Spinsolve
for polymer chemistry:

Characterization of monomers and polymers

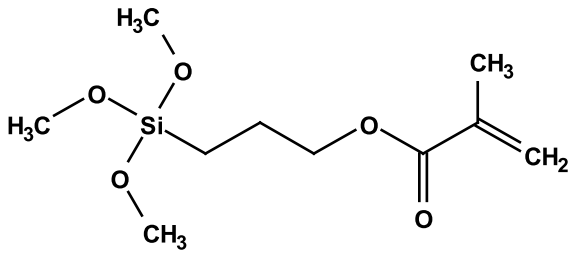
Summary

- Characterisation of monomers: Structure confirmation
- Characterisation of a RAFT initiator
- Molecular weight determination
- Chemical structure
- Comparisons with a 400 MHz

NMR study of

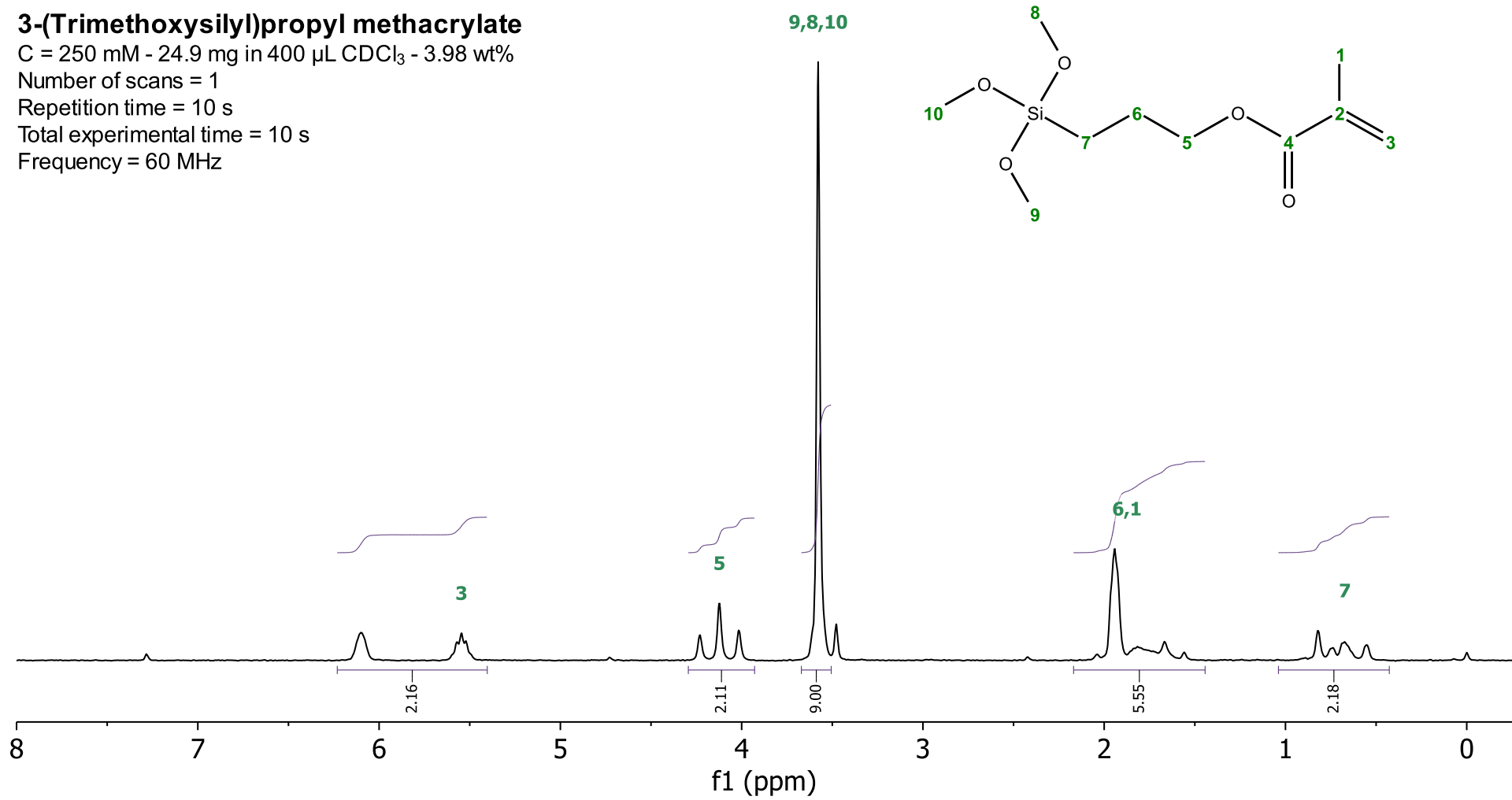
3-(Trimethoxysilyl)propyl methacrylate

on a Spinsolve Carbon 60 MHz



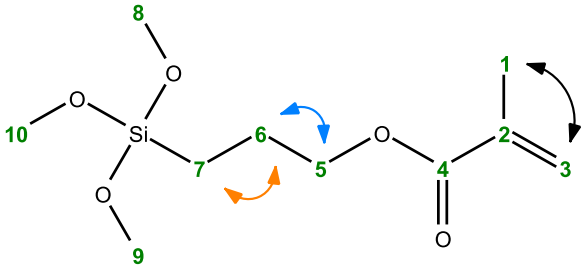
3-(Trimethoxysilyl)propyl methacrylate; 13 wt%; 1D 1H

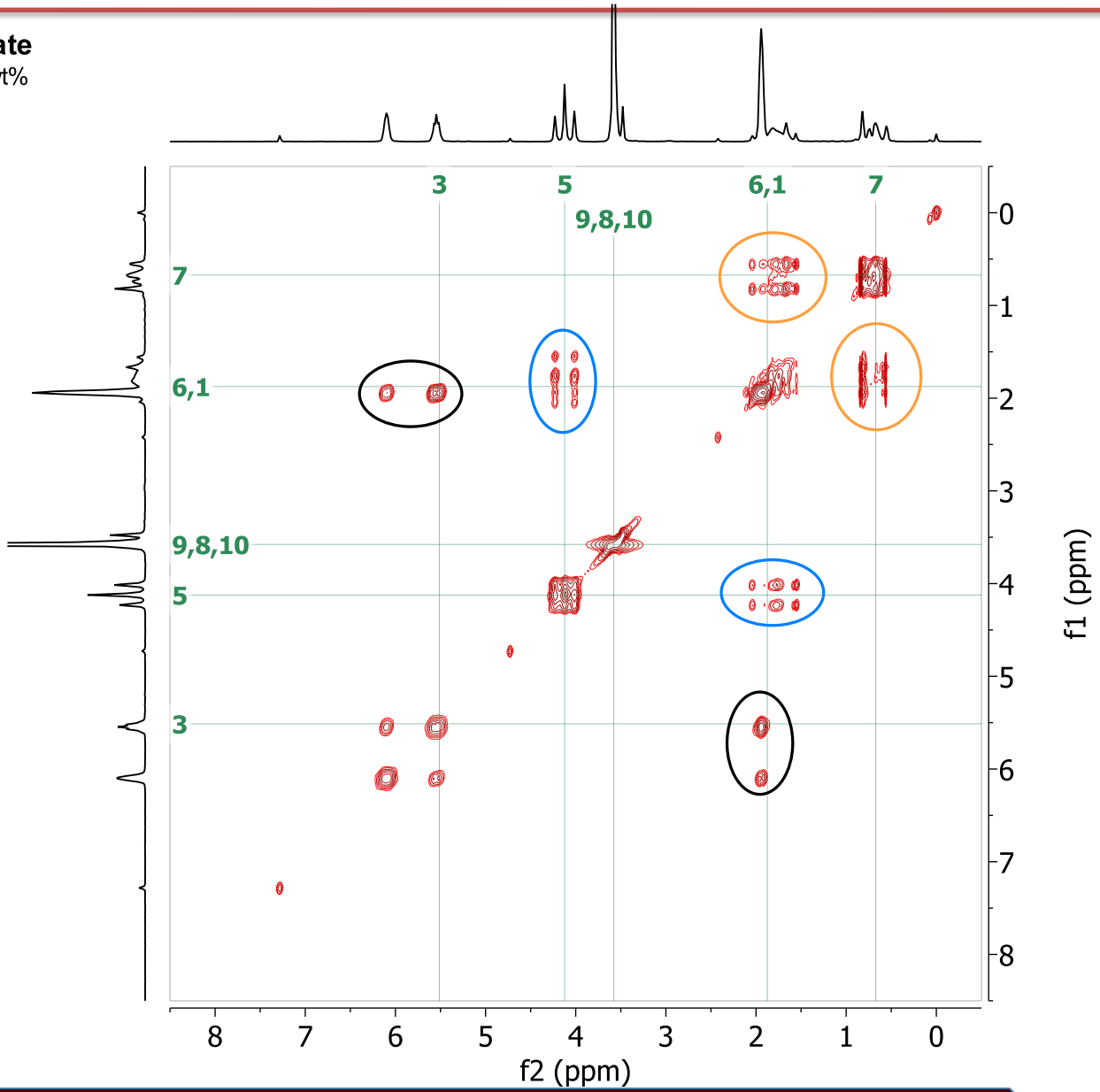
3-(Trimethoxysilyl)propyl methacrylate
C = 250 mM - 24.9 mg in 400 µL $CDCl_3$ - 3.98 wt%
Number of scans = 1
Repetition time = 10 s
Total experimental time = 10 s
Frequency = 60 MHz



3-(Trimethoxysilyl)propyl methacrylate; 13 wt%; 2D COSY

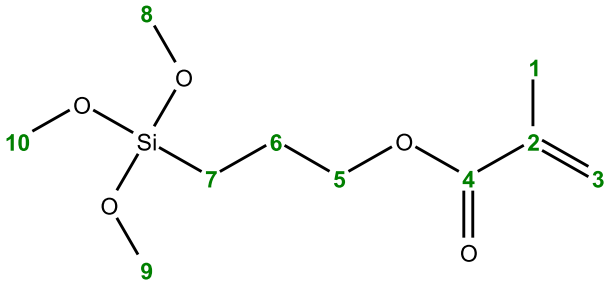
3-(Trimethoxysilyl)propyl methacrylate
C = 250 mM - 24.9 mg in 400 µL CDCl3 - 3.98 wt%
Number of scans = 1
T1 increments = 256
Total experimental time = 10 min
Frequency = 60 MHz

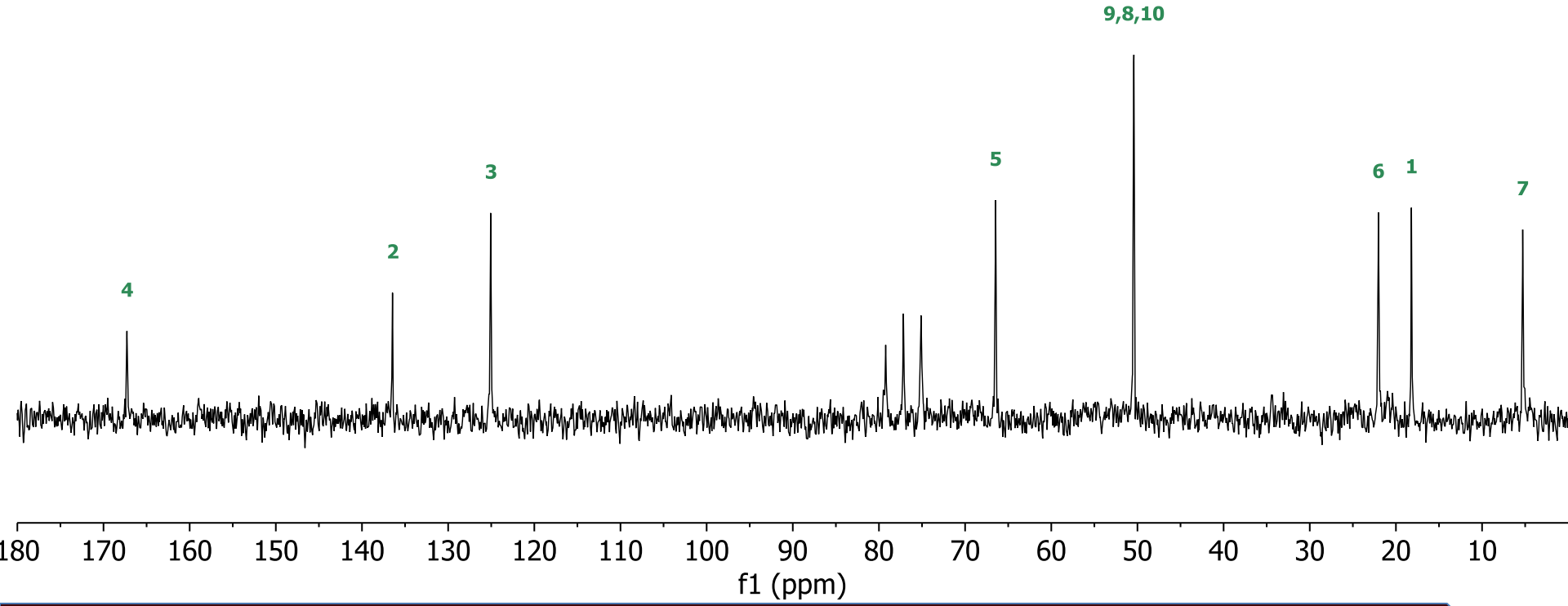




3-(Trimethoxysilyl)propyl methacrylate; 13 wt%; 1D 13C

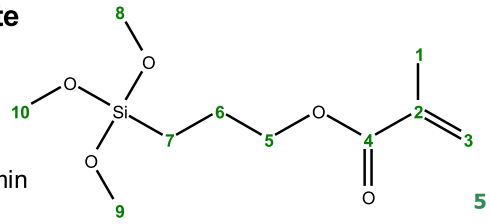
3-(Trimethoxysilyl)propyl methacrylate
C = 1 M - 99.5 mg in 400 µL CDCl3 - 14.2 wt%
number of scans = 1024
repetition time = 5 s
pulse angle = 30 °
measurement time = 85 min
Frequency = 15 MHz



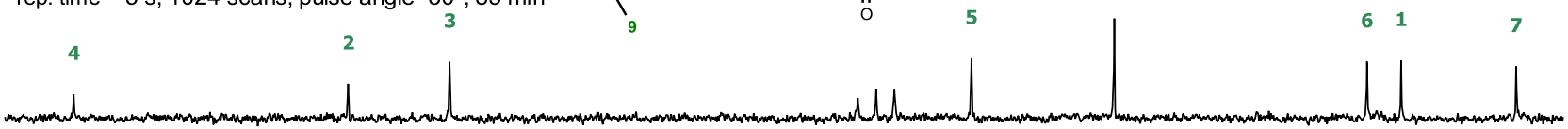


3-(Trimethoxysilyl)propyl methacrylate; 13 wt%; 1D 13C & DEPT

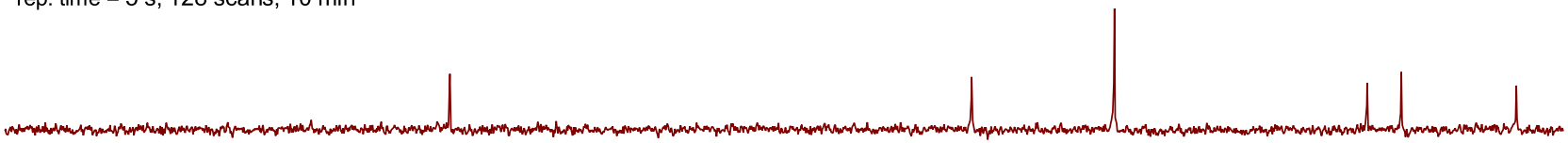
3-(Trimethoxysilyl)propyl methacrylate
C = 1 M - 99.5 mg in 400 µL CDCl3 - 14.2 wt%
Solvent = $CDCl_3$
Frequency = 15 MHz



1D Carbon
rep. time = 5 s, 1024 scans, pulse angle=30°, 85 min

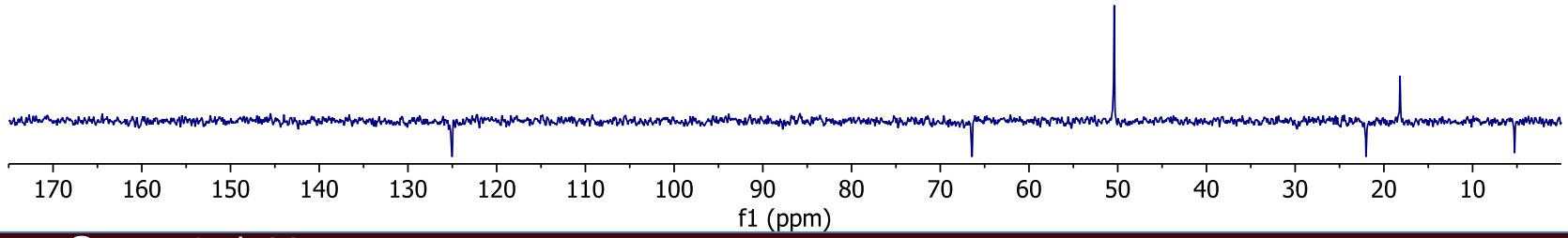


DEPT 45
rep. time = 5 s, 128 scans, 10 min



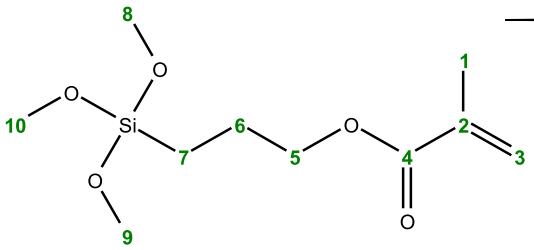
DEPT 90
rep. time = 5 s, 128 scans, 10 min

DEPT 135
rep. time = 5 s, 128 scans, 10 min



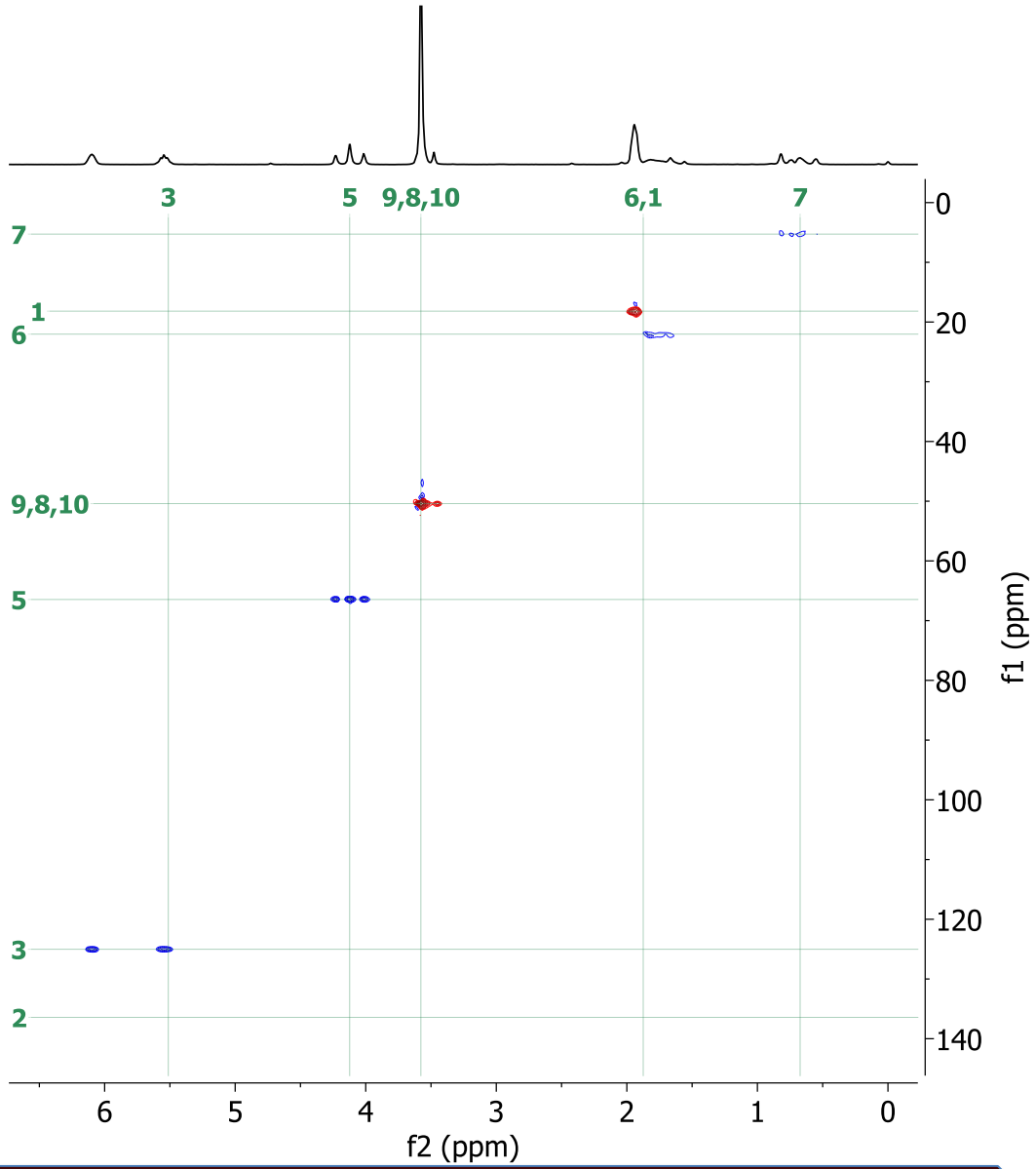
3-(Trimethoxysilyl)propyl methacrylate; 13 wt%; HSQC-ME

3-(Trimethoxysilyl)propyl methacrylate
C = 1 M - 99.5 mg in 400 µL CDCl3 - 14.2 wt%
number of scans = 4
repetition time = 2 s
T1 increments = 128
measurement time = 34 min
Frequency = 15 MHz



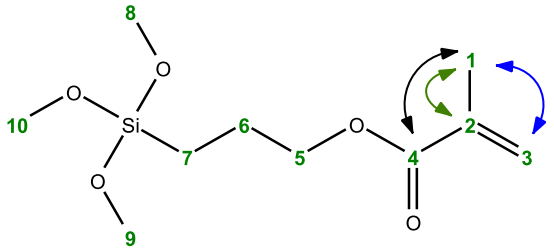
Red = CH and CH_3

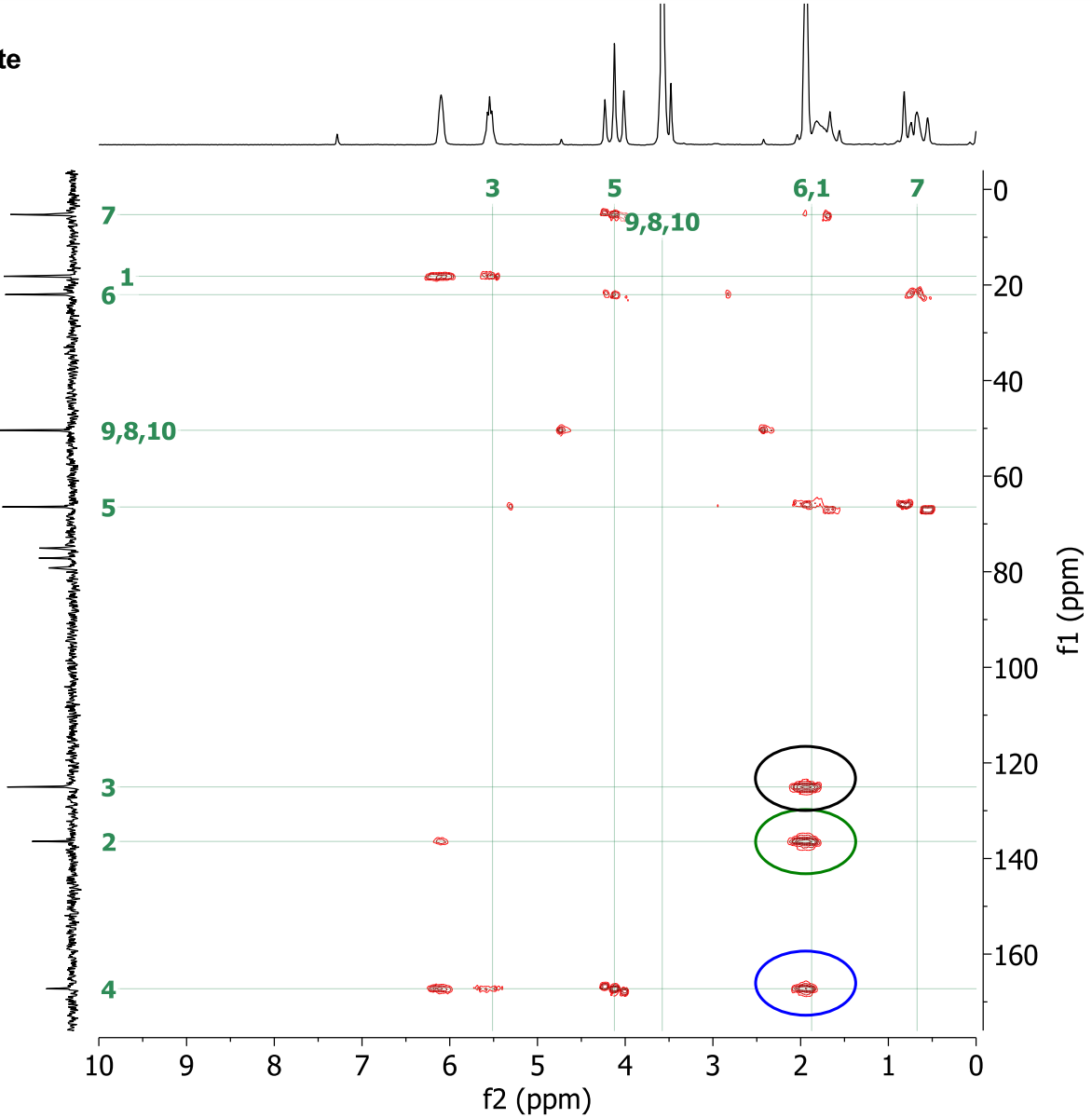
Blue = CH_2



3-(Trimethoxysilyl)propyl methacrylate; 13 wt%; HMBC

3-(Trimethoxysilyl)propyl methacrylate
C = 1 M - 99.5 mg in 400 µL CDCl3 - 14.2 wt%
number of scans = 16
repetition time = 2 s
measurement time = 68 min
Frequency = 15 MHz

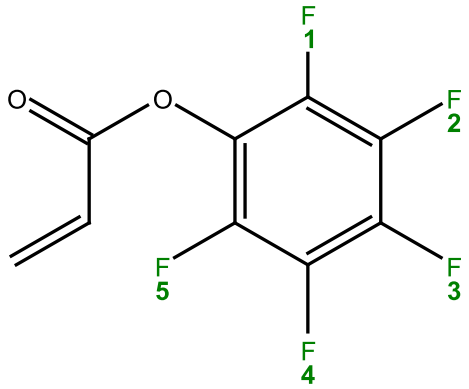




NMR study of

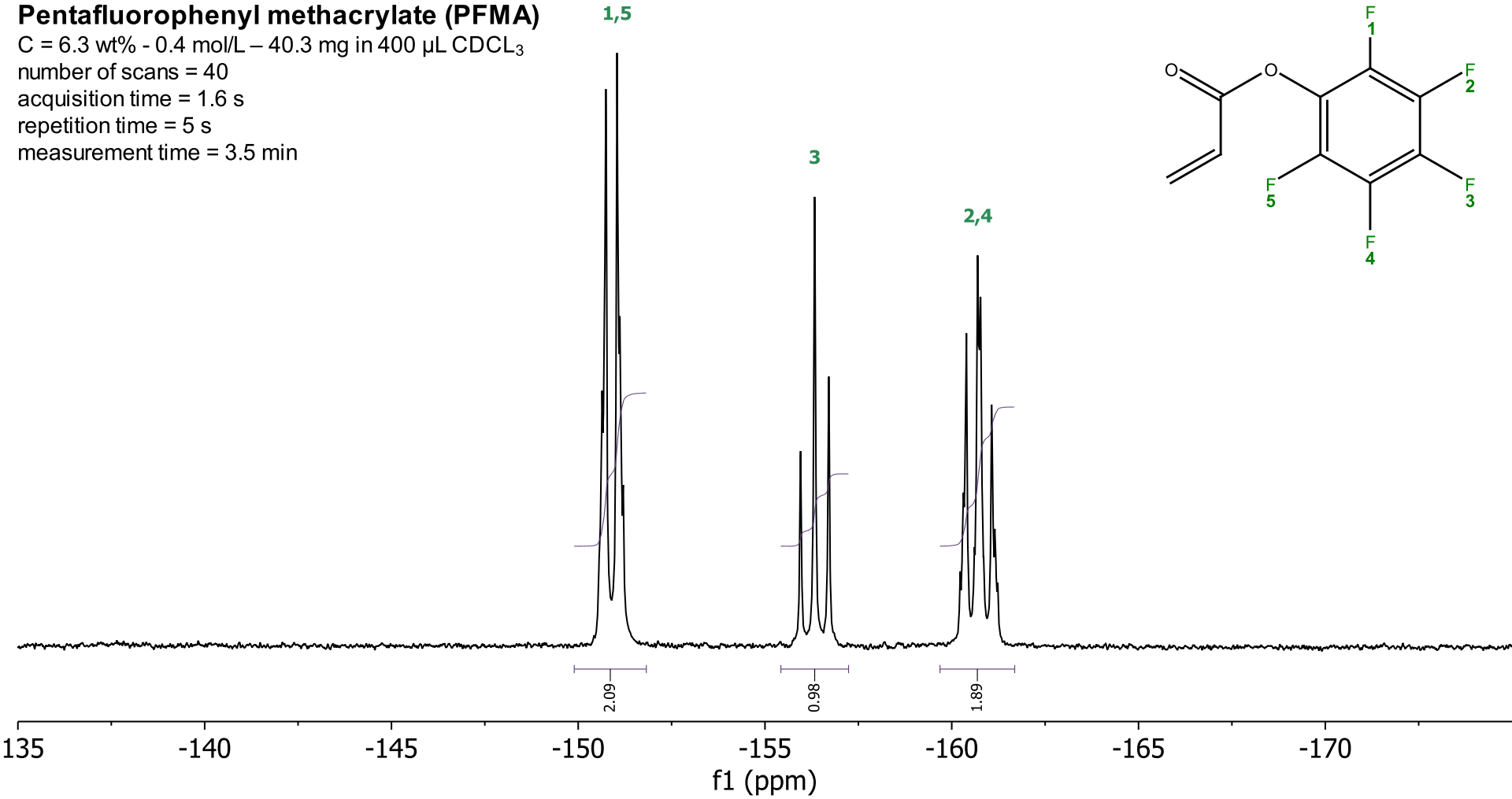
Pentafluorophenyl methacrylate (PFMA)

on a Spinsolve Carbon 60 MHz



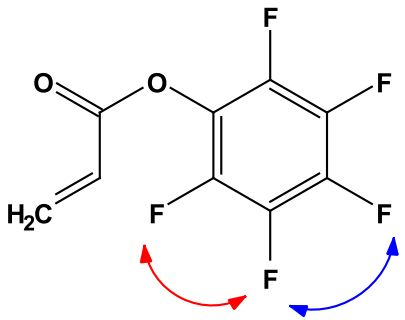
Pentafluorophenyl methacrylate (PFMA); 1D 19F

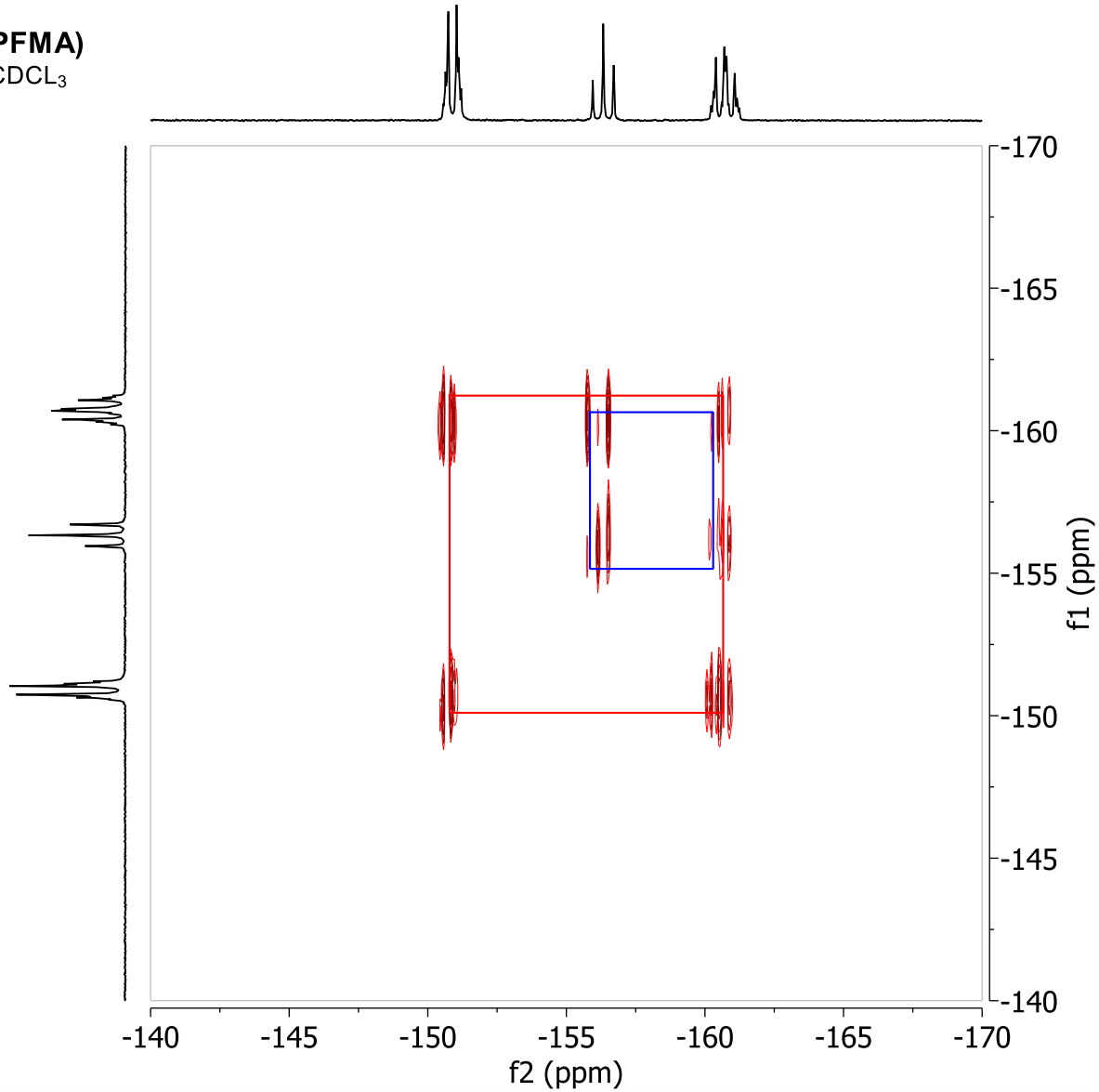
Pentafluorophenyl methacrylate (PFMA)
C = 6.3 wt% - 0.4 mol/L – 40.3 mg in 400 µL $CDCL_3$
number of scans = 40
acquisition time = 1.6 s
repetition time = 5 s
measurement time = 3.5 min



Pentafluorophenyl methacrylate (PFMA); 2D F-F COSY

Pentafluorophenyl methacrylate (PFMA)
C = 6.3 wt% - 0.4 mol/L – 40.3 mg in 400 µL $CDCL_3$
number of scans = 1
T1 increments = 256
total experimental time = 10 min

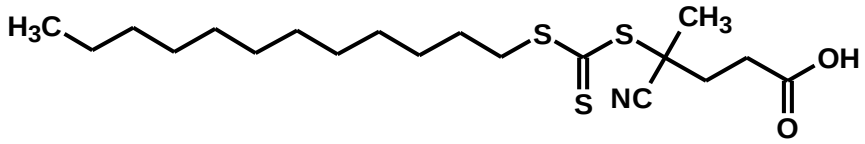




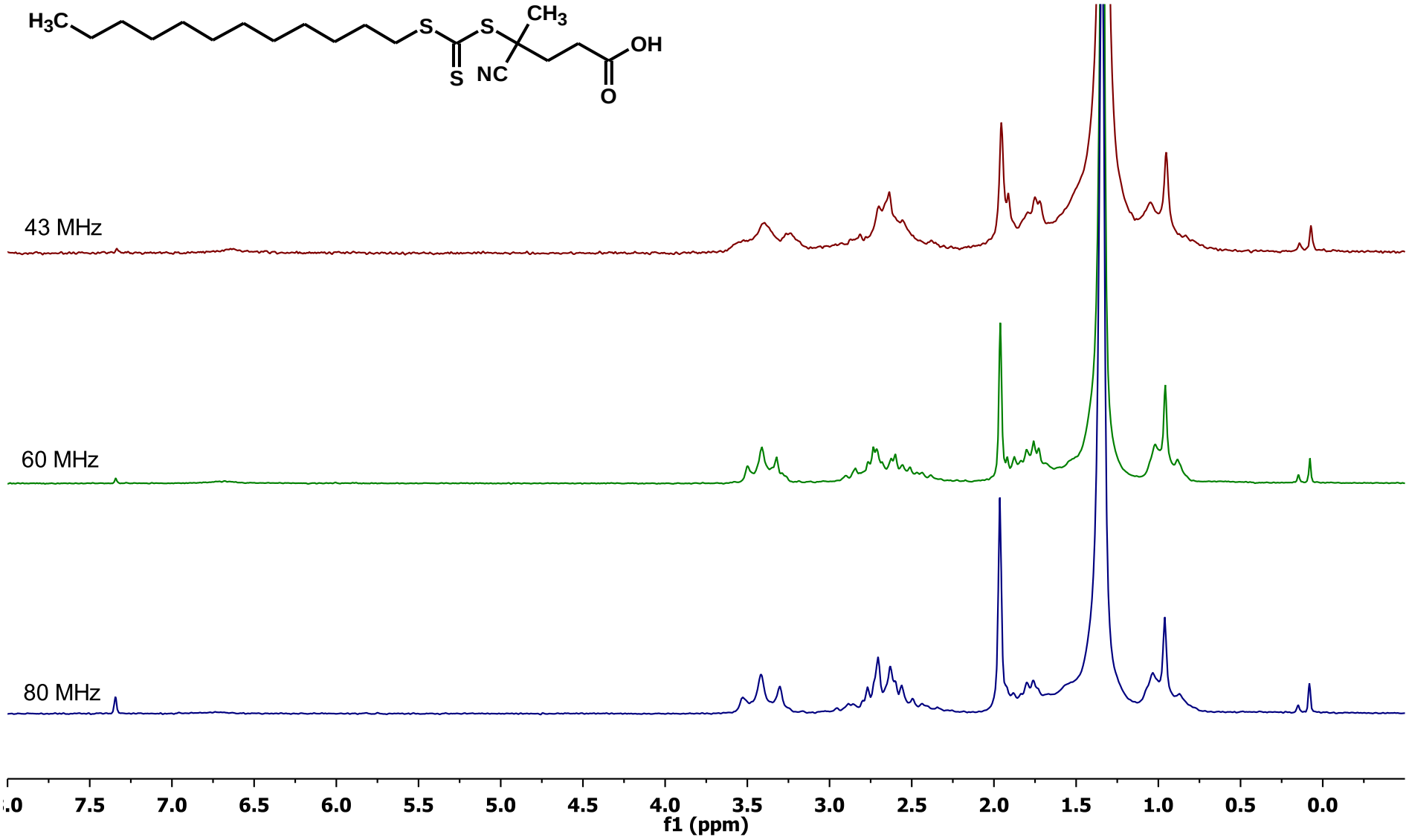
1D 1H of

4-Cyano-4-[(dodecylsulfanylthiocarbonyl)sulfanyl]pentanoic acid:

43 vs 60 vs 80 MHz



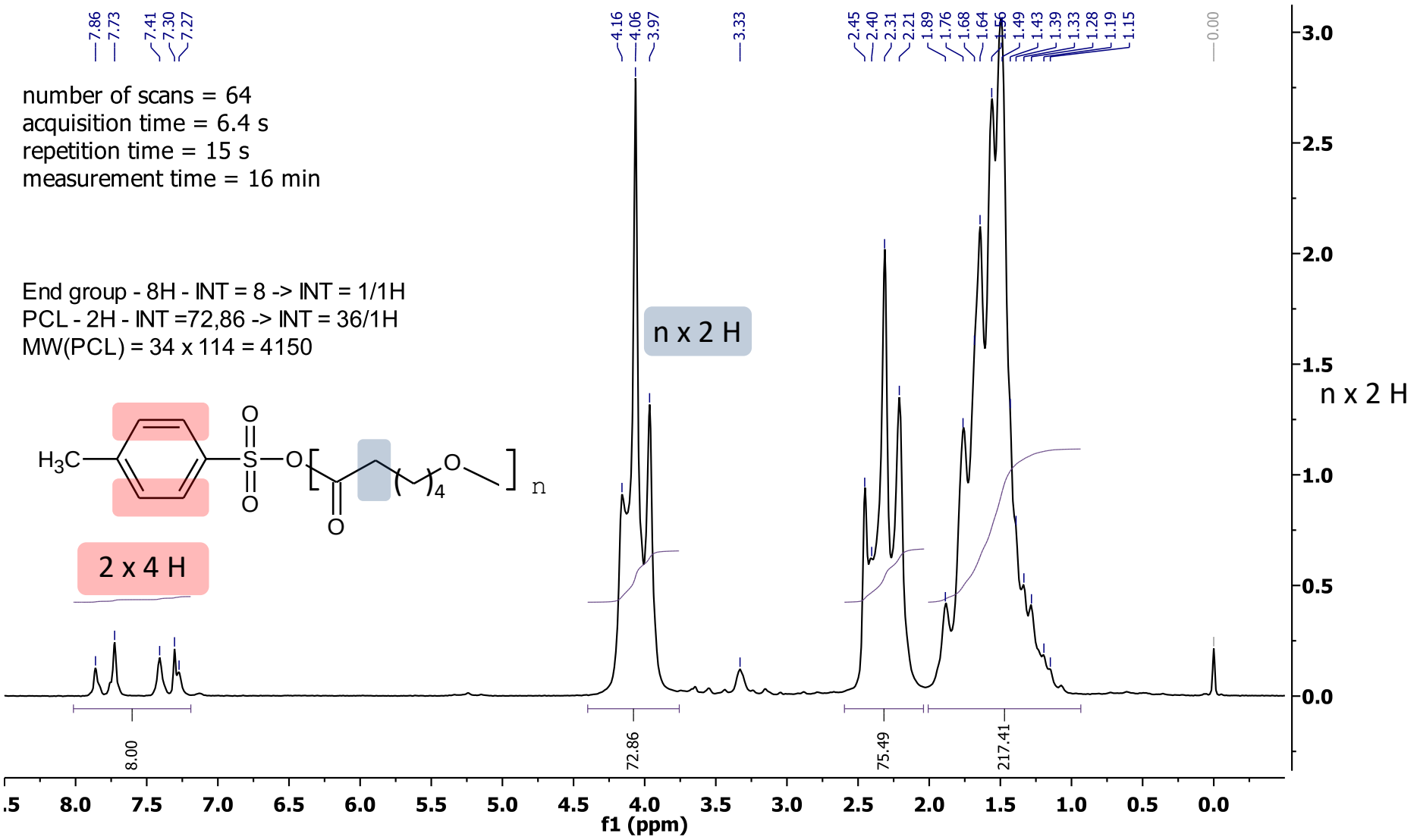
4-Cyano-4-[(dodecylsulfanylthiocarbonyl)sulfanyl]pentanoic acid; 48 mg/mL $CDCl_3$; 1D 1H; 43 vs 60 vs 80 MHz



End-group analysis of polyesters and polyethylene glycol

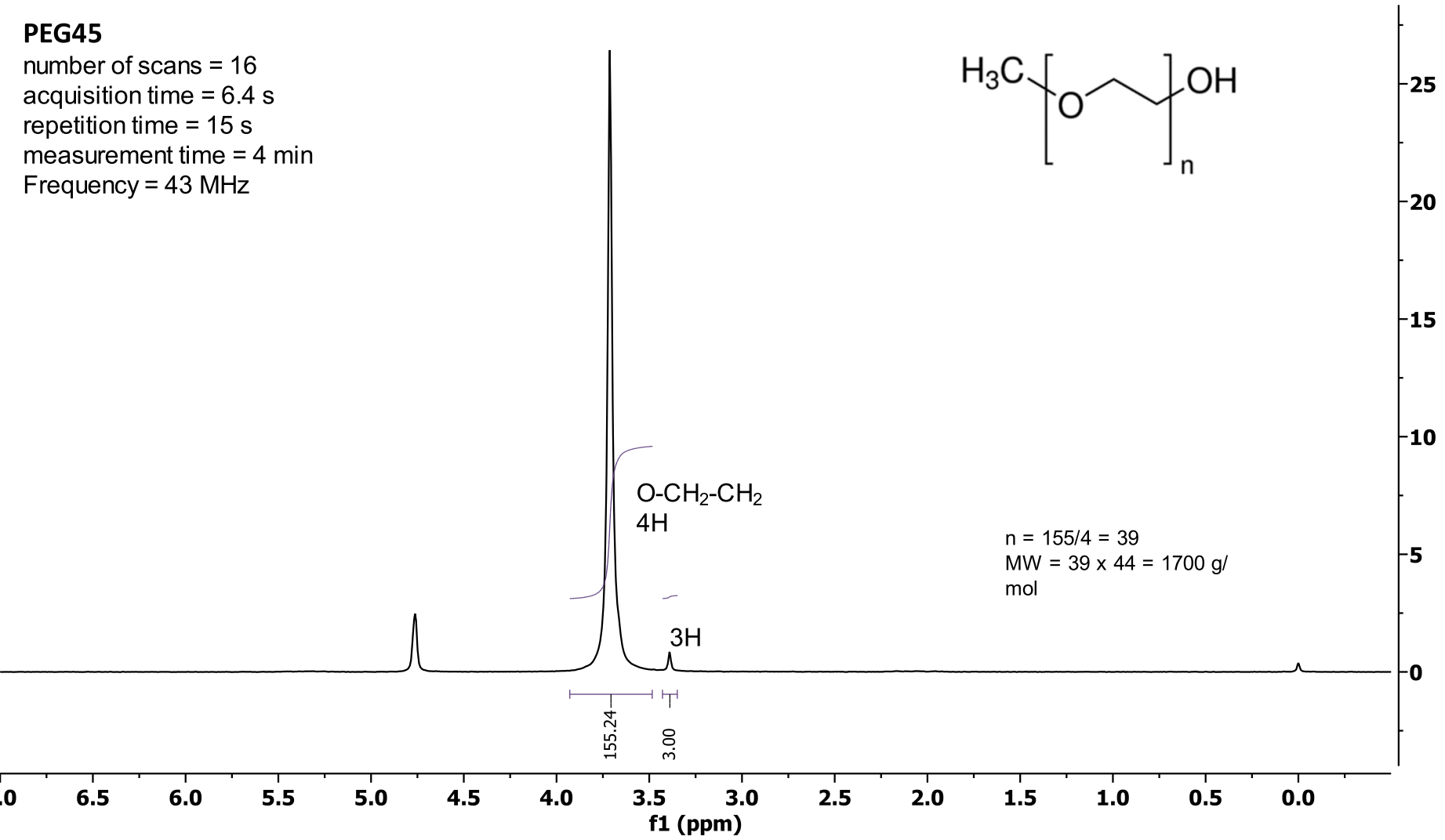
on a Spinsolve Carbon 60 MHz

PCL-(Ots)2; C = 89 g/L; 1D 1H



PEG; 1D 1H

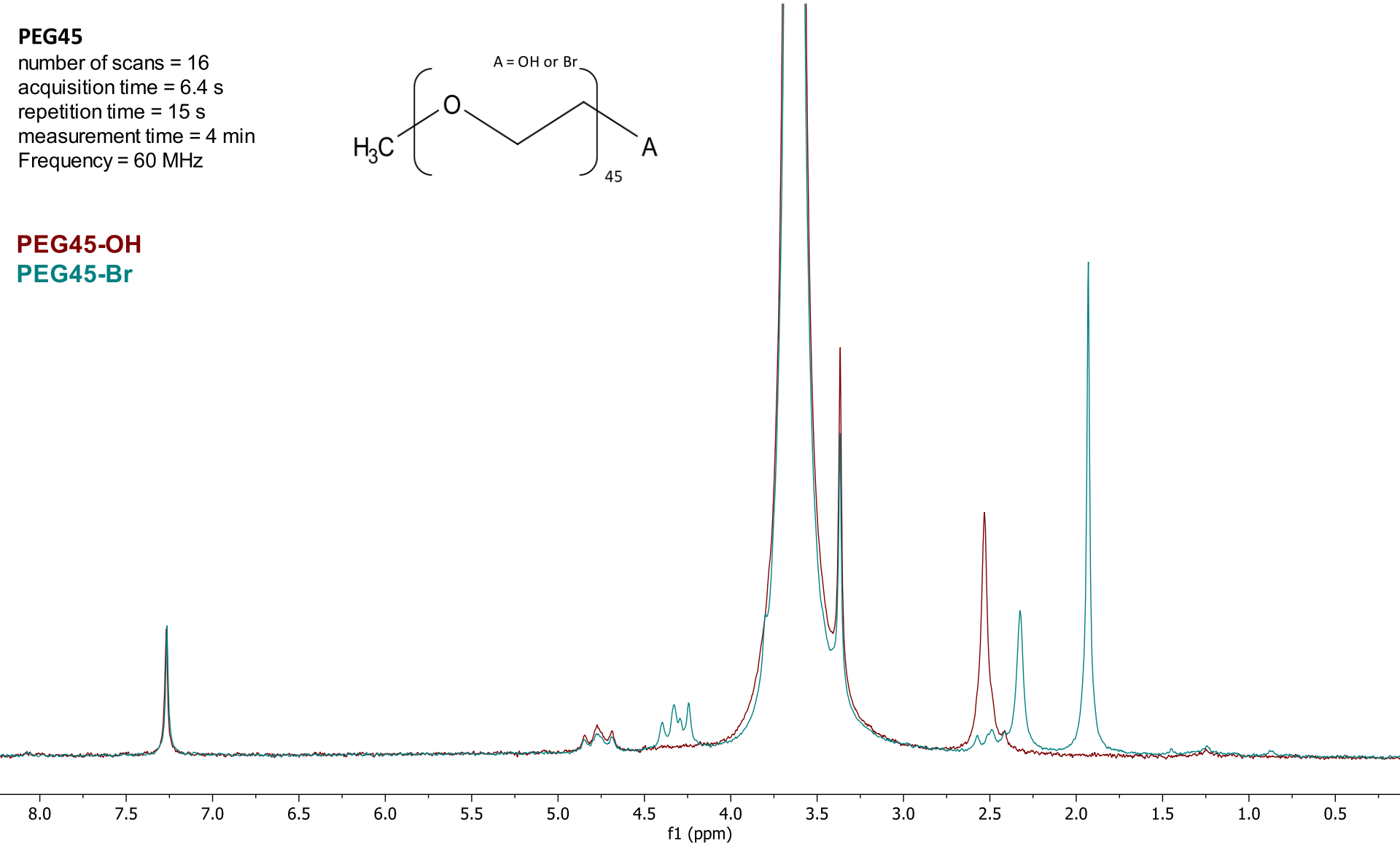
PEG45
number of scans = 16
acquisition time = 6.4 s
repetition time = 15 s
measurement time = 4 min
Frequency = 43 MHz



PEG; 1D 1H

PEG45
number of scans = 16
acquisition time = 6.4 s
repetition time = 15 s
measurement time = 4 min
Frequency = 60 MHz

PEG45-OH
PEG45-Br

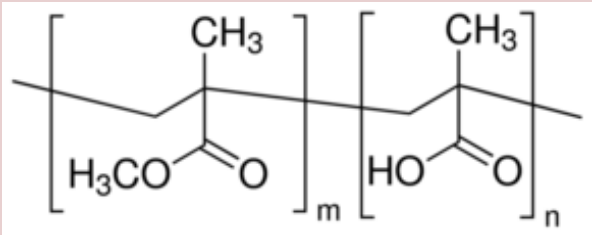
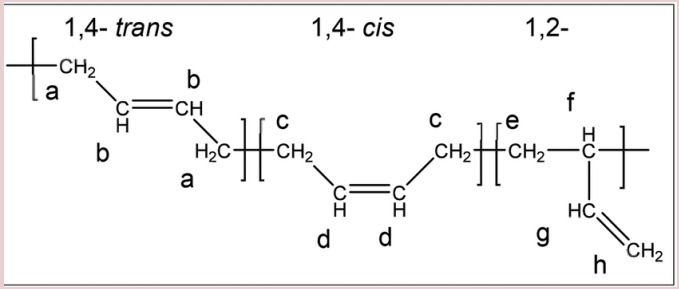
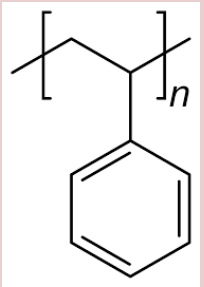


magritek
www.magritek.com

Composition determination of Eudragit® and SBR copolymers

on a Spinsolve Carbon 60 MHz

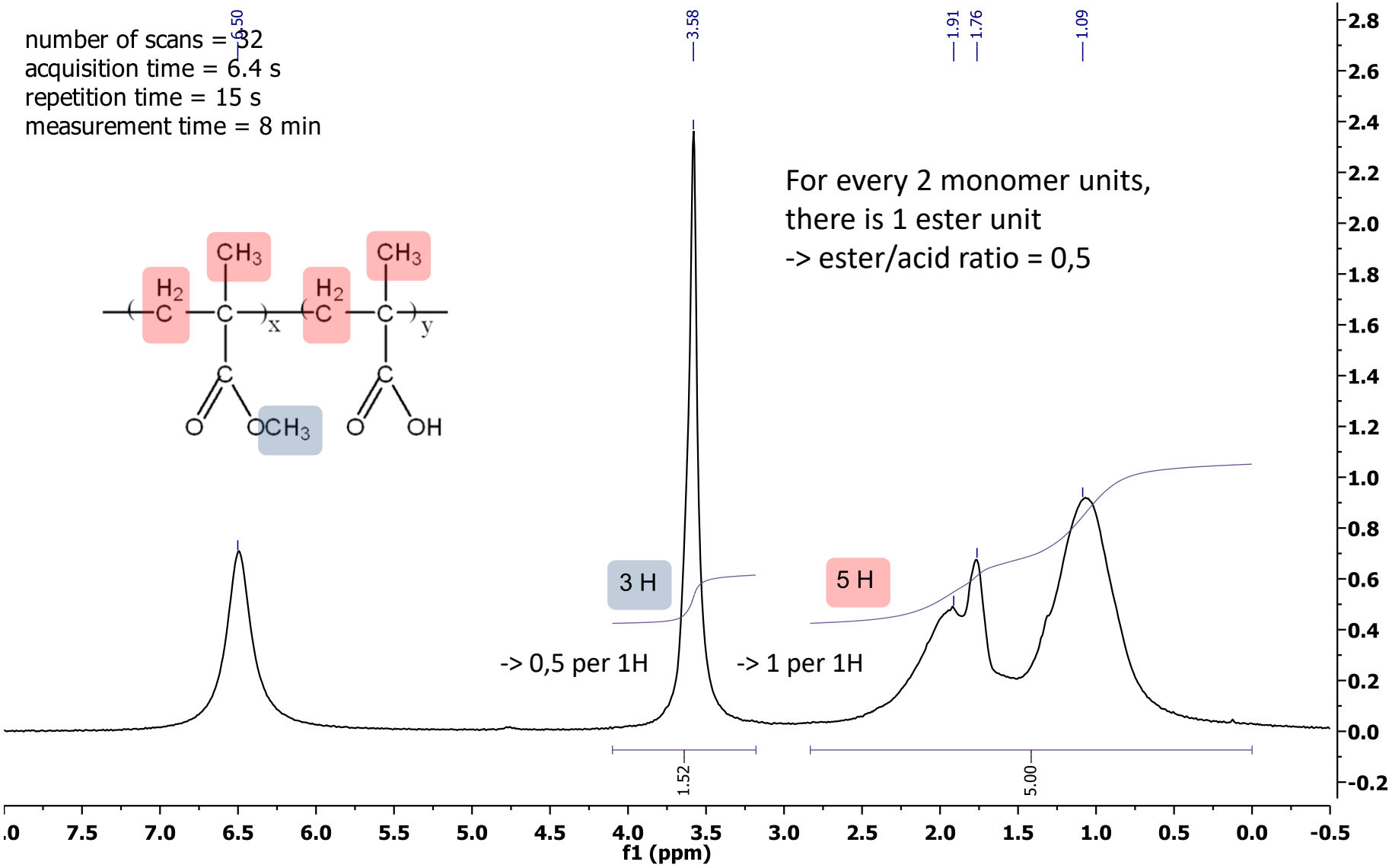
List of polymers and polymers information

Polymer	Concentration	Solvent
Poly(methacrylic acid, methyl methacrylate) 1:1	~ 100 mg/mL	THF-d8
Poly(methacrylic acid, methyl methacrylate) 1:2	~70 mg/mL	THF-d8
		
Styrene butadiene rubber	~ 12,6 mg/mL	CD_2Cl_2
Polybutadiene (3 different configurations) 		Polystyrene 

Poly(methacrylic acid, methyl methacrylate) 1:1

number of scans = 32
acquisition time = 6.4 s
repetition time = 15 s
measurement time = 8 min

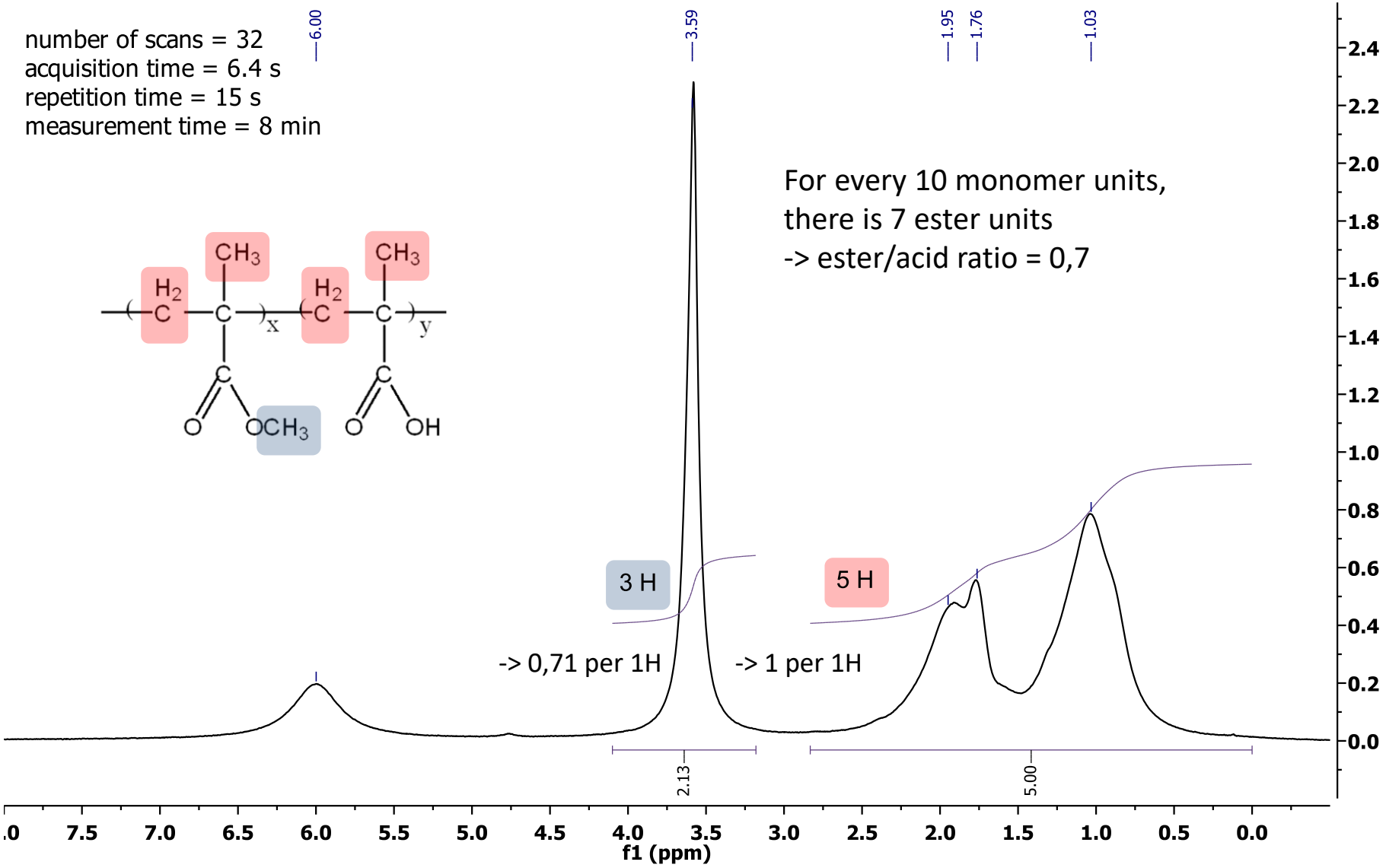
For every 2 monomer units,
there is 1 ester unit
-> ester/acid ratio = 0,5



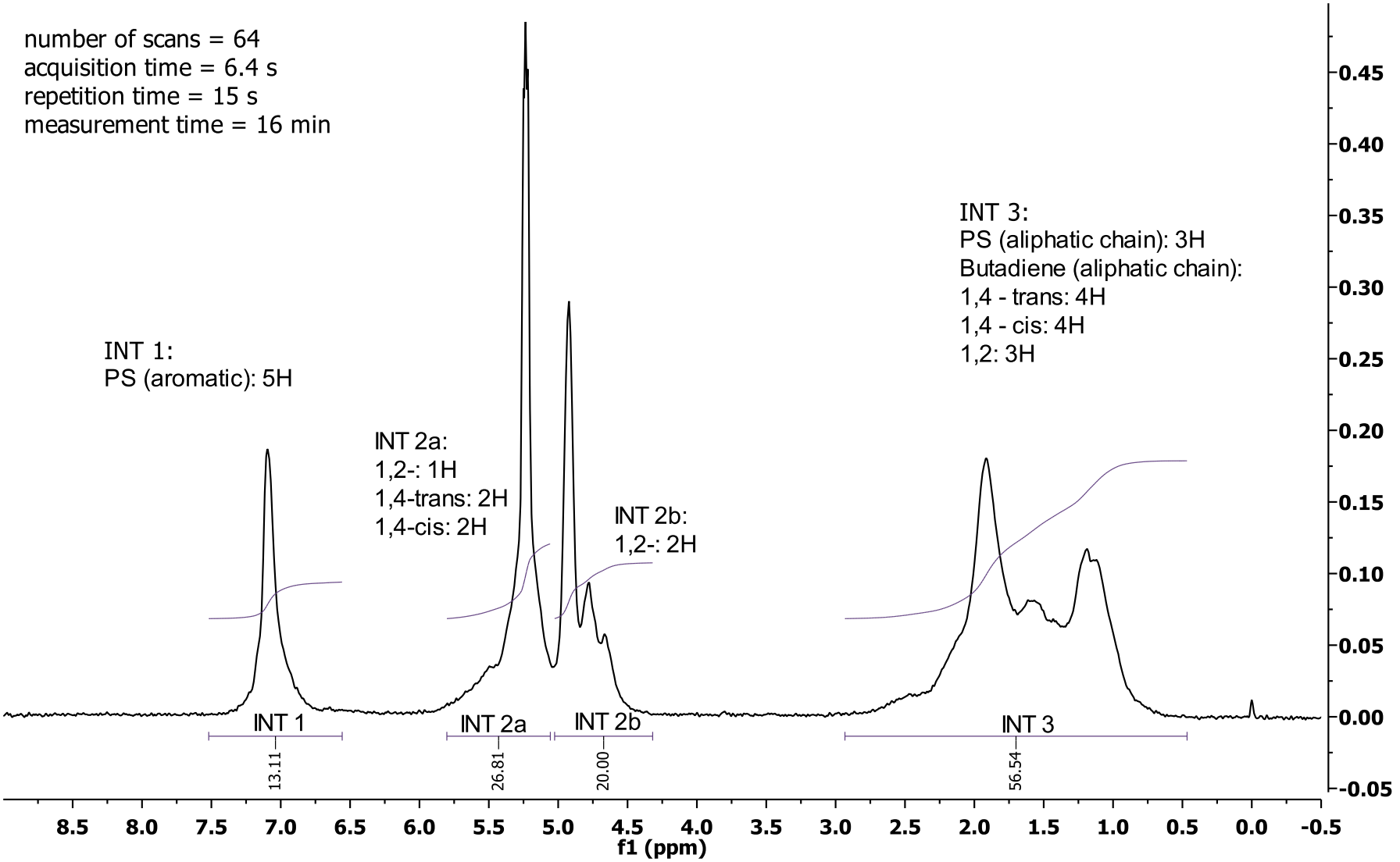
Poly(methacrylic acid, methyl methacrylate) 1:2

number of scans = 32
acquisition time = 6.4 s
repetition time = 15 s
measurement time = 8 min

For every 10 monomer units,
there is 7 ester units
-> ester/acid ratio = 0,7



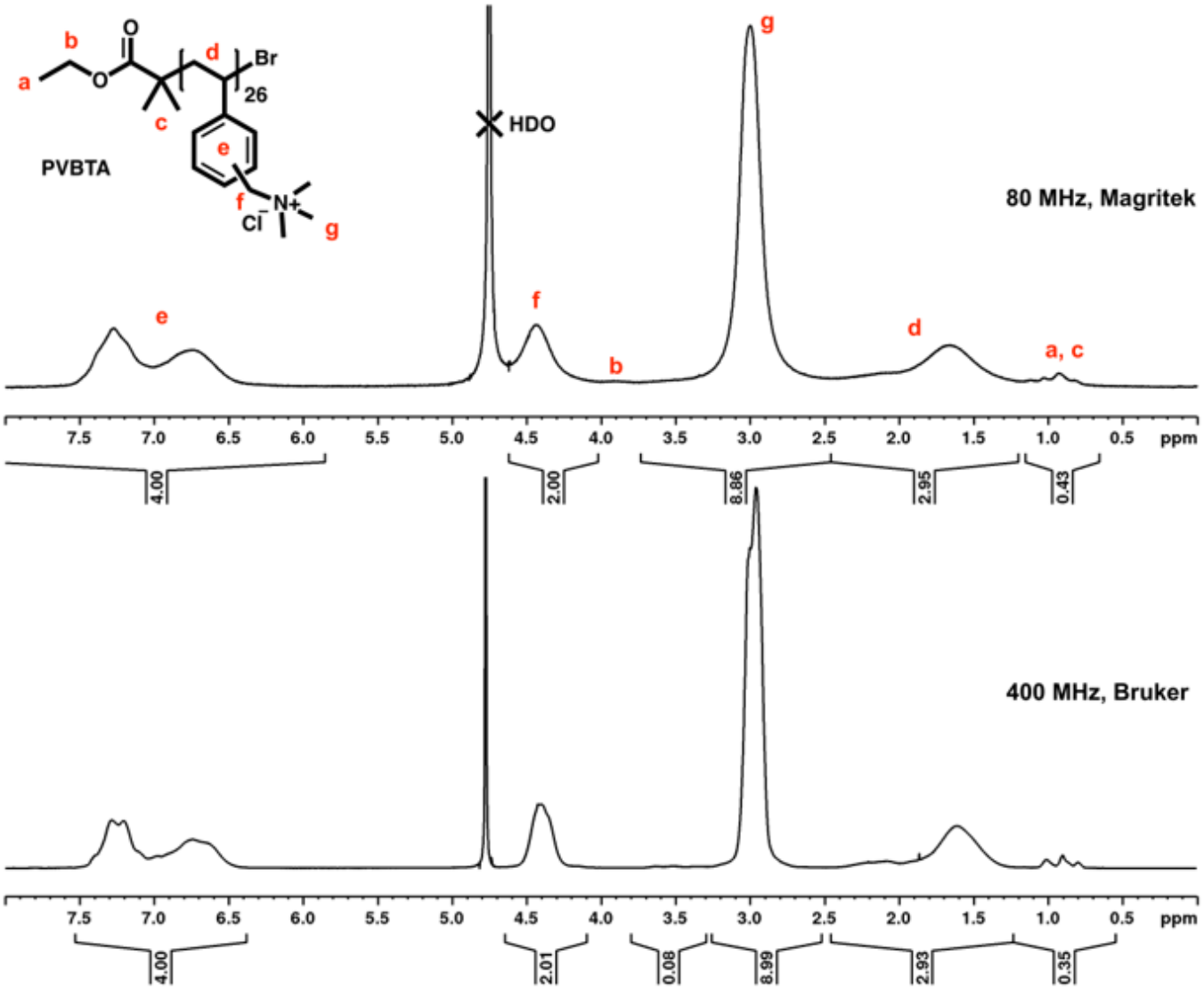
Styrene butadiene rubber



Comparison of polymers synthesised by Jakob Stensgaard Diget

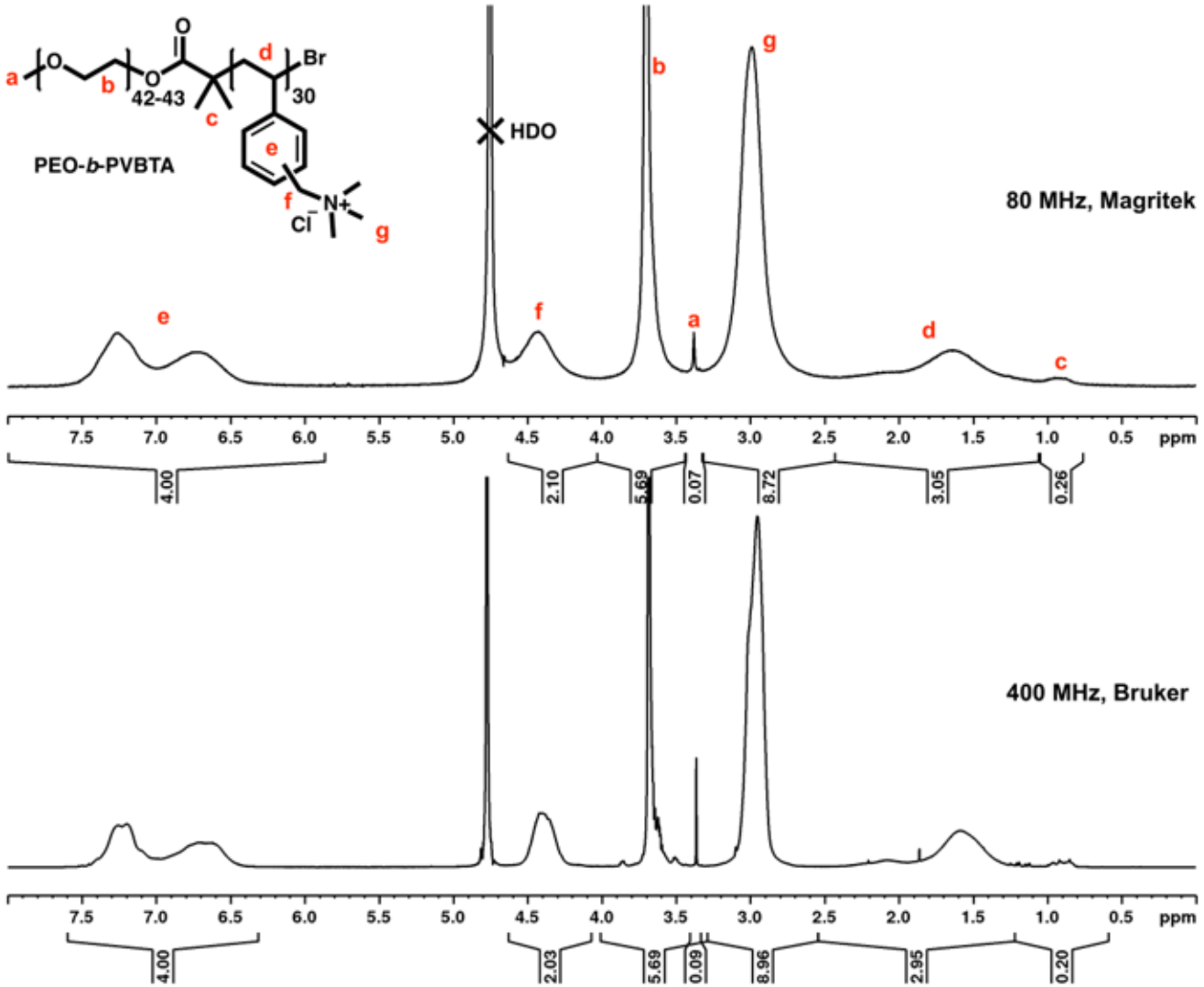
on a Spinsolve Carbon 80 MHz and on a Bruker 400 MHz

Comparison 80 vs 400 MHz



Synthesized by Jakob Stensgaard Diget

Comparison 80 vs 400 MHz



Synthesized by Jakob Stensgaard Diget

Helpful links

http://www.magritek.com/products/spinsolve/ -
Brochures, case studies and much more
http://www.magritek.com/products/spinsolve/nmr-spectra-examples/ -
http://www.magritek.com/products/spinsolve/performance-benchmarks/ -
Example spectra measured by our Spinsolve
https://www.youtube.com/watch?v=eSQJn9mqJ6U -
Reaction monitoring with Spinsolve
http://www.magritek.com/support/videos/ -
Explanation about the principles of NMR

Any more Questions?
Please contact helene@magritek.com

GERMANY
Philipsstraße 8
52068 Aachen, Germany
Tel: +49 (241) 70525-6000
Fax: +49 (241) 963 1429

NEW ZEALAND
6 Hurring Place, Unit 3
Newlands, Wellington 6037, NZ
Tel: +64 4 477 7096
Fax: +64 4 471 4665

UNITED STATES
6440 Lusk Blvd (D108)
San Diego, CA 92121, USA
Tel: +1 (855) 667-6835
+1 (866) NMR-MTEK