

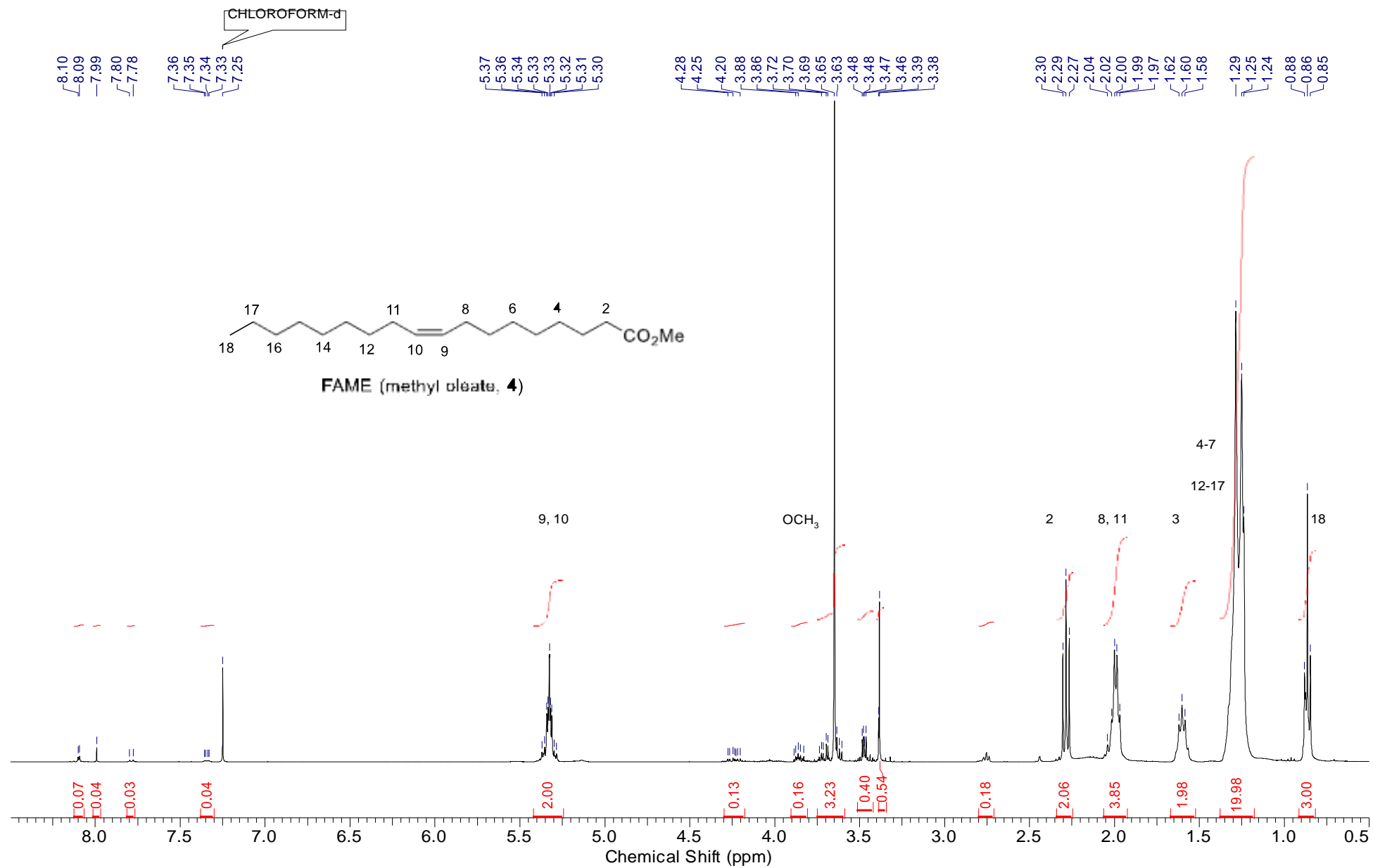
Synthesis of allyl alcohol as a method to valorise glycerol from the biodiesel production

Michael Wormann,^a and Martin E. Maier ^{*a}

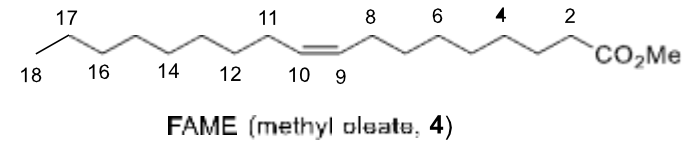
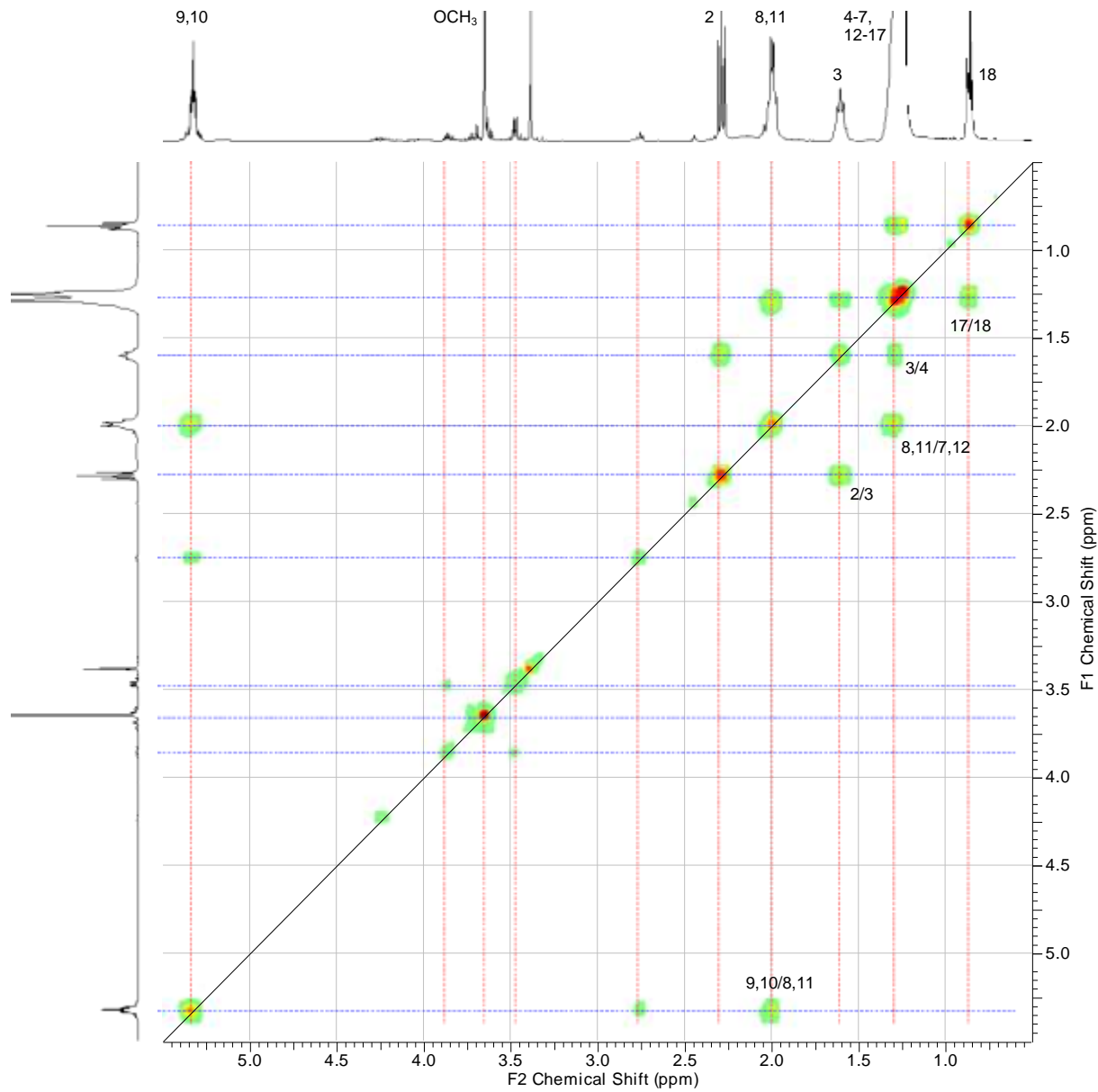
^a Eberhard Karls Universität Tübingen, Institut für Organische Chemie, Auf der Morgenstelle 18, 72976 Tübingen, Germany

Table of Contents

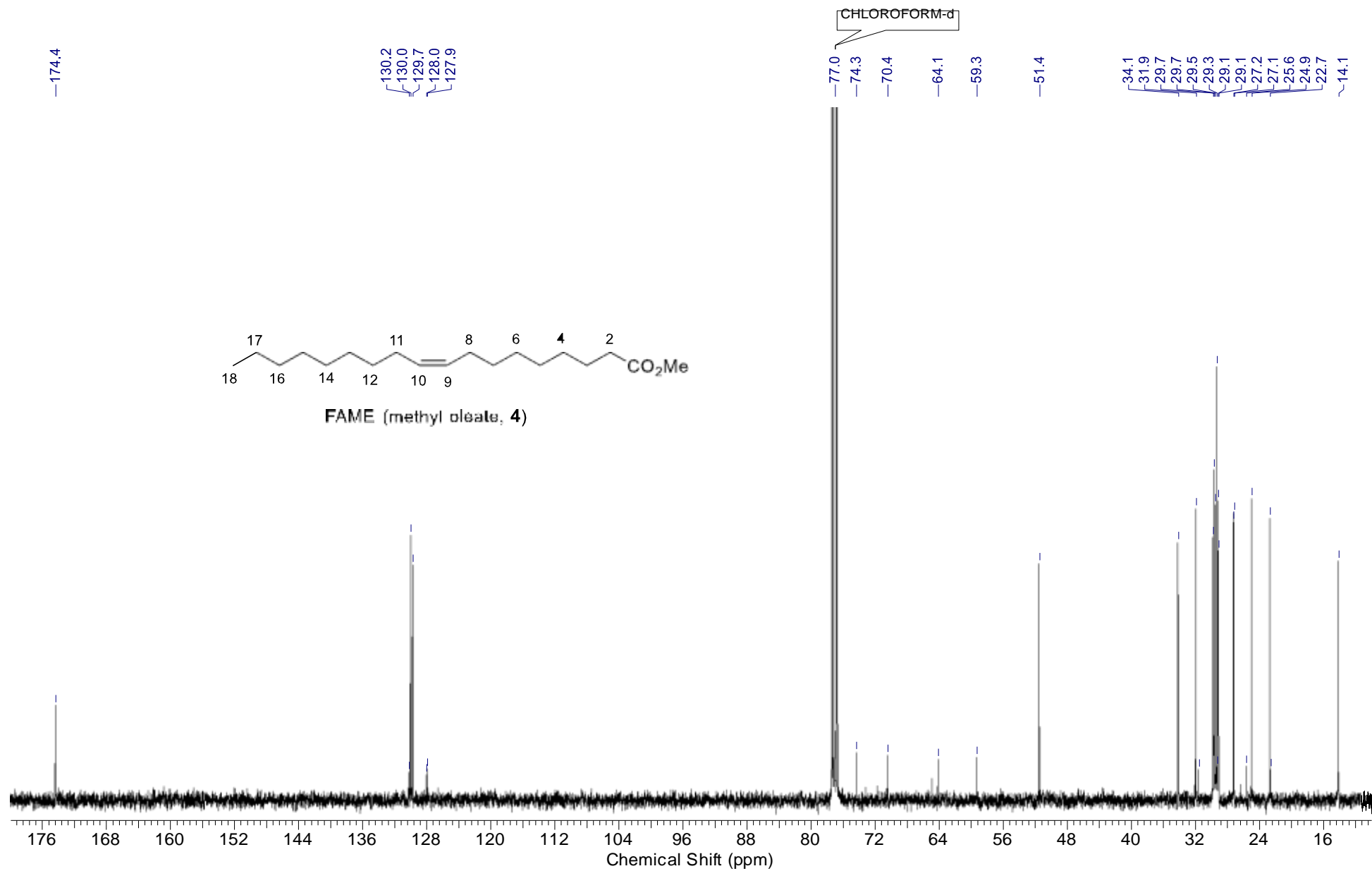
NMR-Spectra	S2
-------------------	----



¹H NMR (400 MHz) of FAME (**4**) in CDCl₃ (0.5 – 8.5 ppm)



H-H COSY (400 MHz) spectrum of FAME (**4**) in CDCl₃ (0.5 – 5.5 ppm)



¹³C NMR (100 MHz) spectrum of FAME (4) in CDCl₃ (10 – 180 ppm)

—34.1

—31.9

—31.5

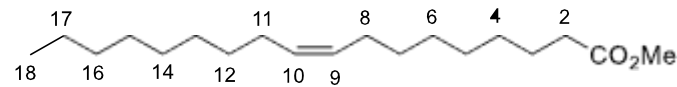
∫29.7
—29.7
—29.5
—29.3
29.1
29.1
29.1

∫27.2
—27.1

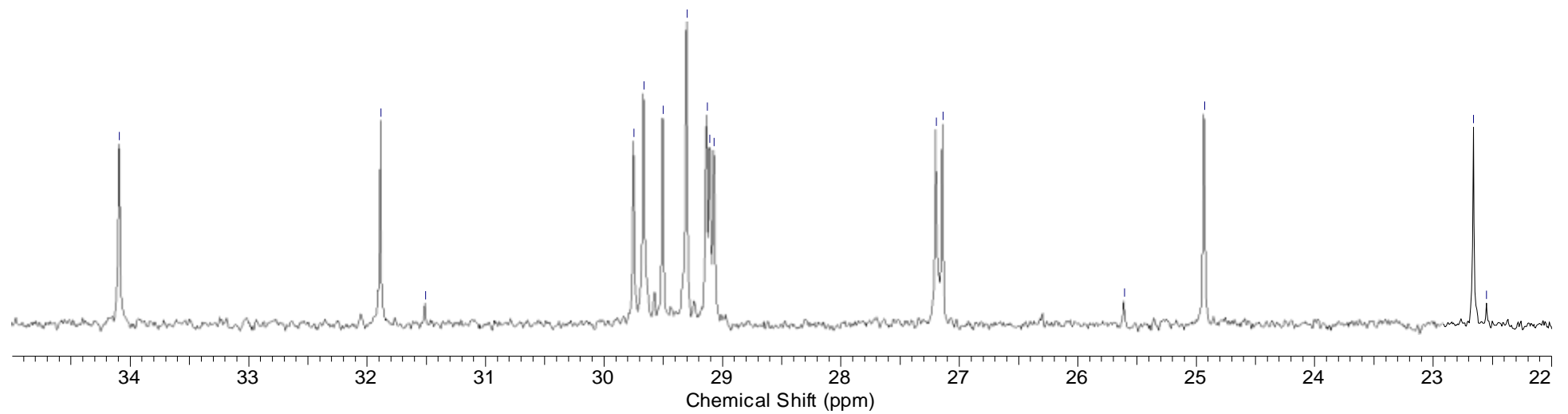
—25.6

—24.9

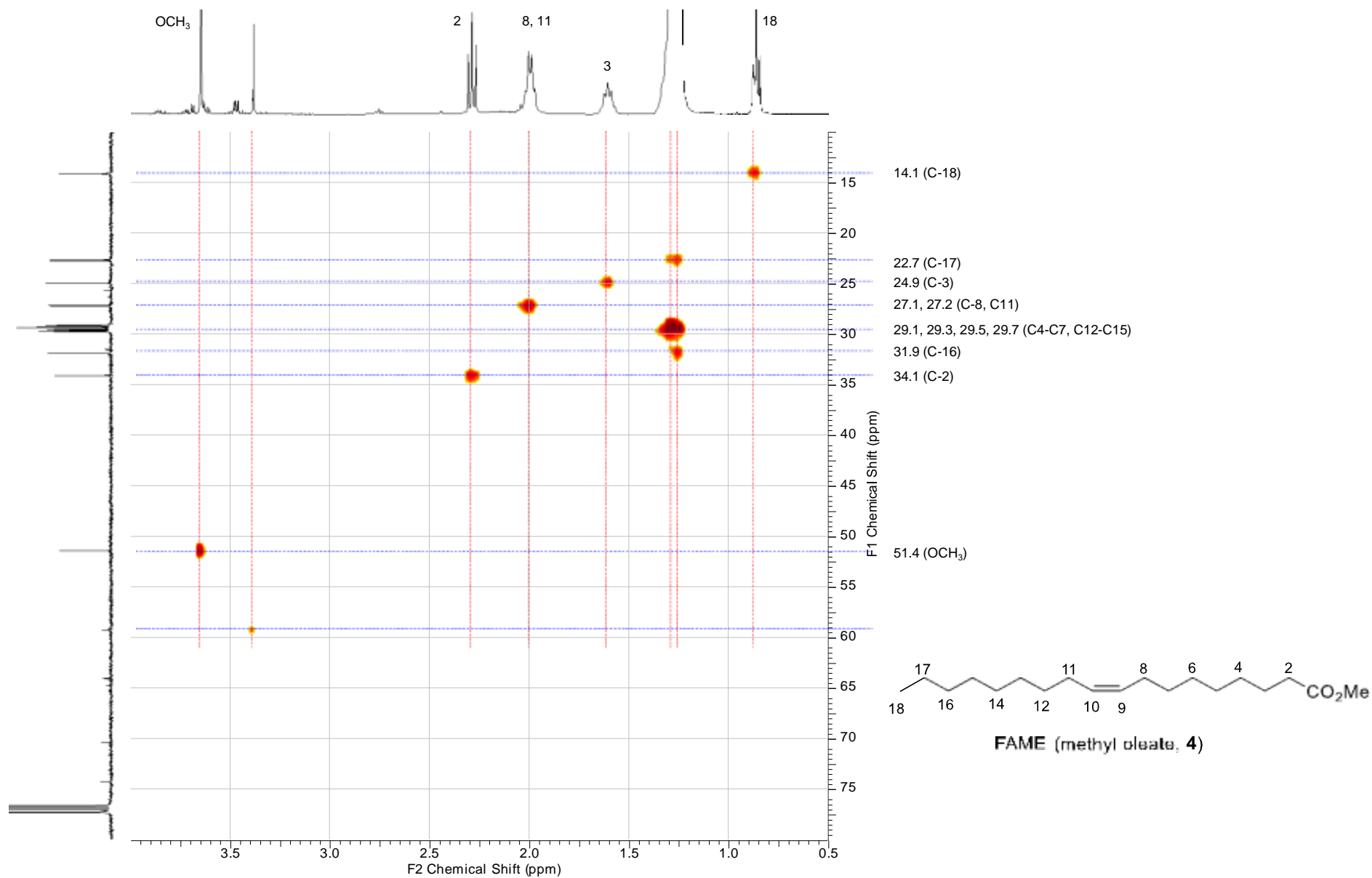
—22.7
~22.6



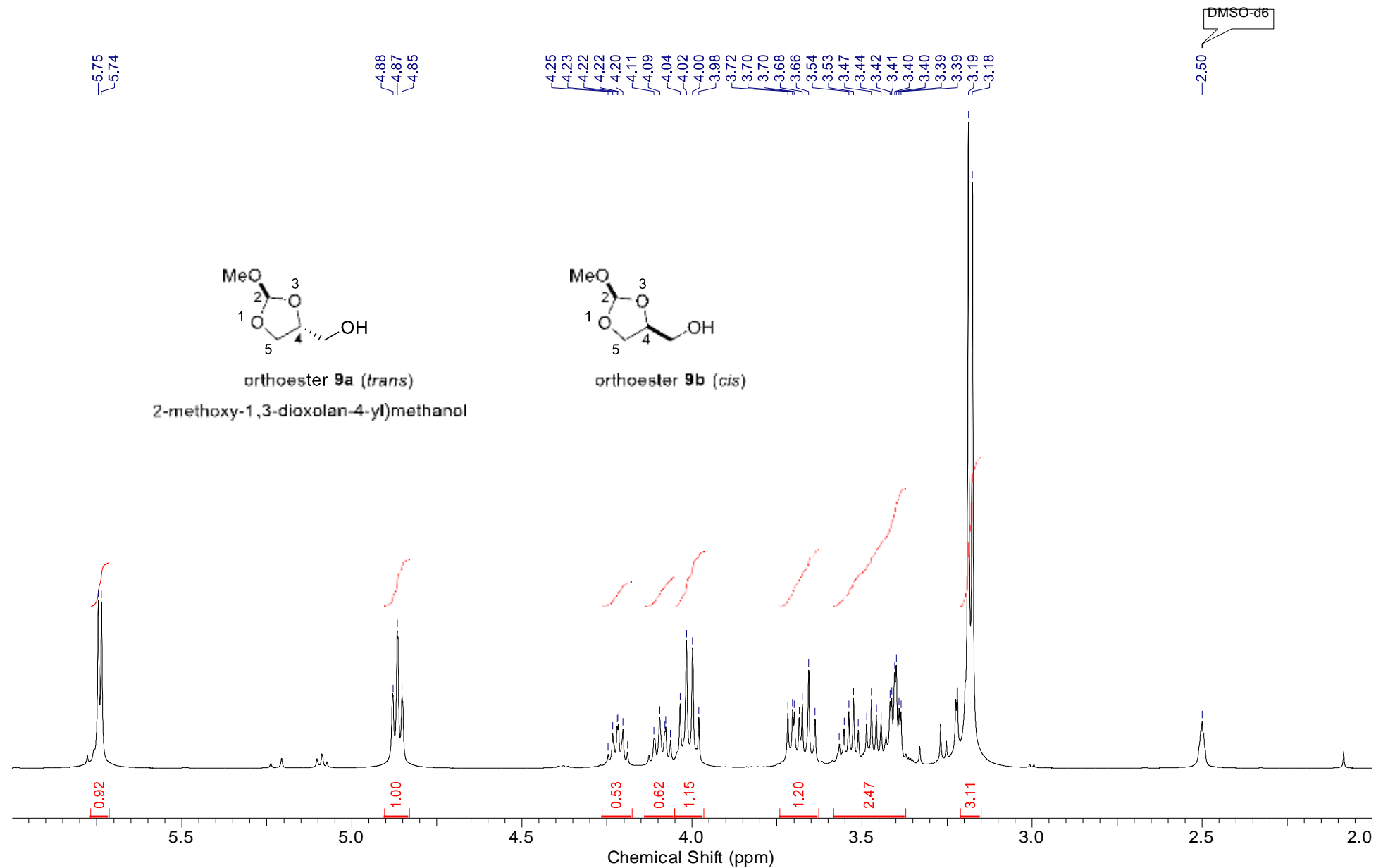
FAME (methyl oleate, 4)



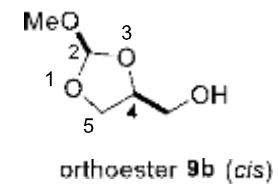
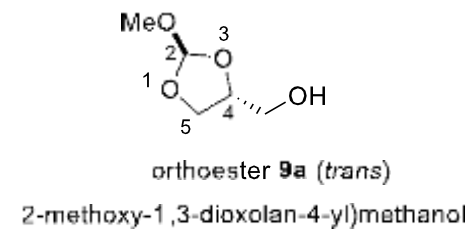
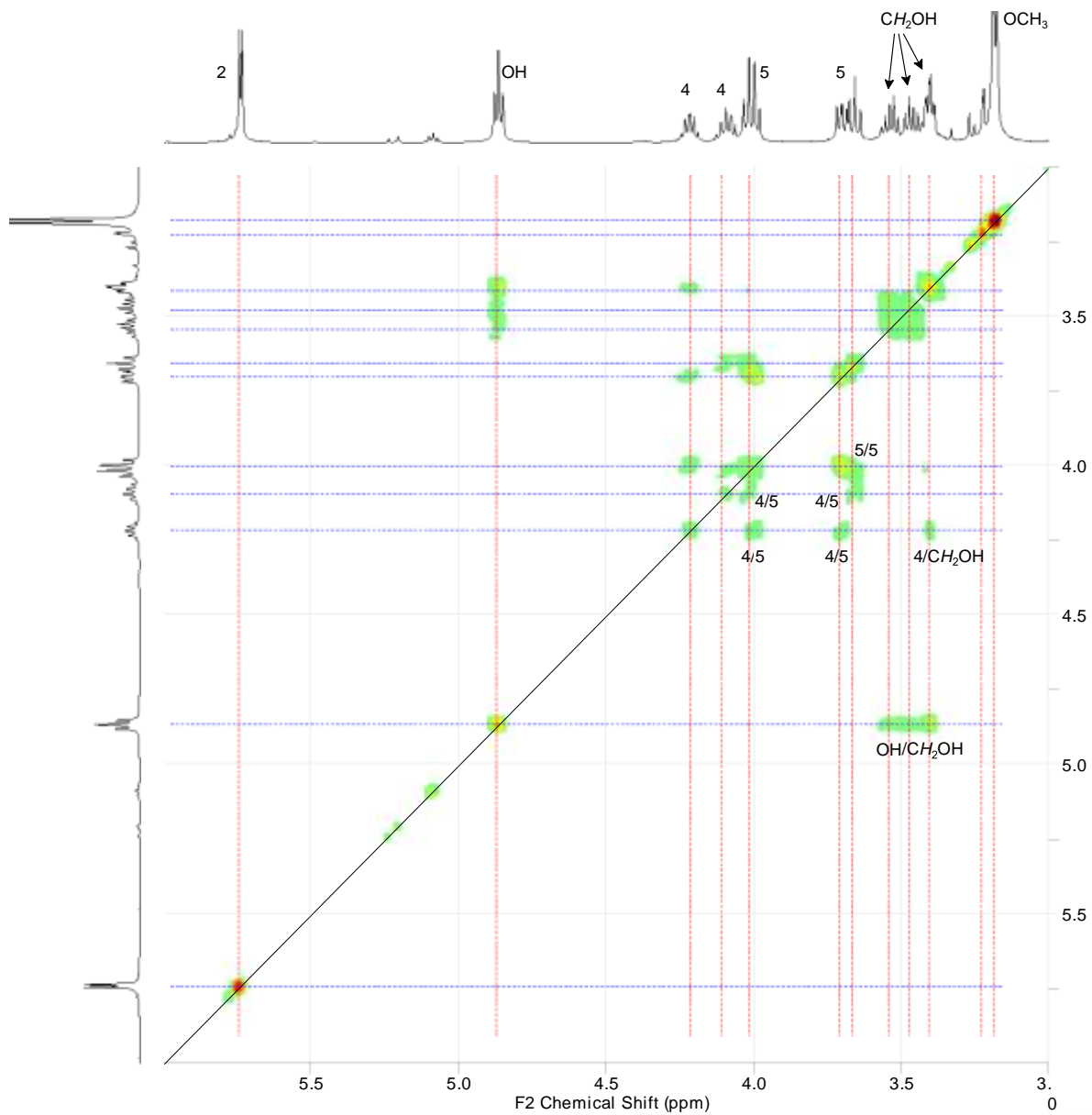
¹³C NMR (100 MHz) spectrum of FAME (4) in CDCl₃ (22 – 35 ppm)



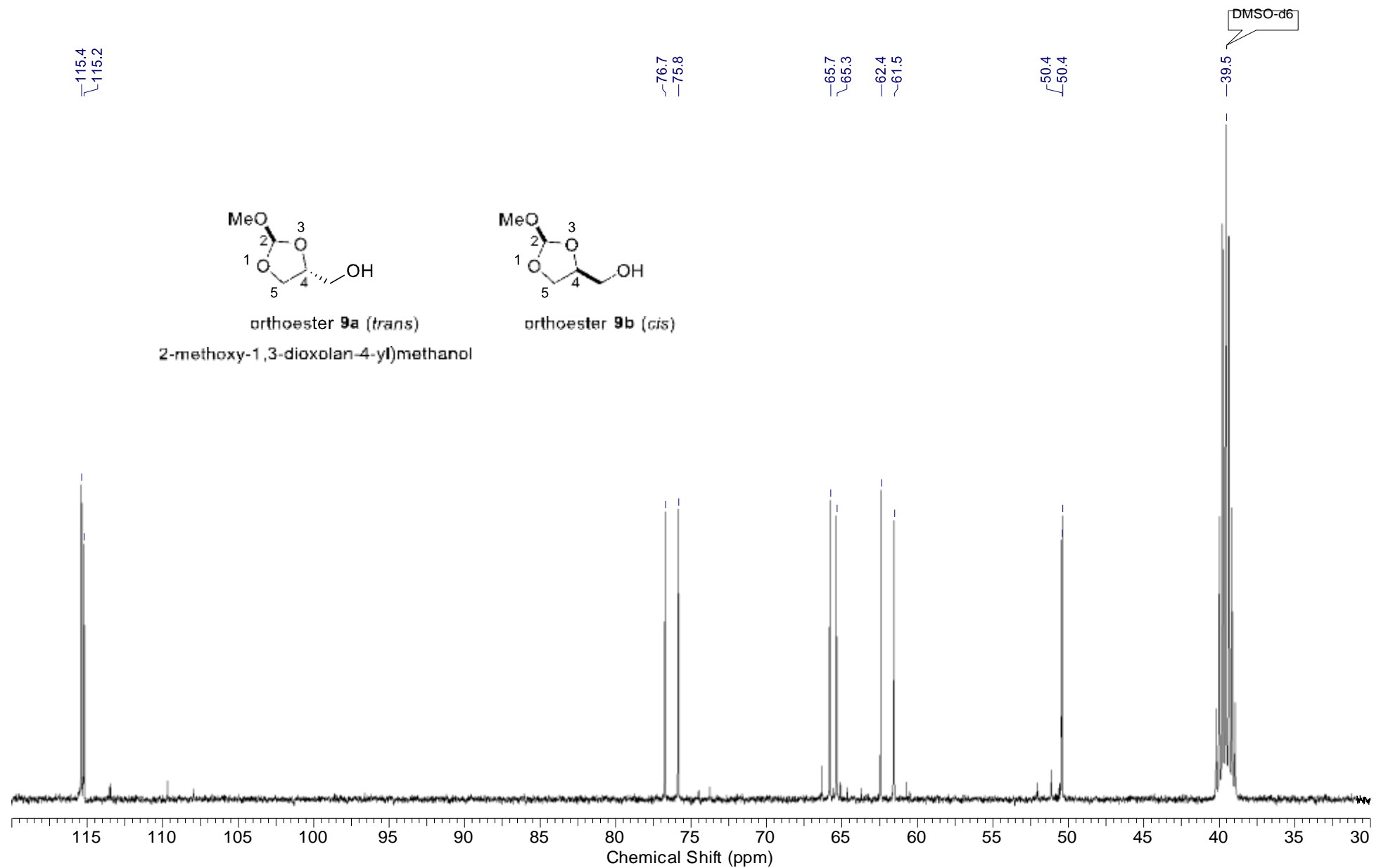
HSQC spectrum of FAME (**4**) in CDCl₃ (0.5 – 4.0, 10 – 80 ppm)



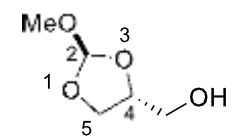
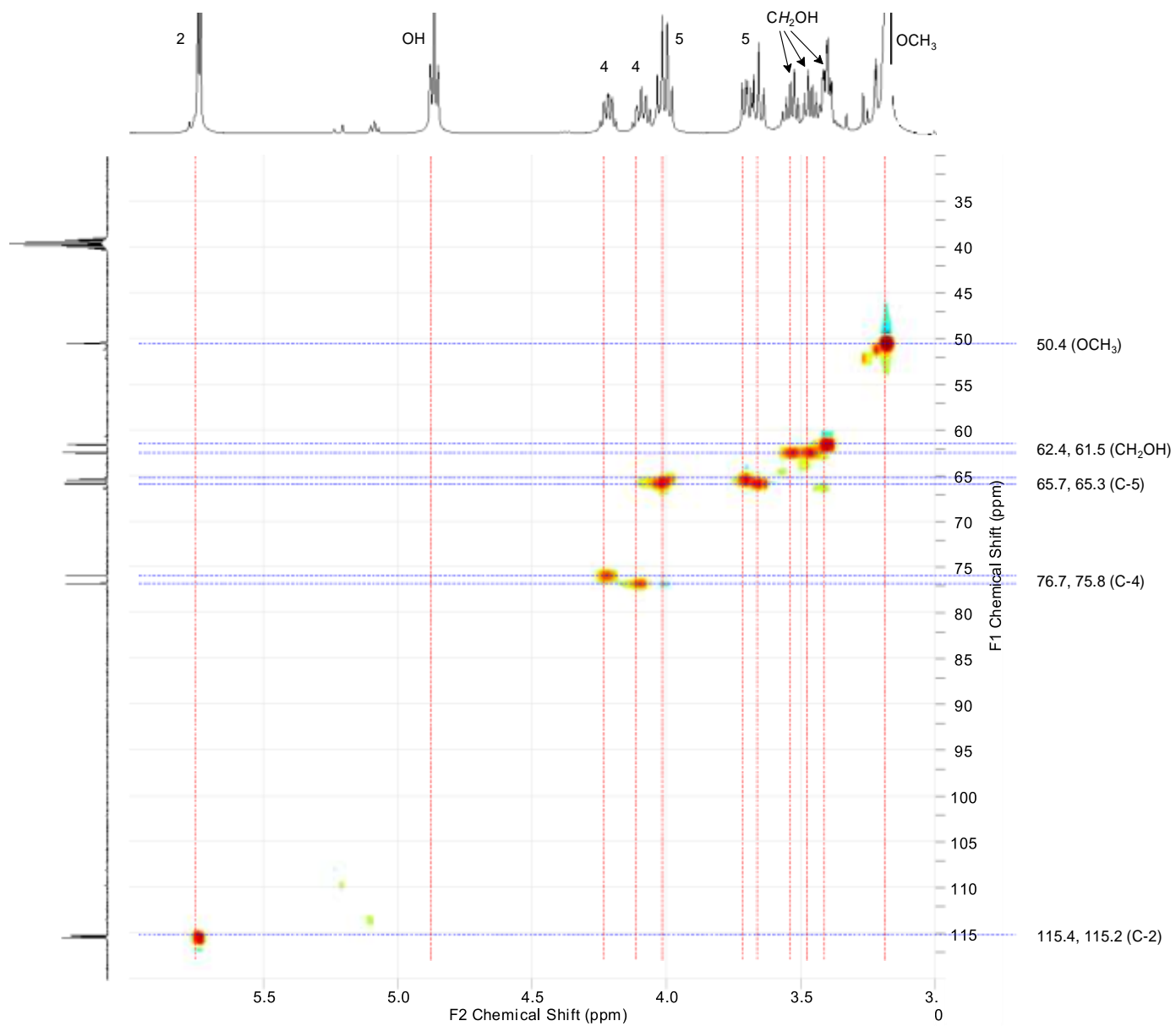
¹H NMR (400 MHz) spectrum of orthoesters **9a** (*trans*) and **9b** (*cis*) in DMSO-d₆ (2.0 – 6.0 ppm)



H-H COSY (400 MHz) spectrum of orthoesters **9a** (*trans*) and **9b** (*cis*) in DMSO-d₆ (3.0 – 6.0 ppm)

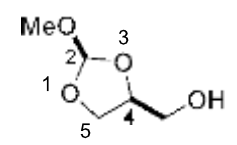


^{13}C NMR (100 MHz) spectrum of orthoesters **9a** (*trans*) and **9b** (*cis*) in DMSO-d₆ (30 – 120 ppm)



orthoester **9a** (*trans*)

2-methoxy-1,3-dioxolan-4-yl)methanol



orthoester **9b** (*cis*)

HSQC spectrum of orthoesters **9a** (*trans*) and **9b** (*cis*) in DMSO- d_6 (3.0 – 6.0, 30 – 120 ppm)
S10

Display Report

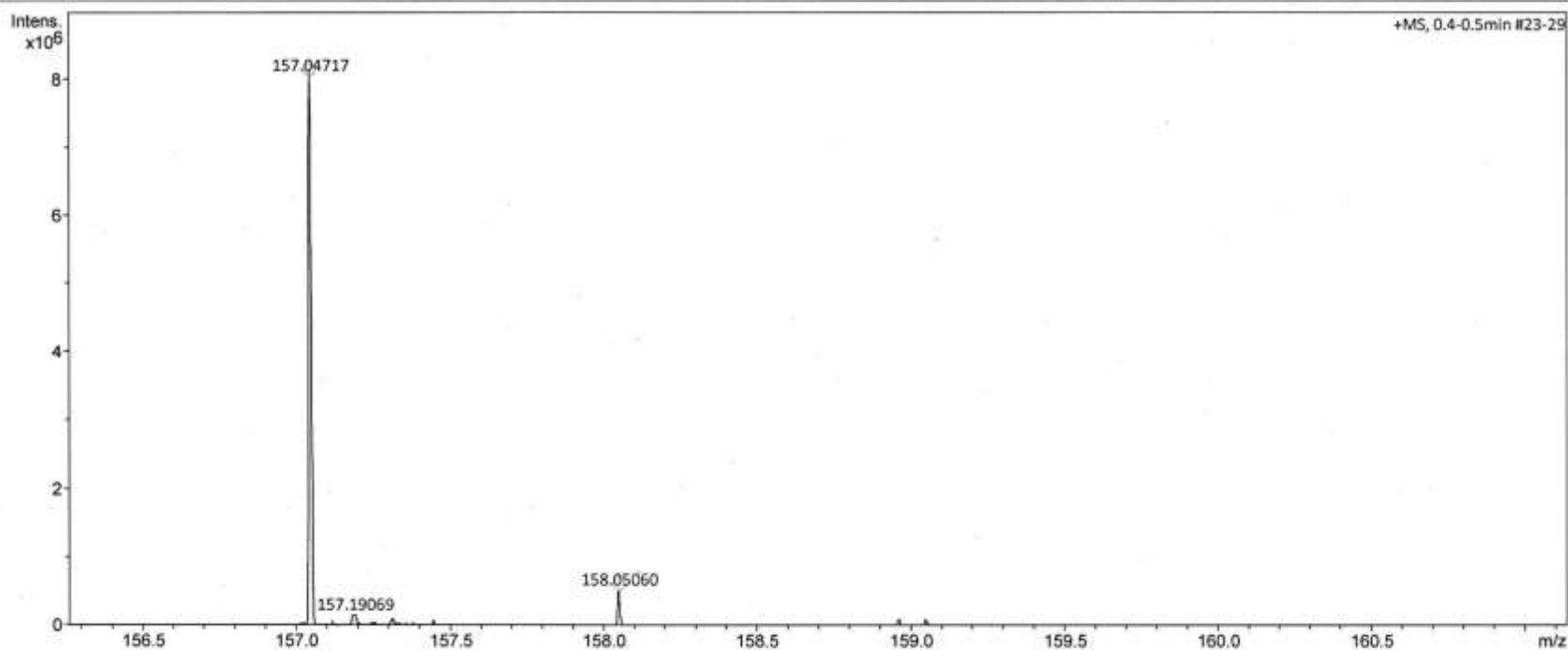
Analysis Info

Analysis Name D:\Data\oi\Wormann_71-20_GB2_01_31193.d
Method fia_ms_80-1000_pos_neu.m
Sample Name Wormann_71-20
Comment

Acquisition Date 4/26/2019 11:45:29 AM
Operator BDAL@DE
Instrument maXis 288882.21253

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	80 m/z	Set End Plate Offset	-500 V	Set Dry Gas	6.0 l/min
Scan End	1000 m/z	Set Charging Voltage	0 V	Set Divert Valve	Waste
		Set Corona	0 nA	Set APCI Heater	0 °C



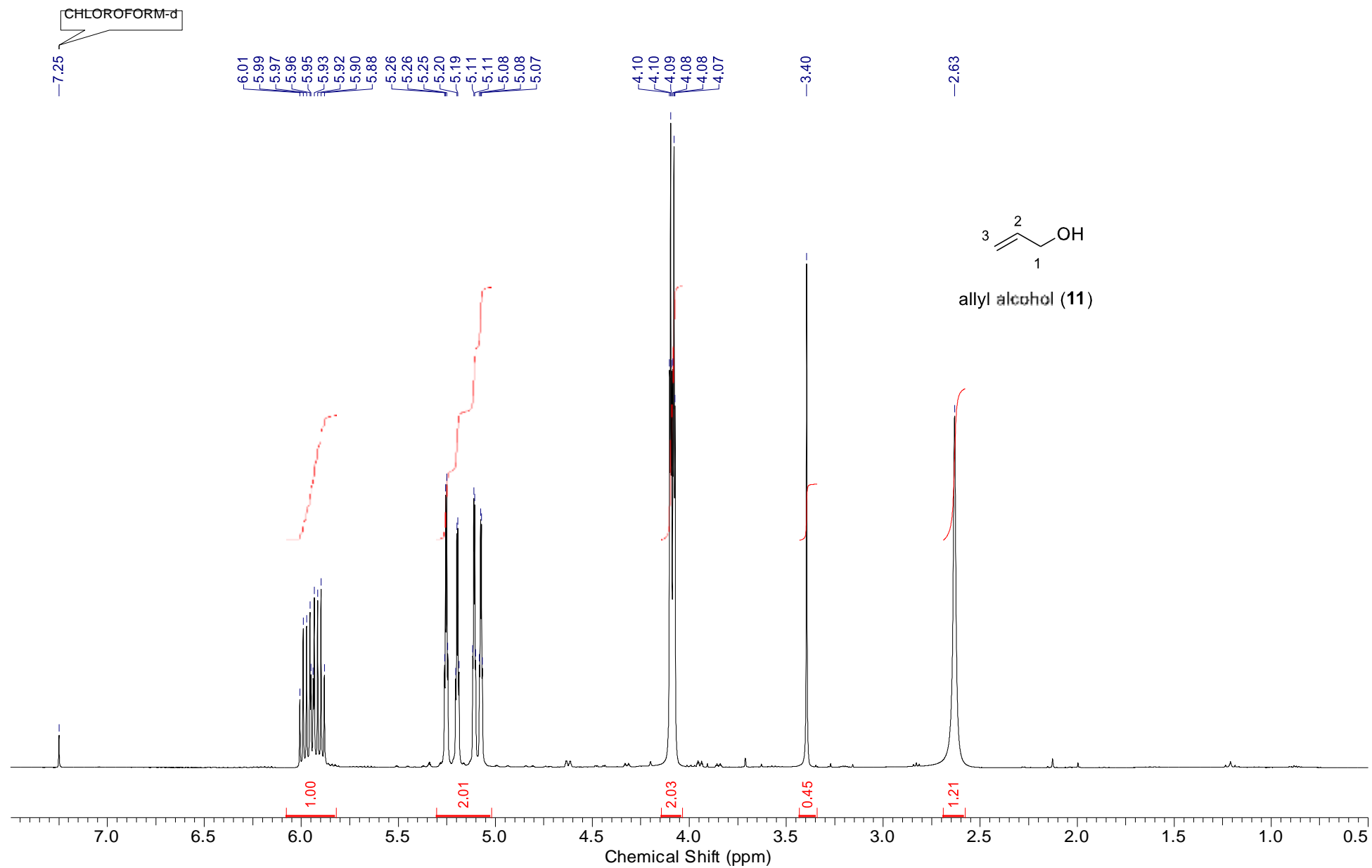
Wormann_71-20_GB2_01_31193.d
Bruker Compass DataAnalysis 4.2

printed: 4/26/2019 12:24:04 PM

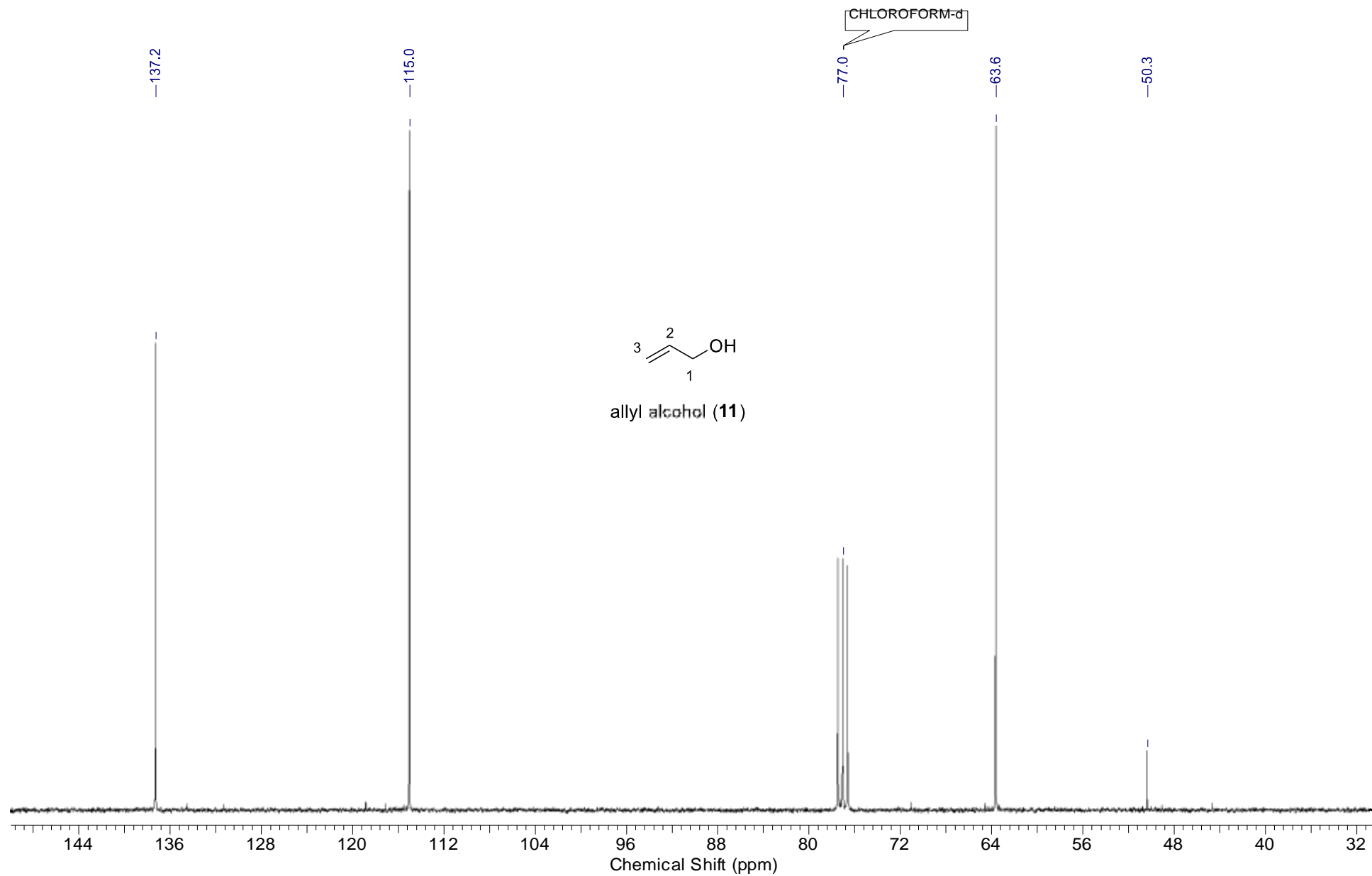
by: BDAL@DE

Page 1 of 1

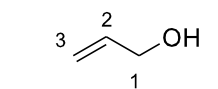
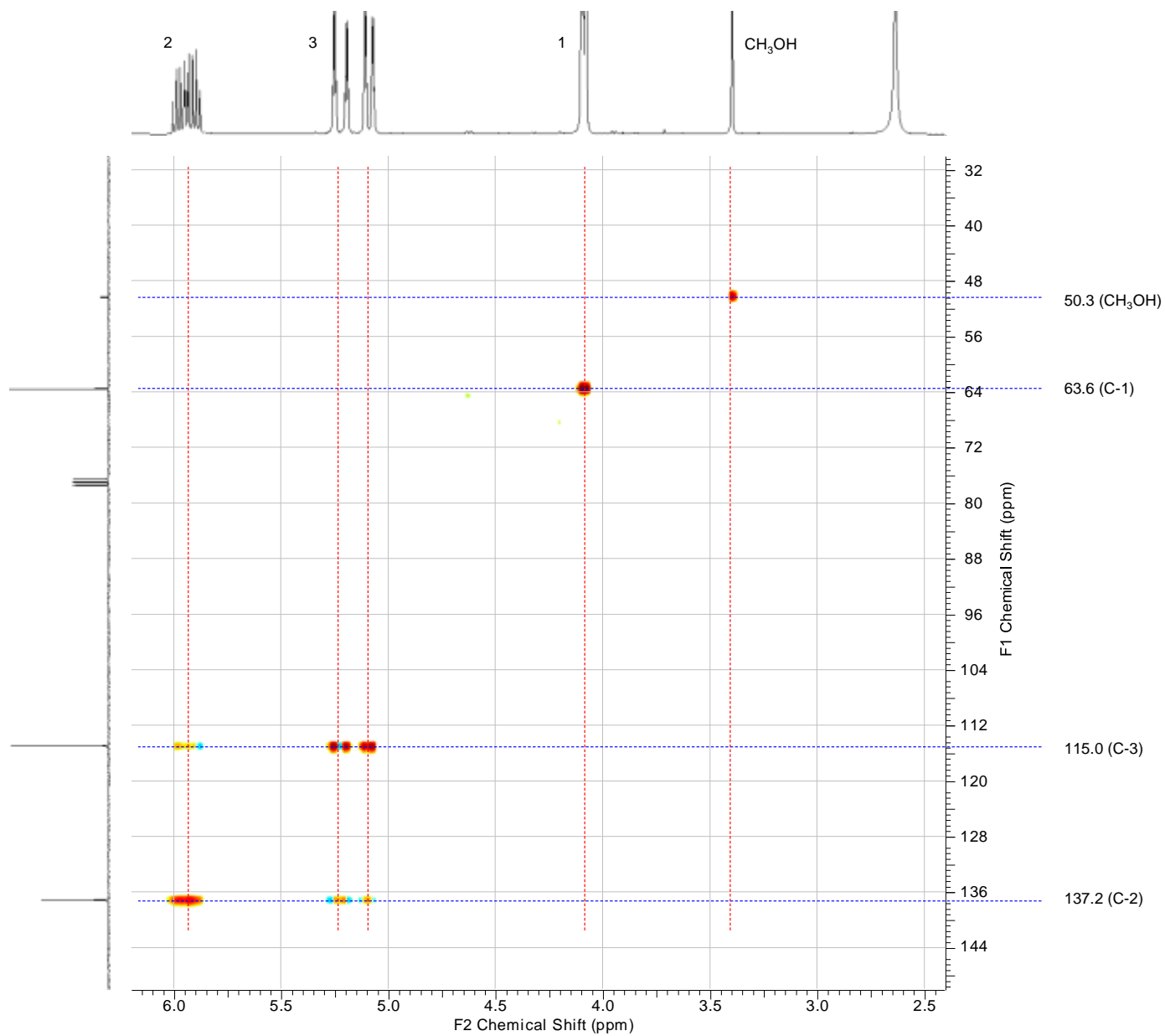
HRMS (ESI-TOF) of cyclic orthoester **9** (mixture of **9a** and **9b**).



^1H NMR (400 MHz) spectrum of allyl alcohol (**11**) in CDCl_3 (0.5 – 7.5 ppm)



^{13}C NMR (100 MHz) spectrum of allyl alcohol (**11**) in CDCl_3 (30 – 150 ppm)



allyl alcohol (**11**)

HSQC spectrum of allyl alcohol (**11**) in CDCl₃ (2.4 – 6.2; 30 – 150 ppm)