Synthesis of Structurally Simplified Analogues of Pancratistatin: Truncation of the Cyclitol Ring

Supplemental Information

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General Experimental Details: Unless otherwise noted all commercially obtained reagents were used without purification. THF was distilled from sodium-benzophenone ketyl prior to use. Dichloromethane and methanol were distilled from calcium hydride. Reactions were carried out under a nitrogen atmosphere in oven-dried glassware using standard syringe, cannula and septa techniques. Reactions were monitored by TLC (Silica Gel 60 F254, 250 μm) and visualized with UV light and ceric ammonium molybdate solution. Flash chromatography was performed on silica gel (32-63 μm, 60 Å pore size). ^{1}H and ^{13}C NMR spectra were recorded on 300 MHz spectrometer.

X-ray structure determination.

Details of the crystal data, data collection and structure refinement parameters for compound **23** are presented in Table 1. Single crystal X-ray diffraction experiment was carried out with a diffractometer, with CCD area detector at 250K. Semiempirical method SADABS1 was applied for absorption correction. The structure was solved by direct methods and refined by the full-matrix least-squares technique against *F2* with the anisotropic temperature parameters for all non-hydrogen atoms. Data reduction and further calculations were performed using SAINT+2 and SHELXTL NT3 program packages.

References

1. Sheldrick G.M. SADABS v.2.03, Bruker/Siemens Area Detector Absorption Correction Program, (2003) Bruker AXS, Madison, Wisconsin, USA.

2. SAINTP+ for NT. Data Reduction and Correction Program v. 6.2, (2001) Bruker AXS, Madison, Wisconsin, USA.

3. Sheldrick G.M. SHELXTL NT v. 6.12, Structure Determination Software Suite, (2001) Bruker AXS, Madison, Wisconsin, USA.

Table 1. Crystal data and structure refinement for **23**.

Identification code	paul17a
Empirical formula	C20 H23 N O9
Formula weight	421.39
Temperature	273(2) K
Wavelength	1.54178 Å

Crystal system	Monoclinic	
Space group	P2(1)	
Unit cell dimensions	a = 10.0278(2) Å	α= 90°.
	b = 7.7271(2) Å	β= 92.6880(10)°.
	c = 12.6670(2) Å	γ = 90°.
Volume	980.43(4) $Å^3$	
Z	2	
Density (calculated)	1.427 Mg/m^3	
Absorption coefficient	0.963 mm^{-1}	
F(000)	444	
Crystal size	0.40 x 0.19 x 0.14 mm^3	
Theta range for data collection	3.49 to 68.92°.	
Index ranges	-11<=h<=12, -9<=k<=8, -14<=l<=15	
Reflections collected	8357	
Independent reflections	3188 [R(int) = 0.0225]	
Completeness to theta = 68.92°	99.0 %	
Absorption correction	Numerical	
Max. and min. transmission	0.8769 and 0.6993	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3188 / 1 / 271	
Goodness-of-fit on F^2	1.036	
Final R indices [I>2sigma(I)]	R1 = 0.0404, wR2 = 0.1021	
R indices (all data)	R1 = 0.0475, wR2 = 0.1077	
Absolute structure parameter	0.3(2)	
Largest diff. peak and hole	0.172 and -0.177 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **23**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	-756(4)	-5172(4)	-8921(2)	118(1)
N(1)	-3857(2)	-4995(3)	-7817(2)	57(1)
C(1)	-2921(2)	-2224(3)	-7341(2)	47(1)
O(2)	140(3)	498(3)	-5427(2)	88(1)
C(2)	-3911(2)	-3178(4)	-8071(2)	51(1)

O(3)	-603(2)	-43(2)	-7083(1)	53(1)
C(3)	-3677(2)	-5626(4)	-6835(2)	57(1)
O(4)	-1329(2)	-2464(2)	-8678(1)	52(1)
C(4)	-3472(2)	-4336(3)	-5968(2)	53(1)
O(5)	-6175(2)	-3309(3)	-8744(2)	65(1)
C(5)	-3592(3)	-4870(4)	-4915(2)	62(1)
O(6)	-5616(3)	-1559(4)	-10024(2)	96(1)
C(6)	-3438(3)	-3655(5)	-4158(2)	64(1)
O(7)	-3691(2)	-7172(3)	-6657(2)	75(1)
C(7)	-3205(4)	-2164(6)	-2649(2)	87(1)
O(8)	-3464(3)	-3828(4)	-3077(2)	88(1)
C(8)	-3227(3)	-1960(4)	-4405(2)	59(1)
O(9)	-3171(2)	-988(4)	-3498(2)	78(1)
C(9)	-3092(2)	-1413(4)	-5415(2)	54(1)
C(10)	-3191(2)	-2661(3)	-6209(2)	50(1)
C(11)	-1509(2)	-2757(3)	-7565(2)	48(1)
C(12)	-452(2)	-1862(3)	-6899(2)	51(1)
C(13)	924(3)	-2395(4)	-7155(3)	73(1)
C(14)	-255(3)	989(3)	-6267(2)	55(1)
C(15)	-410(3)	2825(4)	-6570(3)	69(1)
C(16)	-1020(3)	-3813(4)	-9272(2)	65(1)
C(17)	-1090(4)	-3417(6)	-10417(2)	84(1)
C(18)	-5273(3)	-2461(4)	-7999(2)	62(1)
C(19)	-6250(3)	-2748(4)	-9733(2)	66(1)
C(20)	-7166(4)	-3770(5)	-10418(3)	93(1)

Table 3. Bond lengths [Å] and angles [°] for **23**.

O(1)-C(16)	1.166(4)
N(1)-C(3)	1.341(4)
N(1)-C(2)	1.440(4)
N(1)-HN1	0.8600
C(1)-C(2)	1.515(3)
C(1)-C(10)	1.510(3)
C(1)-C(11)	1.514(3)
C(1)-H(1)	0.9800
O(2)-C(14)	1.181(3)
C(2)-C(18)	1.481(4)
C(2)-H(2)	0.9800
O(3)-C(14)	1.339(3)
O(3)-C(12)	1.432(3)
C(3)-O(7)	1.216(3)
C(3)-C(4)	1.490(4)
O(4)-C(16)	1.331(3)
O(4)-C(11)	1.448(3)
C(4)-C(10)	1.363(4)
C(4)-C(5)	1.407(4)
O(5)-C(19)	1.324(3)
O(5)-C(18)	1.436(3)
C(5)-C(6)	1.346(5)
C(5)-H(5)	0.9300
O(6)-C(19)	1.185(4)
C(6)-O(8)	1.377(3)
C(6)-C(8)	1.366(5)
C(7)-O(8)	1.415(5)
C(7)-O(9)	1.410(4)
C(7)-H(7A)	0.9700
C(7)-H(7B)	0.9700
C(8)-O(9)	1.372(3)
C(8)-C(9)	1.360(4)
C(9)-C(10)	1.393(4)
C(9)-H(9)	0.9300

C(11)-C(12)	1.493(3)
C(11)-H(11)	0.9800
C(12)-C(13)	1.490(4)
C(12)-H(12)	0.9800
C(13)-H(13A)	0.9600
C(13)-H(13B)	0.9600
C(13)-H(13C)	0.9600
C(14)-C(15)	1.476(4)
C(15)-H(15A)	0.9600
C(15)-H(15B)	0.9600
C(15)-H(15C)	0.9600
C(16)-C(17)	1.481(4)
C(17)-H(17A)	0.9600
C(17)-H(17B)	0.9600
C(17)-H(17C)	0.9600
C(18)-H(18A)	0.9700
C(18)-H(18B)	0.9700
C(19)-C(20)	1.466(4)
C(20)-H(20A)	0.9600
C(20)-H(20B)	0.9600
C(20)-H(20C)	0.9600
C(3)-N(1)-C(2)	124.3(2)
C(3)-N(1)-HN1	117.9
C(2)-N(1)-HN1	117.9
C(2)-C(1)-C(10)	109.2(2)
C(2)-C(1)-C(11)	110.18(19)
C(10)-C(1)-C(11)	109.15(19)
C(2)-C(1)-H(1)	109.4
C(10)-C(1)-H(1)	109.4
C(11)-C(1)-H(1)	109.4
N(1)-C(2)-C(18)	112.1(2)
N(1)-C(2)-C(1)	108.8(2)
C(18)-C(2)-C(1)	111.1(2)
N(1)-C(2)-H(2)	108.3
C(18)-C(2)-H(2)	108.3
C(1)-C(2)-H(2)	108.3

C(14)-O(3)-C(12)	115.89(19)
O(7)-C(3)-N(1)	121.8(3)
O(7)-C(3)-C(4)	121.6(3)
N(1)-C(3)-C(4)	116.6(2)
C(16)-O(4)-C(11)	118.1(2)
C(10)-C(4)-C(5)	121.2(3)
C(10)-C(4)-C(3)	119.6(2)
C(5)-C(4)-C(3)	119.2(2)
C(19)-O(5)-C(18)	118.6(2)
C(6)-C(5)-C(4)	117.3(3)
C(6)-C(5)-H(5)	121.3
C(4)-C(5)-H(5)	121.3
C(5)-C(6)-O(8)	129.3(3)
C(5)-C(6)-C(8)	121.3(3)
O(8)-C(6)-C(8)	109.4(3)
O(8)-C(7)-O(9)	107.7(2)
O(8)-C(7)-H(7A)	110.2
O(9)-C(7)-H(7A)	110.2
O(8)-C(7)-H(7B)	110.2
O(9)-C(7)-H(7B)	110.2
H(7A)-C(7)-H(7B)	108.5
C(6)-O(8)-C(7)	106.3(3)
O(9)-C(8)-C(9)	128.0(3)
O(9)-C(8)-C(6)	109.5(3)
C(9)-C(8)-C(6)	122.5(3)
C(8)-O(9)-C(7)	106.5(3)
C(8)-C(9)-C(10)	117.2(3)
C(8)-C(9)-H(9)	121.4
C(10)-C(9)-H(9)	121.4
C(4)-C(10)-C(9)	120.4(2)
C(4)-C(10)-C(1)	118.2(2)
C(9)-C(10)-C(1)	121.4(2)
O(4)-C(11)-C(12)	111.1(2)
O(4)-C(11)-C(1)	107.47(18)
C(12)-C(11)-C(1)	114.4(2)
O(4)-C(11)-H(11)	107.9

C(12)-C(11)-H(11)	107.9
C(1)-C(11)-H(11)	107.9
O(3)-C(12)-C(11)	107.22(19)
O(3)-C(12)-C(13)	109.2(2)
C(11)-C(12)-C(13)	112.9(2)
O(3)-C(12)-H(12)	109.2
C(11)-C(12)-H(12)	109.2
C(13)-C(12)-H(12)	109.2
C(12)-C(13)-H(13A)	109.5
C(12)-C(13)-H(13B)	109.5
H(13A)-C(13)-H(13B)	109.5
C(12)-C(13)-H(13C)	109.5
H(13A)-C(13)-H(13C)	109.5
H(13B)-C(13)-H(13C)	109.5
O(2)-C(14)-O(3)	124.7(3)
O(2)-C(14)-C(15)	124.7(3)
O(3)-C(14)-C(15)	110.6(2)
C(14)-C(15)-H(15A)	109.5
C(14)-C(15)-H(15B)	109.5
H(15A)-C(15)-H(15B)	109.5
C(14)-C(15)-H(15C)	109.5
H(15A)-C(15)-H(15C)	109.5
H(15B)-C(15)-H(15C)	109.5
O(1)-C(16)-O(4)	123.0(3)
O(1)-C(16)-C(17)	124.1(3)
O(4)-C(16)-C(17)	113.0(3)
C(16)-C(17)-H(17A)	109.5
C(16)-C(17)-H(17B)	109.5
H(17A)-C(17)-H(17B)	109.5
C(16)-C(17)-H(17C)	109.5
H(17A)-C(17)-H(17C)	109.5
H(17B)-C(17)-H(17C)	109.5
O(5)-C(18)-C(2)	110.1(2)
O(5)-C(18)-H(18A)	109.6
C(2)-C(18)-H(18A)	109.6
O(5)-C(18)-H(18B)	109.6

C(2)-C(18)-H(18B)	109.6
H(18A)-C(18)-H(18B)	108.2
O(6)-C(19)-O(5)	122.8(3)
O(6)-C(19)-C(20)	124.3(3)
O(5)-C(19)-C(20)	112.9(3)
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

Symmetry transformations used to generate equivalent atoms:

DFT Optimizations

Geometry optimizations were carried out using B3LYP method of density functional theory. B3LYP denotes a three-parameter hybrid density functional that combines Becke's exchange functional with the correlation functional by Lee, Yang, and Parr.[1-3] The basis set 6-31g* was employed for the optimization. The crystallographic molecular structure[4] was used for the initial geometry of pancratistatin. All calculations were carried out with Gaussian03, Revision B.01.[5]

References

[1] A. D. Becke, *Phys. Rev.* **1988**, *A38*, 3098.

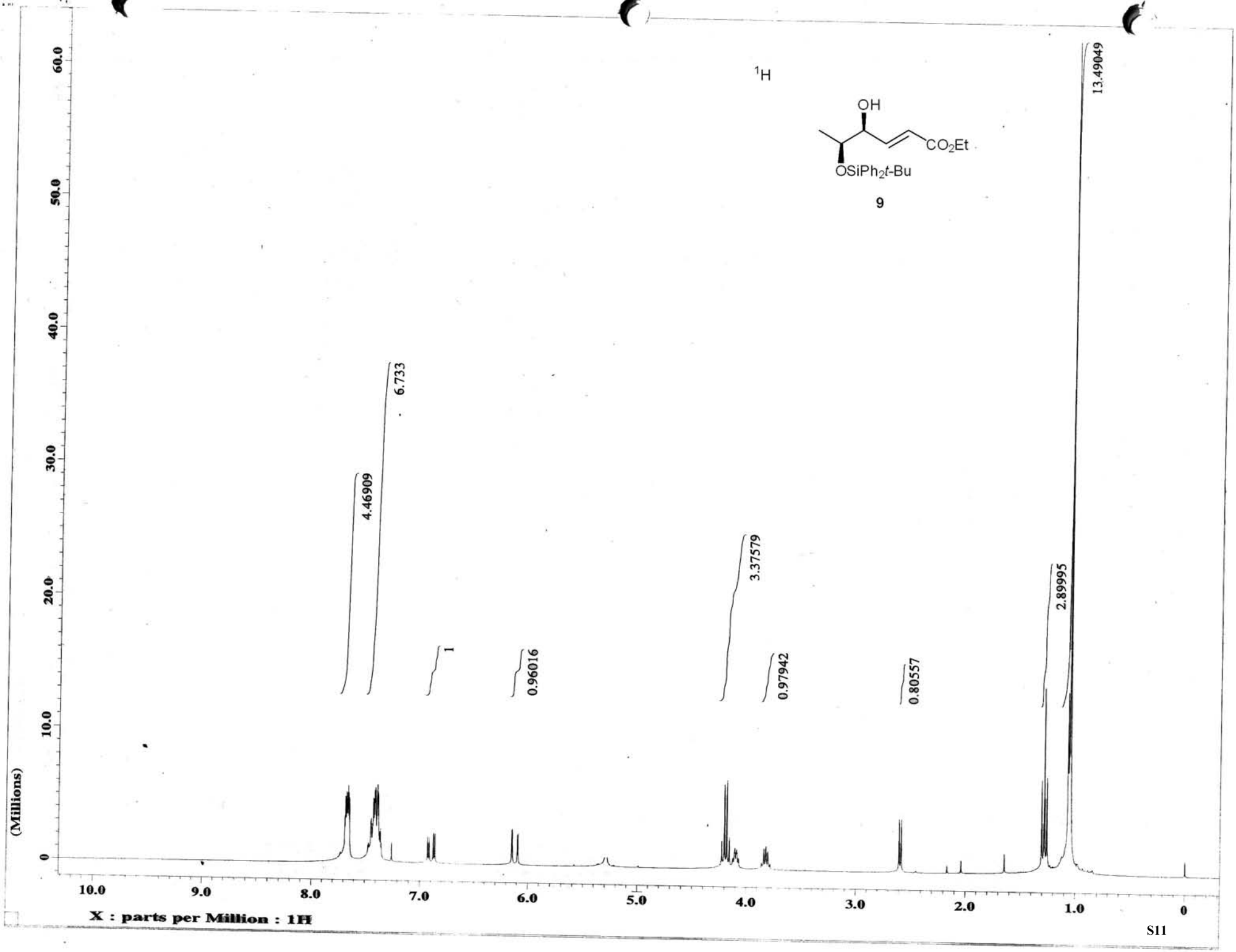
[2] A. D. Becke, *J. Chem. Phys.* **1993**, *98*, 5648.

[3] C. T. Lee, W. T. Yang, R. G. Parr, *Phys. Re*v. **1988**, *B37*, 785.

[4] G. R. Pettit, Y. Meng, D. L. Herald, J. C. Knight, and J. F. Day, *J. Nat. Prod.* **2005**, 68, 1256.

[5] M. J. Frisch, G. W. Trucks, H. B. Schlegel, G. E. Scuseria, M. A. Robb, J. R. Cheeseman, J. A. Montgomery, Jr., T. Vreven, K. N. Kudin, J. C. Burant, J. M. Millam, S. S. Iyengar, J. Tomasi, V. Barone, B. Mennucci, M. Cossi, G. Scalmani, N. Rega, G. A. Petersson, H. Nakatsuji, M. Hada, M. Ehara, K. Toyota, R. Fukuda, J. Hasegawa, M. Ishida, T. Nakajima, Y. Honda, O. Kitao, H. Nakai, M. Klene, X. Li, J. E. Knox, H. P. Hratchian, J. B. Cross, C. Adamo, J. Jaramillo, R. Gomperts, R. E. Stratmann, O. Yazyev, A. J. Austin, R. Cammi, C. Pomelli, J.

W. Ochterski, P. Y. Ayala, K. Morokuma, G. A. Voth, P. Salvador, J. J. Dannenberg, V. G. Zakrzewski, S. Dapprich, A. D. Daniels, M. C. Strain, O. Farkas, D. K. Malick, A. D. Rabuck, K. Raghavachari, J. B. Foresman, J. V. Ortiz, Q. Cui, A. G. Baboul, S. Clifford, J. Cioslowski, B. B. Stefanov, G. Liu, A. Liashenko, P. Piskorz, I. Komaromi, R. L. Martin, D. J. Fox, T. Keith, M. A. Al-Laham, C. Y. Peng, A. Nanayakkara, M. Challacombe, P. M. W. Gill, B. Johnson, W. Chen, M. W. Wong, C. Gonzalez, and J. A. Pople, Gaussian, Inc., Pittsburgh PA, 2003.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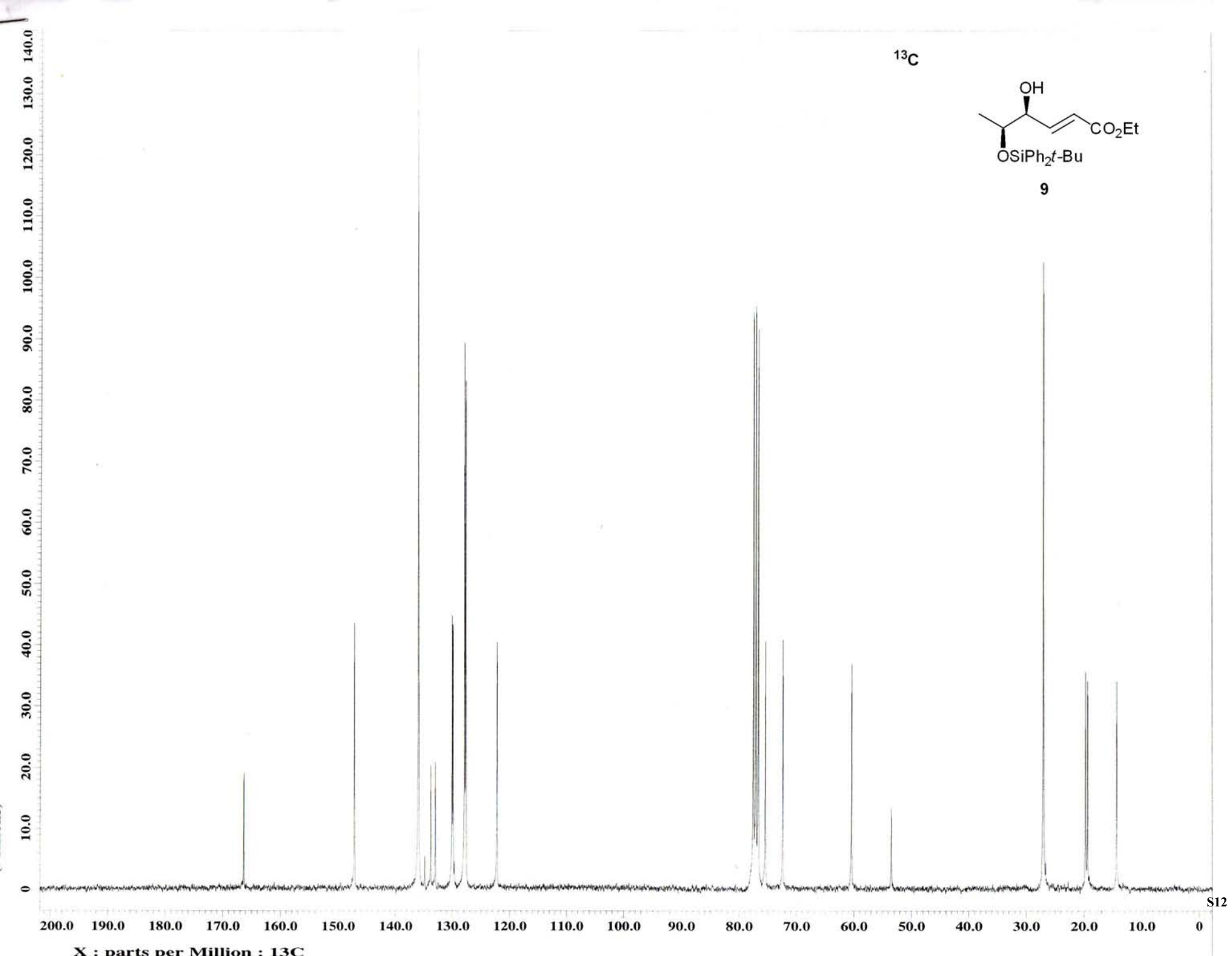
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70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
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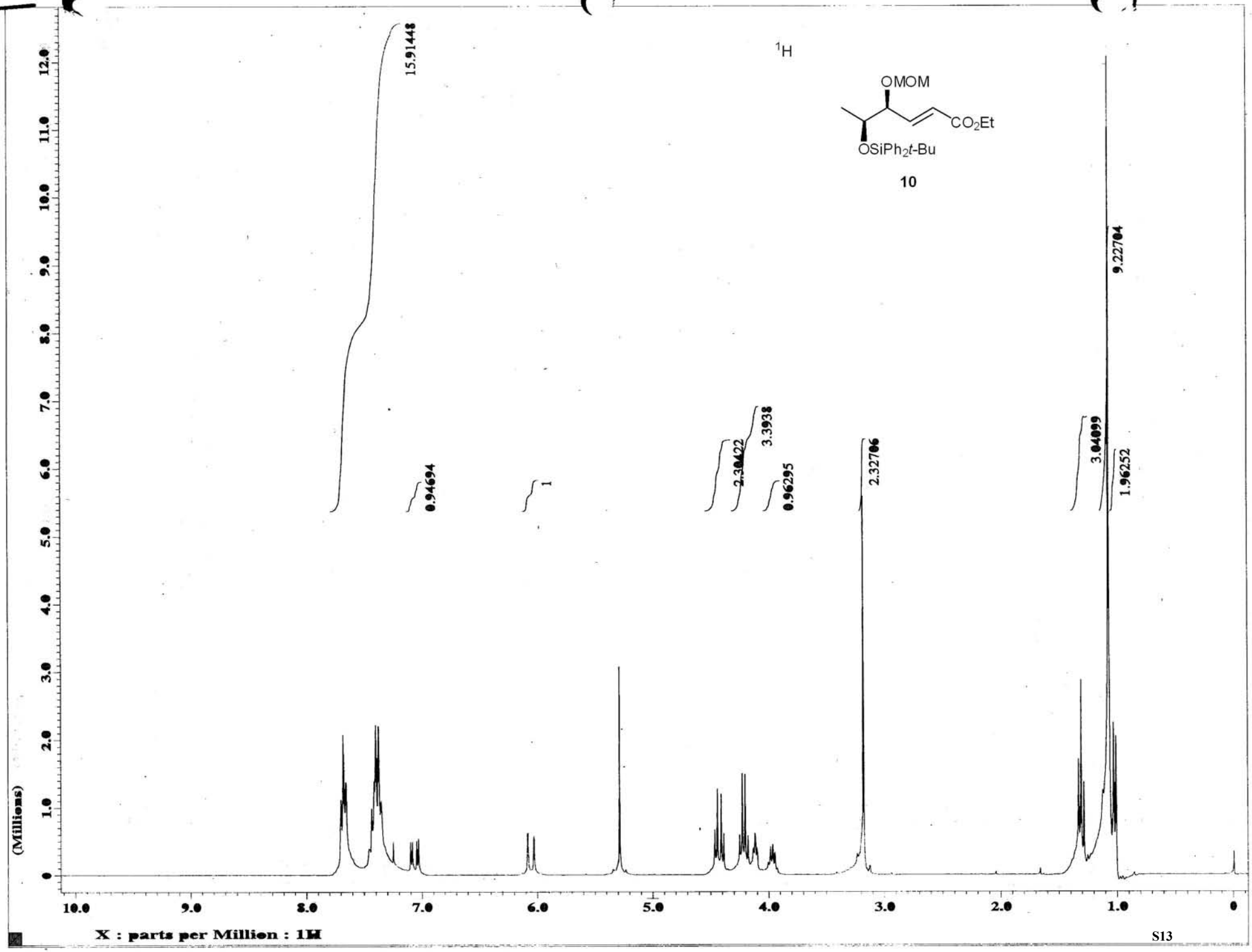
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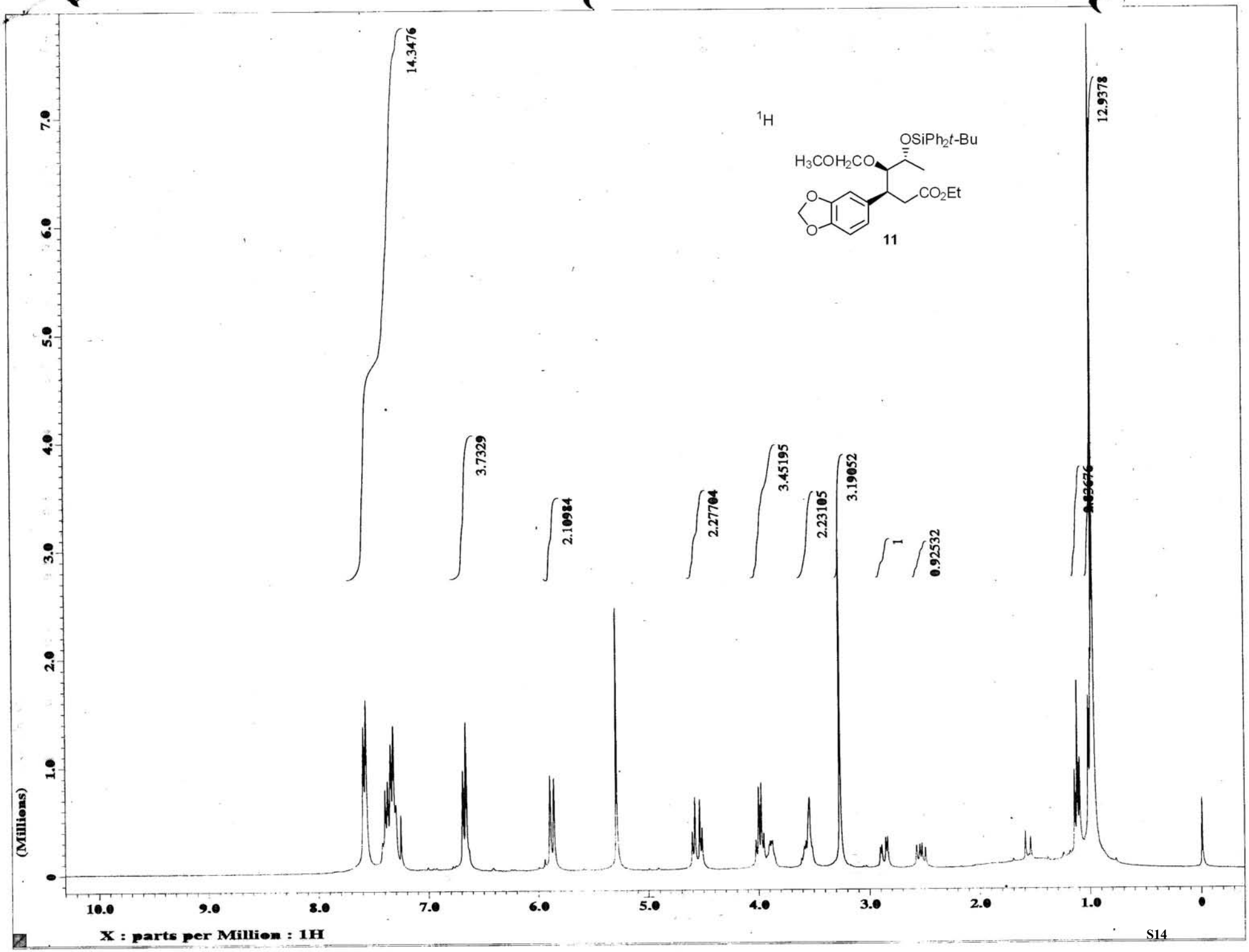
(Millions)
X : parts per Million : 1H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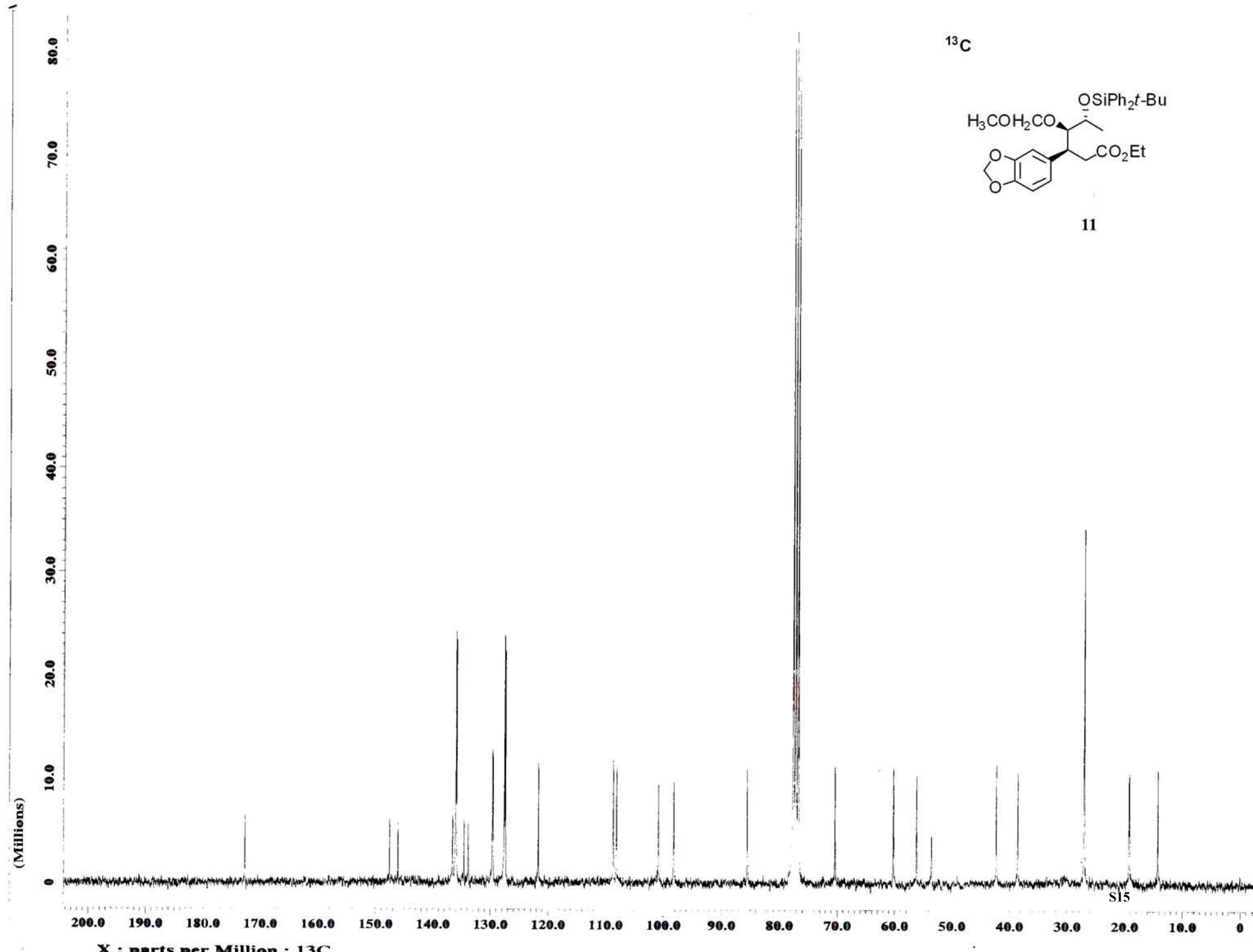
CO$_2$Et
11
(Millions)
X : parts per Million : 1H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H$_3$COH$_2$CO
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O
O
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(Millions)
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50.0
40.0
30.0
20.0
10.0
0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
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50.0
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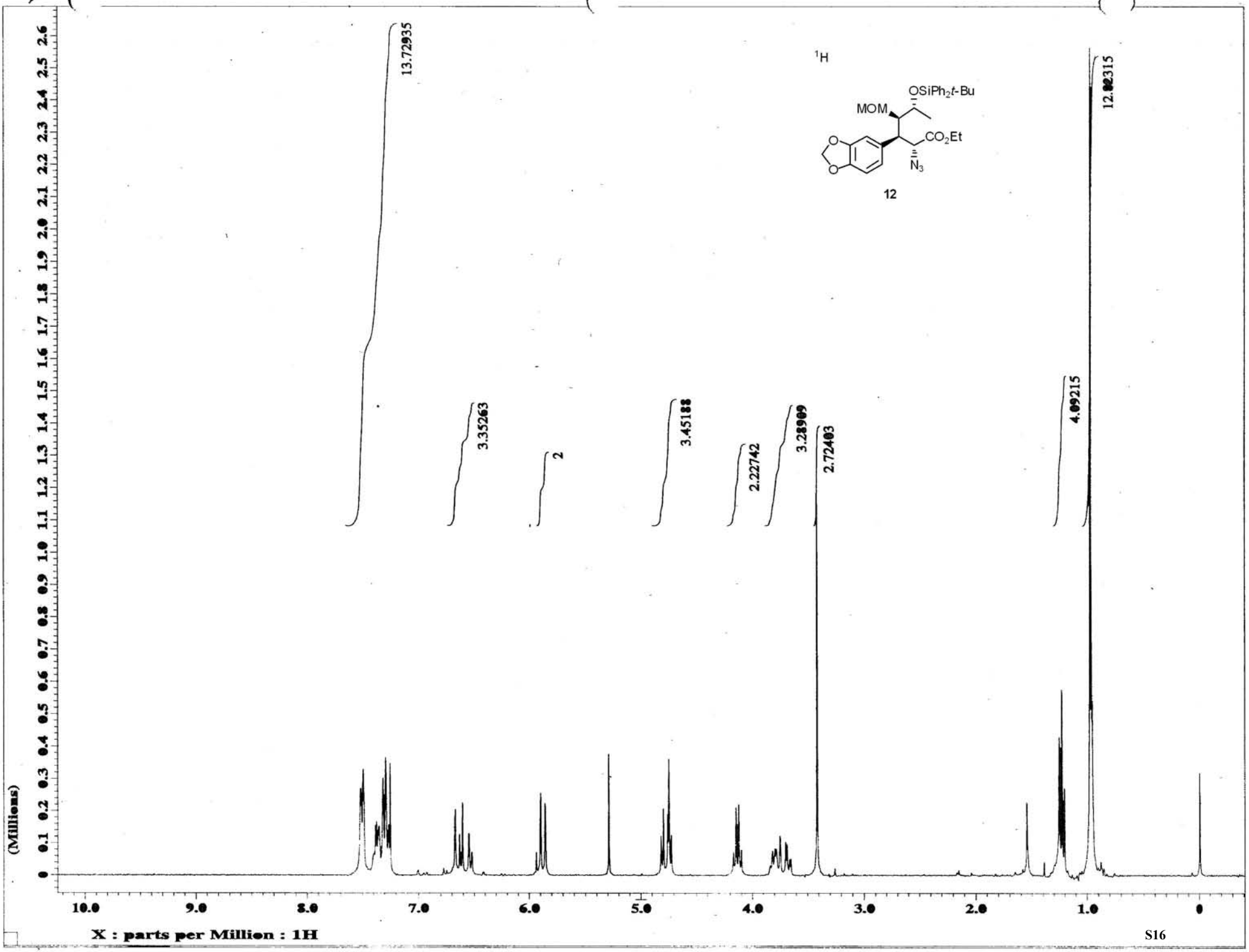
20.0
10.0
0
X : parts per Million : 13C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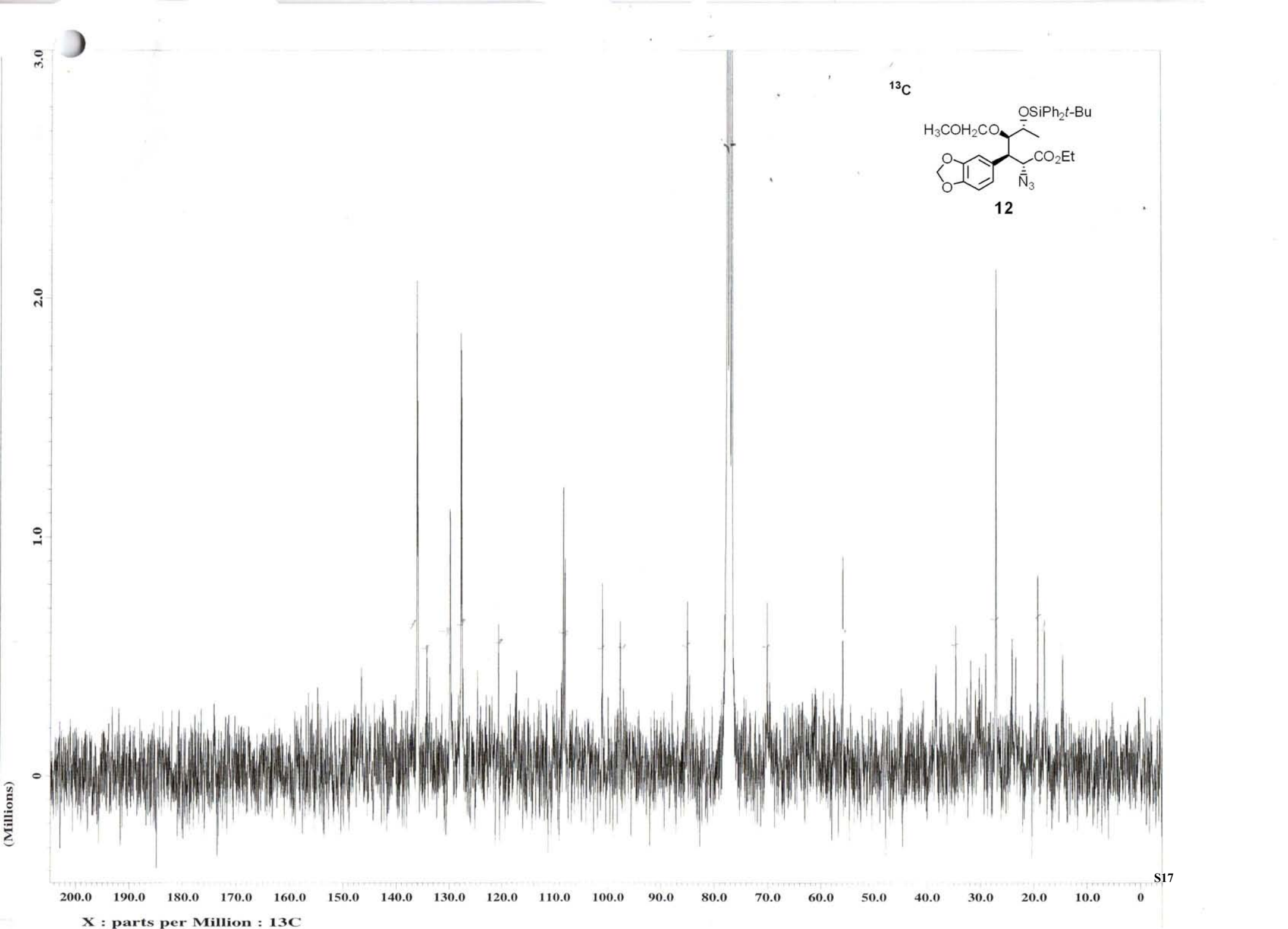
(Millions)
X : parts per Million : 1H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H$_3$COH$_2$CO
CO$_2$Et
N$_3$
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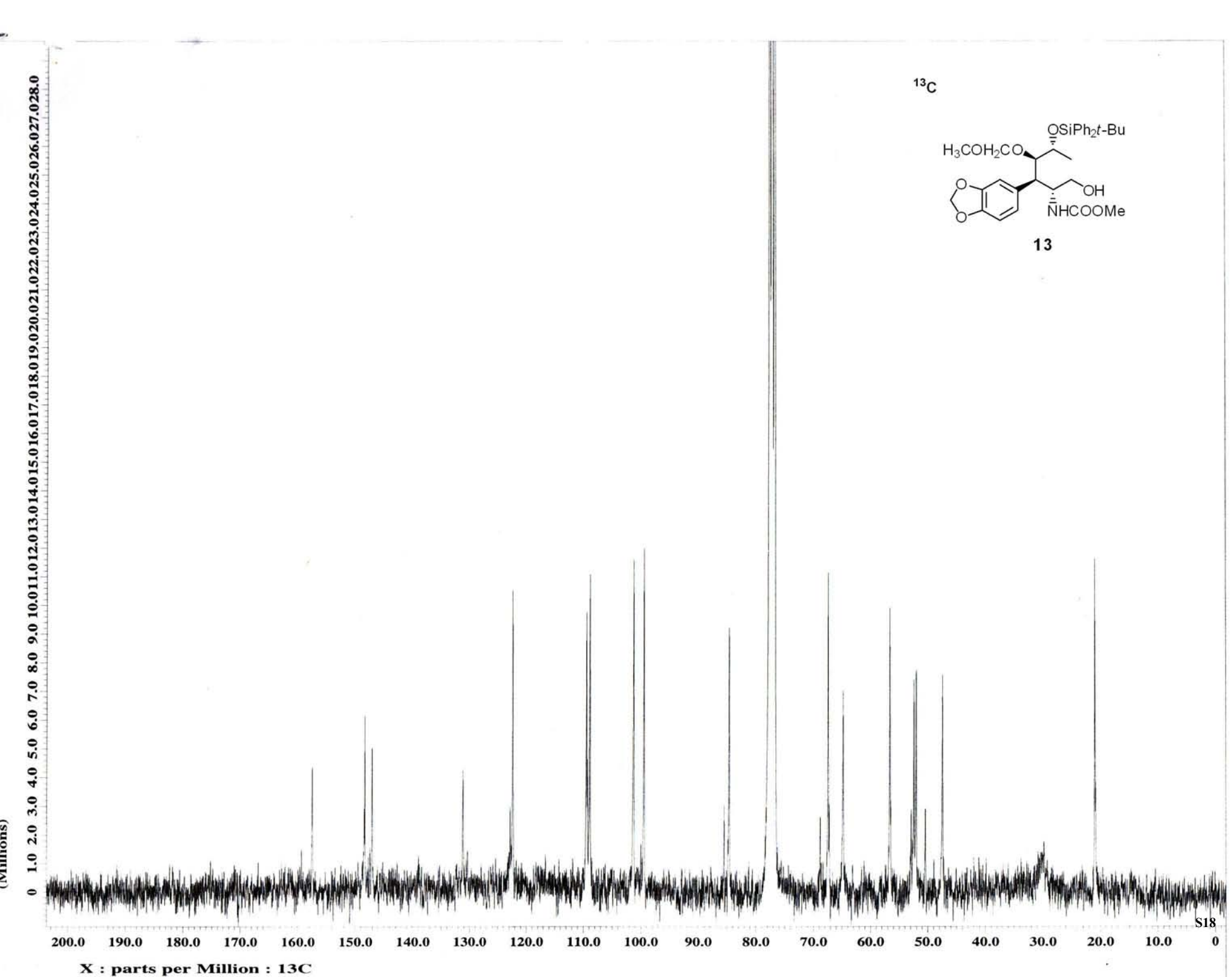
3.0
2.0
1.0
0
(Millions)
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
X : parts per Million : 13C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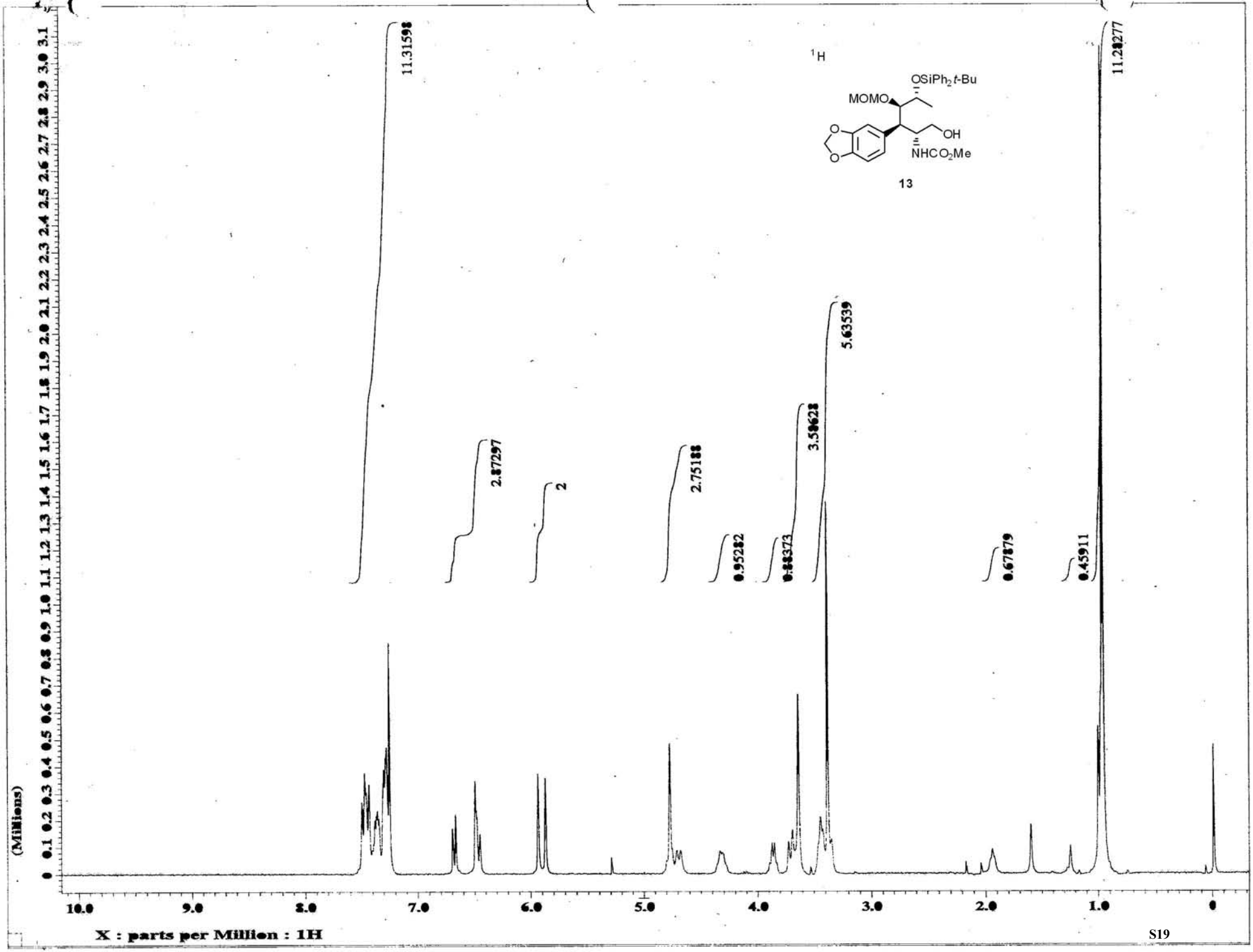
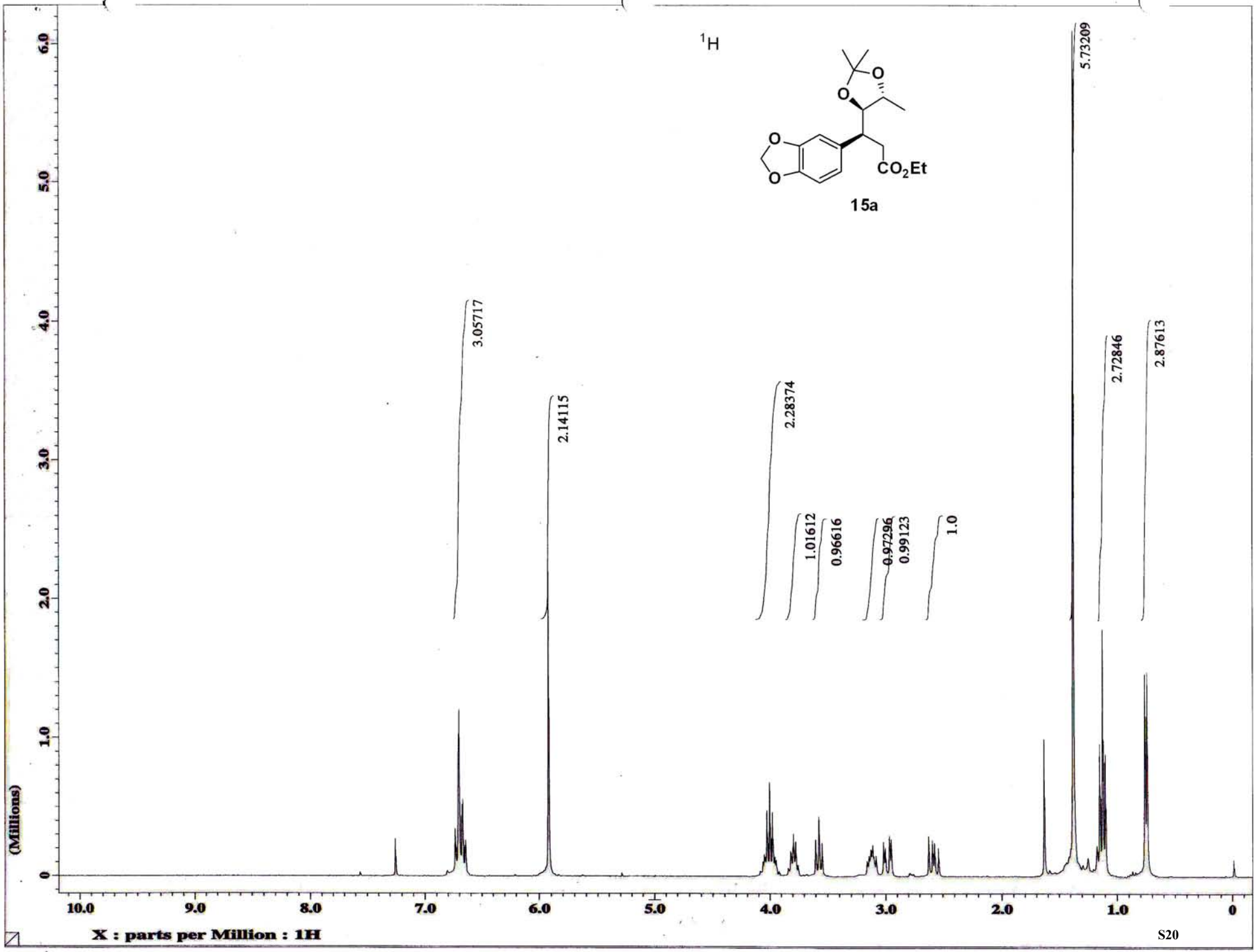
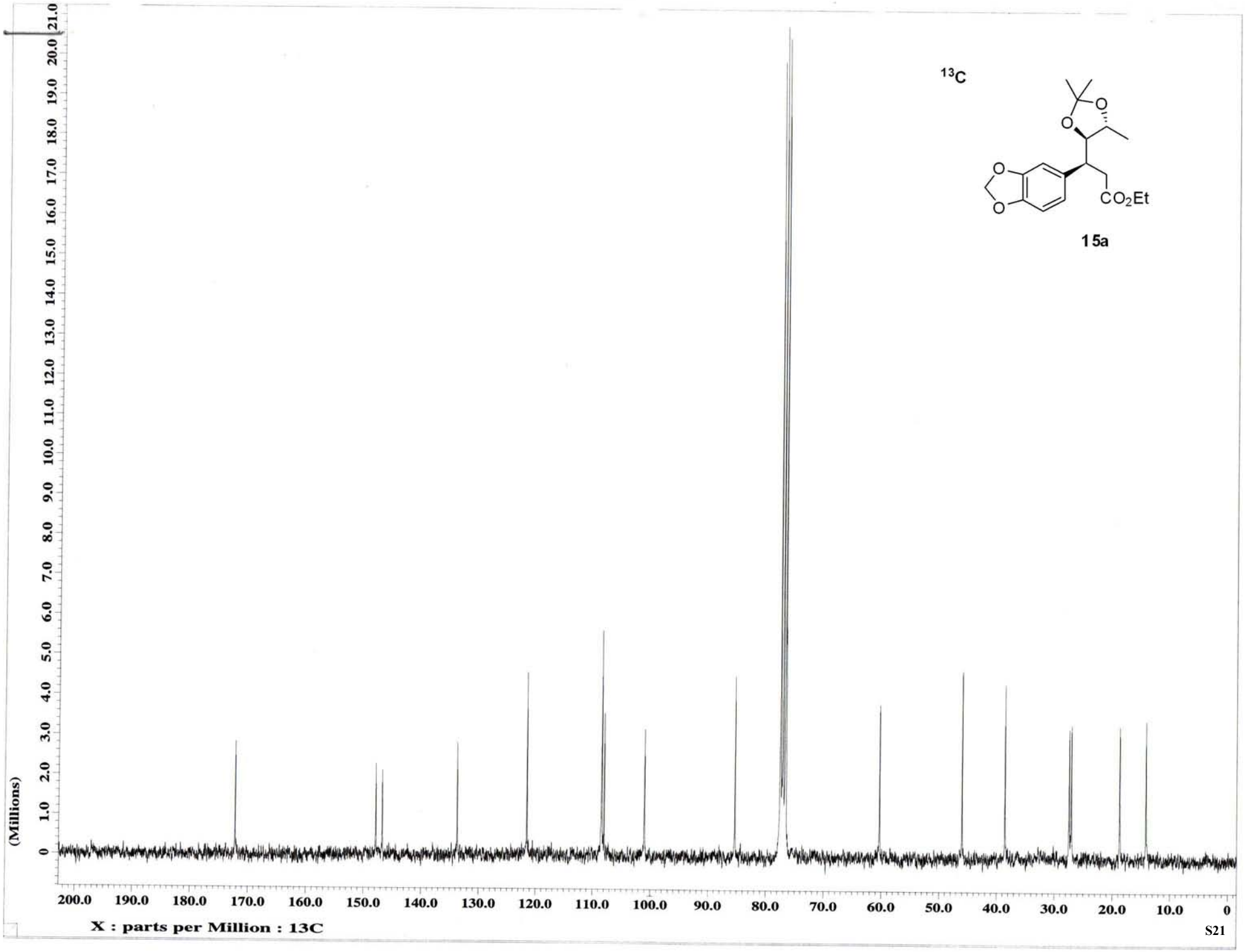
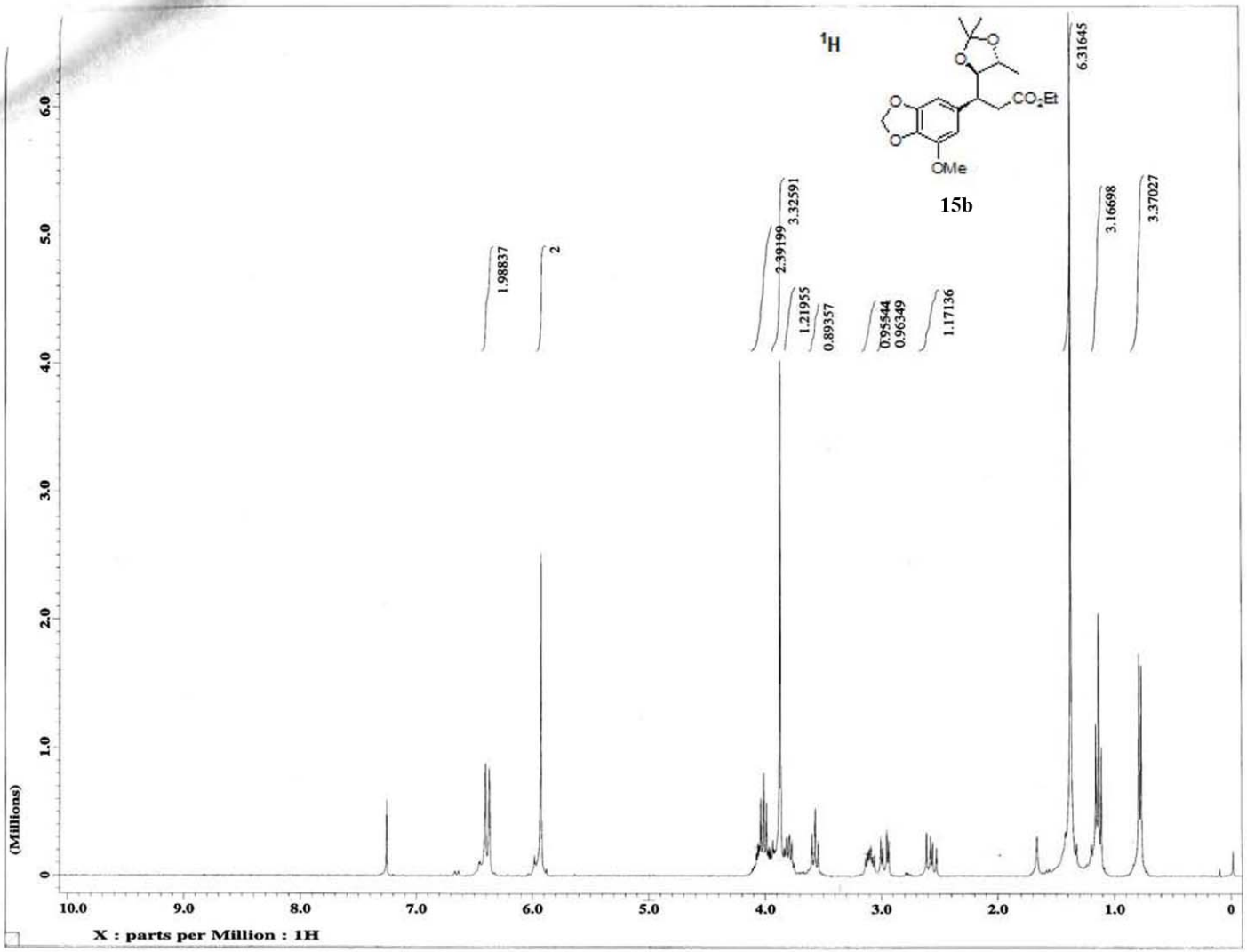
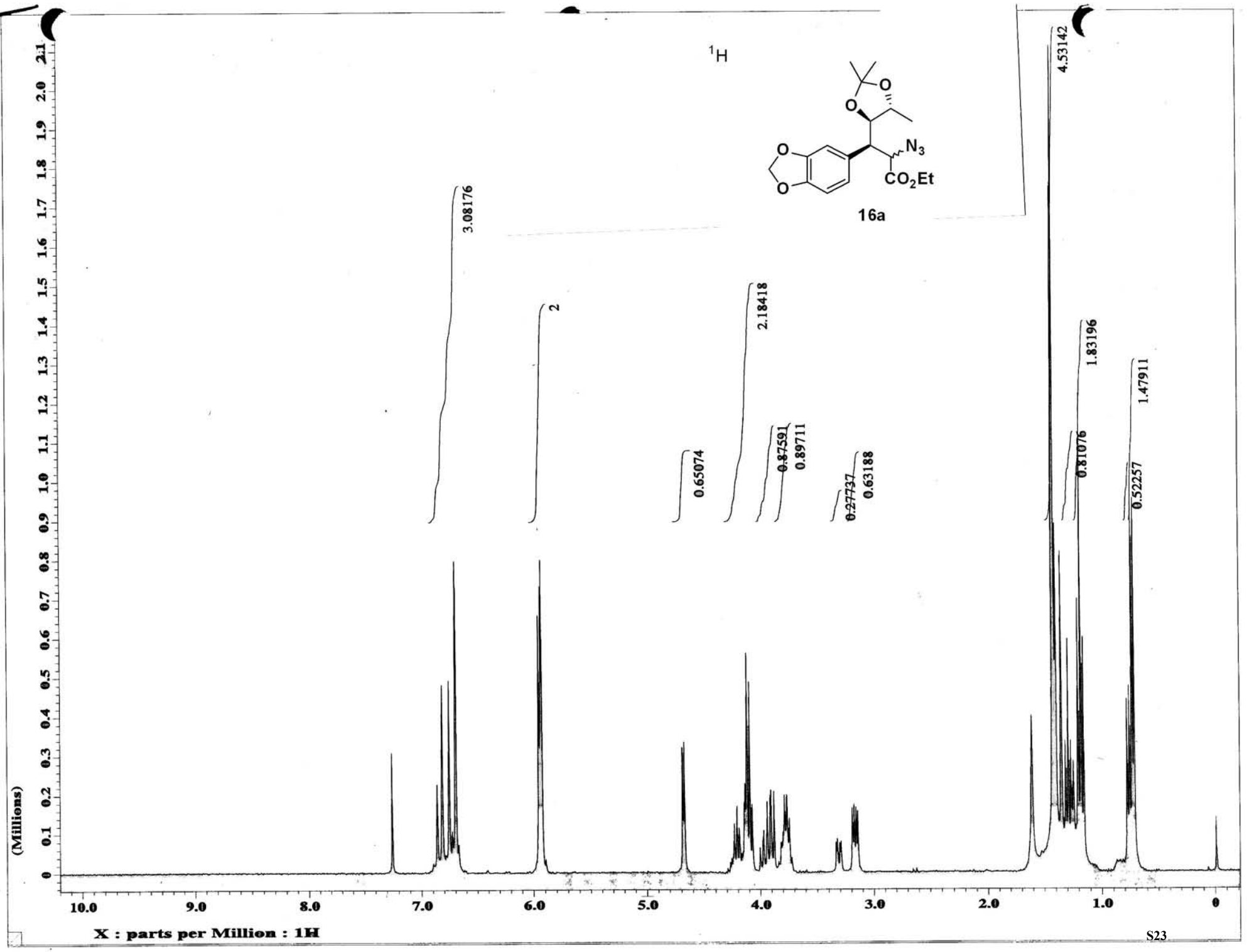
H$_3$COH$_2$CO
OH
NHCOOMe
13
(Millions)
0 1.0 2.0 3.0 4.0 5.0 6.0 7.0 8.0 9.0 10.0 11.0 12.0 13.0 14.0 15.0 16.0 17.0 18.0 19.0 20.0 21.0 22.0 23.0 24.0 25.0 26.0 27.0 28.0
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
X : parts per Million : 13C

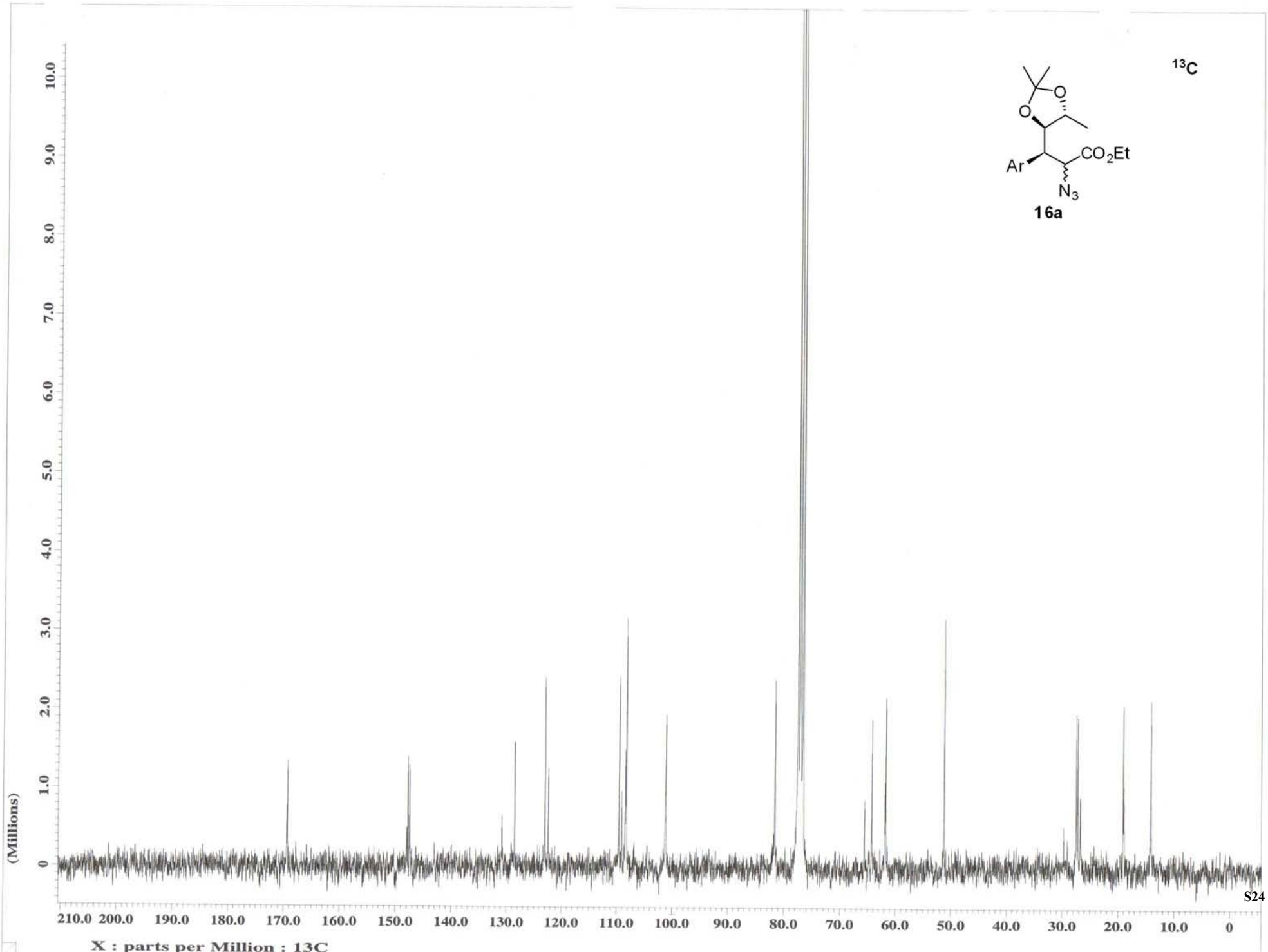

^{1}H
OSiPh$_2$t-Bu
MOMO
OH
NHCO$_2$Me
13
11.31598
2.87297
2
2.75188
0.95282
3.58628
5.63539
0.67879
0.45911
11.28277
(Millions)
X : parts per Million : 1H

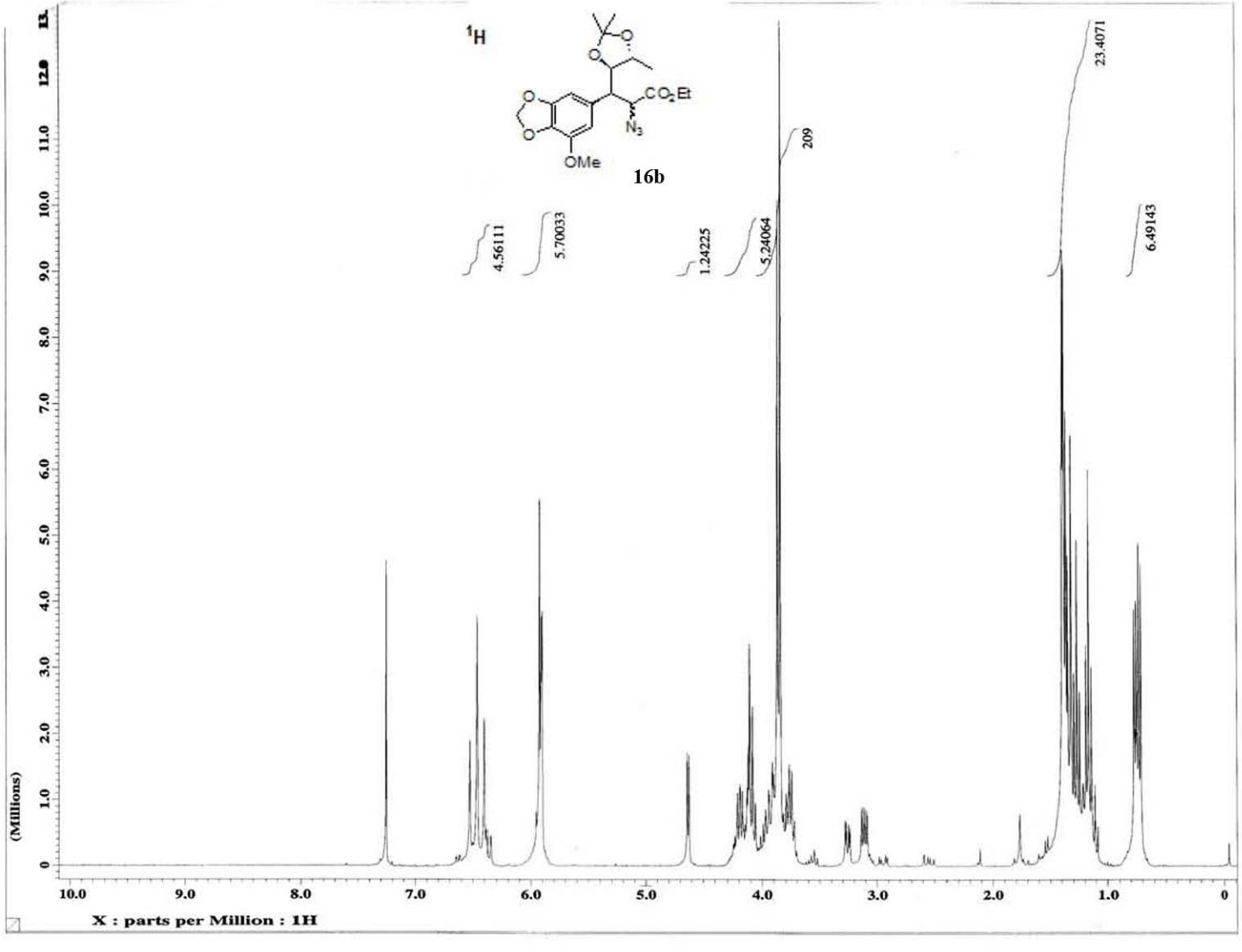

^{1}H
CO$_2$Et
15a
5.73209
3.05717
2.14115
2.28374
2.72846
2.87613
1.01612
0.96616
0.97296
0.99123
1.0
(Millions)
X : parts per Million : 1H

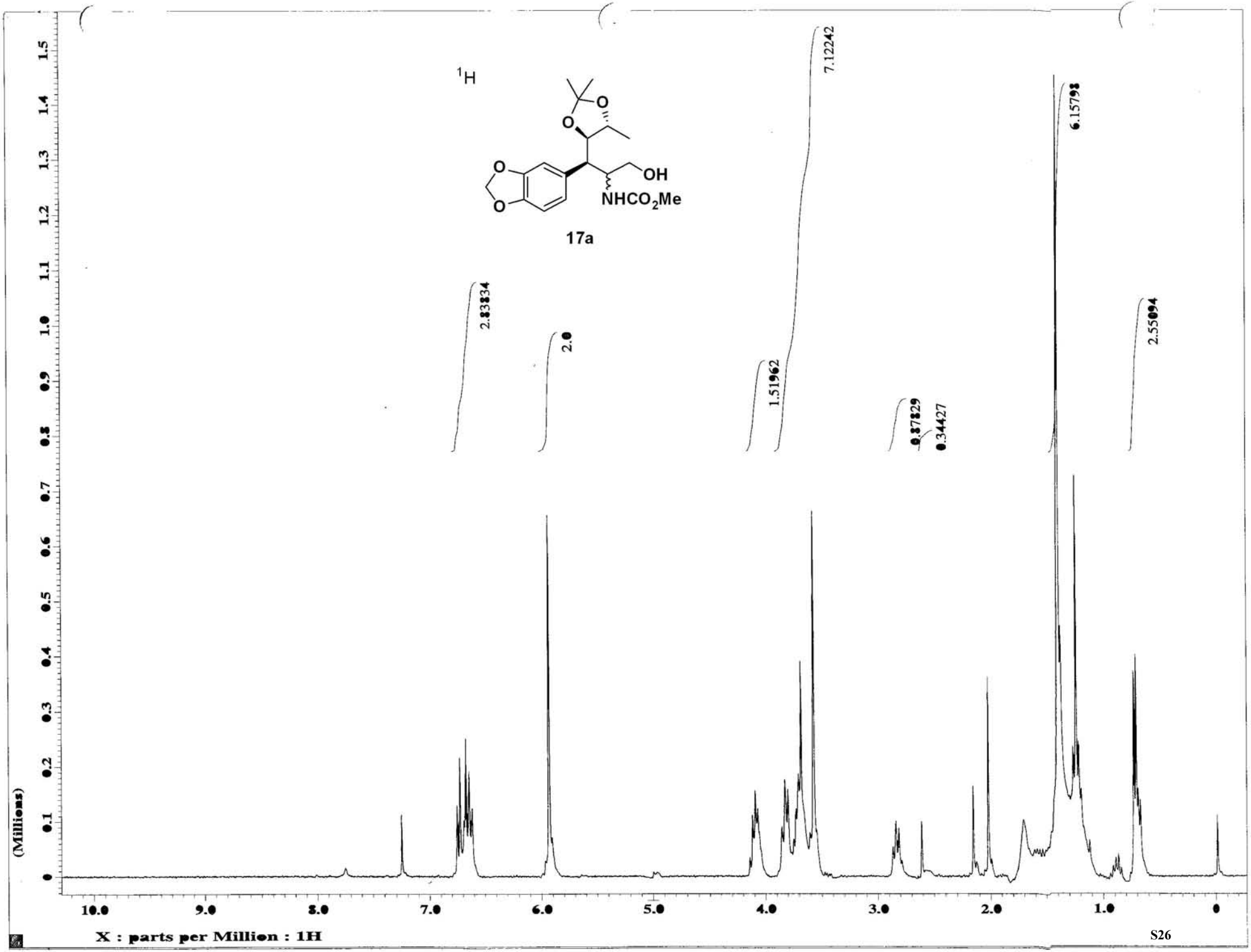

^{13}C
CO_2Et
15a
(Millions)
21.0
20.0
19.0
18.0
17.0
16.0
15.0
14.0
13.0
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
X : parts per Million : 13C

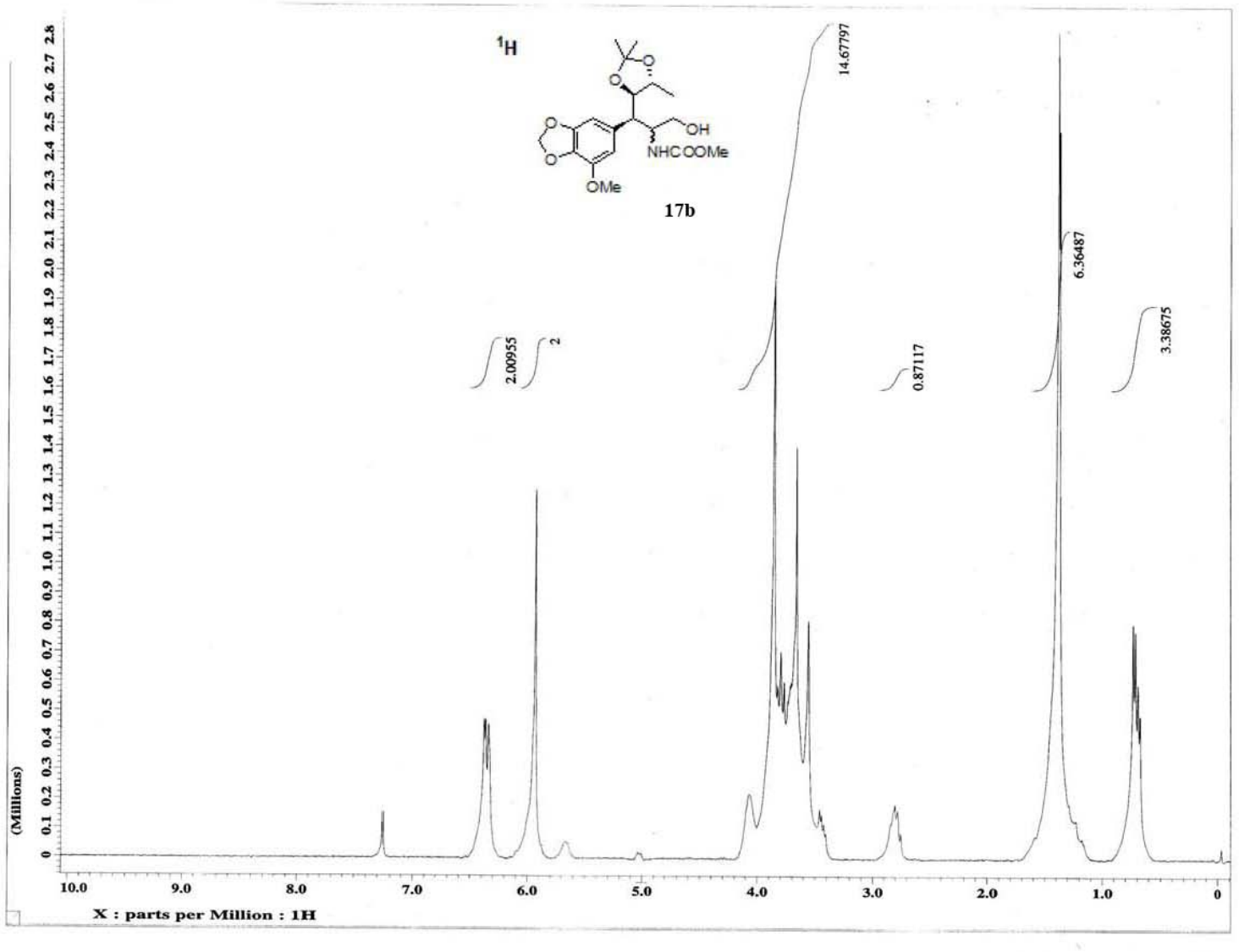

^{1}H
15b
1.98837
2
2.39199
3.32591
1.21955
0.89357
0.95544
0.96349
1.17136
6.31645
3.16698
3.37027
(Millions)
X : parts per Million : 1H

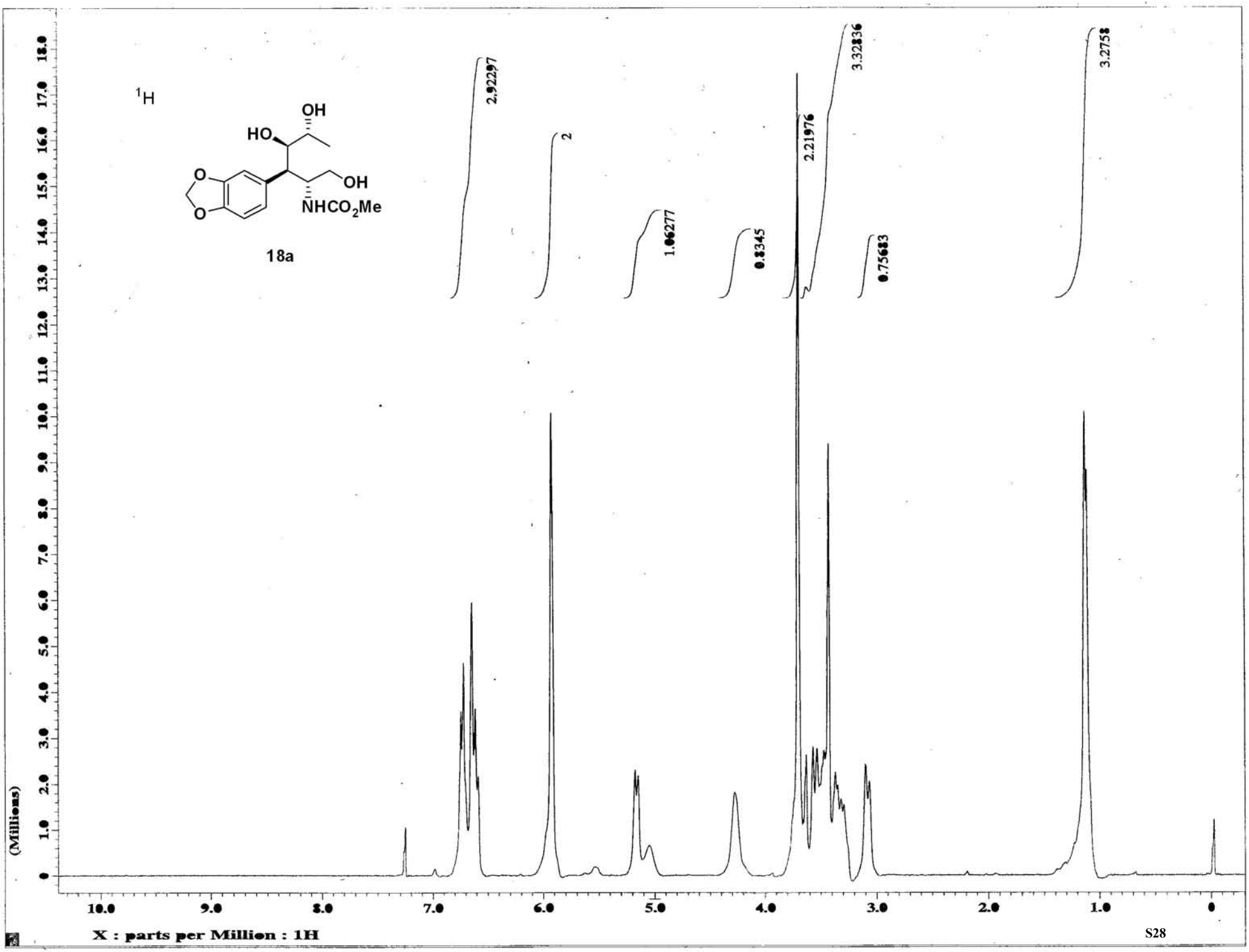

^{1}H
16a
3.08176
2
0.65074
2.18418
0.87591
0.89711
0.27737
0.63188
4.53142
0.81076
1.83196
0.52257
1.47911
(Millions)
X : parts per Million : 1H

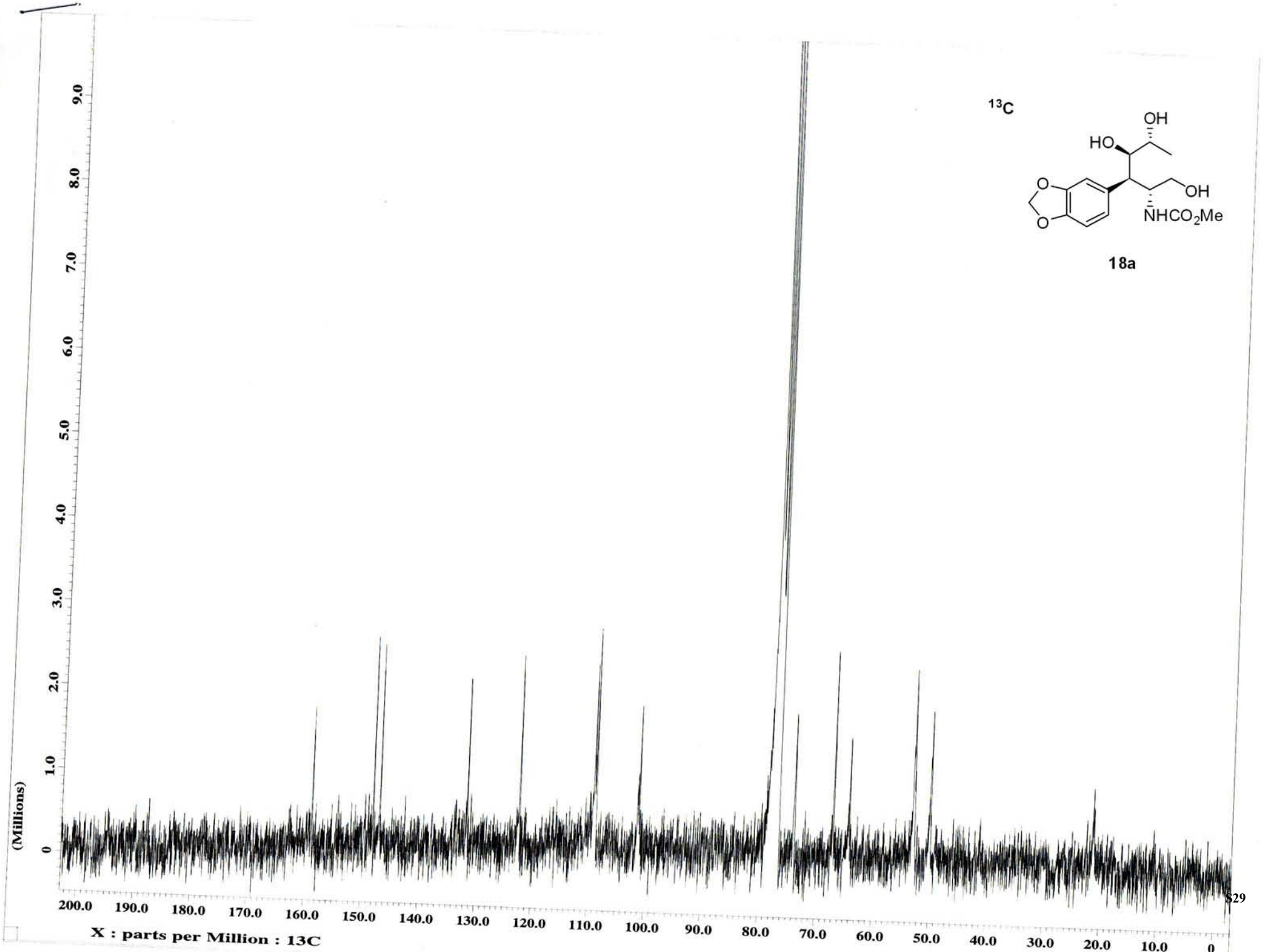

^{13}C
Ar
CO_2Et
N_3
16a
(Millions)
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
X : parts per Million : 13C

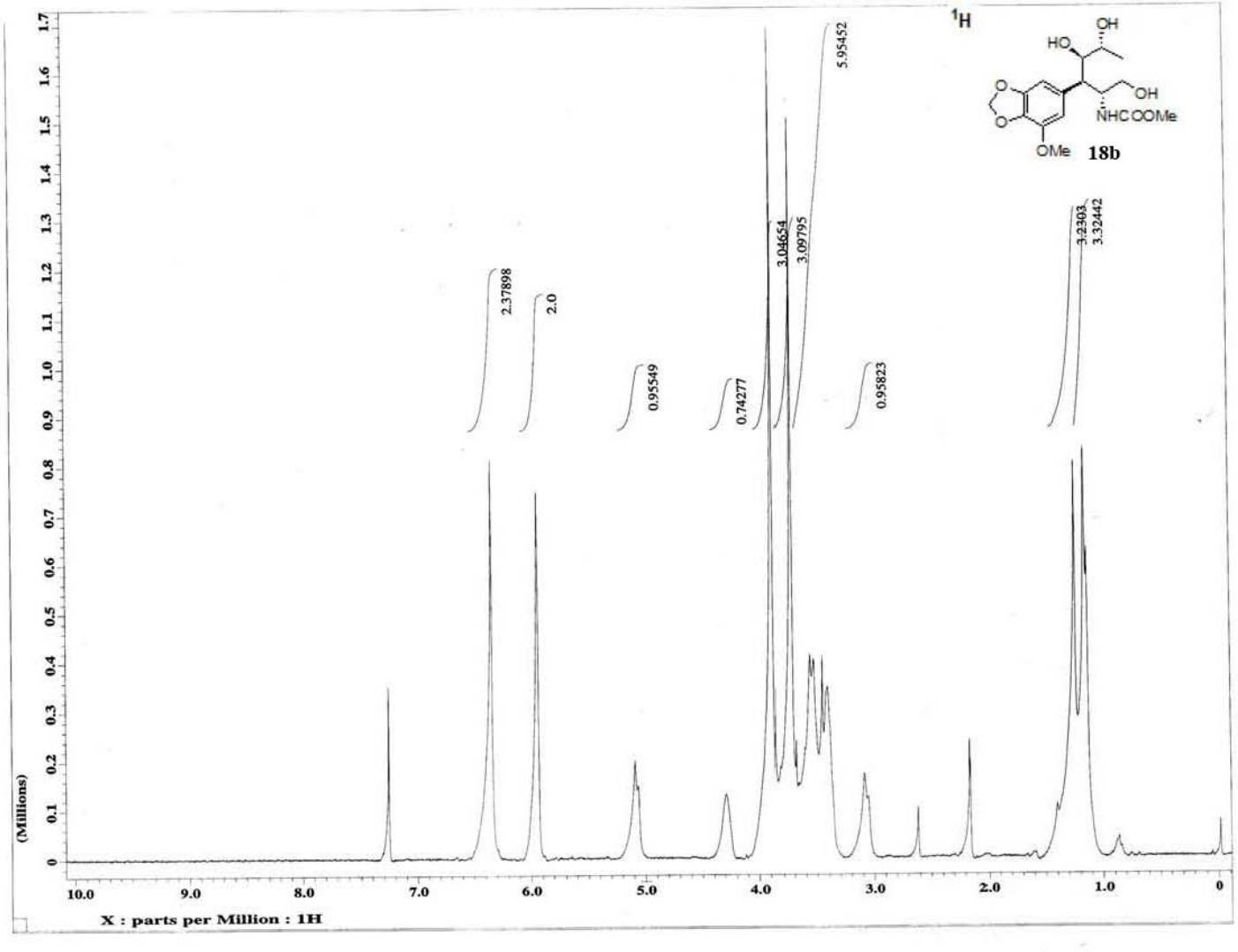

^{1}H
16b
4.56111
5.70033
1.24225
5.24064
209
23.4071
6.49143
(Millions)
X : parts per Million : 1H

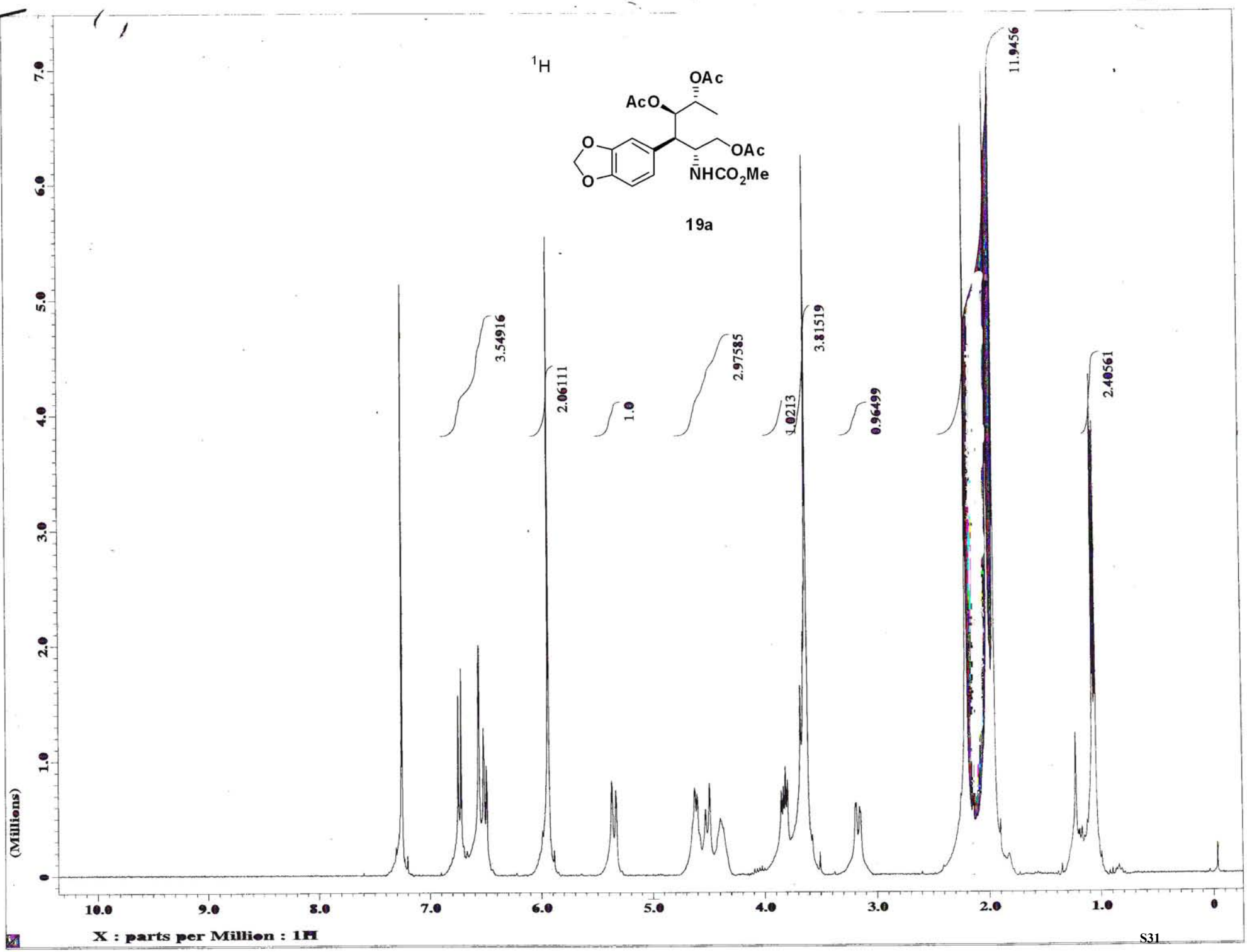

^{1}H
OH
NHCO$_2$Me
17a
7.12242
6.15798
2.83834
2.0
1.51962
0.87829
0.34427
2.55094
(Millions)
X : parts per Million : 1H

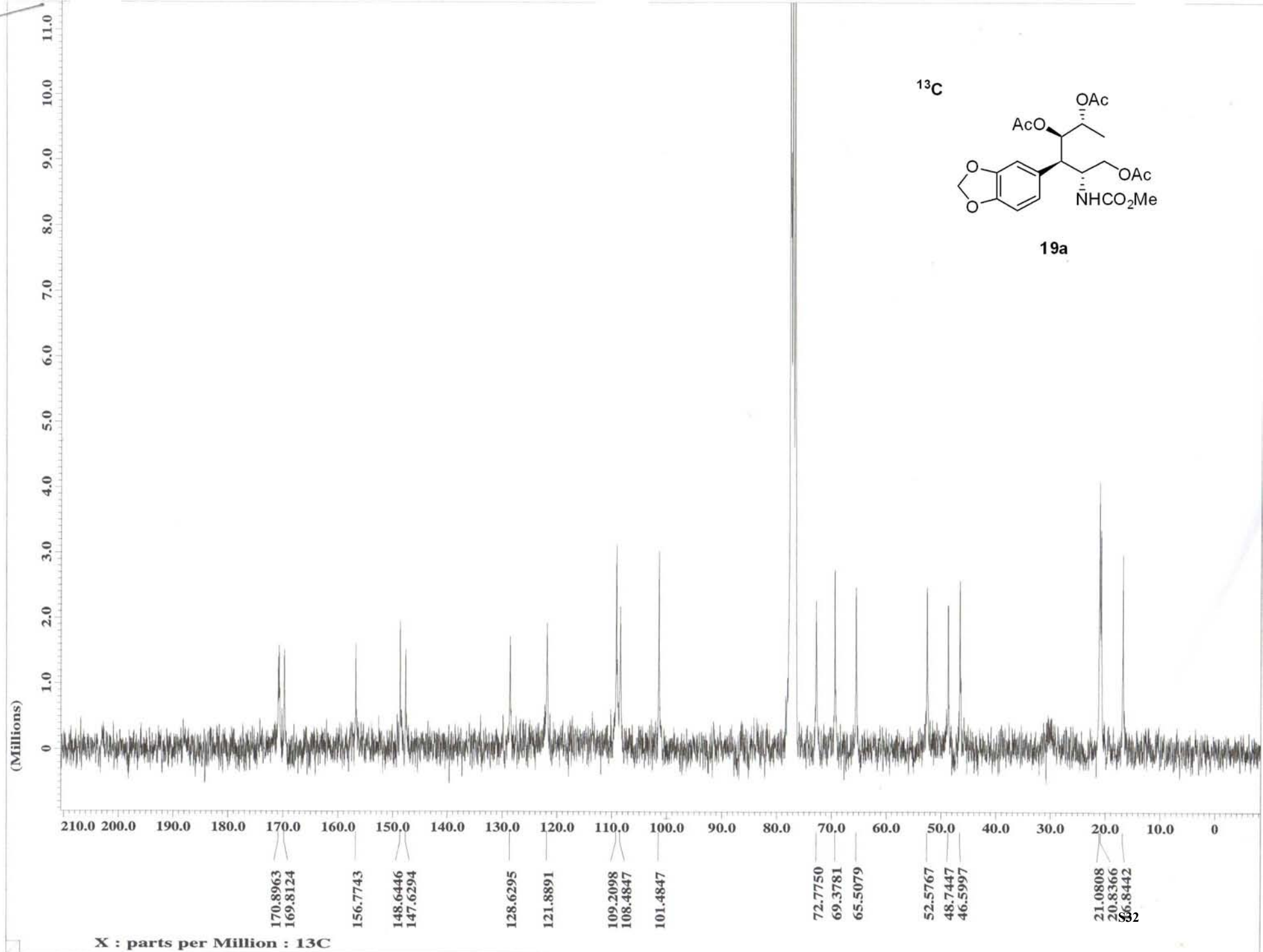

^{1}H
OH
NHCOOMe
OMe
17b
14.67797
6.36487
3.38675
2.00955
2
0.87117
(Millions)
X : parts per Million : 1H

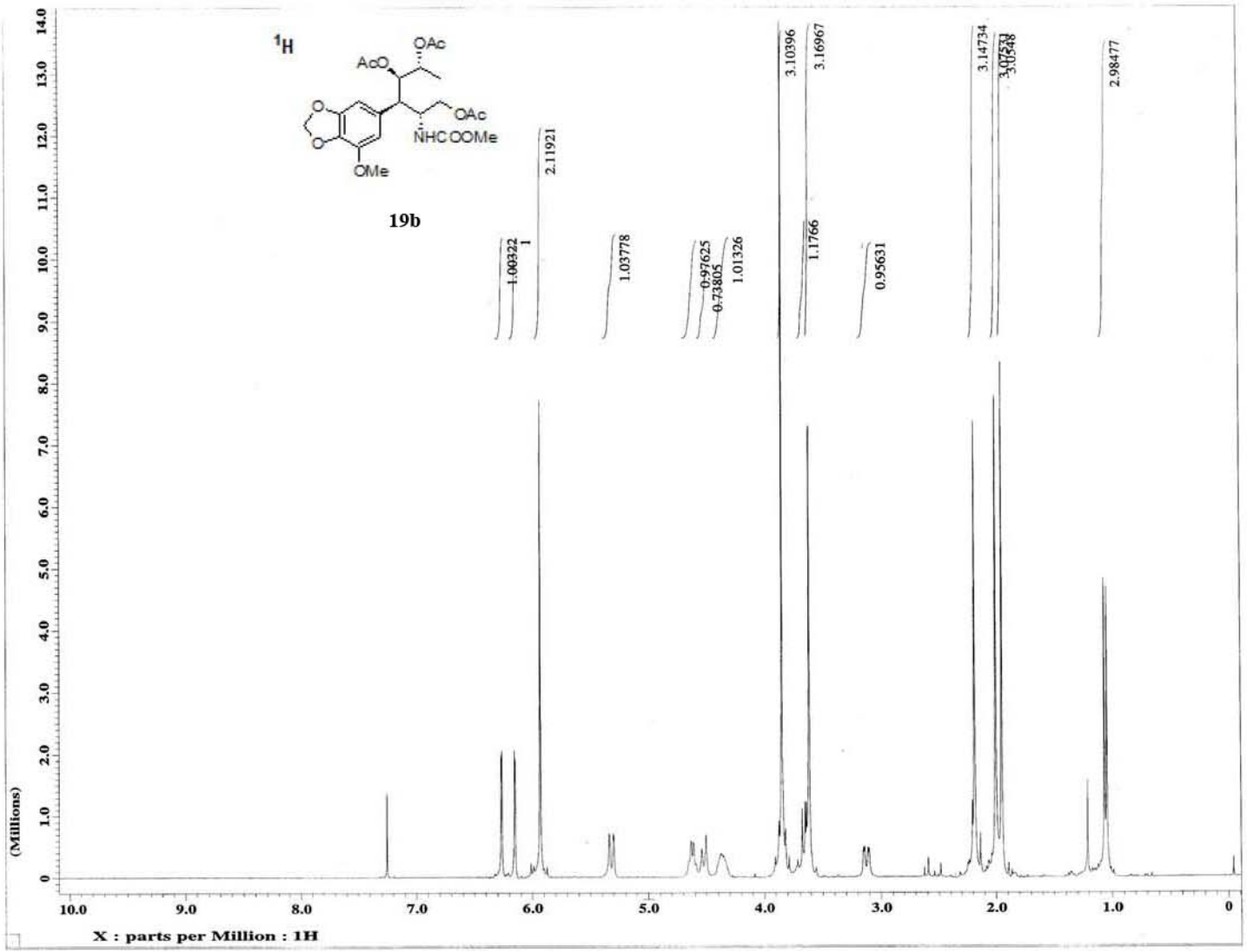

^{1}H
OH
HO
OH
O
O
NHCO$_2$Me
18a
2.92297
2
1.06277
0.8345
2.21976
3.32836
0.75683
3.27558
(Millions)
X : parts per Million : 1H

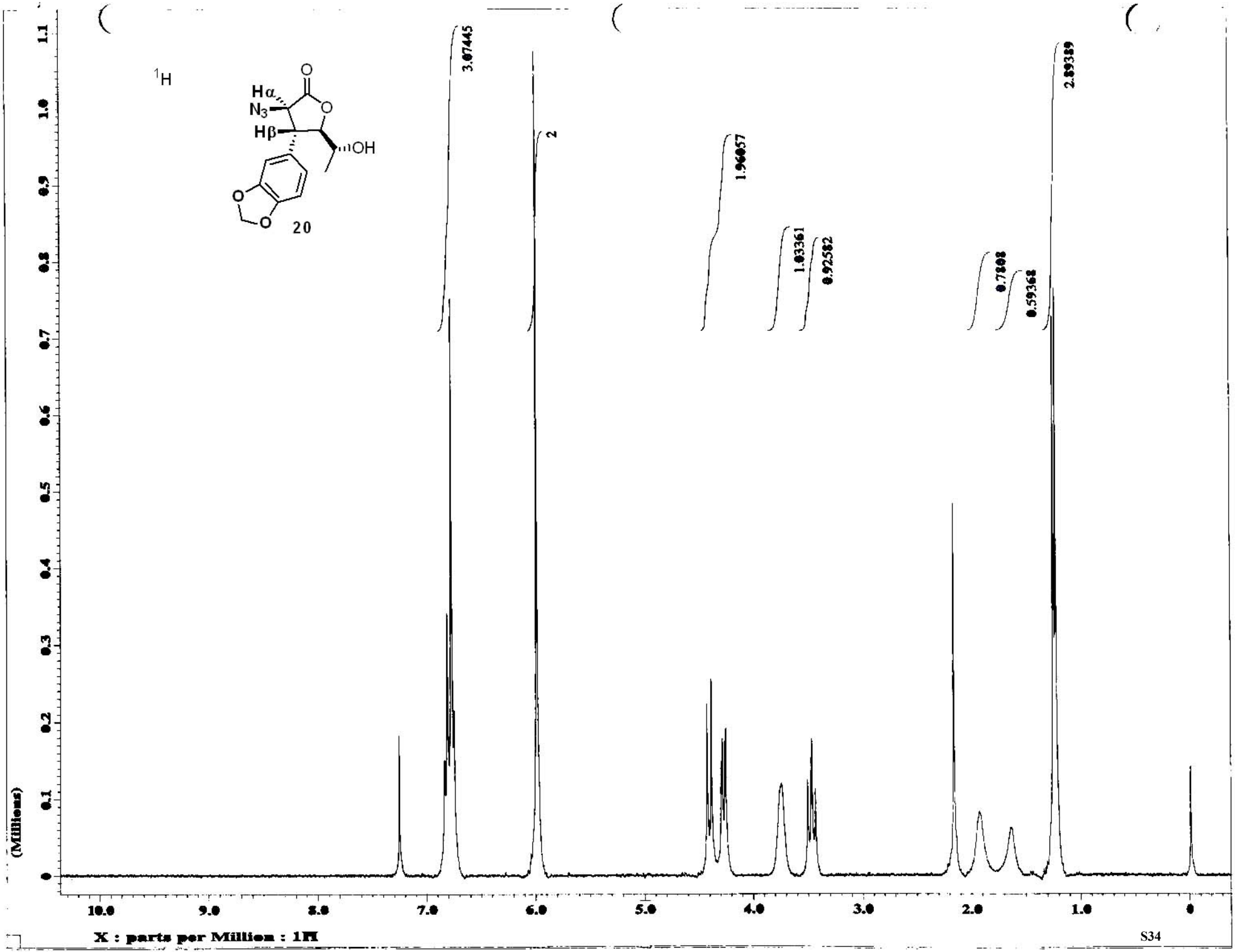

^{13}C
OH
HO
O
O
OH
NHCO$_2$Me
18a
(Millions)
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
X : parts per Million : 13C

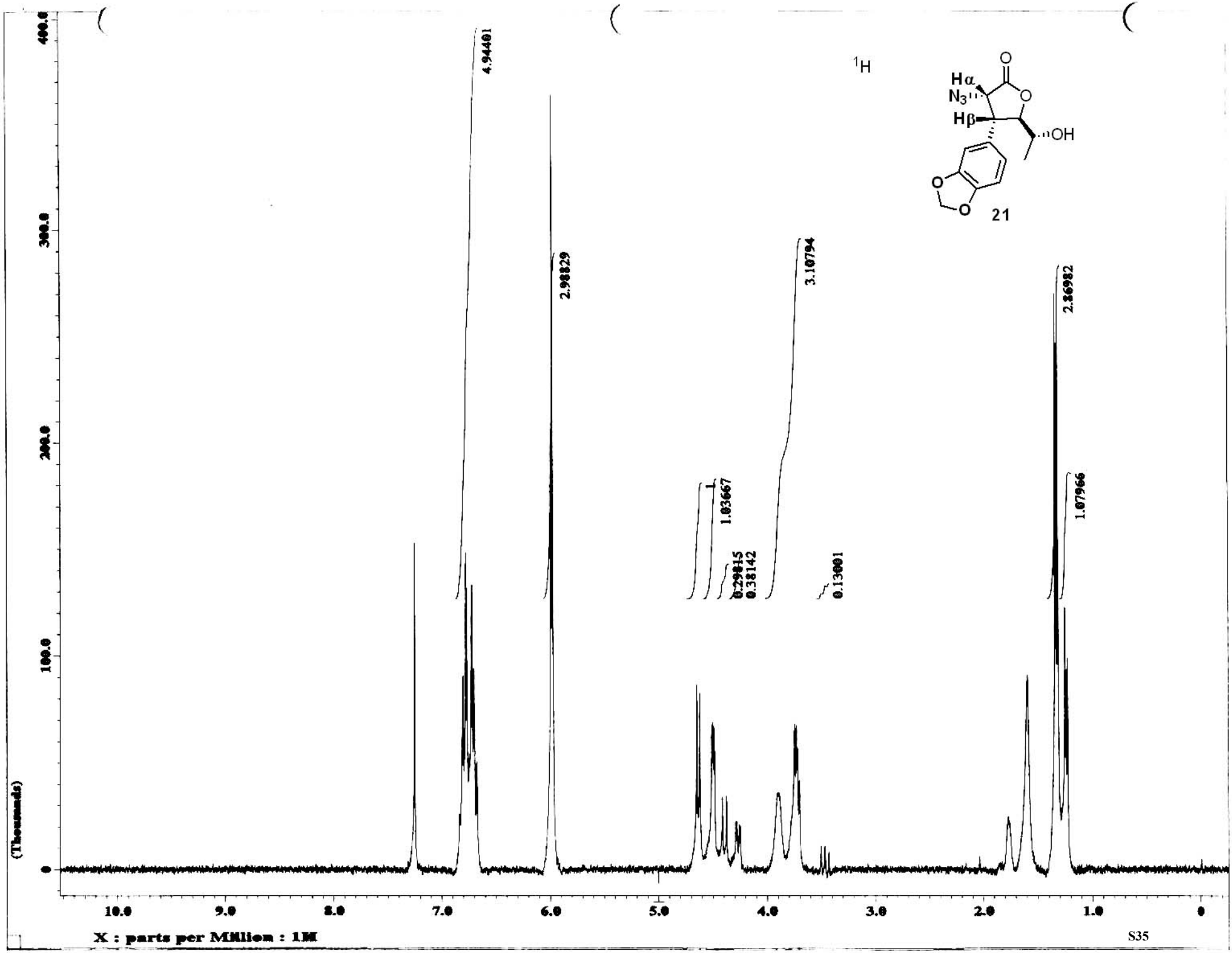

1H
HO
OH
OH
NHCOOMe
OMe
18b
2.37898
2.0
0.95549
0.74277
3.04654
3.09795
5.95452
0.95823
3.2303
3.32442
(Millions)
X : parts per Million : 1H

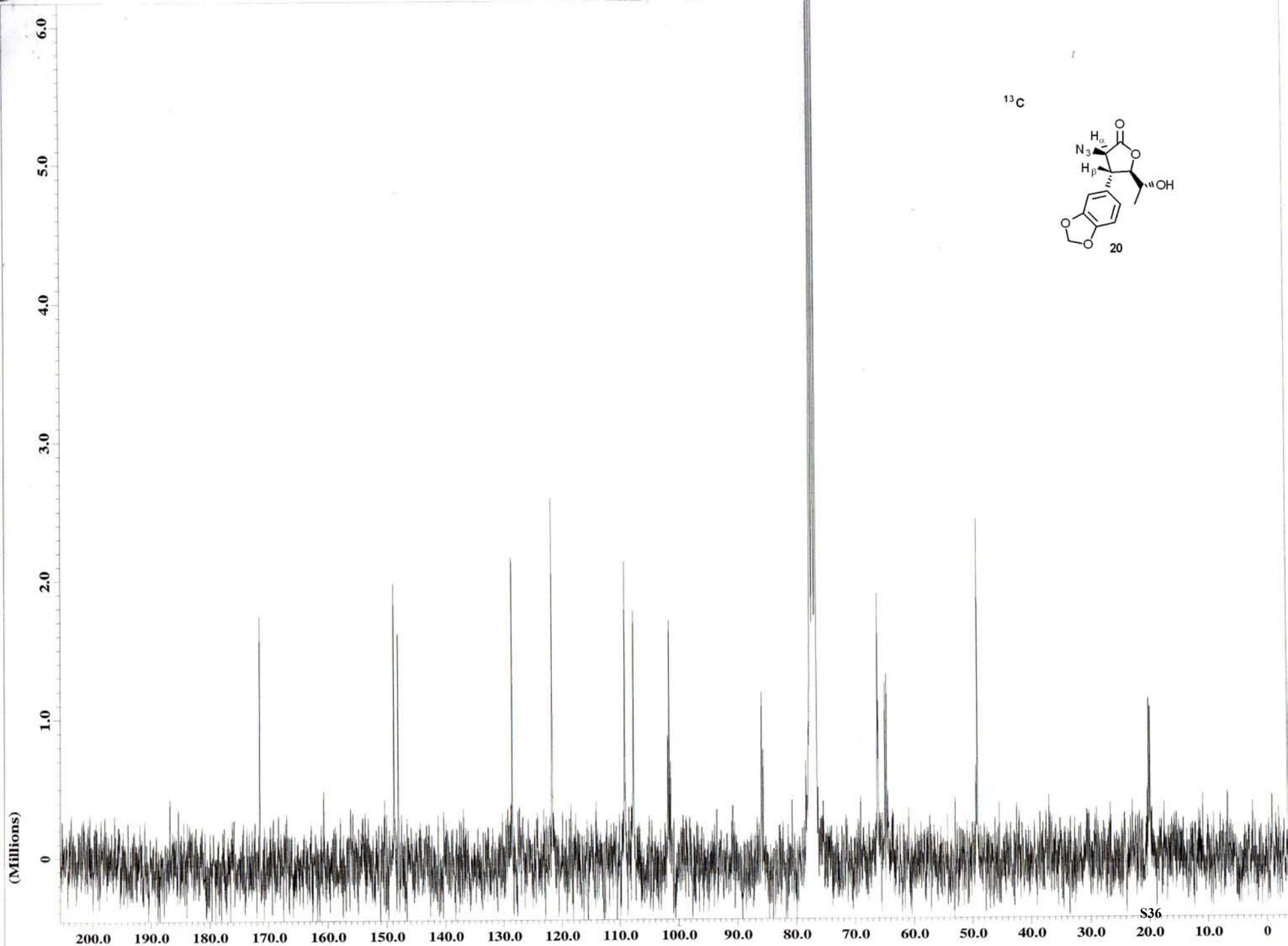

^{1}H
OAc
AcO
OAc
NHCO$_2$Me
19a
3.54916
2.06111
1.0
2.97585
1.0213
3.81519
0.96499
11.9456
2.40561
(Millions)
X : parts per Million : 1H

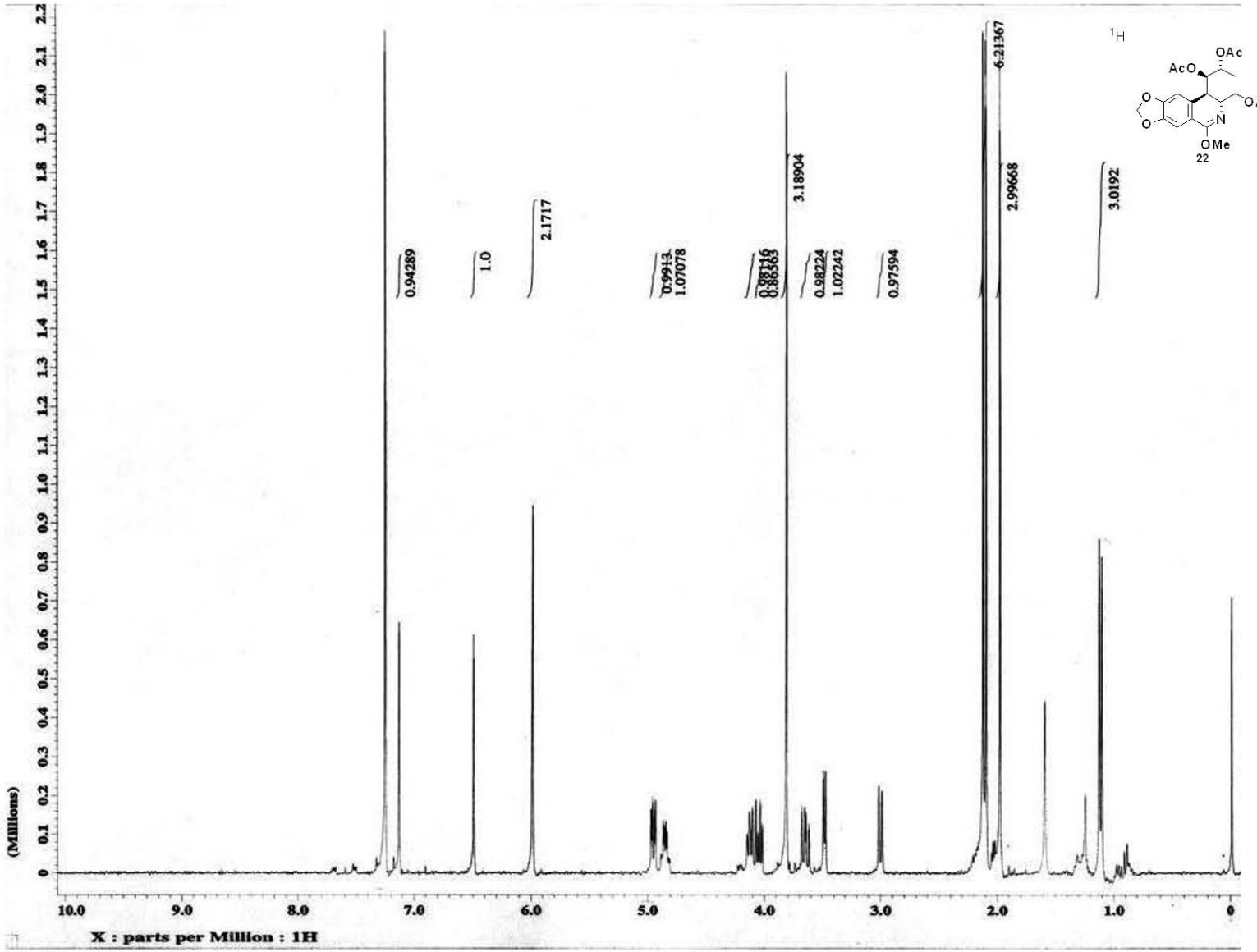

^{13}C
OAc
AcO
OAc
NHCO$_2$Me
19a
(Millions)
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
170.8963
169.8124
156.7743
148.6446
147.6294
128.6295
121.8891
109.2098
108.4847
101.4847
72.7750
69.3781
65.5079
52.5767
48.7447
46.5997
21.0808
20.8366
16.8442
X : parts per Million : 13C

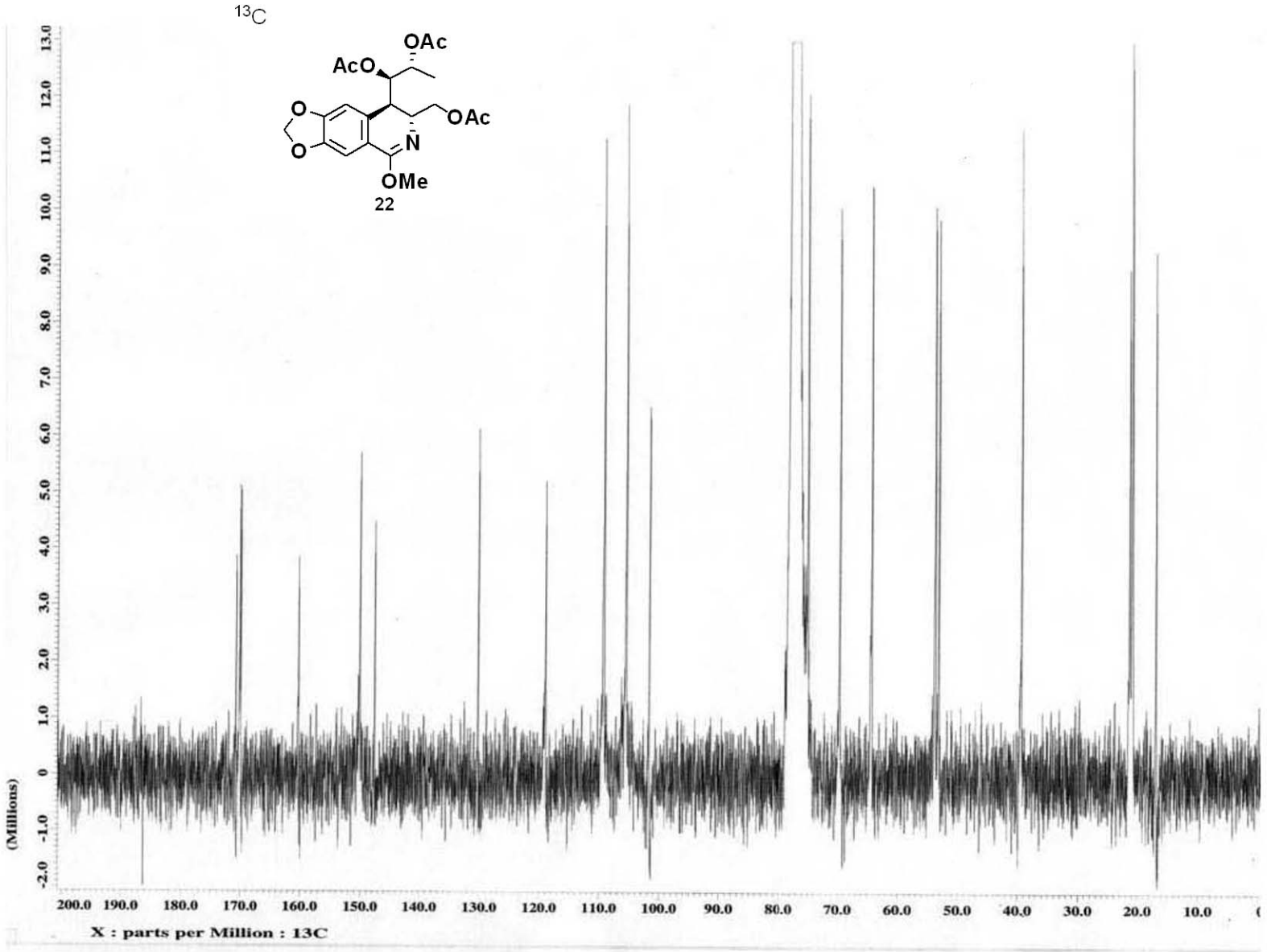

1H
AcO
OAc
OAc
NHCOOMe
OMe
19b
1.00322
1
2.11921
1.03778
0.97625
0.73805
1.01326
3.10396
3.16967
1.1766
0.95631
3.14734
3.07531
3.05348
2.98477
(Millions)
X : parts per Million : 1H

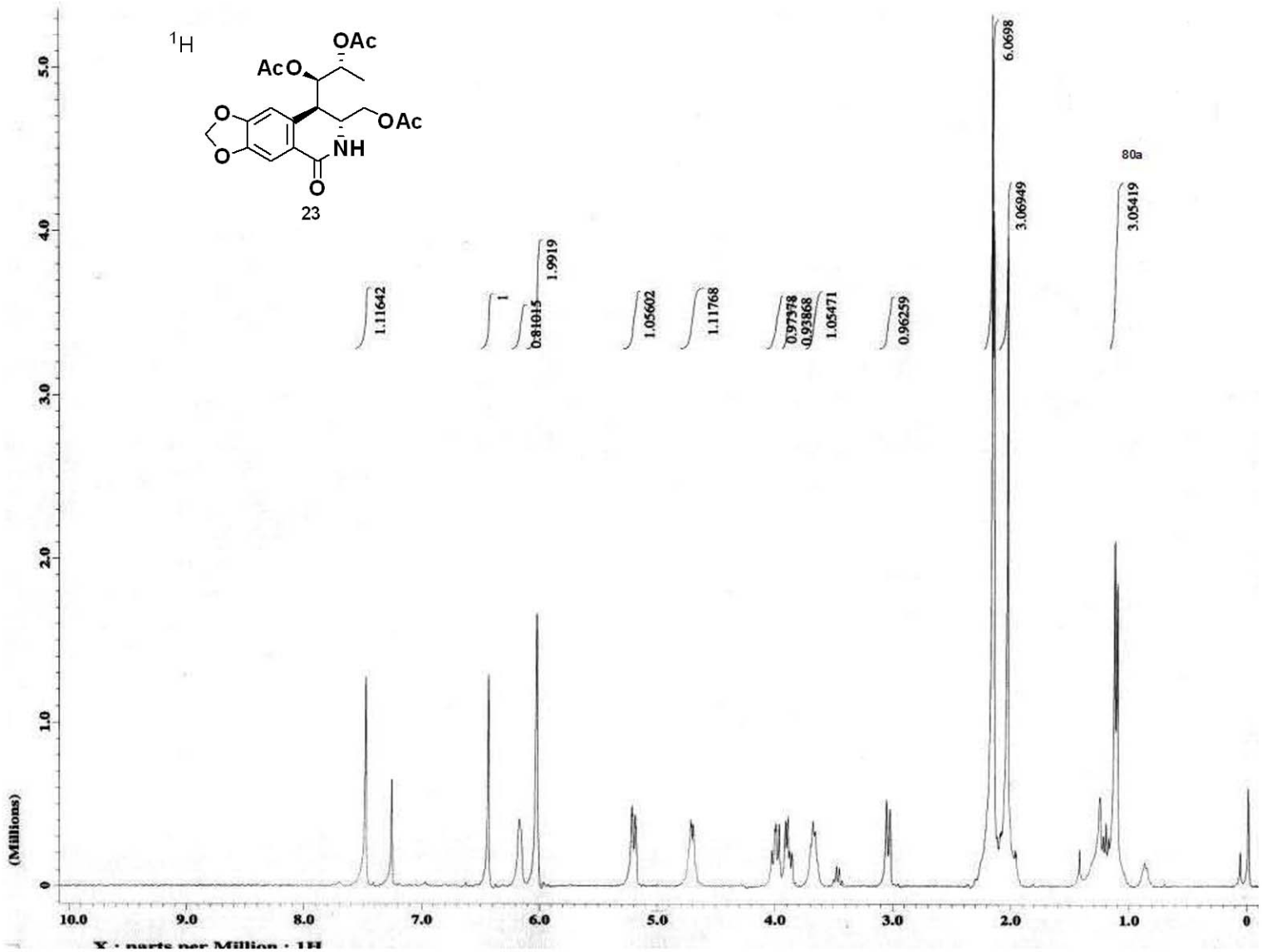

^{1}H
Hα
N3
Hβ
OH
O
O
O
O
20
3.07445
2
1.96057
1.03361
0.92582
0.78008
0.59368
2.89389
(Millions)
X : parts per Million : 1H

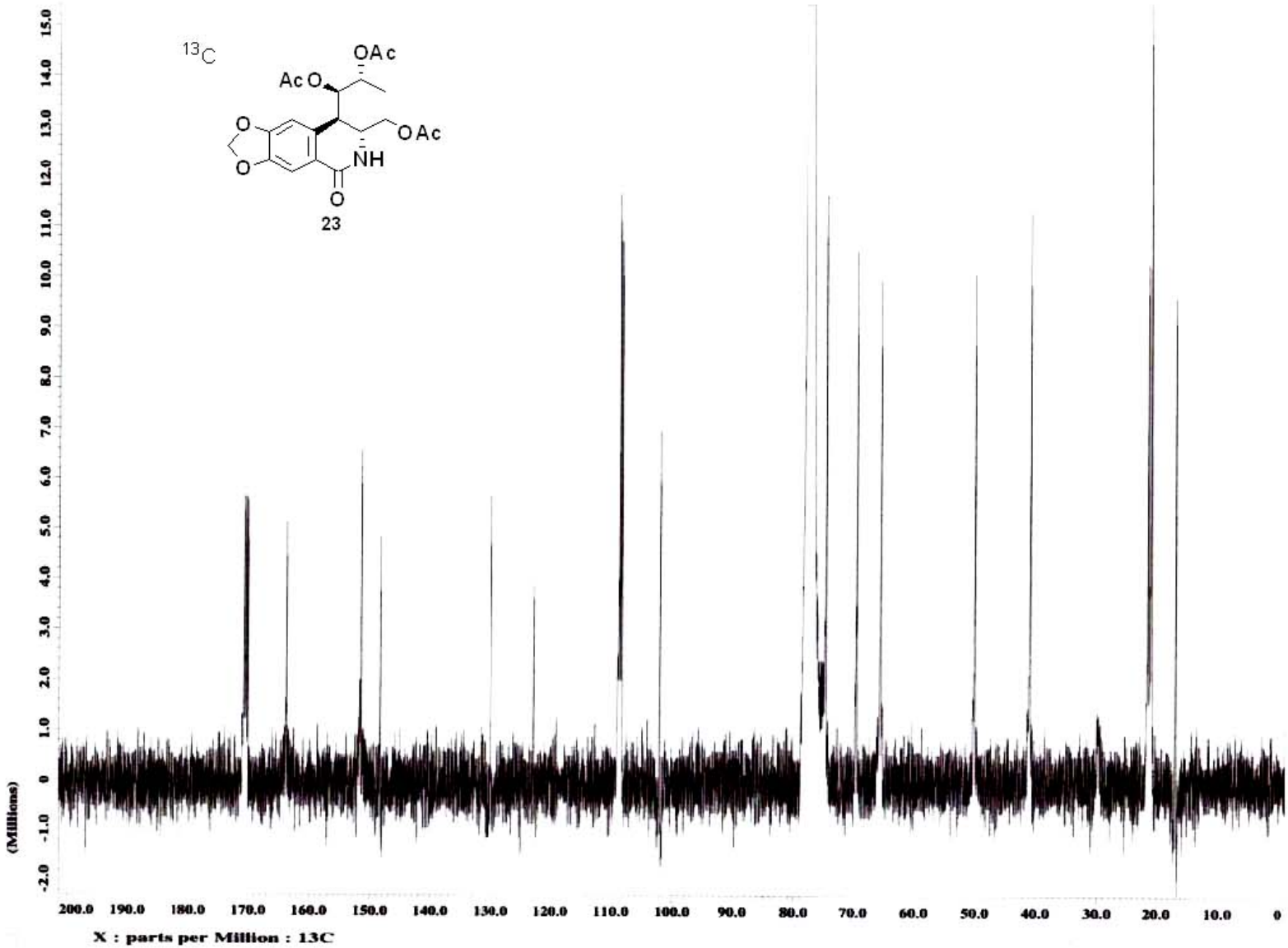

^{1}H
Hα
N3
Hβ
OH
21
4.94401
2.98829
1.03667
0.29815
0.38142
3.10794
0.13001
2.86982
1.07966
(Thousands)
X : parts per Million : 1M

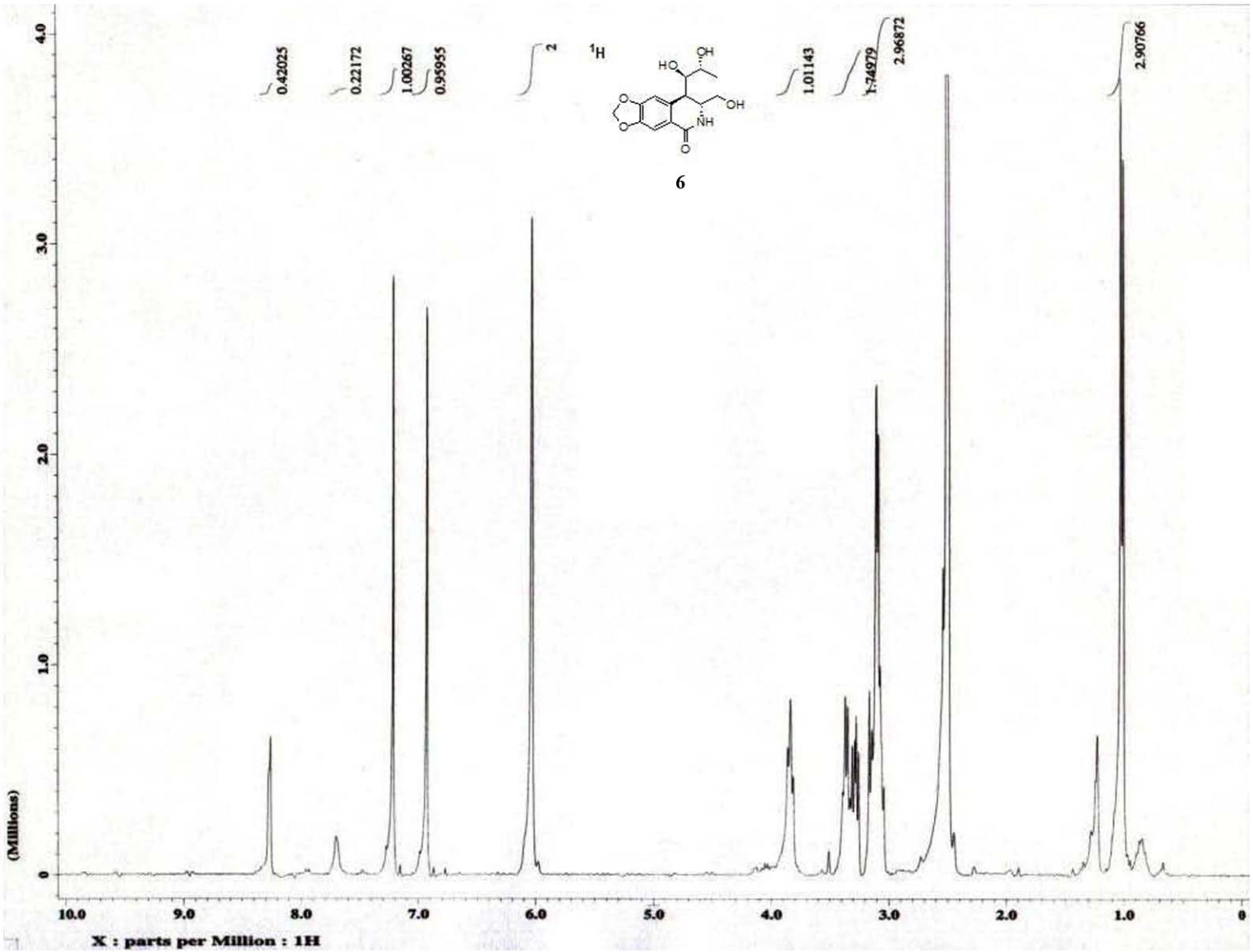

13C
20
(Millions)
6.0
5.0
4.0
3.0
2.0
1.0
0
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
X : parts per Million : 13C

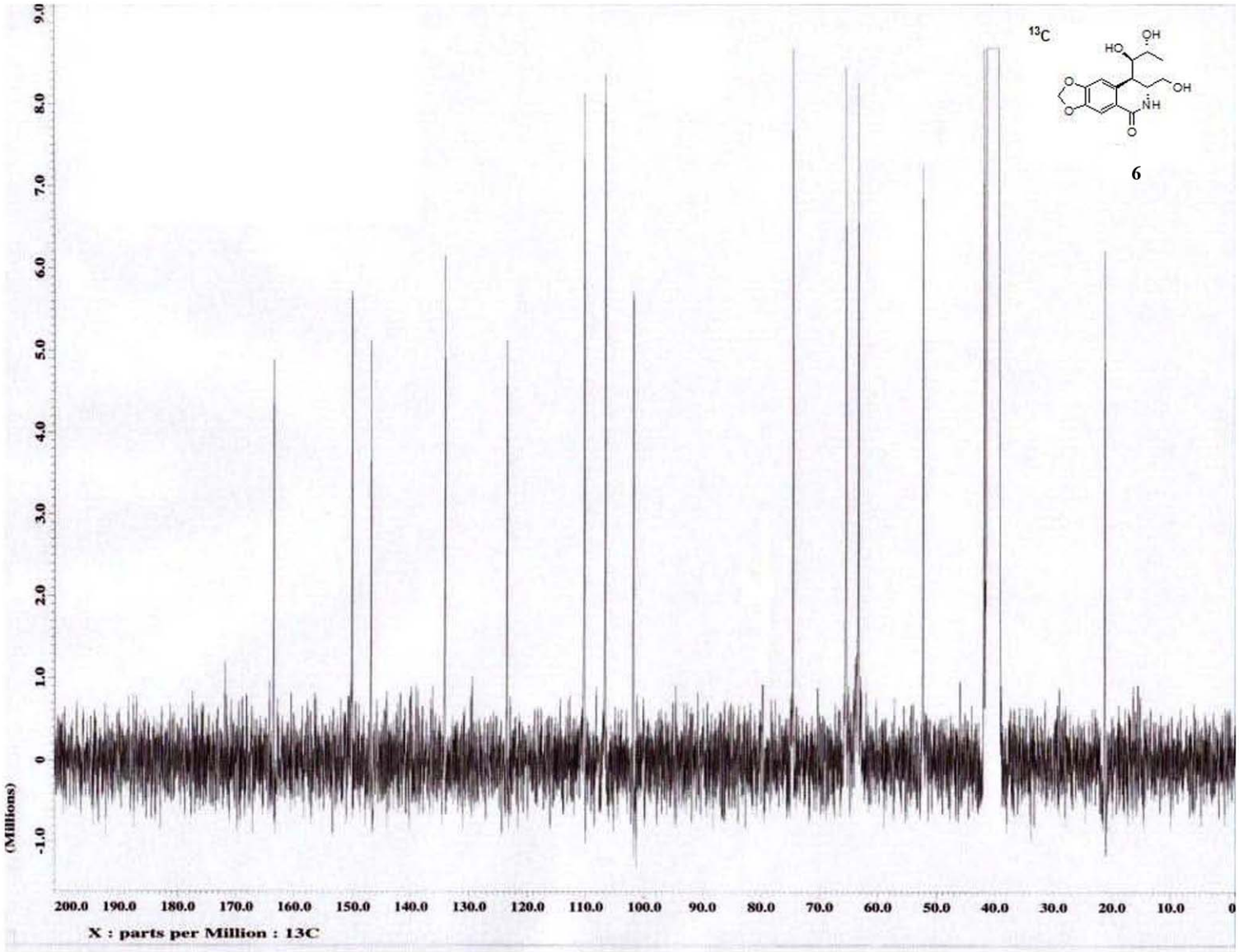

(Millions)
X : parts per Million : 1H
0.94289
1.0
2.1717
0.99113
1.07078
3.18904
0.98224
1.02242
0.97594
6.21367
2.99668
3.0192
1H
AcO
OAc
OAc
OMe
22


13C
OAc
AcO
OAc
O
O
N
OMe
22
(Millions)
13.0
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
-1.0
-2.0
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
X : parts per Million : 13C

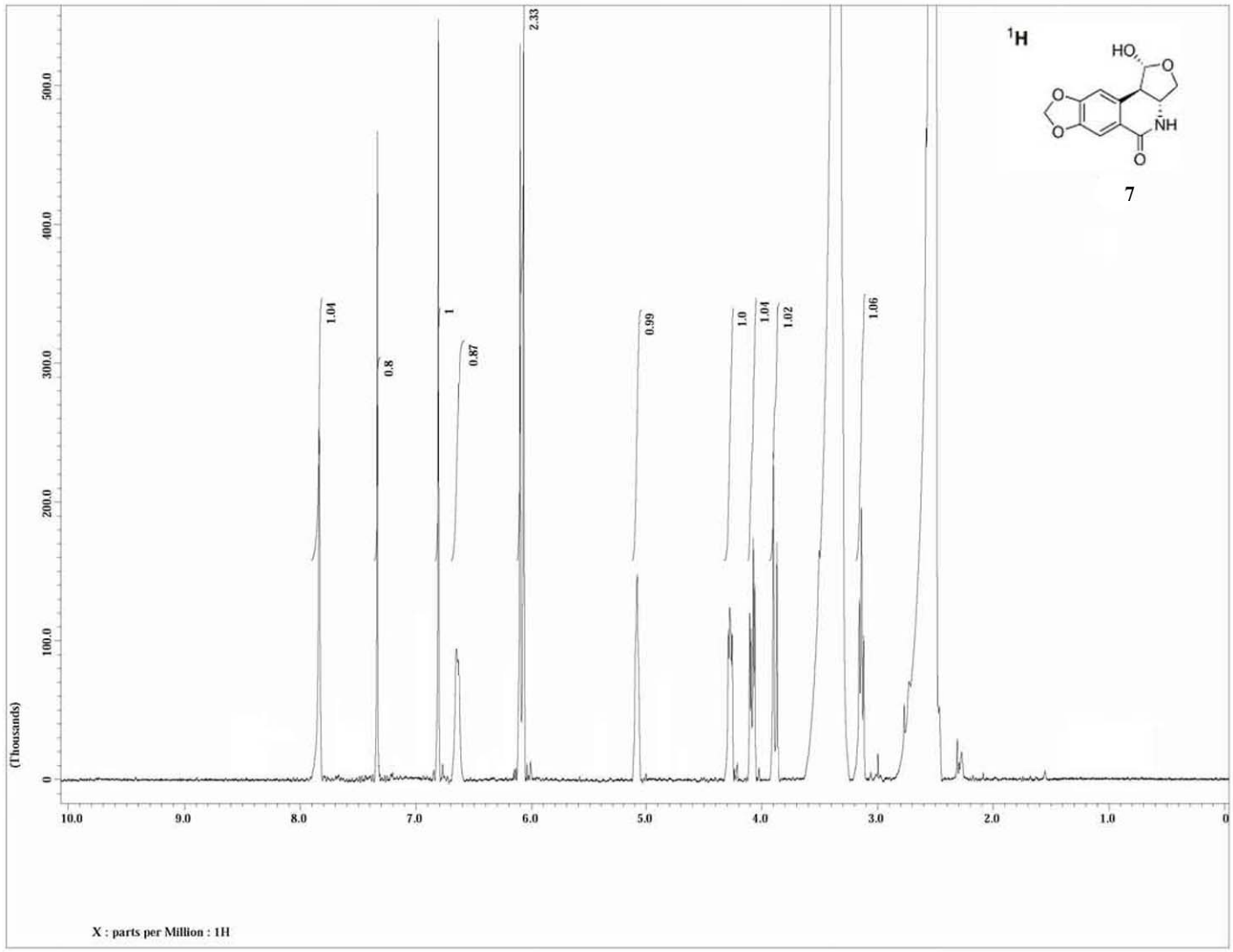

1H
OAc
AcO
OAc
O
O
NH
O
23
80a
6.0698
3.06949
3.05419
1.9919
1.11642
1.05602
1.11768
0.97378
0.93868
1.05471
0.96259
0.81015
(Millions)
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0

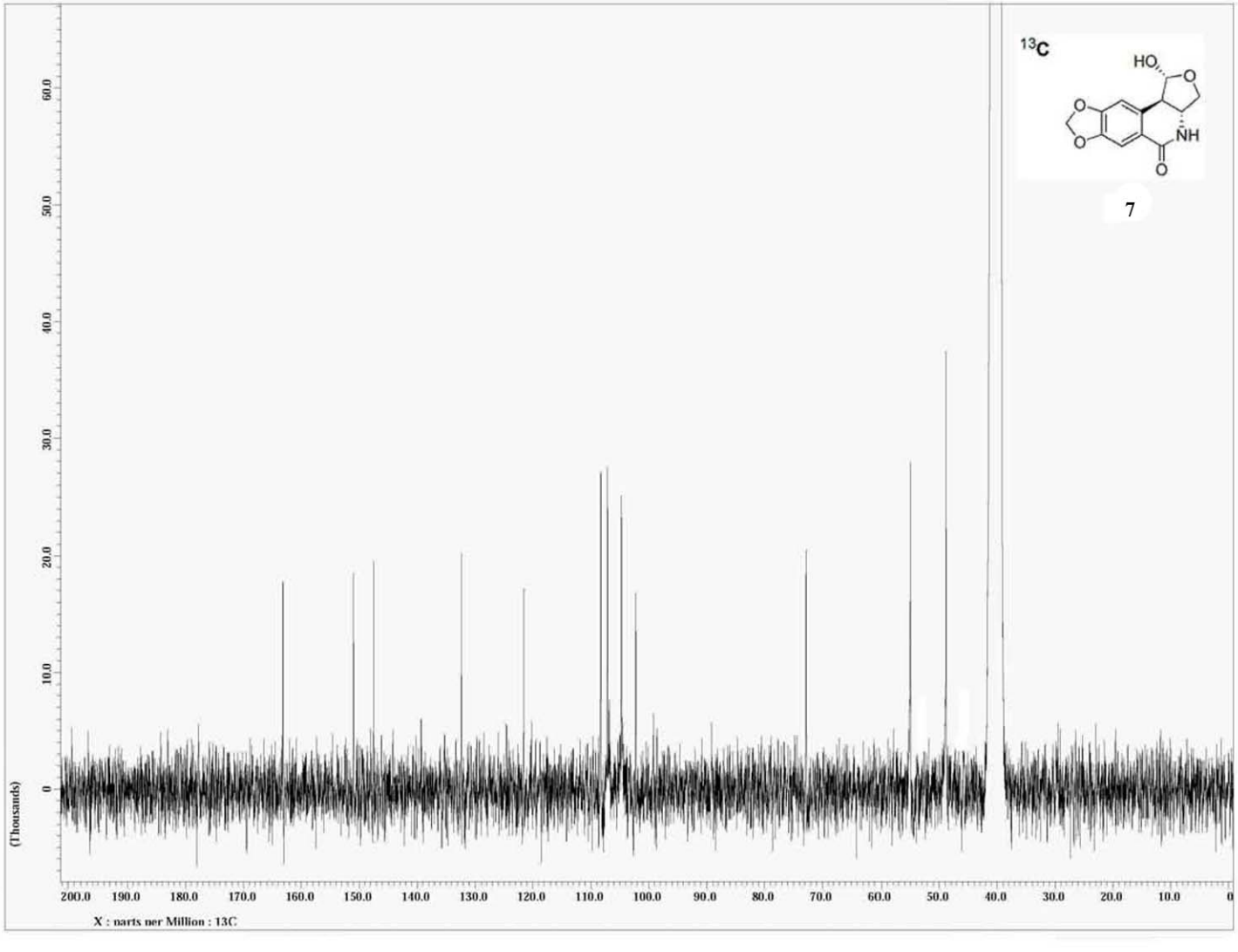

13C
OAc
AcO
OAc
O
O
NH
O
23
(Millions)
15.0
14.0
13.0
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
-1.0
-2.0
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
X : parts per Million : 13C

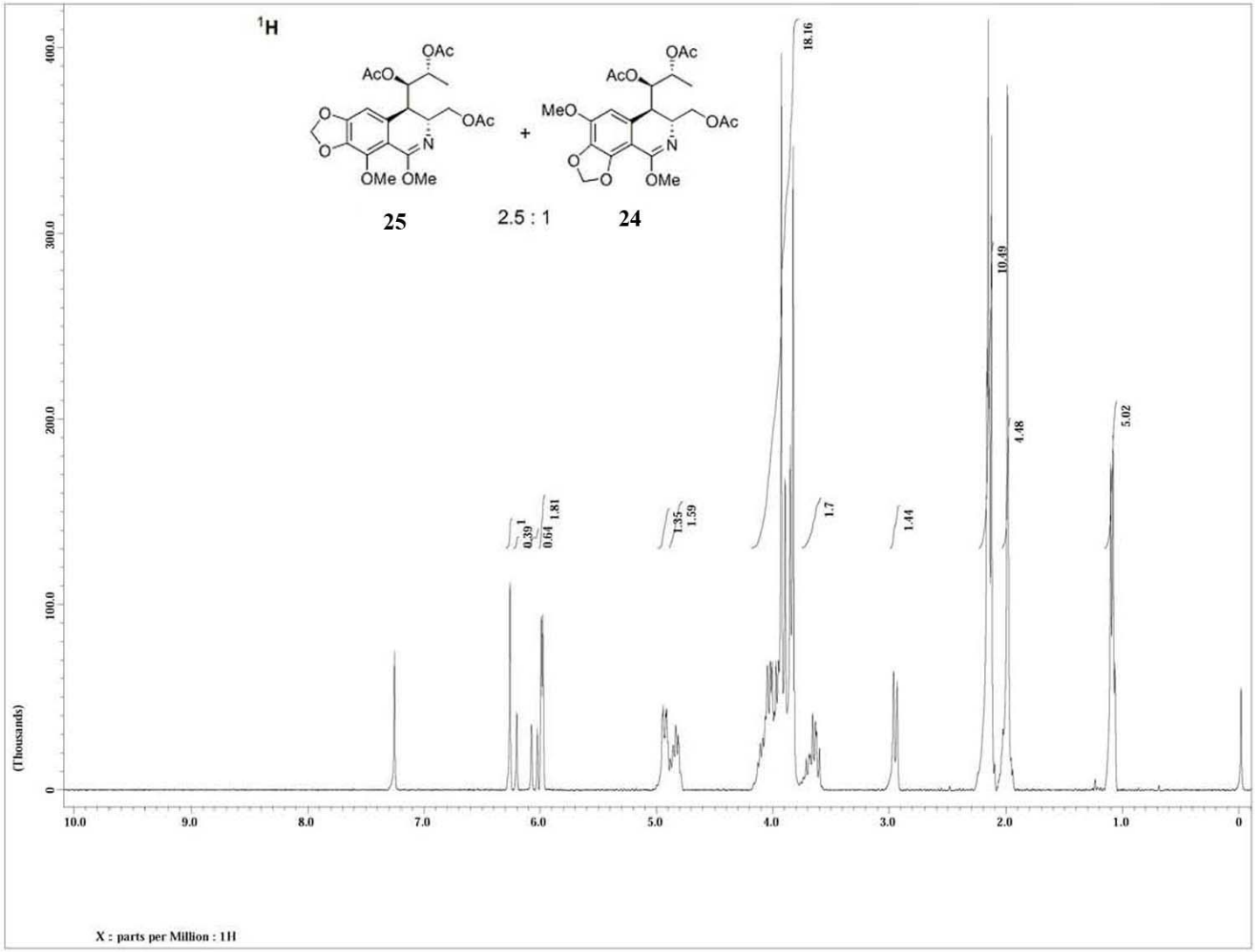

0.42025
0.22172
1.00267
0.95955
2
1.01143
1.74979
2.96872
2.90766
1H
HO
OH
OH
NH
O
6
(Millions)
4.0
3.0
2.0
1.0
0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
X : parts per Million : 1H

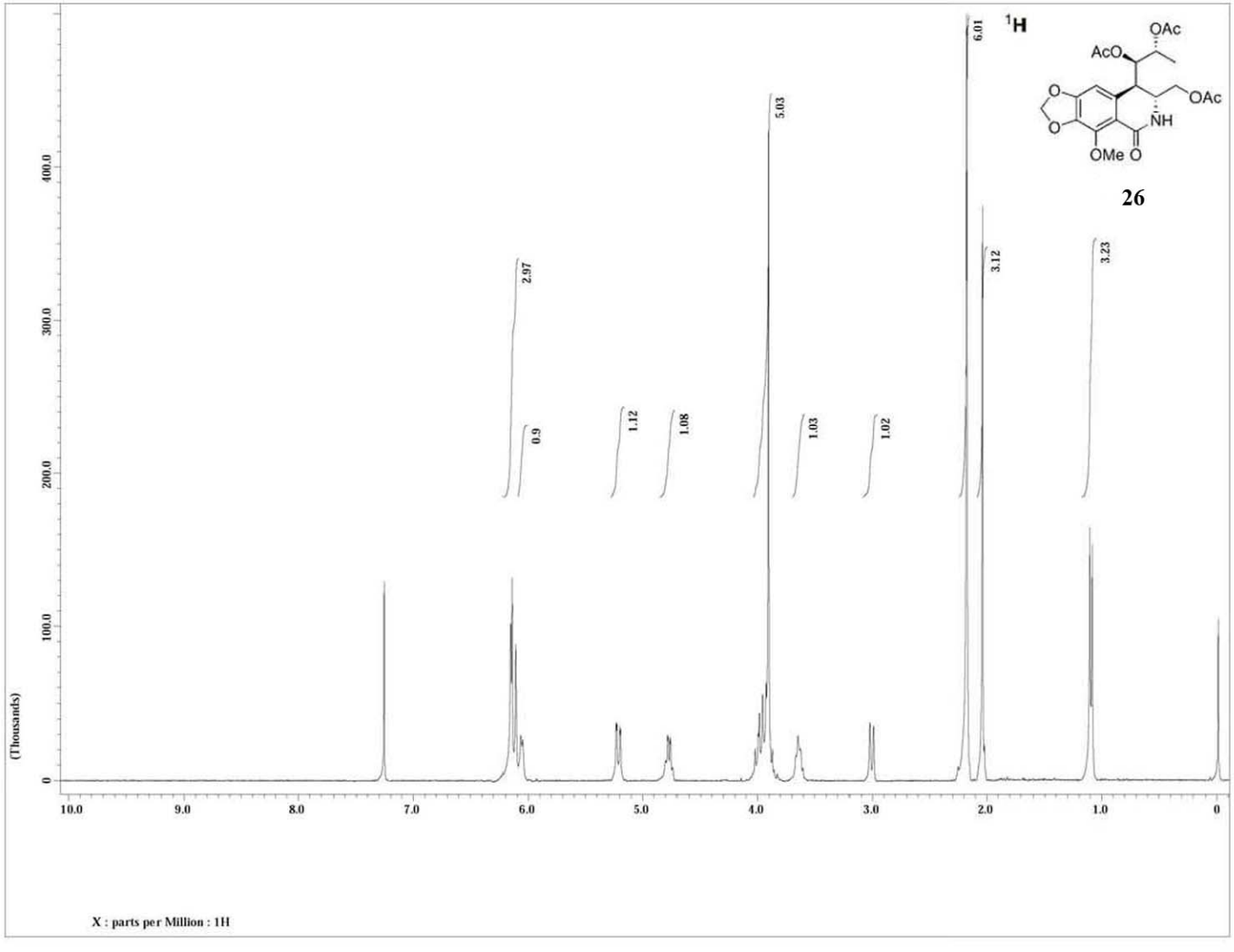

13C
OH
HO
OH
O
O
NH
O
(Millions)
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
-1.0
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
X : parts per Million : 13C

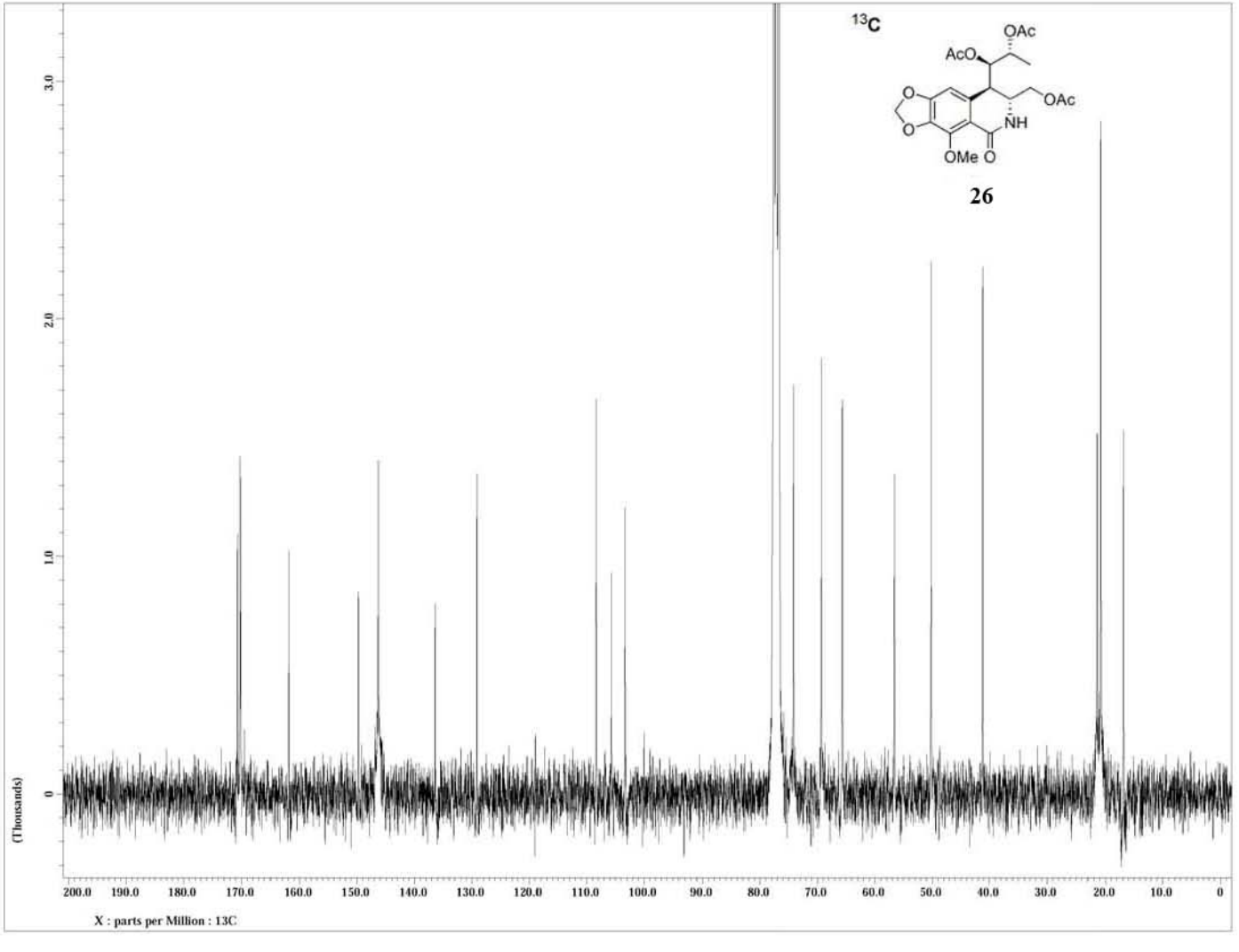
6

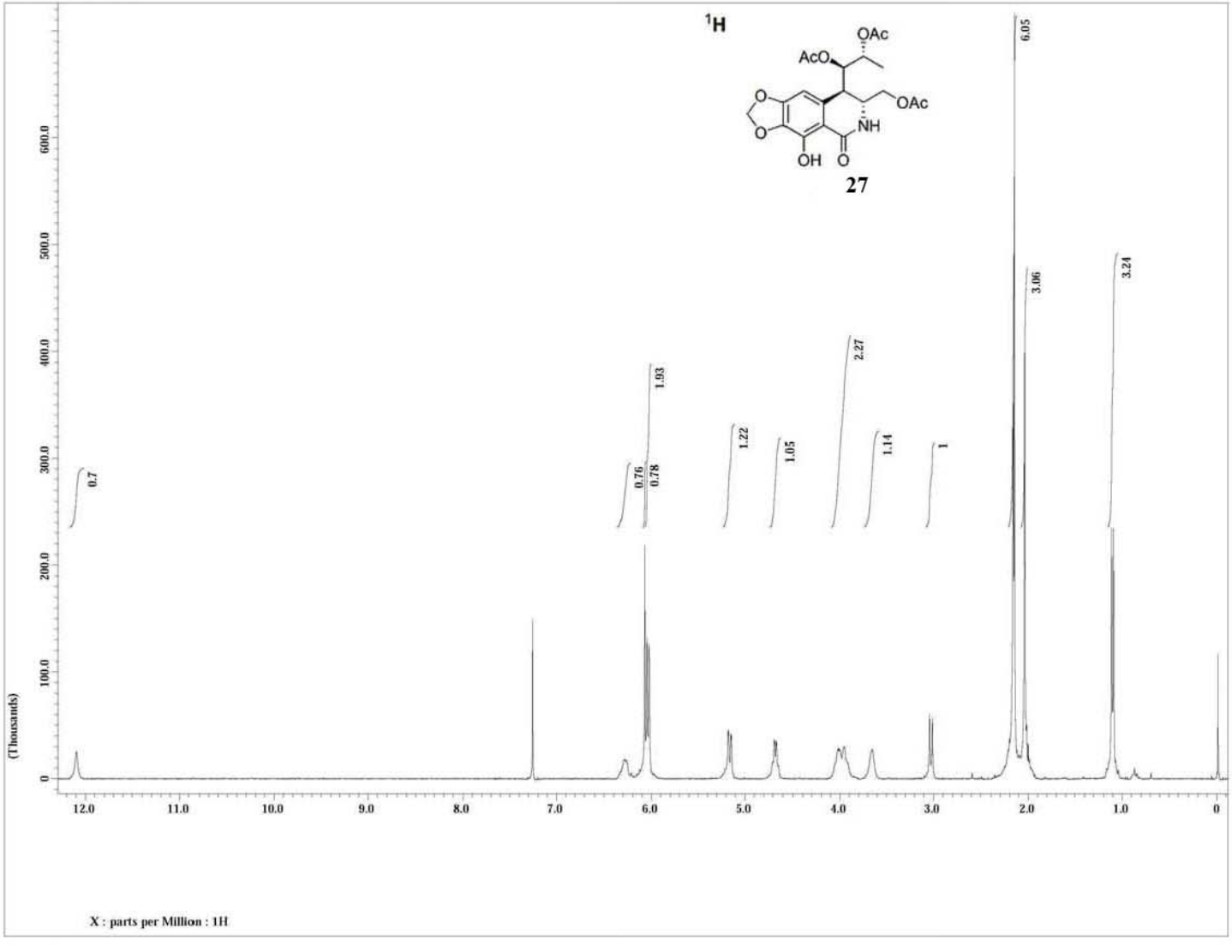

1H
HO
O
O
O
NH
O
7
(Thousands)
500.0
400.0
300.0
200.0
100.0
0
1.04
0.8
1
0.87
2.33
0.99
1.0
1.04
1.02
1.06
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
X : parts per Million : 1H

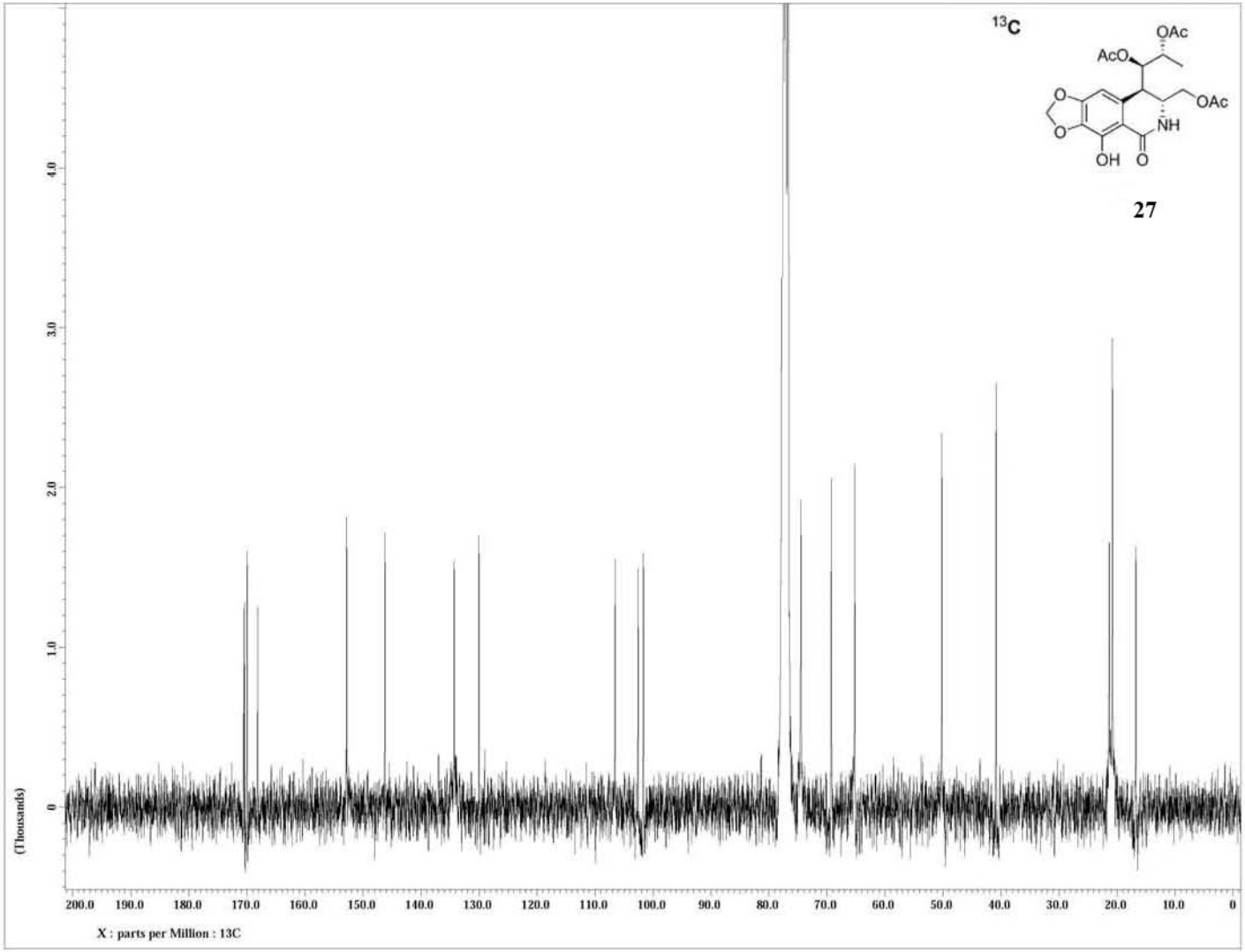

13C
HO
O
O
O
NH
O
7
60.0
50.0
40.0
30.0
20.0
10.0
0
(Thousands)
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
X : parts per Million : 13C

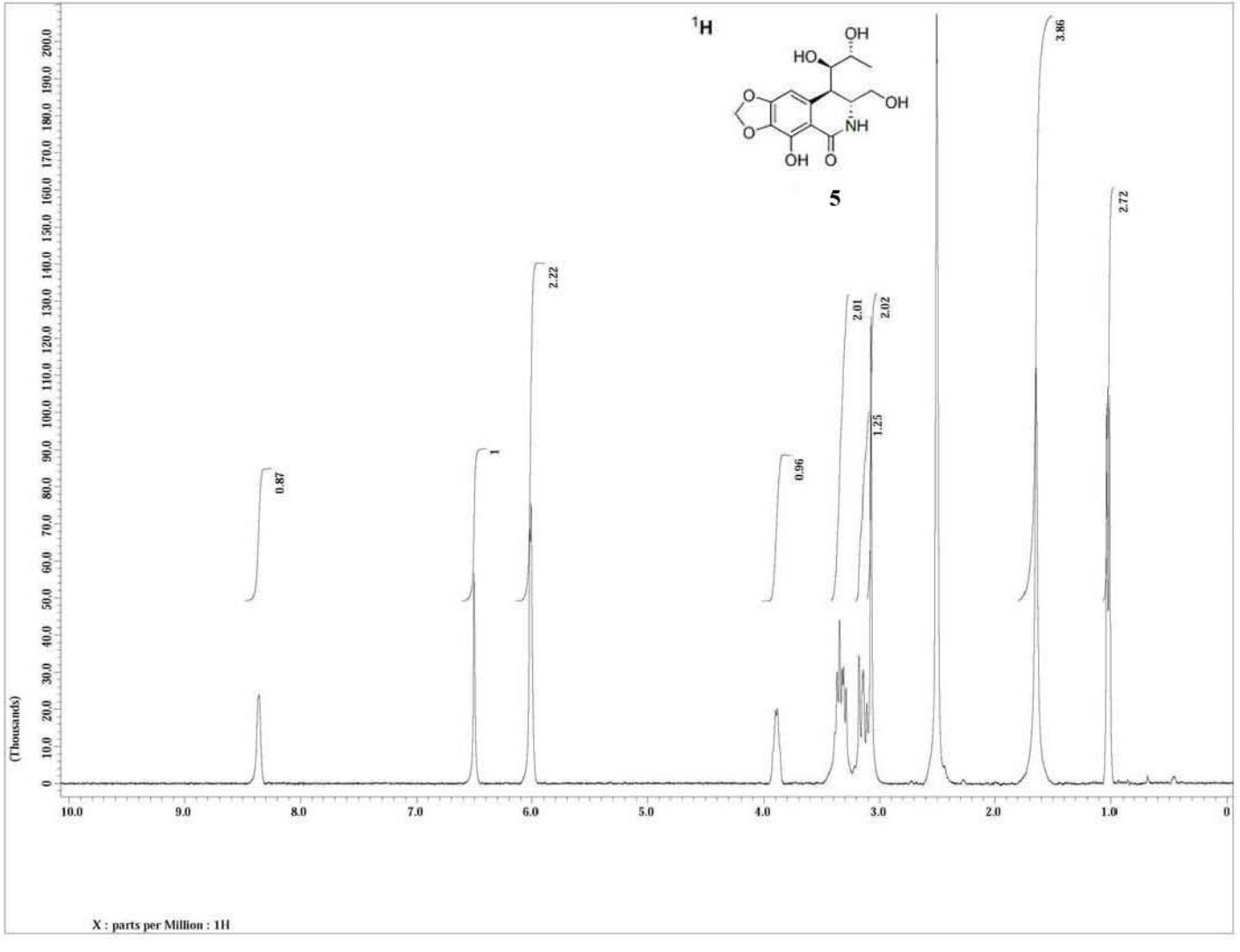

^{1}H
OAc
AcO
OAc
O
O
N
OMe OMe
25
2.5 : 1
MeO
OMe
24
18.16
10.49
4.48
5.02
1.81
1.59
1.7
1.44
(Thousands)
X : parts per Million : 1H

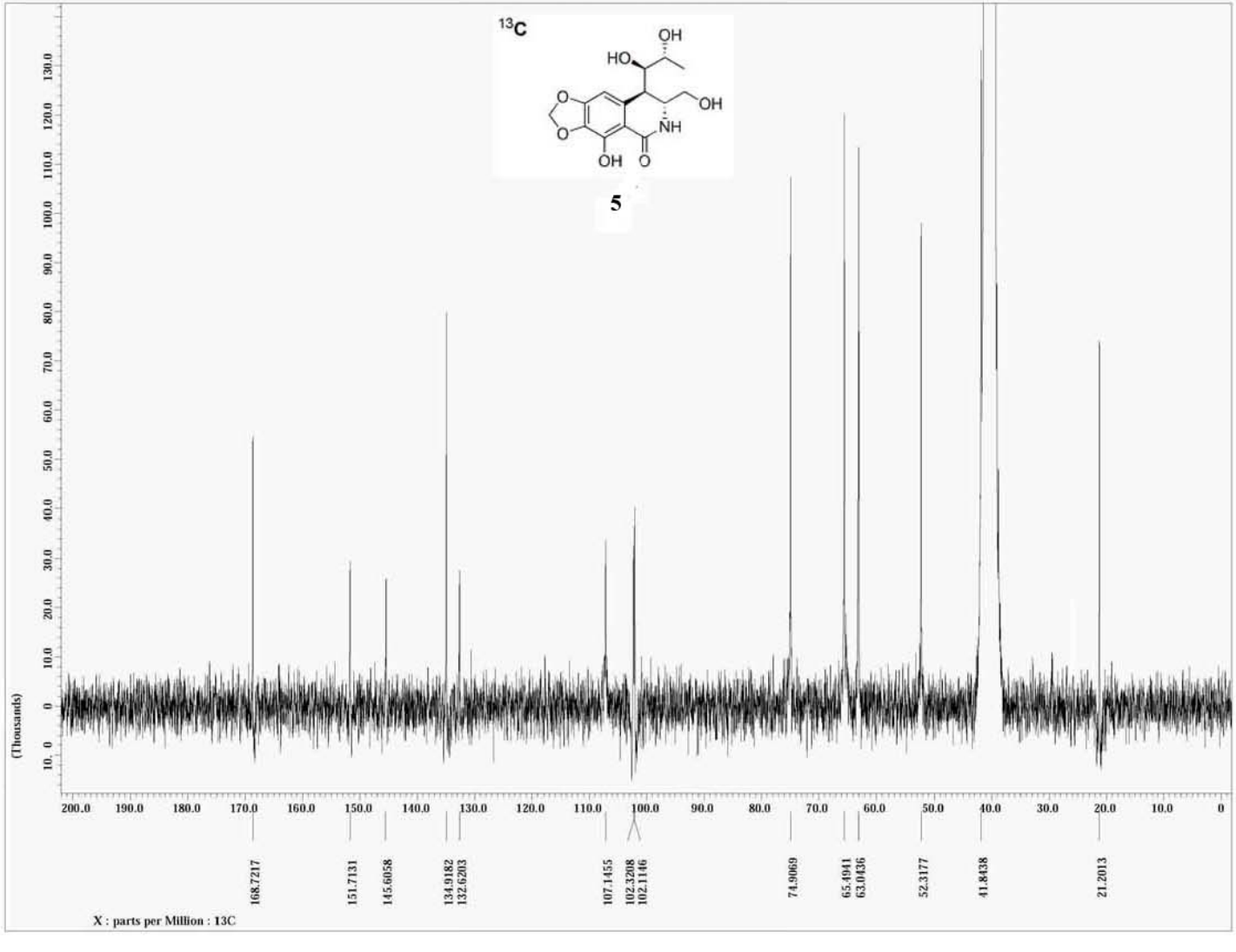

1H
OAc
AcO
OAc
O
O
NH
OMe O
26
6.01
5.03
3.23
3.12
2.97
0.9
1.12
1.08
1.03
1.02
(Thousands)
400.0
300.0
200.0
100.0
0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
X : parts per Million : 1H


13C
OAc
AcO
OAc
O
O
NH
OMe O
26
3.0
2.0
1.0
0
(Thousands)
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
X : parts per Million : 13C


1H
OAc
AcO
OAc
NH
OH
O
27
6.05
3.06
3.24
2.27
1.93
1.22
1.05
1.14
1
0.7
0.76
0.78
(Thousands)
600.0
500.0
400.0
300.0
200.0
100.0
0
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
X : parts per Million : 1H


13C
OAc
AcO
OAc
O
O
NH
OH
O
27
(Thousands)
4.0
3.0
2.0
1.0
0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
X : parts per Million : 13C


^{1}H
OH
HO
OH
O
O
NH
OH
O
5
3.86
2.72
2.22
2.01
2.02
1.25
1
0.96
0.87
(Thousands)
X : parts per Million : 1H


13C
OH
HO
OH
O
O
NH
OH
O
5
(Thousands)
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
10. 0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
168.7217
151.7131
145.6058
134.9182
132.6203
107.1455
102.3208
102.1146
74.9069
65.4941
63.0436
52.3177
41.8438
21.2013
X : parts per Million : 13C