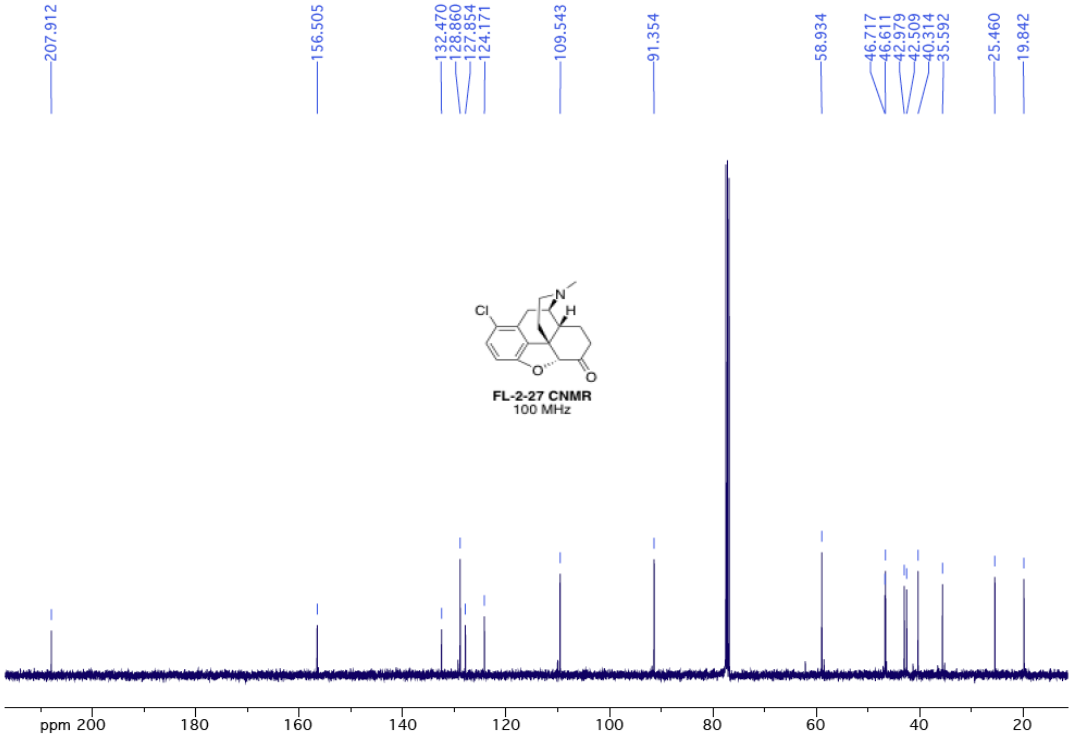
Compound 7a


7.260
7.243
6.698
6.681
4.644
3.255
3.252
2.991
2.953
2.597
2.591
2.571
2.554
2.550
2.448
2.429
2.419
2.399
2.390
2.371
2.362
2.224
2.213
2.185
2.174
2.117
2.112
2.092
2.071
2.062
1.871
1.863
1.844
1.837
1.777
1.756
1.220
1.214
1.193
1.187
Br
N
H
O
O
FL-2-23
1
1.18
2.55
1.31
1.34
1.23
1.37
0.945
1.07
5.07
1.25
1.35
1.23
8
7
6
5
4
3
2
1
0

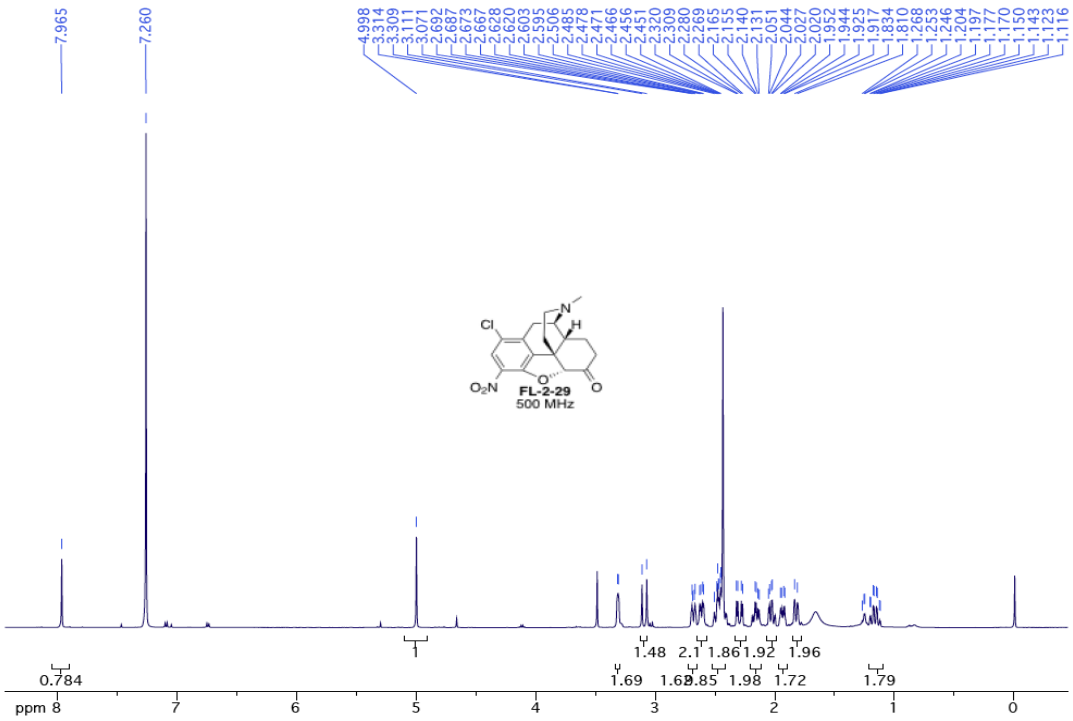

207.850
157.172
134.353
131.934
128.115
113.421
110.216
91.264
77.477
77.160
76.842
59.230
46.874
46.613
42.994
42.540
40.304
35.617
25.462
22.222
Br
N
H
O
O
FL-2-23 CNMR
100 MHz
ppm 200
180
160
140
120
100
80
60
40
20

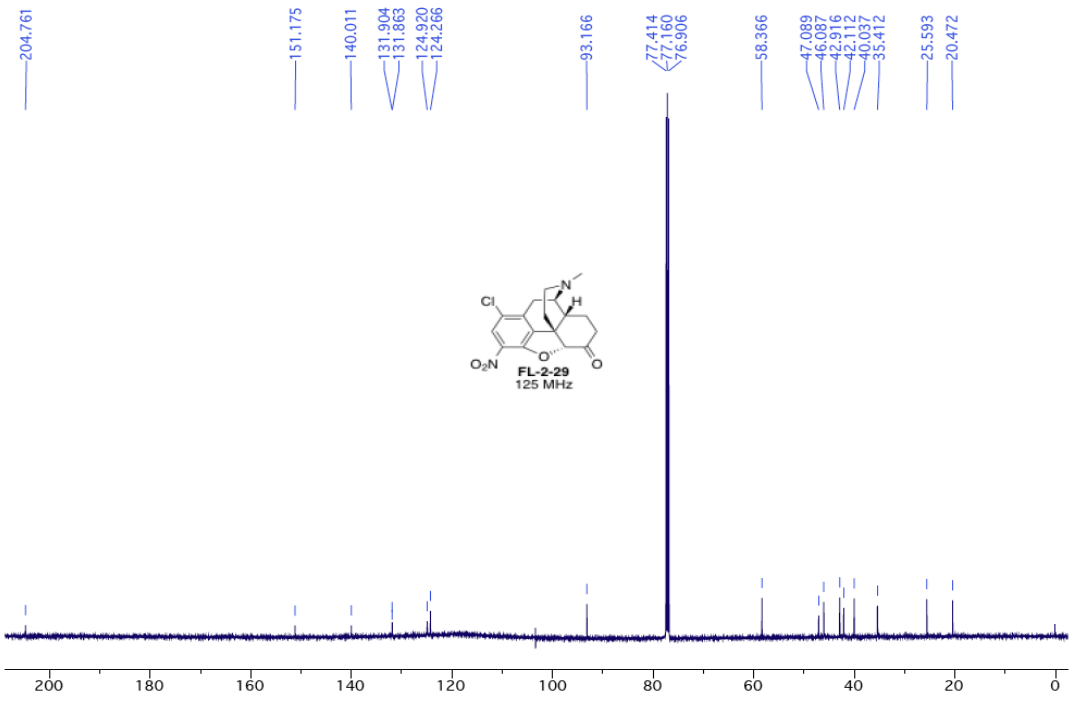
Compound 7b


7.260
7.075
7.058
6.725
6.709
4.641
3.248
3.243
3.038
3.000
2.595
2.590
2.569
2.562
2.542
2.537
2.431
2.415
2.390
2.381
2.362
2.353
2.254
2.243
2.215
2.204
2.137
2.132
2.113
2.107
2.085
2.078
2.062
2.053
2.038
2.029
1.860
1.852
1.843
1.834
1.826
1.768
1.745
1.228
1.207
1.201
1.181
1.174
1.154
1.147
Cl
N
H
O
O
FL-2-27
0.947
1.15
2.07
1.05
2.32
1.14
1
1.01
1.07
4.63
1.18
0.887
1.23
8
7
6
5
4
3
2
1
0

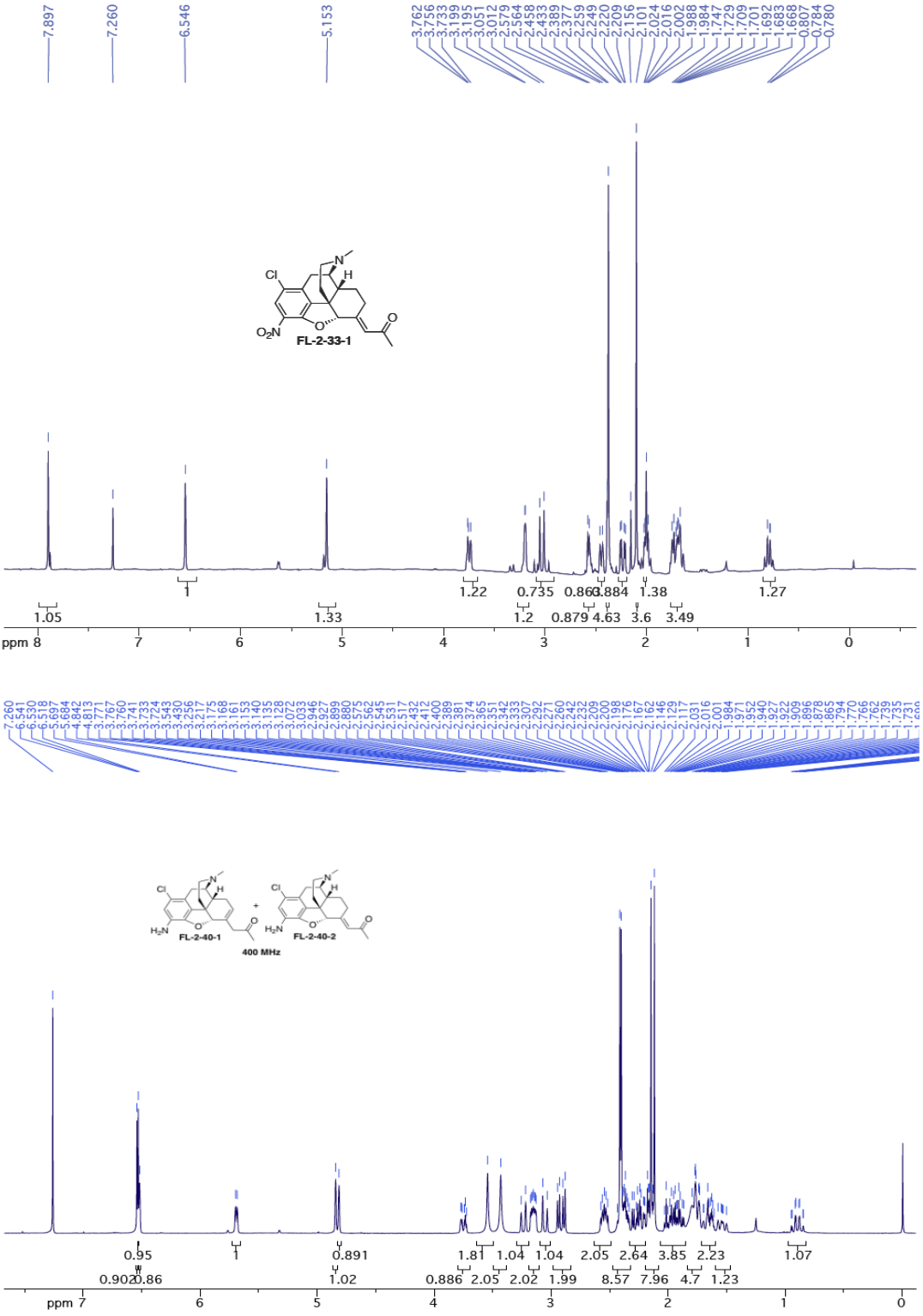

207.912
156.505
132.470
128.860
127.854
124.171
109.543
91.354
58.934
46.717
46.611
42.979
42.509
40.314
35.592
25.460
19.842
Cl
N
H
O
O
FL-2-27 CNMR
100 MHz
ppm 200
180
160
140
120
100
80
60
40
20

Compound 8b

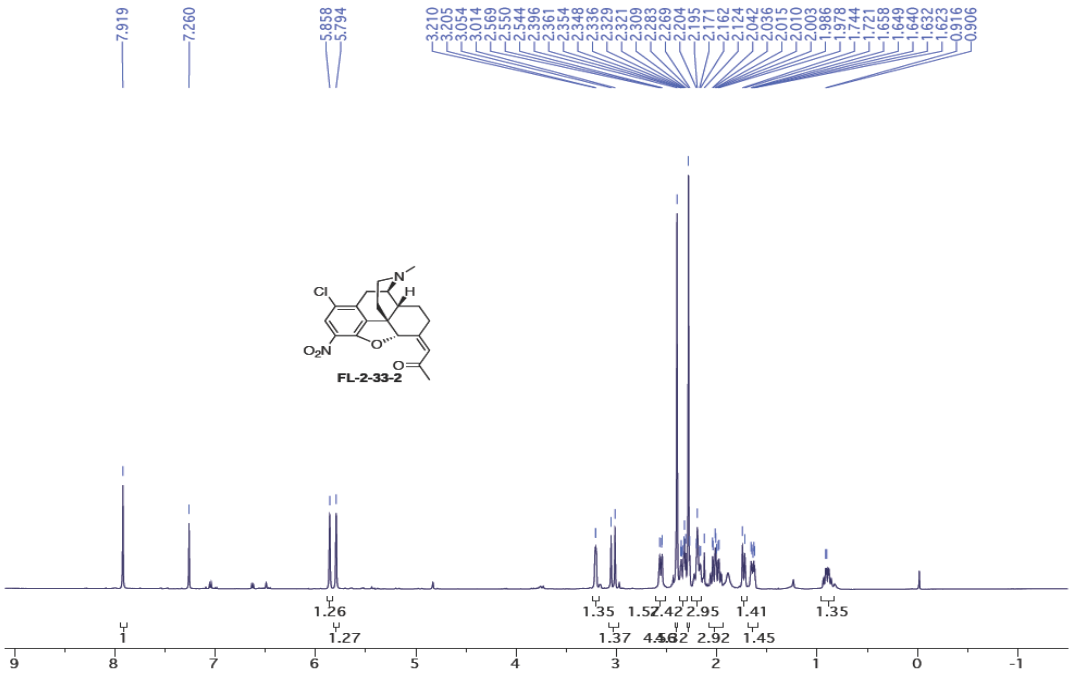

7.965
7.260
4.998
3.314
3.309
3.111
3.071
2.692
2.687
2.673
2.667
2.628
2.620
2.603
2.595
2.506
2.485
2.478
2.471
2.466
2.456
2.451
2.320
2.309
2.280
2.269
2.165
2.155
2.140
2.131
2.051
2.044
2.027
2.020
1.952
1.944
1.925
1.917
1.834
1.810
1.268
1.253
1.246
1.204
1.197
1.177
1.170
1.150
1.143
1.123
1.116
FL-2-29
500 MHz
0.784
1
1.48
2.1
1.86
1.92
1.96
1.69
1.98
1.72
1.79
ppm 8
7
6
5
4
3
2
1
0

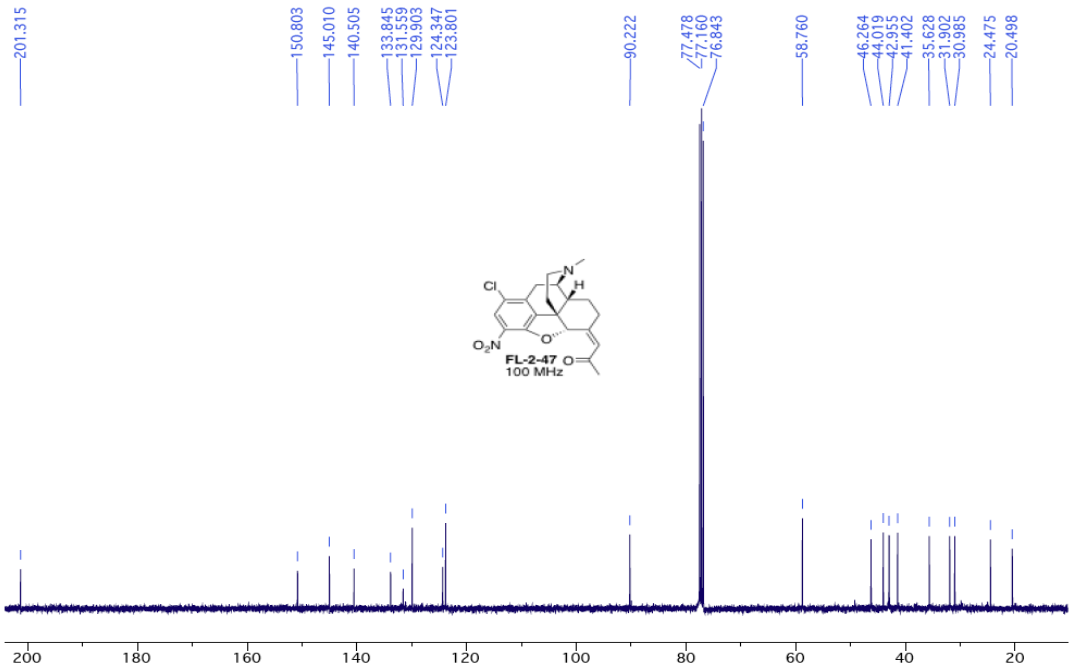

204.761
151.175
140.011
131.904
131.863
124.920
124.266
93.166
77.414
77.160
76.906
58.366
47.089
46.087
42.916
42.112
40.037
35.412
25.593
20.472
FL-2-29
125 MHz
200
180
160
140
120
100
80
60
40
20
0

Compound 9a

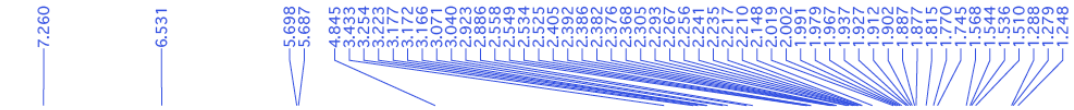

7.897
7.260
6.546
5.153
FL-2-33-1
1.05
1
1.33
1.22
0.735
0.860
0.884
1.38
1.27
1.2
0.879
4.63
3.6
3.49
ppm 8
7
6
5
4
3
2
1
0
FL-2-40-1
FL-2-40-2
400 MHz
0.95
0.902
0.86
1
0.891
1.02
1.81
1.04
1.04
0.886
2.05
2.02
1.99
2.05
2.64
3.85
2.23
8.57
7.96
4.7
1.23
1.07
ppm 7
6
5
4
3
2
1
0

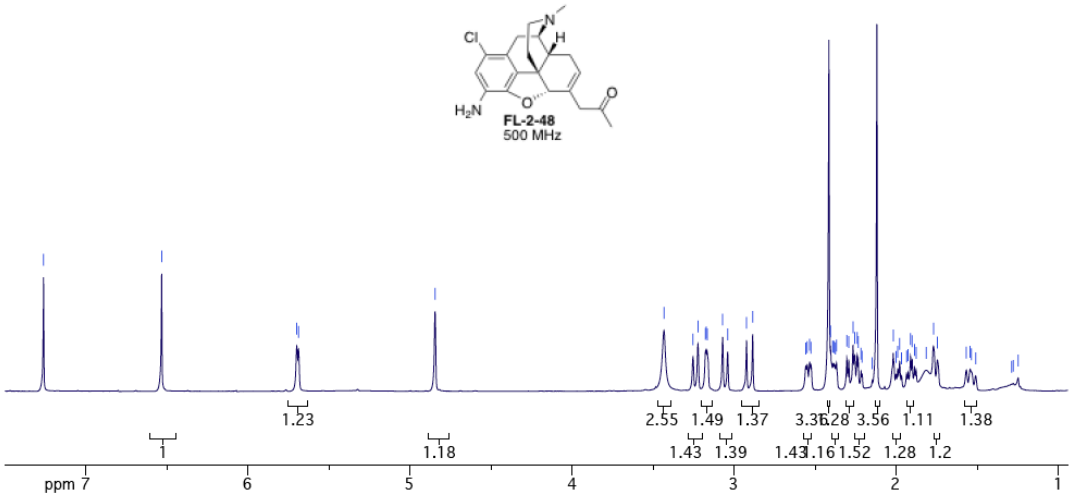
Compound 9b

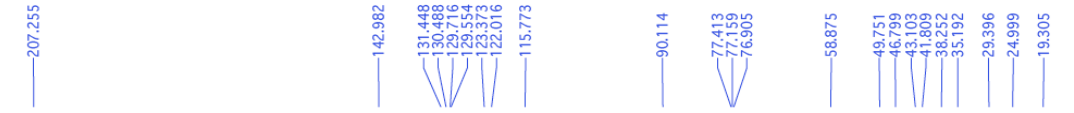

7.919
7.260
5.858
5.794
3.210
3.205
3.054
3.014
2.569
2.550
2.544
2.396
2.361
2.354
2.348
2.336
2.329
2.321
2.309
2.283
2.269
2.204
2.195
2.171
2.162
2.124
2.042
2.036
2.015
2.010
2.003
1.996
1.978
1.744
1.721
1.658
1.649
1.640
1.632
1.623
0.916
0.906
FL-2-33-2
1.26
1.35
1.52
2.42
2.95
1.41
1.35
1
1.27
1.37
2.92
1.45

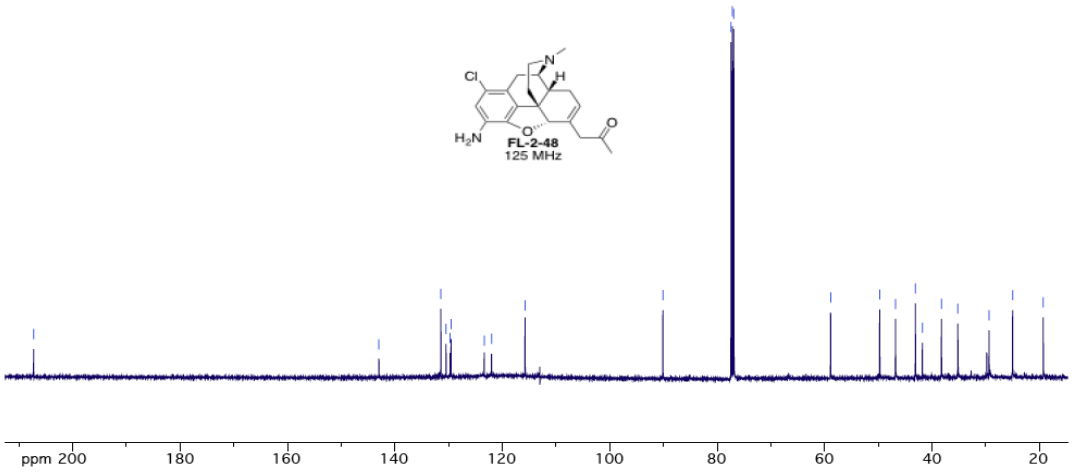

201.315
150.803
145.010
140.505
133.845
131.559
129.903
124.347
123.801
90.222
77.478
77.160
76.843
58.760
46.264
44.019
42.955
41.402
35.628
31.902
30.985
24.475
20.498
FL-2-47
100 MHz

Compound 10

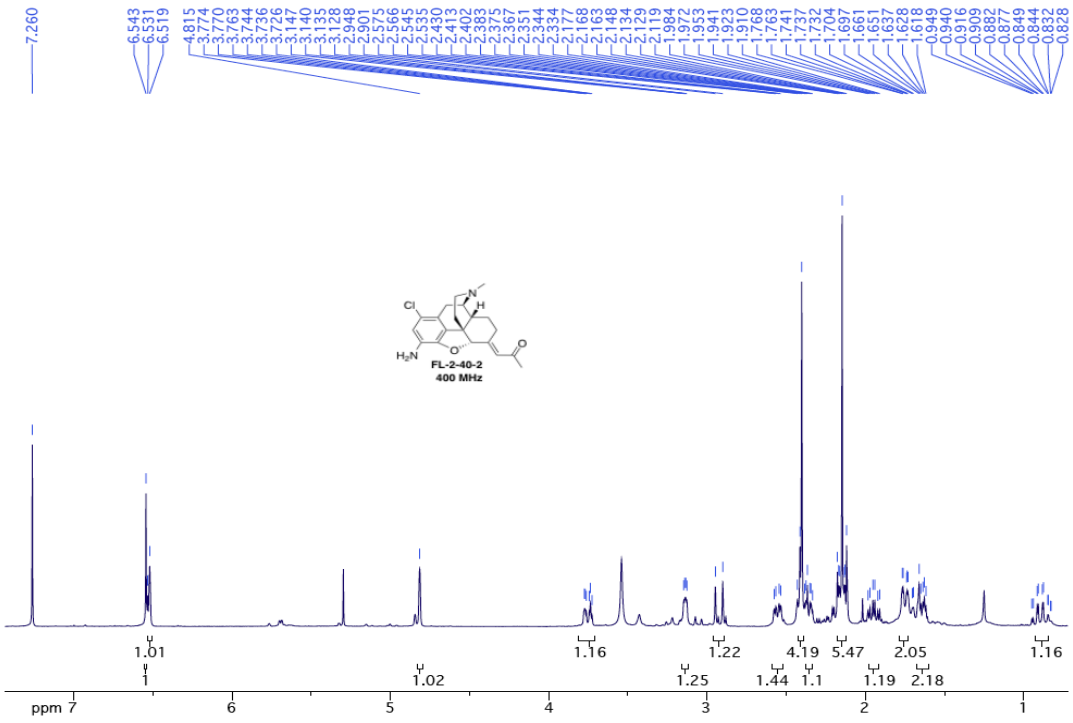

7.260
6.531
5.698
5.687
4.845
3.433
3.354
3.223
3.177
3.172
3.166
3.071
3.040
2.923
2.886
2.558
2.549
2.534
2.525
2.405
2.392
2.386
2.382
2.376
2.368
2.305
2.293
2.267
2.256
2.241
2.235
2.217
2.210
2.148
2.019
2.002
1.991
1.979
1.967
1.937
1.927
1.912
1.902
1.887
1.877
1.815
1.770
1.745
1.568
1.544
1.536
1.510
1.299
1.279
1.248


FL-2-48
500 MHz
1.23
1.18
2.55
1.49
1.37
1.43
1.39
1.43
1.16
1.52
1.28
1.2
1.38
1.11
3.56
3.28
ppm 7
6
5
4
3
2
1

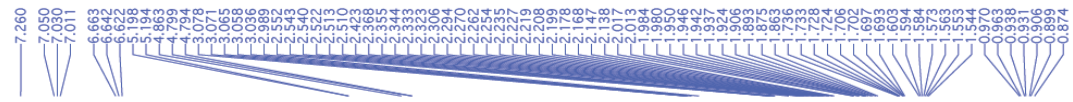

207.255
142.982
131.448
130.488
129.716
129.554
123.373
122.016
115.773
90.114
77.413
77.159
76.905
58.875
49.751
46.799
43.103
41.809
38.252
35.192
29.396
24.999
19.305

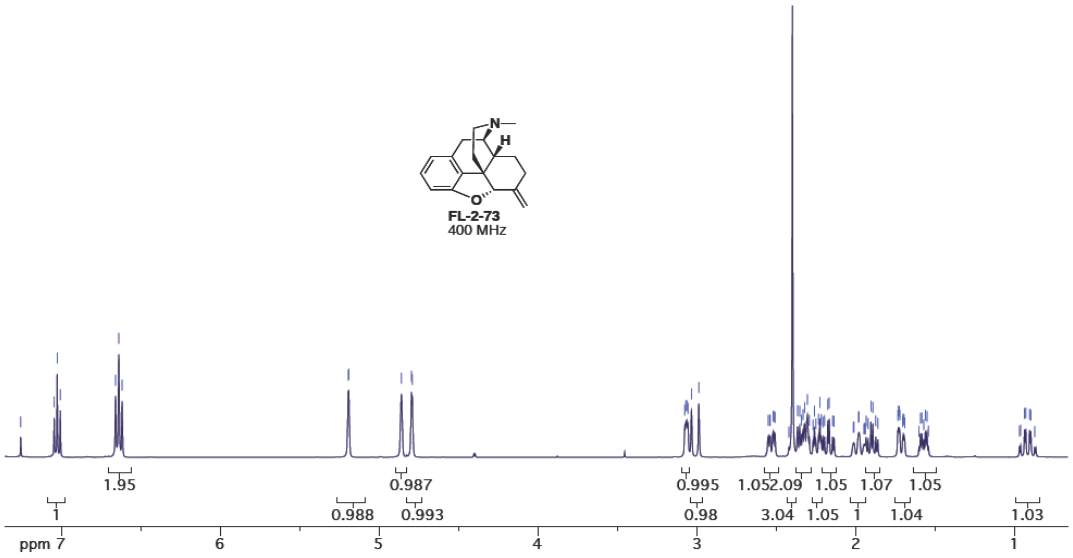

FL-2-48
125 MHz
ppm 200
180
160
140
120
100
80
60
40
20

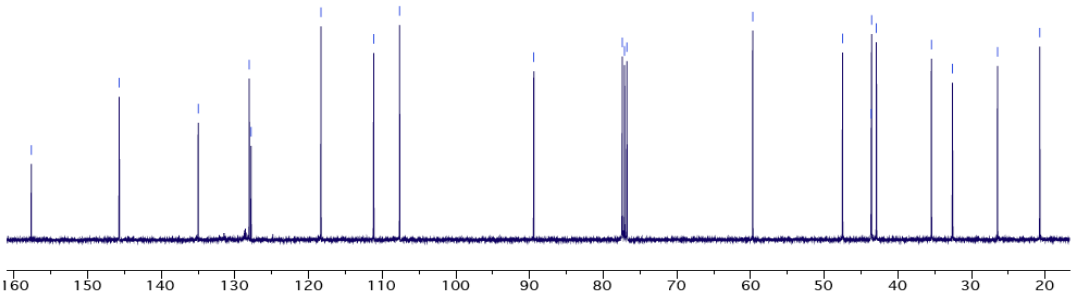
Compound 11


7.260
6.543
6.531
6.519
4.815
3.774
3.770
3.763
3.744
3.736
3.726
3.147
3.140
3.135
3.128
2.948
2.901
2.575
2.566
2.545
2.535
2.430
2.413
2.402
2.383
2.375
2.367
2.351
2.344
2.334
2.177
2.168
2.163
2.148
2.134
2.129
2.119
1.984
1.972
1.953
1.941
1.923
1.910
1.768
1.763
1.741
1.737
1.732
1.704
1.697
1.661
1.651
1.637
1.628
1.618
0.949
0.940
0.916
0.909
0.882
0.877
0.849
0.844
0.832
0.828
FL-2-40-2
400 MHz
1.01
1.16
1.22
4.19
5.47
2.05
1.16
1
1.02
1.25
1.44
1.1
1.19
2.18
ppm 7
6
5
4
3
2
1

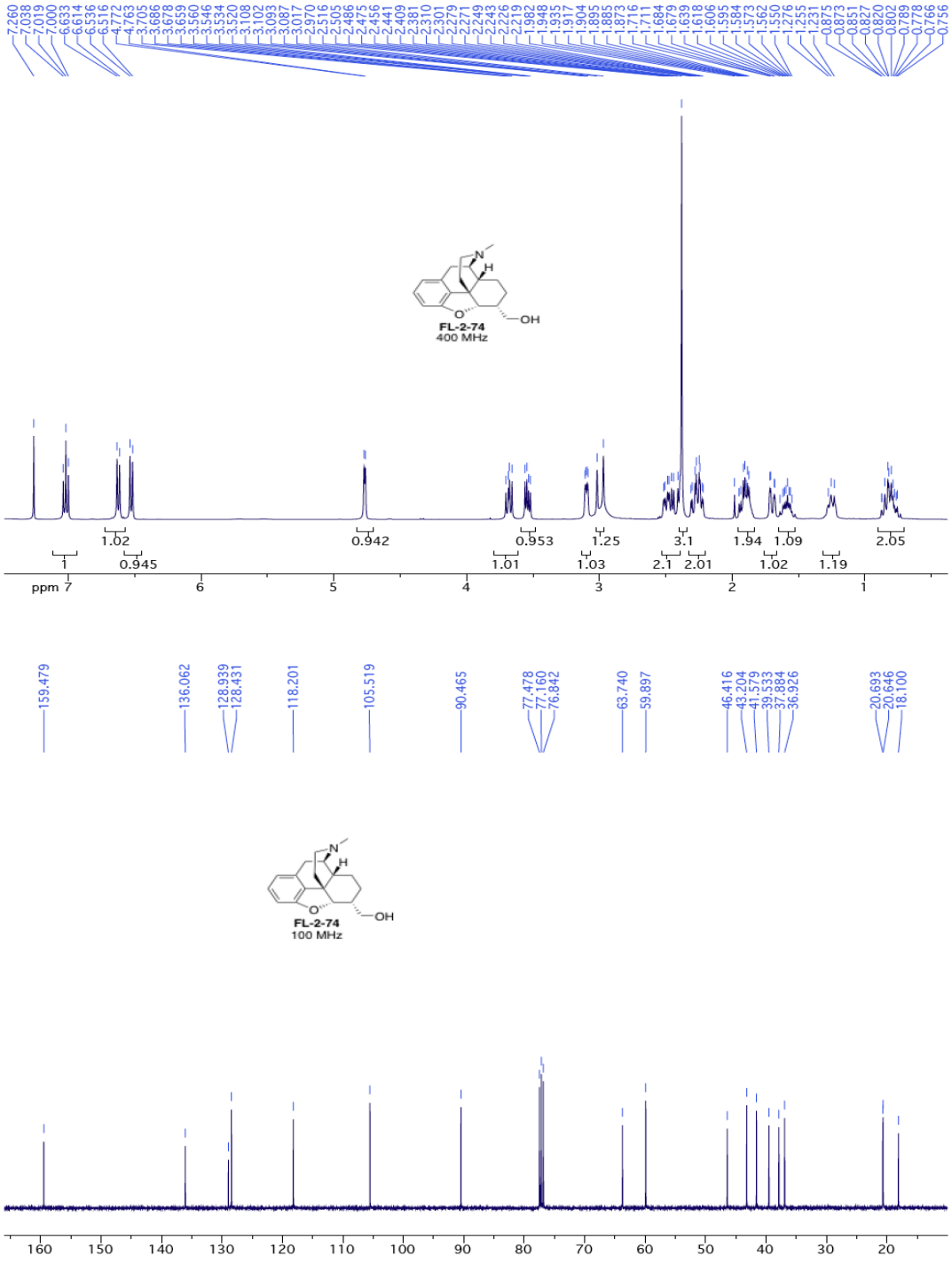
Compound 12a



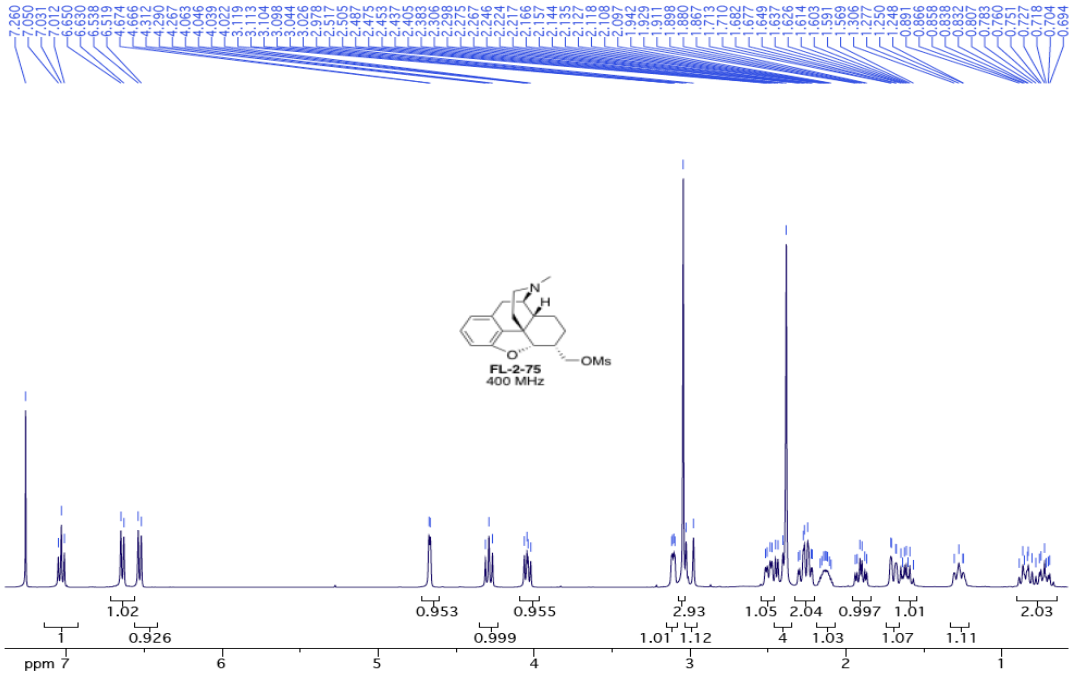


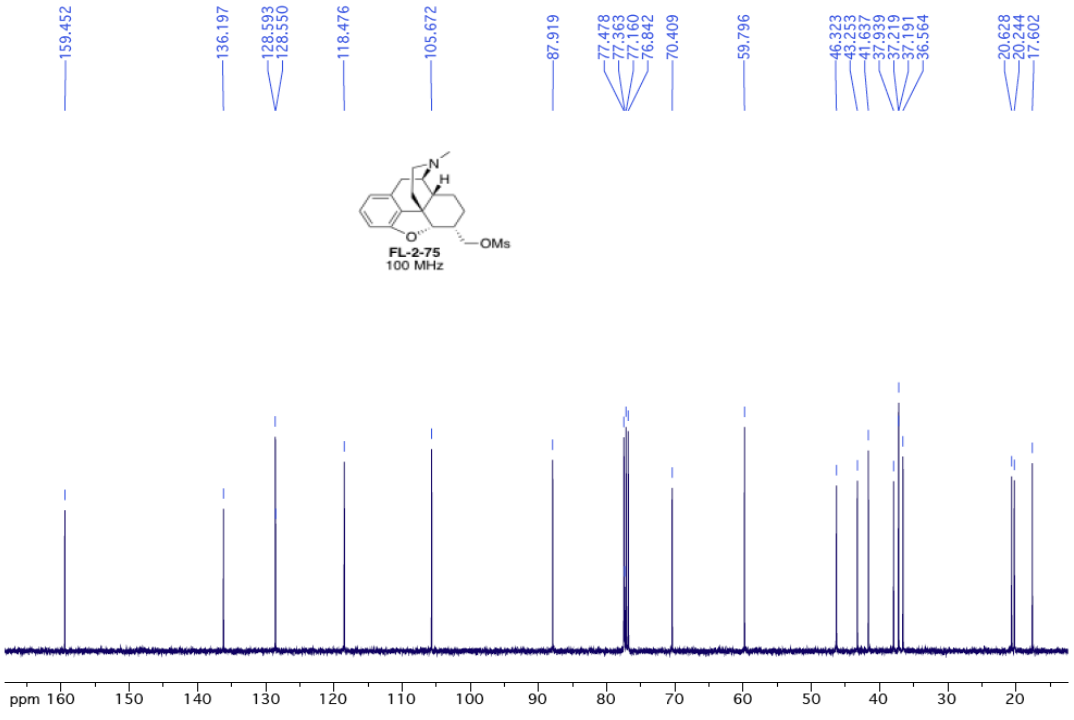


Compound 13a

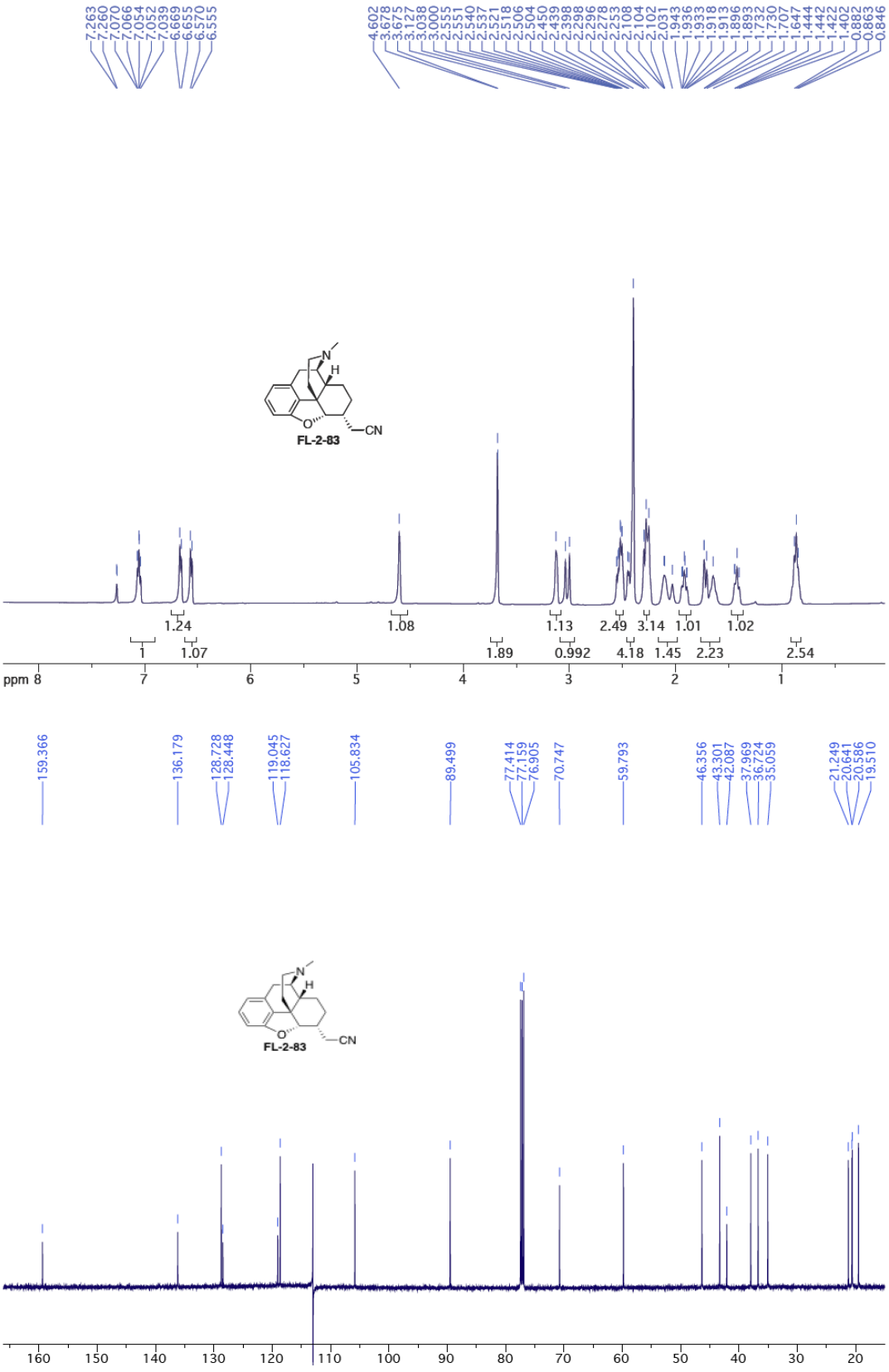


Compound 14a

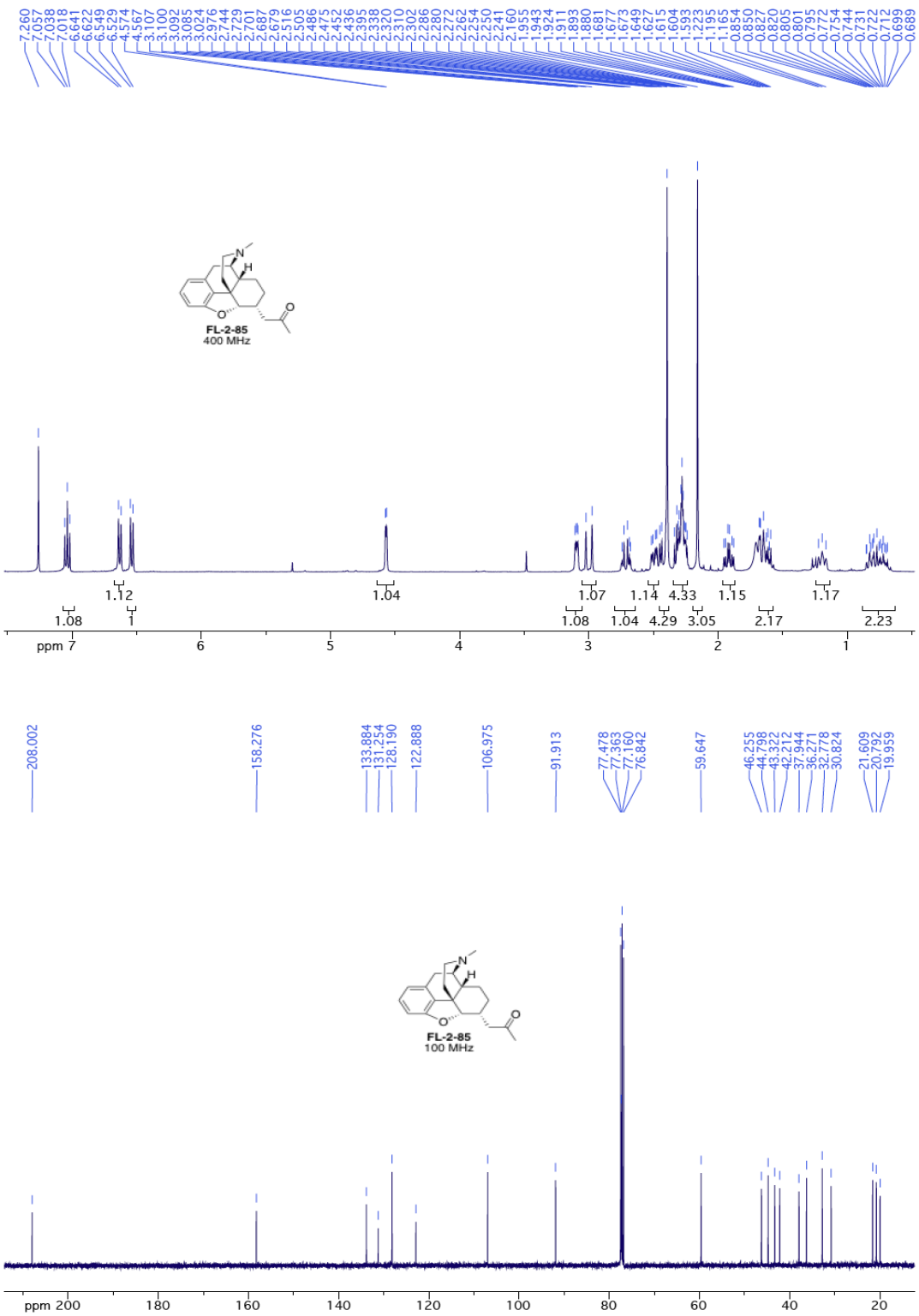




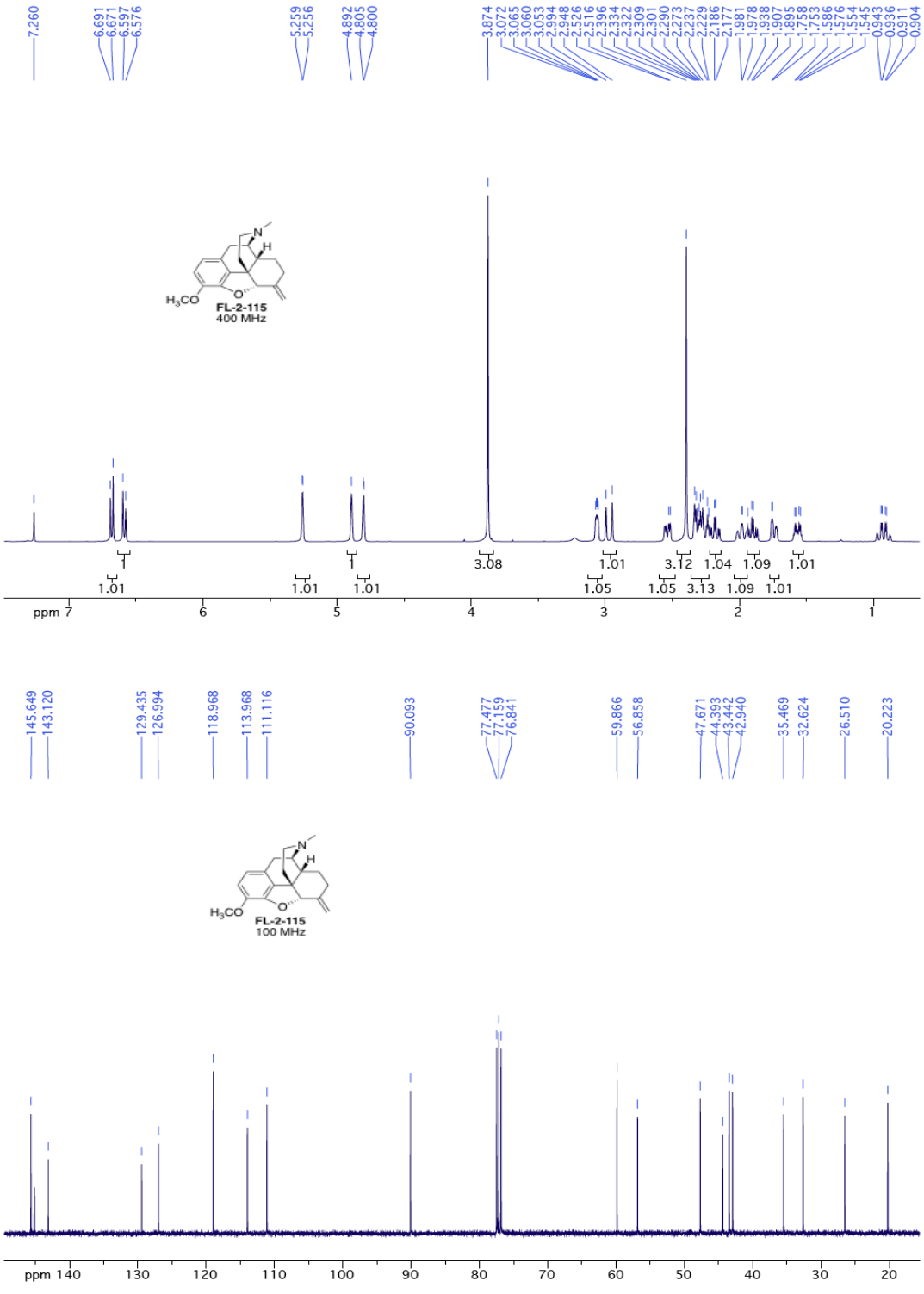
Compound 15a



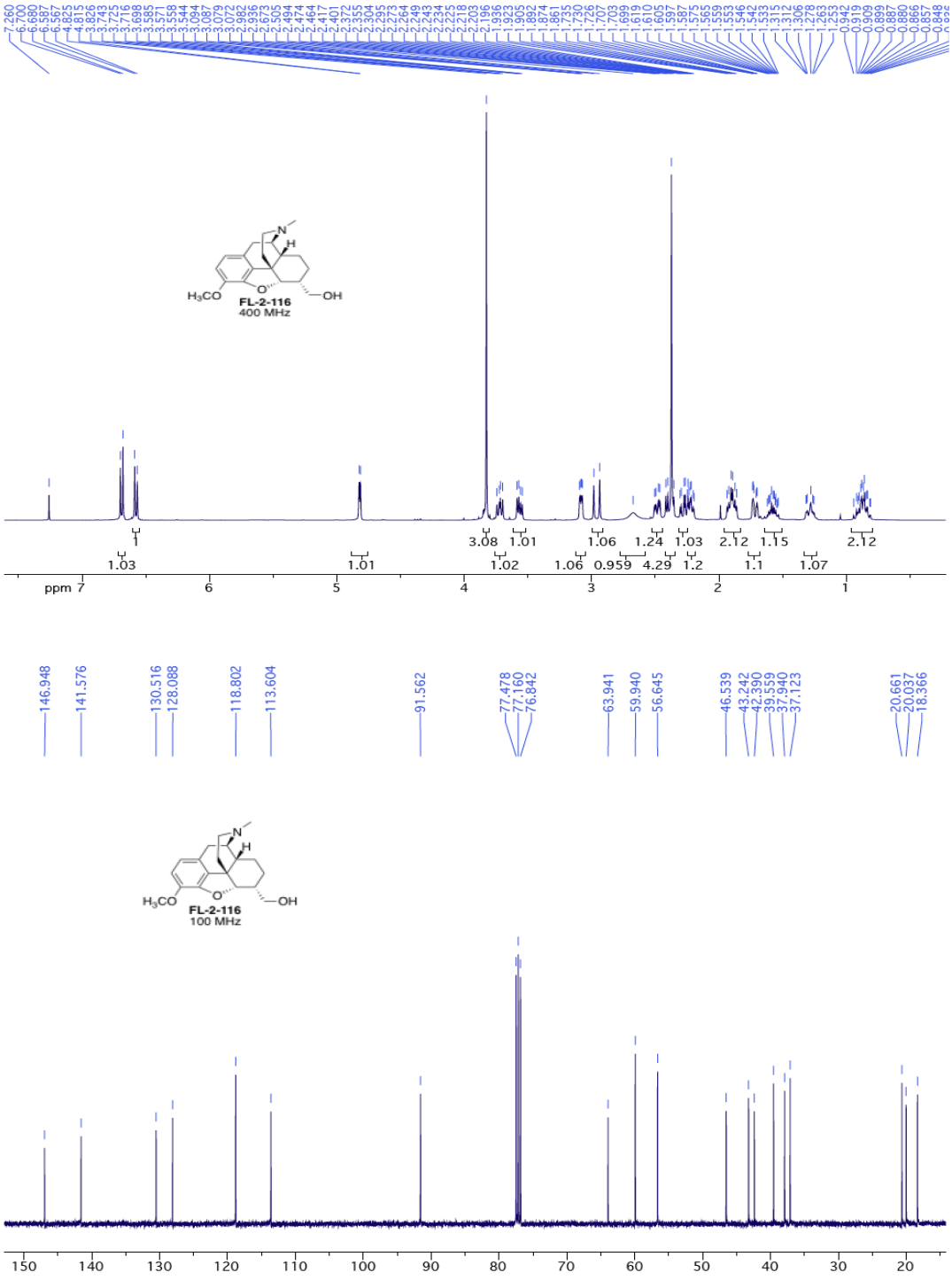
Compound 16a



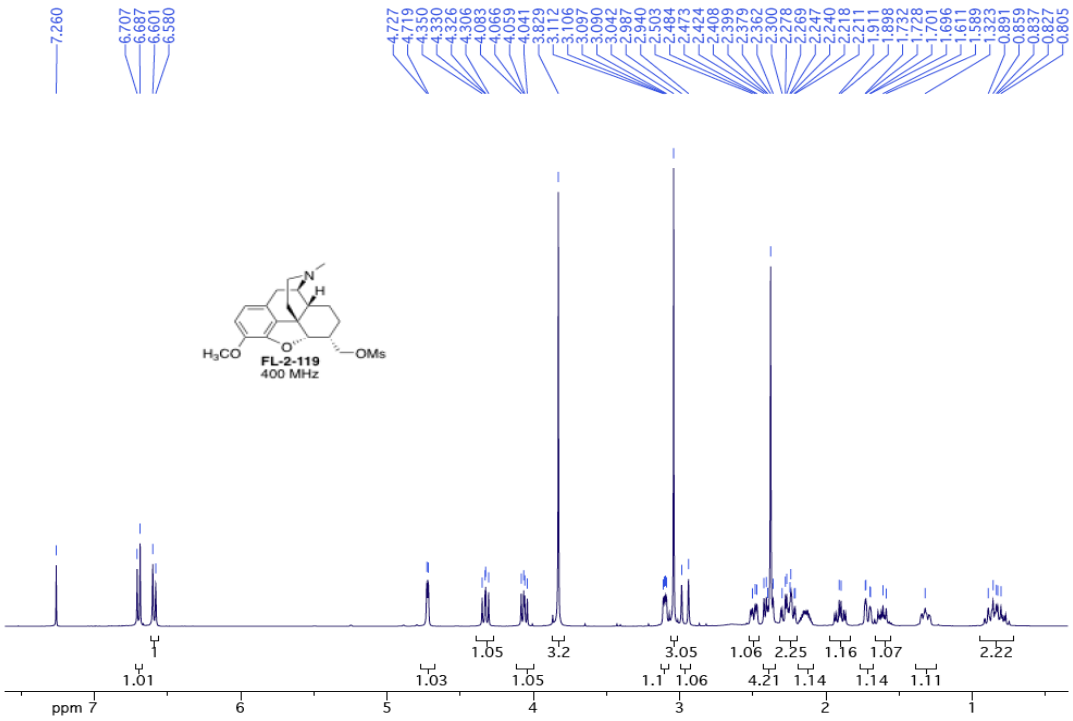
Compound 12b

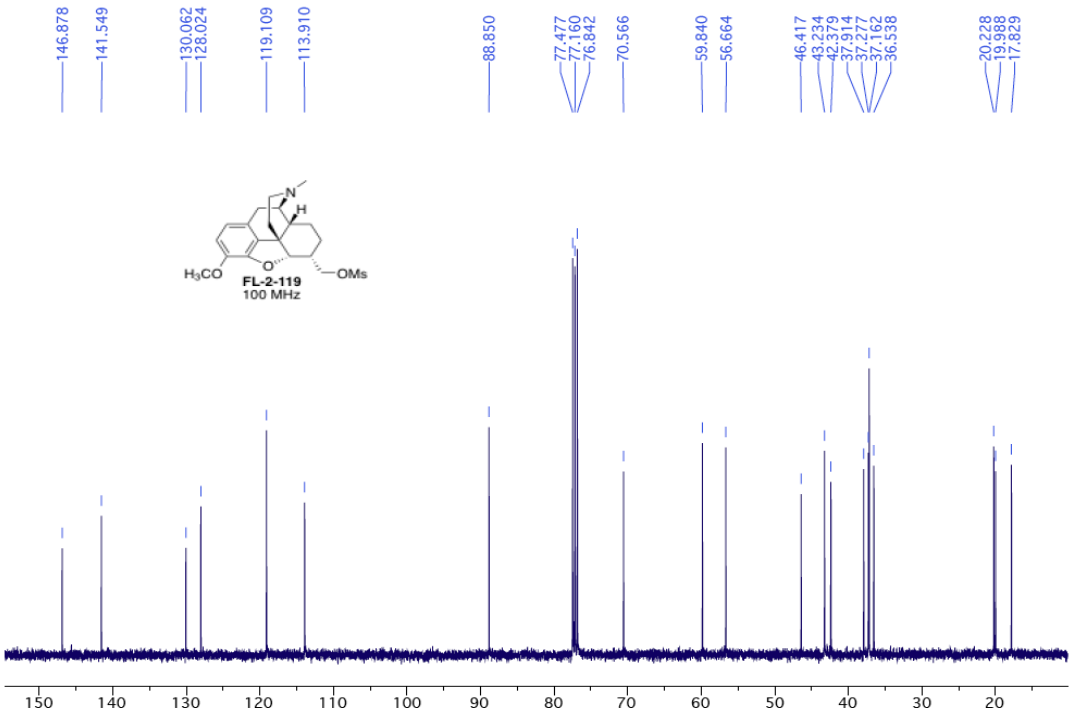


Compound 13b

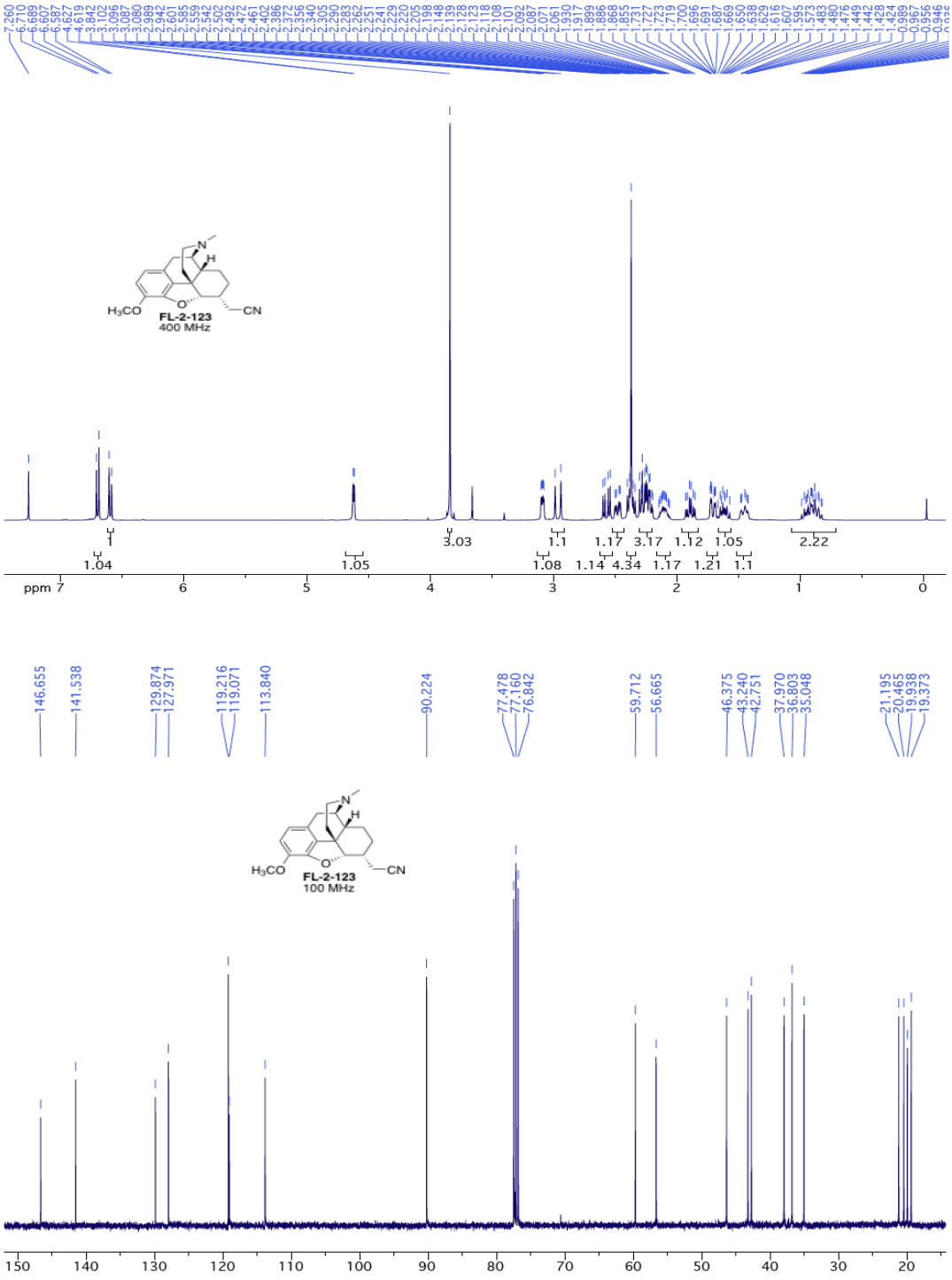


Compound 14b

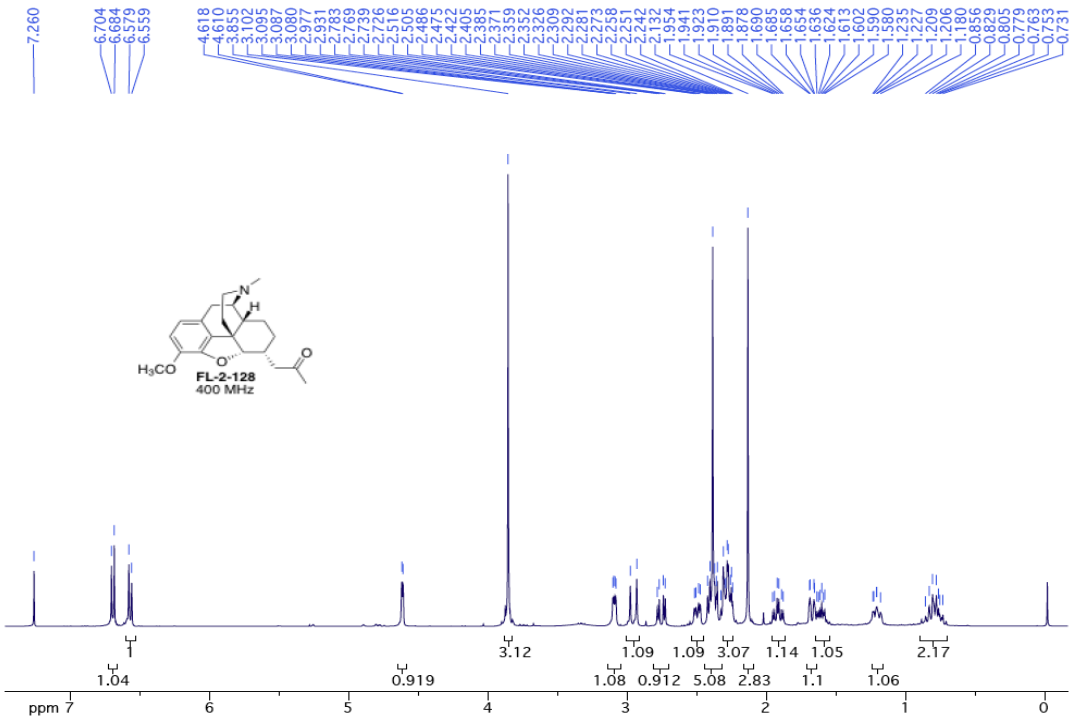


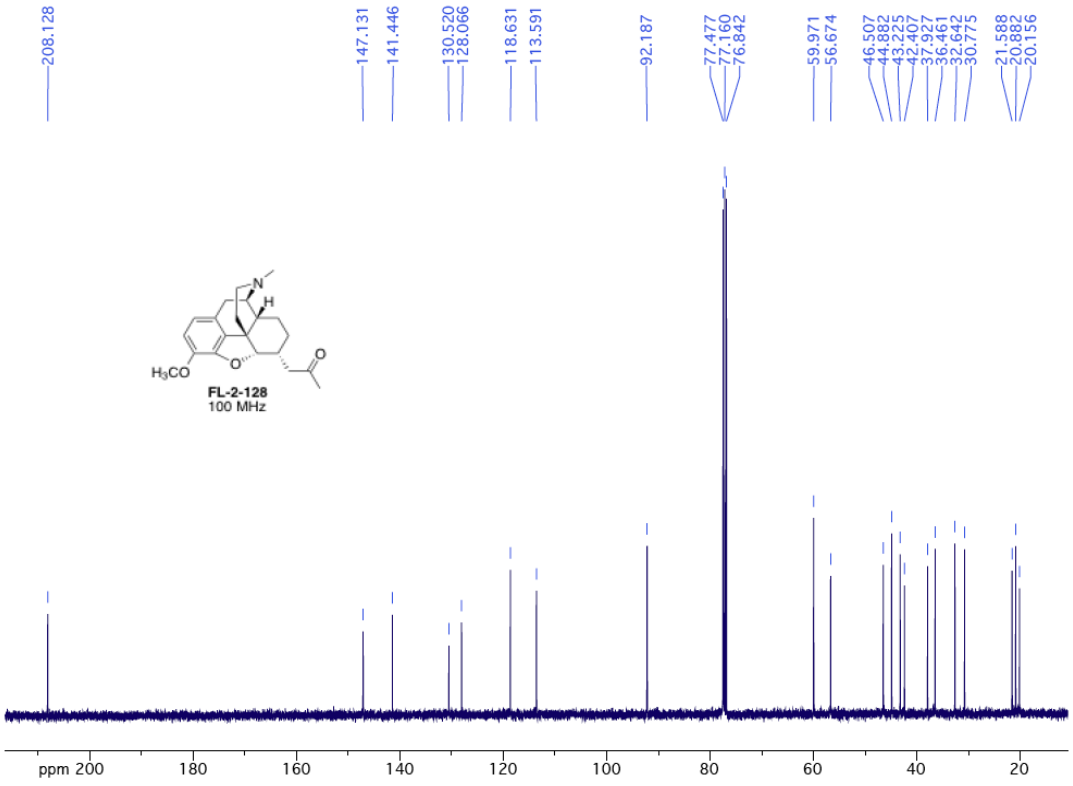


Compound 15b

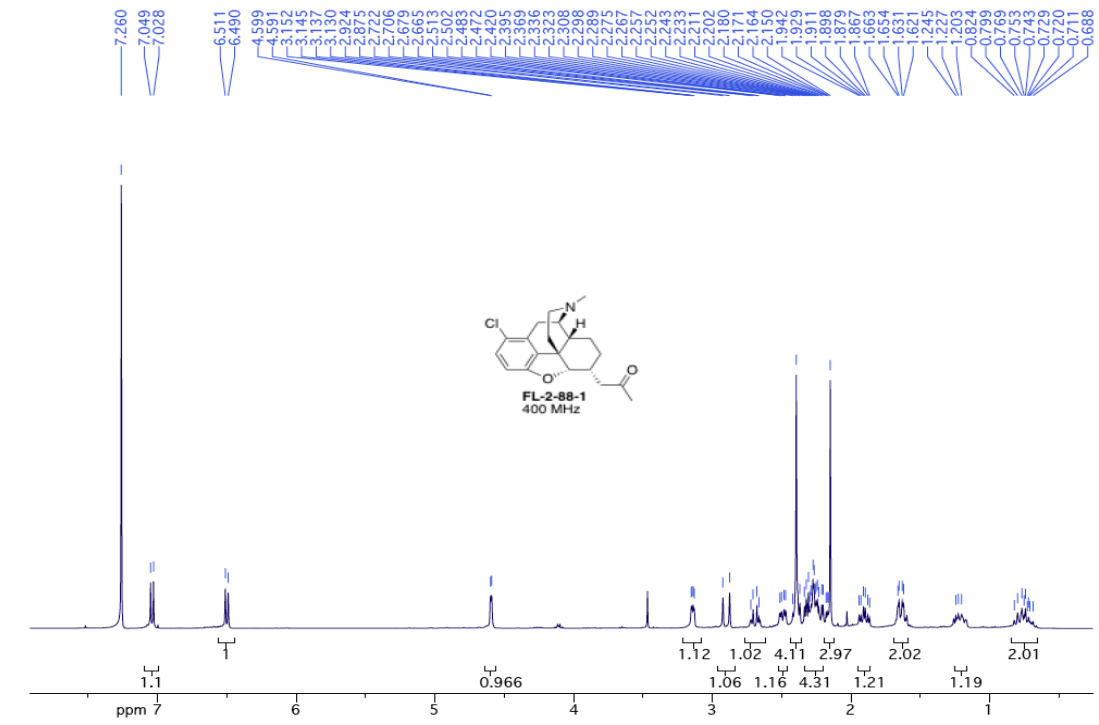


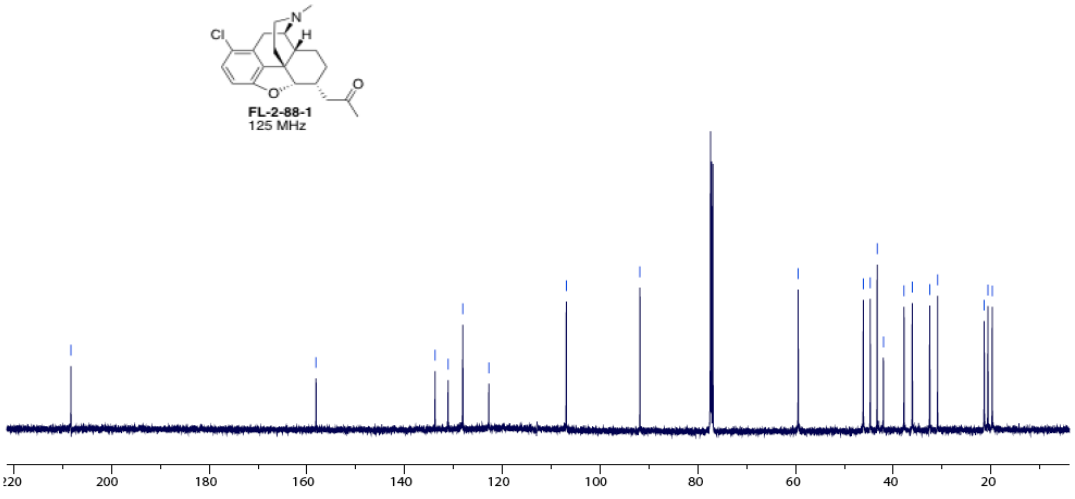
Compound 16b



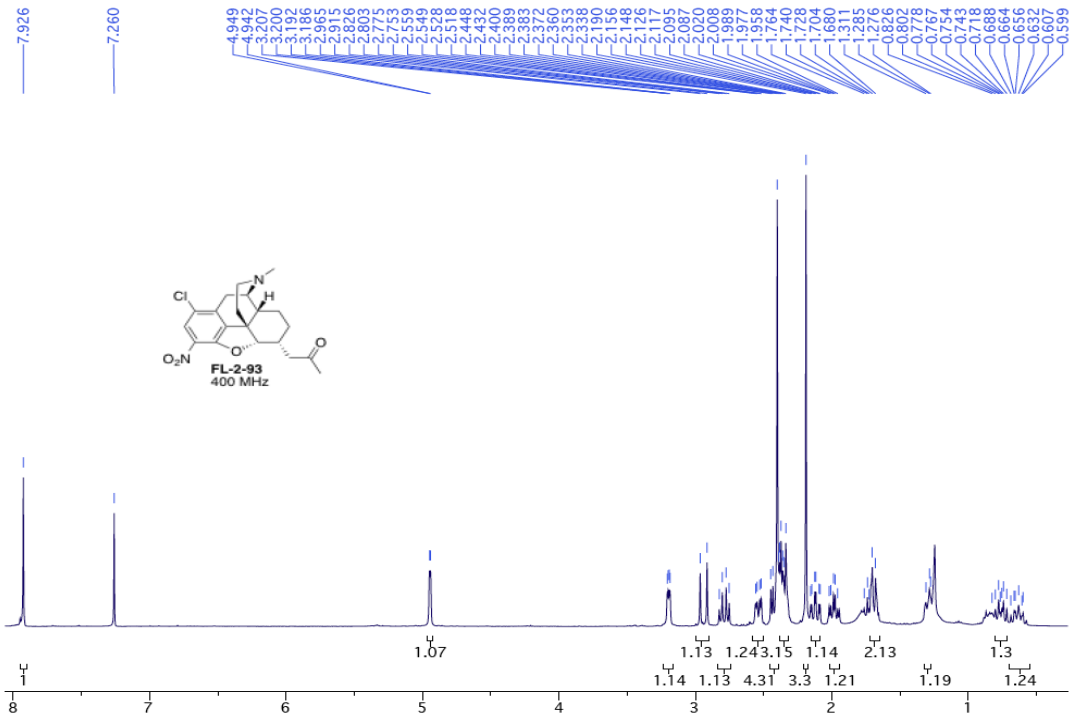


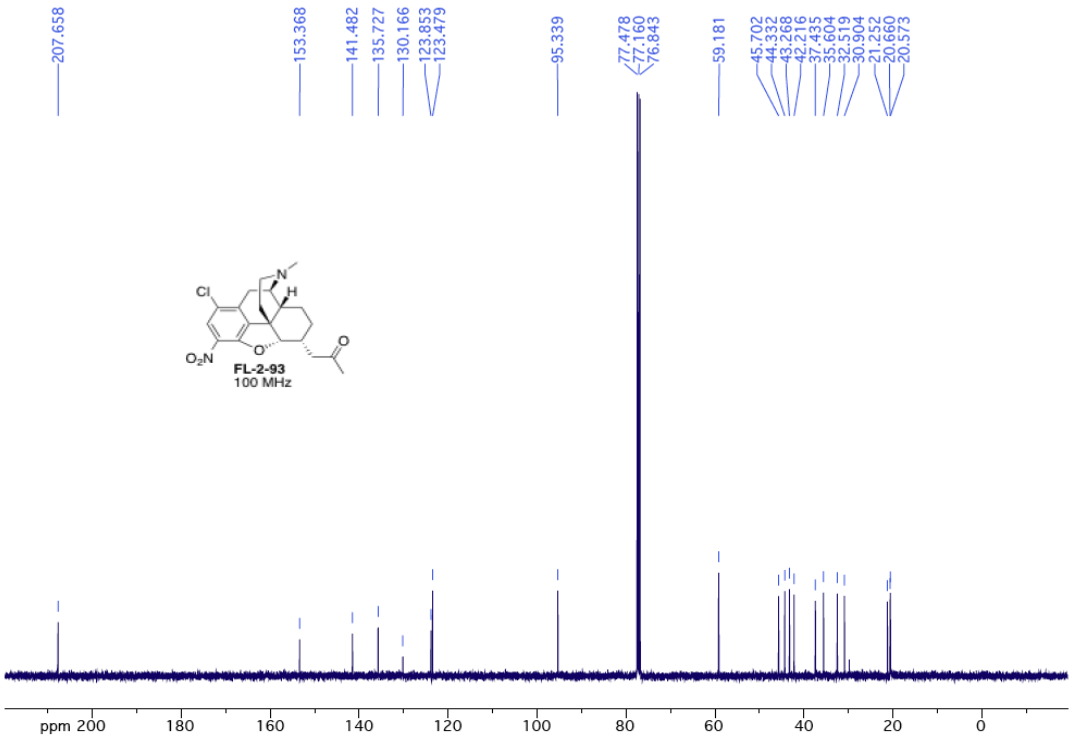
Compound 17



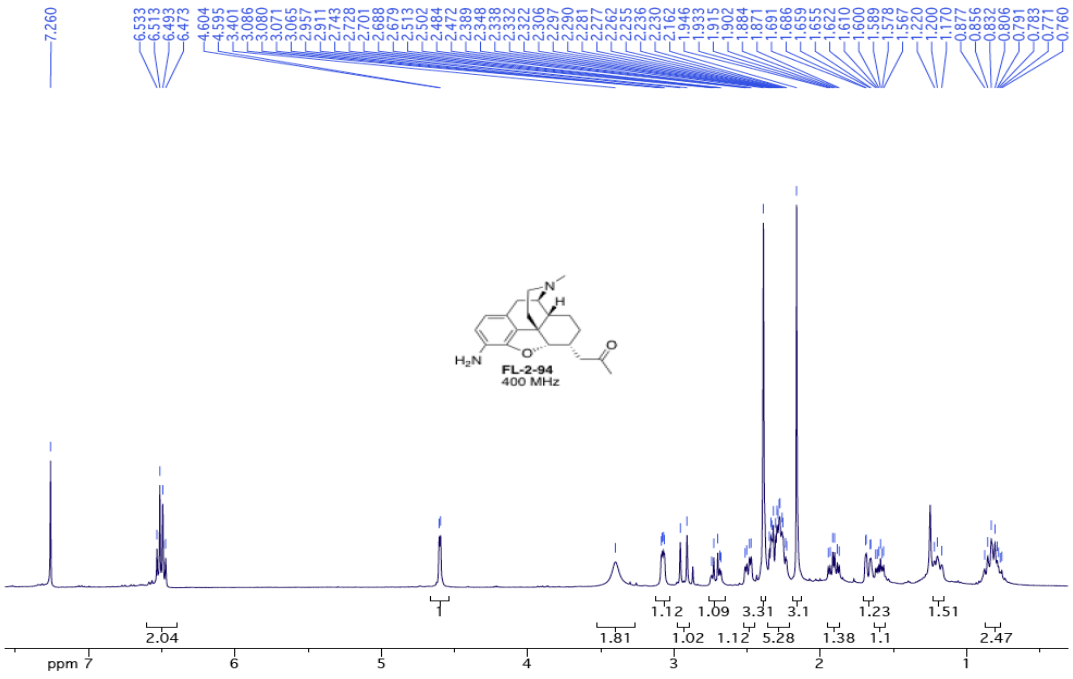


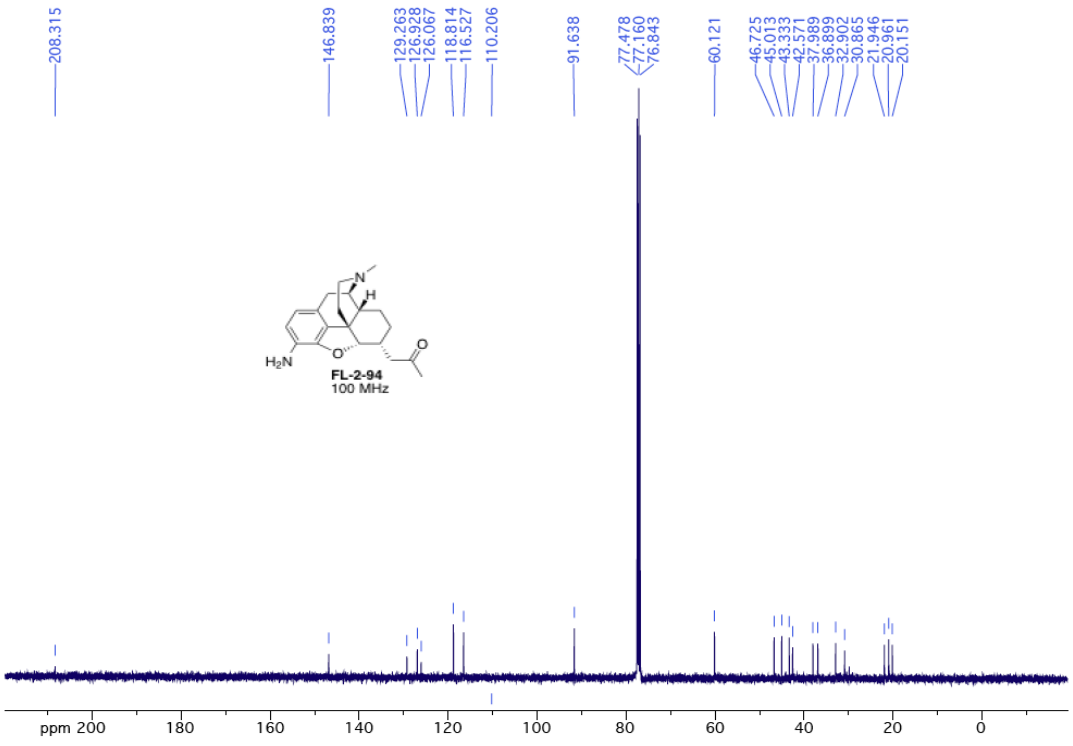
Compound 18


7.926
7.260
4.949
4.942
3.207
3.200
3.192
3.186
2.965
2.915
2.826
2.803
2.775
2.753
2.559
2.549
2.528
2.518
2.448
2.432
2.400
2.389
2.383
2.372
2.360
2.353
2.338
2.190
2.156
2.148
2.126
2.117
2.095
2.087
2.020
2.008
1.989
1.977
1.958
1.764
1.740
1.728
1.704
1.680
1.311
1.285
1.276
0.826
0.802
0.778
0.767
0.754
0.743
0.718
0.688
0.664
0.656
0.632
0.607
0.599
FL-2-93
400 MHz
1.07
1.13
1.24
3.15
1.14
2.13
1.3
1
1.14
1.13
4.31
3.3
1.21
1.19
1.24
8
7
6
5
4
3
2
1

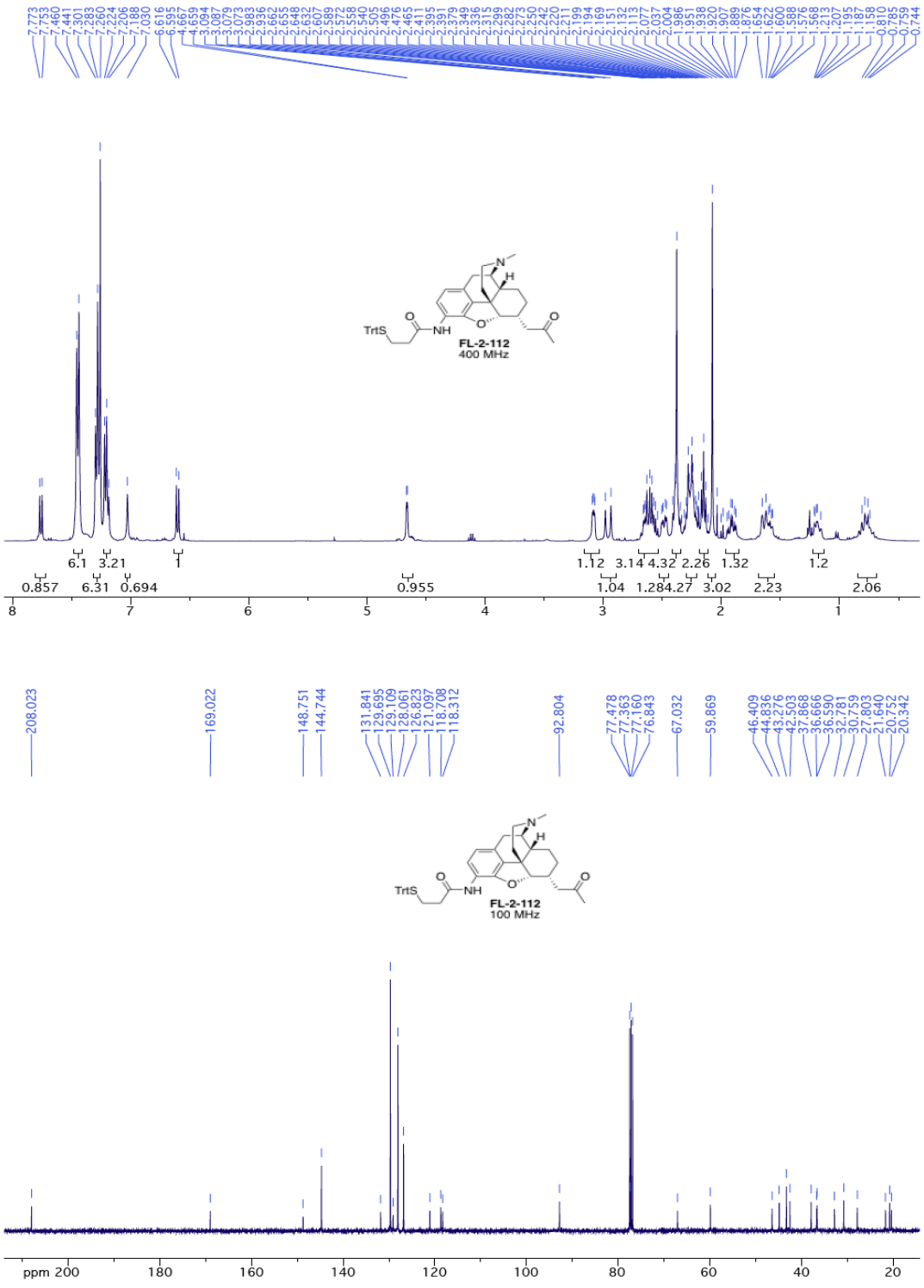

207.658
153.368
141.482
135.727
130.166
123.853
123.479
95.339
77.478
77.160
76.843
59.181
45.702
44.332
43.268
42.216
37.435
35.604
32.519
30.904
21.252
20.660
20.573
FL-2-93
100 MHz
ppm 200
180
160
140
120
100
80
60
40
20
0

Compound 19

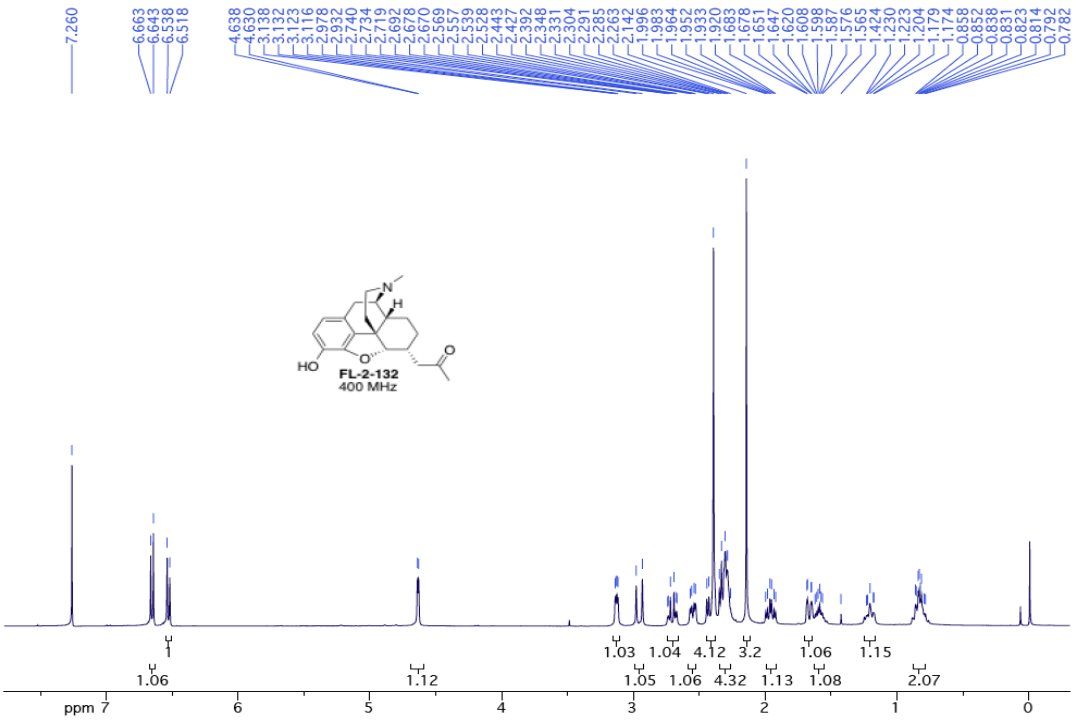

FL-2-94
400 MHz
ppm 7
6
5
4
3
2
1

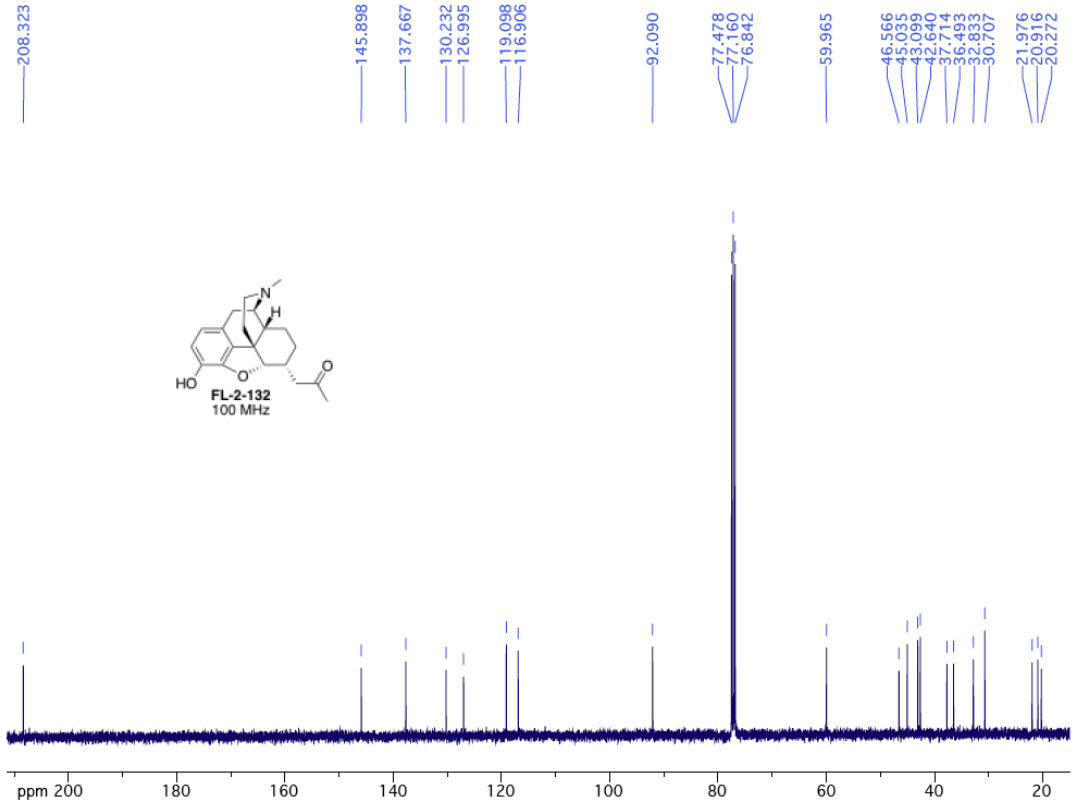

FL-2-94
100 MHz
ppm 200
180
160
140
120
100
80
60
40
20
0

Compound 1 (6-PrOxyHap)

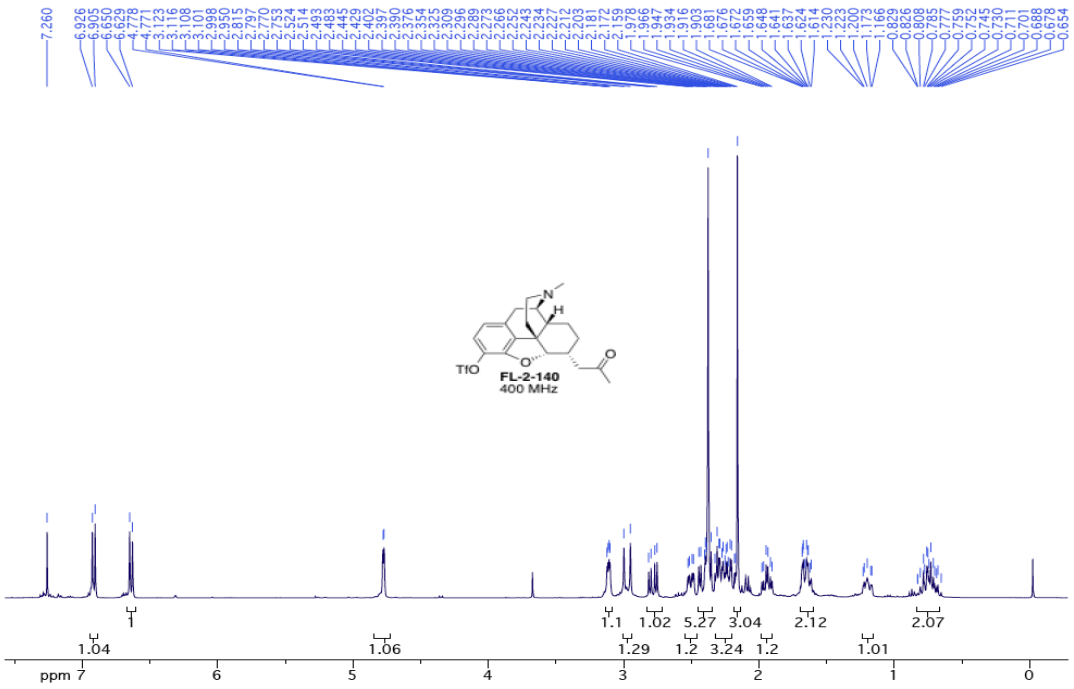


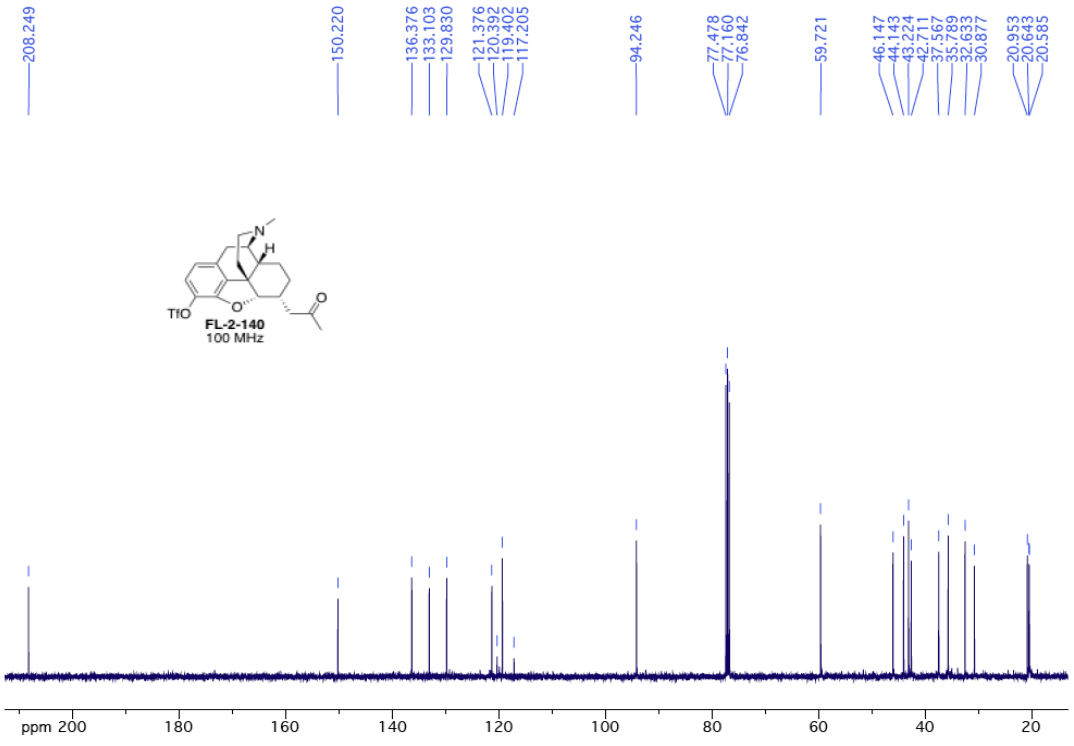
Compound 20


7.260
6.663
6.643
6.538
6.518
4.638
4.630
3.138
3.132
3.123
3.116
2.978
2.932
2.740
2.734
2.719
2.692
2.678
2.670
2.569
2.557
2.539
2.528
2.443
2.427
2.392
2.348
2.331
2.304
2.291
2.285
2.263
2.142
1.996
1.983
1.964
1.952
1.333
1.320
1.683
1.678
1.651
1.647
1.620
1.608
1.598
1.587
1.576
1.565
1.424
1.230
1.223
1.204
1.179
1.174
0.858
0.852
0.838
0.831
0.823
0.814
0.792
0.782
FL-2-132
400 MHz
1
1.06
1.12
1.03
1.04
4.12
3.2
1.06
1.15
1.05
1.06
4.32
1.13
1.08
2.07
ppm 7
6
5
4
3
2
1
0

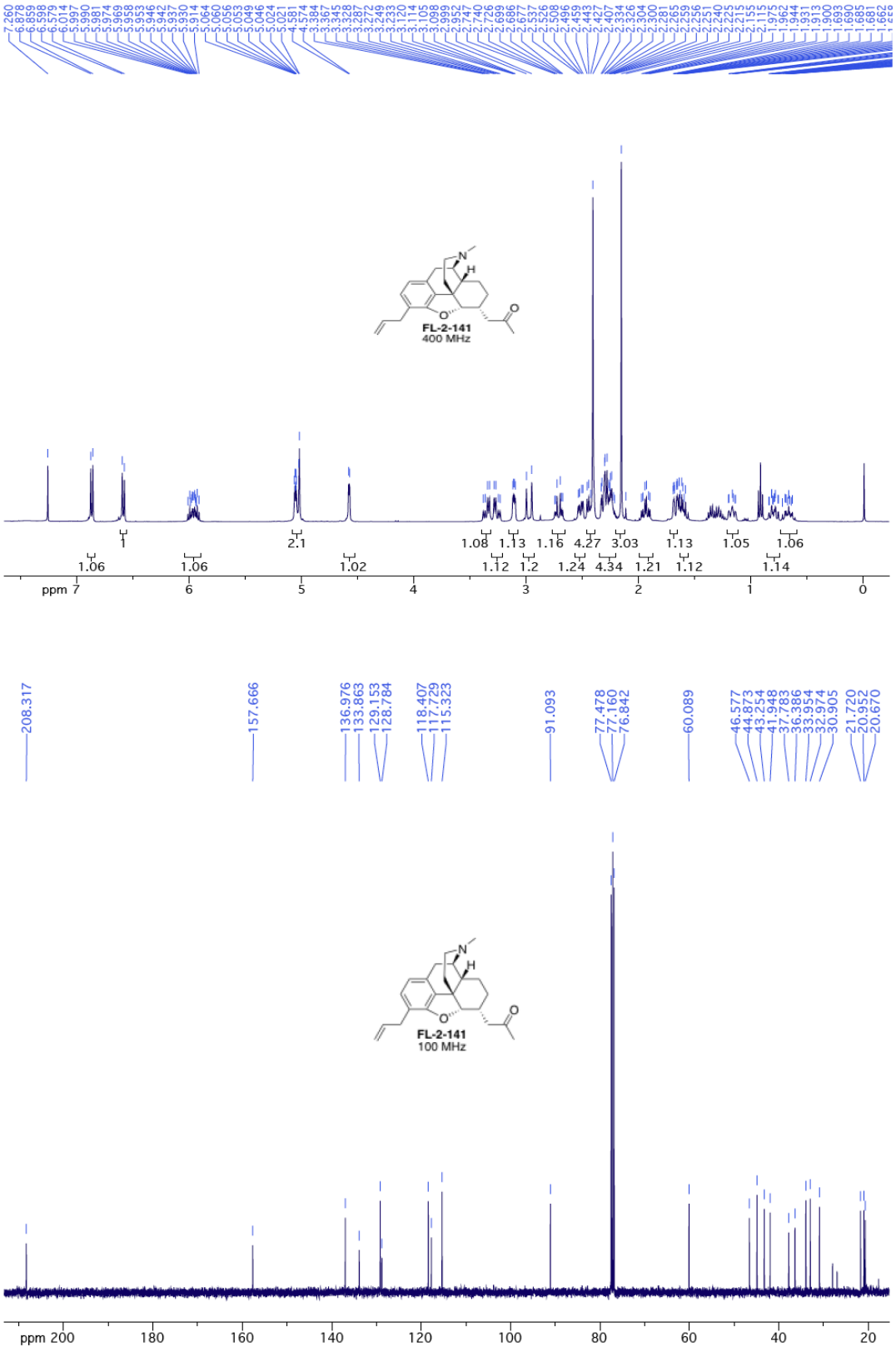

208.323
145.898
137.667
130.232
126.995
119.098
116.906
92.090
77.478
77.160
76.842
59.965
46.566
45.035
43.099
42.640
37.714
36.493
32.833
30.707
21.976
20.916
20.272
FL-2-132
100 MHz
ppm 200
180
160
140
120
100
80
60
40
20

Compound 21

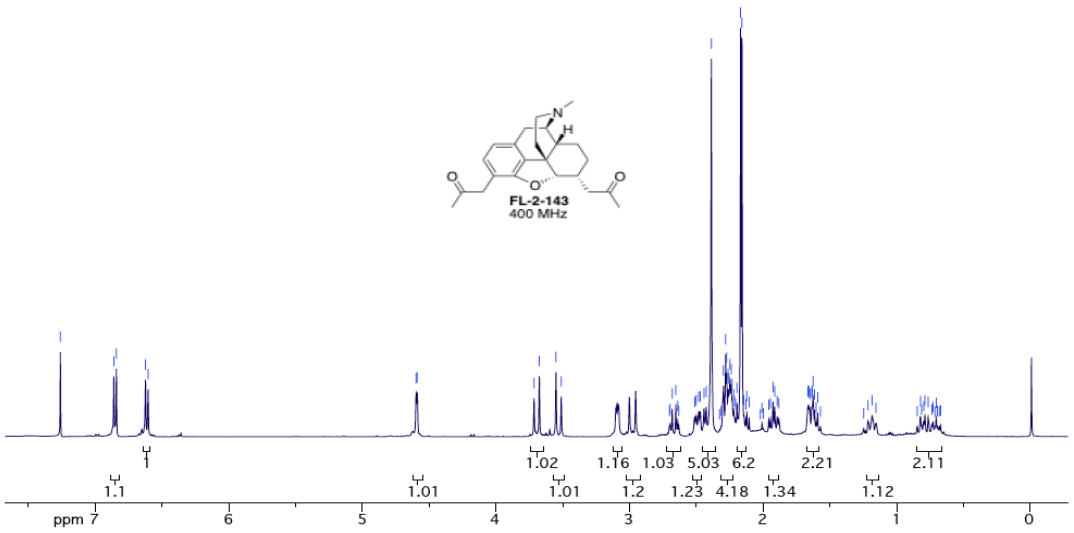


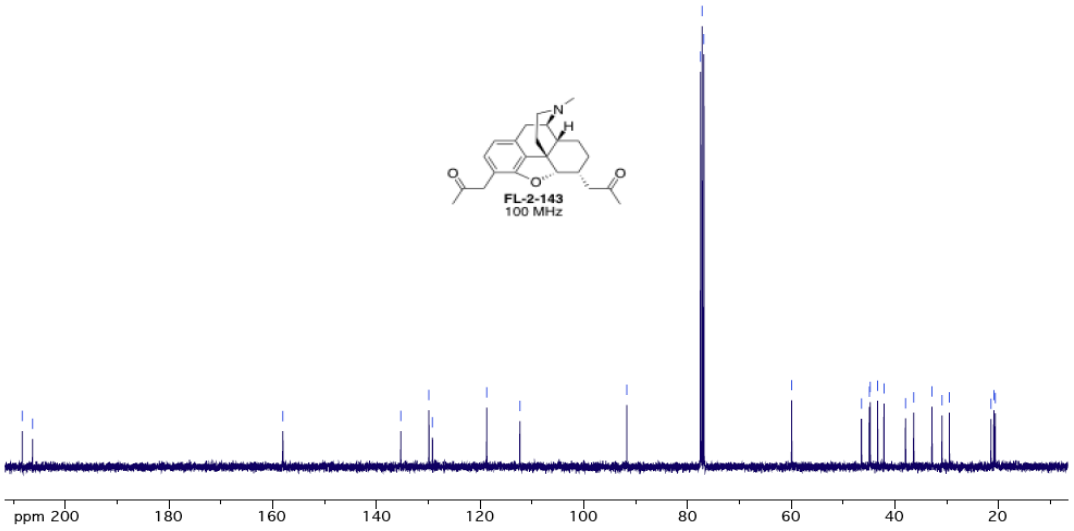


Compound 22

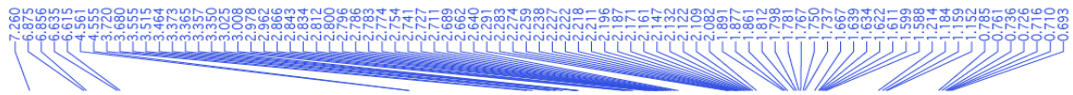


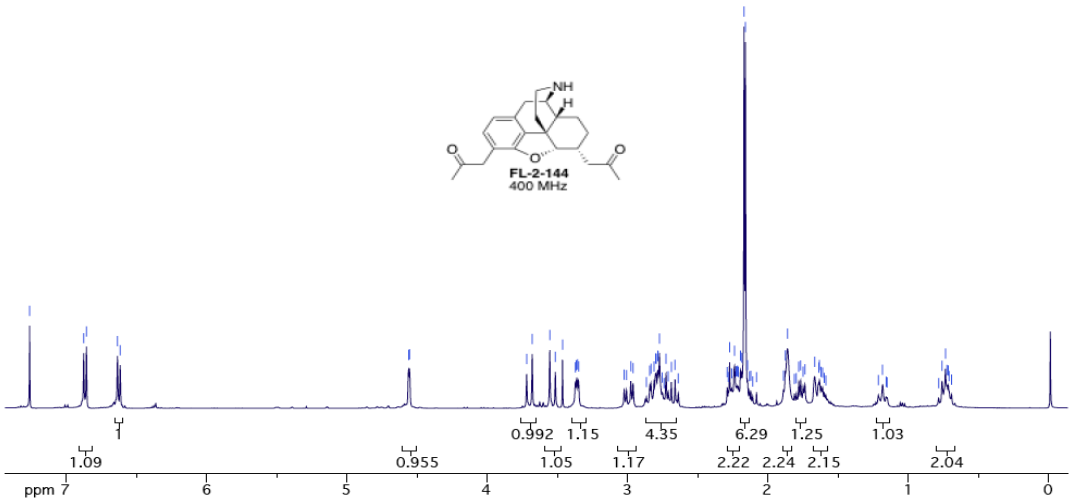
Compound 23

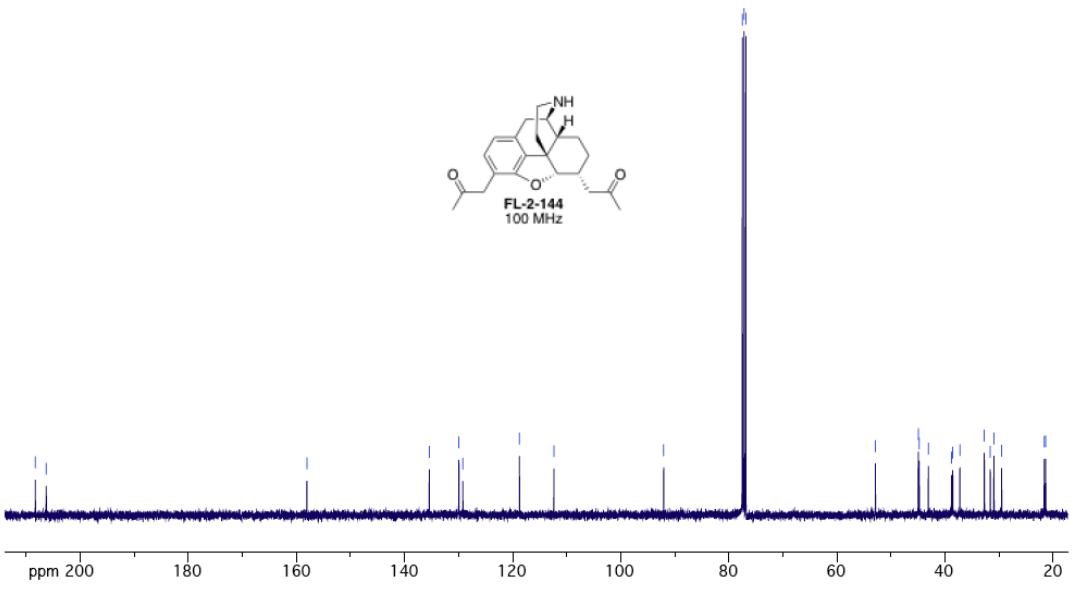

FL-2-143
400 MHz
1
1.02
1.16
1.03
5.03
6.2
2.21
2.11
1.1
1.01
1.01
1.2
1.23
4.18
1.34
1.12
ppm 7
6
5
4
3
2
1
0


FL-2-143
100 MHz
ppm 200
180
160
140
120
100
80
60
40
20

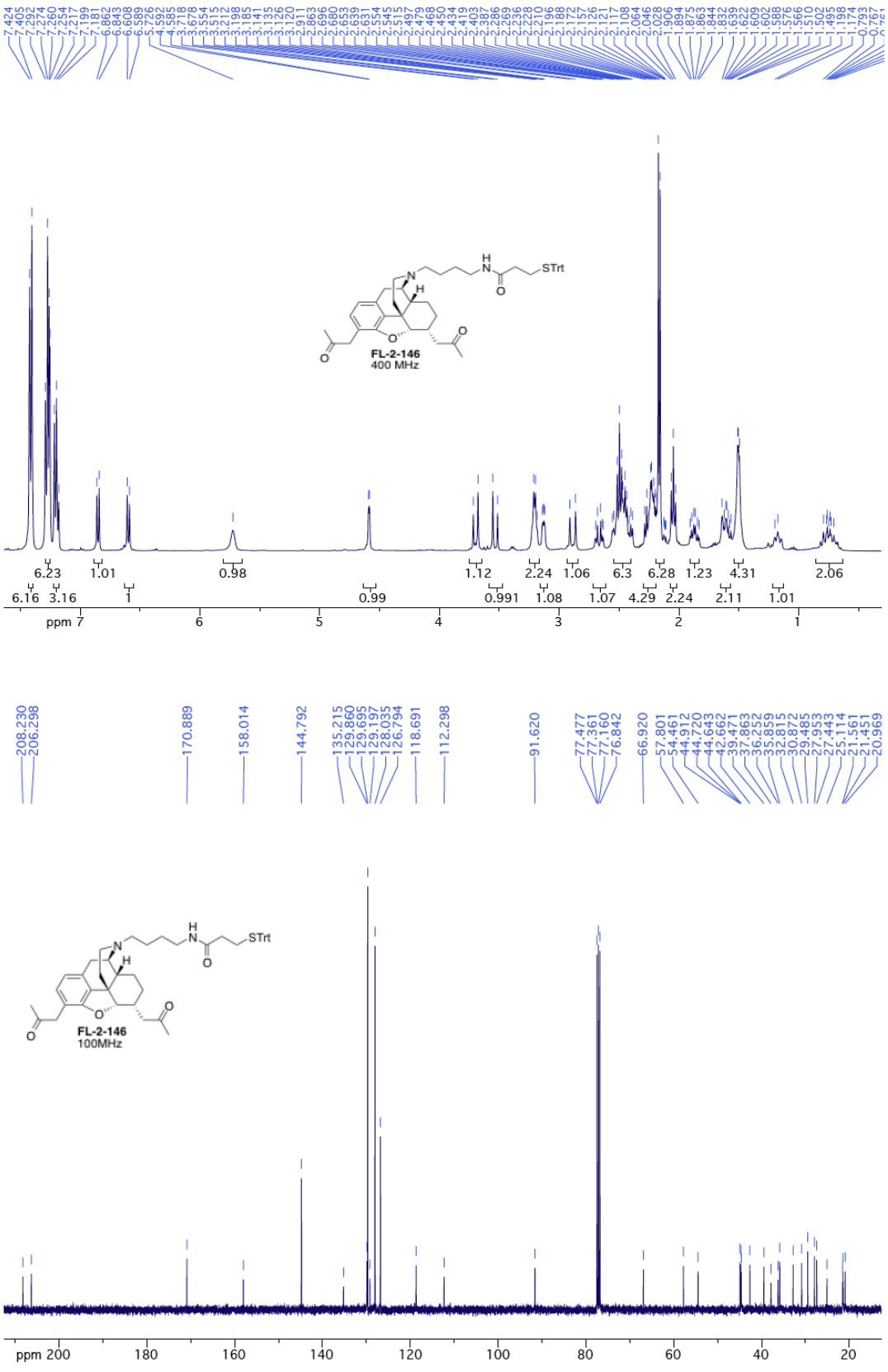
Compound 24



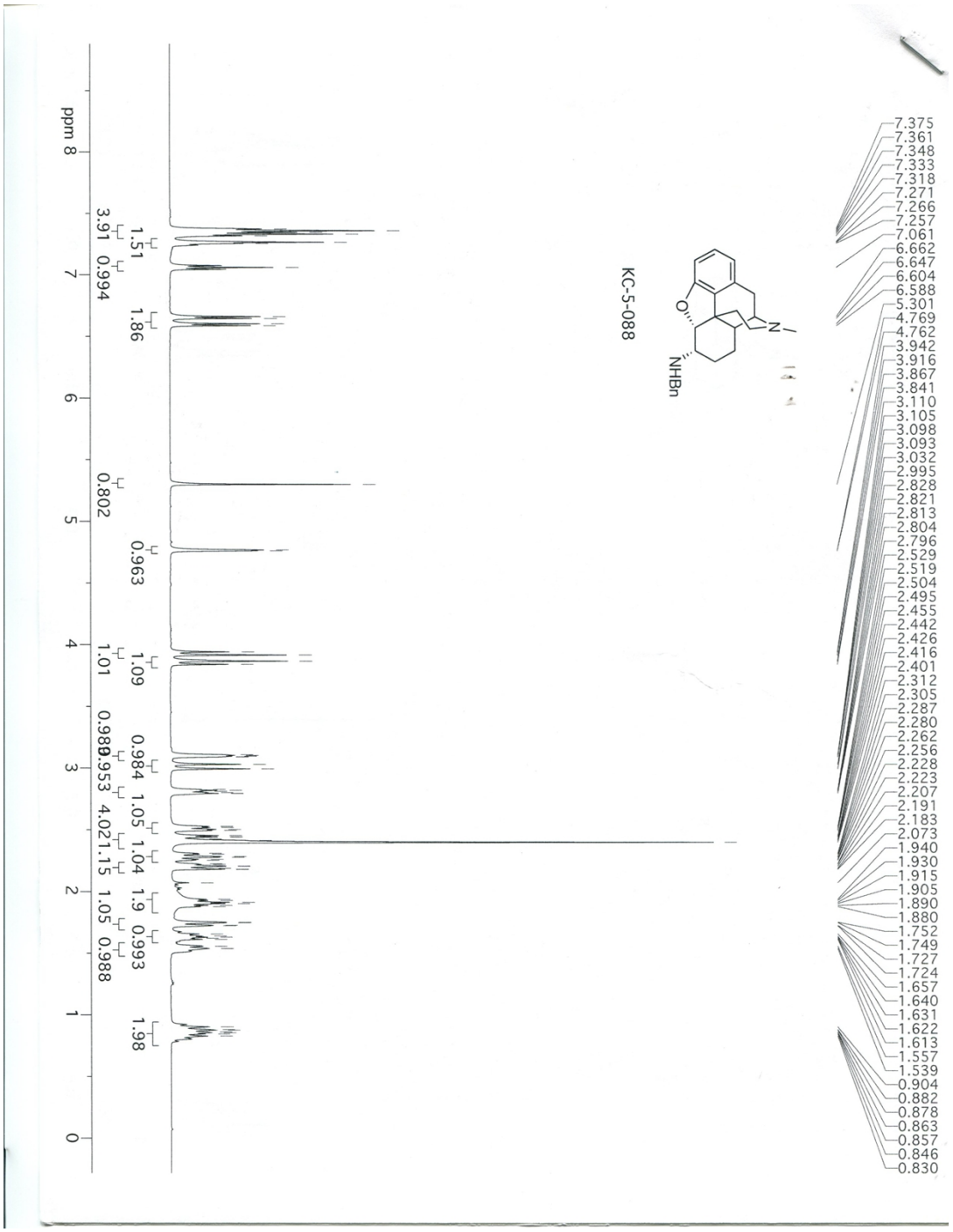

NH
H
O
O
O
FL-2-144
400 MHz
1
0.992
1.15
4.35
6.29
1.25
1.03
1.09
0.955
1.05
1.17
2.22
2.24
2.15
2.04
ppm 7
6
5
4
3
2
1
0


NH
H
O
O
O
FL-2-144
100 MHz
ppm 200
180
160
140
120
100
80
60
40
20

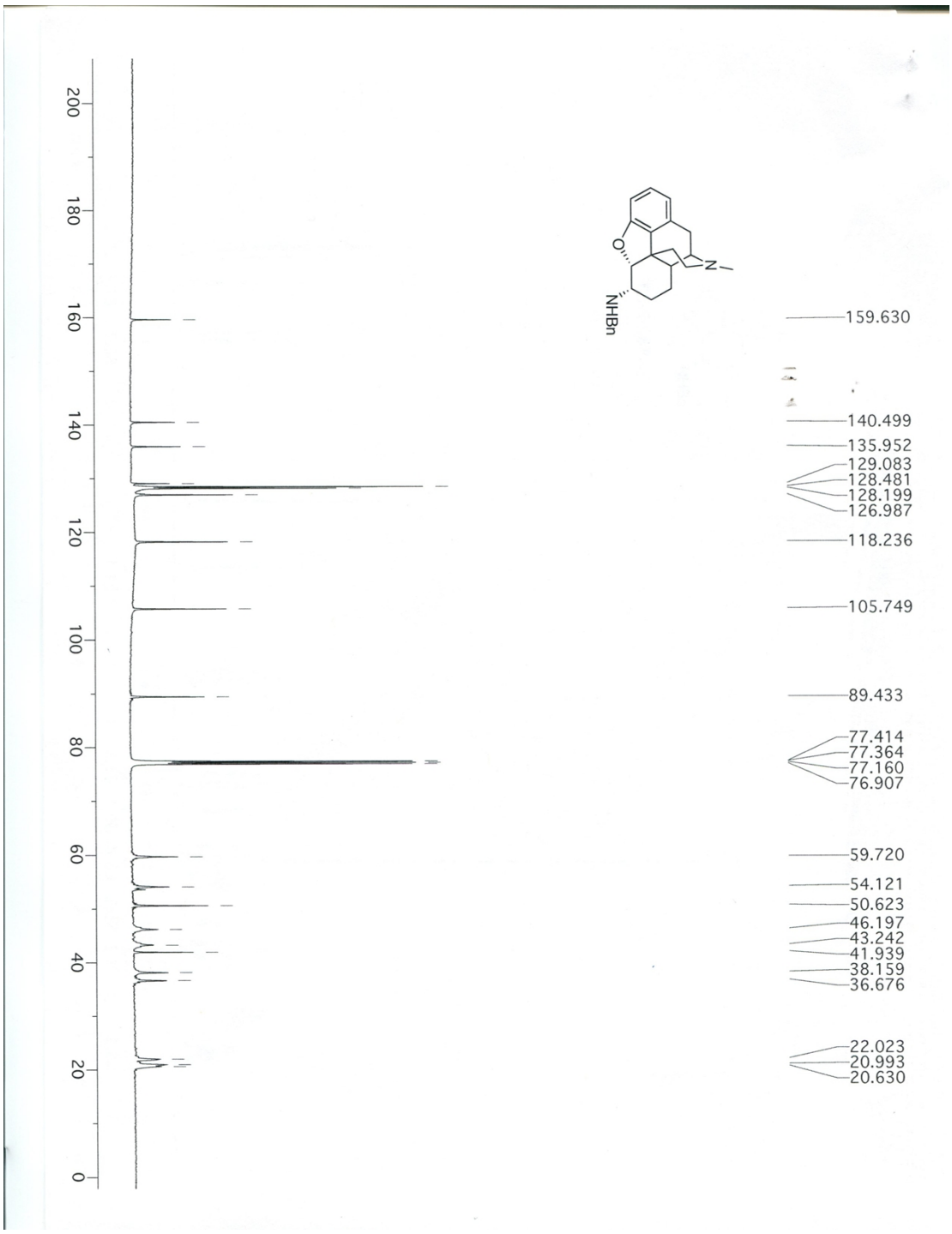
Compound 2 (DiPrOxyHap)



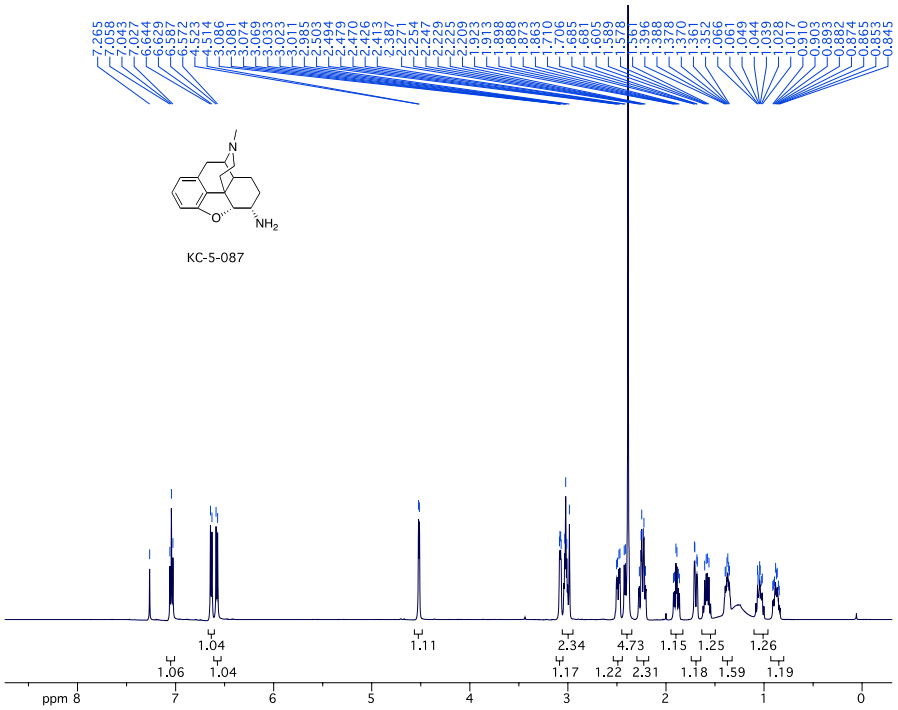
Compound 26

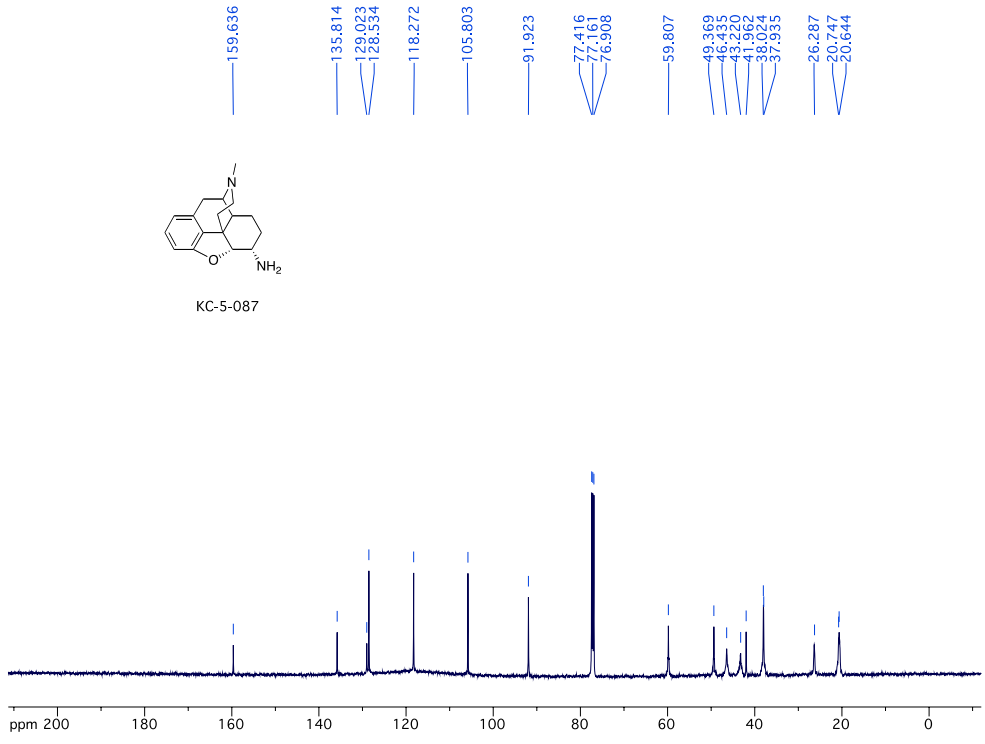

KC-5-088
NHBn
ppm
7.375
7.361
7.348
7.333
7.318
7.271
7.266
7.257
7.061
6.662
6.647
6.604
6.588
5.301
4.769
4.762
3.942
3.916
3.867
3.841
3.110
3.105
3.098
3.093
3.032
2.995
2.828
2.821
2.813
2.804
2.796
2.529
2.519
2.504
2.495
2.455
2.442
2.426
2.416
2.401
2.312
2.305
2.287
2.280
2.262
2.256
2.228
2.223
2.207
2.191
2.183
2.073
1.940
1.930
1.915
1.905
1.890
1.880
1.752
1.749
1.727
1.724
1.657
1.640
1.631
1.622
1.613
1.557
1.539
0.904
0.882
0.878
0.863
0.857
0.846
0.830

^{13}C NMR spectrum of Compound **26**

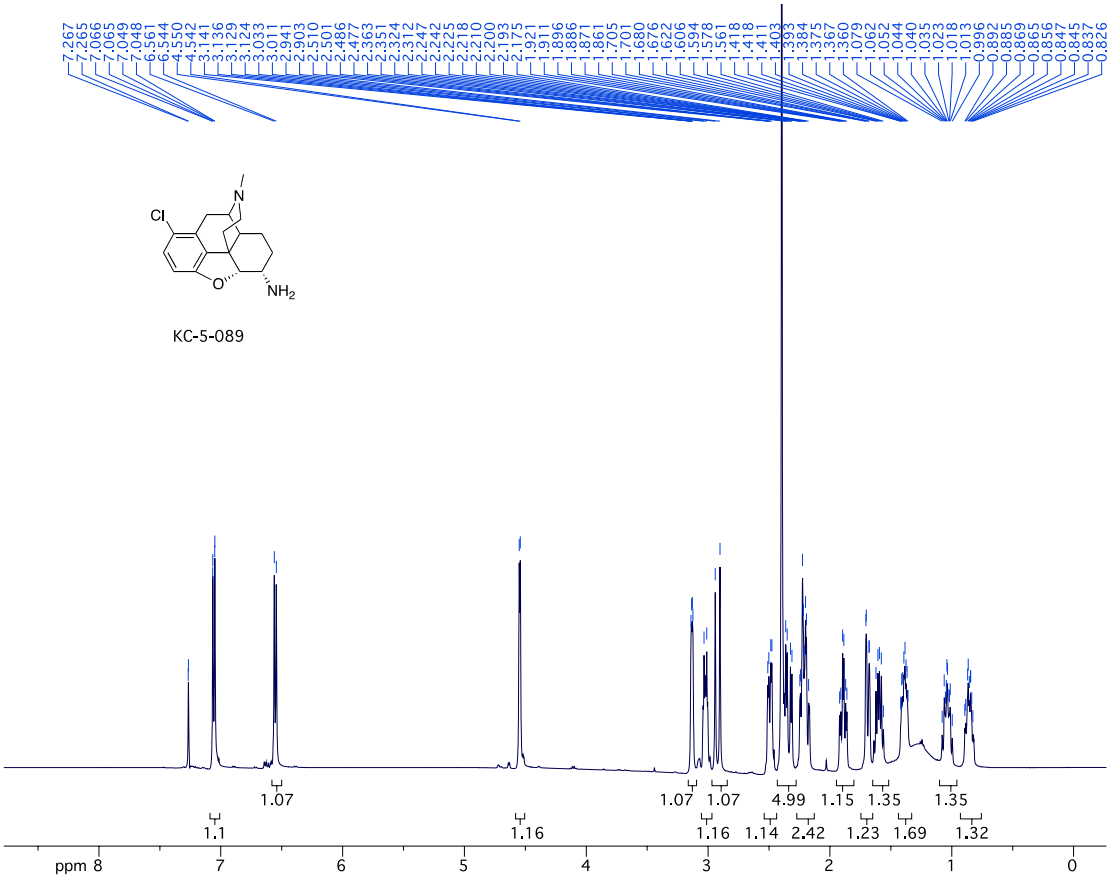


Compound **27**

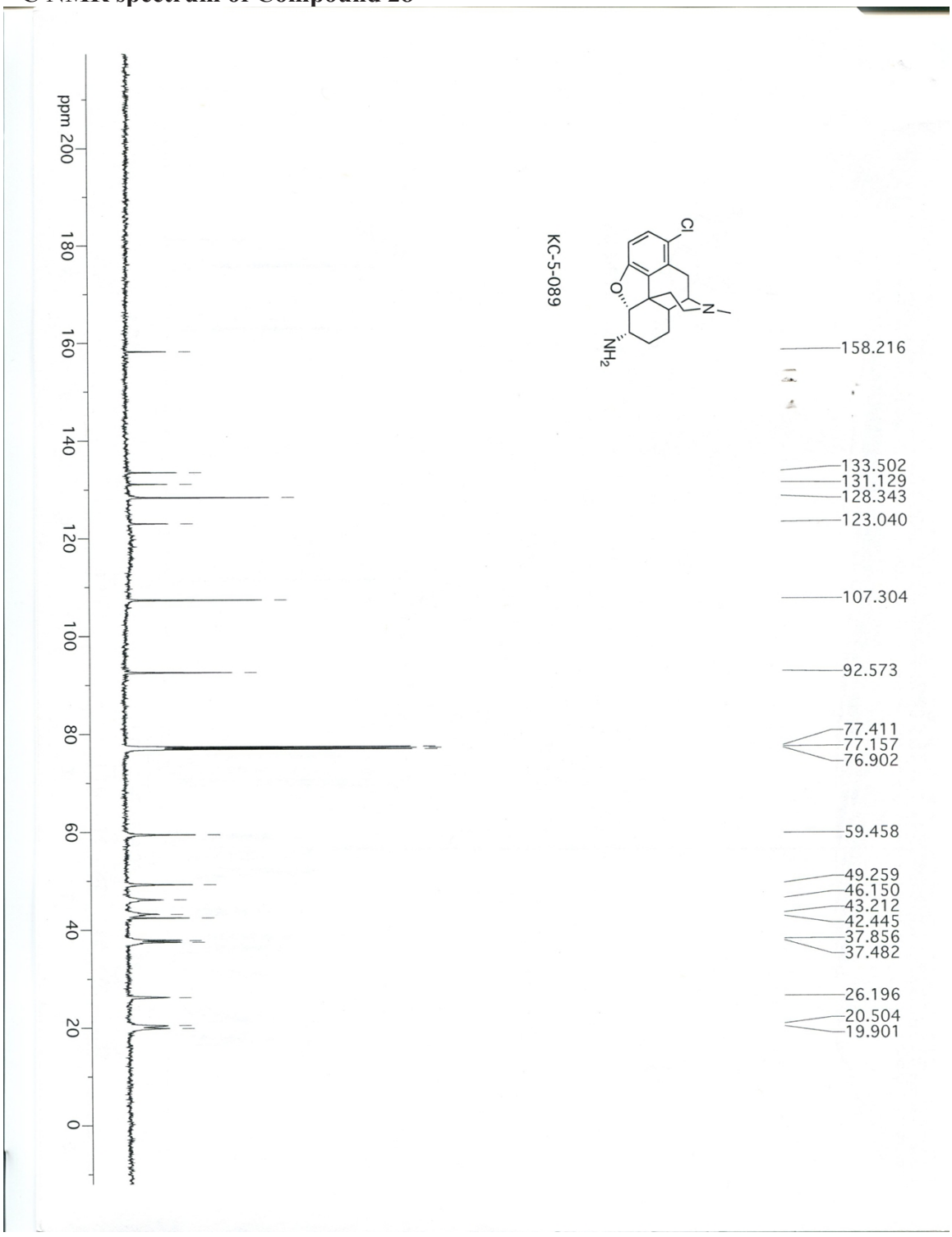

KC-5-087
ppm 8
7
6
5
4
3
2
1
0


KC-5-087
ppm 200
180
160
140
120
100
80
60
40
20
0

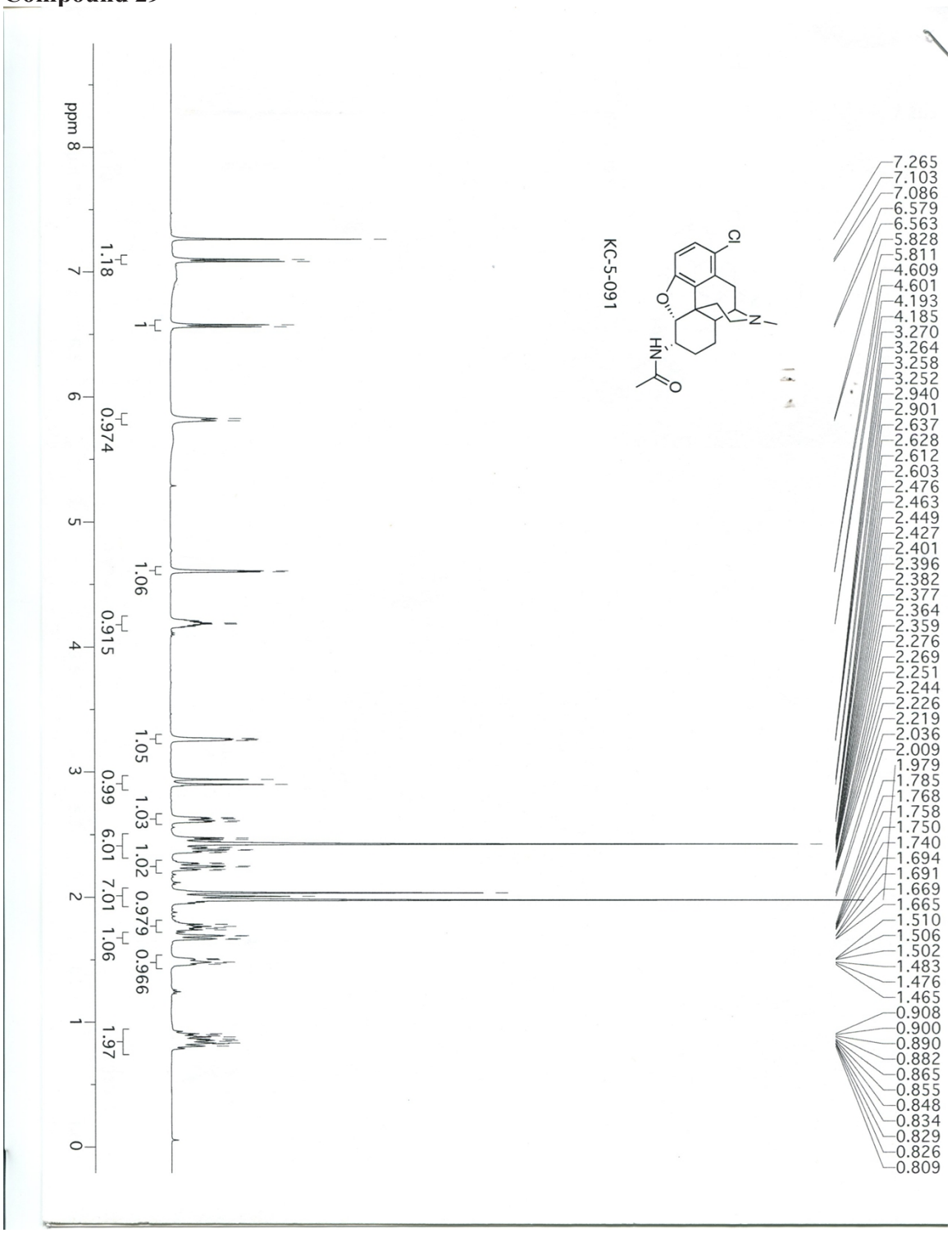
Compound 28


7.267
7.265
7.066
7.065
7.049
7.048
6.561
6.544
4.550
4.542
3.141
3.136
3.129
3.124
3.033
3.011
2.941
2.903
2.510
2.501
2.486
2.477
2.363
2.351
2.324
2.312
2.247
2.242
2.225
2.218
2.210
2.200
2.193
2.175
1.921
1.911
1.896
1.886
1.871
1.861
1.705
1.701
1.680
1.676
1.662
1.606
1.594
1.578
1.561
1.418
1.411
1.403
1.393
1.384
1.375
1.367
1.360
1.079
1.062
1.052
1.044
1.040
1.035
1.023
1.018
1.013
0.996
0.892
0.885
0.869
0.865
0.856
0.847
0.845
0.837
0.826
Cl
N
O
NH2
KC-5-089
1.07
1.07
1.07
4.99
1.15
1.35
1.35
1.1
1.16
1.16
1.14
2.42
1.23
1.69
1.32
ppm 8
7
6
5
4
3
2
1
0

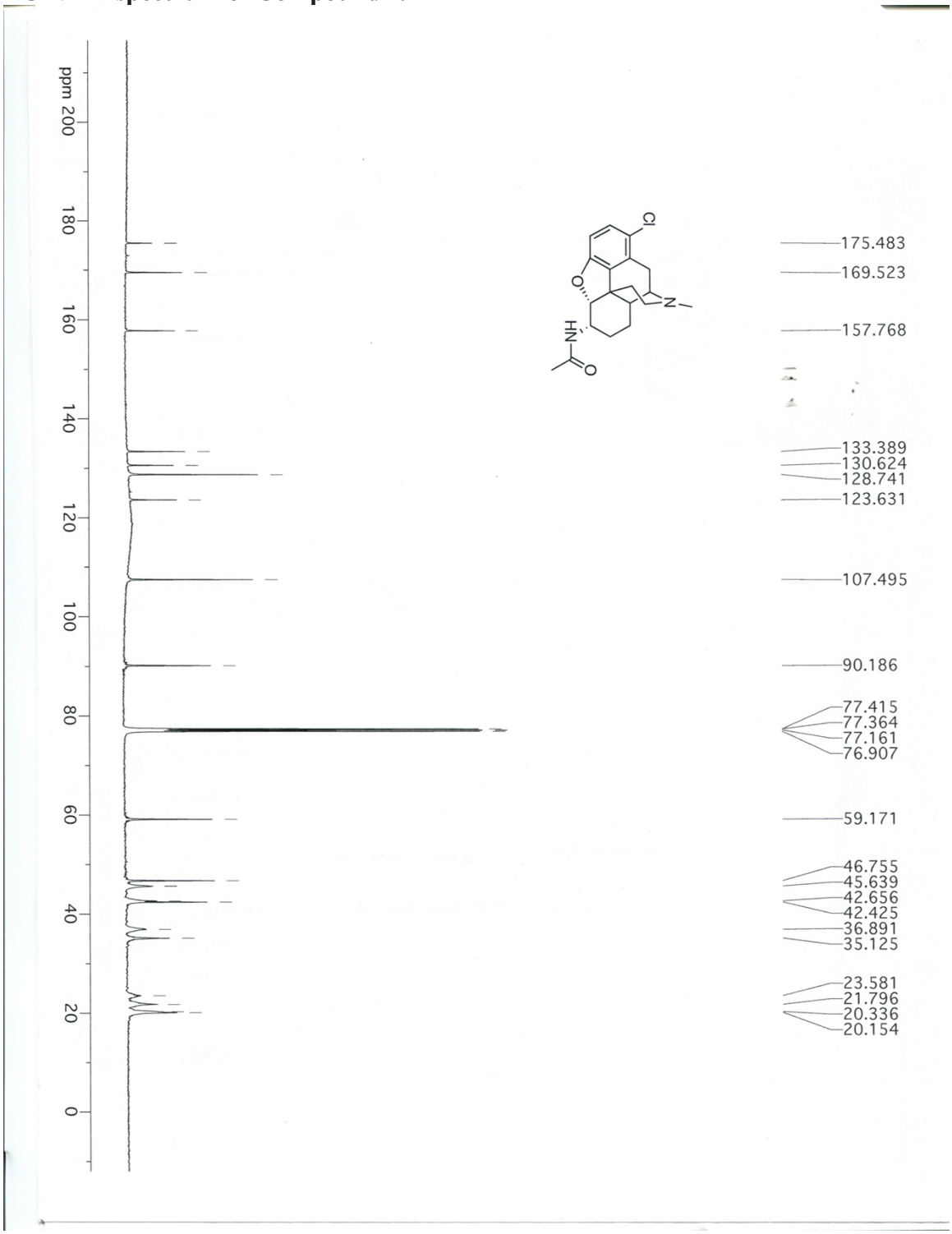
^{13}C NMR spectrum of Compound 28



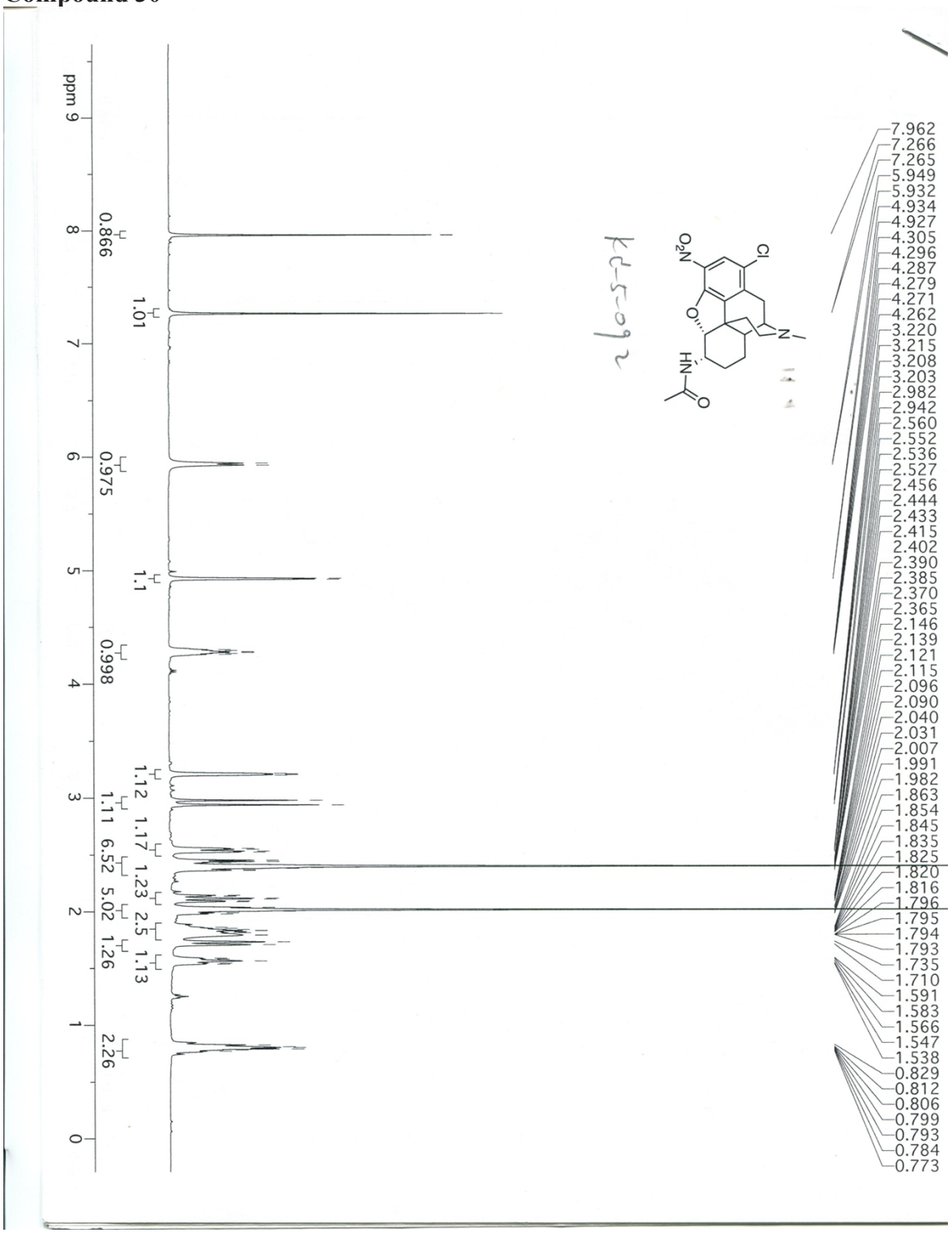
Compound 29


KC-5-091
Cl
O
N
HN
O
ppm 8
7
6
5
4
3
2
1
0
1.18
1
0.974
1.06
0.915
1.05
0.99
1.03
6.01
1.02
7.01
0.979
1.06
0.966
1.97
7.265
7.103
7.086
6.579
6.563
5.828
5.811
4.609
4.601
4.193
4.185
3.270
3.264
3.258
3.252
2.940
2.901
2.637
2.628
2.612
2.603
2.476
2.463
2.449
2.427
2.401
2.396
2.382
2.377
2.364
2.359
2.276
2.269
2.251
2.244
2.226
2.219
2.036
2.009
1.979
1.785
1.768
1.758
1.750
1.740
1.694
1.691
1.669
1.665
1.510
1.506
1.502
1.483
1.476
1.465
0.908
0.900
0.890
0.882
0.865
0.855
0.848
0.834
0.829
0.826
0.809

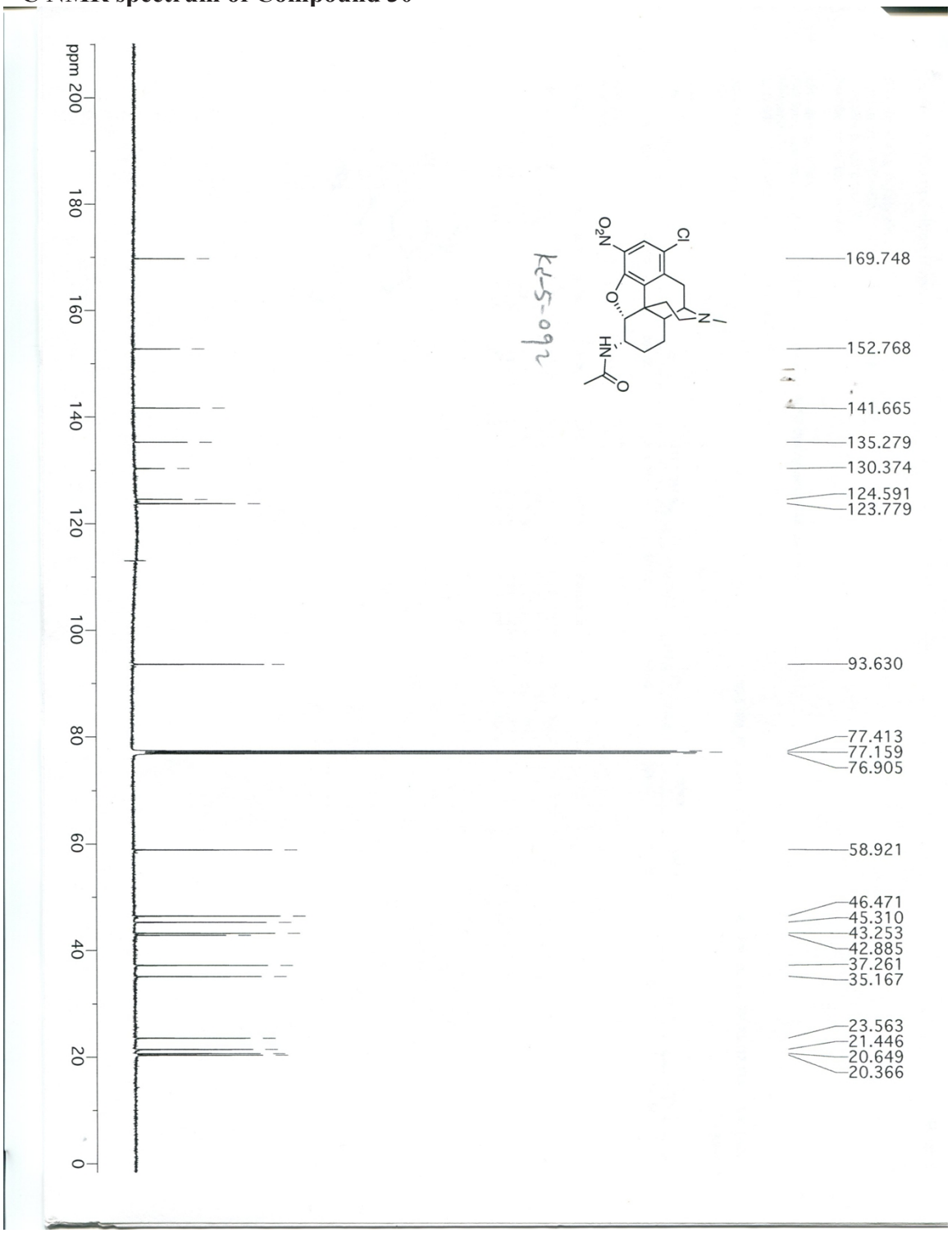
^{13}C NMR spectrum of Compound 29



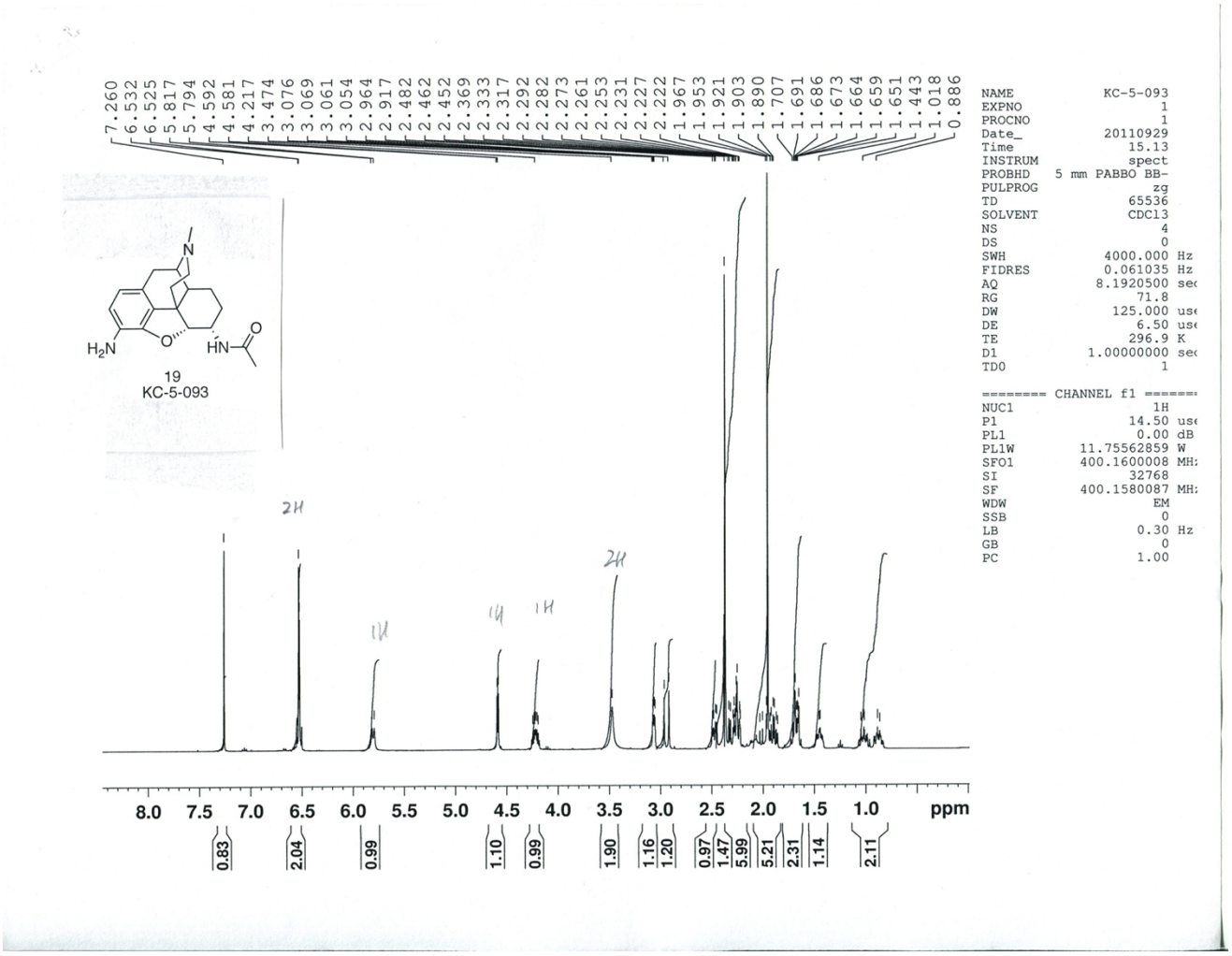
Compound 30


ppm 9
8
7
6
5
4
3
2
1
0
0.866
1.01
0.975
1.1
0.998
1.12
1.11
1.17
6.52
1.23
5.02
2.5
1.26
1.13
2.26
7.962
7.266
7.265
5.949
5.932
4.934
4.927
4.305
4.296
4.287
4.279
4.271
4.262
3.220
3.215
3.208
3.203
2.982
2.942
2.560
2.552
2.536
2.527
2.456
2.444
2.433
2.415
2.402
2.390
2.385
2.370
2.365
2.146
2.139
2.121
2.115
2.096
2.090
2.040
2.031
2.007
1.991
1.982
1.863
1.854
1.845
1.835
1.825
1.820
1.816
1.796
1.795
1.794
1.793
1.735
1.710
1.591
1.583
1.566
1.547
1.538
0.829
0.812
0.806
0.799
0.793
0.784
0.773

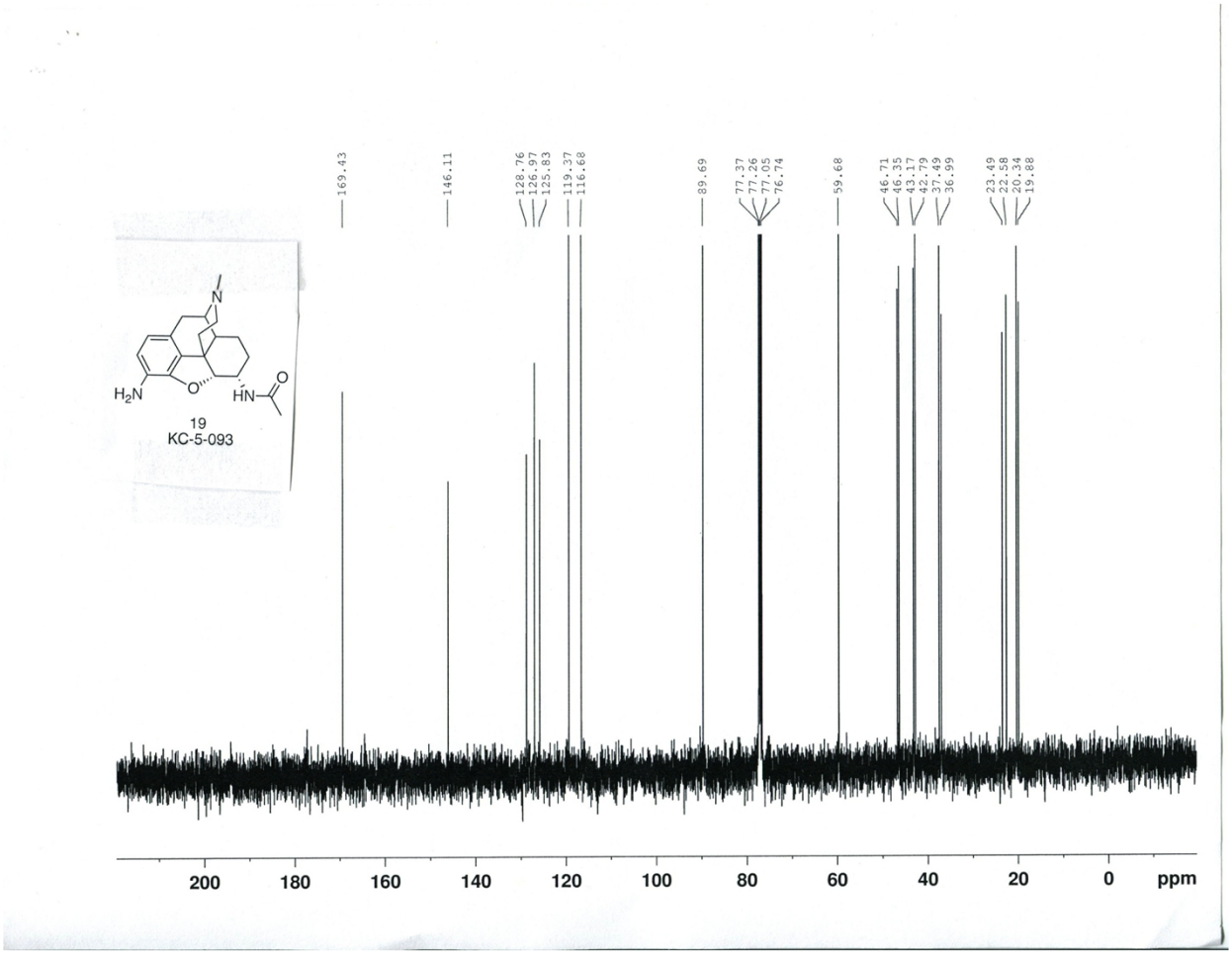
^{13}C NMR spectrum of Compound 30



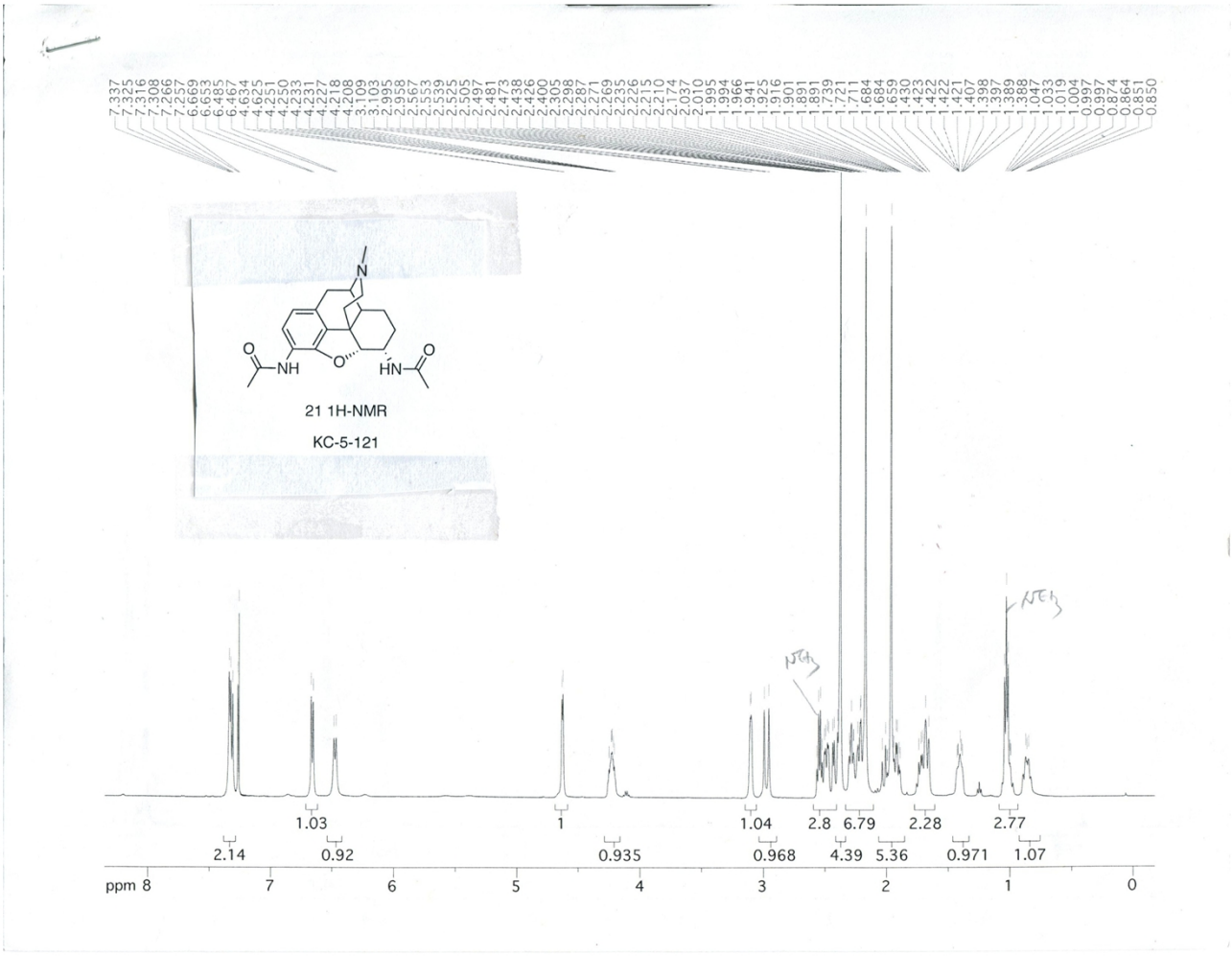
Compound 31 (400 MHz)



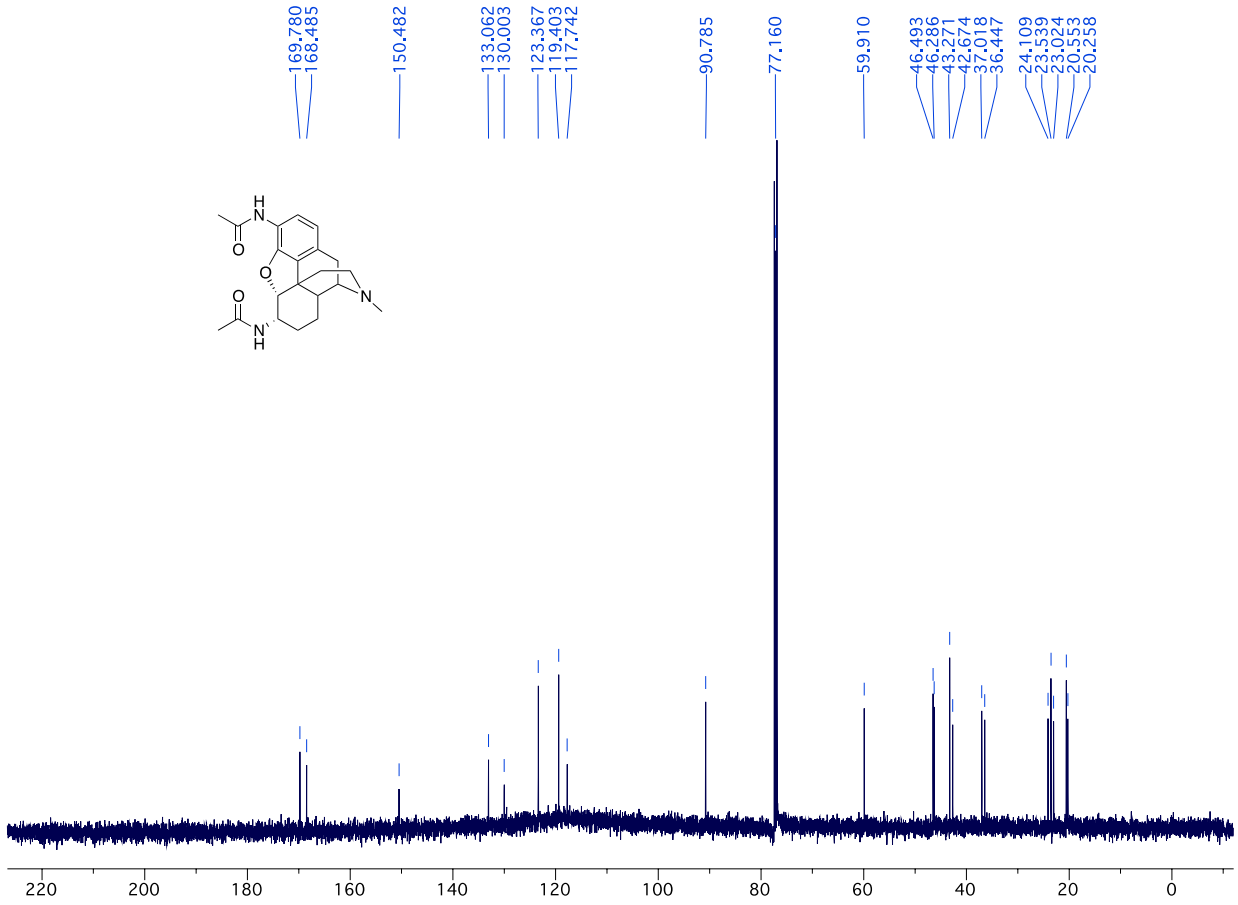
^{13}C NMR spectrum of Compound 31 (100 MHz)



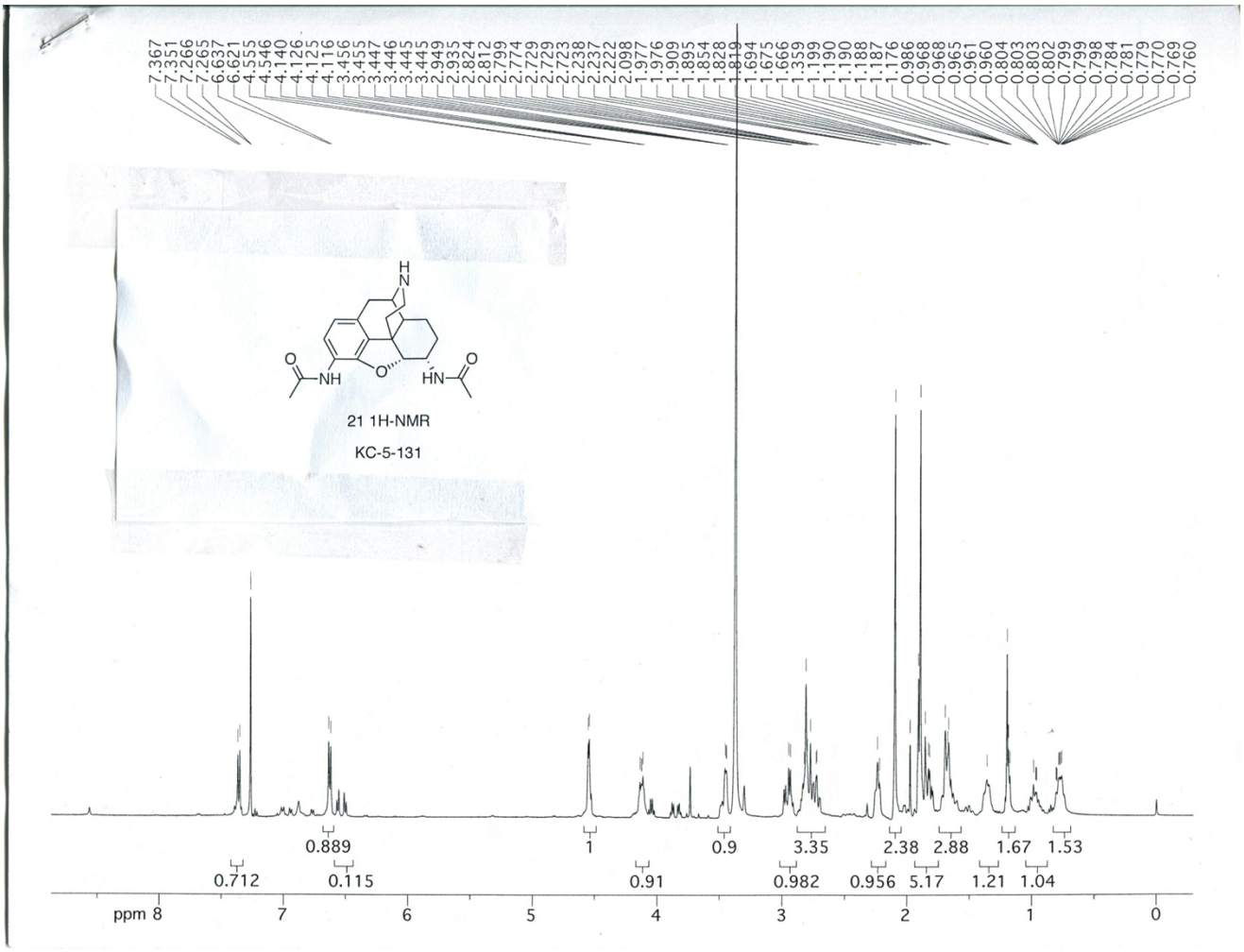
Compound 32


21 1H-NMR
KC-5-121
1.03
1
1.04
2.8
6.79
2.28
2.77
2.14
0.92
0.935
0.968
4.39
5.36
0.971
1.07
ppm 8
7
6
5
4
3
2
1
0

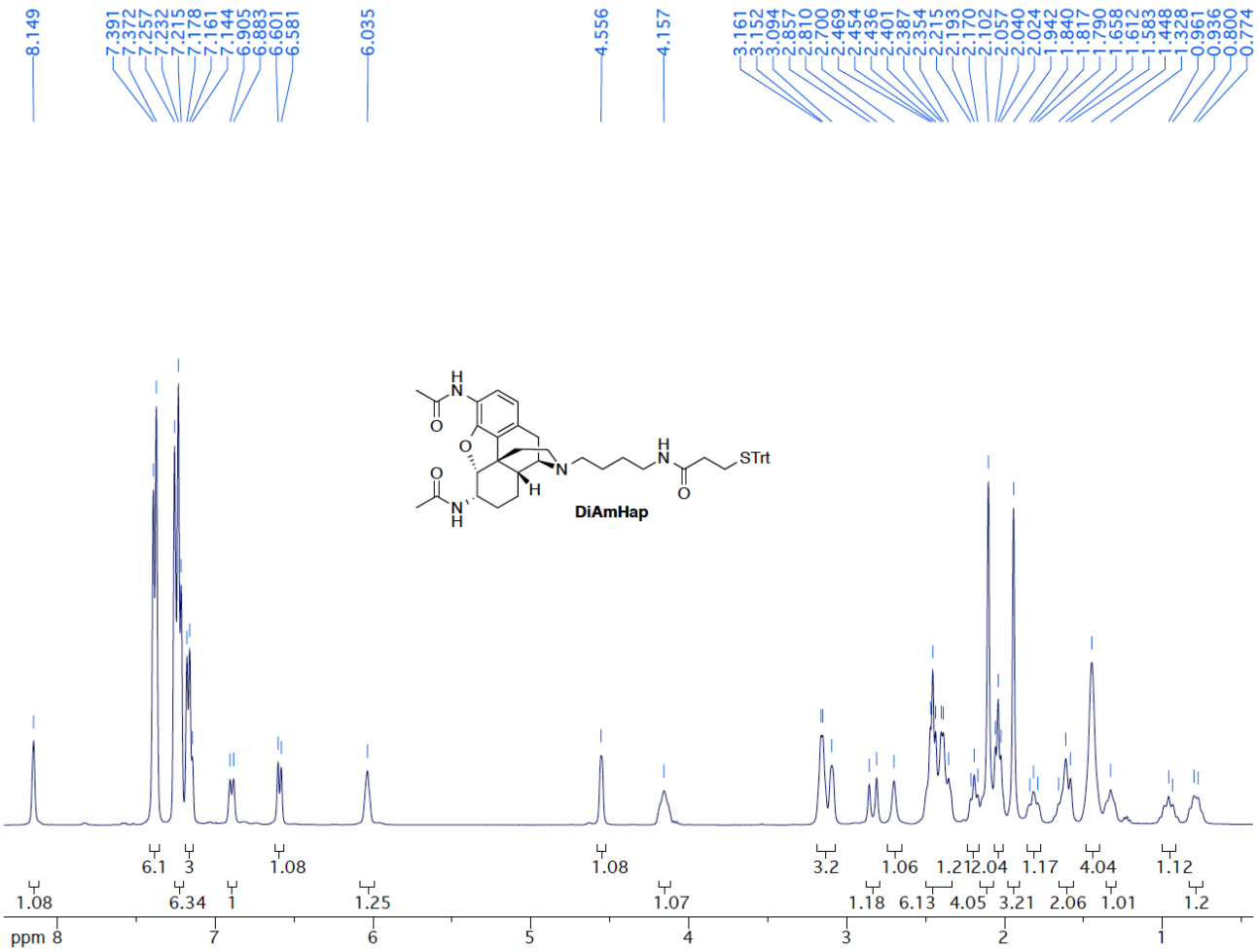
^{13}C NMR spectrum of Compound 32



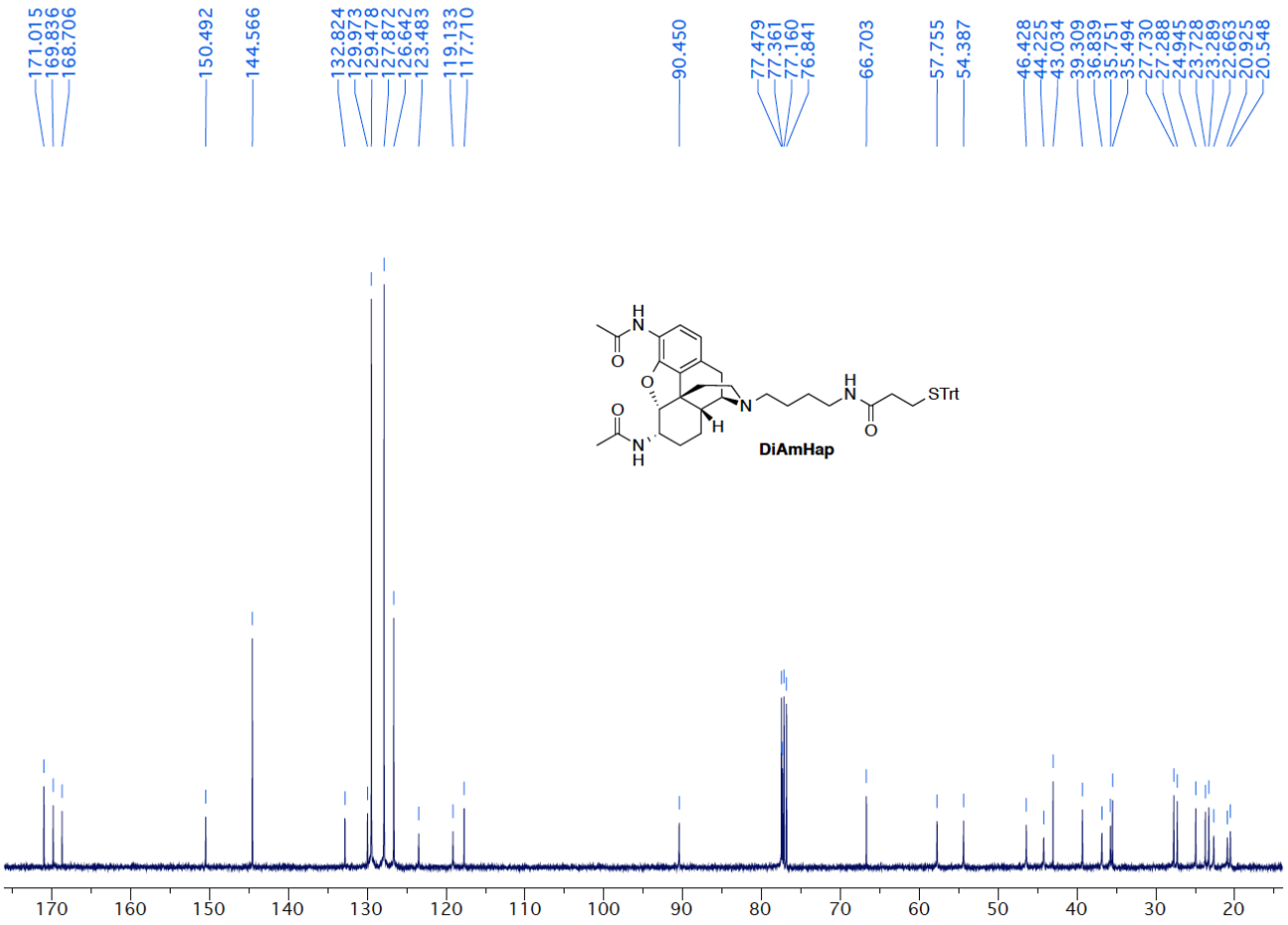
Compound 33


7.367
7.351
7.266
7.265
6.637
6.621
4.555
4.546
4.140
4.126
4.125
4.116
3.456
3.455
3.447
3.446
3.445
3.445
2.949
2.935
2.824
2.812
2.799
2.774
2.729
2.729
2.723
2.238
2.237
2.222
2.098
1.977
1.976
1.909
1.895
1.854
1.828
1.819
1.694
1.675
1.666
1.359
1.199
1.190
1.190
1.188
1.187
1.176
0.986
0.968
0.968
0.965
0.961
0.960
0.804
0.803
0.802
0.799
0.799
0.798
0.784
0.781
0.779
0.770
0.769
0.760
21 1H-NMR
KC-5-131
0.889
1
0.9
3.35
2.38
2.88
1.67
1.53
0.712
0.115
0.91
0.982
0.956
5.17
1.21
1.04
ppm 8
7
6
5
4
3
2
1
0

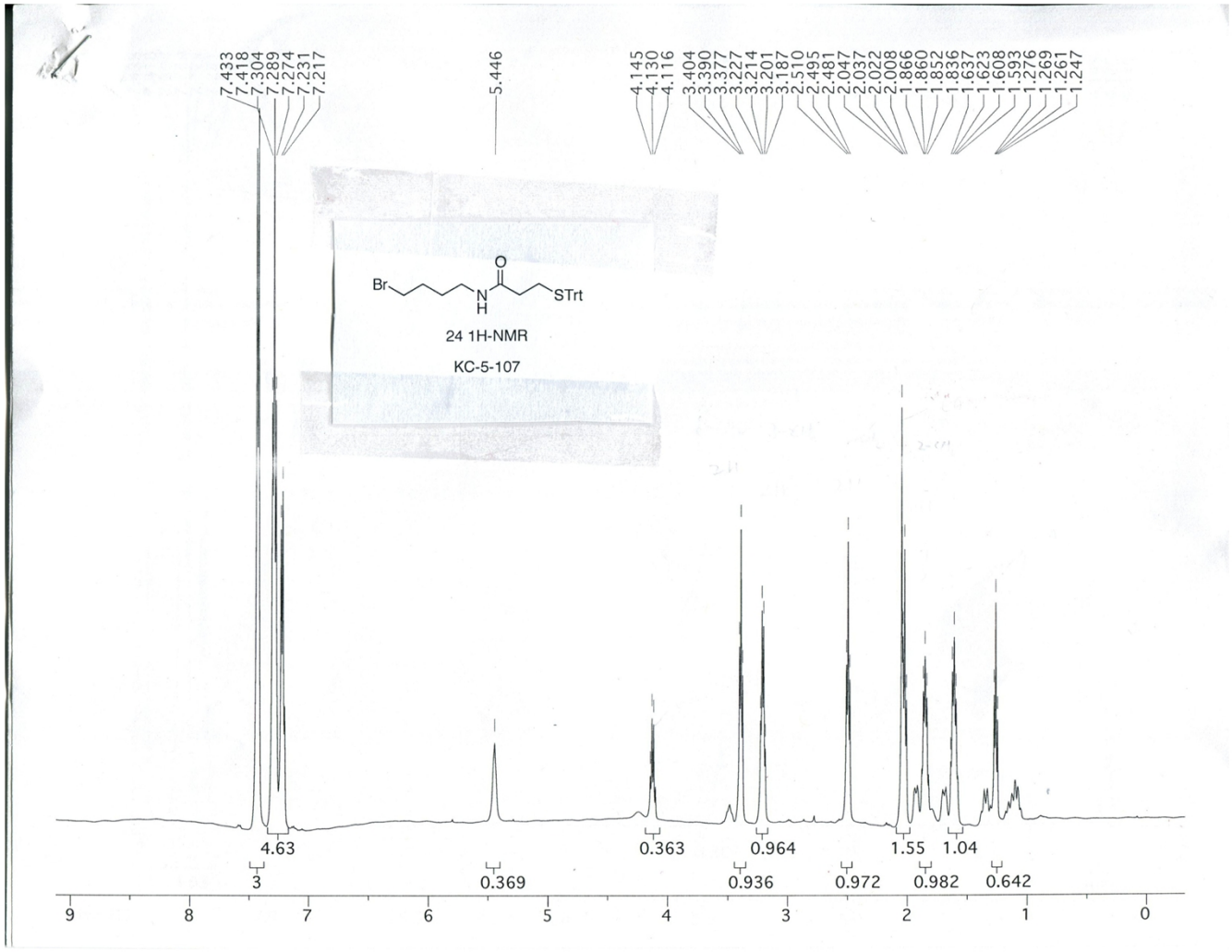
Compound 3 (DiAmHap, 400 MHz)



^{13}C NMR spectrum of Compound 3 (DiAmHap, 100 MHz)



Compound **36**


7.433
7.418
7.304
7.289
7.274
7.231
7.217
5.446
4.145
4.130
4.116
3.404
3.390
3.377
3.227
3.214
3.201
3.187
2.510
2.495
2.481
2.047
2.037
2.022
2.008
1.866
1.860
1.852
1.836
1.637
1.623
1.608
1.593
1.276
1.269
1.261
1.247
Br
O
N
H
STrt
24 1H-NMR
KC-5-107
4.63
0.363
0.964
1.55
1.04
3
0.369
0.936
0.972
0.982
0.642
9
8
7
6
5
4
3
2
1
0

^{13}C NMR spectrum of Compound 36