

Unexpected Cleavage of Thiacalix[4]arene Sulfoxides

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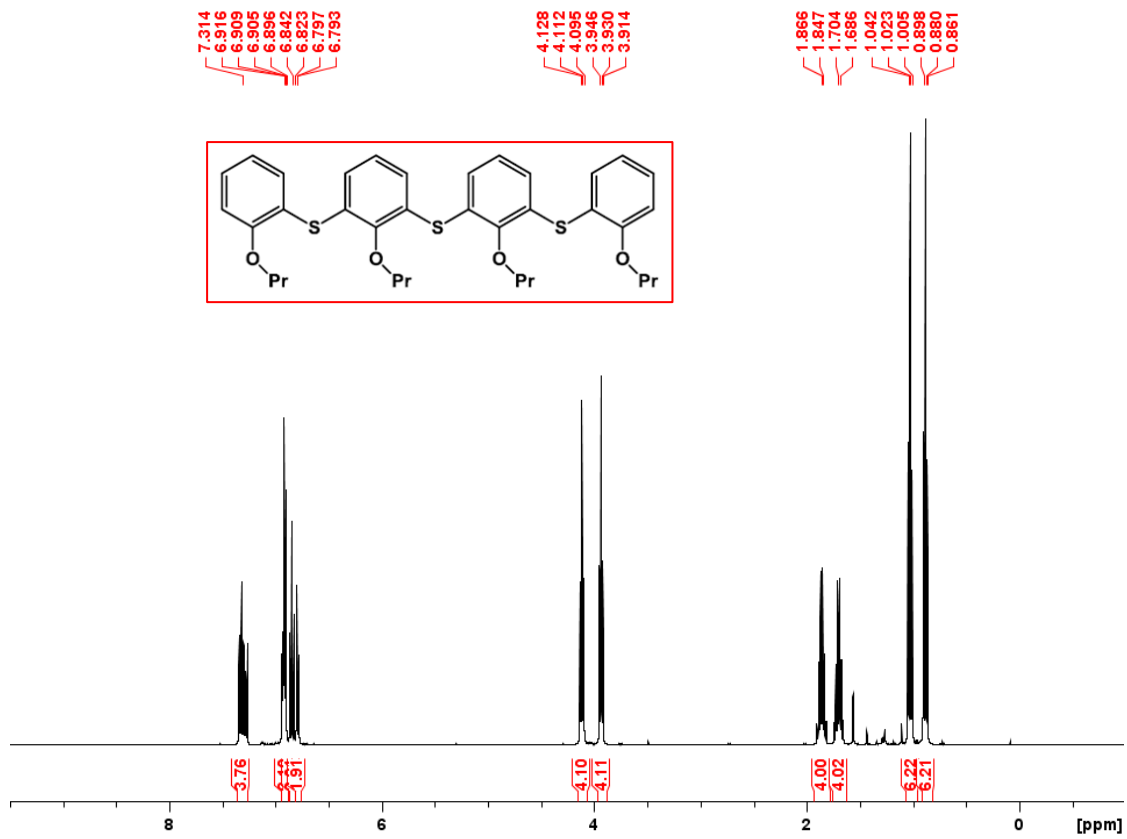
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Supporting information

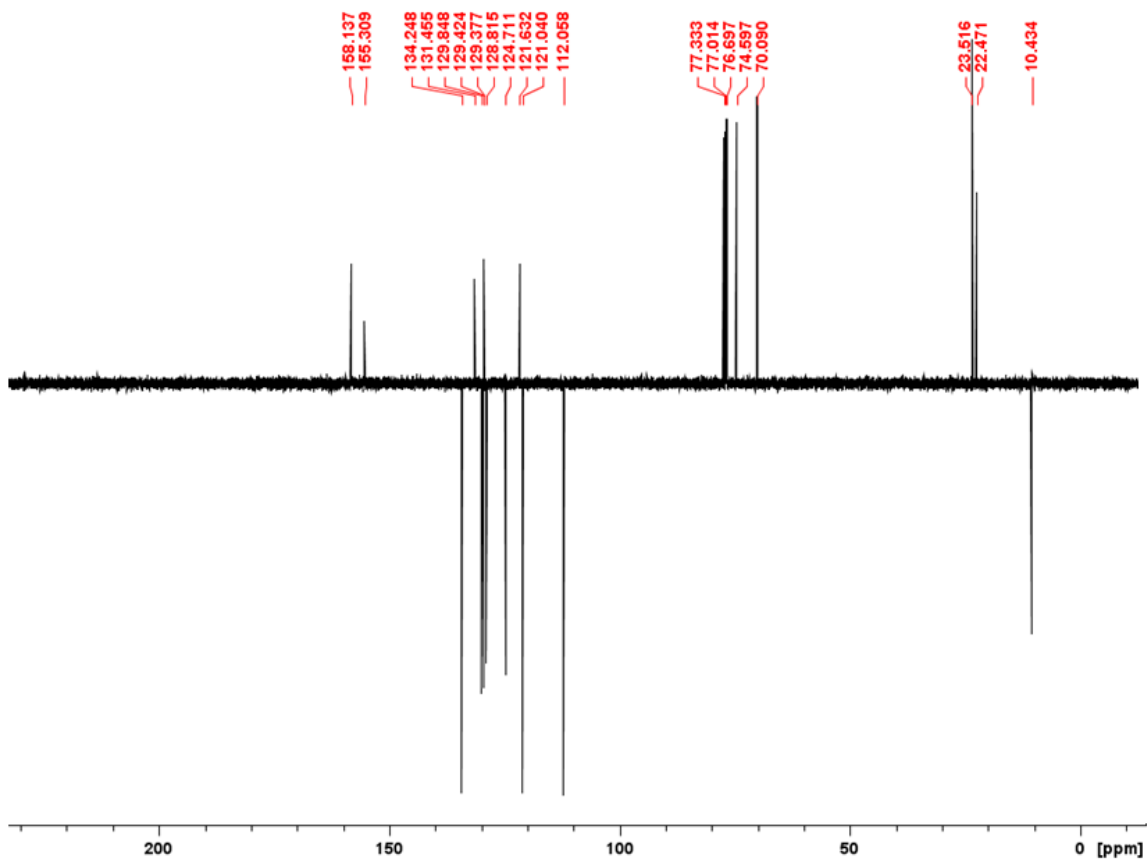
Table of Contents

Spectra of compounds	p. 2-34
X-ray structure of compound 6	p. 35

Compound 4

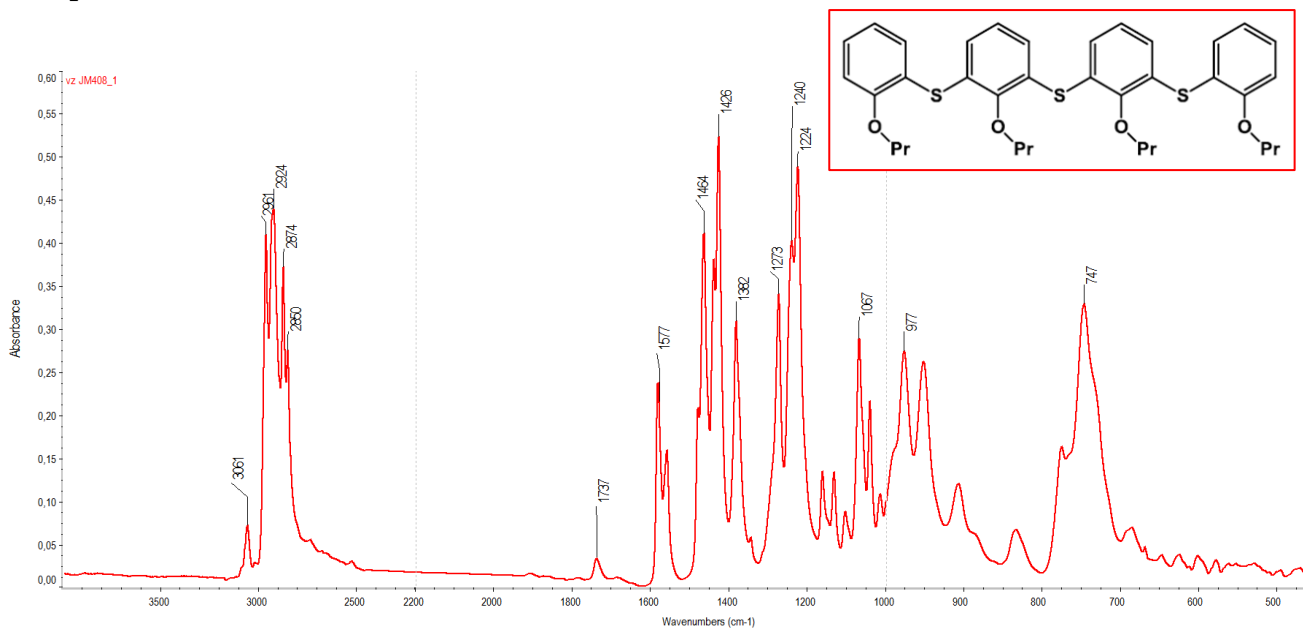


¹H NMR spectrum of compound 4 (CDCl₃, 298K, 400 MHz)

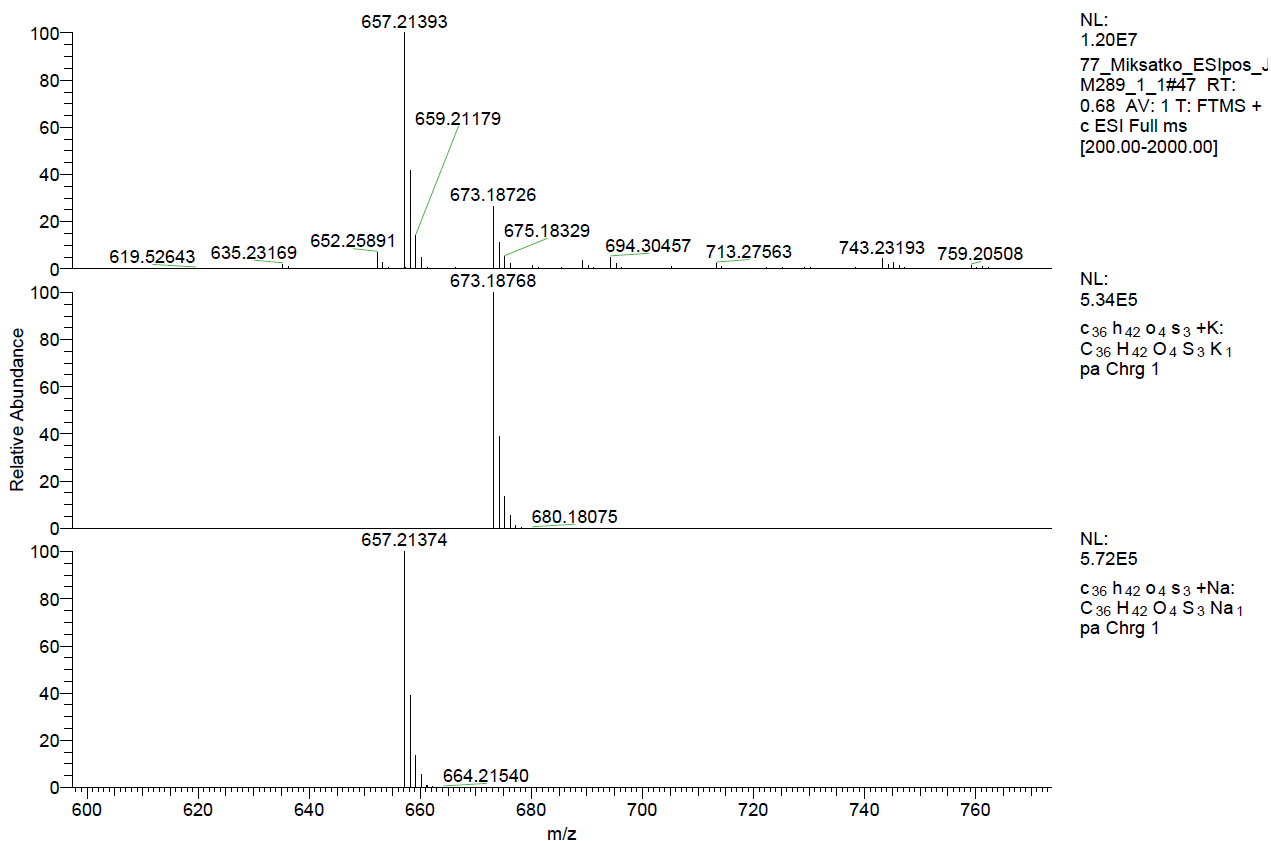


¹³C NMR (APT) spectrum of compound 4 (CDCl₃, 298K, 100 MHz)

Compound 4

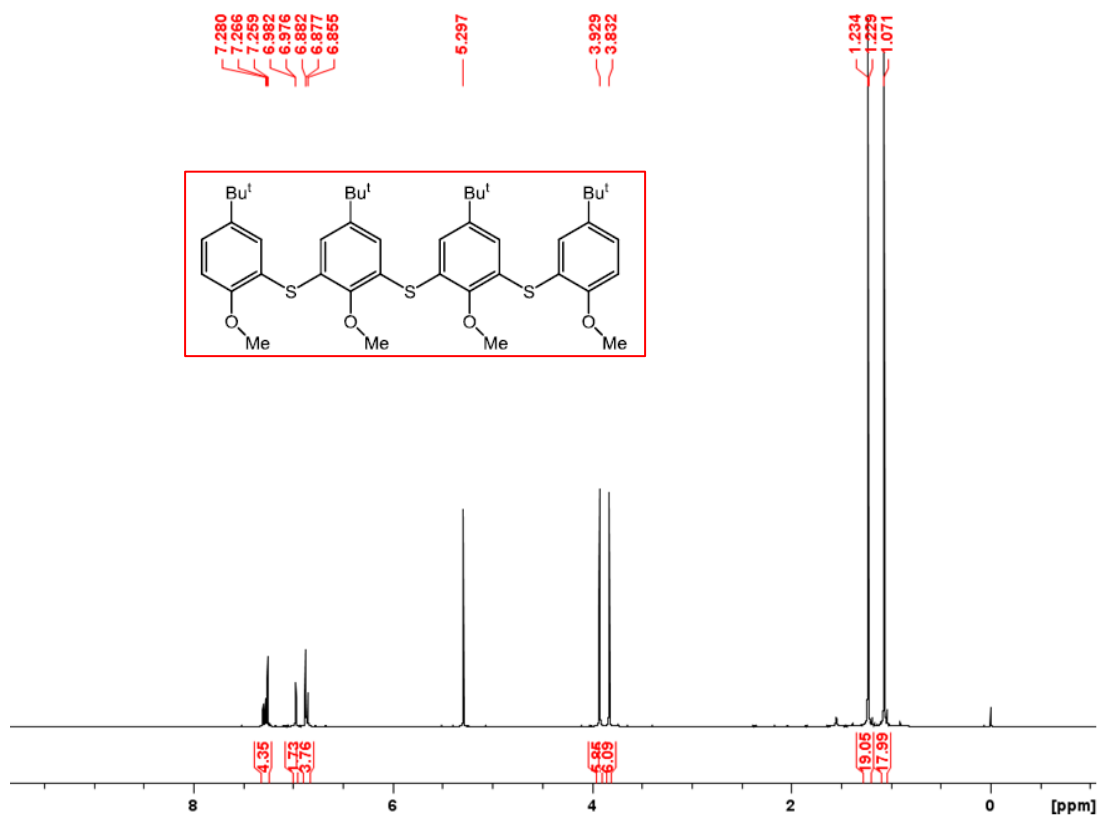


IR (ATR) spectrum of compound 4

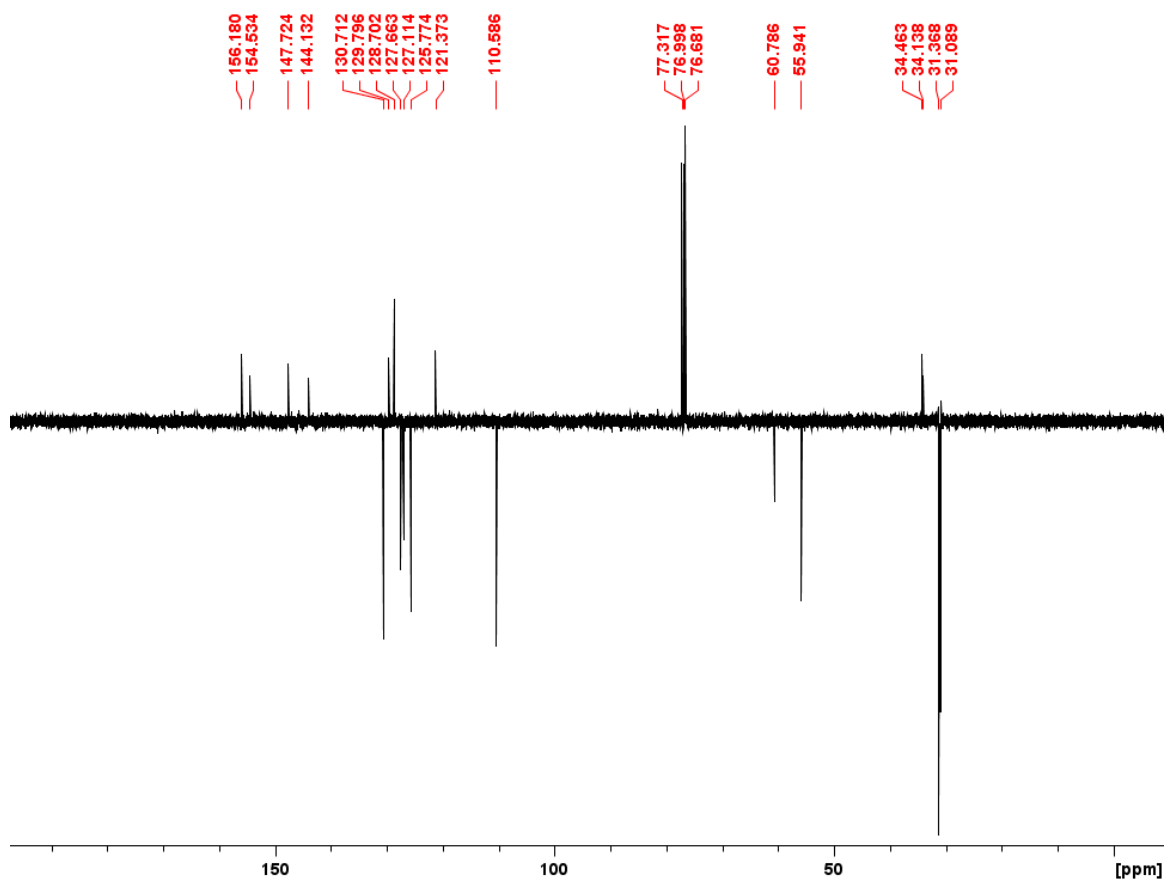


HRMS (ESI⁺) spectrum of compound 4

Compound 6

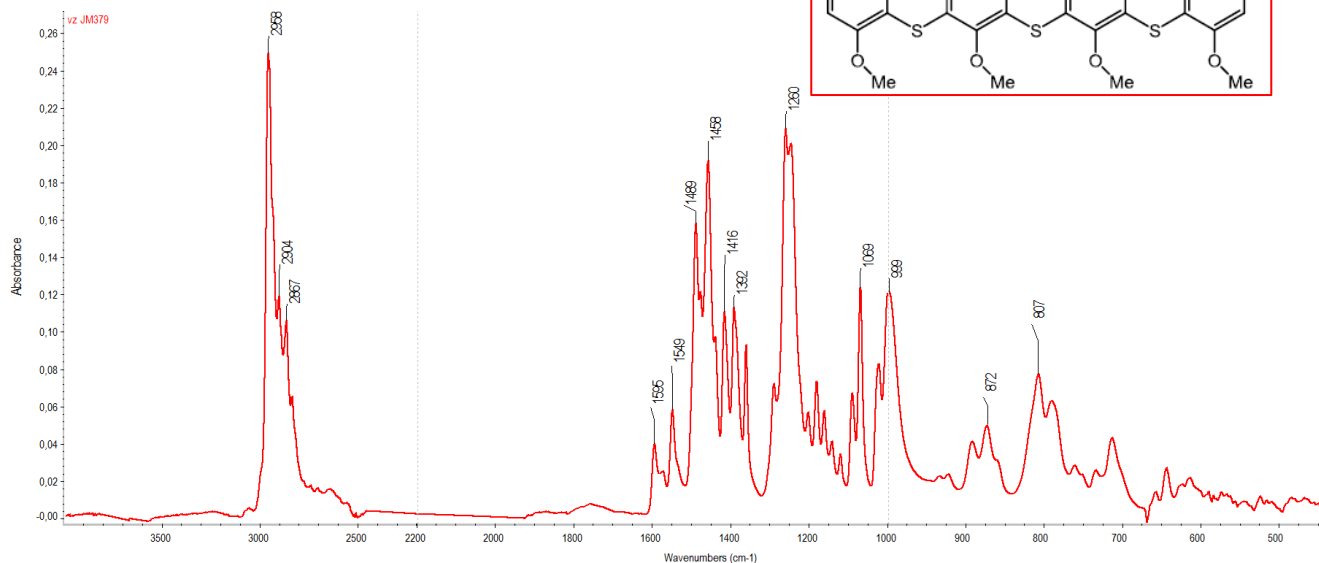


¹H NMR spectrum of compound 6 (CDCl₃, 298K, 400 MHz)

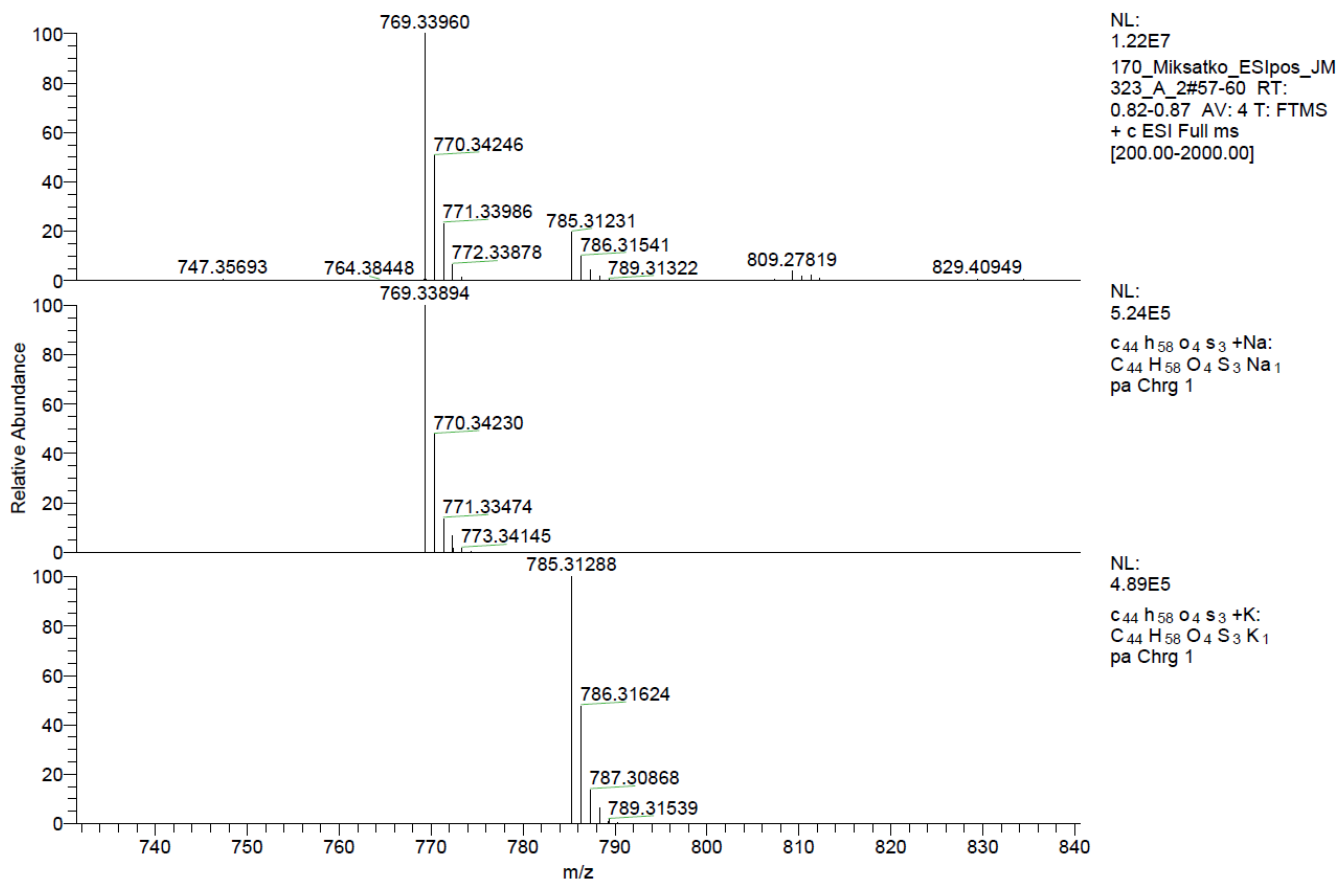


¹³C NMR (APT) spectrum of compound 6 (CDCl₃, 298K, 100 MHz)

Compound 6

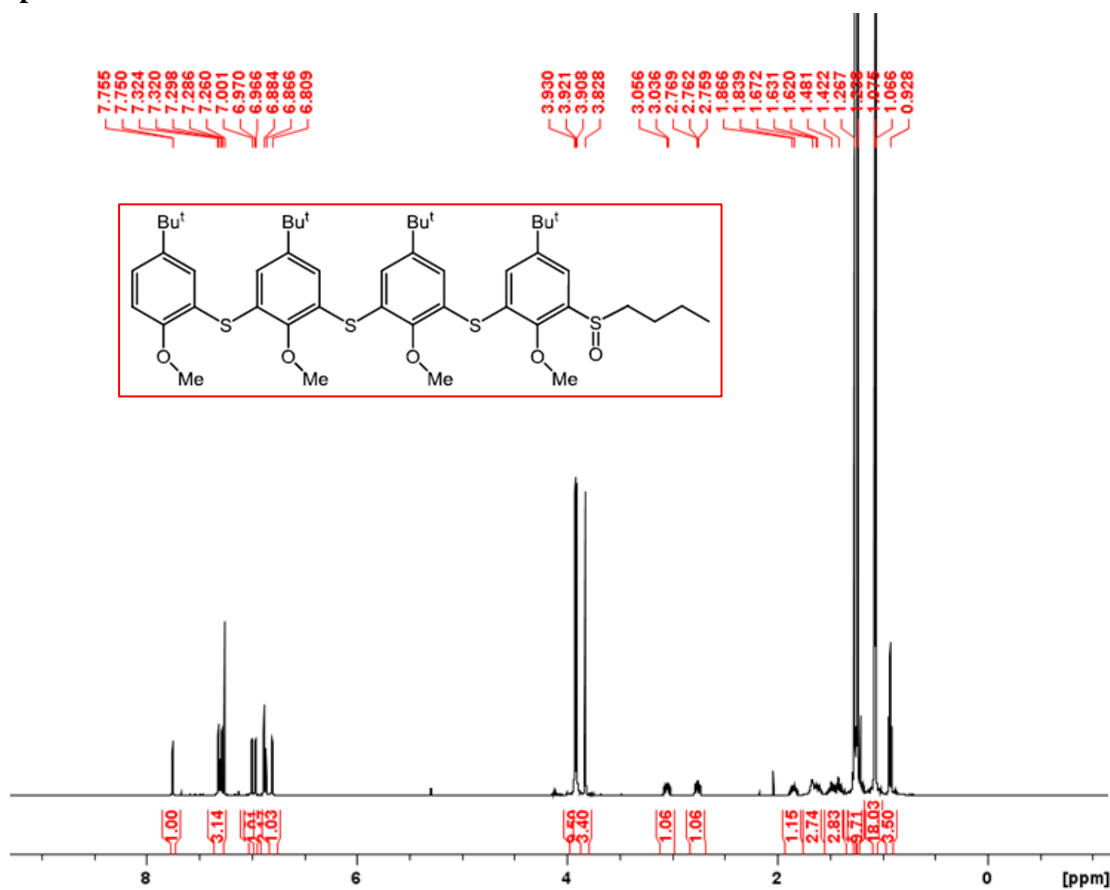


IR (ATR) spectrum of compound 6

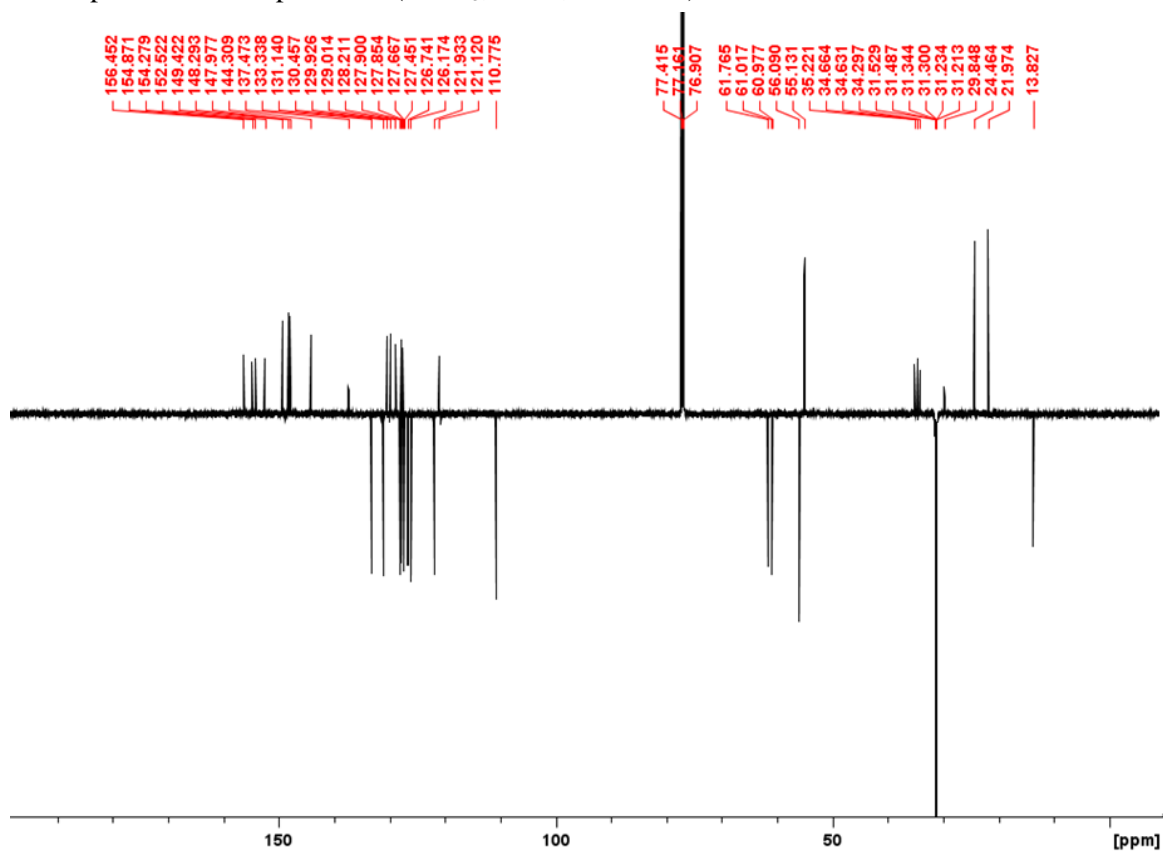


HRMS (ESI⁺) spectrum of compound 6

Compound 7a

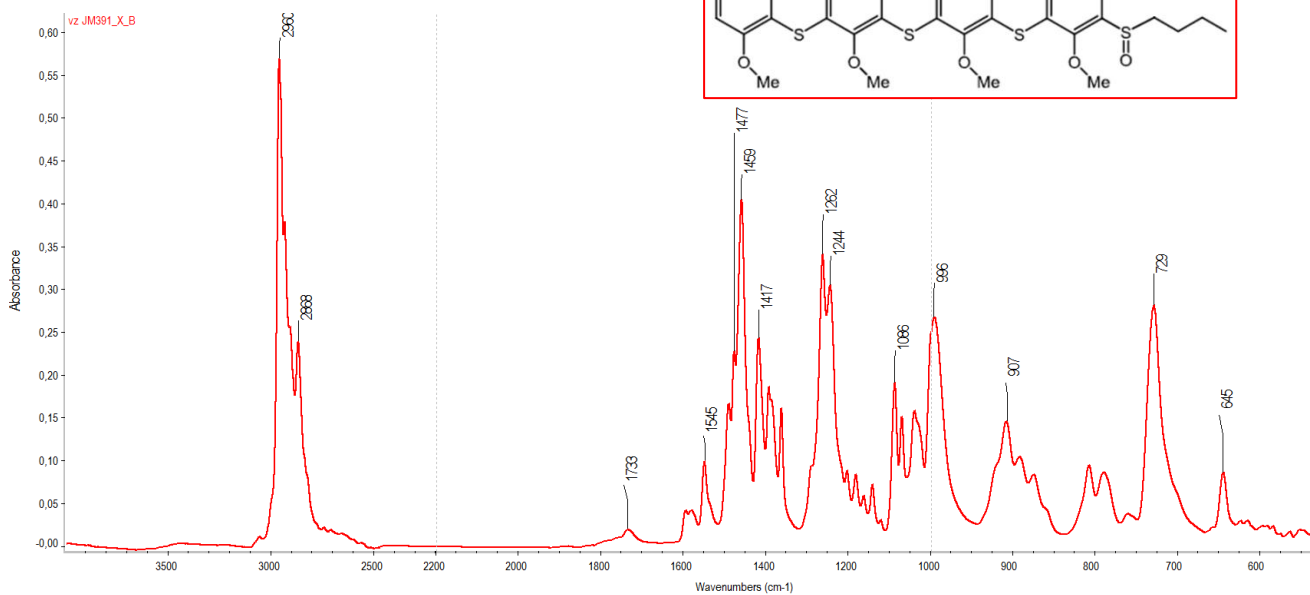
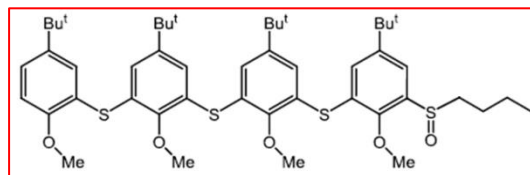


¹H NMR spectrum of compound 7a (CDCl₃, 298K, 400 MHz)

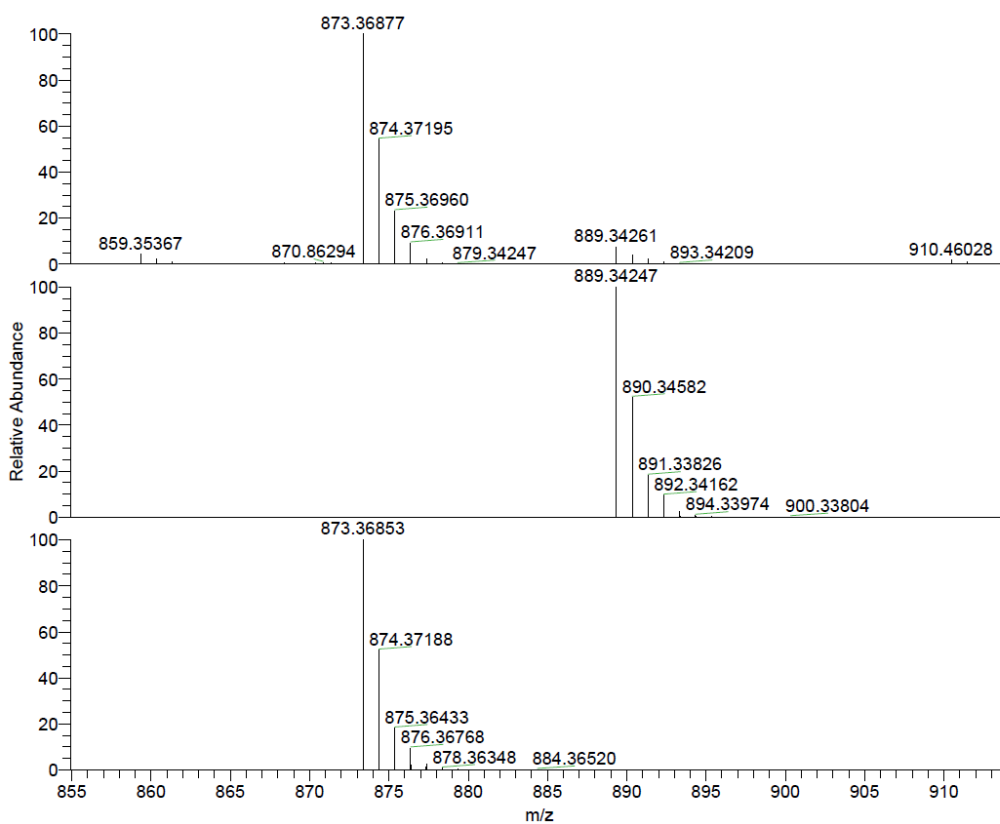


¹³C NMR (APT) spectrum of compound 7a (CDCl₃, 298K, 100 MHz)

Compound 7a



IR (ATR) spectrum of compound 7a



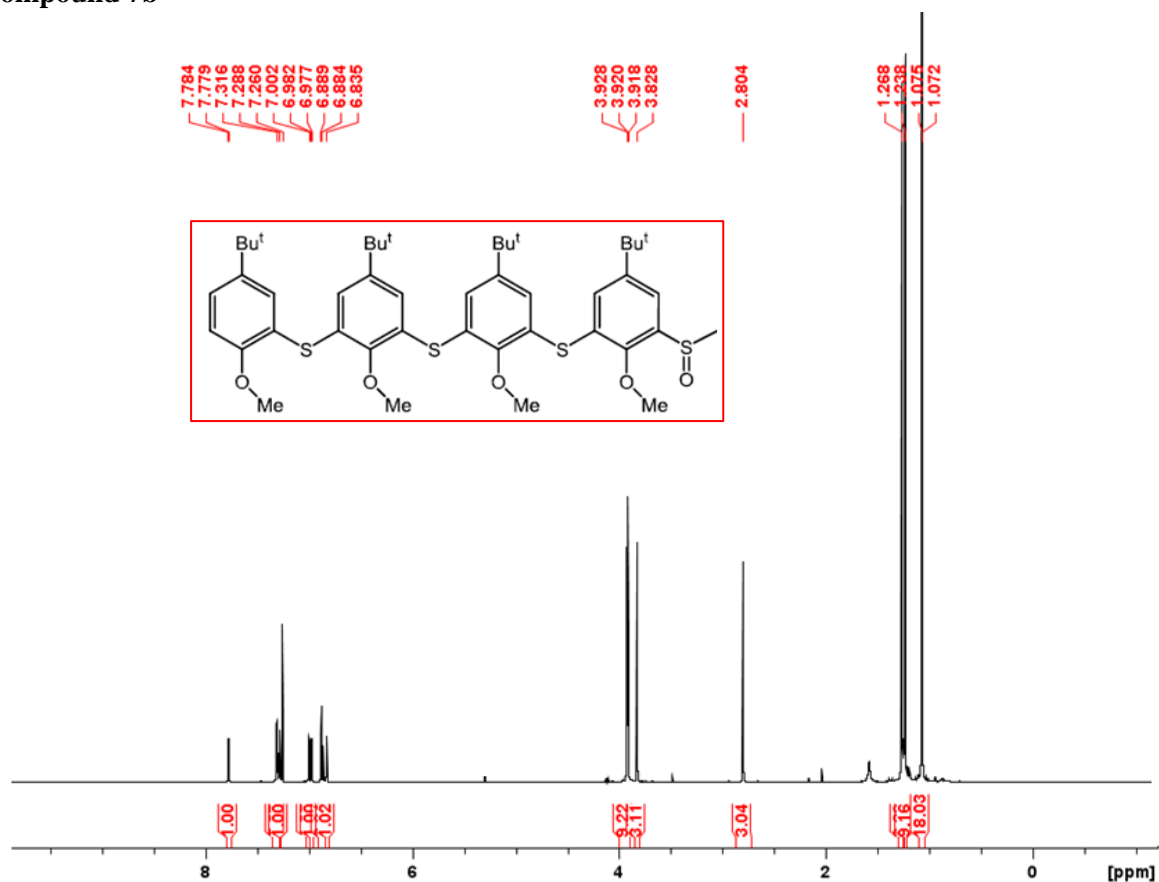
NL:
5.20E6
8_Miksatko_ESIpos_JM3
91_X_1#86-98 RT:
1.29-1.48 AV: 13 T:
FTMS + c ESI Full ms
[100.00-1500.00]

NL:
4.43E5
C₄₈H₆₆O₅S₄+K:
C₄₈H₆₆O₅S₄K₁
pa Chrg 1

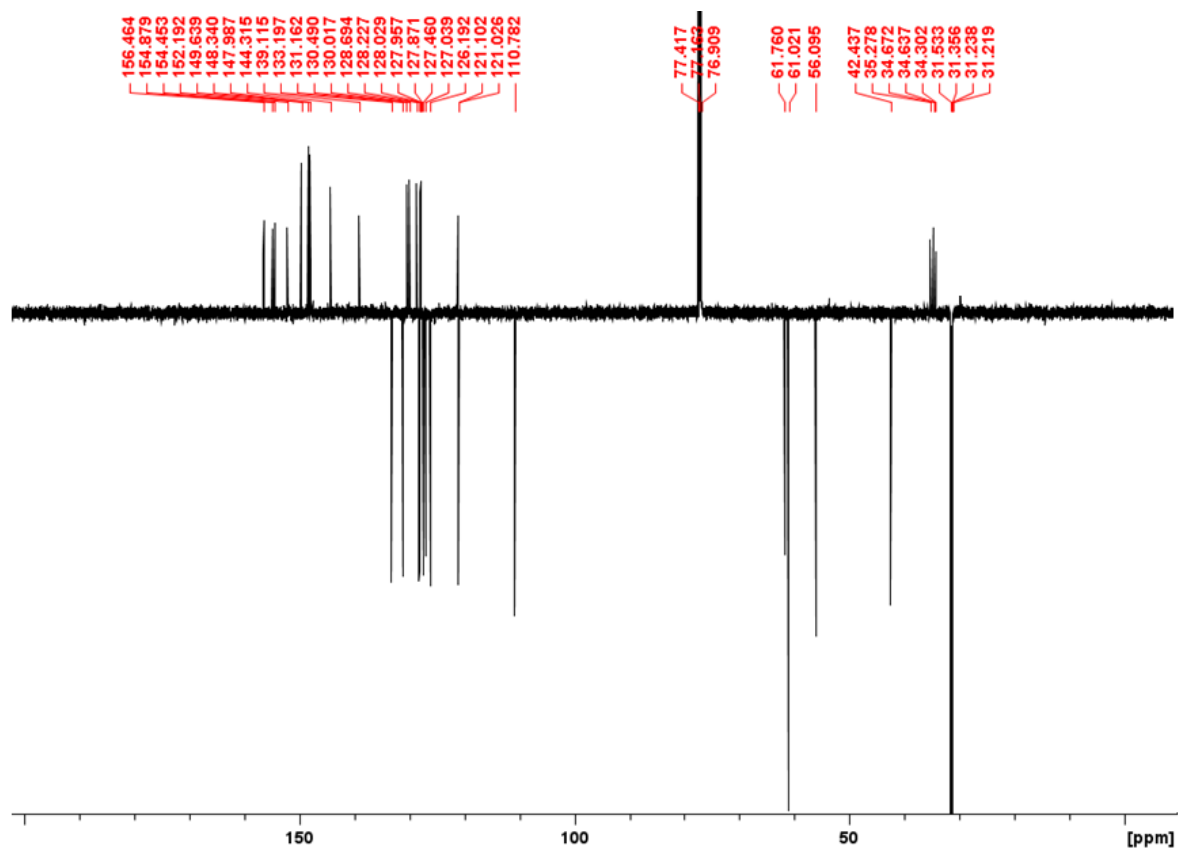
NL:
4.75E5
C₄₈H₆₆O₅S₄+Na:
C₄₈H₆₆O₅S₄Na₁
pa Chrg 1

HRMS (ESI⁺) spectrum of compound 7a

Compound 7b

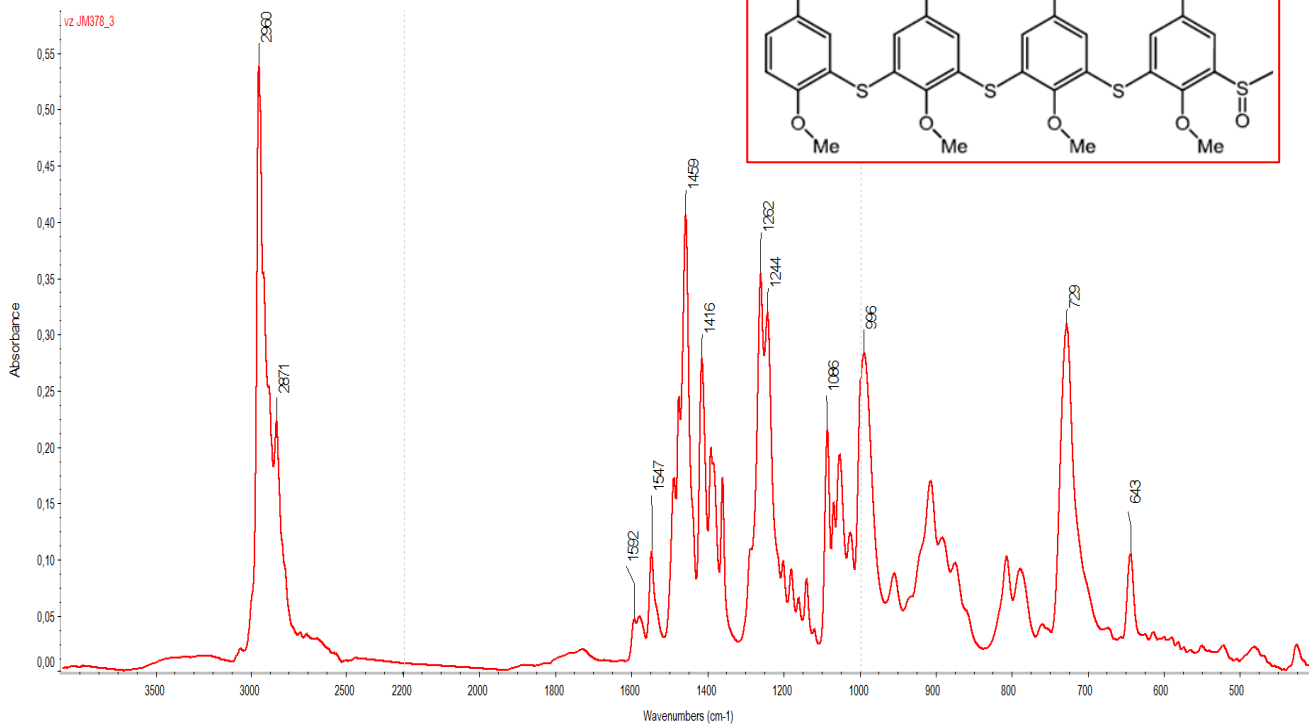
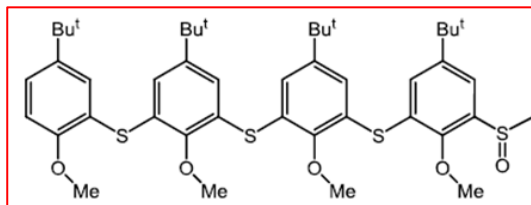


¹H NMR spectrum of compound 7b (CDCl₃, 298K, 400 MHz)

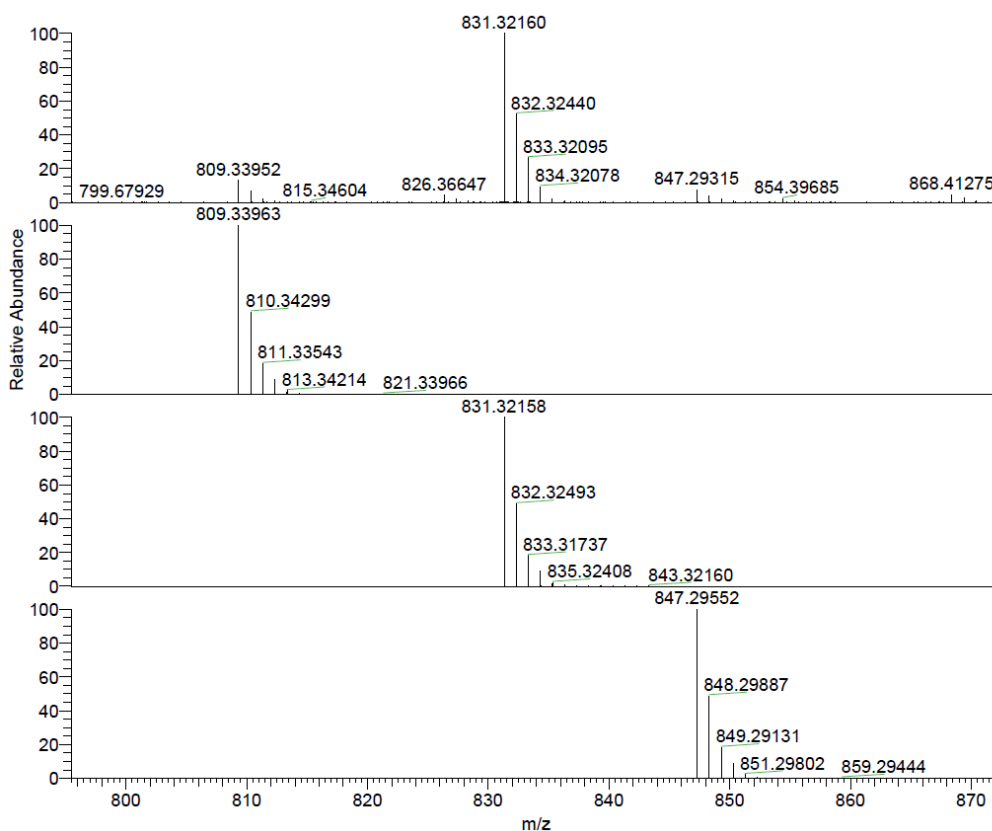


¹³C NMR (APT) spectrum of compound 7b (CDCl₃, 298K, 100 MHz)

Compound 7b



IR (ATR) spectrum of compound 7b



NL:
1.55E7
27_Miksatko_ESIpos_JM37
8_3_1#25-27 RT:
0.53-0.57 AV: 3 T: FTMS +
c ESI Full ms
[200.00-2000.00]

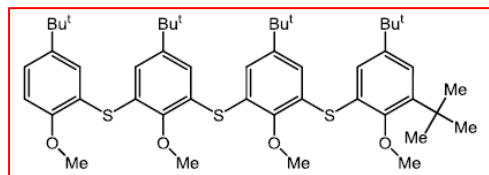
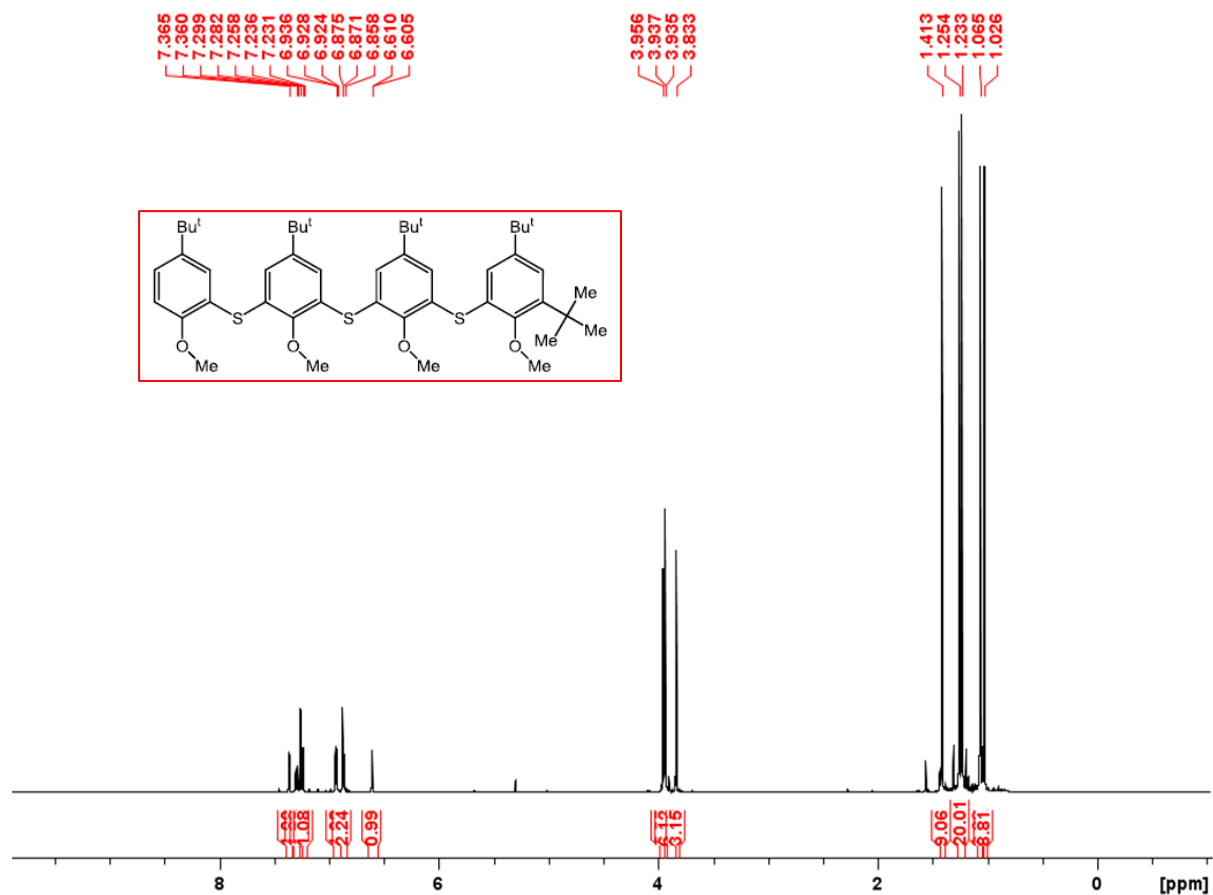
NL:
4.91E5
C₄₅H₆₀O₅S₄+H:
C₄₅H₆₁O₅S₄
pa Chrg 1

NL:
4.91E5
C₄₅H₆₀O₅S₄+Na:
C₄₅H₆₀O₅S₄Na₁
pa Chrg 1

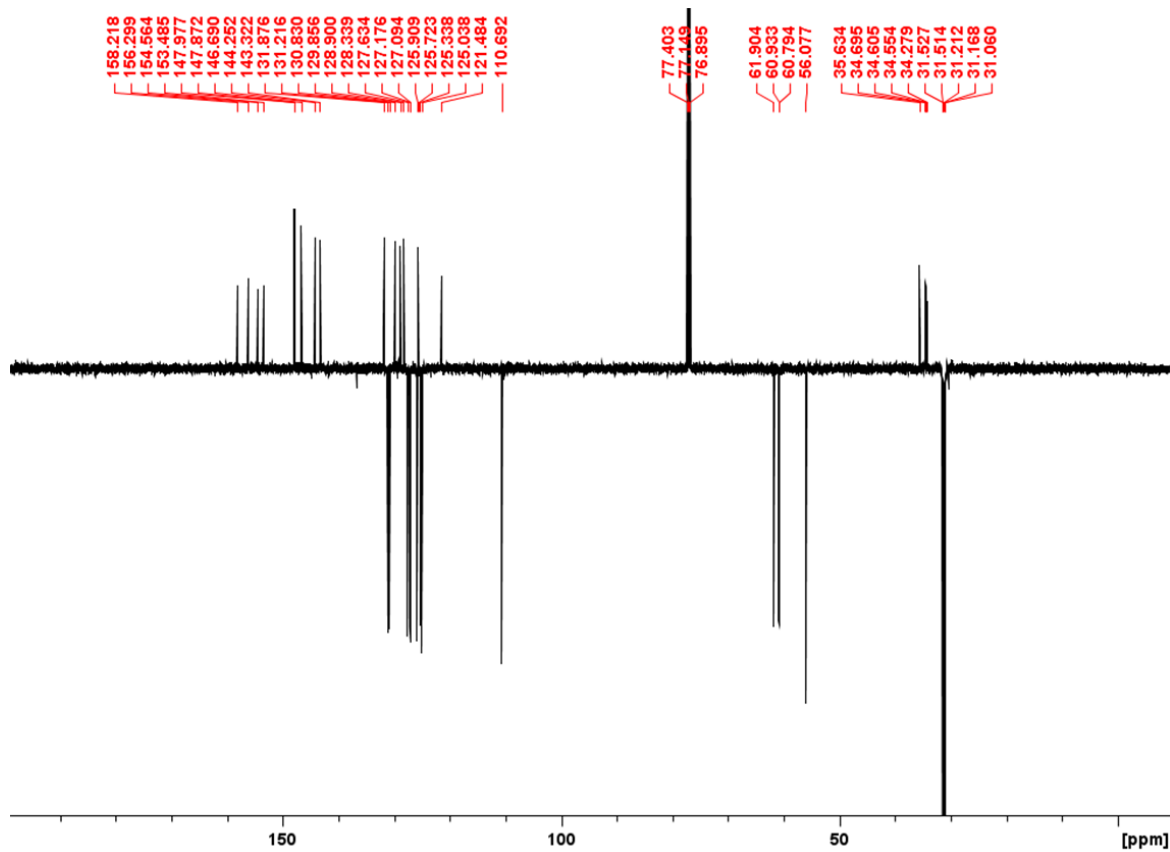
NL:
4.58E5
C₄₅H₆₀O₅S₄+K:
C₄₅H₆₀O₅S₄K₁
pa Chrg 1

HRMS (ESI⁺) spectrum of compound 7b

Compound 8

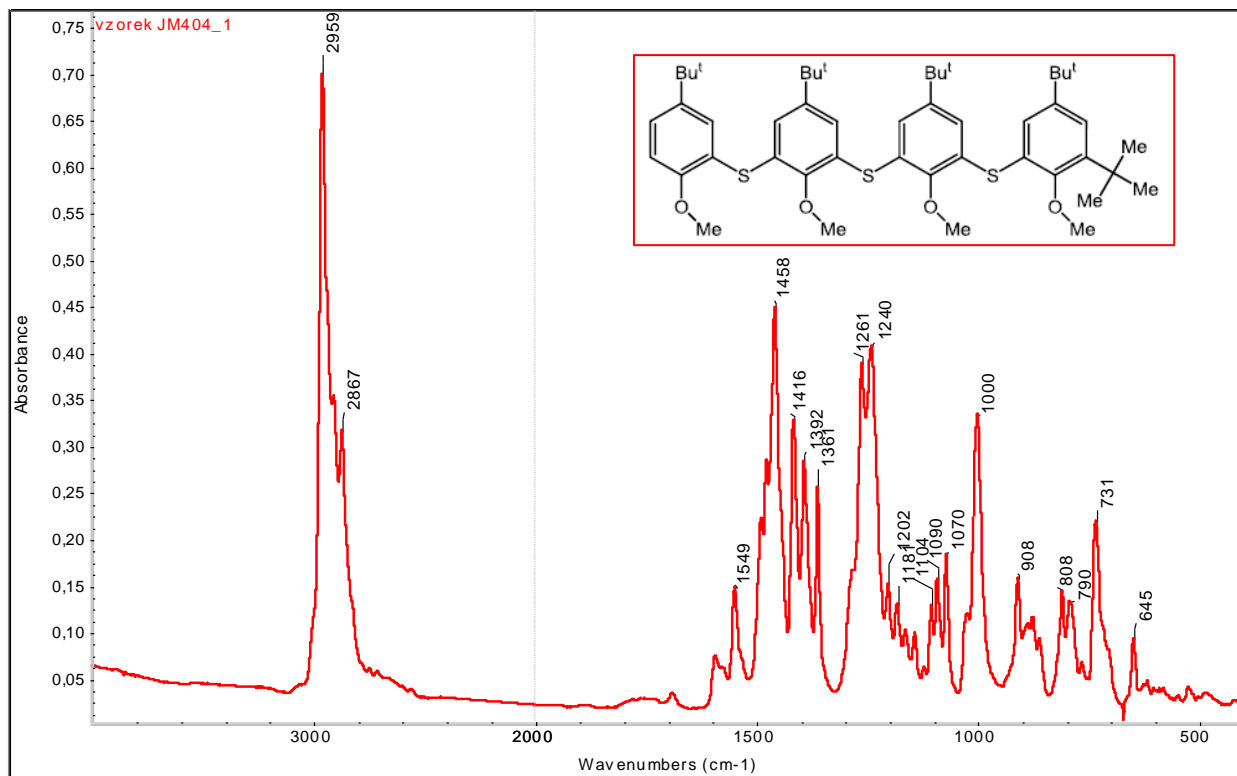


¹H NMR spectrum of compound 8 (CDCl₃, 298K, 400 MHz)

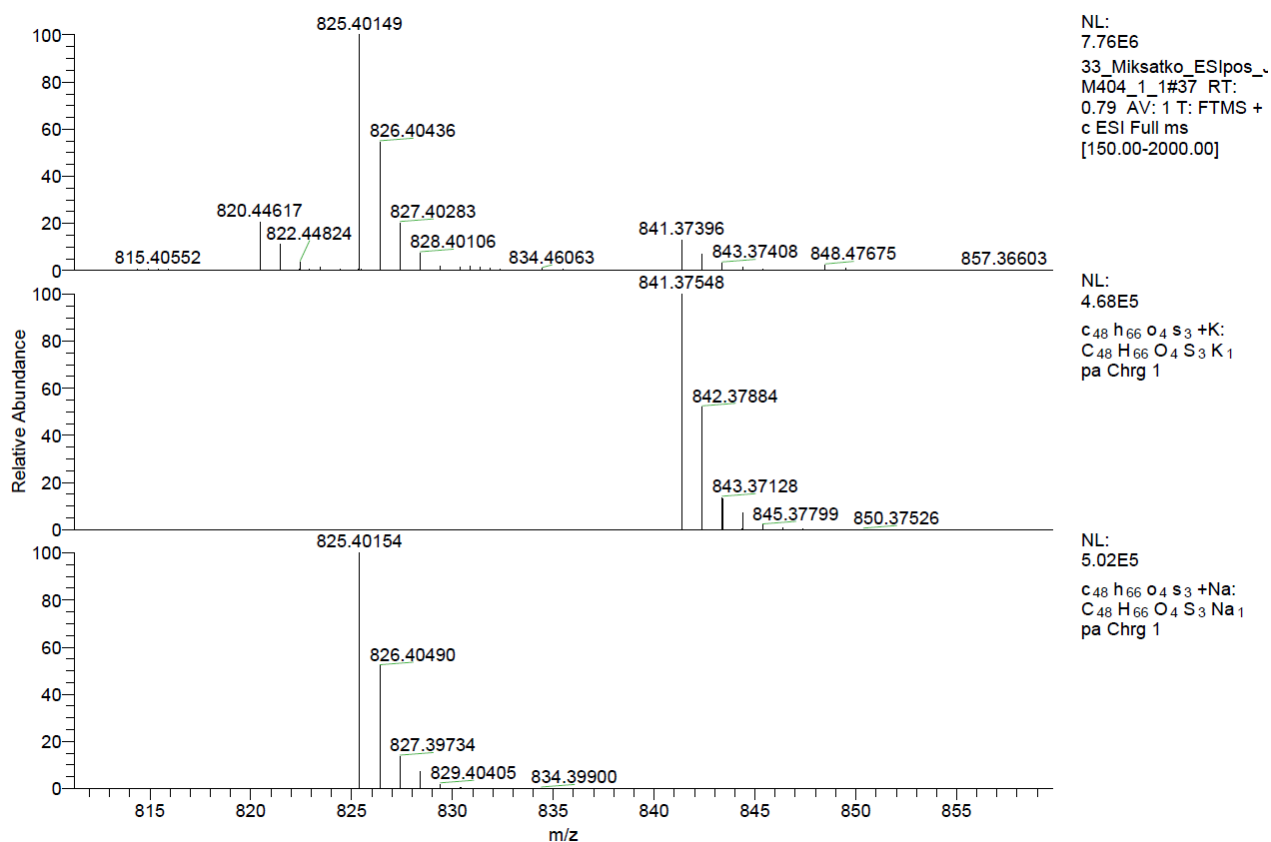


¹³C NMR (APT) spectrum of compound 8 (CDCl₃, 298K, 100 MHz)

Compound 8

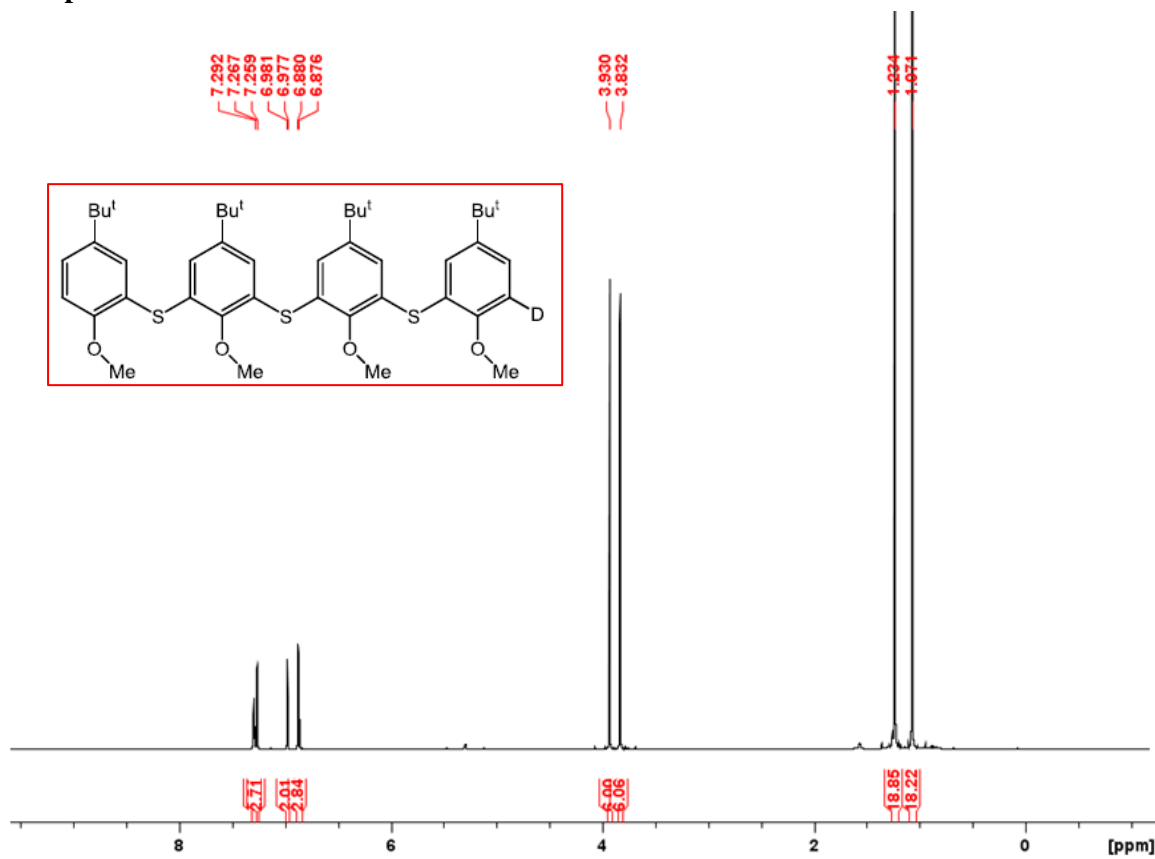


IR (ATR) spectrum of compound 8

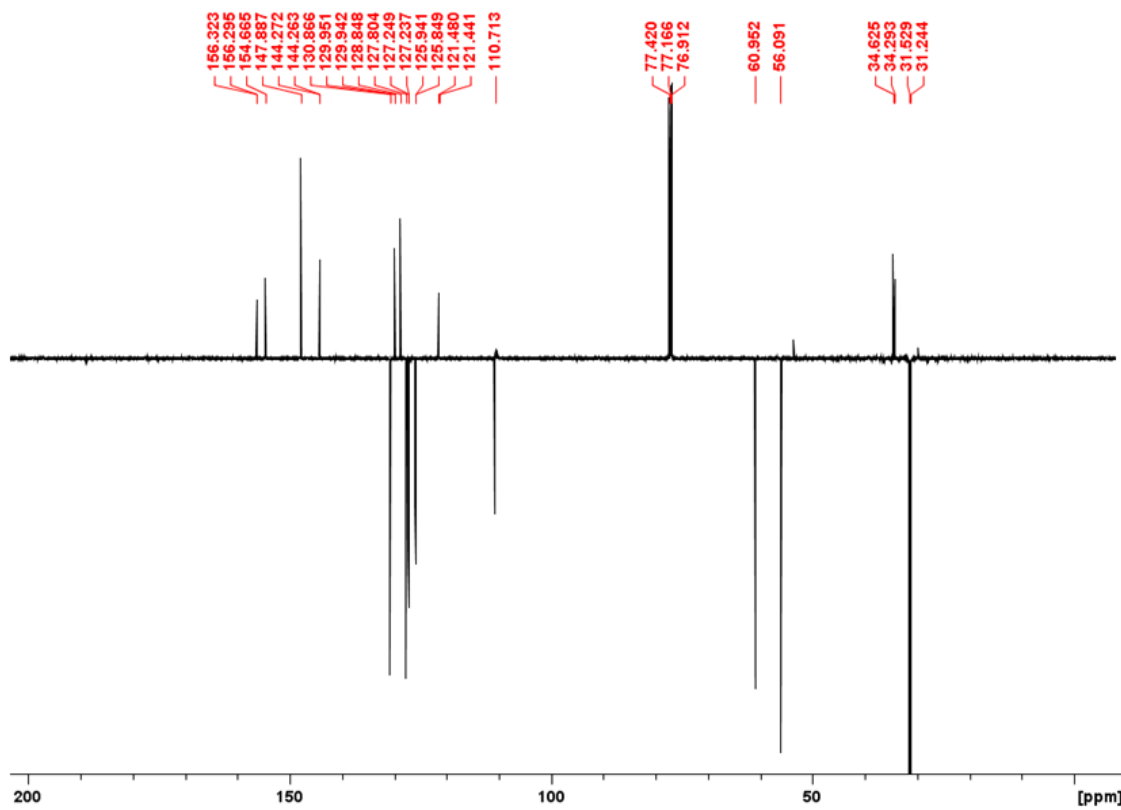


HRMS (ESI⁺) spectrum of compound 8

Compound 9

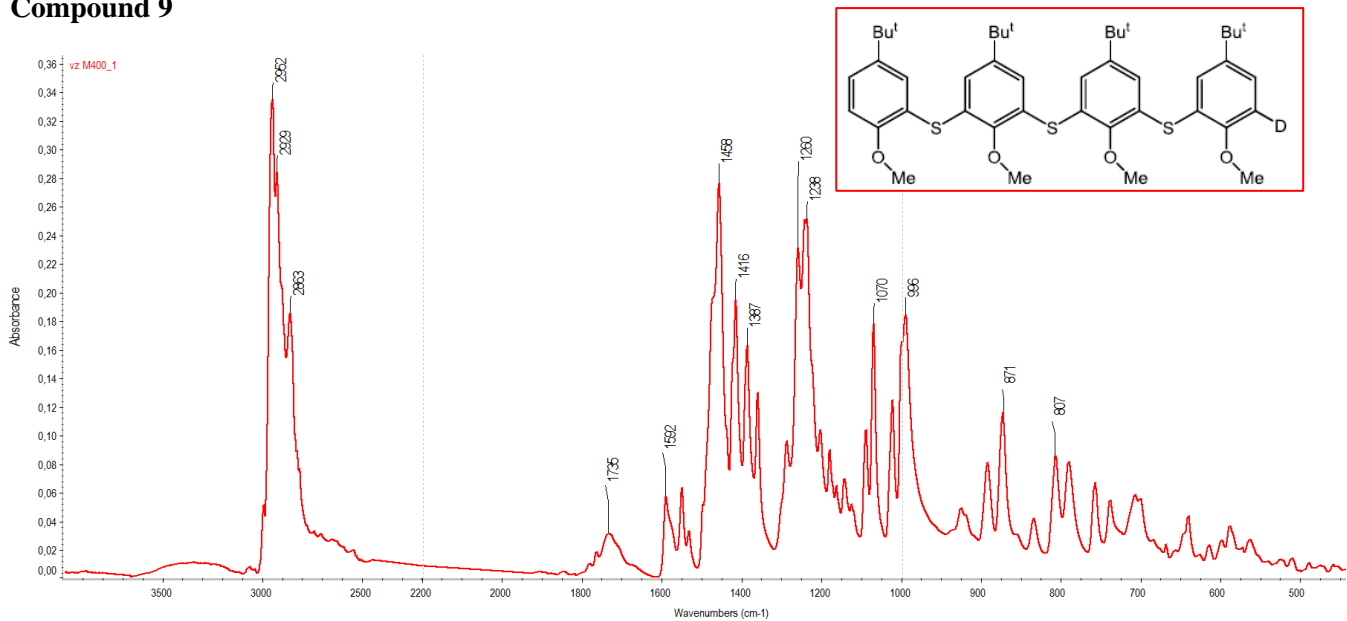


¹H NMR spectrum of compound **9** (CDCl₃, 298K, 400 MHz)

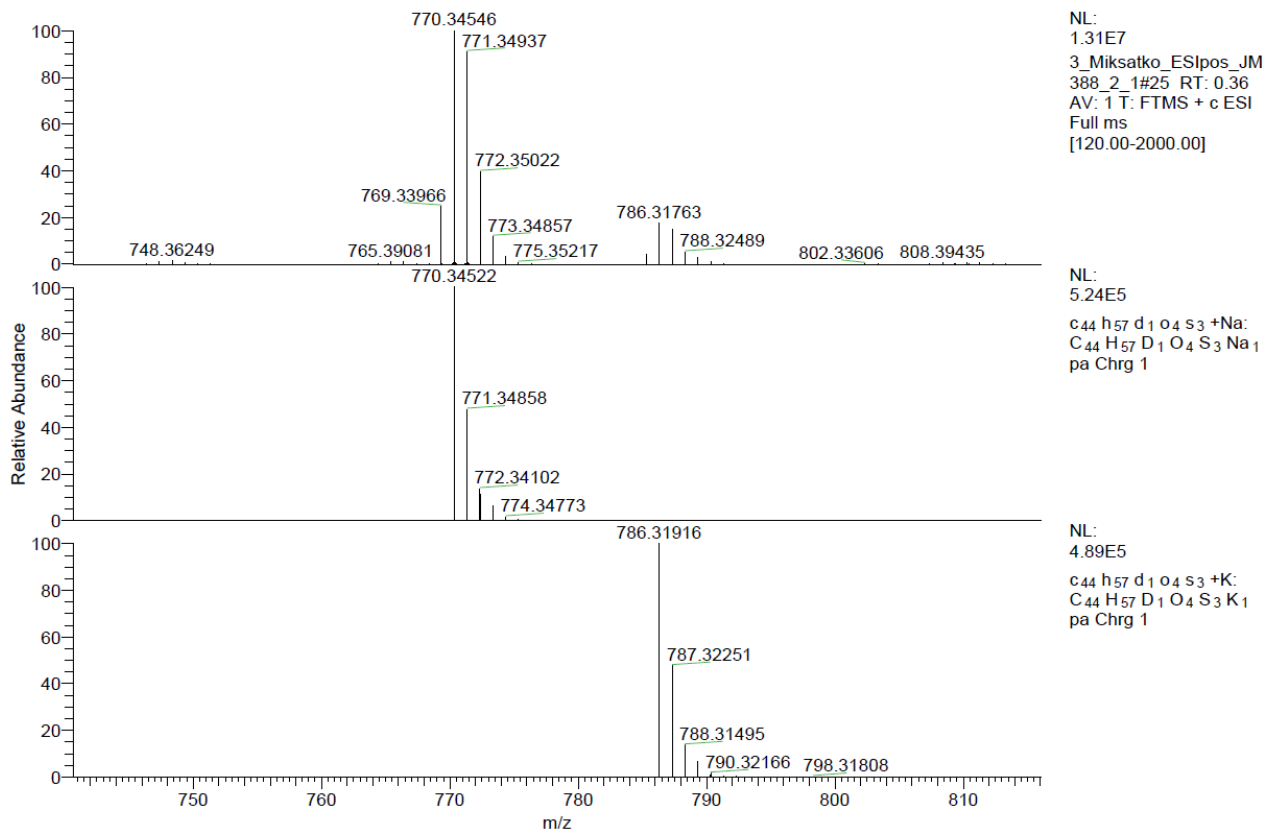


¹³C NMR (APT) spectrum of compound **9** (CDCl₃, 298K, 100 MHz)

Compound 9

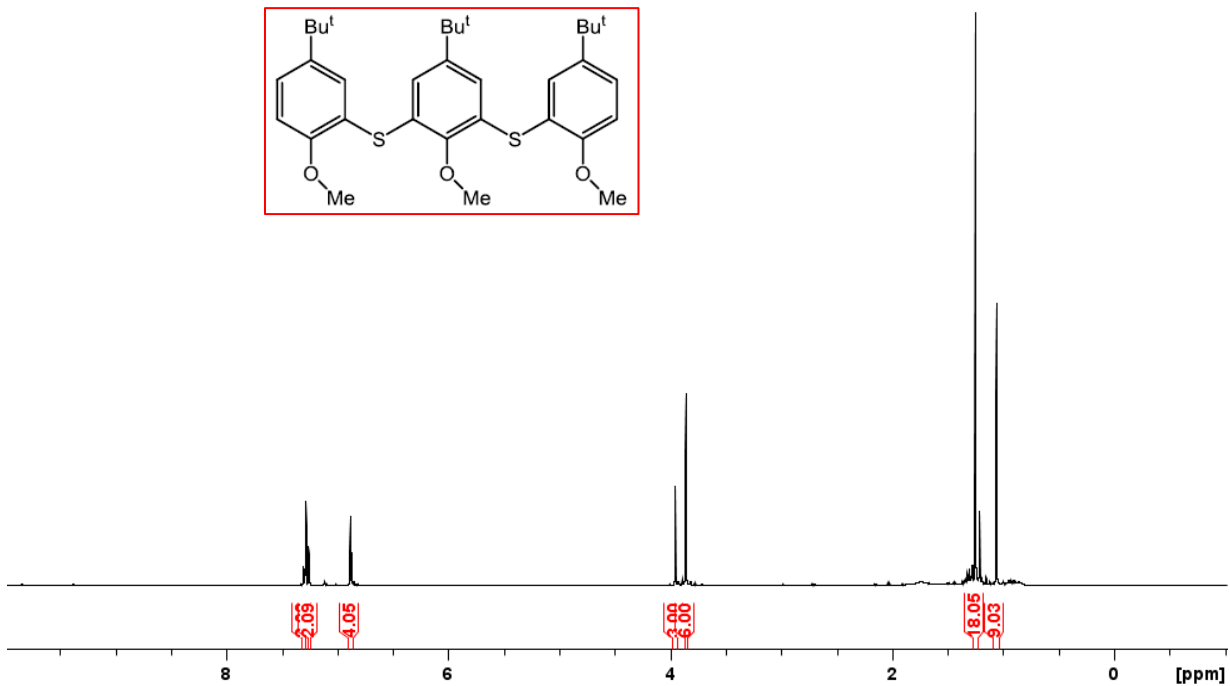
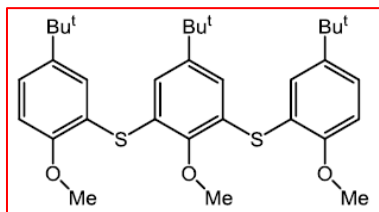


IR (ATR) spectrum of compound 9

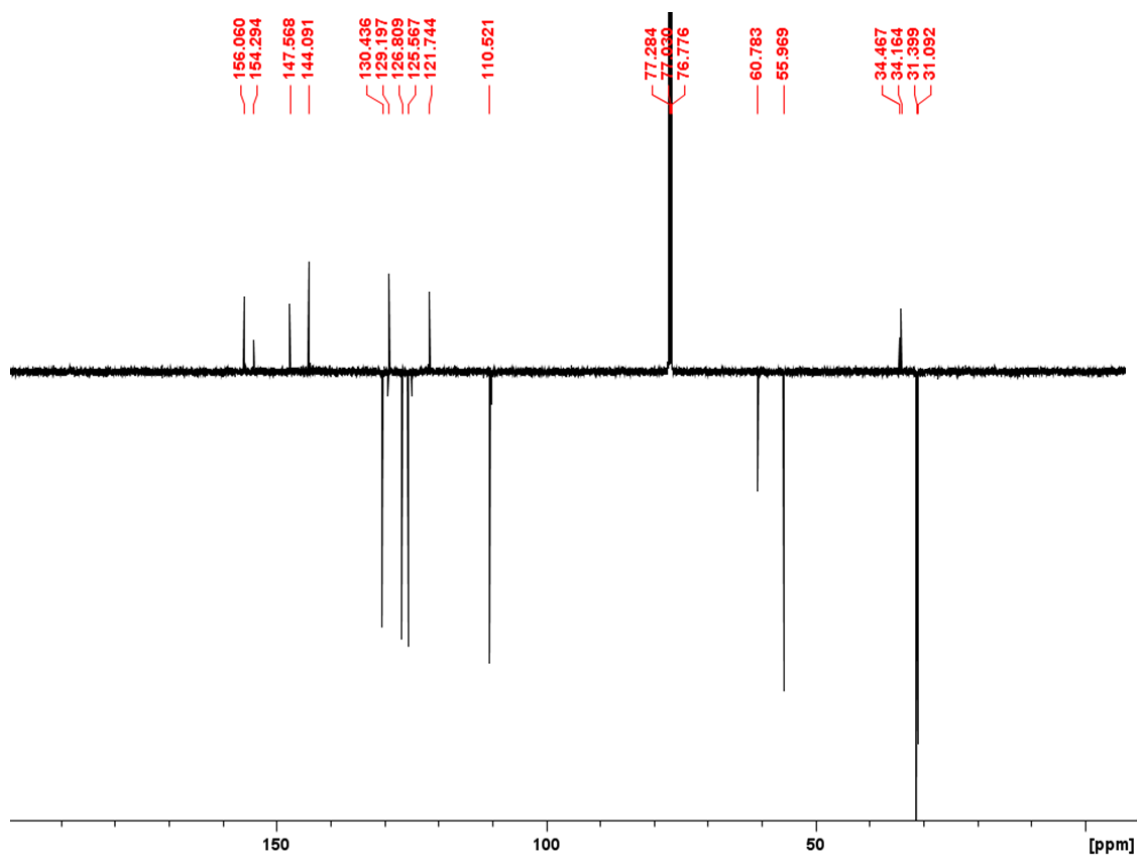


HRMS (ESI⁺) spectrum of compound 9

Compound 17

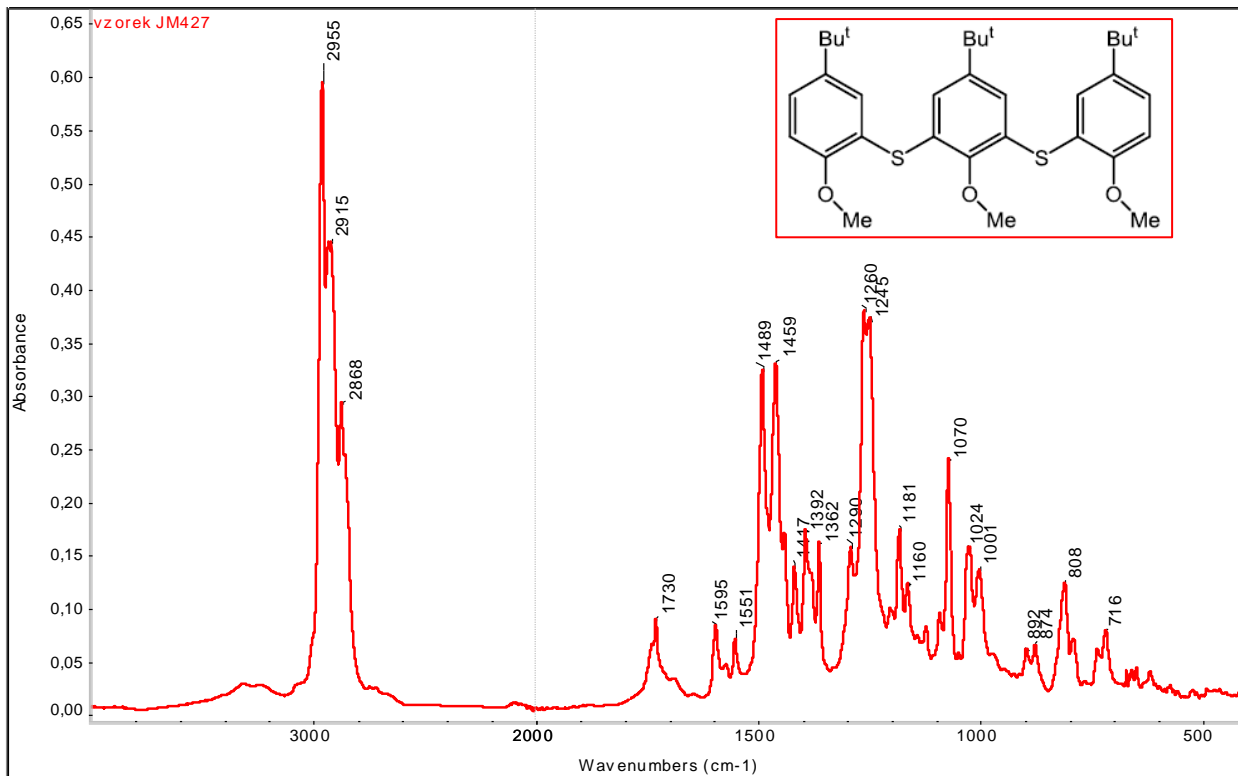


¹H NMR spectrum of compound 17 (CDCl₃, 298K, 400 MHz)

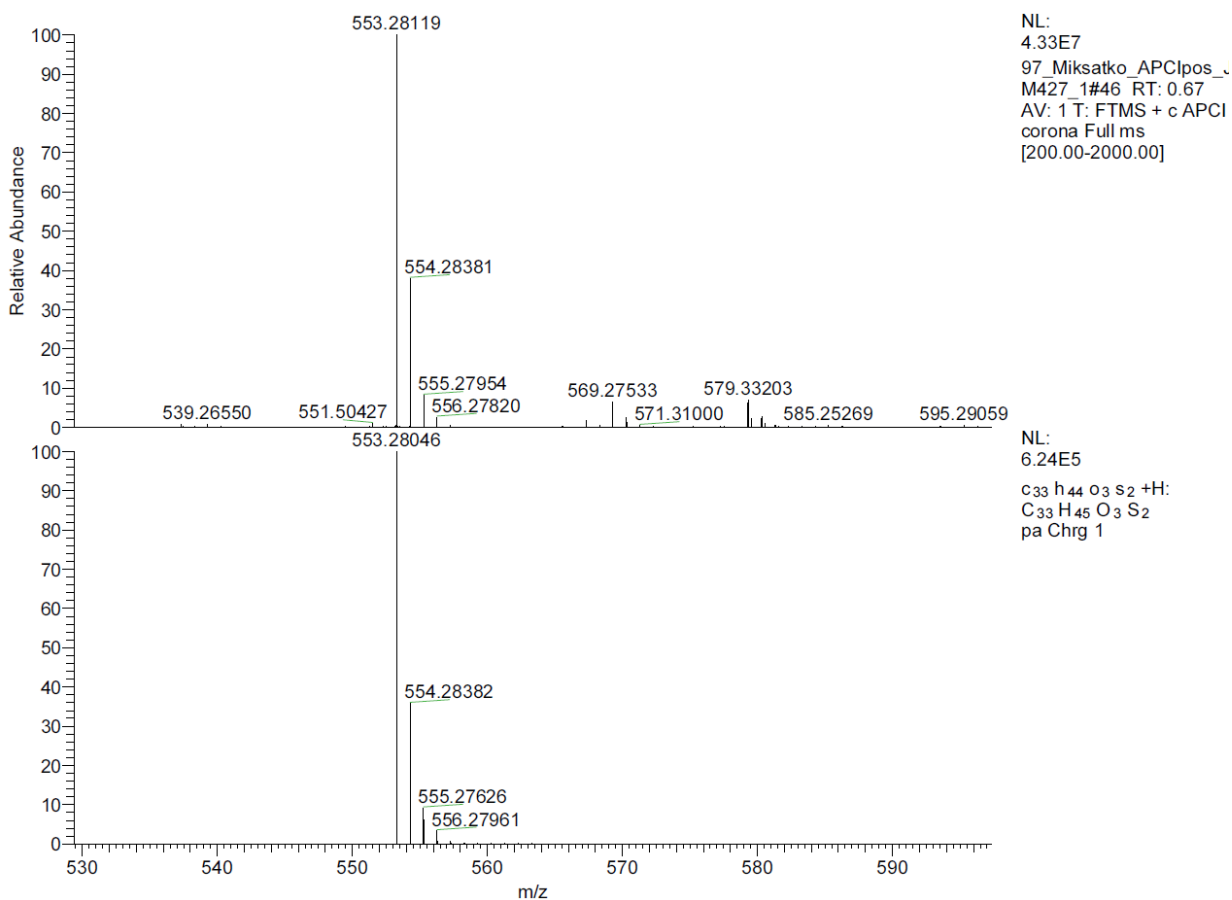


¹³C NMR (APT) spectrum of compound 17 (CDCl₃, 298K, 100 MHz)

Compound 17

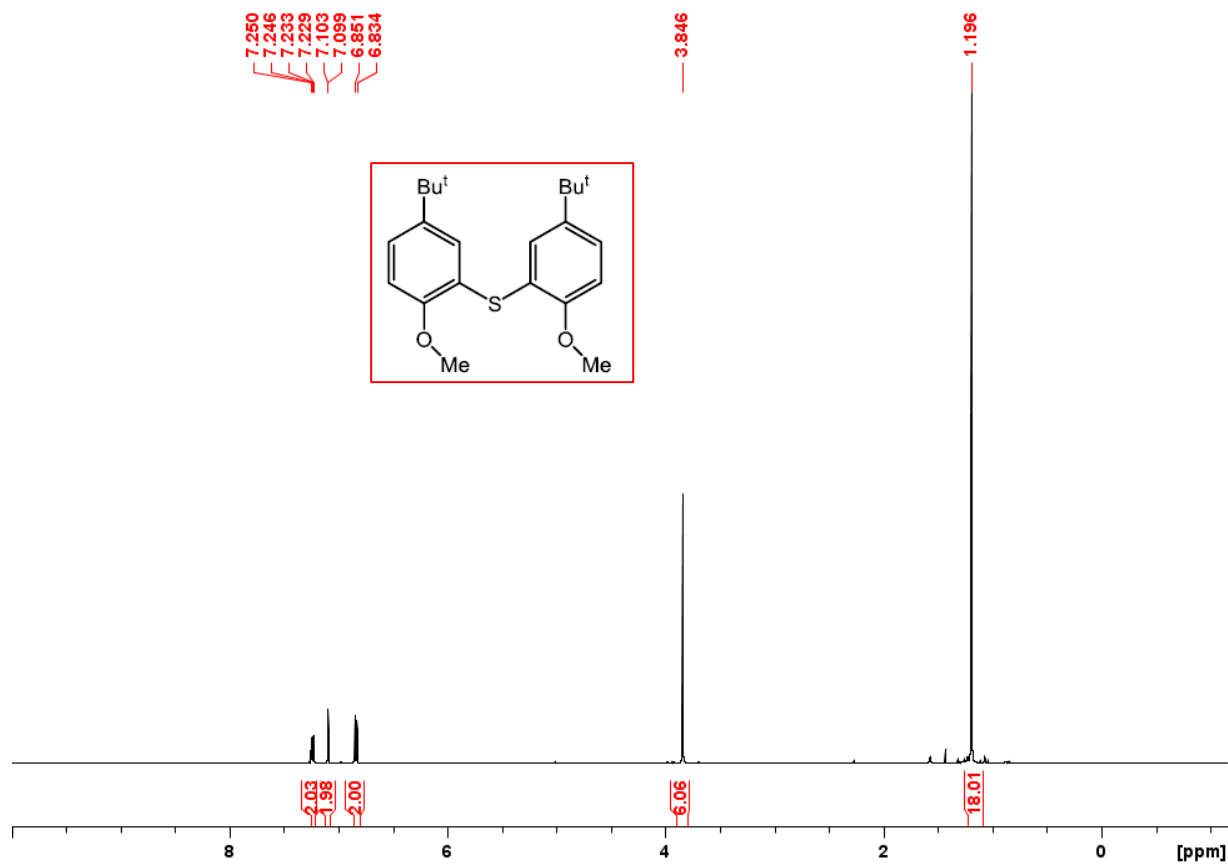


IR (ATR) spectrum of compound 17

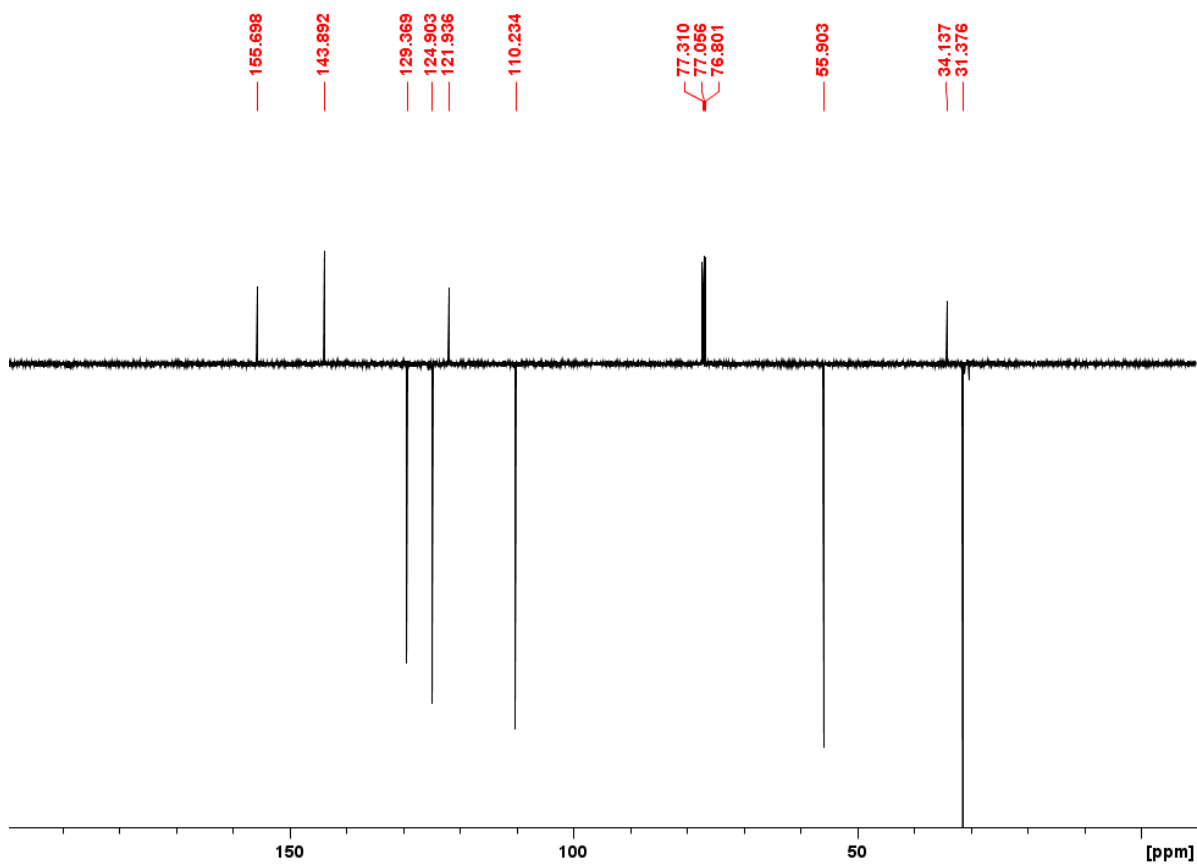


HRMS (ESI⁺) spectrum of compound 17

Compound 18

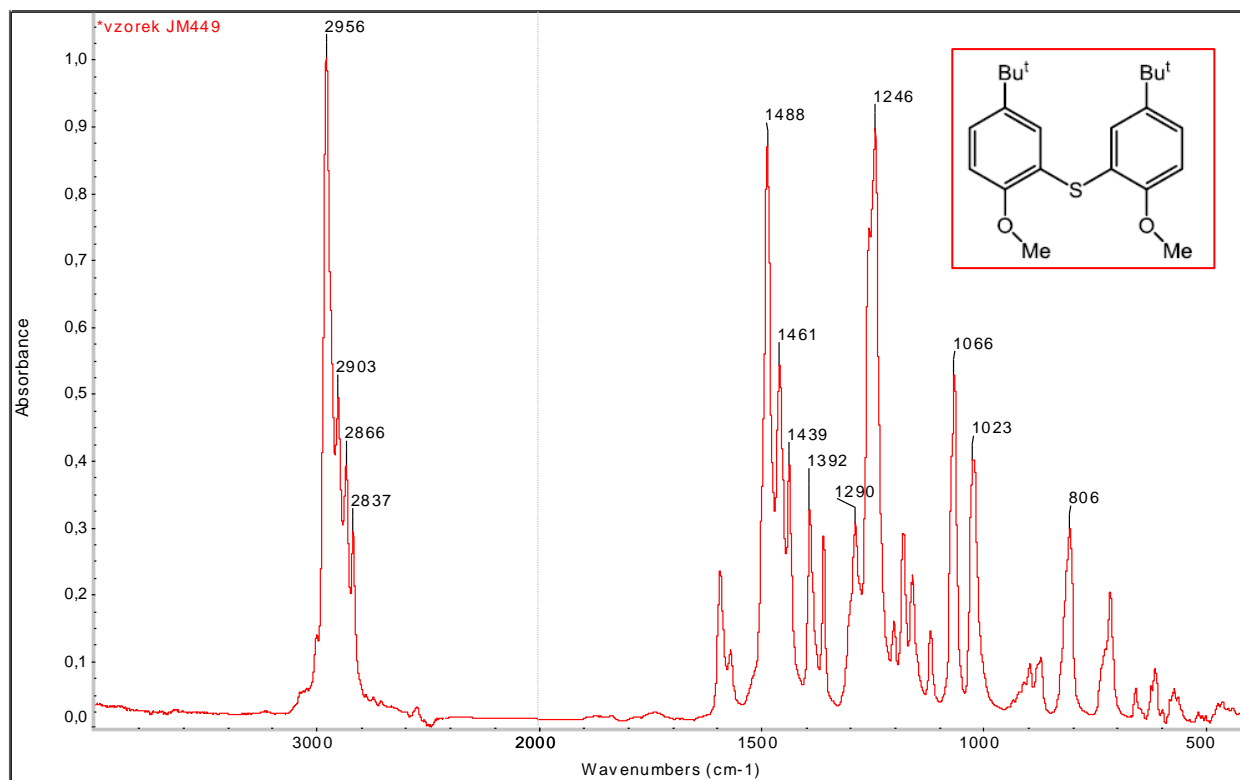


¹H NMR spectrum of compound **18** (CDCl₃, 298K, 400 MHz)

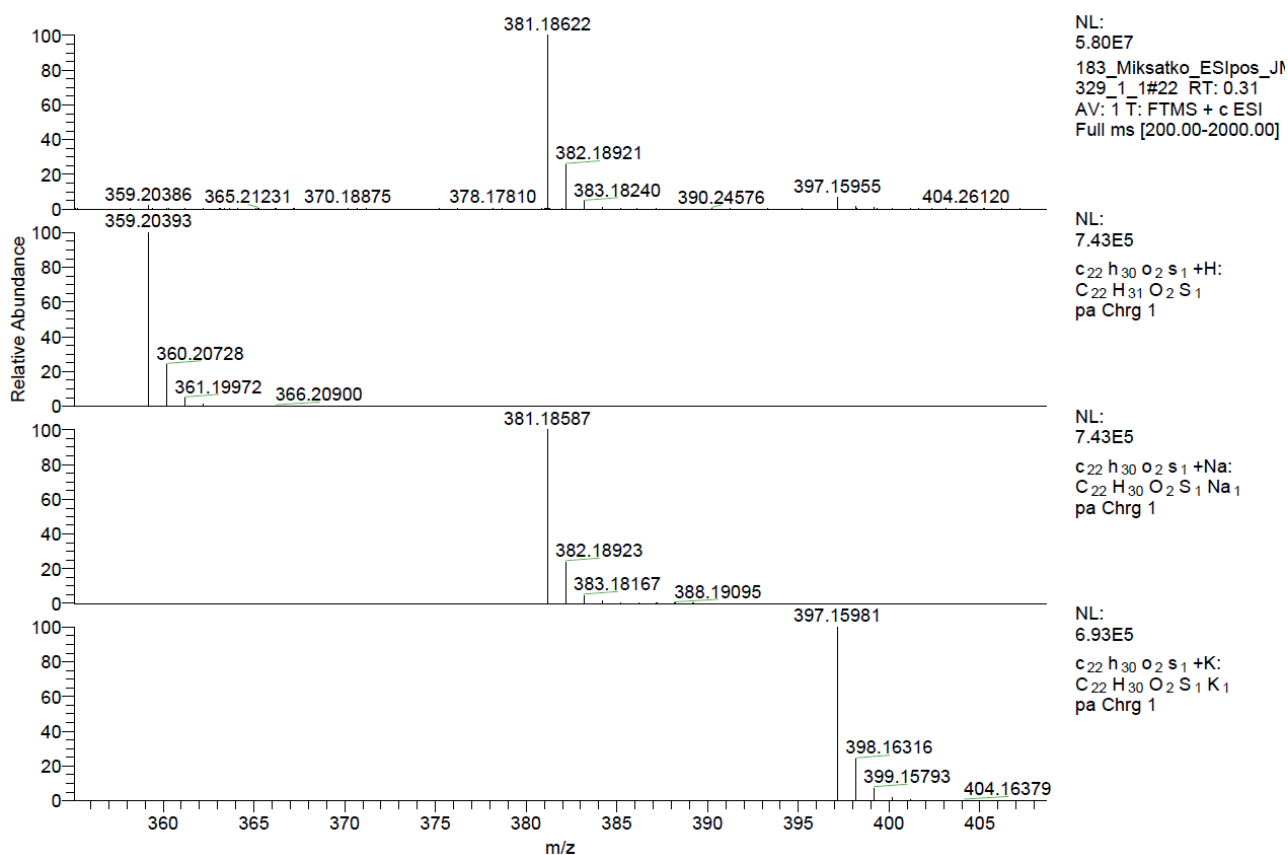


¹³C NMR (APT) spectrum of compound **18** (CDCl₃, 298K, 100 MHz)

Compound 18

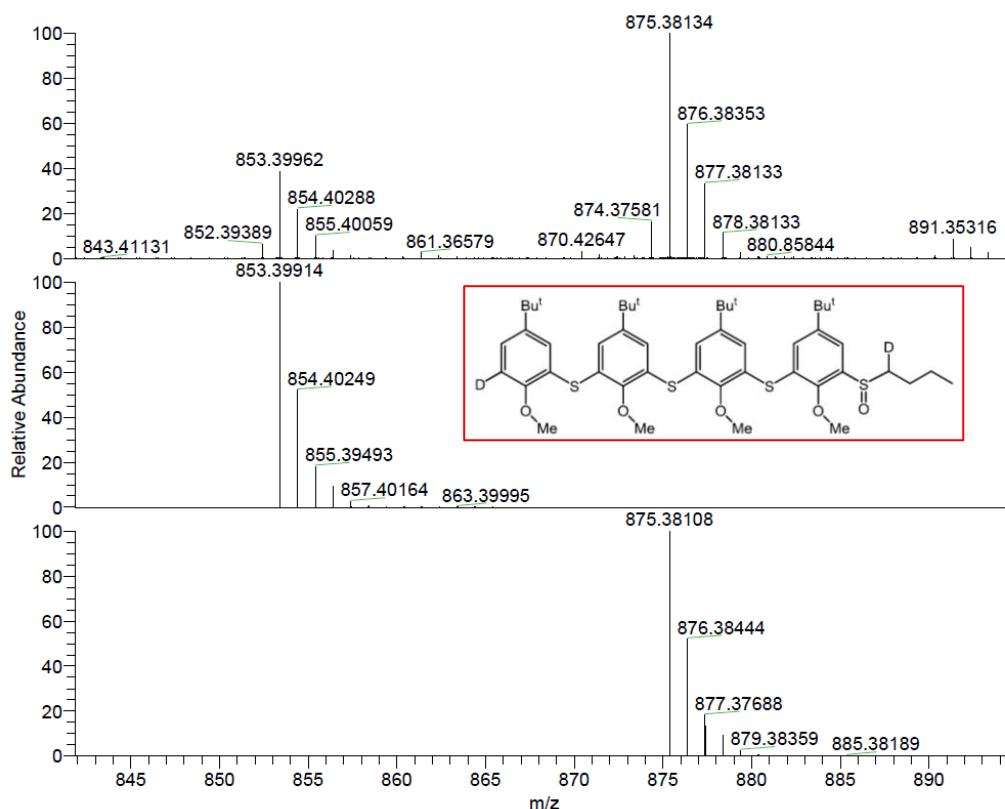


IR (ATR) spectrum of compound **18**



HRMS (ESI⁺) spectrum of compound **18**

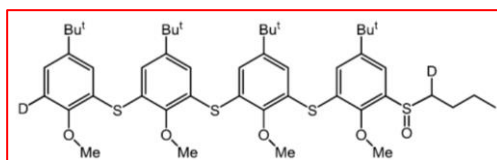
Compound 10



NL:
1.52E7
167_Miksatko_ESIpos_JM
447_2_1#31-34 RT:
0.66-0.73 AV: 4 T: FTMS
+ c ESI Full ms
[200.00-2000.00]

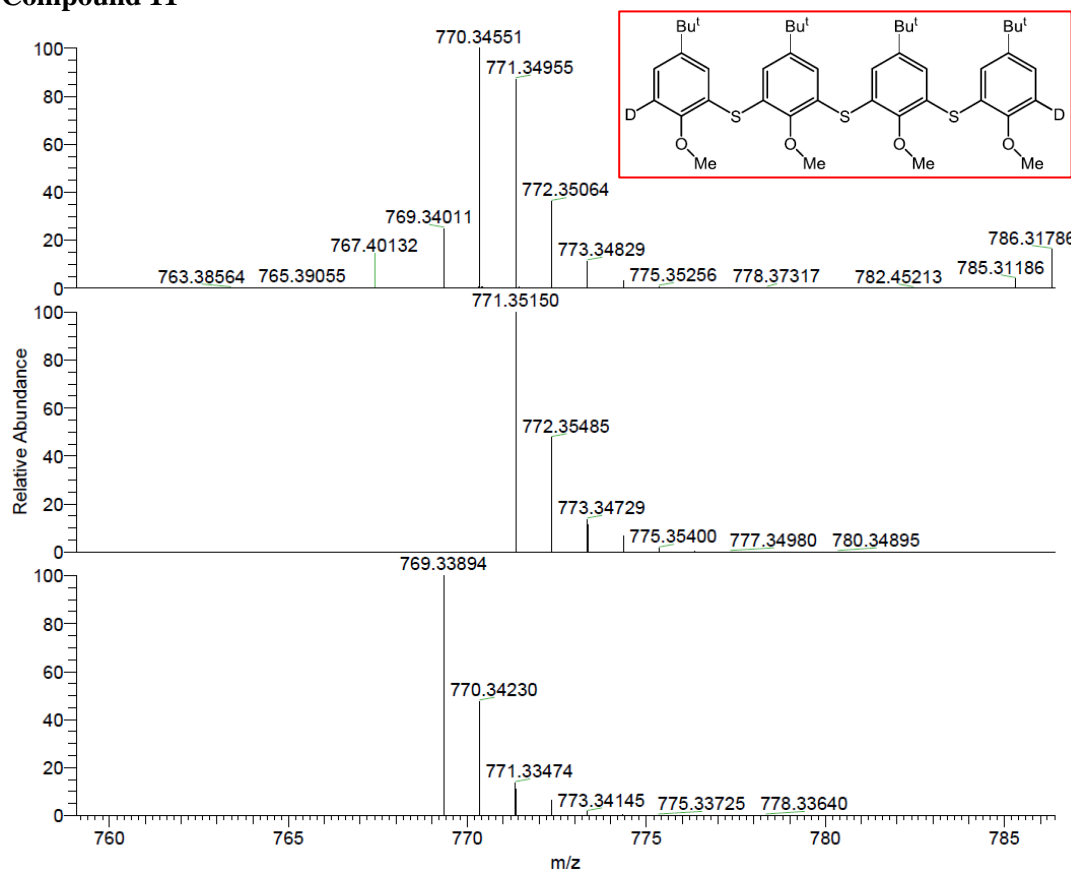
NL:
4.75E5
C₄₈ H₆₄ D₂ O₅ S₄ +H:
C₄₈ H₆₄ D₂ O₅ S₄
pa Chrg 1

NL:
4.75E5
C₄₈ H₆₄ D₂ O₅ S₄ +Na:
C₄₈ H₆₄ D₂ O₅ S₄ Na₁
pa Chrg 1



HRMS (ESI⁺) spectrum of compound 10

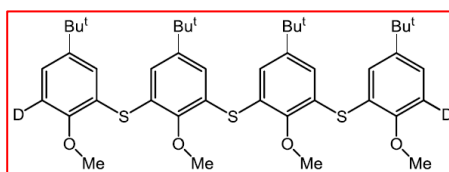
Compound 11



NL:
9.39E6
124_Miksatko_ESIpos_J
M434_2#23-31 RT:
0.52-0.73 AV: 9 T:
FTMS + c ESI Full ms
[200.00-2000.00]

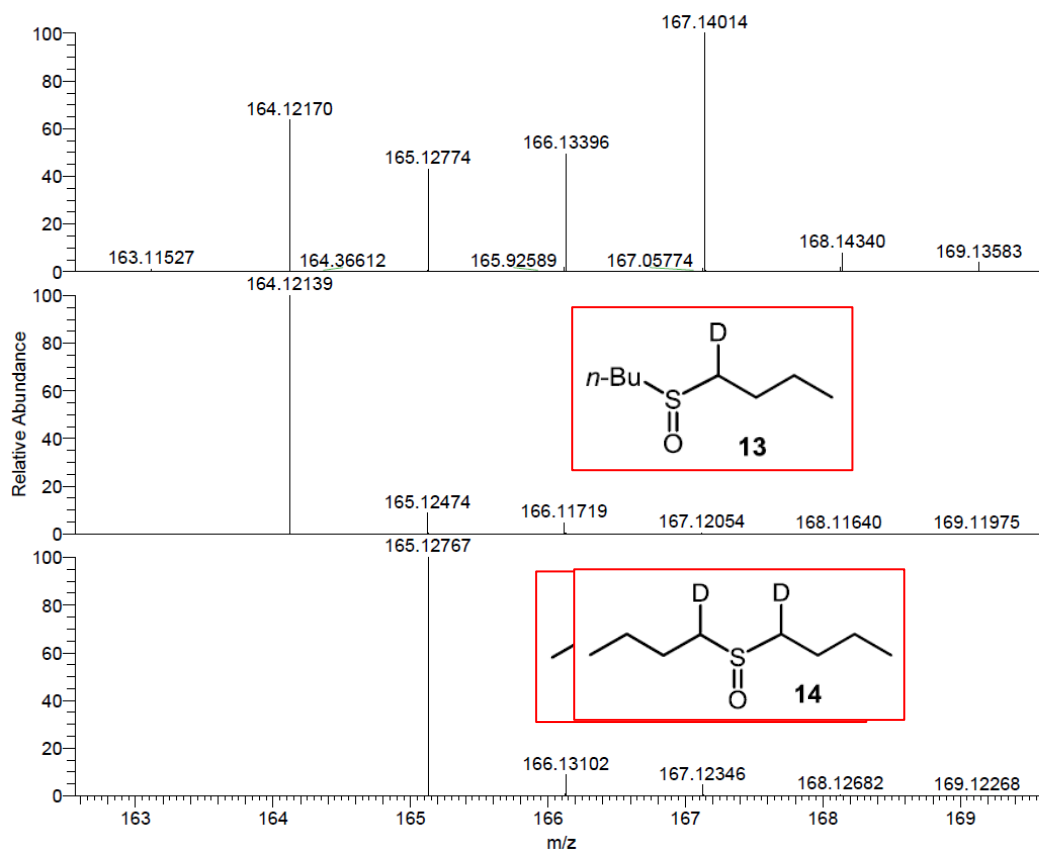
NL:
5.24E5
C₄₄ H₅₆ D₂ O₄ S₃ +Na:
C₄₄ H₅₆ D₂ O₄ S₃ Na₁
pa Chrg 1

NL:
5.24E5
C₄₄ H₅₈ O₄ S₃ +Na:
C₄₄ H₅₈ O₄ S₃ Na₁
pa Chrg 1



HRMS (ESI⁺) spectrum of compound 11

Compounds 13 and 14



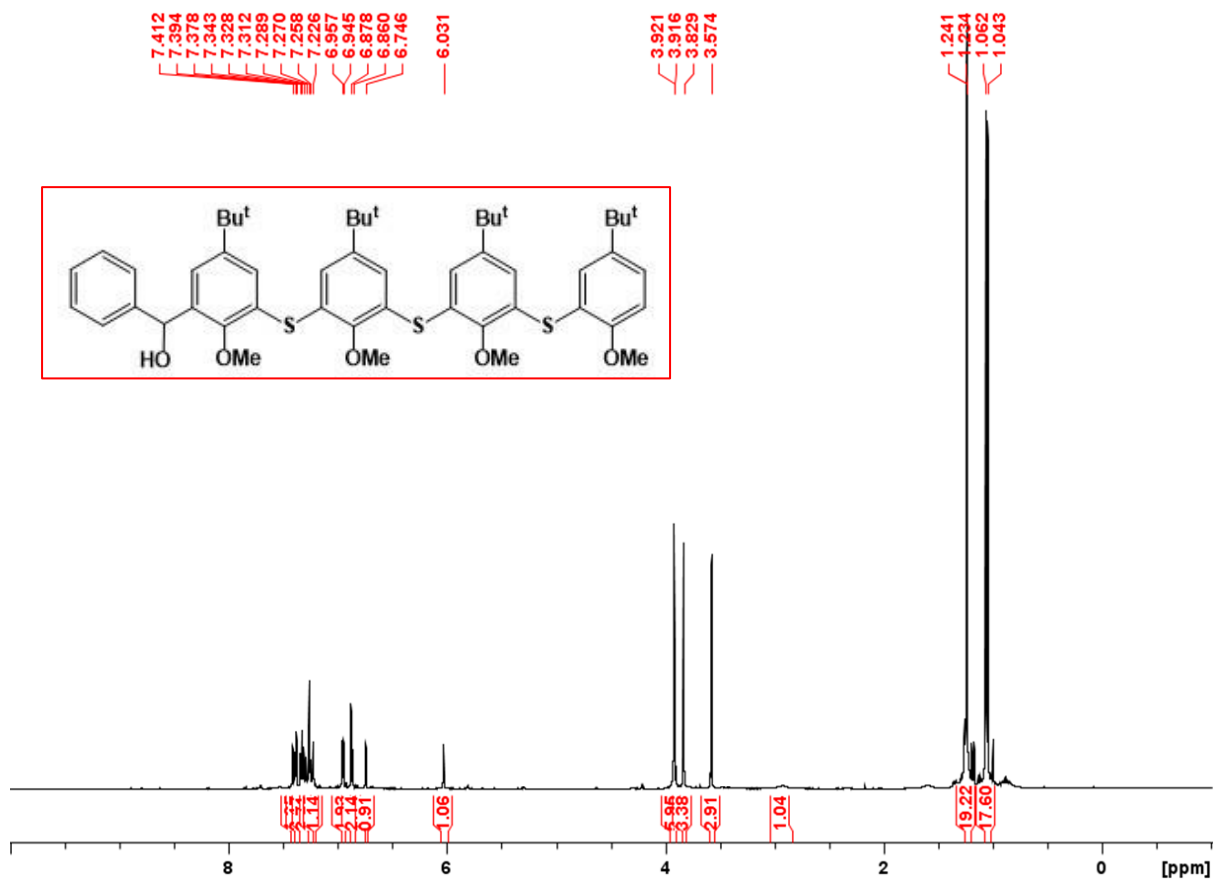
NL:
8.69E6
167_Miksatko_ESIpos_J
M447_X_2#26-28 RT:
0.55-0.59 AV: 3 T: FTMS
+ c ESI Full ms
[50.00-1000.00]

NL:
8.67E5
C₈H₁₇D₁O₁S₁+H:
C₈H₁₈D₁O₁S₁
pa Chrg 1

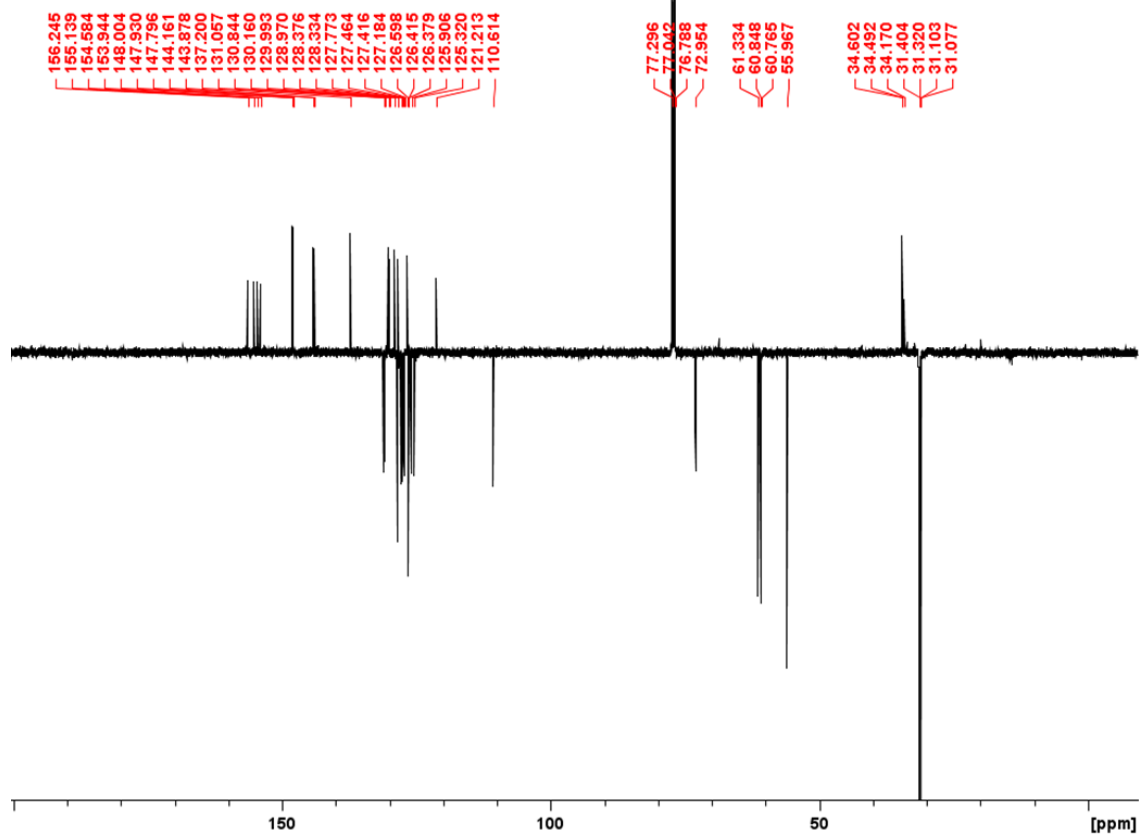
NL:
8.67E5
C₈H₁₆D₂O₁S₁+H:
C₈H₁₇D₂O₁S₁
pa Chrg 1

HRMS (ESI⁺) spectrum of compounds **13** and **14**

Compound 19

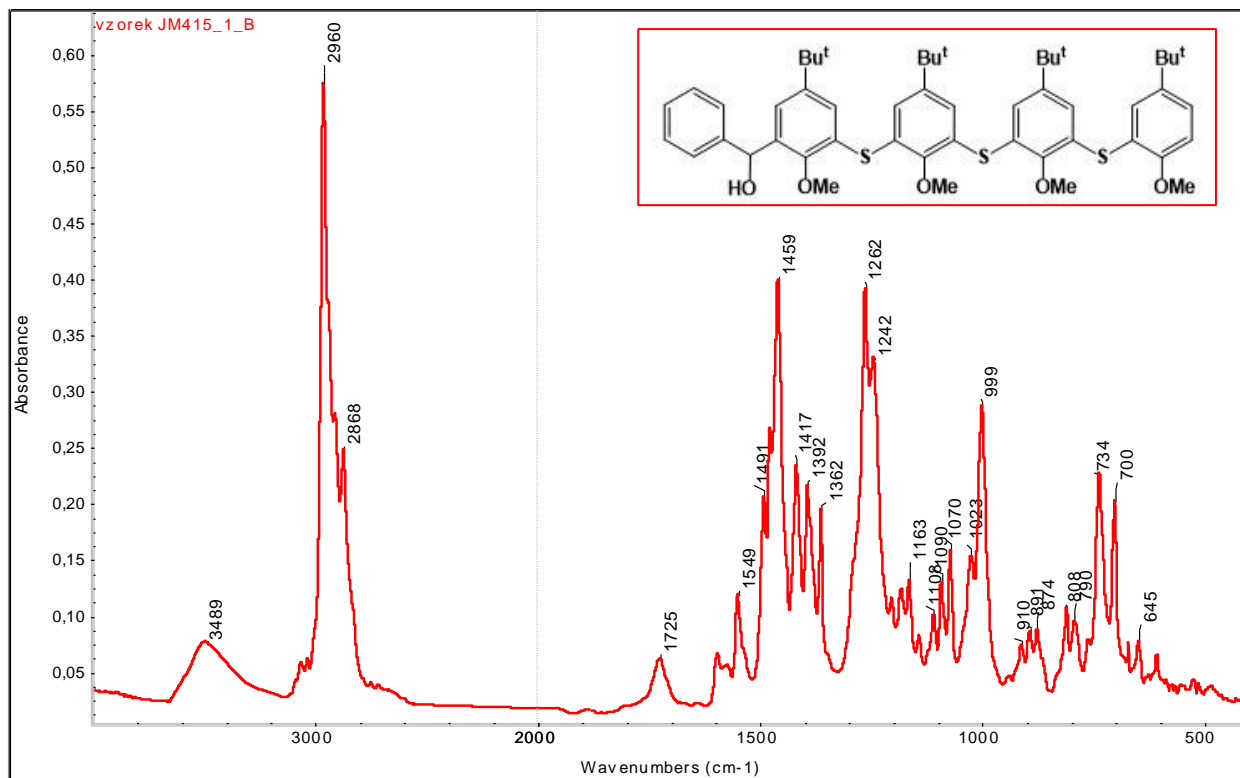


¹H NMR spectrum of compound 19 (CDCl₃, 298K, 400 MHz)

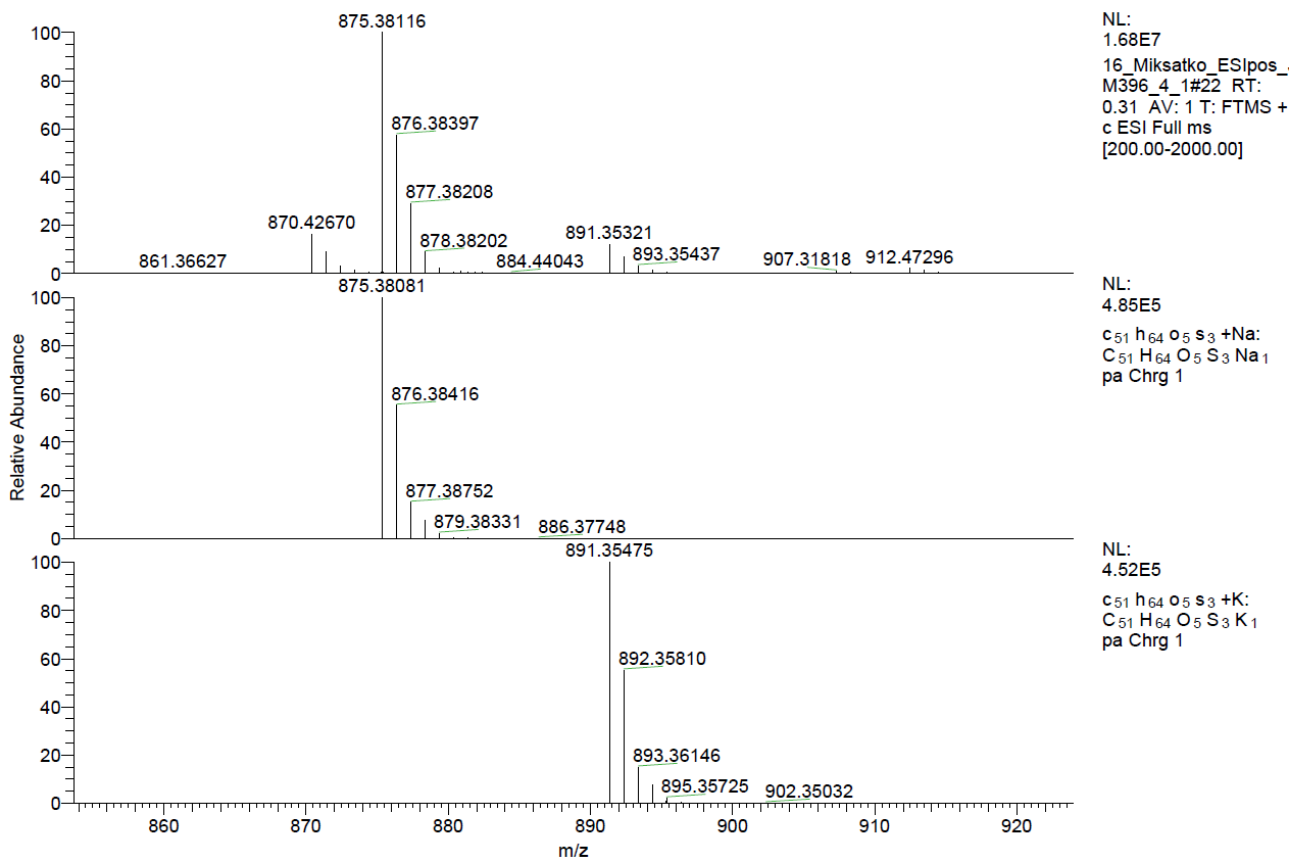


¹³C NMR (APT) spectrum of compound 19 (CDCl₃, 298K, 100 MHz)

Compound 19

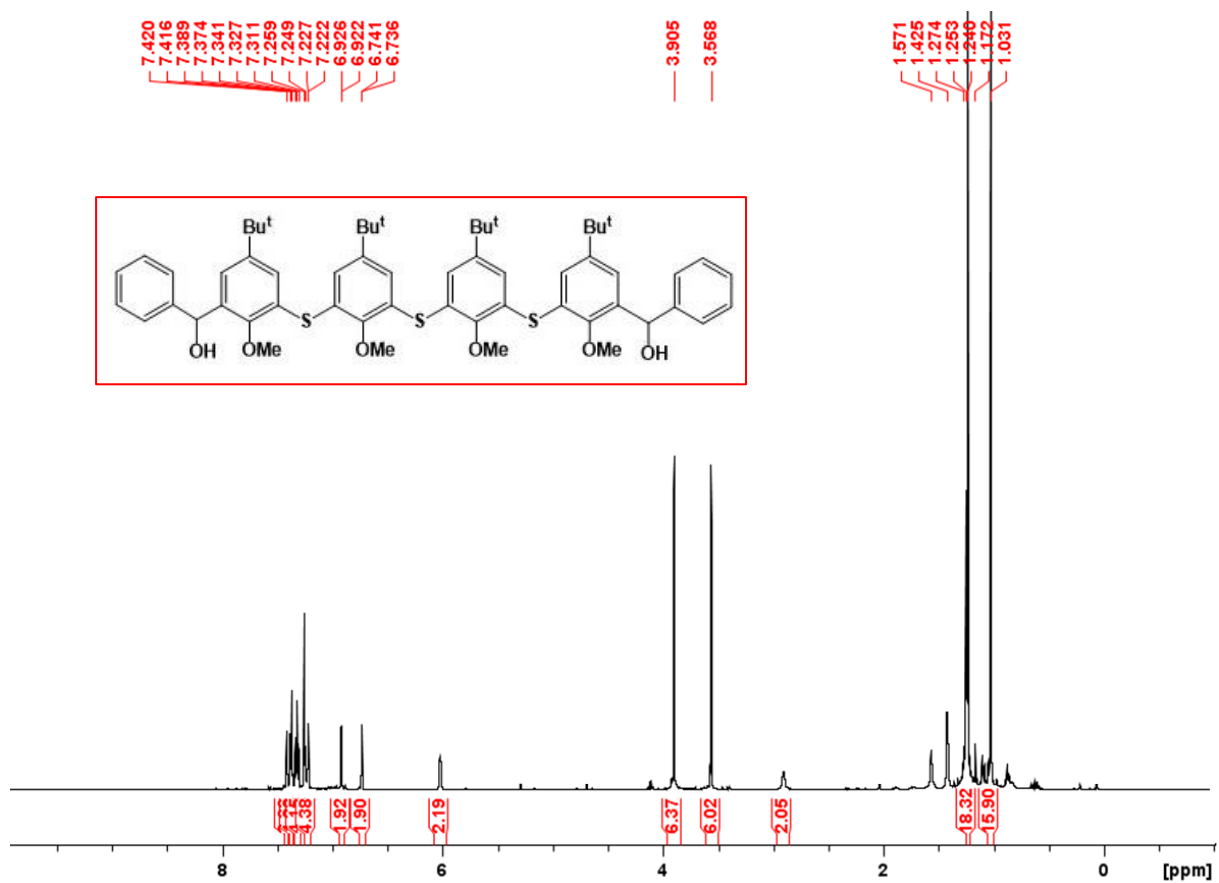


IR (ATR) spectrum of compound 19

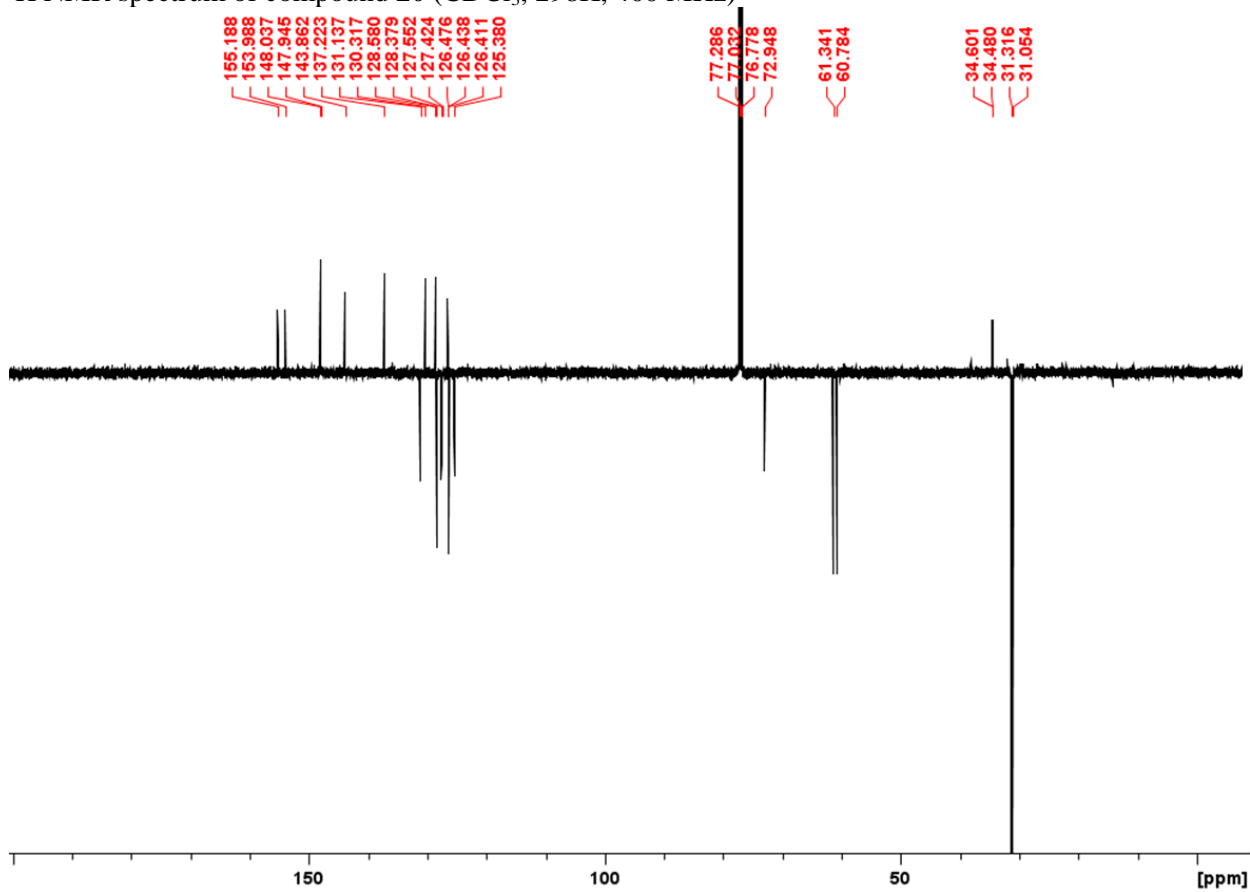


HRMS (ESI⁺) spectrum of compound 19

Compound 20

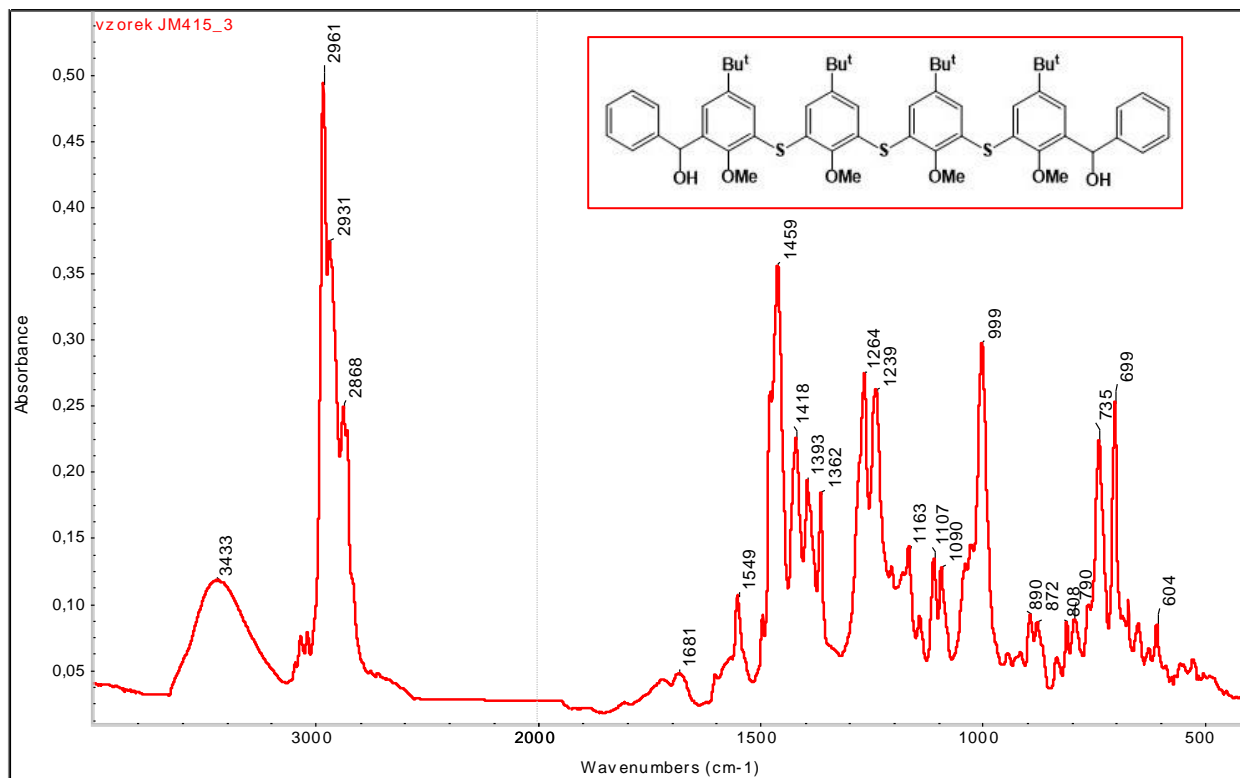


¹H NMR spectrum of compound **20** (CDCl₃, 298K, 400 MHz)

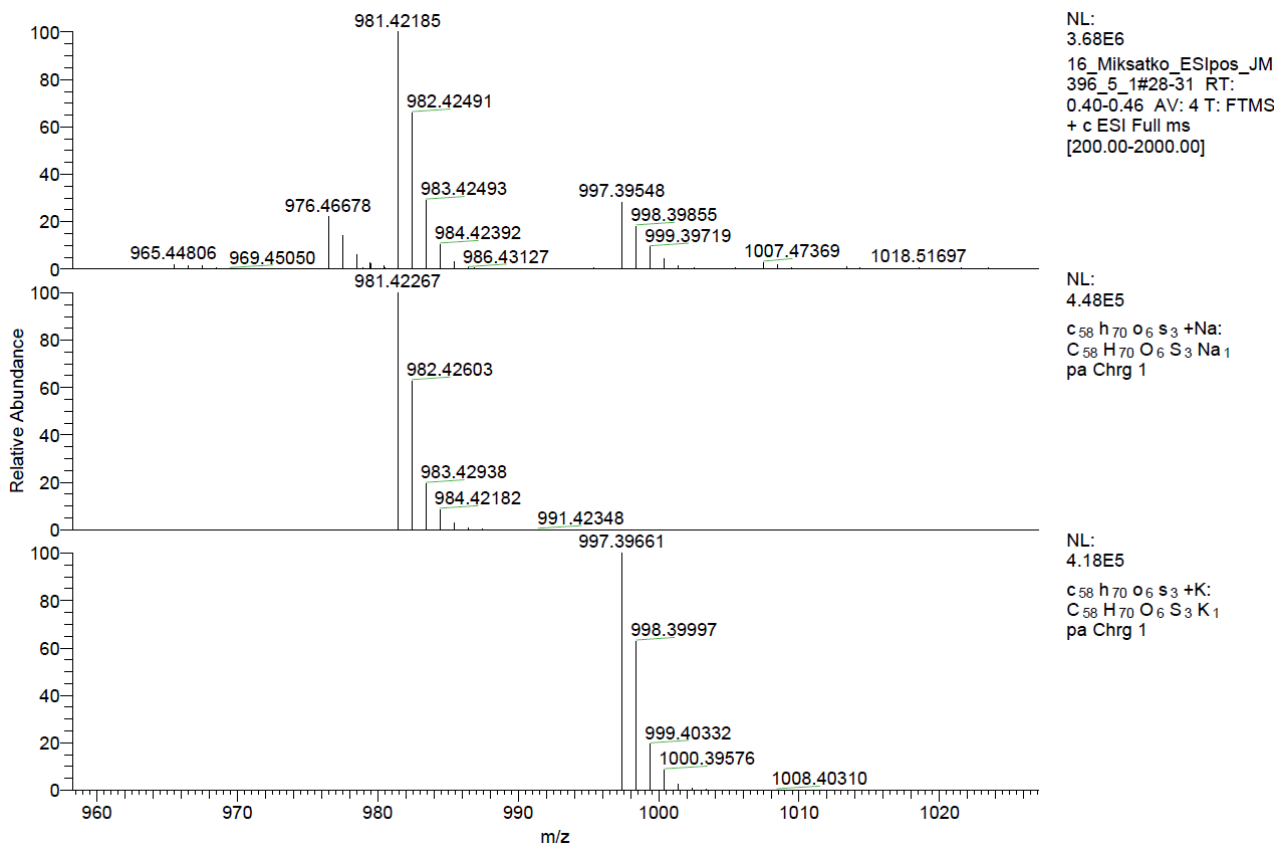


¹³C NMR (APT) spectrum of compound **20** (CDCl₃, 298K, 100 MHz)

Compound 20

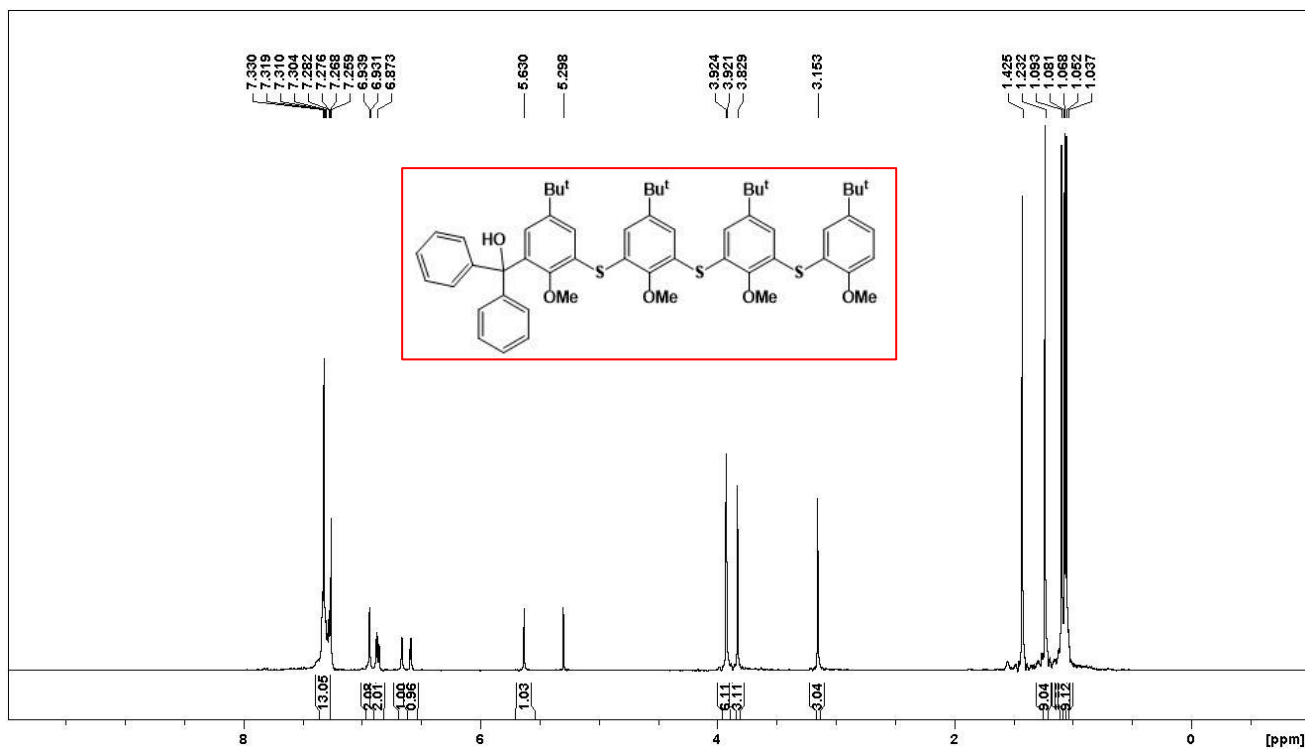


IR (ATR) spectrum of compound 20

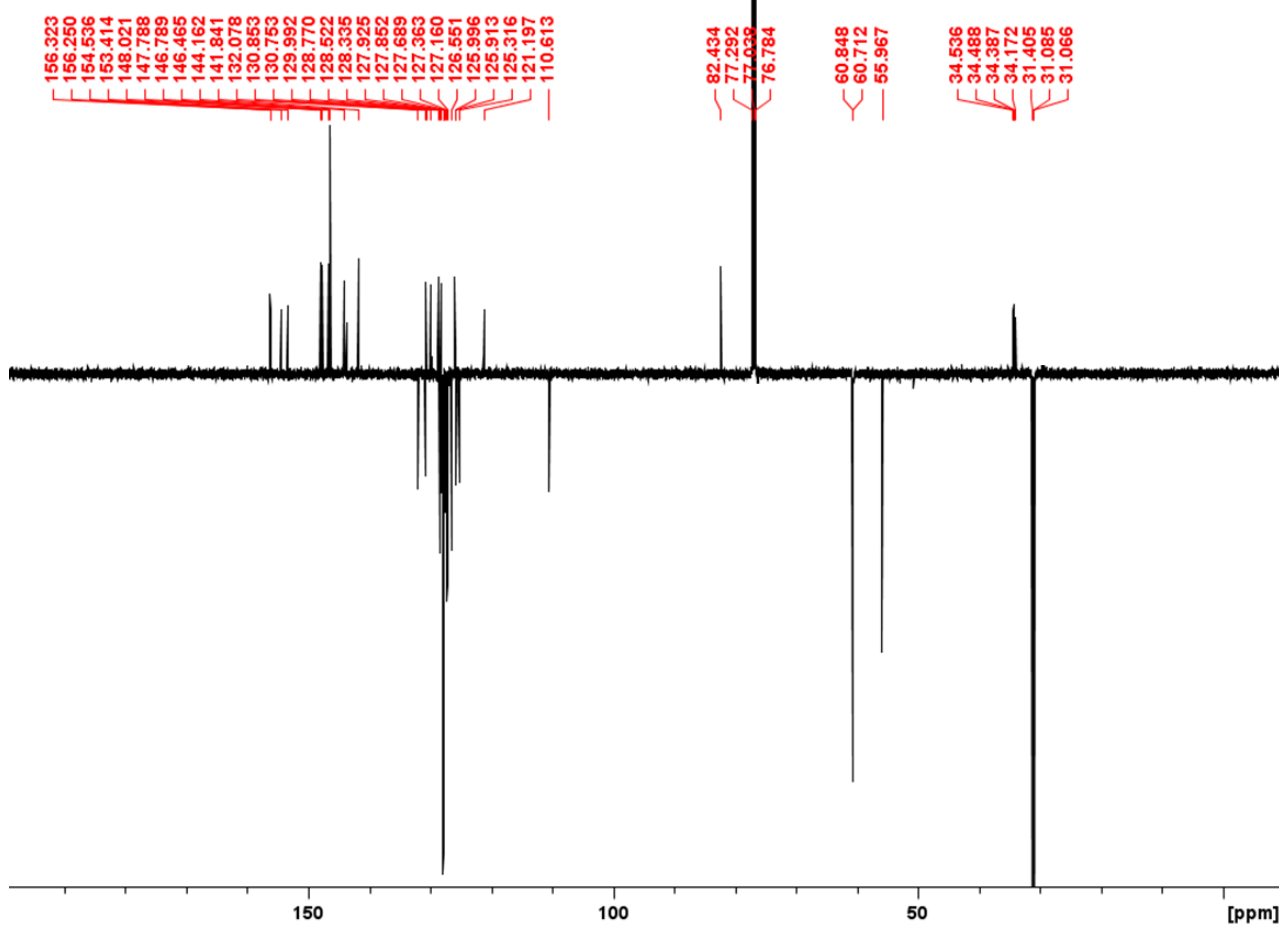


HRMS (ESI⁺) spectrum of compound 20

Compound 21

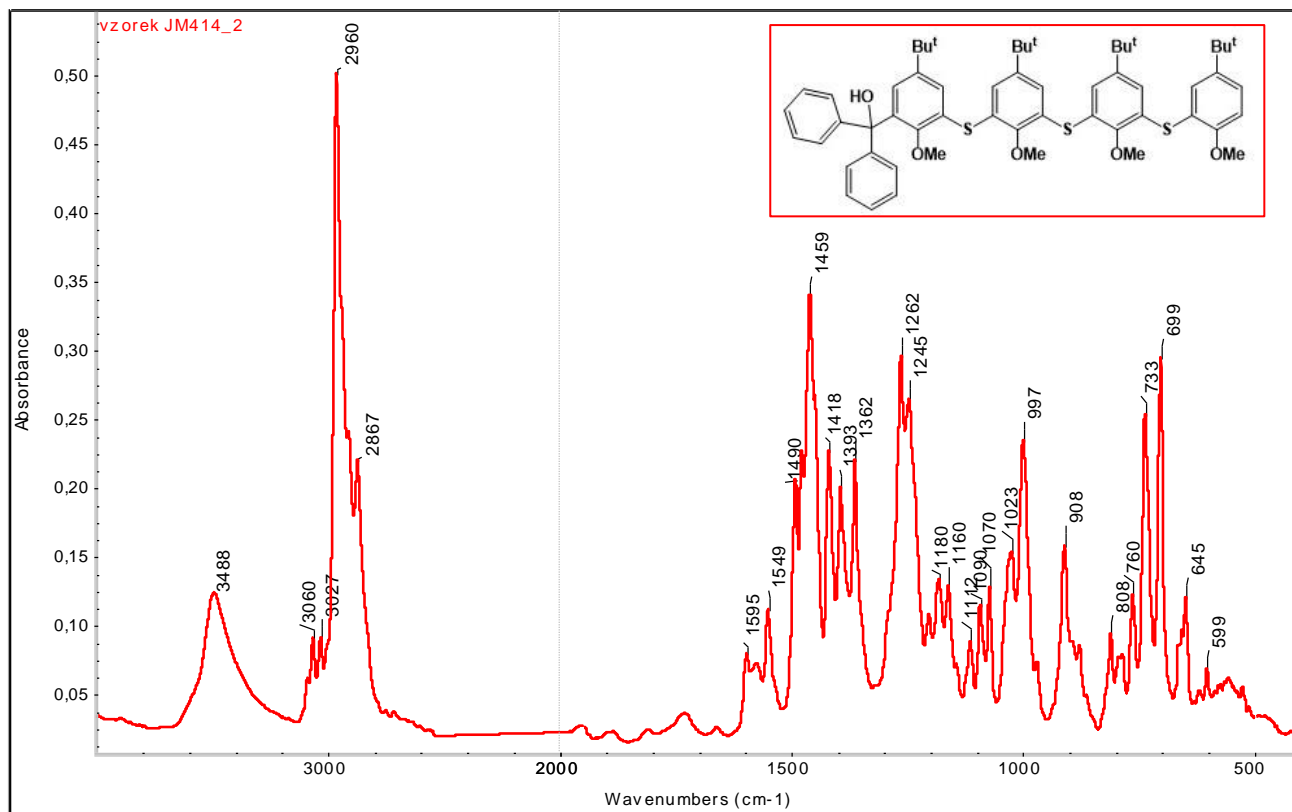


¹H NMR spectrum of compound **21** (CDCl₃, 298K, 400 MHz)

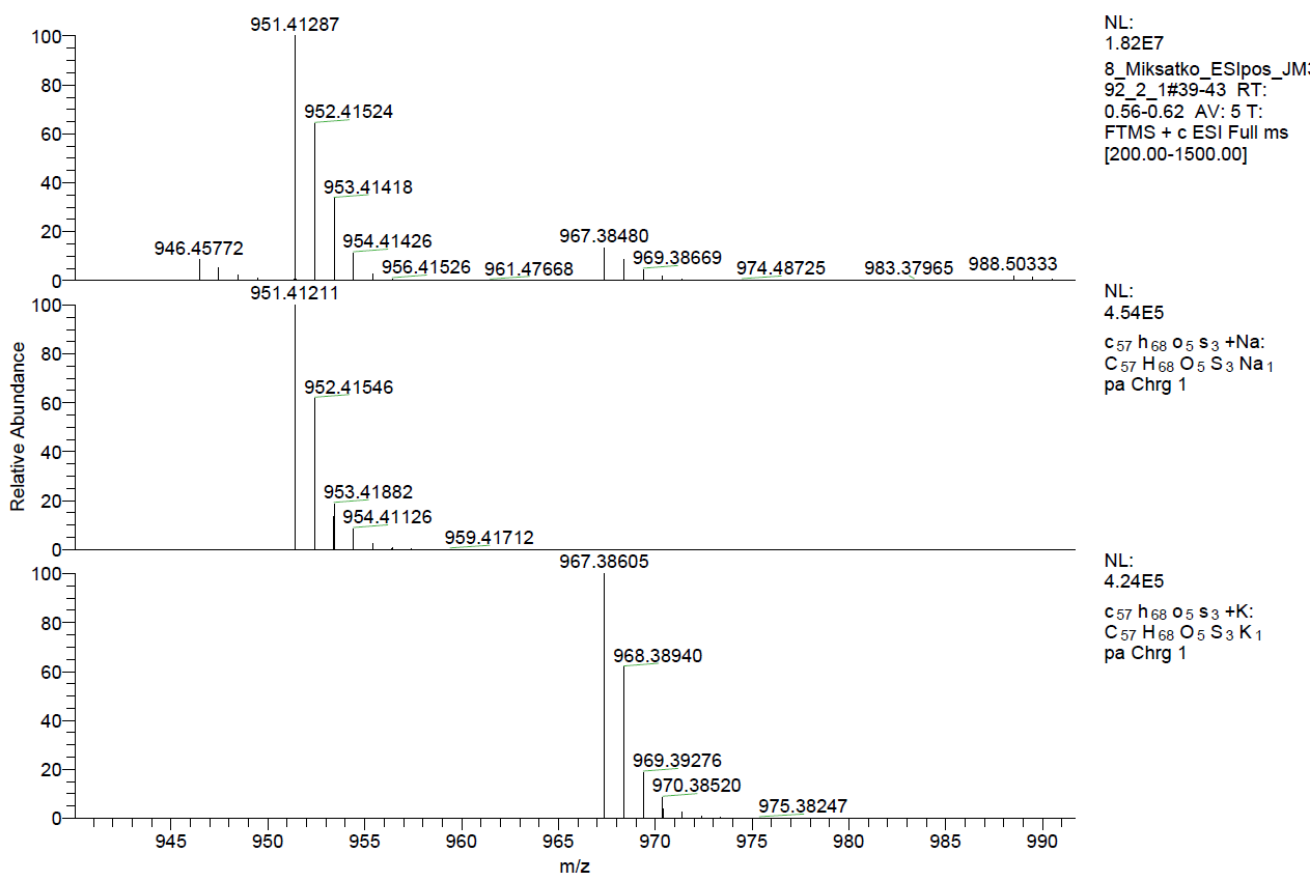


¹³C NMR (APT) spectrum of compound **21** (CDCl₃, 298K, 100 MHz)

Compound 21

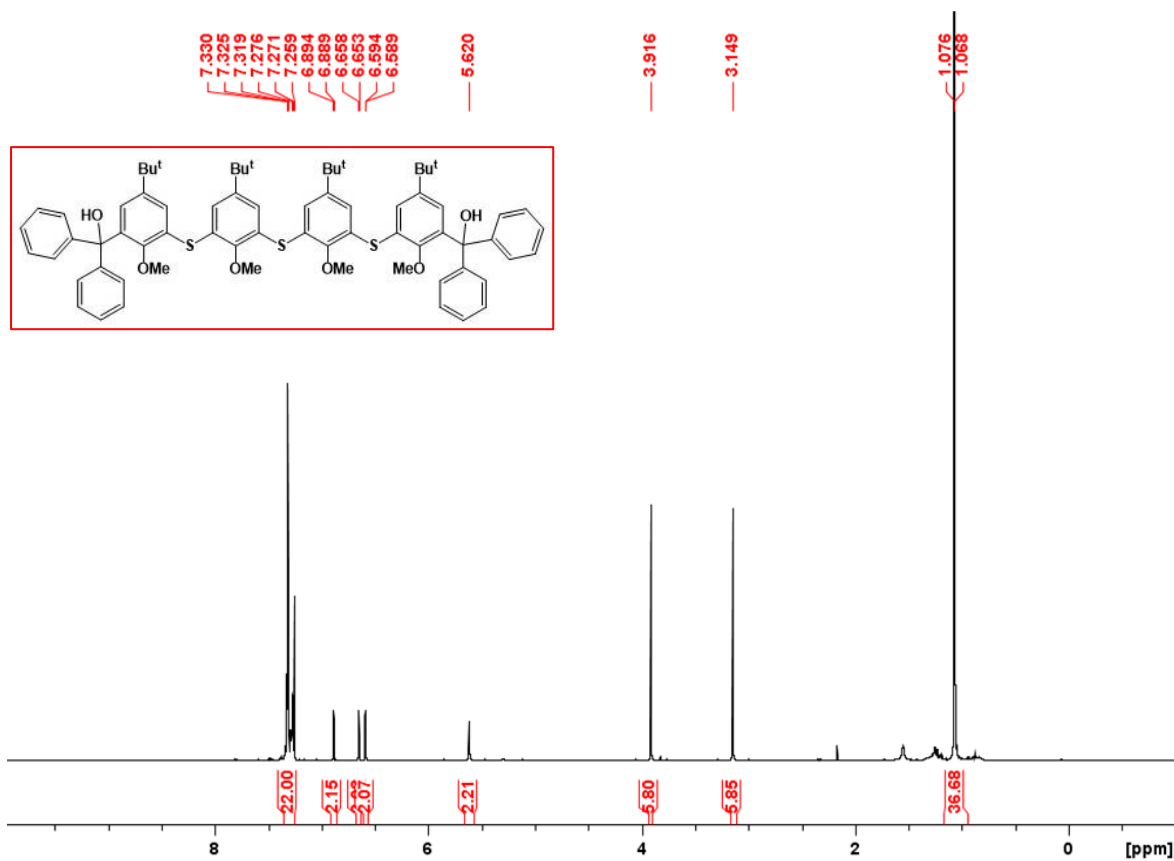


IR (ATR) spectrum of compound 21

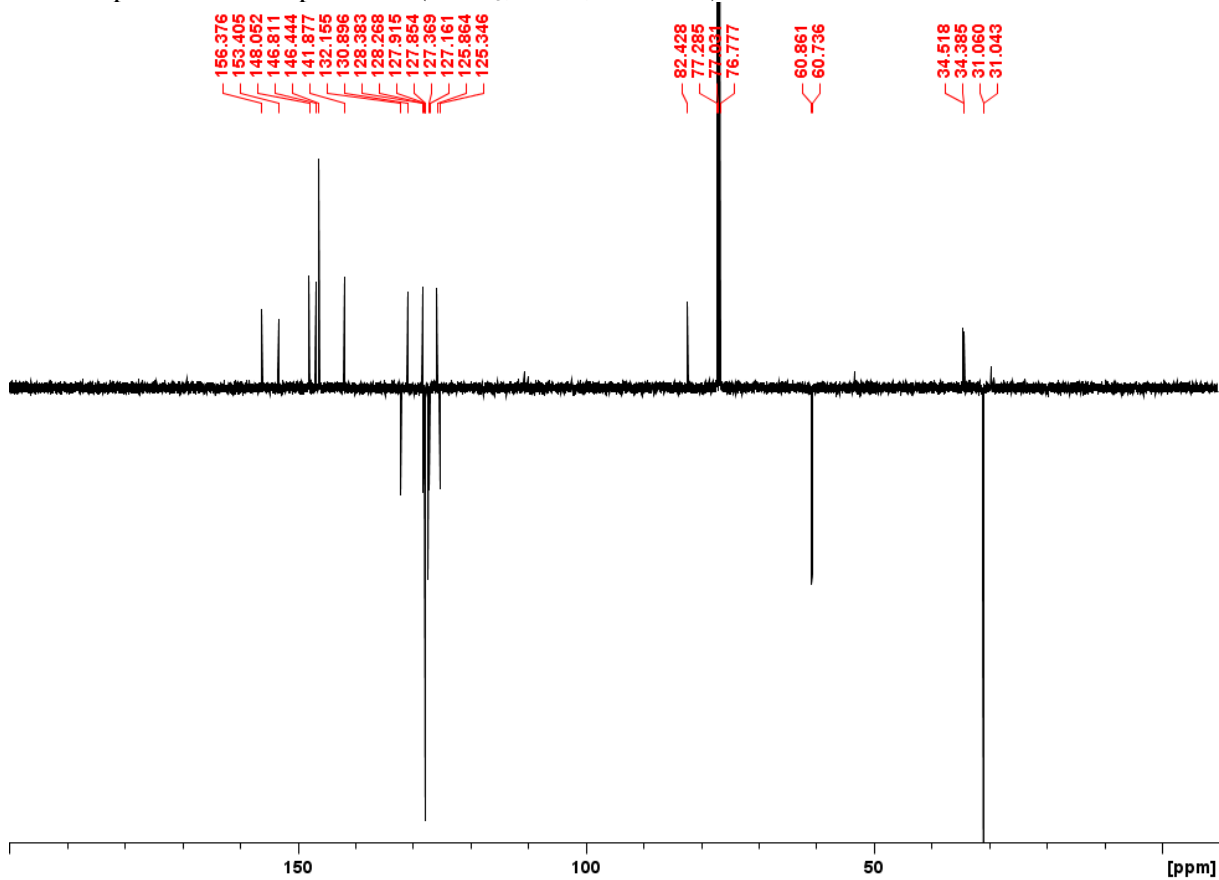


HRMS (ESI⁺) spectrum of compound 21

Compound 22

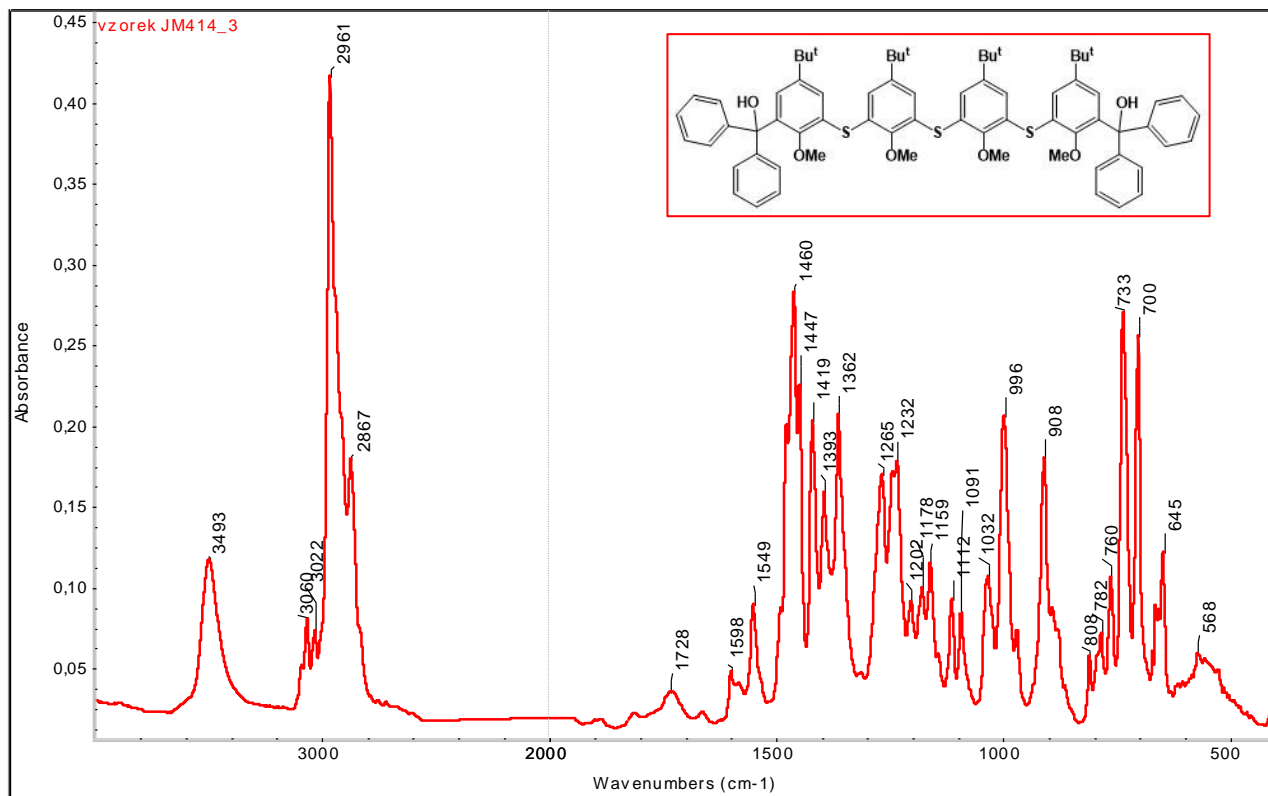


¹H NMR spectrum of compound 22 (CDCl₃, 298K, 400 MHz)

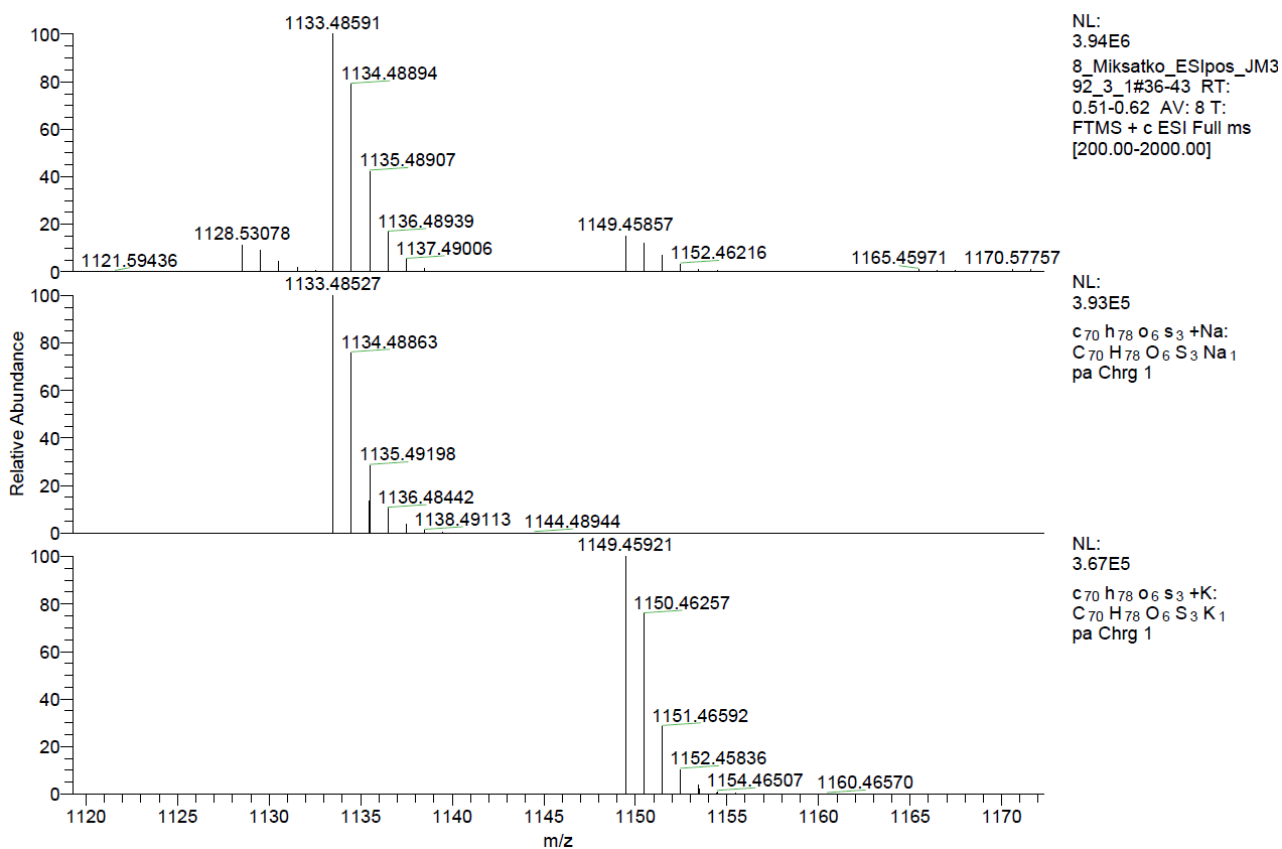


¹³C NMR (APT) spectrum of compound 22 (CDCl₃, 298K, 100 MHz)

Compound 22

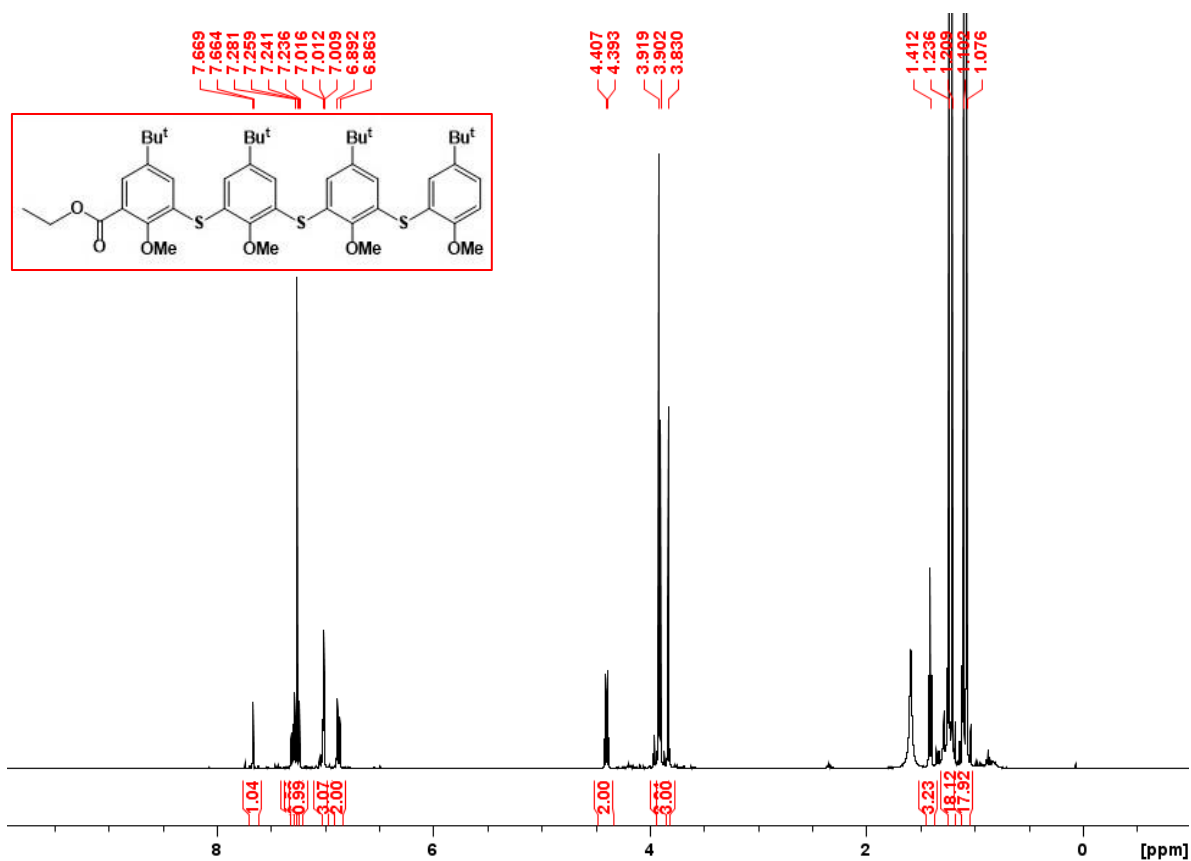


IR (ATR) spectrum of compound 22

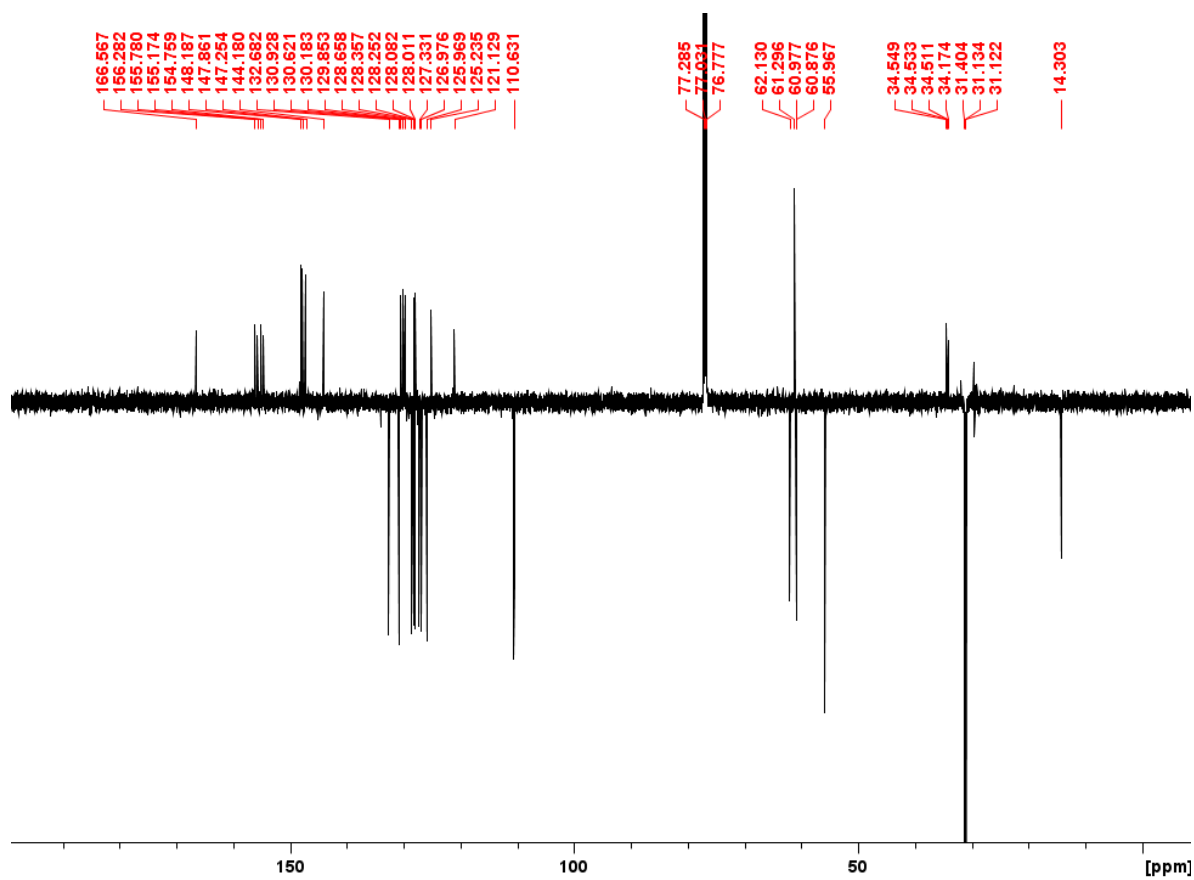


HRMS (ESI⁺) spectrum of compound 22

Compound **23**

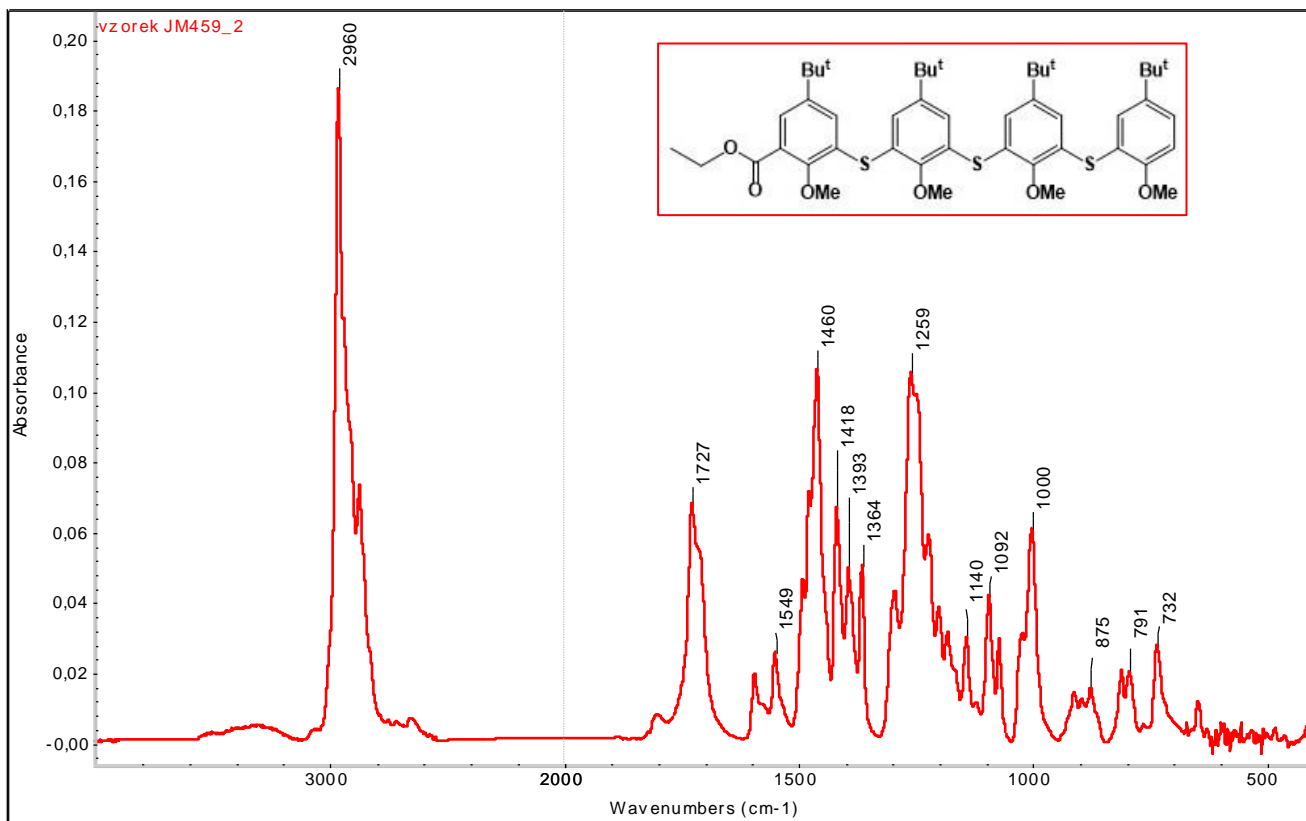


¹H NMR spectrum of compound **23** (CDCl₃, 298K, 400 MHz)

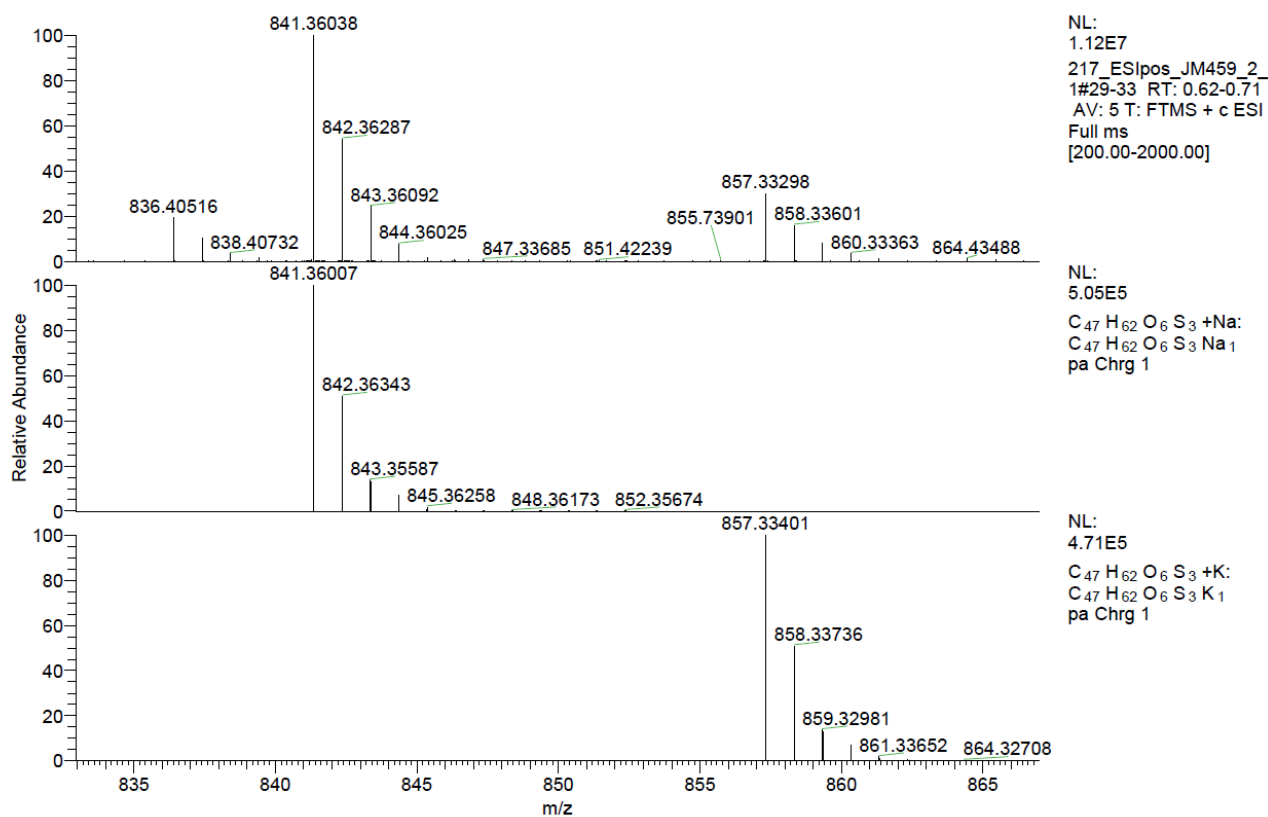


¹³C NMR (APT) spectrum of compound **23** (CDCl₃, 298K, 100 MHz)

Compound 23

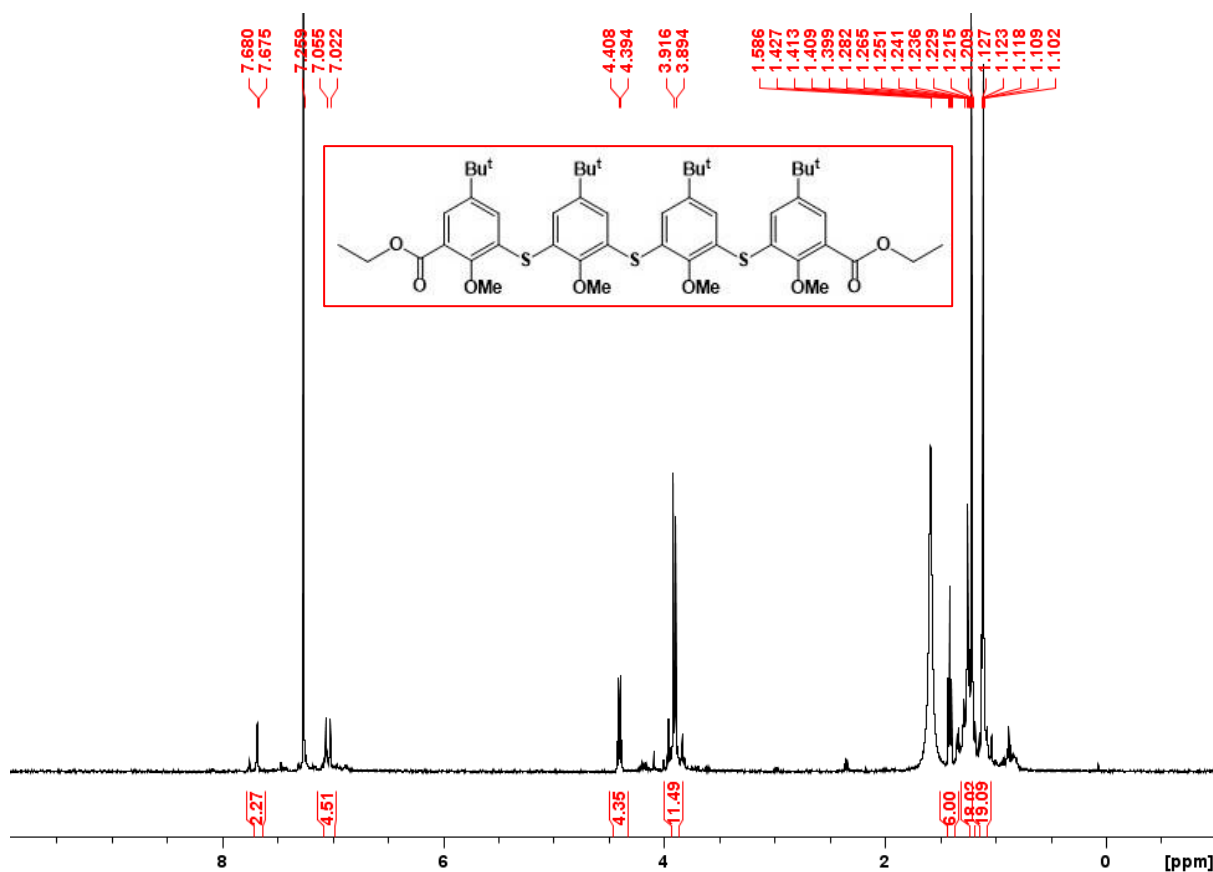


IR (ATR) spectrum of compound 23

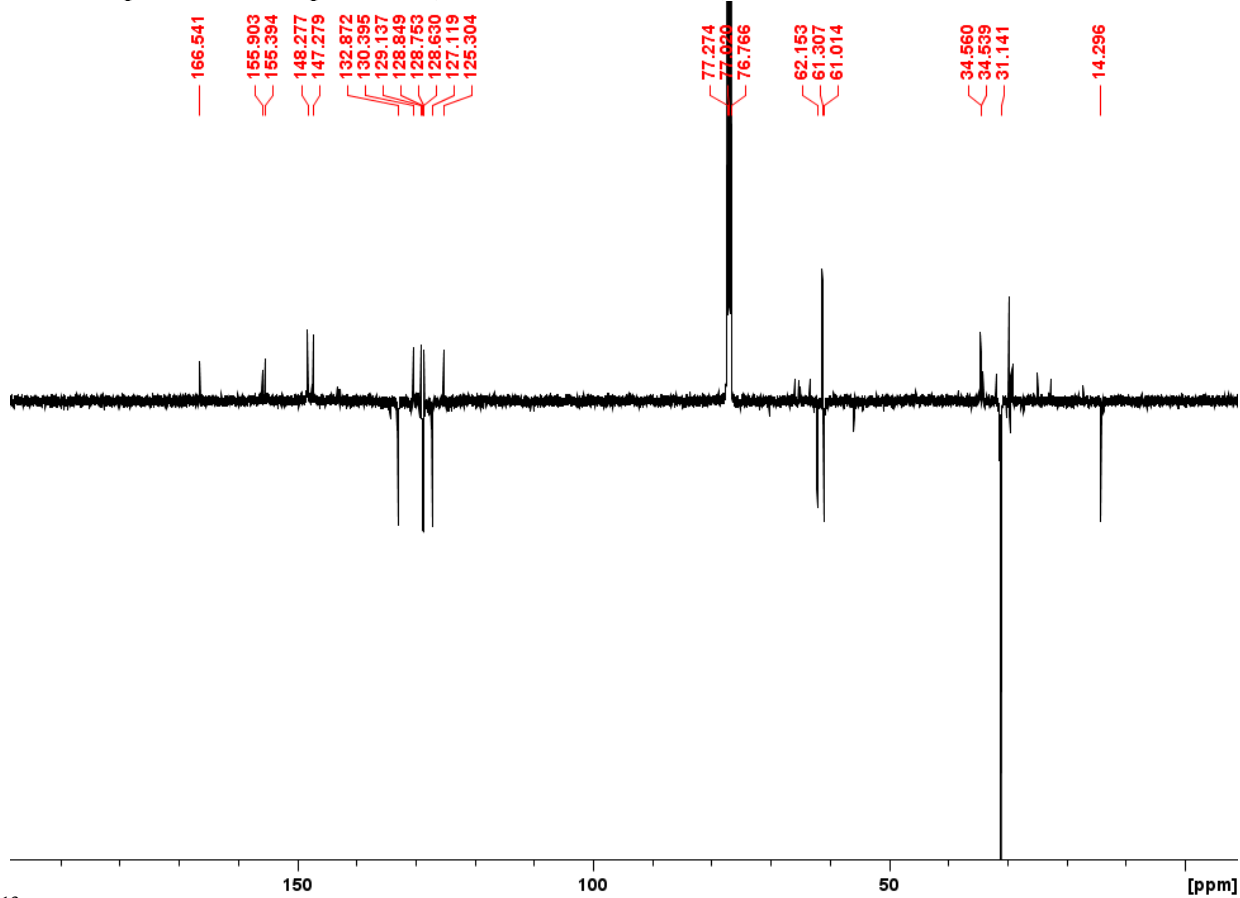


HRMS (ESI⁺) spectrum of compound 23

Compound 24

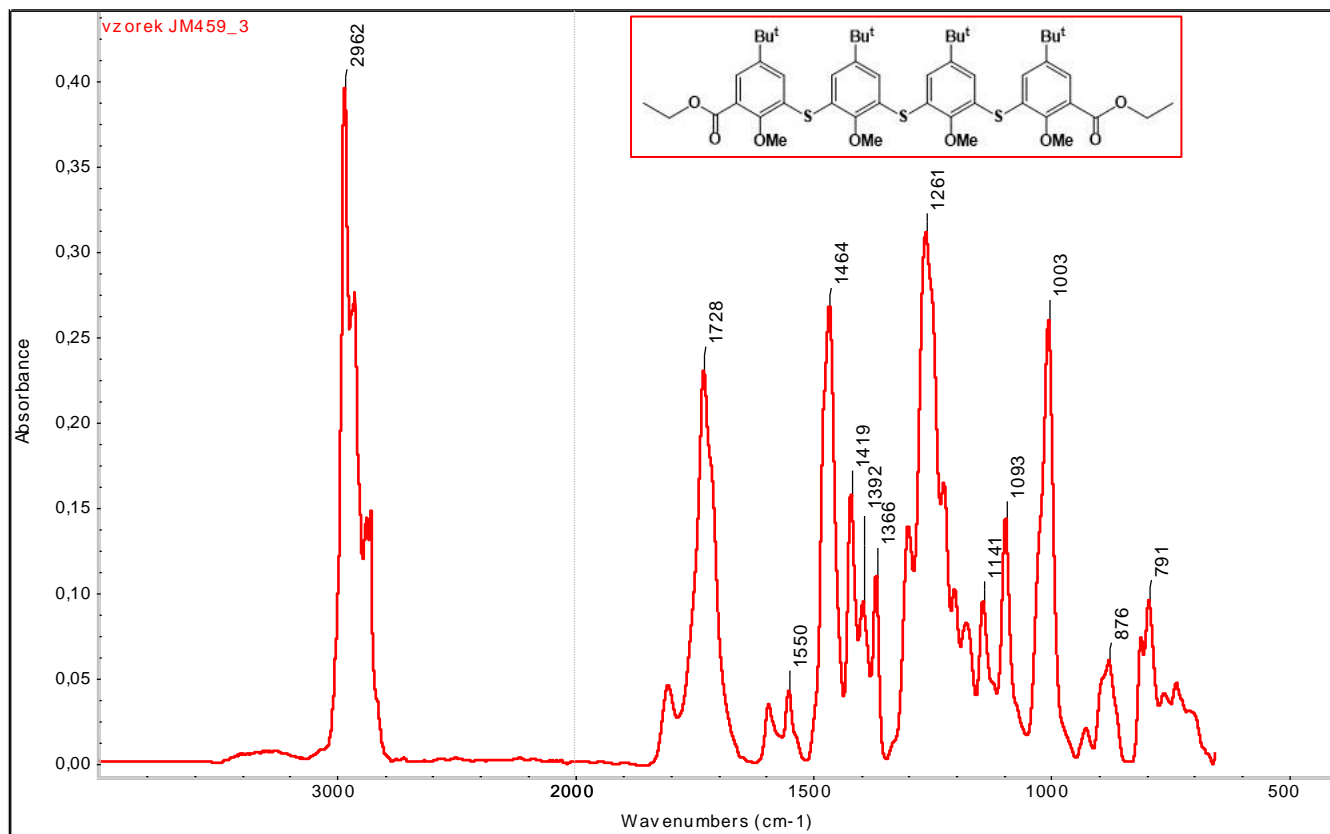


¹H NMR spectrum of compound **24** (CDCl₃, 298K, 400 MHz)

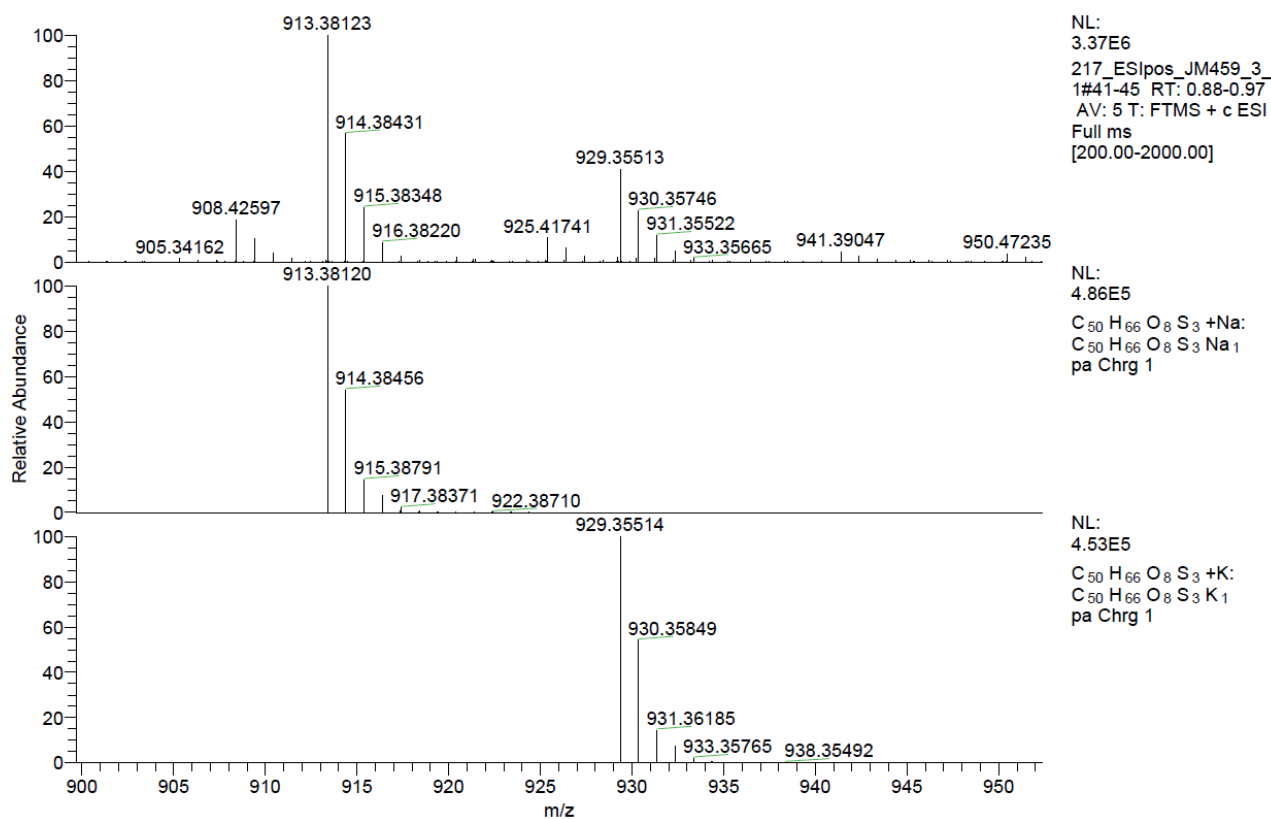


¹³C NMR (APT) spectrum of compound **24** (CDCl₃, 298K, 100 MHz)

Compound 24

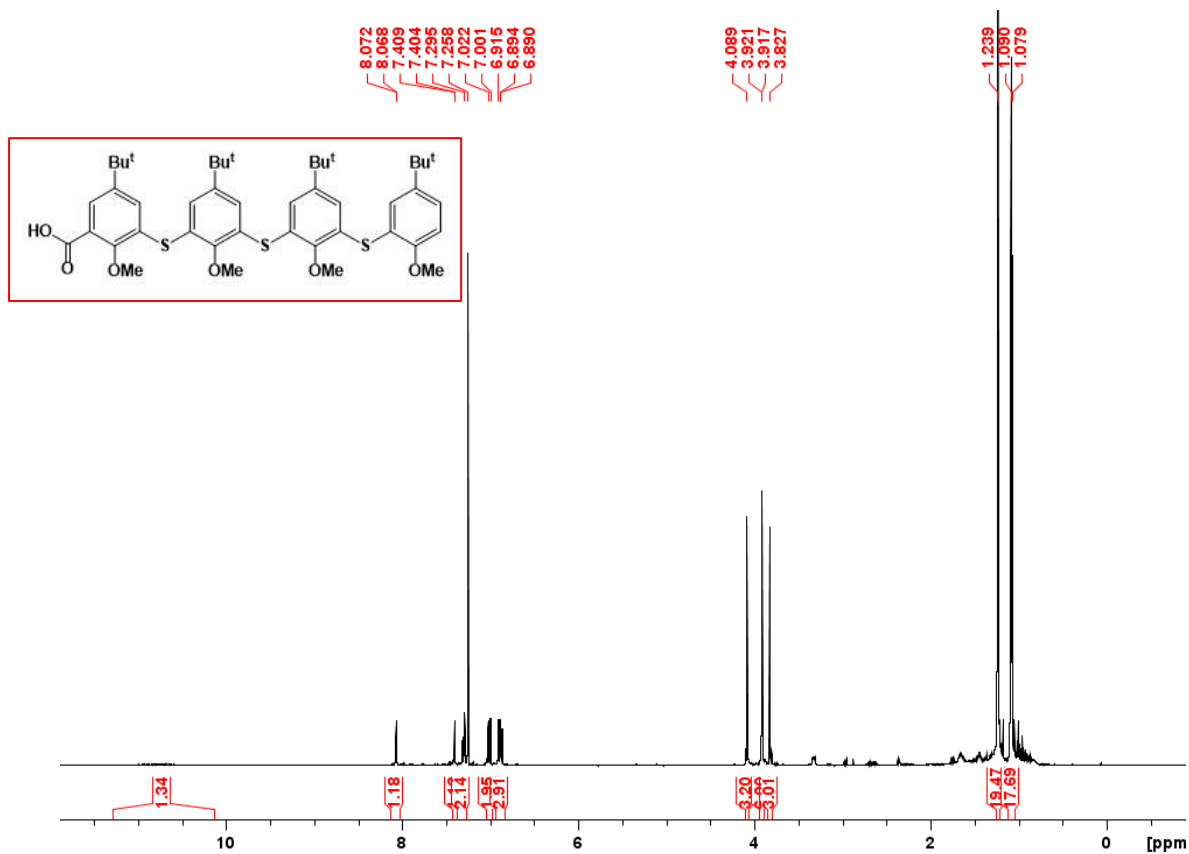


IR (ATR) spectrum of compound 24

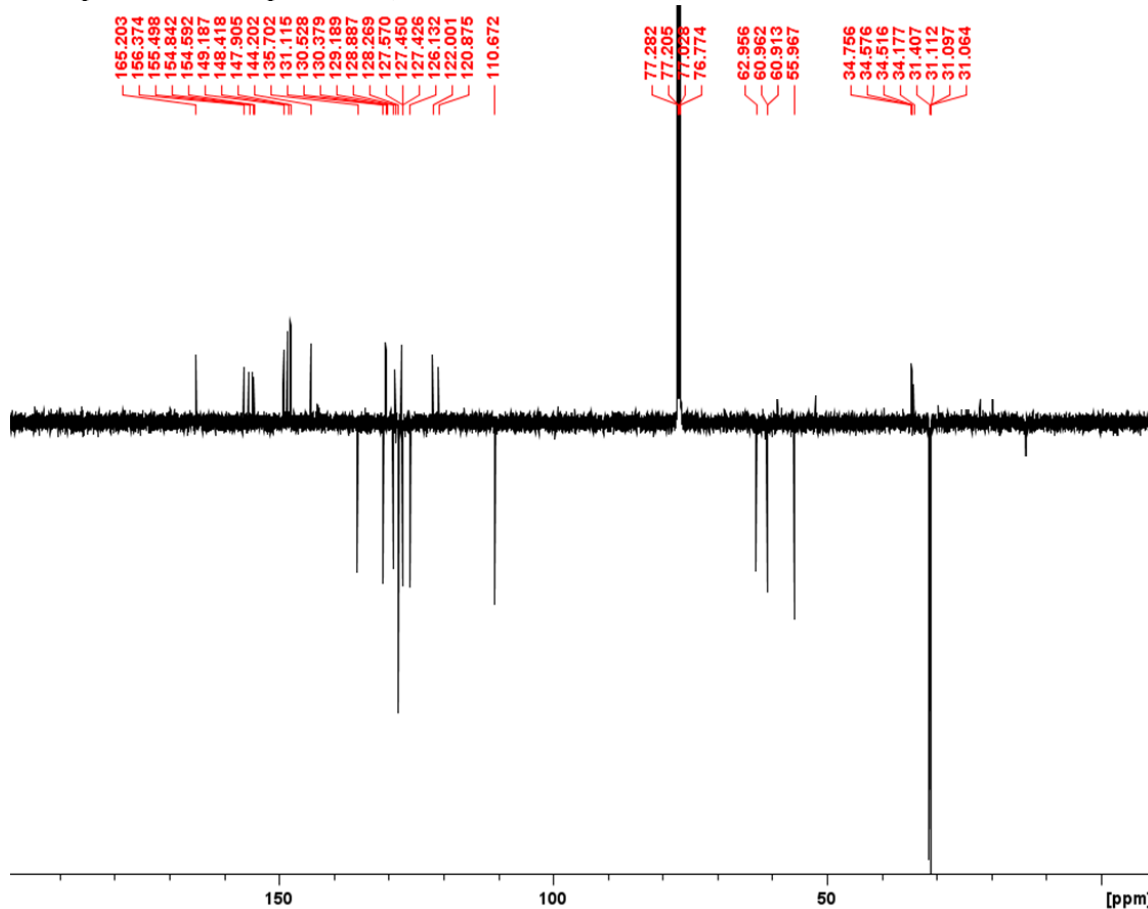


HRMS (ESI⁺) spectrum of compound 24

Compound 25

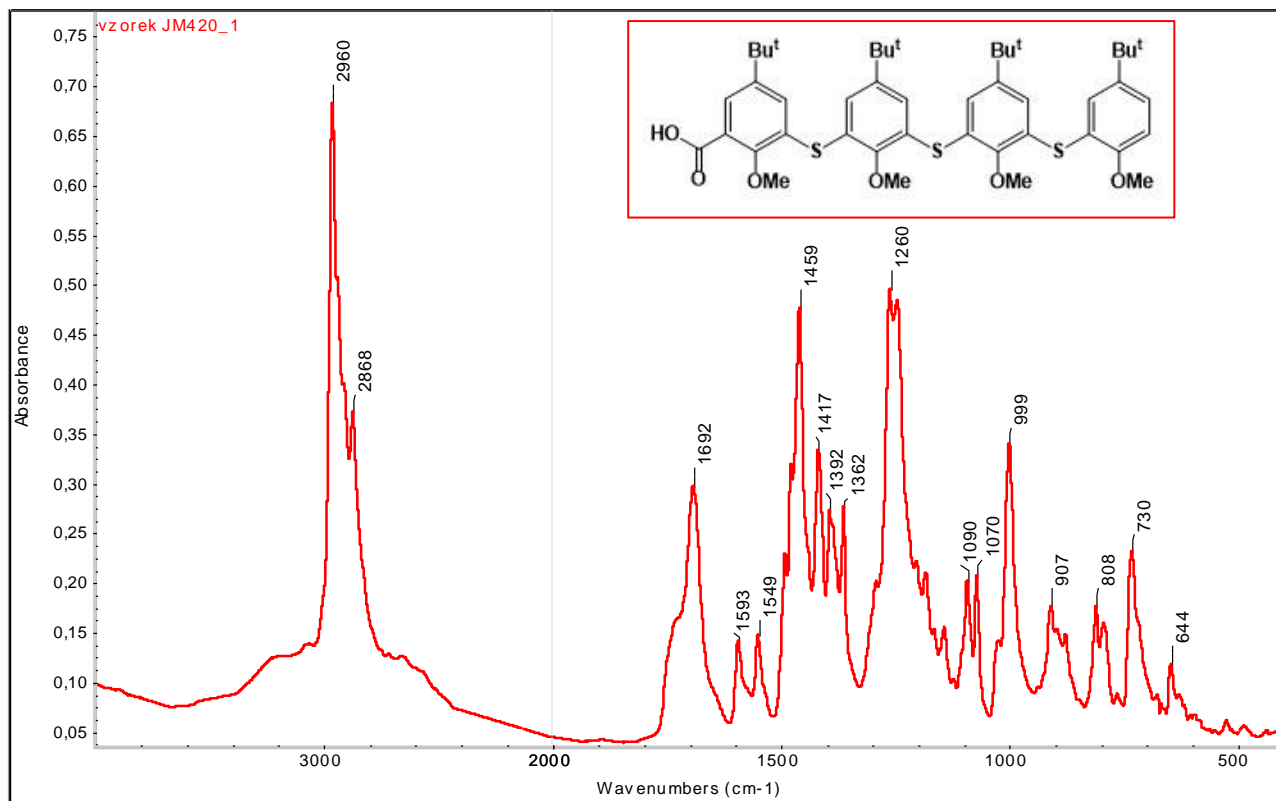


¹H NMR spectrum of compound 25 (CDCl₃, 298K, 400 MHz)

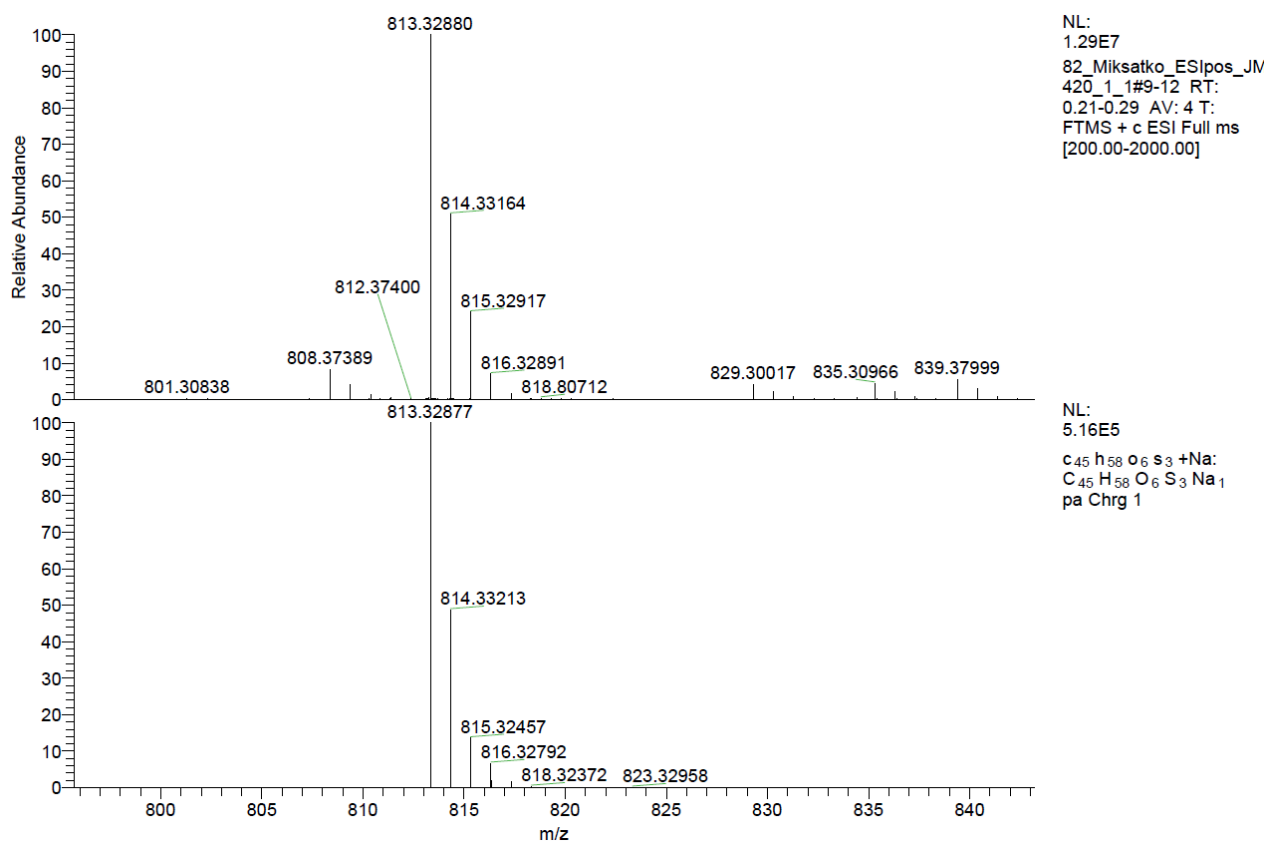


¹³C NMR (APT) spectrum of compound 25 (CDCl₃, 298K, 100 MHz)

Compound 25

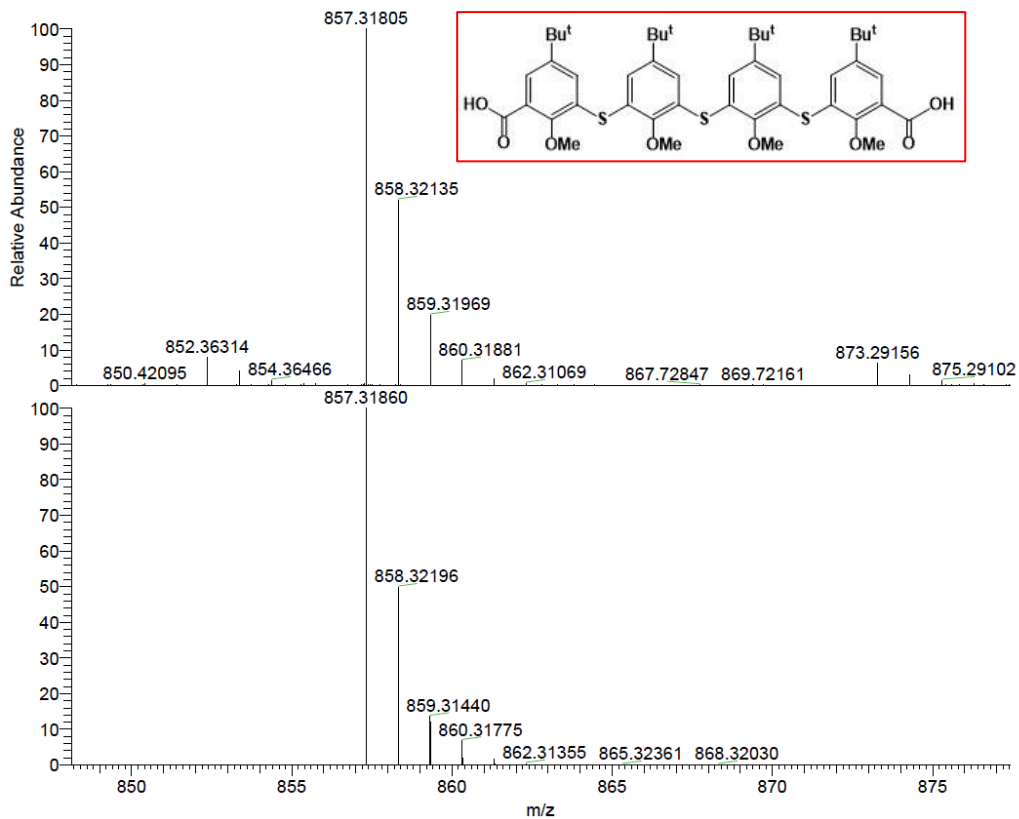


IR (ATR) spectrum of compound 25



HRMS (ESI⁺) spectrum of compound 25

Compound 26



NL:
3.08E6
82_Miksatko_ESIpos_JM
420_2_1#10-12 RT:
0.21-0.27 AV: 3 T: FTMS
+ c ESI Full ms
[200.00-2000.00]

NL:
5.08E5
C₄₆H₅₈S₃O₈+Na:
C₄₆H₅₈S₃O₈Na₁
pa Chrg 1

HRMS (ESI⁺) spectrum of compound 26

X-ray structure

Finally, the structure of the corresponding linear oligophenol ether derivative **6** was determined unambiguously using X-ray crystallography. This revealed that a monocrystal of **6** belonged to the monoclinic system, space group C_c . As shown in Fig. 1, the corresponding phenolic moieties are arranged in such a way that every succeeding unit is almost perpendicular to the previous one. Thus, according to Fig 1a, the corresponding interplanar angles ϕ between the succeeding aromatic subunits were 81.49° , 70.95° , and 71.83° (from the left to right).

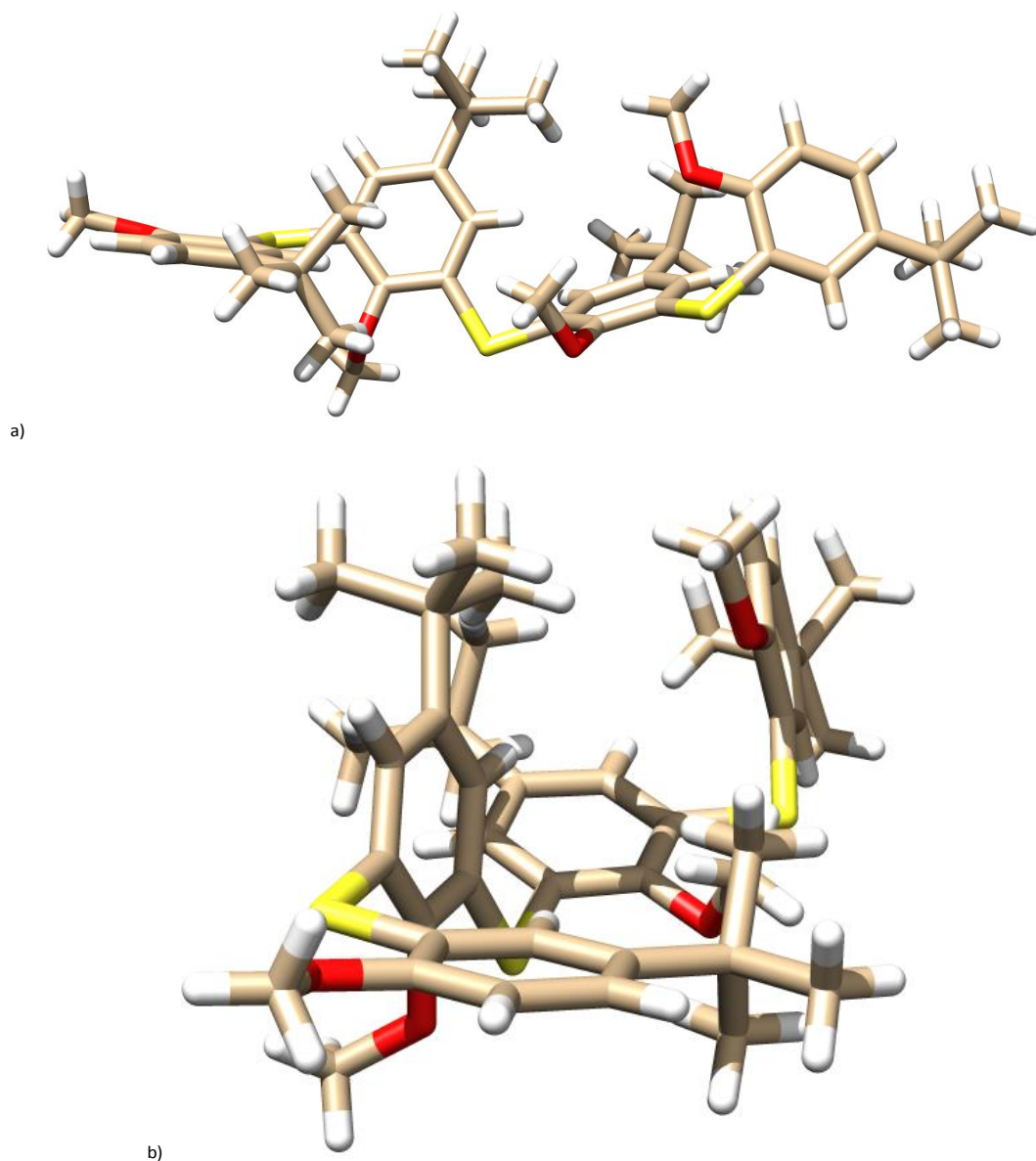


Figure: Single crystal X-ray structure of oligophenol **6**: (a) side-view, (b) front-view..
