Electronic Supplementary Material (ESI) for Chemical Communications

Confirming the existence of π-allyl-palladium intermediates during the reaction of *meta* photocycloadducts with palladium(II) compounds

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Supplementary Information

Contents

REAGENTS AND SOLVENTS

All starting materials and reagents were purchased from commercial sources and were used after verification of purity by NMR. The solvents used in reactions and all forms of chromatography were subjected to rotary evaporation before use to remove impurities, with the exception of halogenated solvents. Petroleum Ether used was from the fraction of boiling range 40-60 ºC. Unless stated all solvents used were not rigorously dried.

SPECTROSCOPY

^{1}H NMR spectra were recorded at either 500 or 600 MHz. NMR spectra performed at 600 MHz were recorded by Dr I. J. Day. Chemical shifts (δ) are quoted in ppm using deuterated chloroform ($CDCl_3$) as a reference (δ = 7.24 ppm). Coupling constants (*J*) are quoted in hertz (Hz) and the following abbreviations are used to describe the signal multiplicity: s=singlet, d=doublet, t=triplet, q=quartet, m=multiplet, br=broad. In some instances dm is used to describe the major d signal multiplicity, although the m signifies that other unresolved minor splitting signals are also present in the NMR spectrum. ^{13}C NMR spectra were recorded on the same spectrometers at 125 and 150 MHz respectively, using the same technique ($CDCl_3$ δ = 77.0 ppm). Full proton and carbon assignments have been made using a combination of COSY, DQF-COSY, multiplicity-edited HSQC and standard HMBC correlation spectroscopies. Stereochemical assignments were performed using rotating-frame Overhauser effect spectroscopy (ROESY). In instances where a signal's identity is ambiguous, a best guess is offered.

Infra-Red (IR) spectra were recorded with frequencies (ν_{max}) quoted in wavenumbers (cm^{-1}). Liquid samples were recorded using sodium chloride plates, while solids were recorded using a KBr disc.

High Resolution mass spectra data were captured by Dr. A. K. Abdul-Sada.

CHROMATOGRAPHY

Where reactions were monitored using TLC, the solvent system is indicated. Chromatography were executed using glass backed plates with a 250 µm layer of 60 Å silica gel with fluorescent indicator. Visualization was carried out using ultraviolet light (254 nm), combined with either potassium permanganate ($KMnO_4$) or vanillin (4-hydroxy-3-methoxybenzaldehyde) dips. Most flash column chromatography was run using silica 60 Å (particle size 35 – 70 µm). Solvent systems for separations are given for each procedure or product, where applicable.

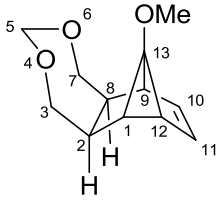
PHOTOCHEMISTRY

Irradiations were carried out in quartz immersion-well reactors fitted with 6 W or 16 W low pressure mercury vapor lamps or 125 W or 400 W medium pressure mercury vapor lamps. Oxygen free solvent for the irradiation experiments was simply obtained by passing a vigorous stream of nitrogen gas through a sintered glass tube into the solvent at rt. Experiments were conducted with gentle stirring of the reaction solution under an atmosphere of nitrogen and with cold-water cooling of the lamp and vessel contents throughout.

Irradiation of anisole and *cis*-4,7-dihydro-1,3-dioxepin[10(b),(c)]

A solution of anisole (6.8 g, 63 mmol) and *cis*-4,7-dihydro-1,3-dioxepin (6.3 g, 63 mmol) in cyclohexane (165 ml) was added to a quartz immersion well photoreactor and degassed by passing a stream of nitrogen through it for 10 min. The solution was irradiated with 254 nm UV light for 18 hours using a 6 W low-pressure mercury vapour lamp. The solvent was removed in vacuo and the residue subjected to column chromatography (silica, petrol/ether 5:1) to obtain the *exo*-isomer **9** (0.70 g, 5.4 %) and *endo*-isomer **10** (0.50 g, 3.8 %) as light green oils.

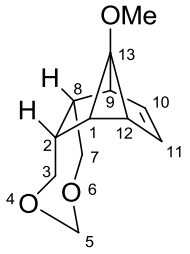
rac-(1*S*,2*S*,8*R*,9*S*,12*R*,13*R*)-13-Methoxy-4,6-dioxatetracyclo[7.3.1.0.^{2,8}0,^{12,13}]tridec-10-ene **9**
(The *exo* adduct)



^{1}H NMR (500 MHz, $CDCl_3$): δ 5.64 (1H, dd, *J* 2.6, 5.7, H-11), 5.58 (1H, ddd, *J* 1.4, 2.7, 5.7, H-10), 4.7 (2H, s, H-5), 4.04 (1H, t, *J* 11.7, H-7), 3.97 (1H, dd, *J* 2.7, 12.4, H-3), 3.90 (1H, dd, *J* 3.4,12.4, H-3), 3.72 (1H, ddd, *J* 0.9, 4.5, 12.0, H-7), 3.30 (3H, s, -OCH_3), 2.79 (1H, d, *J* 2.7, H-9), 2.22 (1H, ddd, *J* 1.4, 2.4, 8.4, H-12), 2.21 (1H, m, H-8), 2.09 (1H, dd, *J* 1.6, 8.4, H-1), 1.92 (1H, m, H-2); ^{13}C NMR(125 MHz, $CDCl_3$): δ 131.89 (C-10), 127.43 (C-11), 95.56 (C-5), 89.94 (C-13), 67.63 (C-3), 66.62 (C-7), 56.30 (-O***C***H_3), 56.04 (C-12), 52.74 (C-9), 40.71 (C-2), 37.37 (C-1), 36.45 (C-8).

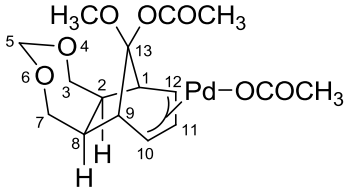
Note: *The correct NMR data for compound **10** in toluene-d_8 have been reported as a corrigendum*[10(c)]

rac-(1*S*,2*R*,8*S*,9*S*,12*R*,13*R*)-13-Methoxy-4,6-dioxatetracyclo[7.3.1.0.^{2,8}0,^{12,13}]tridec-10-ene **10**
(The *endo* adduct)



^{1}H NMR (500 MHz, $CDCl_3$): δ 5.83 (1H, ddd, *J* 1.4, 2.6, 5.8, H-10), 5.80 (1H, dd, *J* 2.4, 5.8, H-11), 5.02 (1H, d, *J* 6.6, H-5), 4.46 (1H, d, *J* 6.6, H-5), 4.02 (1H, dd, *J* 5.7, 12.5, H-7), 3.79 (1H, dd, *J* 11.2, 12.2, H-3), 3.69 (1H, dd, *J* 3.7, 12.2, H-3), 3.57 (1H, dd, *J* 9.8, 12.5, H-7), 3.39 (3H, s, -OCH_3), 3.19-3.26 (1H, m, H-8), 3.08-3.16 (2H, m, H-2 + H-9), 2.15 (1H, dd, *J* 6.6, 8.4, H-11), 2.06 (1H, ddd, *J* 1.2, 2.4, 8.4, H-12); ^{13}C NMR(125 MHz, $CDCl_3$): δ 133.90 (C-10), 130.94 (C-11), 99.75 (C-5), 91.97 (C-13), 71.32 (C-7), 70.31 (C-3), 56.38 (-O***C***H_3), 54.19 (C-8), 53.21 (C-9), 47.60 (C-2), 37.67 (C-1), 35.31 (C-1).

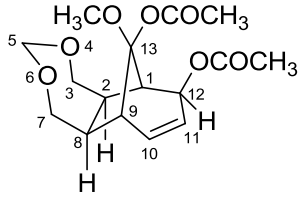
exo π-Allyl palladium intermediate **11**



A solution of *exo* photoadduct **9** (22 mg, 0.11 mmol) and $Pd(OAc)_2$ (31 mg, 0.14 mmol) in acetonitrile (3 ml) was added together in a round bottomed flask and left to react for 4 minutes. The solvent was removed *in vacuo* and to obtain the crude *exo*-π-allyl palladium complex **11** as a dark green residue, which was dissolved in $CDCl_3$ in order to analyze **11** using NMR spectroscopy before significant degradation could occur.

^{1}H NMR (500 MHz, $CDCl_3$): δ 6.54 (1H, t, *J* 5.8, H-11), 5.23 (2H, t, *J* 5.8, H-10/12), 5.01 (1H, d, *J* 6.8, H-5), 4.38 (1H, d, *J* 6.8, H-5), 3.99 (2H, d, *J* 11.7, H-3/7), 3.83-3.88 (2H, m, H-3/7), 3.29 (3H, s, -OCH_3), 2.98-3.03 (2H, br s, H-1/9), 2.71 (2H, d, *J* 10.4, H-2/8), 2.53 (3H, s, (C-13)-OAc), 1.81 (3H, s, -PdOAc); ^{13}C NMR(125 MHz, $CDCl_3$): δ 182.57 (-PdO***C***(O)CH_3), 169.13 ((C-13)-O***C***(O)CH_3), 111.04 (C-13), 99.74 (C-5), 95.96 (C-11), 73.58 (C-10/12), 71.73 (C-3/7), 49.72 (-O***C***H_3), 47.36 (C-2/8), 44.97 (C-1/9), 23.12 (-PdOC(O)***C***H_3), 21.96 ((C-13)-OC(O)***C***H_3); IR 1570, 1749, 2930 cm^{-1}.

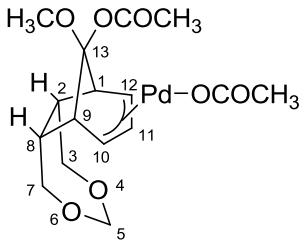
rac-(1*R*,2*R*,8*S*,9*R*,12*S*,13*R*)-12,13-Diacetoxy-13-methoxy-4,6-dioxatricyclo[7.3.1.$0^{2,8}$]-tridec-10-ene **12**.



A solution of *exo* photoadduct **9** (70 mg, 0.33 mmol) and $Pd(OAc)_2$ (90 mg, 0.40 mmol) in deuterated acetonitrile (5 ml) was added together in an NMR tube and left to react for 2 hours. The solution changed colour from clear orange to dark brown with a fine black precipitate of palladium metal coating the internal surface of the tube. The solvent was removed in vacuo and the residue subjected to column chromatography to obtain the *exo* bis-acetate **12** (32 mg, 30%) as fine white crystals.

R_f 0.25 (ether/petrol 3:1); m.p. 143.1-145.9 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 6.16 (1H, ddd, *J* 1.0, 7.2, 9.4, H-10), 5.56 (1H, ddd, *J* 1.4, 3.9, 9.4, H-11), 5.20-5.22 (1H, m, H-12), 5.03 (1H, d, *J* 7.1, H-5), 4.43 (1H, d, *J* 7.1, H-5), 4.10 (1H, dd, *J* 5.3, 12.3, H-3/7), 3.85-3.95 (3H, m, H-3/7), 3.24 (3H, s, -OCH_3), 2.76 (1H, dd, *J* 2.2, 7.2, H-9), 2.64-2.66 (1H, br s, H-1), 2.52 (1H, ddd, *J* 6.5, 9.7, 9.7, H-2), 2.40 (1H, dddd, *J* 1.0, 5.2, 9.4, 12.3, H-8), 2.02 (3H, s, -OAc), 2.00 (3H, s, -OAc); ^{13}C NMR(125 MHz, $CDCl_3$): δ 170.03 (-***C***OCH_3), 169.00 (-***C***OCH_3), 134.83 (C-10), 122.65 (C-11), 107.11 (C-13), 99.75 (C-5), 75.36 (C-12), 73.14 (C-7), 72.77 (C-3), 49.27 (-O***C***H_3), 47.56 (C-8), 44.77 (C-1), 43.55 (C-2), 42.70 (C-9), 21.49 (-CO***C***H_3), 21.05 (-CO***C***H_3); IR 1728, 1752, 2932,cm^{-1}; HRMS (ESI) *m/z* calcd $C_{16}H_{22}NaO_7$ $[M + Na]^+$ 349.1263, found 349.1257.

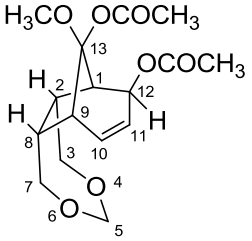
endo π-Allyl palladium intermediate **13**



A solution of *endo* photoadduct **10** (40 mg, 0.19 mmol) and $Pd(OAc)_2$ (47 mg, 0.21 mmol) in acetonitrile (3 ml) was added together in a round bottomed flask and left to react for 4 minutes. The solvent was removed *in vacuo* and to obtain the crude *endo*-π-allyl palladium complex as a dark green residue, which was dissolved in $CDCl_3$ in order to analyze **13** using NMR spectroscopy before significant degradation could occur.

^{1}H NMR (500 MHz, $CDCl_3$): δ 6.67 (1H, t, *J* 5.8, H-11), 5.11 (1H, d, *J* 7.3, H-5), 4.85 (2H, 7, *J* 5.8, H-10/12), 4.49 (1H, d, *J* 7.3, H-4), 4.22 (2H, dm, *J* 12.0, H-3/7), 3.87-3.95 (2H, m, H-3/7), 3.38 (3H, s, -OCH_3), 3.26-3.30 (2H, m, H-1/9), 2.68-2.74 (2H, m, H-2/8), 2.50 (3H, s, (C-13)-OAc), 1.84 (3H, s, -PdOAc); ^{13}C NMR(125 MHz, $CDCl_3$): δ 182.61 (-PdO***C***(O)CH_3), 169.44 ((C-13)-O***C***(O)CH_3), 111.82 (C-13), 100.15 (C-5), 96.45 (C-11), 72.61 (C-3/7), 69.73 (C-10/12), 49.18 (-O***C***H_3), 44.94 (C-2/8), 44.66 (C-1/9), 23.17 (-PdOC(O)***C***H_3), 22.08 ((C-13)-OC(O)***C***H_3).

rac-(1*R*,2*S*,8*R*,9*R*,12*S*,13*R*)-12,13-Diacetoxy-13-methoxy-4,6-dioxatricyclo[7.3.1.$0^{2,8}$]-tridec-10-ene **14**.

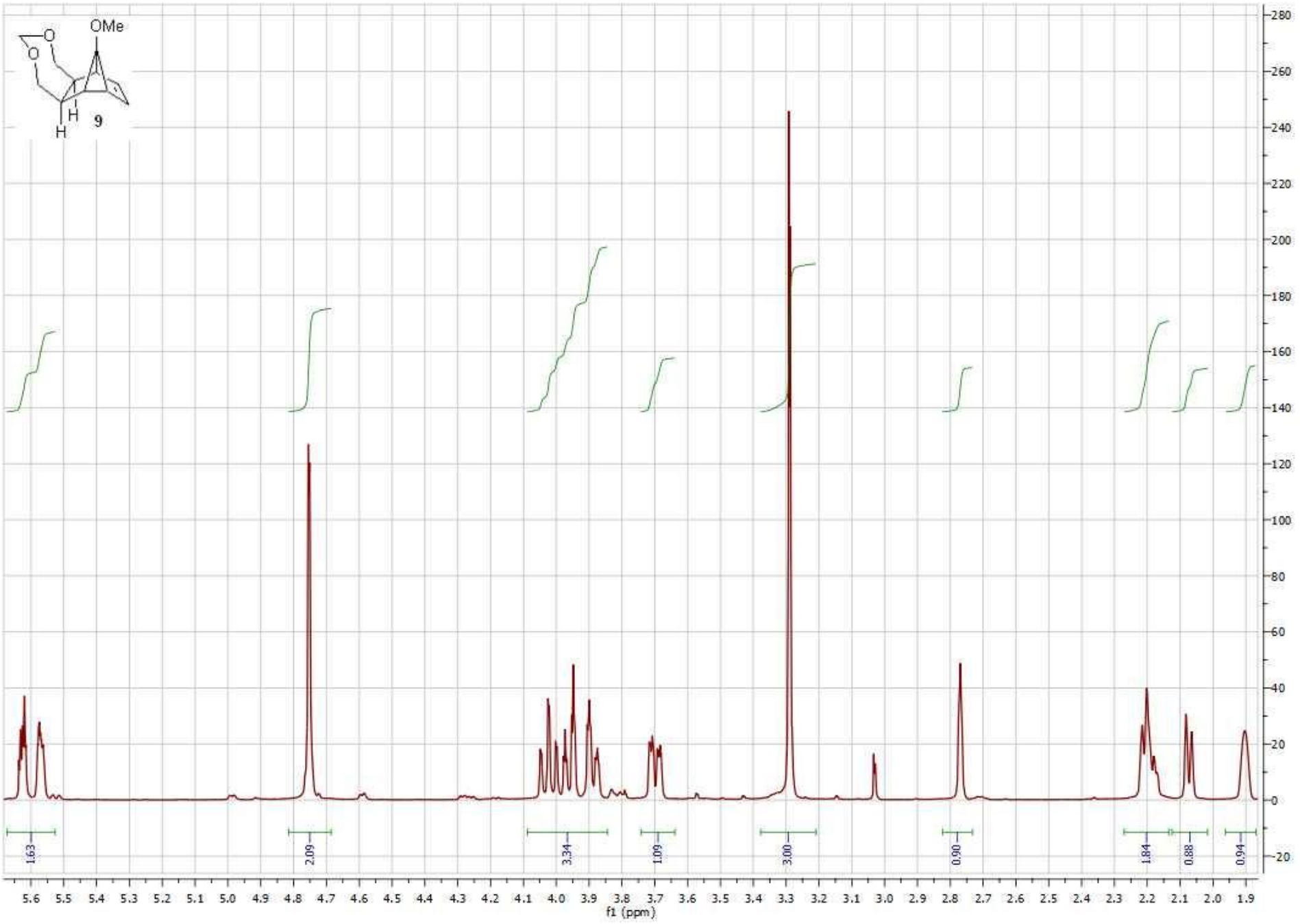


A solution of *endo* photoadduct **10** (50 mg, 0.24 mmol) and $Pd(OAc)_2$ (59 mg, 0.26 mmol) in acetonitrile (2 ml) was left to react for 2 hours. The solution changed colour from clear orange to dark brown with a fine black precipitate of palladium metal coating the internal surface of the tube. The solvent was removed in vacuo and the residue subjected to column chromatography to obtain the *endo* bis-acetate **14** (25 mg, 32%) as a yellow oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ 5.96 (1H, dd, *J* 7.2, 9.6, H-10), 5.77 (1H, ddd, *J* 1.6, 3.8, 9.6, H-11), 5.23-5.25 (1H, m, H-12), 5.07 (1H, d, *J* 7.3, H-5), 4.40 (1H, d, *J* 7.3, H-5), 4.26 (1H, dd, *J* 4.3, 12.1, H-3), 4.06 (1H, dd, *J* 4.6, 12.6, H-7), 3.62 (1H, t, *J* 12.6, H-7), 3.58 (1H, dd, *J* 12.1, 12.6, H-3), 3.31 (3H, s, -OCH_3), 3.12 (1H, dm, *J* 7.4, H-1), 2.96-3.02 (2H, m, H-2/9), 2.74 (1H, dddd, *J* 4.5, 5.6, 10.3, 12.5, H-8), 2.02 (3H, s, -OAc), 2.00 (3H, s, -OAc); ^{13}C NMR(125 MHz, $CDCl_3$): δ 170.05 (-***C***OCH_3), 169.22 (-***C***OCH_3), 132.53 (C-10), 125.48 (C-11), 107.11 (C-13), 100.03 (C-5), 71.67 (C-3/7), 71.63 (C-3/7), 69.65 (C-12), 49.13 (-O***C***H_3), 44.40 (C-1), 43.40 (C-8), 41.52 (C-9), 40.78 (C-2), 21.65 (-CO***C***H_3), 21.04 (-CO***C***H_3); HRMS (ESI) *m/z* calcd $C_{16}H_{22}NaO_7$ $[M + Na]^+$ 349.1263, found 349.1263.

References

10 (b) Penkett, C. S.; Sims, R. O.; Byrne, P. W.; Kingston, L.; French, R.; Dray, L..; Berritt, S.; Lai, J.; Avent, A. G.; Hitchcock, P. B. *Tetrahedron* **2006**, *62*, .3423. (b) Penkett, C. S.; Sims, R. O.; Byrne, P. W.; Kingston, L.; French, R.; Dray, L..; Berritt, S.; Lai, J.; Avent, A. G.; Hitchcock, P. B. *Tetrahedron* **2010**, *66*, .8223.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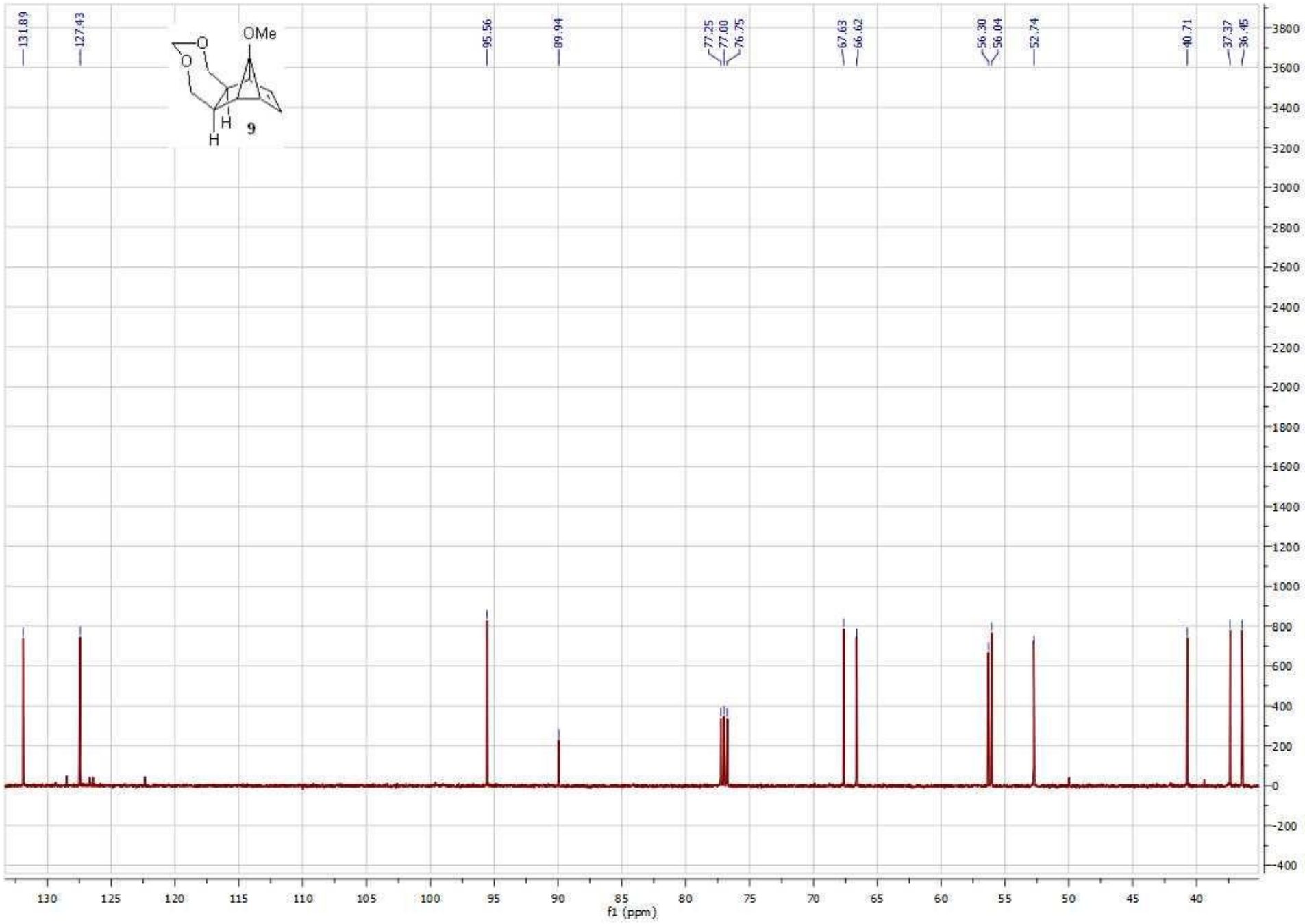
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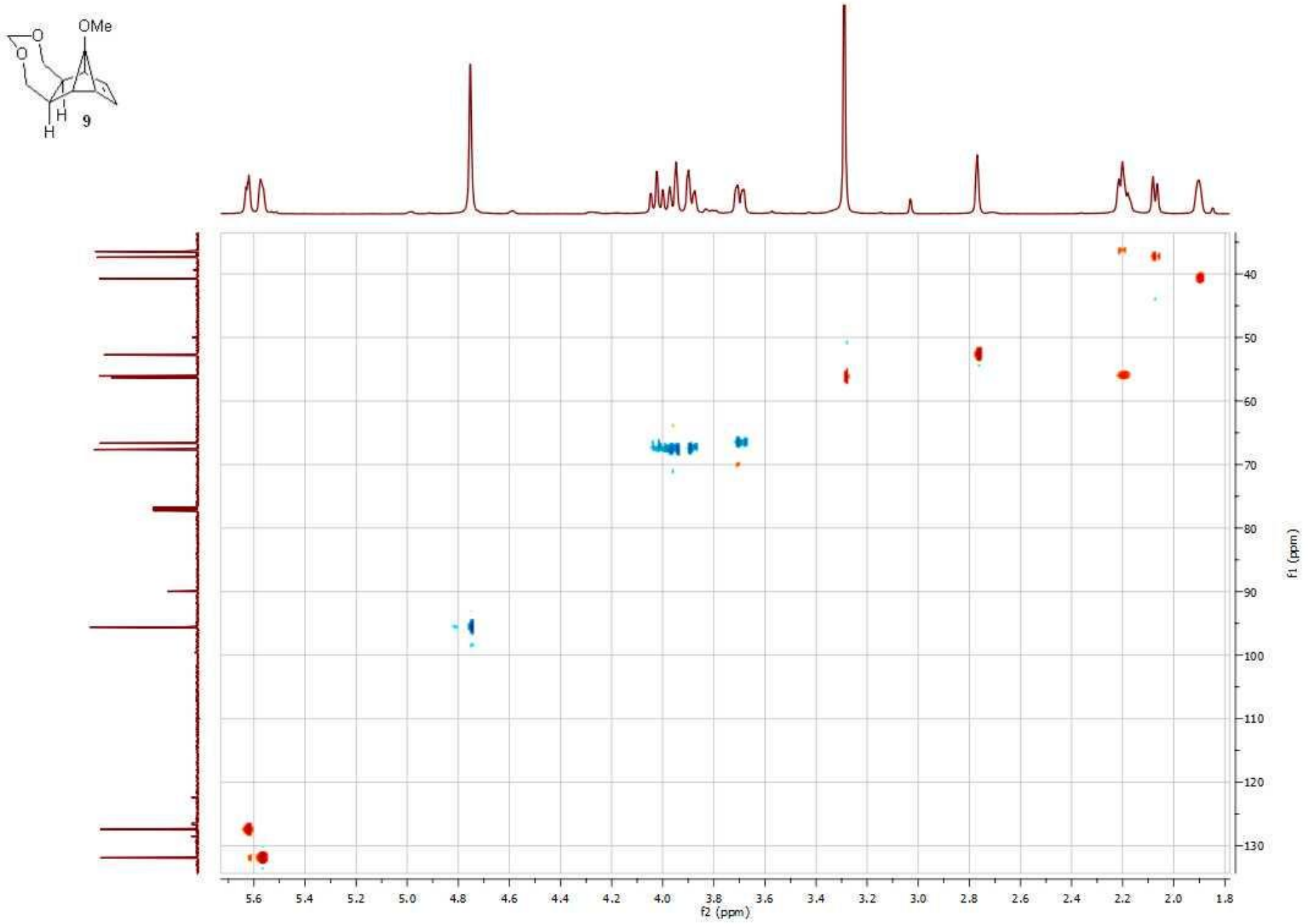
f1 (ppm)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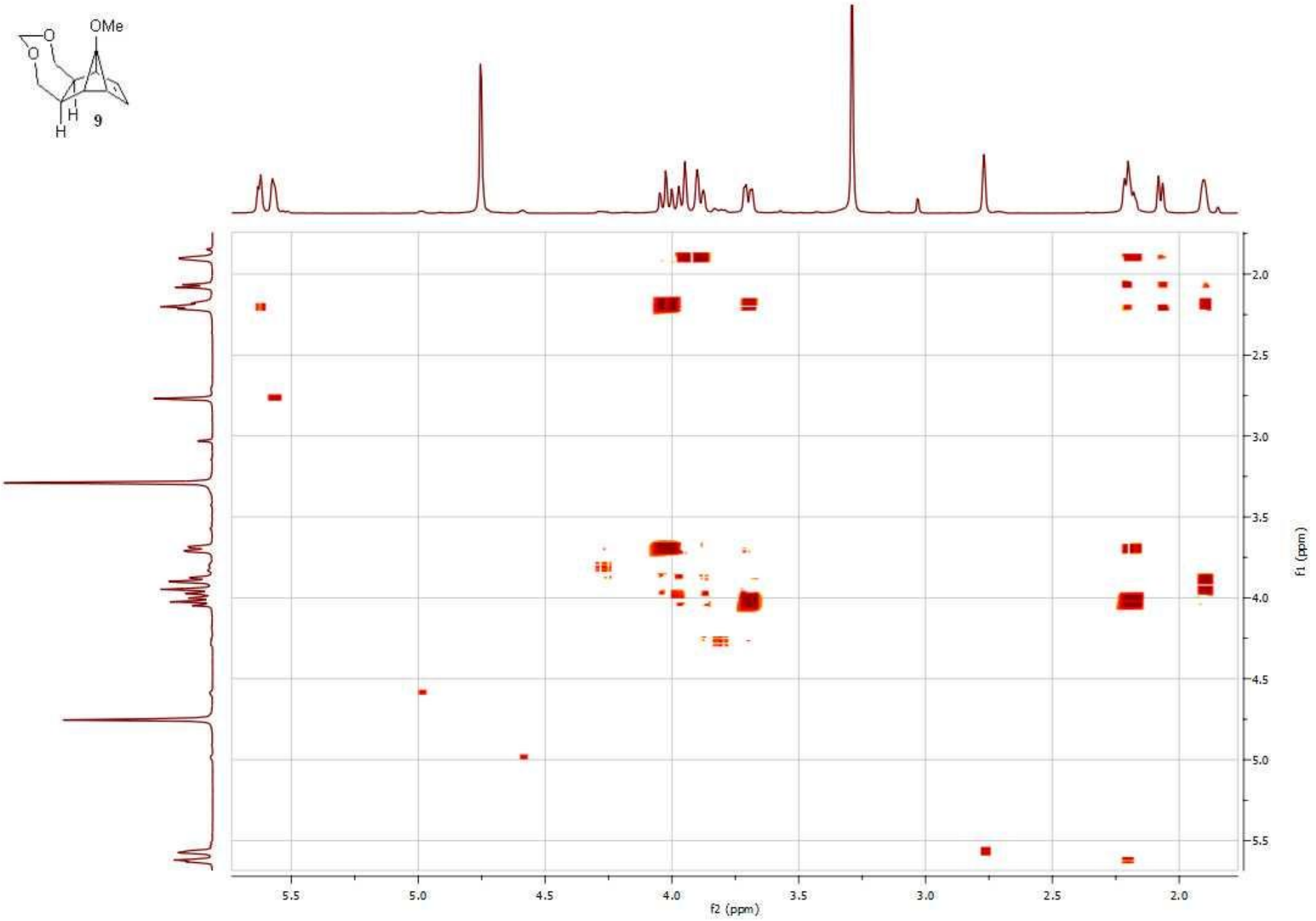
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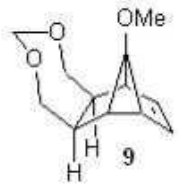
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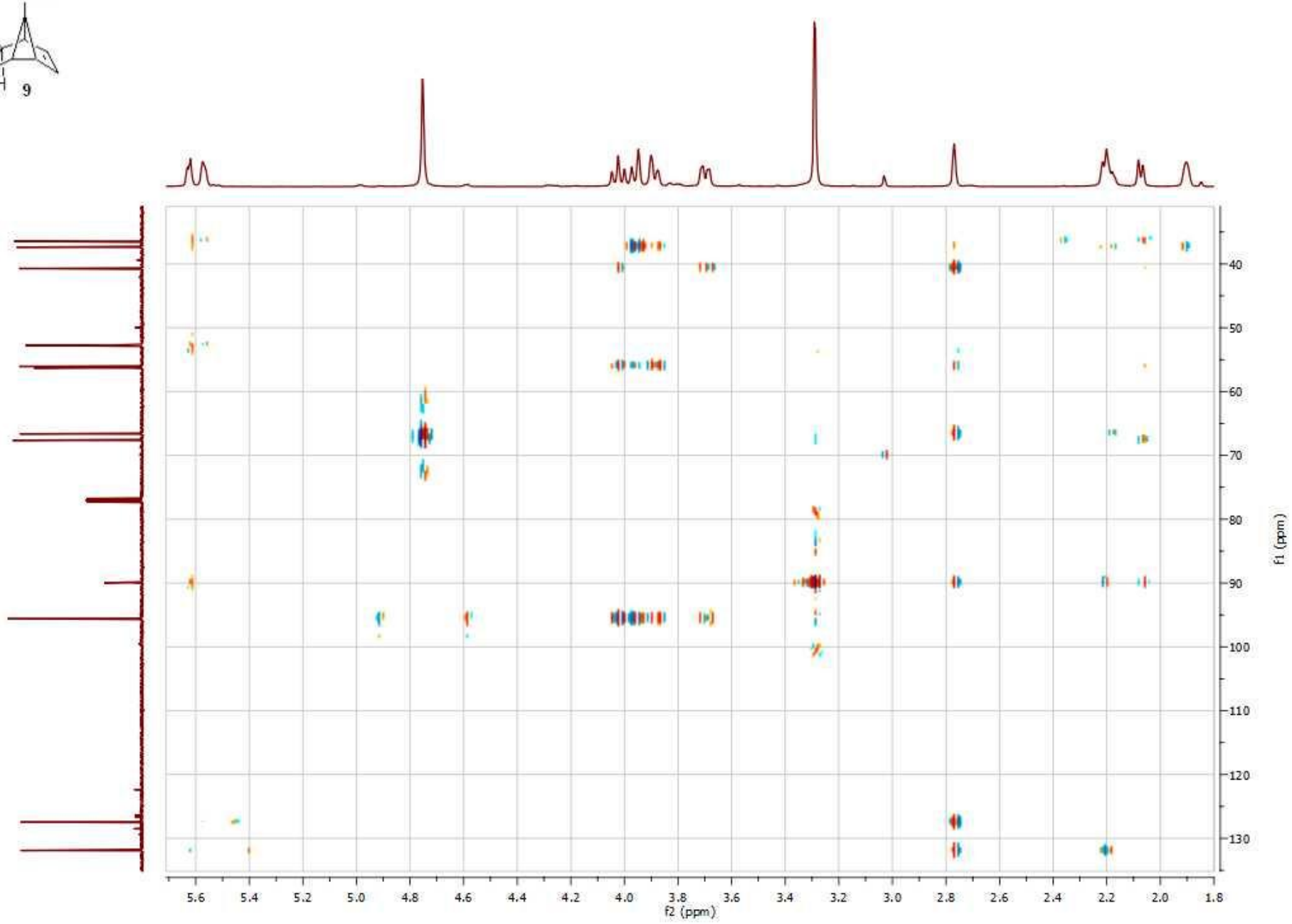
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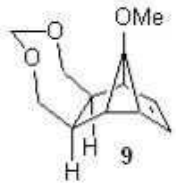

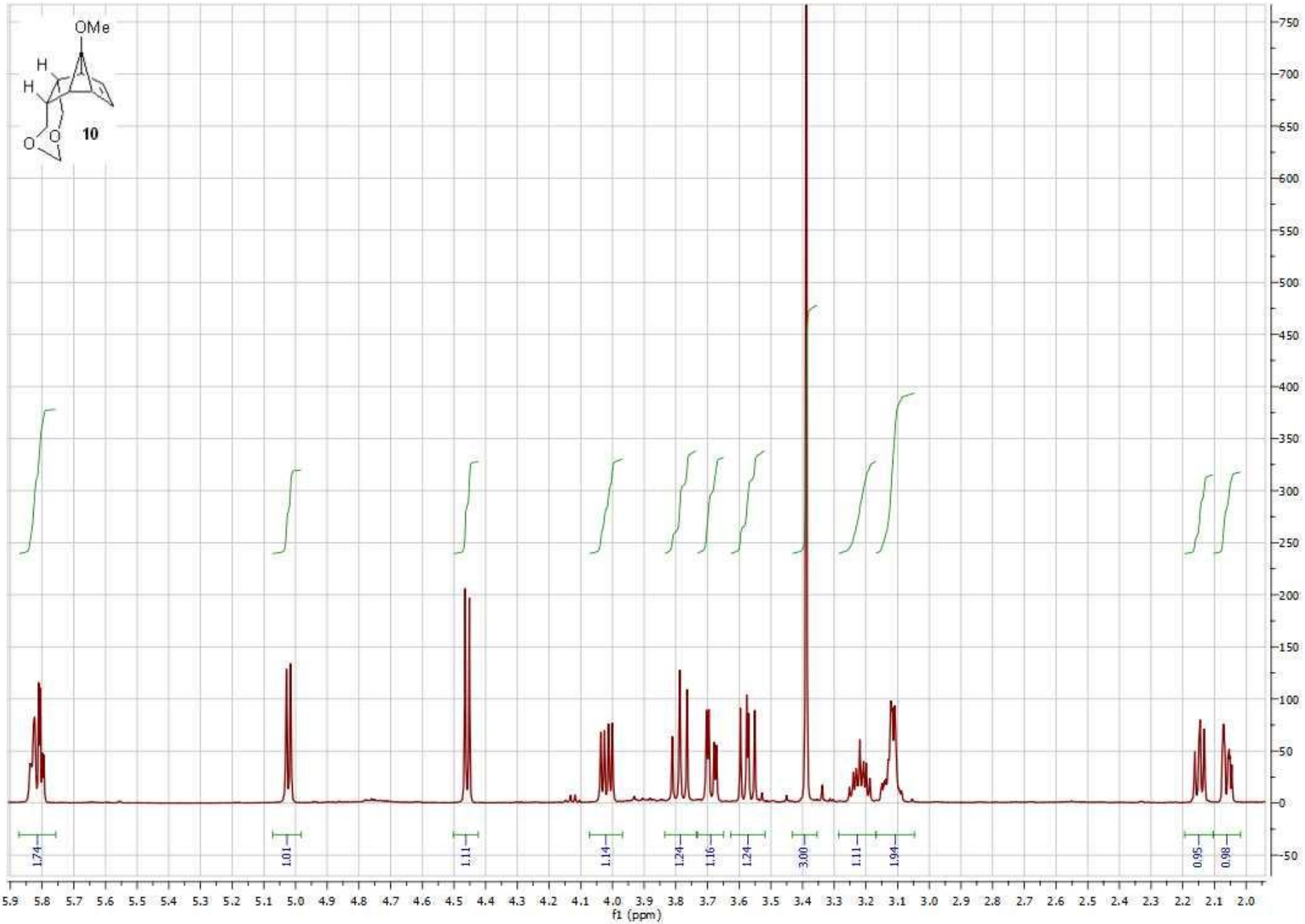
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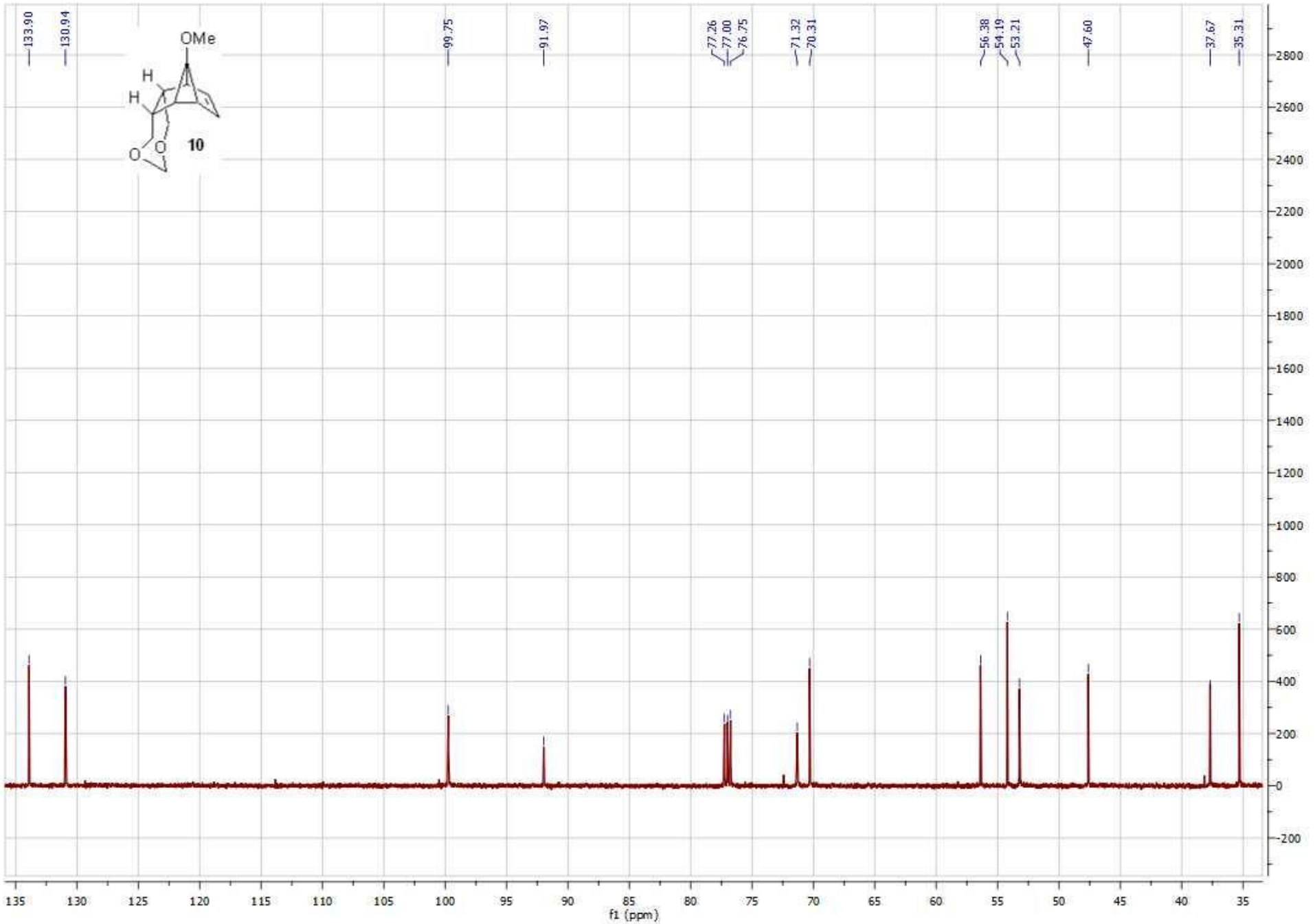
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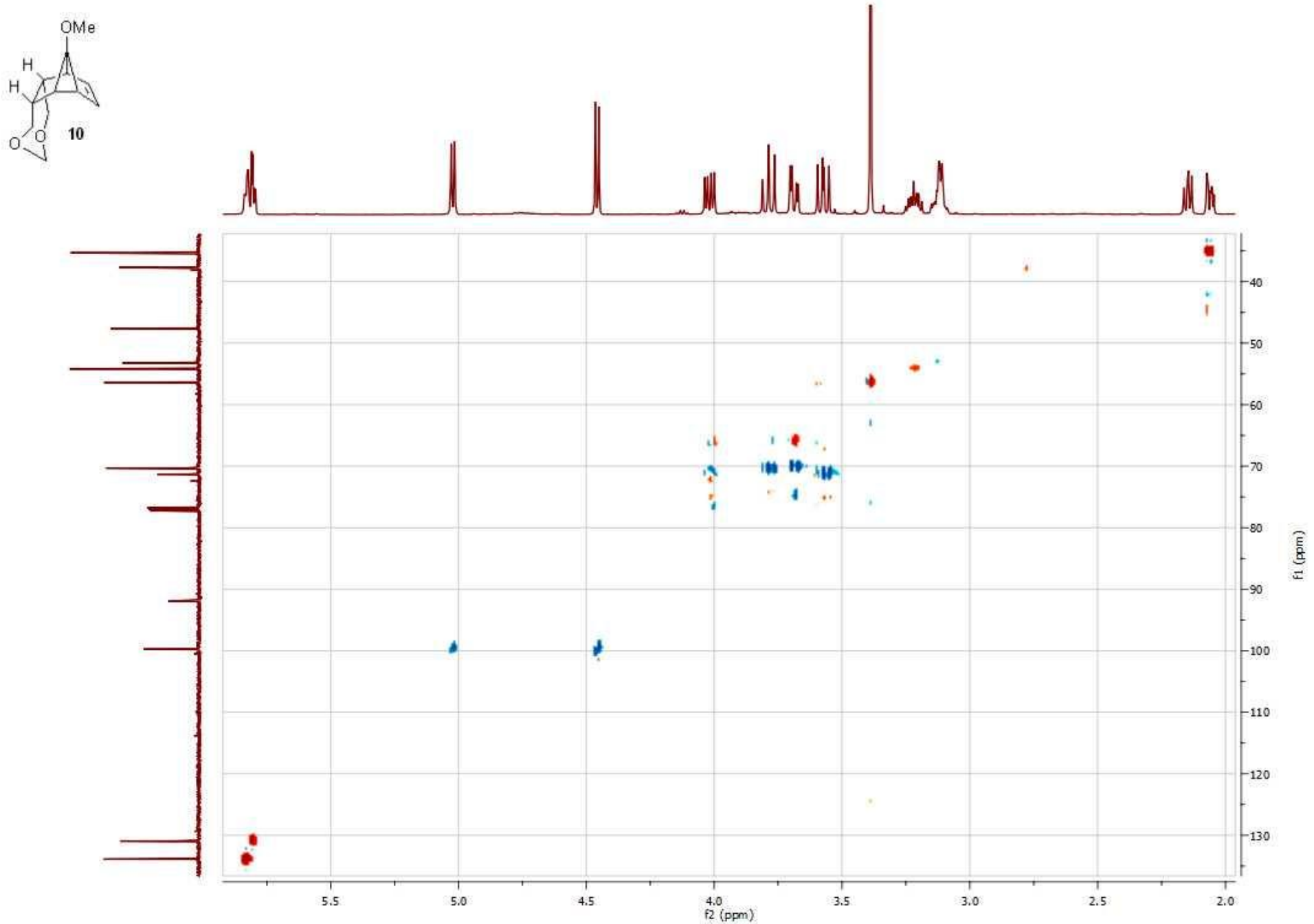

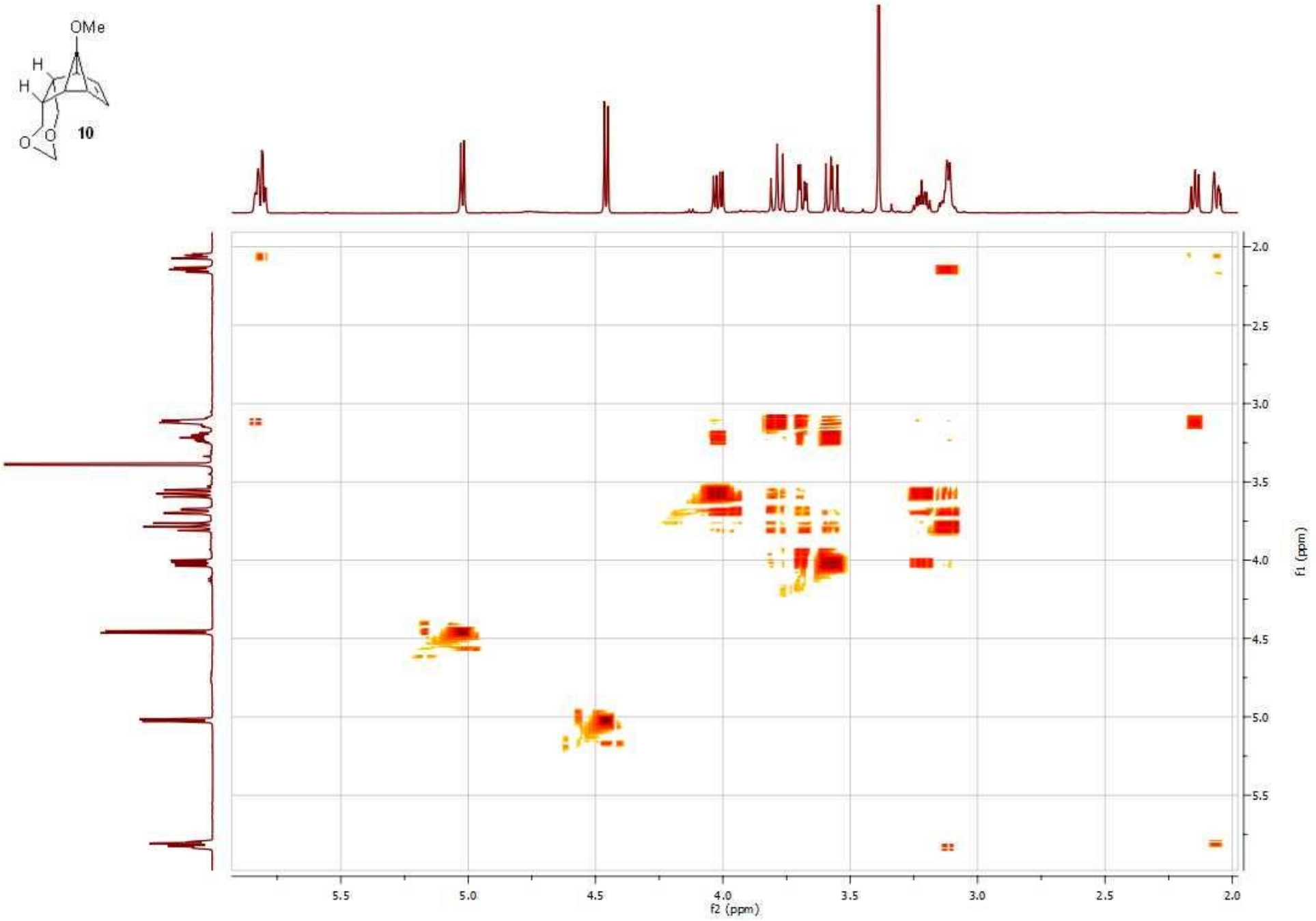
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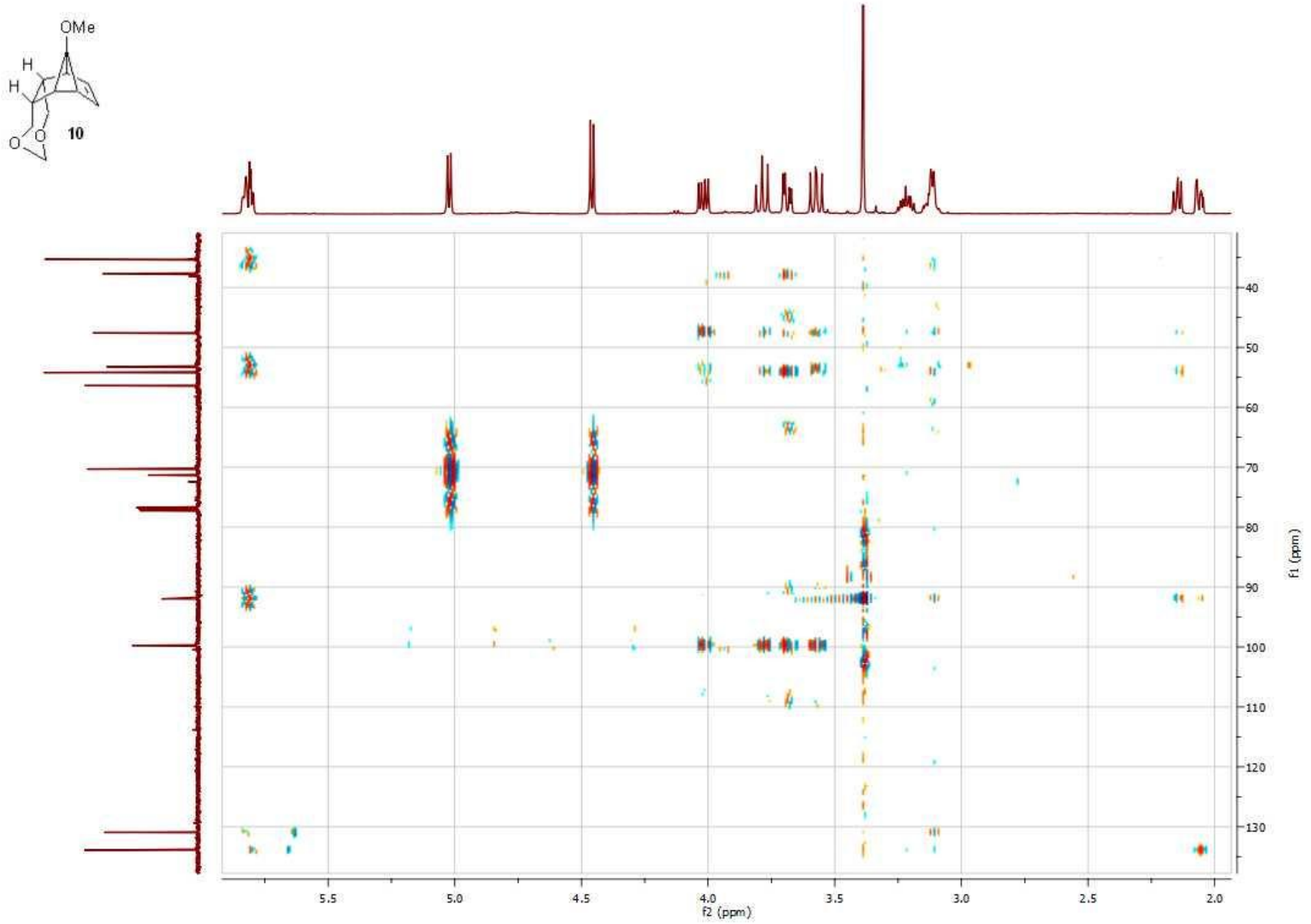
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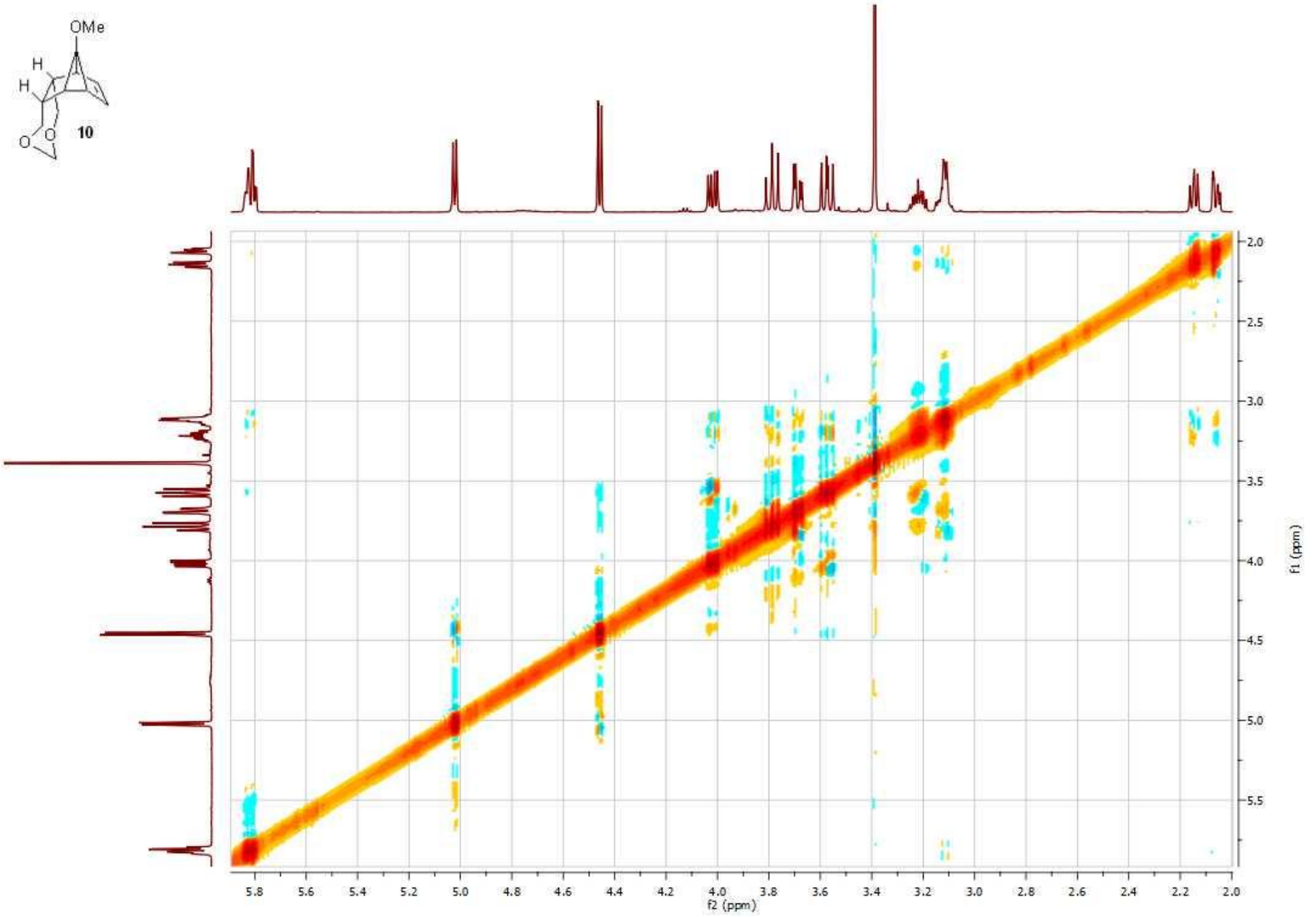
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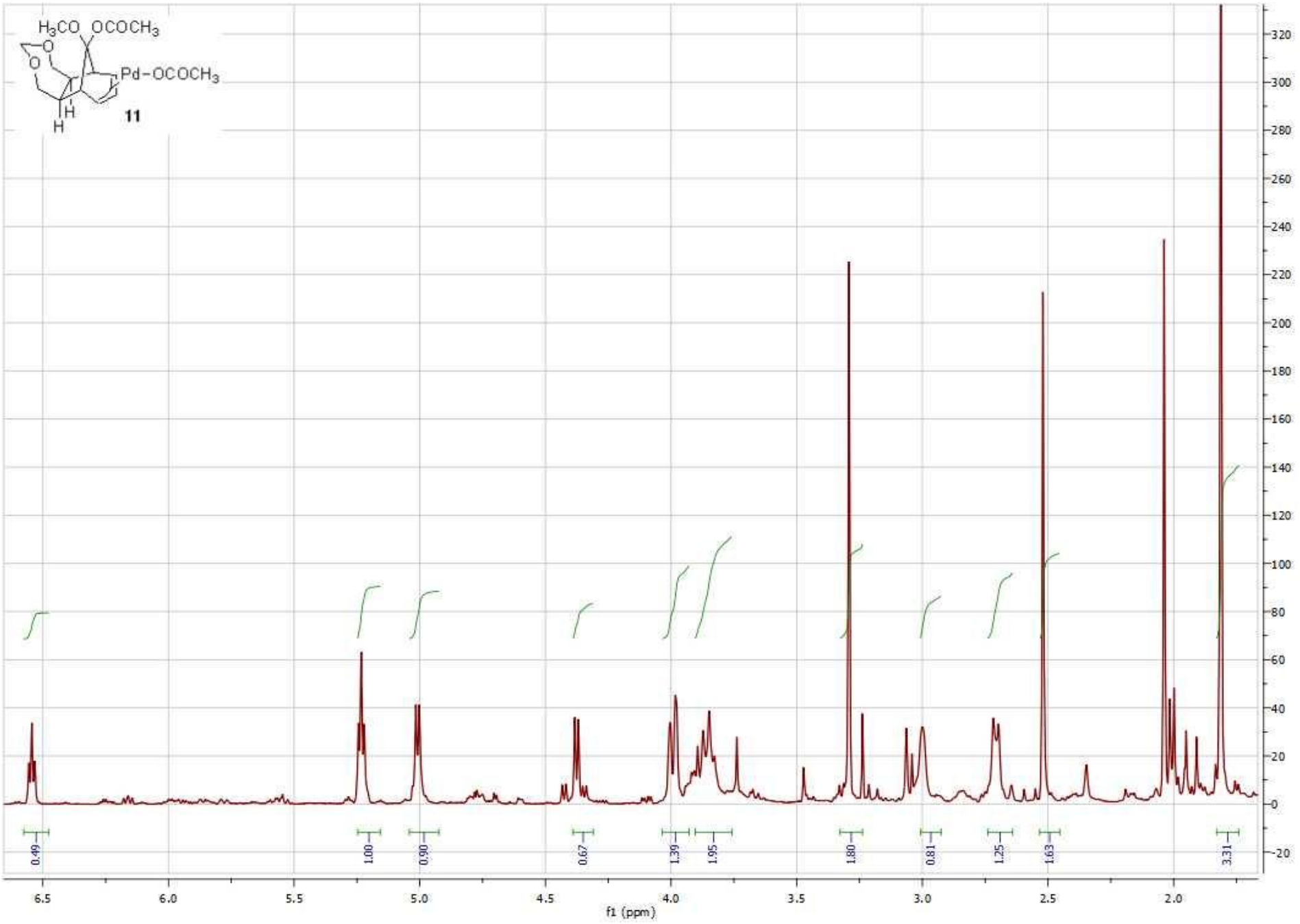
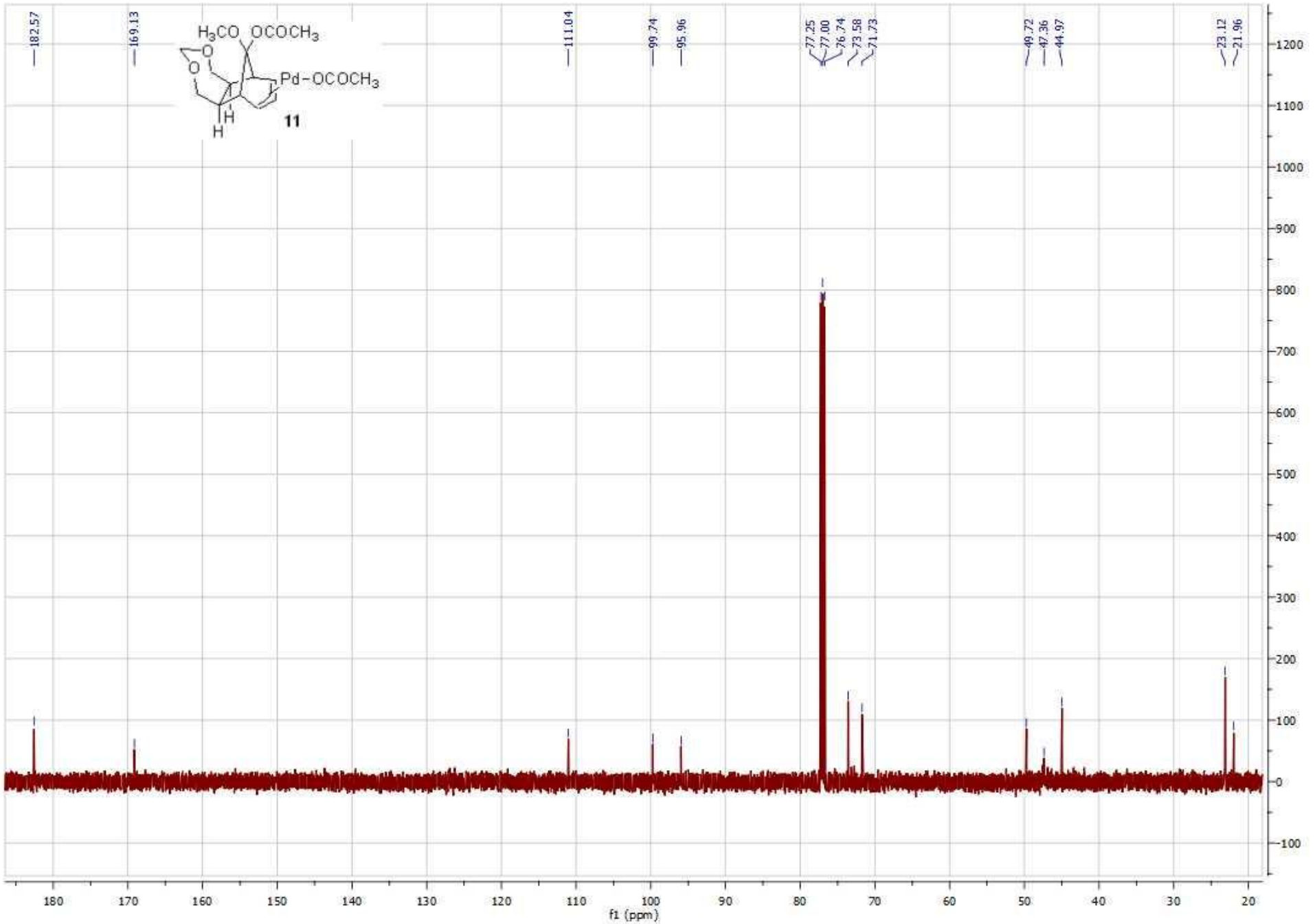
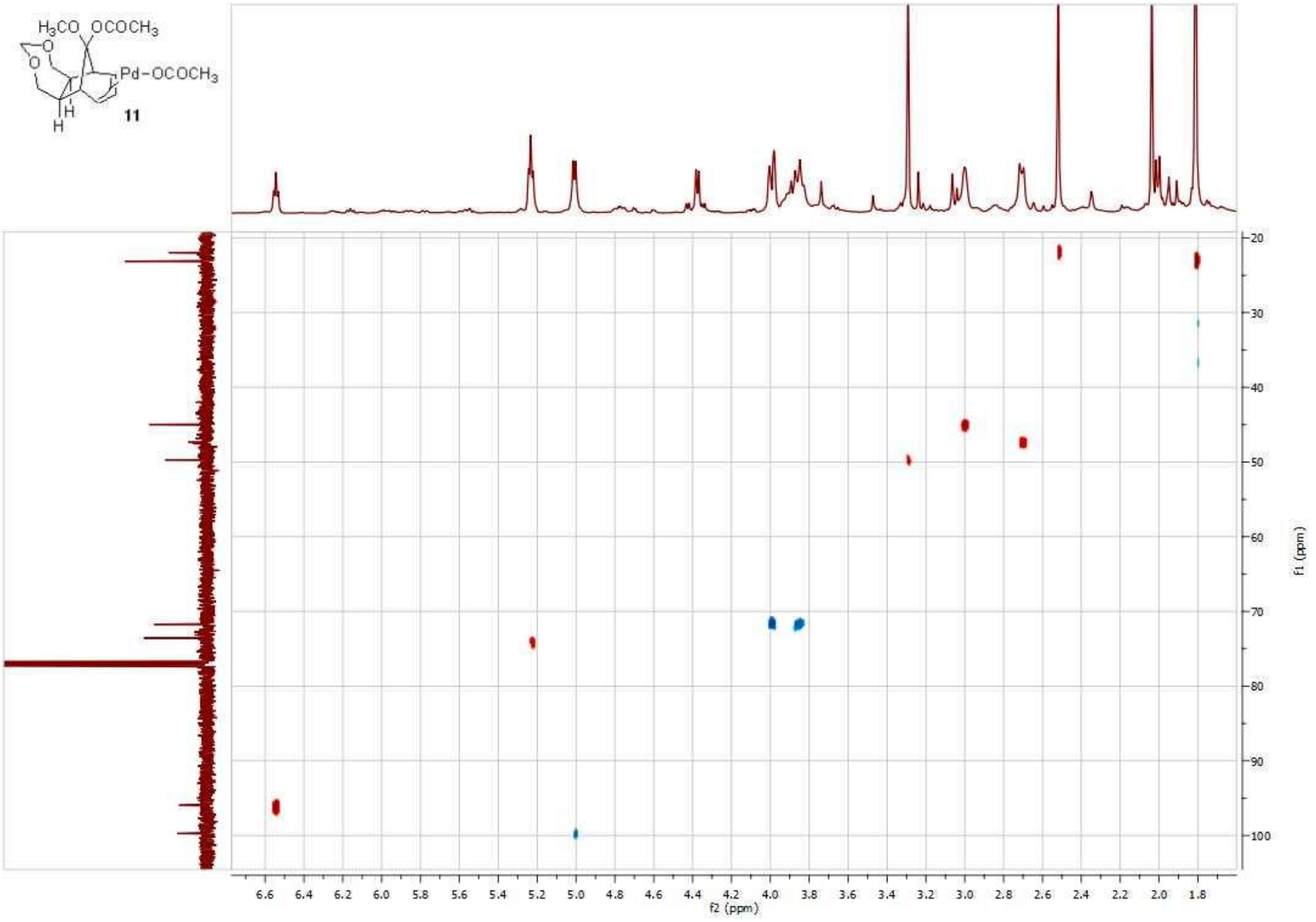
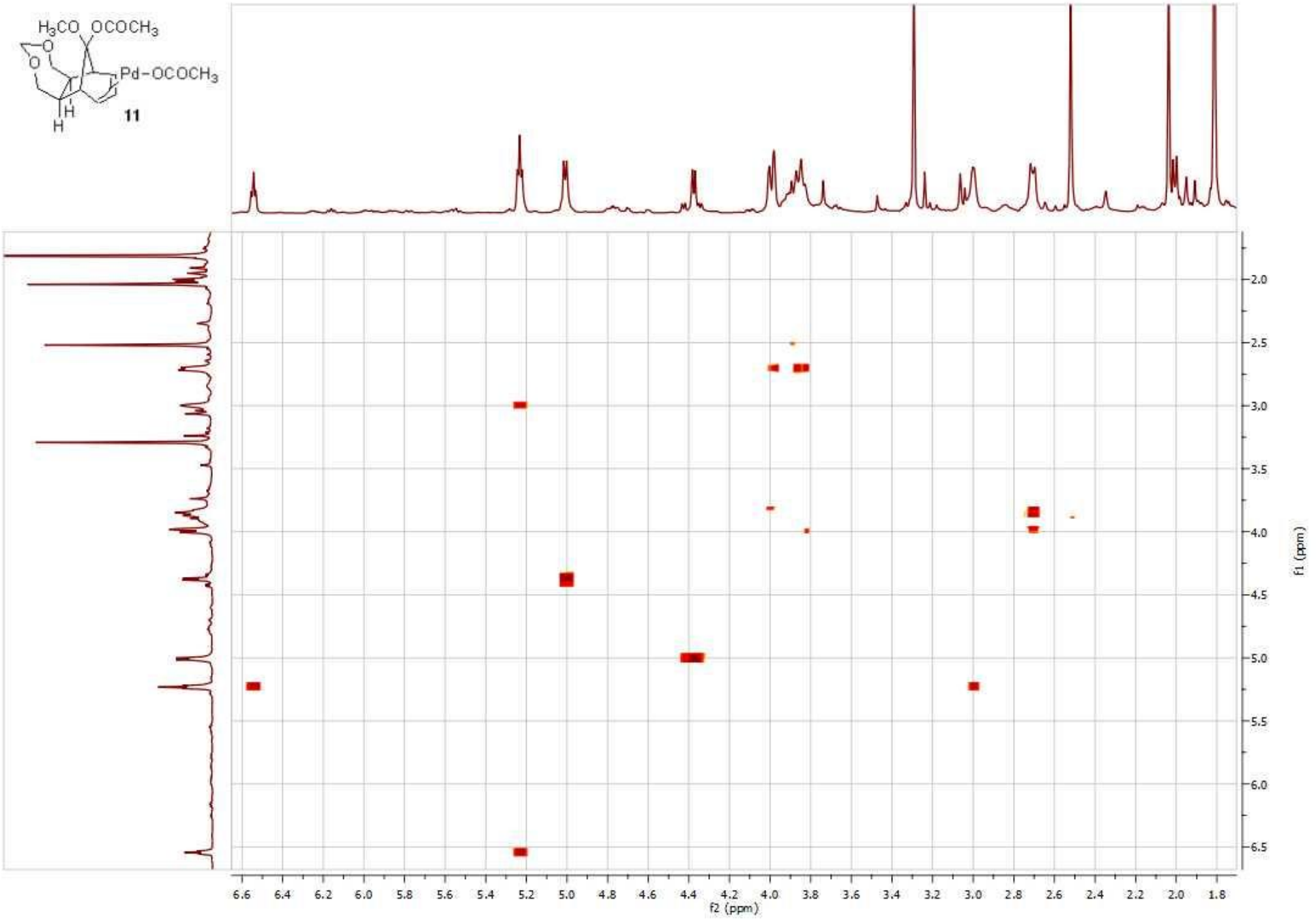
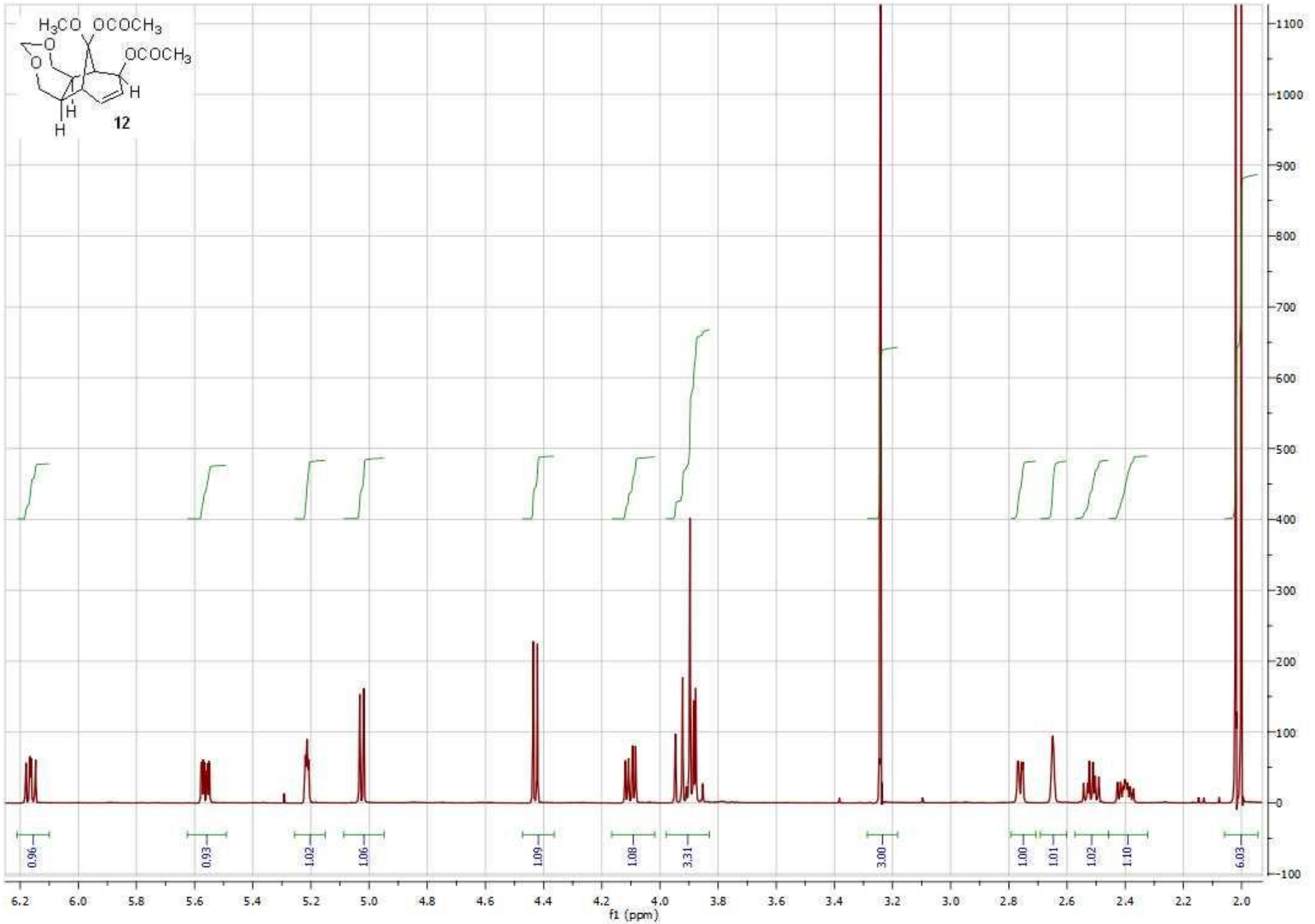
f2 (ppm)
f1 (ppm)

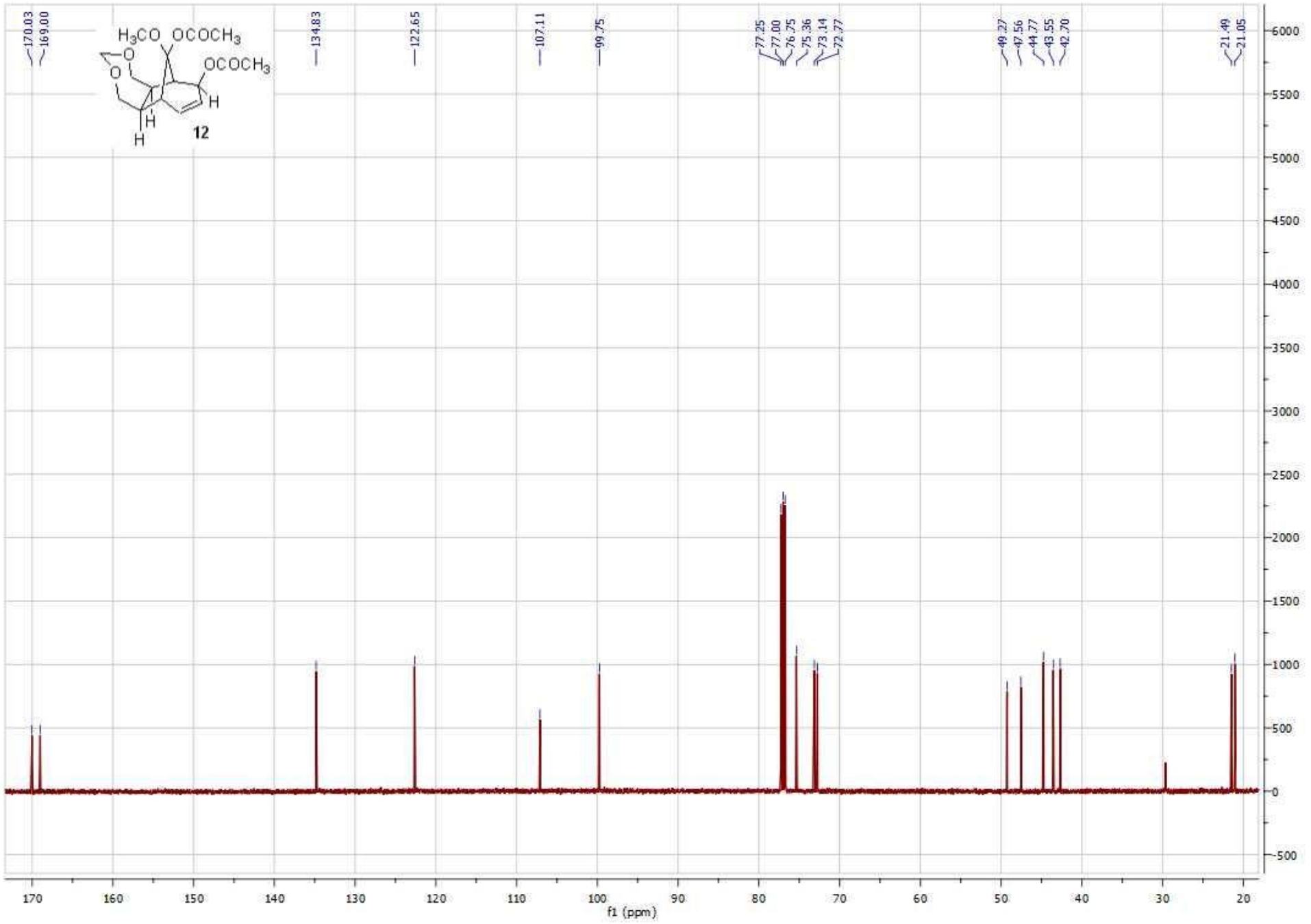

H3CO
OCOCH3
O
O
Pd-OCOCH3
H
H
11
f1 (ppm)
0.49
1.00
0.90
0.67
1.39
1.95
1.80
0.81
1.25
1.63
3.31

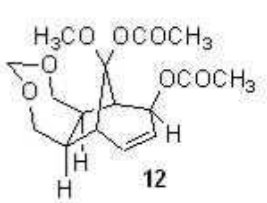

182.57
169.13
111.04
99.74
95.96
77.25
77.00
76.74
73.58
71.73
49.72
47.36
44.97
23.12
21.96
H3CO OCOCH3
Pd-OCOCH3
H
11
f1 (ppm)


H3CO
OCOCH3
O
O
Pd-OCOCH3
H
H
11
f2 (ppm)
f1 (ppm)

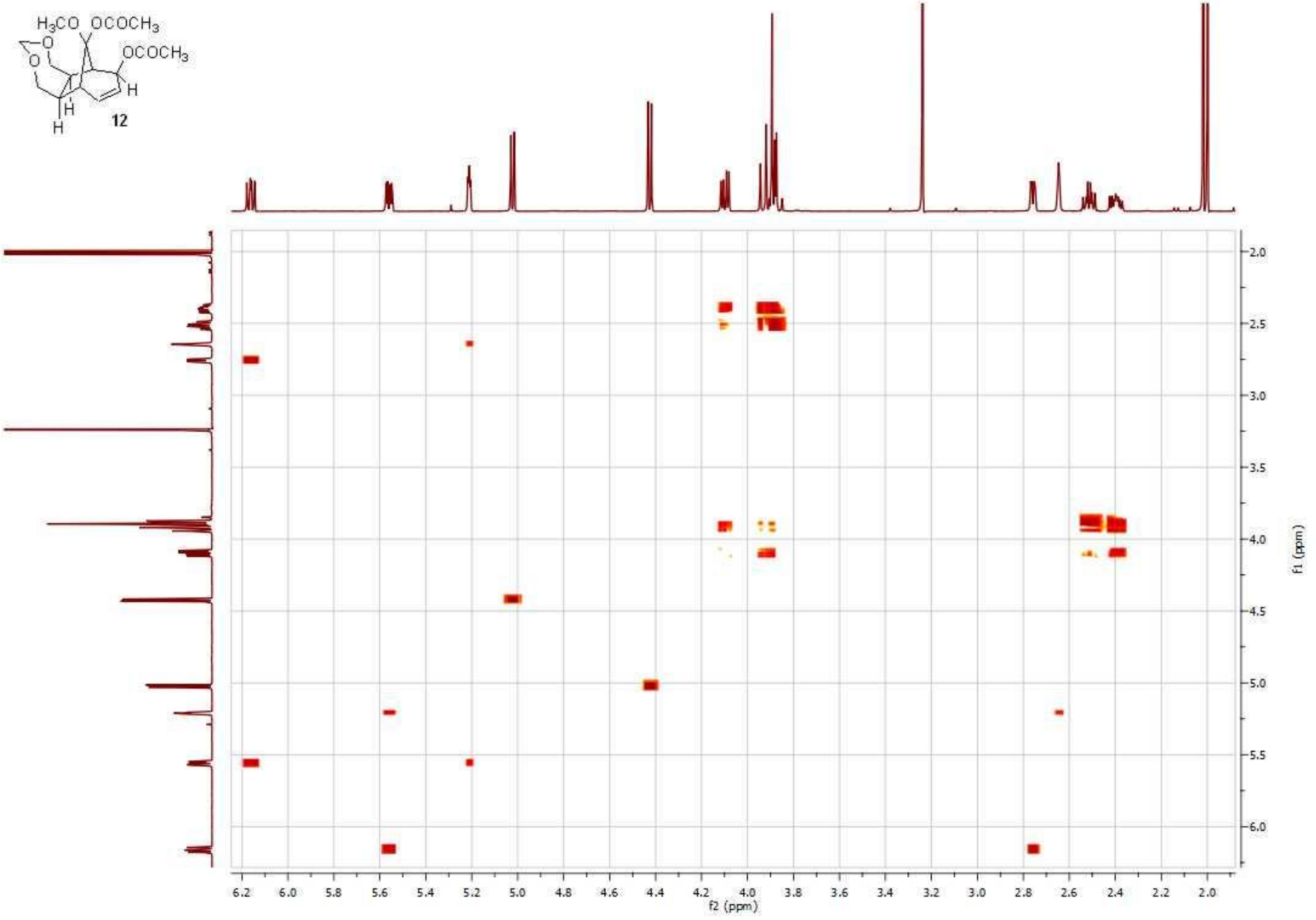

H3CO
OCOCH3
O
O
Pd-OCOCH3
H
H
11
f2 (ppm)
f1 (ppm)

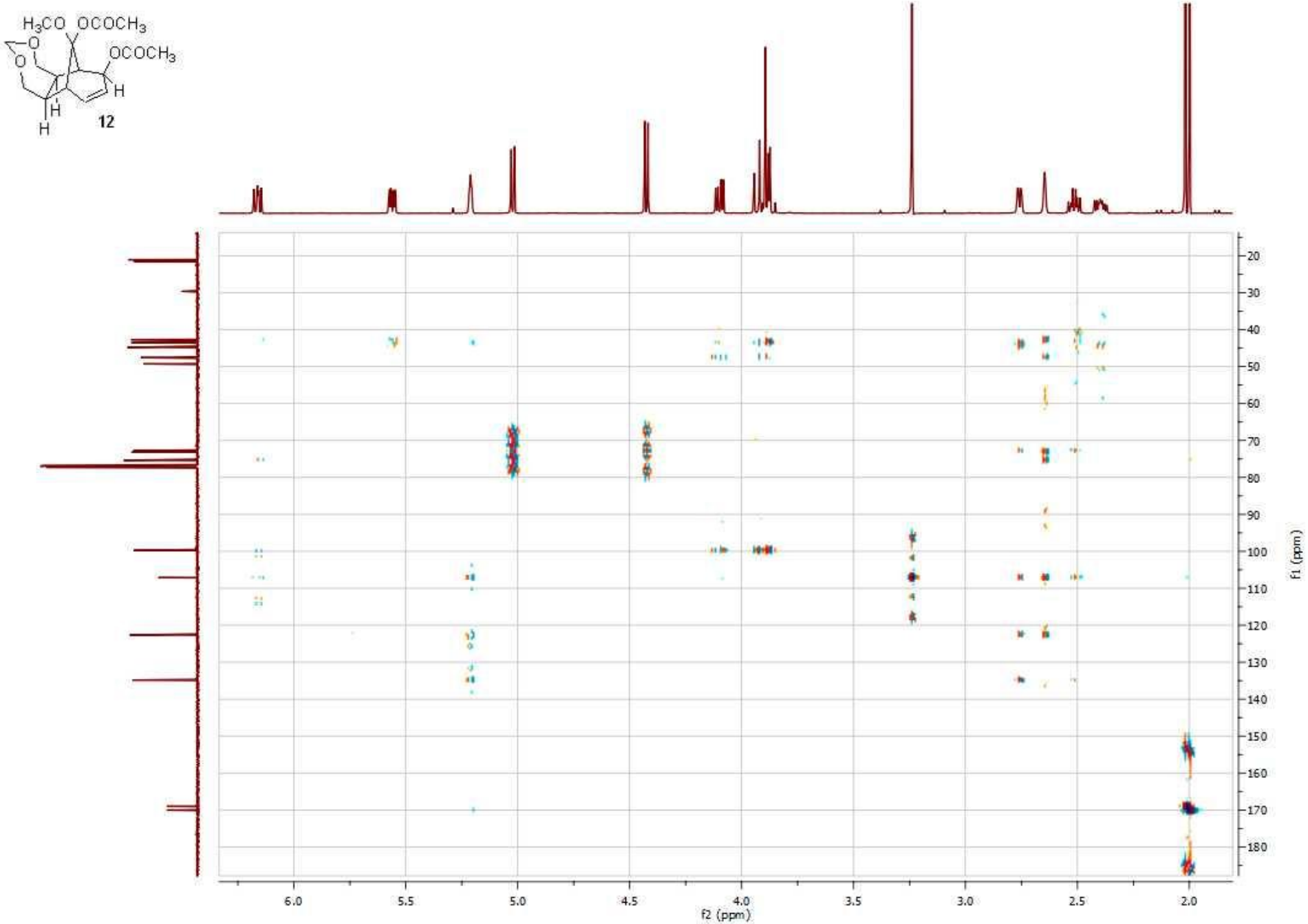


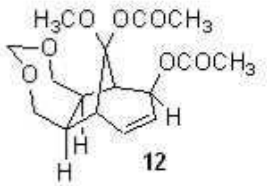

170.03
169.00
134.83
122.65
107.11
99.75
77.25
77.00
76.75
75.36
73.14
72.77
49.27
47.56
44.77
43.55
42.70
21.49
21.05
H3CO OCOCH3
OCOCH3
12
f1 (ppm)



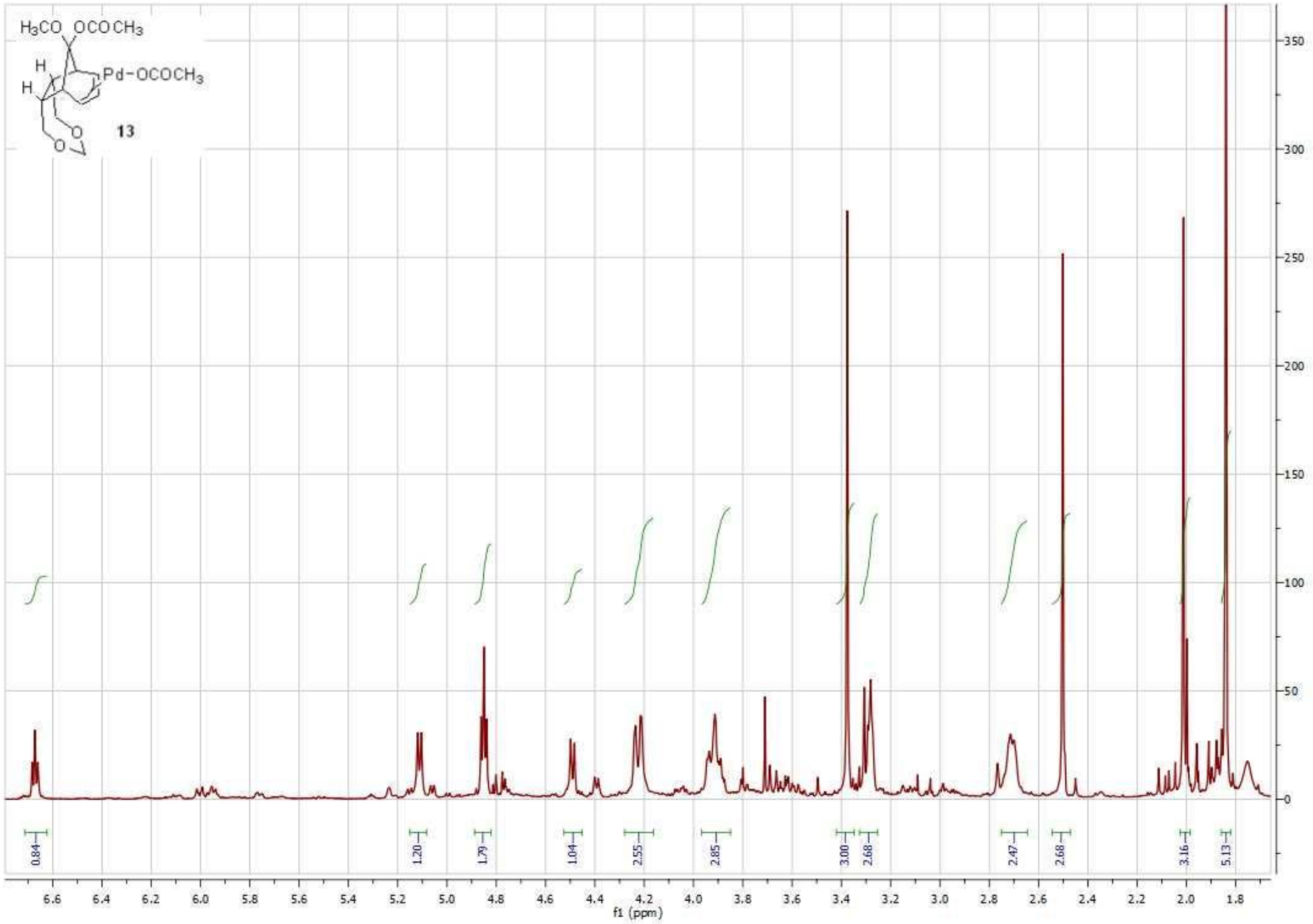
12

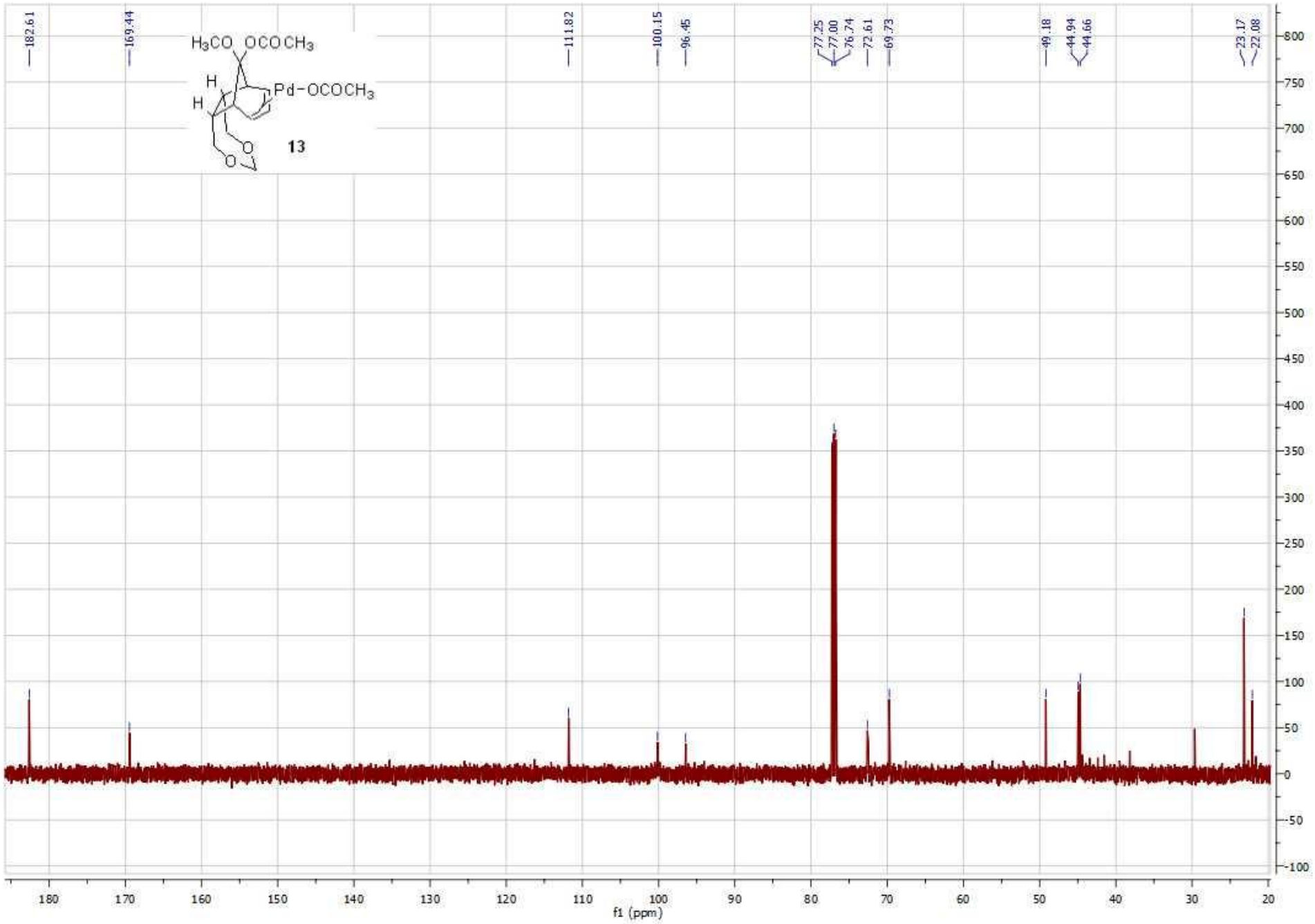


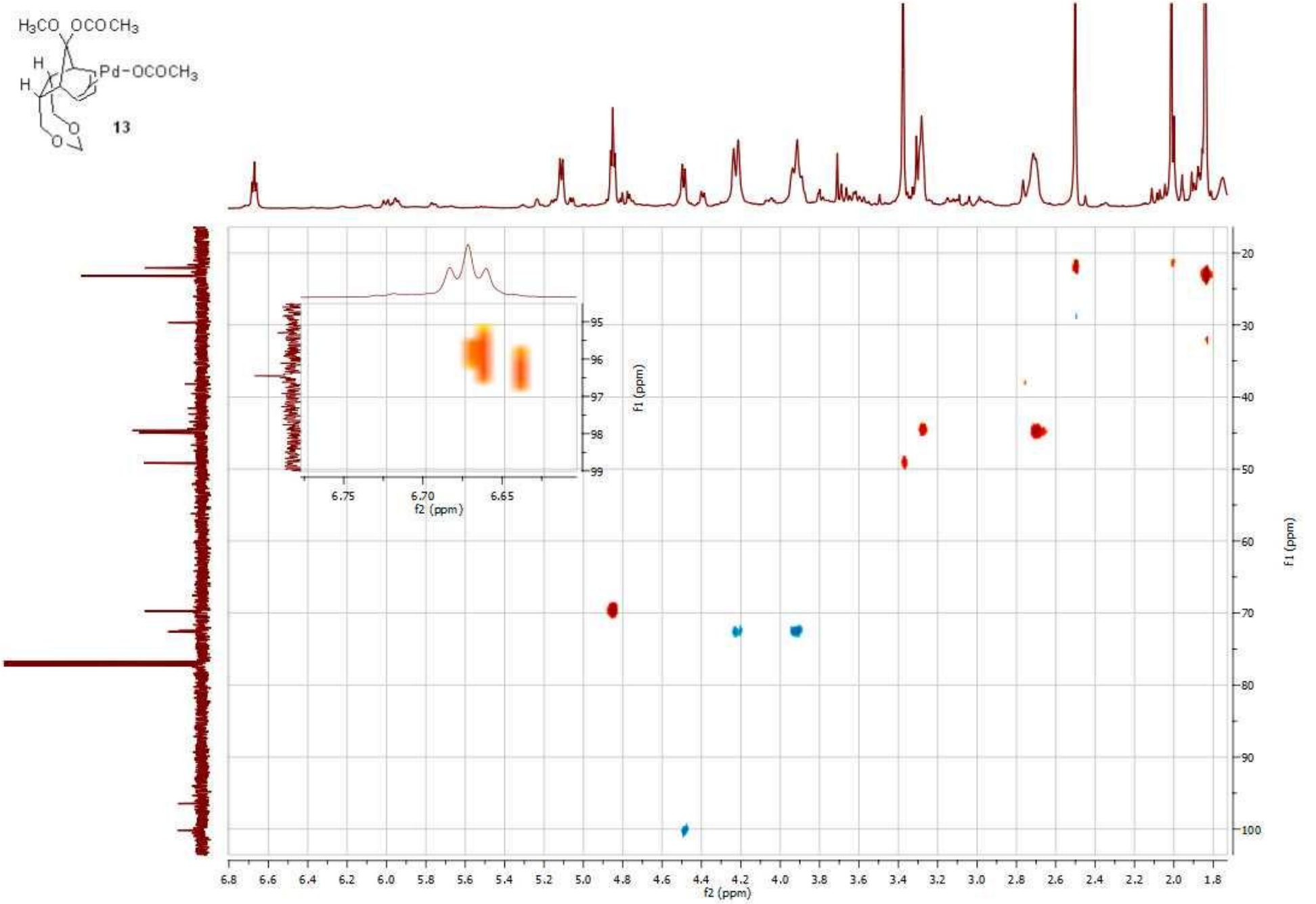

H3CO
OCOCH3
O
O
OCOCH3
H
H
H
12
f2 (ppm)
f1 (ppm)

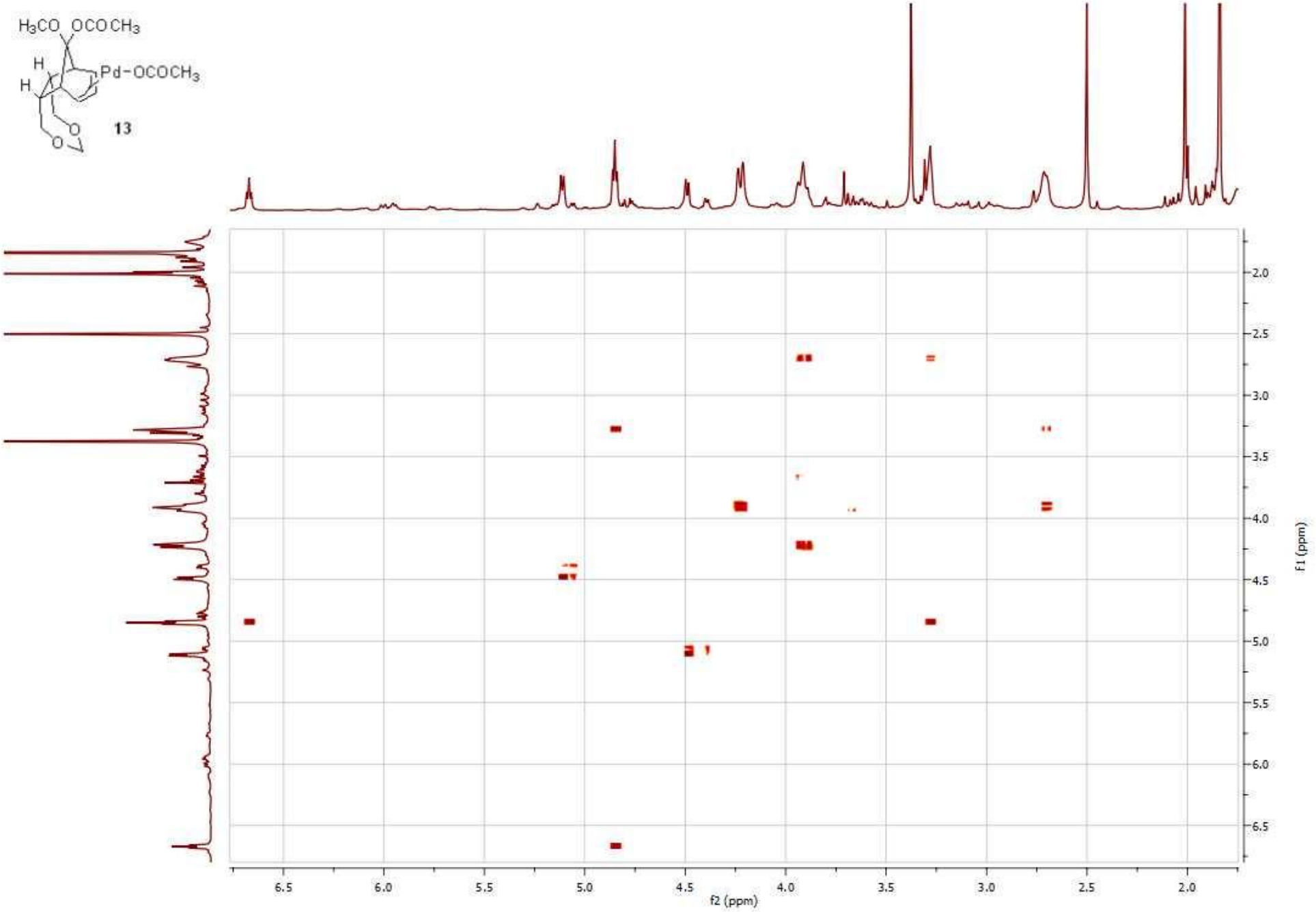


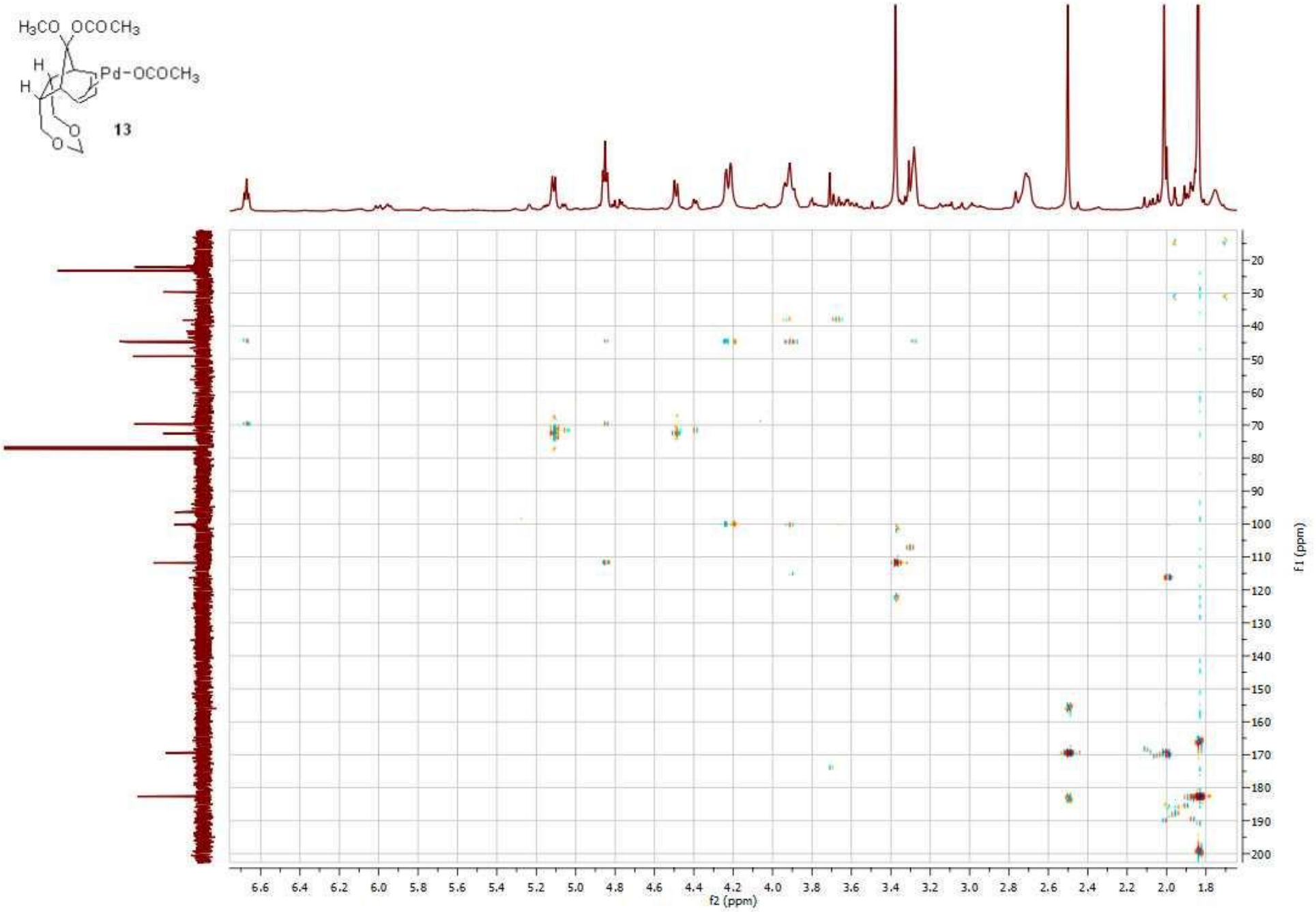
12

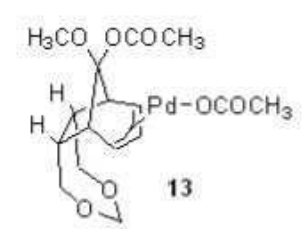

H3CO OCOCH3
H
H
Pd-OCOCH3
O
O
13
0.84
1.20
1.79
1.04
2.55
2.85
3.00
2.68
2.47
2.68
3.16
5.13
f1 (ppm)


182.61
169.44
111.82
100.15
96.45
77.25
77.00
76.74
72.61
69.73
49.18
44.94
44.66
23.17
22.08
H3CO OCOCH3
H
H
Pd-OCOCH3
O
O
13
f1 (ppm)

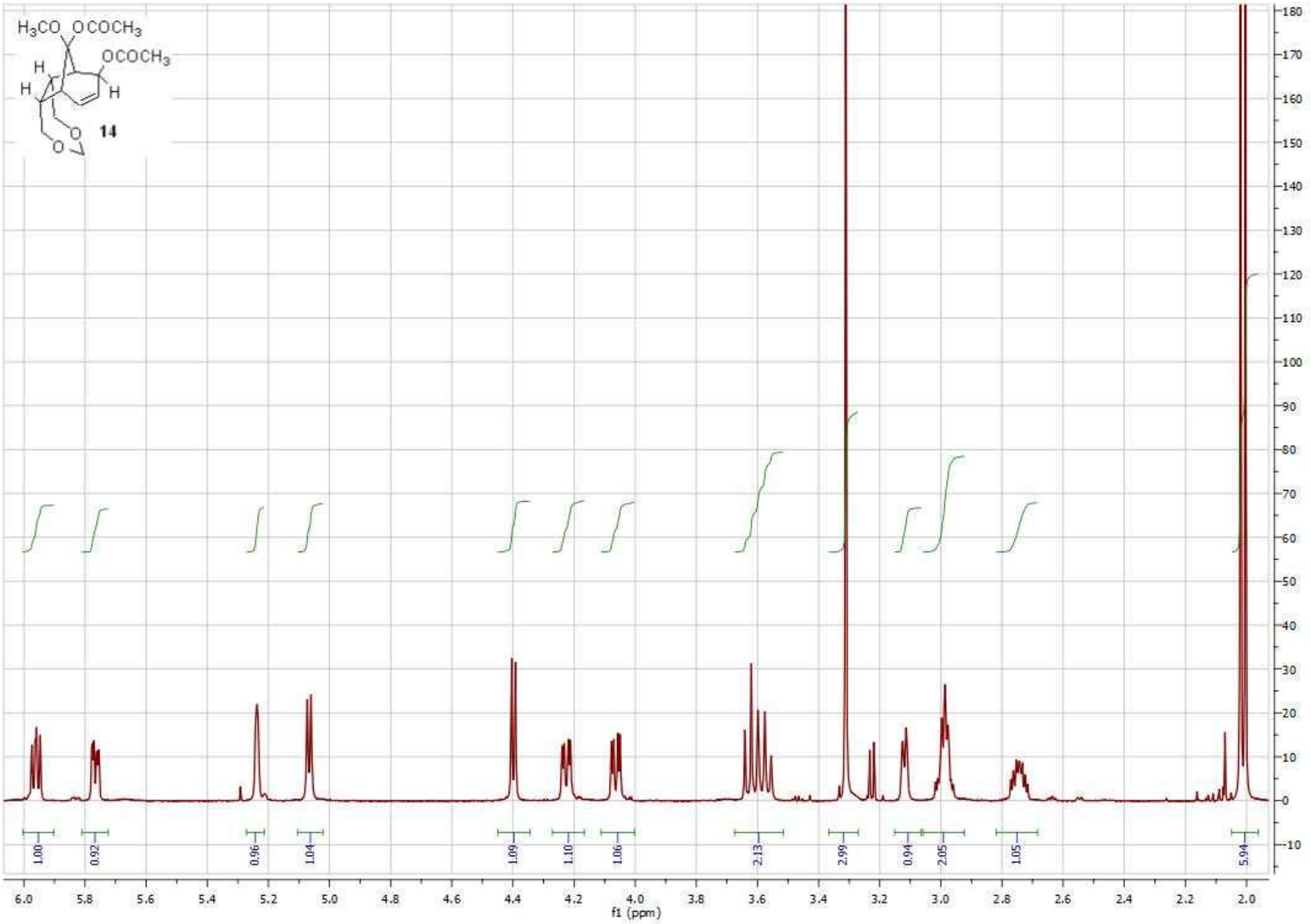

H3CO OCOCH3
H
H
Pd-OCOCH3
O
O
13
f1 (ppm)
f2 (ppm)
6.75
6.70
6.65
95
96
97
98
99
20
30
40
50
60
70
80
90
100
6.8
6.6
6.4
6.2
6.0
5.8
5.6
5.4
5.2
5.0
4.8
4.6
4.4
4.2
4.0
3.8
3.6
3.4
3.2
3.0
2.8
2.6
2.4
2.2
2.0
1.8

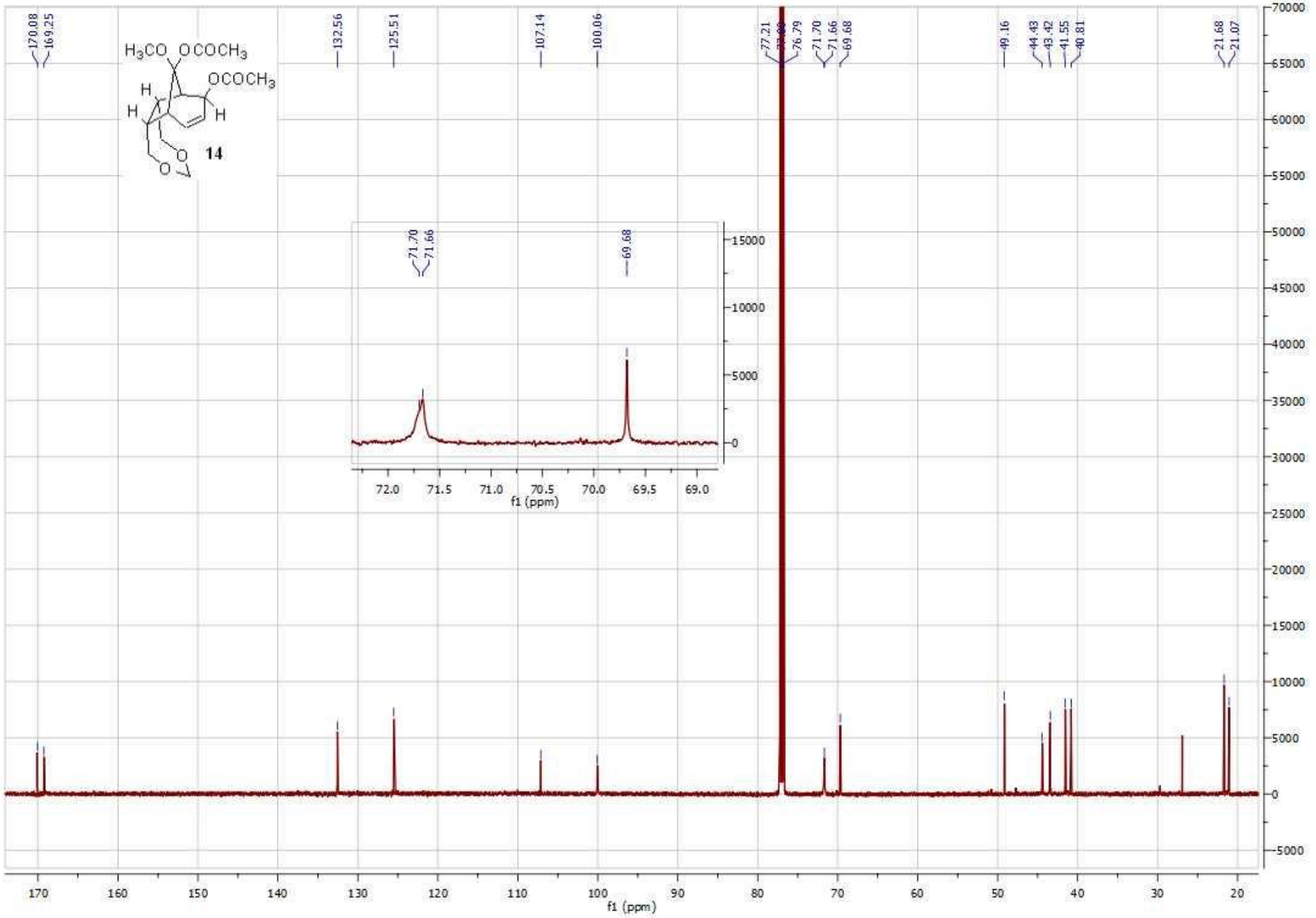

H3CO
OCOCH3
H
H
Pd-OCOCH3
O
O
13
f2 (ppm)
f1 (ppm)

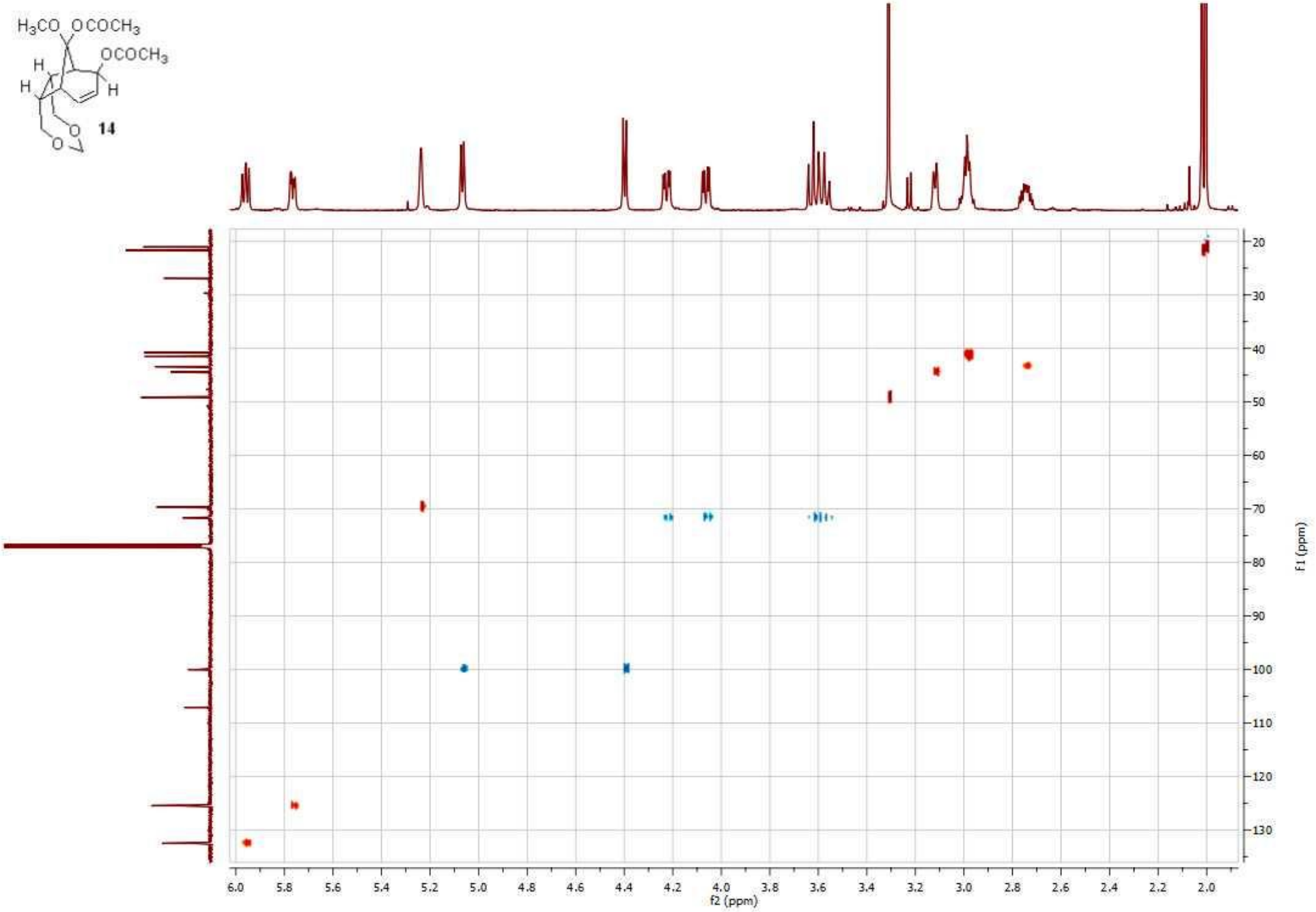

H3CO
OCOCH3
H
H
Pd-OCOCH3
O
O
13
f2 (ppm)
f1 (ppm)

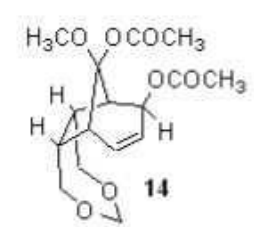


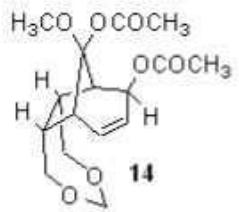
13


H3CO OCOCH3
OCOCH3
H
H
H
O
O
14
f1 (ppm)
1.00
0.92
0.96
1.04
1.09
1.10
1.06
2.13
2.99
0.94
2.05
1.05
5.94

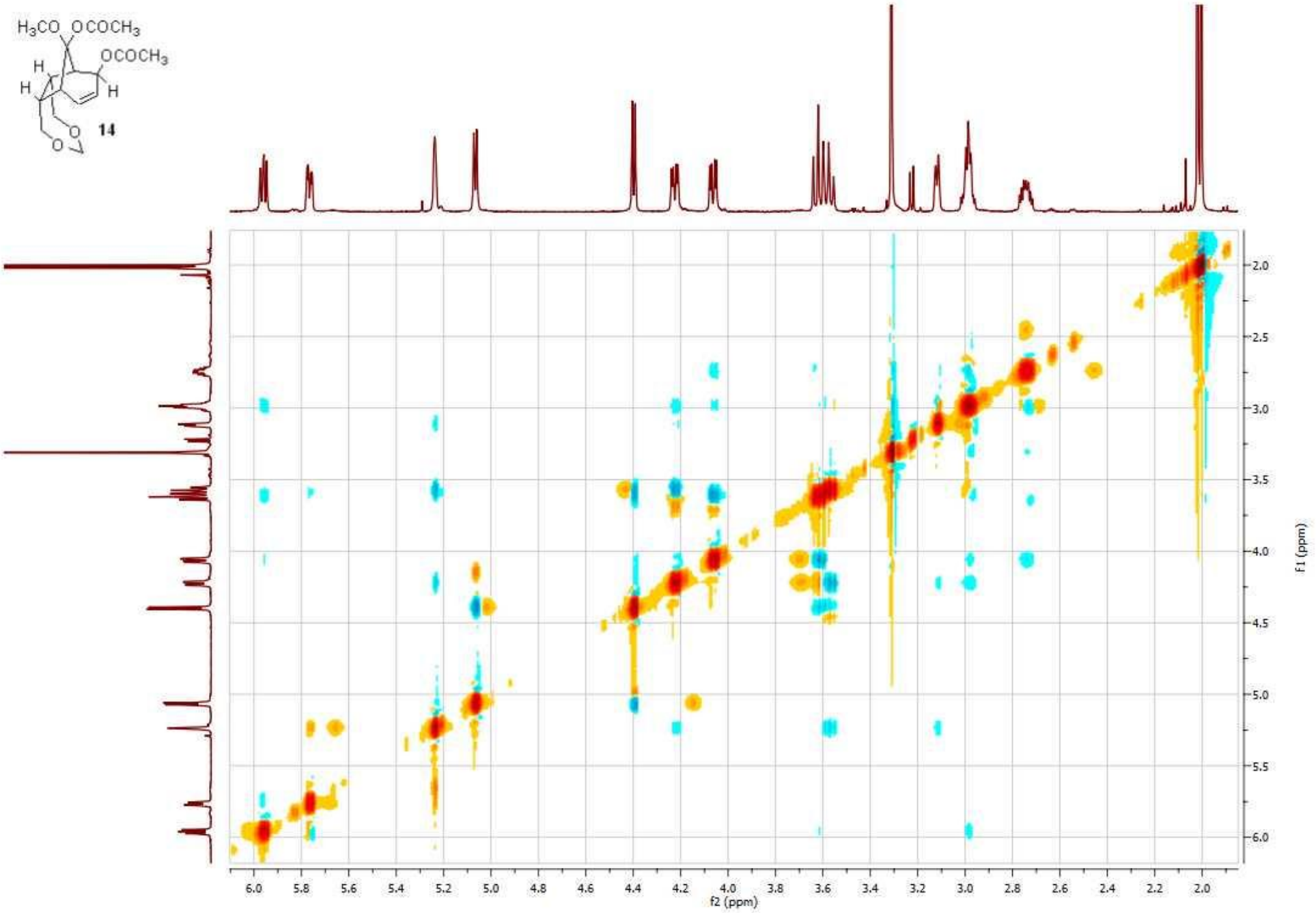


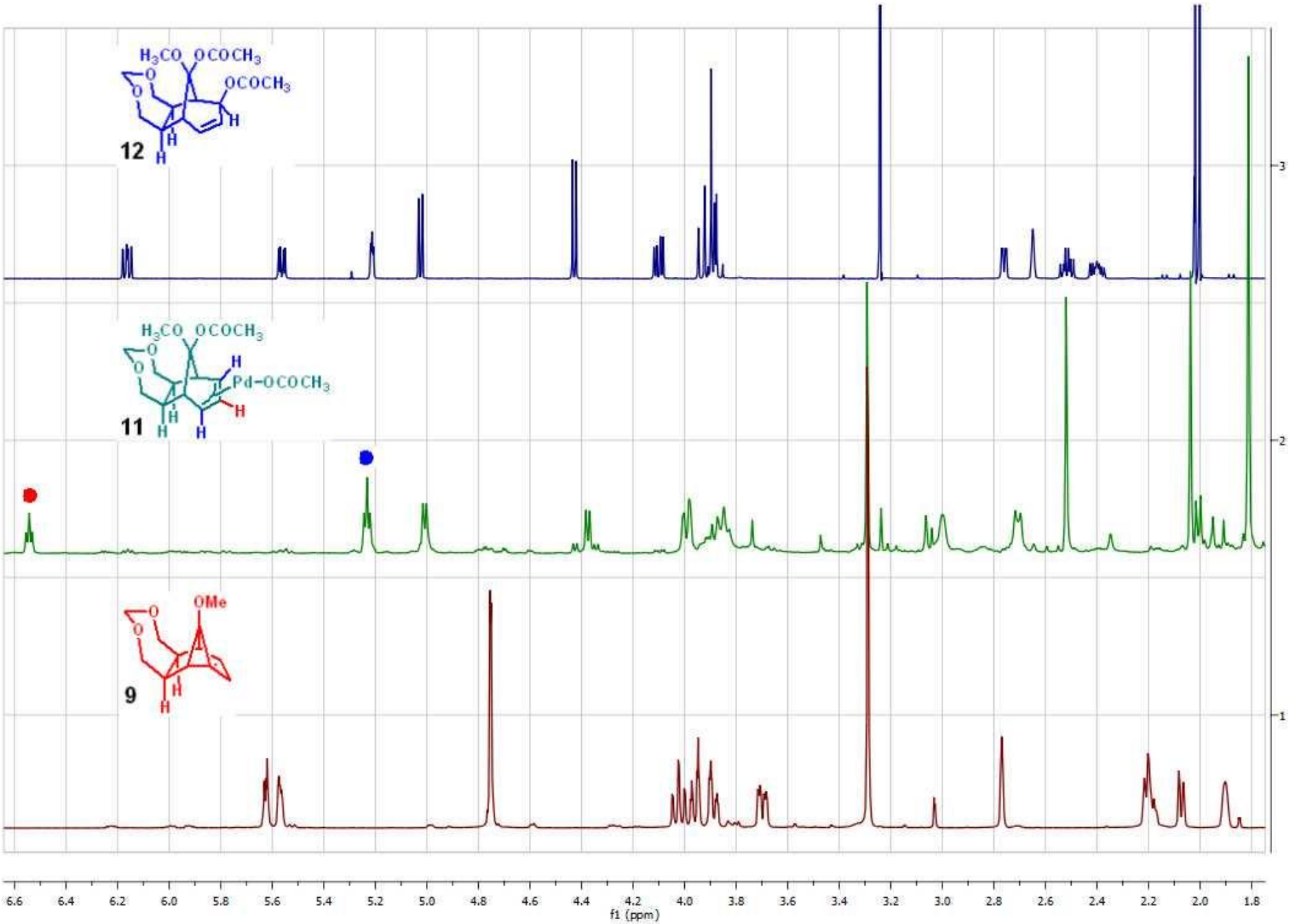


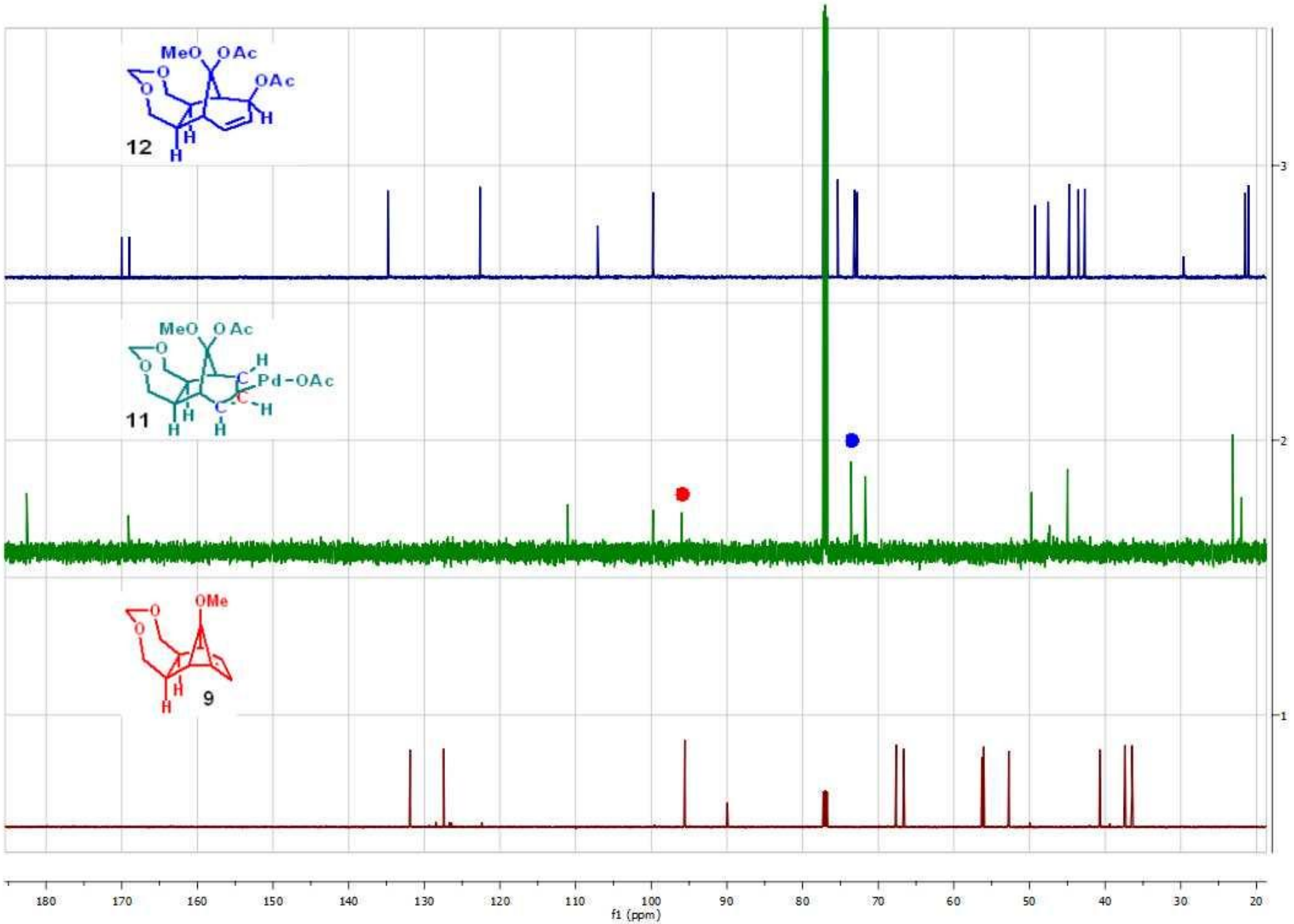


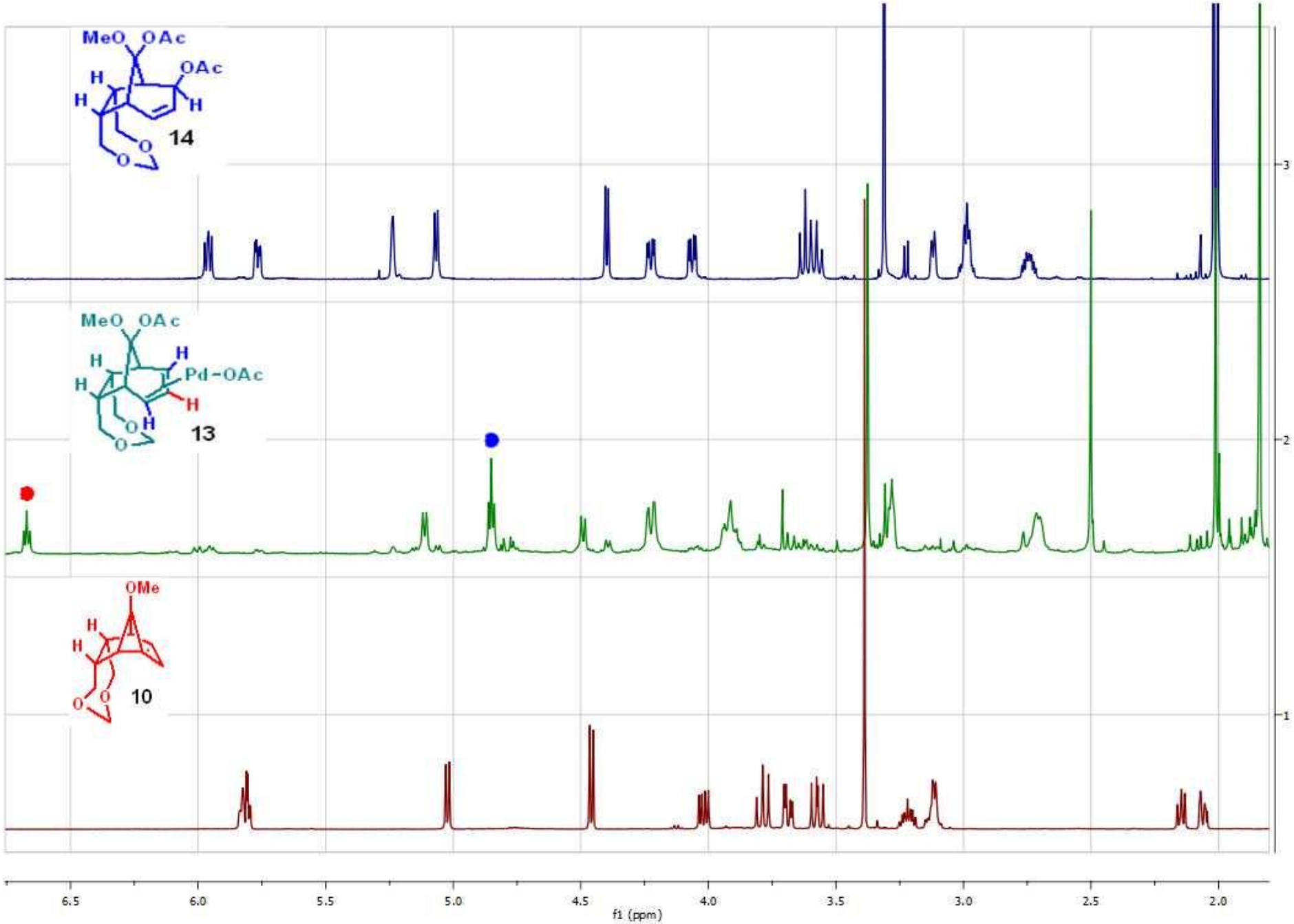


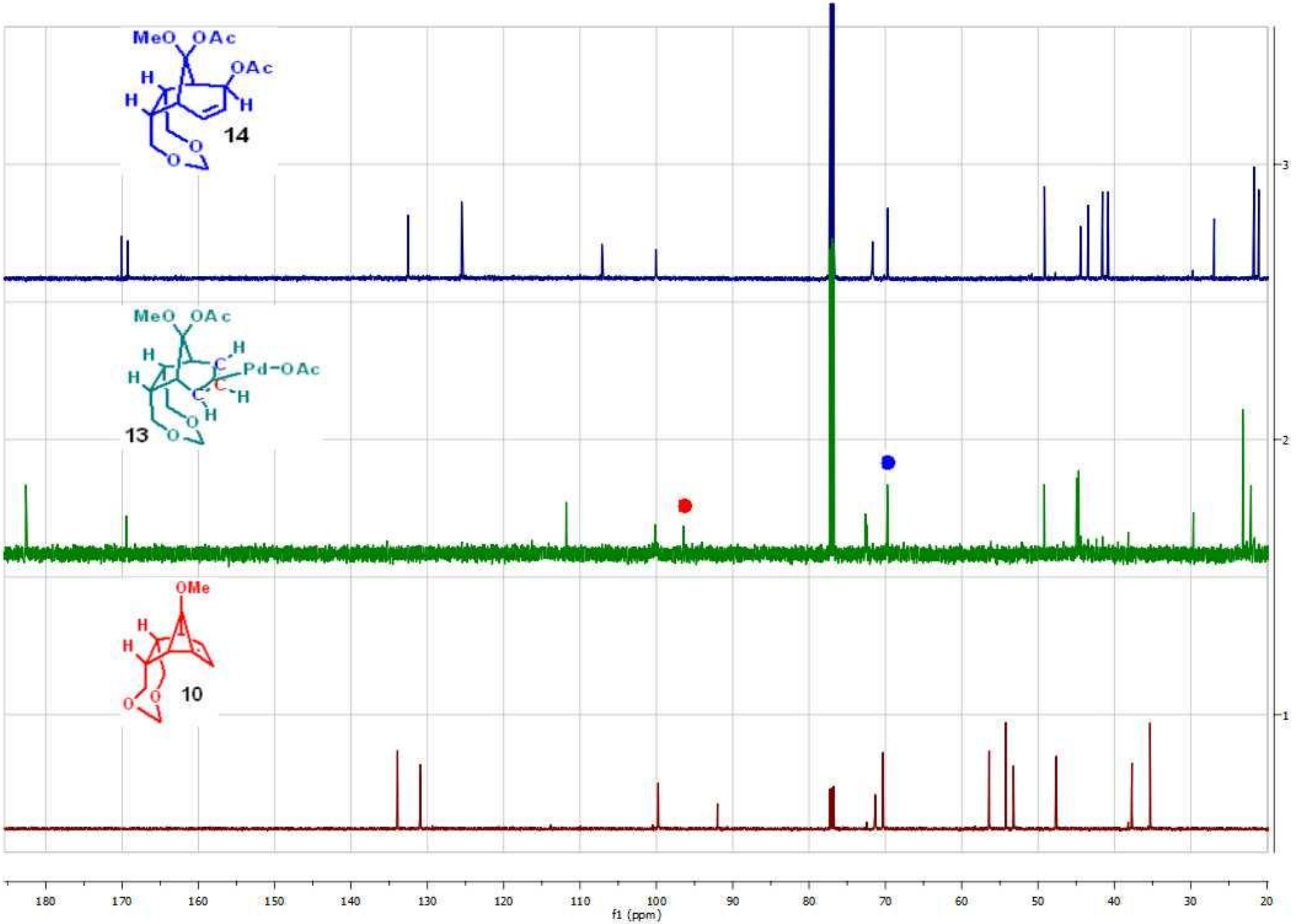
14


H3CO
OCOCH3
OCOCH3
H
H
H
O
O
14
f2 (ppm)
f1 (ppm)


H3CO OCOCH3
OCOCH3
12
H3CO OCOCH3
Pd-OCOCH3
11
OMe
9
6.6 6.4 6.2 6.0 5.8 5.6 5.4 5.2 5.0 4.8 4.6 4.4 4.2 4.0 3.8 3.6 3.4 3.2 3.0 2.8 2.6 2.4 2.2 2.0 1.8
f1 (ppm)
3
2
1


MeO
OAc
OAc
O
O
H
H
H
12
MeO
OAc
O
O
H
C
Pd-OAc
C
C
H
H
H
H
11
OMe
O
O
H
H
9
3
2
1
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
f1 (ppm)


MeO
OAc
OAc
H
H
H
O
O
14
MeO
OAc
H
H
H
Pd-OAc
H
O
H
O
13
OMe
H
H
O
O
10
3
2
1
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
f1 (ppm)


MeO
OAc
OAc
H
H
H
O
O
14
MeO
OAc
H
H
C
H
Pd–OAc
C
C
H
O
H
O
13
OMe
H
H
O
O
10
3
2
1
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
f1 (ppm)

Crystal structure of compound **12**

The crystallographic data for compound **12** have been deposited at the Cambridge Crystallographic Data Centre as supplementary publication number CCDC 698743.

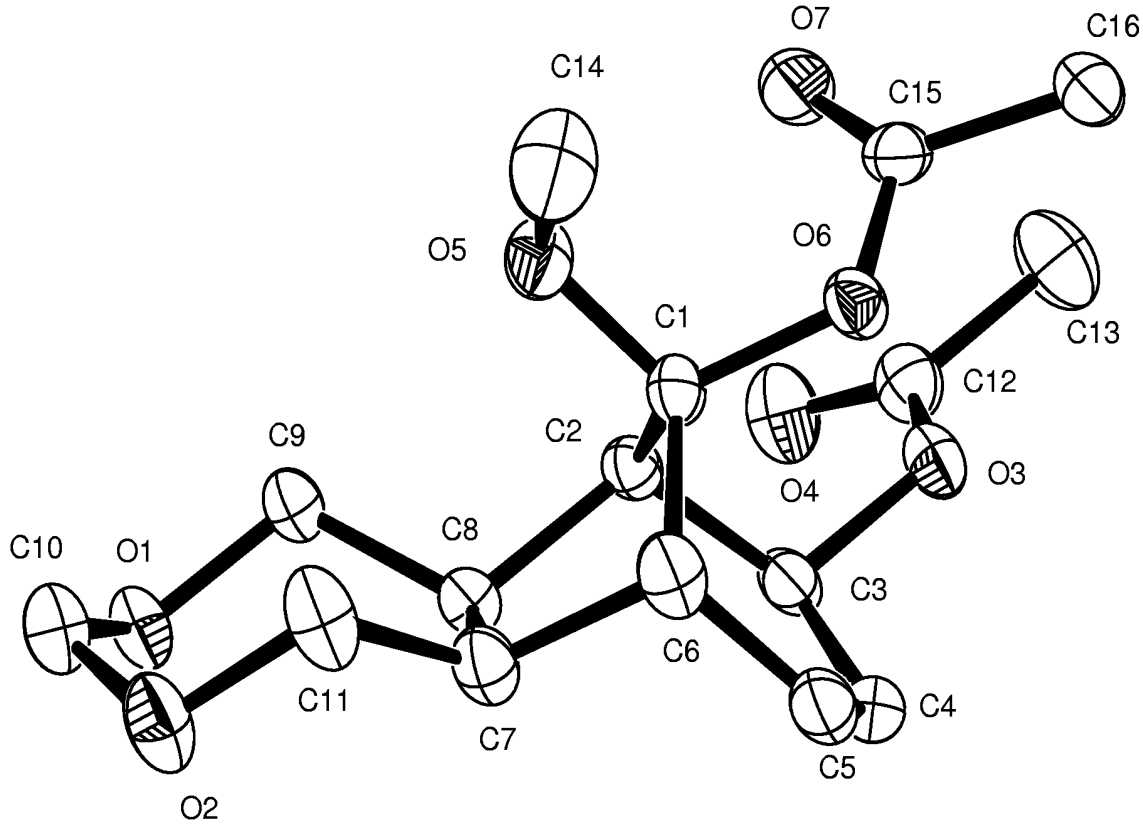


Table 1. Crystal data and structure refinement .

Identification code	feb1108	
Empirical formula	C16 H22 O7	
Formula weight	326.34	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$ (No.14)	
Unit cell dimensions	a = 9.0913(5) Å	α= 90°.
	b = 10.8004(4) Å	β= 91.343(2)°.
	c = 16.2056(9) Å	γ = 90°.
Volume	1590.79(14) $Å^3$	
Z	4	
Density (calculated)	1.36 Mg/m^3	
Absorption coefficient	0.11 mm^{-1}	
F(000)	696	
Crystal size	0.30 x 0.30 x 0.25 mm^3	
Theta range for data collection	3.41 to 26.03°.	
Index ranges	-11<=h<=9, -11<=k<=13, -15<=l<=19	
Reflections collected	7843	
Independent reflections	3058 [R(int) = 0.044]	
Reflections with I>2sigma(I)	2421	
Completeness to theta = 26.03°	97.5 %	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3058 / 0 / 210	
Goodness-of-fit on F^2	0.966	
Final R indices [I>2sigma(I)]	R1 = 0.043, wR2 = 0.098	
R indices (all data)	R1 = 0.060, wR2 = 0.109	
Largest diff. peak and hole	0.21 and -0.20 $e.Å^{-3}$	

Data collection KappaCCD , Program package WinGX , Abs correction not applied ,

Refinement using SHELXL-97 , Drawing using ORTEP-3 for Windows

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for feb1108. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	3214(1)	913(1)	4927(1)	29(1)
O(2)	2242(1)	2941(1)	4895(1)	38(1)
O(3)	8958(1)	2019(1)	3175(1)	29(1)
O(4)	8980(1)	-2(1)	3502(1)	40(1)
O(5)	4391(1)	2421(1)	2599(1)	29(1)
O(6)	6660(1)	3318(1)	2312(1)	25(1)
O(7)	6863(1)	1402(1)	1759(1)	37(1)
C(1)	5689(2)	2897(1)	2951(1)	24(1)
C(2)	6320(2)	1903(1)	3532(1)	22(1)
C(3)	7885(2)	2241(2)	3823(1)	27(1)
C(4)	8026(2)	3579(2)	4044(1)	33(1)
C(5)	6939(2)	4367(2)	3910(1)	35(1)
C(6)	5461(2)	3965(2)	3556(1)	30(1)
C(7)	4616(2)	3320(2)	4263(1)	29(1)
C(8)	5265(2)	1978(1)	4271(1)	23(1)
C(9)	4142(2)	935(2)	4224(1)	25(1)
C(10)	1955(2)	1664(2)	4849(1)	32(1)
C(11)	2951(2)	3428(2)	4181(1)	37(1)
C(12)	9384(2)	834(2)	3074(1)	30(1)
C(13)	10421(2)	706(2)	2379(1)	46(1)
C(14)	3615(2)	3231(2)	2037(1)	50(1)
C(15)	7165(2)	2484(2)	1761(1)	26(1)
C(16)	8179(2)	3094(2)	1174(1)	31(1)

Table 3. Bond lengths [Å] and angles [°] for feb1108.

O(1)-C(10)	1.406(2)
O(1)-C(9)	1.4333(19)
O(2)-C(10)	1.405(2)
O(2)-C(11)	1.437(2)
O(3)-C(12)	1.348(2)
O(3)-C(3)	1.4695(19)
O(4)-C(12)	1.201(2)
O(5)-C(1)	1.397(2)
O(5)-C(14)	1.436(2)
O(6)-C(15)	1.356(2)
O(6)-C(1)	1.4494(18)
O(7)-C(15)	1.200(2)
C(1)-C(2)	1.530(2)
C(1)-C(6)	1.532(2)
C(2)-C(3)	1.533(2)
C(2)-C(8)	1.553(2)
C(3)-C(4)	1.494(2)
C(4)-C(5)	1.317(3)
C(5)-C(6)	1.512(3)
C(6)-C(7)	1.558(2)
C(7)-C(11)	1.521(3)
C(7)-C(8)	1.565(2)
C(8)-C(9)	1.521(2)
C(12)-C(13)	1.493(2)
C(15)-C(16)	1.494(2)

C(10)-O(1)-C(9)	114.34(12)
C(10)-O(2)-C(11)	113.81(14)
C(12)-O(3)-C(3)	116.01(13)
C(1)-O(5)-C(14)	115.64(13)
C(15)-O(6)-C(1)	118.85(12)
O(5)-C(1)-O(6)	110.26(12)
O(5)-C(1)-C(2)	107.12(12)
O(6)-C(1)-C(2)	115.76(13)
O(5)-C(1)-C(6)	114.39(14)
O(6)-C(1)-C(6)	108.33(12)
C(2)-C(1)-C(6)	100.86(13)
C(1)-C(2)-C(3)	110.73(13)

C(1)-C(2)-C(8)	102.09(12)
C(3)-C(2)-C(8)	109.64(13)
O(3)-C(3)-C(4)	105.95(13)
O(3)-C(3)-C(2)	111.63(12)
C(4)-C(3)-C(2)	112.19(14)
C(5)-C(4)-C(3)	121.69(16)
C(4)-C(5)-C(6)	122.24(16)
C(5)-C(6)-C(1)	109.08(14)
C(5)-C(6)-C(7)	107.35(14)
C(1)-C(6)-C(7)	102.13(13)
C(11)-C(7)-C(6)	114.14(15)
C(11)-C(7)-C(8)	116.50(14)
C(6)-C(7)-C(8)	103.22(13)
C(9)-C(8)-C(2)	110.41(13)
C(9)-C(8)-C(7)	115.64(13)
C(2)-C(8)-C(7)	106.26(12)
O(1)-C(9)-C(8)	112.28(13)
O(2)-C(10)-O(1)	114.24(14)
O(2)-C(11)-C(7)	111.50(15)
O(4)-C(12)-O(3)	123.49(16)
O(4)-C(12)-C(13)	124.96(17)
O(3)-C(12)-C(13)	111.55(16)
O(7)-C(15)-O(6)	124.66(15)
O(7)-C(15)-C(16)	124.84(16)
O(6)-C(15)-C(16)	110.48(14)