Supporting Information

Solvent-free synthesis of pillar[6]arenes

S. Santra,[a] D. S. Kopchuk,[a,b] I. S. Kovalev,[a] G. V. Zyryanov,*[a,b] A. Majee,[c] V. N. Charushin[a,b] and O. N. Chupakhin[a,b]

[a] Ural Federal University, Chemical Engineering Institute, Yekaterinburg, 620002, Russian Federation.

[b] I. Ya. Postovskiy Institute of Organic Synthesis, Ural Division of the Russian Academy of Sciences, 22 S. Kovalevskoy Str., Yekaterinburg, 620219, Russian Federation.

[c] Department of Chemistry, Visva-Bharati (A Central University), Santiniketan-731235, India.

Table of Contents

General experimental methods: ^{1}H NMR spectra were determined on a Bruker 400 (400 MHz) spectrometer as solutions in $CDCl_3$. Chemical shifts are expressed in parts per million (δ) and are referenced to tetramethylsilane (TMS) as internal standard and the signals were reported as s (singlet), d (doublet), t (triplet), m (multiplet) and coupling constants *J* were given in Hz. ^{13}C NMR spectra were recorded at 100 MHz in $CDCl_3$ solution. ESI-HRMS data were collected at Brucker MicrOTOF-Q II Instrument. MALDI-TOF data were obtained on Bruker Ultraflex III MALDI-TOF instrument using 4-nitroaniline or matrixes Melting points were determined using the Boetius Block apparatus. A PM 100, Retsch GmbH Germany, apparatus was used for ball-milling. All organic solvents were purified and dried in accordance with standard procedures before use. Additional control of the purity of compounds and monitoring of the reaction were carried out by thin-layer chromatography using Silica G, 200 μm plates, UV 254. Commercially available substrates were freshly distilled before the reaction. Solvents, reagents and chemicals were purchased from Aldrich, Fluka, Merck, SRL, Spectrochem and Process Chemicals and used as received without additional purification.

General procedure for the synthesis of 1,4-dialkoxybenzenes:

1,4-Dialkoxybenzenes were synthesized by the reported method.[1] The adequate amounts of hydroquinone, TBAB and KOH were mixed at room temperature. The appropriate alkyl halide was then added and the reaction mixture was heated to 60-80 °C in an oil bath for the required time. The crude mixture was extracted with diethyl ether (50 mL). Filtration and evaporation of the solvent afforded the pure 1,4-dialkoxybenzene.

For 1,4-diethoxybenzene: 3 Equiv. of ethyl iodide, 9 mol% of TBAB and 2.5 equiv. of KOH were used with respect to hydroquinone. The reaction mixture was heated at 60 °C for 24 h.

For 1,4-dipropoxybenzene: 3 Equiv. of *n*-propyl bromide, 9 mol% of TBAB and 2.5 equiv. of KOH were used with respect to hydroquinone. The reaction mixture was heated at 70 °C for 5 h.

For 1,4-dibutoxybenzene: 2.5 Equiv. of *n*-butyl bromide, 9 mol% of TBAB and 2.5 equiv. of KOH were used with respect to hydroquinone. The reaction mixture was heated at 80 °C for 4 h.

For 1,4-di-(2-ethylhexyloxy)benzene: 2.5 Equiv. of 2-ethylhexyl bromide, 9 mol% of TBAB and 2.5 equiv. of KOH were used with respect to hydroquinone. The reaction mixture was heated at 80 °C for 4 h.

For 1,4-diheptyloxy benzene: 2.5 Equiv. of heptyl iodide, 9 mol% of TBAB and 2.5 equiv. of KOH were used with respect to hydroquinone. The reaction mixture was heated at 80 °C for 4 h.

General procedure for the synthesis of per-alkylated pillar[6]arenes 2a-2e:

A mixture of the appropriate 1,4-dialkoxy benzene (5 mmol), paraformaldehyde (0.46 g, 15 mmol) and 98% H_2SO_4 (30 µL, 10 mol%) was ground in a mortar and pestle at room temperature. Continuous grinding for 10 minutes by hand, a deep green gummy mass was observed as a crude product. 10 mL of ethyl alcohol and 0.5 mL of water were poured into the vessel. The resulting precipitate was filtered off and washed with ethyl alcohol by several times to get the pure per-alkylated pillar[6]arenes without any purification by column chromatography or recrystallization.

Typical procedure for the synthesis of 1,4-bis(ethoxy)pillar[6]arene (2a):[2] A mixture of the 1,4-diethoxy benzene (0.83 g, 5 mmol), paraformaldehyde (0.46 g, 15 mmol) and 98% H_2SO_4 (30 µL, 10 mol%) was ground in a mortar and pestle at room temperature. Continuous grinding for 10 minutes by hand, a deep green gummy mass was observed as a crude product. 10 mL of ethyl alcohol and 0.5 mL of water were poured into the vessel. The resulting precipitate was filtered off and washed with ethyl alcohol by several times to get the pure 1,4-bis(ethoxy)pillar[6]arene (**2a**) as white solid. Yield 84%, mp. 170-172 ºC (Lit. 172-173 ºC); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 6.66 (s, 12H, phenyl protons), 3.91-3.84 (m, 36H, methylene protons and methylene bridges), 1.30 (t, J = 6.6 Hz, 36H, methyl protons); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 150.65, 128.11, 114.99 (C of phenyl), 64.36 (C of oxymethylene groups), 29.70 (C of methylene bridge), 15.13 (C of methyl groups). Calcd. For $C_{66}H_{84}O_{12}$: m/z = 1068.60; Found: m/z = 1086.63 $[M+NH_4]^+$. Anal. Calcd for $C_{66}H_{84}O_{12}$: C, 74.13; H, 7.92. Found: C, 73.97; H, 8.05. HPLC Retention time = 22.7 min.

Typical procedure for the synthesis of 1,4-bis(ethoxy)pillar[6]arene (2a) on a gram-scale: A mixture of the 1,4-diethoxy benzene (1.66 g, 10 mmol), paraformaldehyde (0.93 g, 30 mmol) and 98% H_2SO_4 (60 µL, 10 mol%) was ground in a mortar and pestle at room temperature. Continuous grinding for 10 minutes by hand, a deep green gummy mass was observed as a crude product. 20 mL of ethyl alcohol and 1 mL of water were poured into the vessel. The resulting precipitate was filtered off and washed with ethyl alcohol by several times to get the pure 1,4-bis(ethoxy)pillar[6]arene (**2a**) as white solid. Yield 84%.

Typical procedure for the synthesis of 1,4-bis(ethoxy)pillar[6]arene (2a) under ball-milling:

A mixture of the 1,4-diethoxy benzene (1 g, 6 mmol), paraformaldehyde (0.56 g, 18 mmol) and 98% H_2SO_4 (36 µL, 10 mol%) was subjected to ball-milling at 600 rpm using a 25 mL stainless steel beaker with six balls (d = 10 mm) of the same material for 10 min. 20 mL of ethyl alcohol

and 1 mL of water were poured into the crude reaction mixture. The resulting precipitate was filtered off and washed with ethyl alcohol by several times to get the pure 1,4-bis(ethoxy)pillar[6]arene (**2a**) as white solid. Yield 85%.

Typical procedure for the synthesis of 1,4-bis(propoxy)pillar[6]arene (2b):[2] A mixture of 1,4-dipropoxy benzene (0.97 g, 5 mmol), paraformaldehyde (0.46 g, 15 mmol) and 98% H_2SO_4 (30 µL, 10 mol%) was ground in a mortar and pestle at room temperature. Continuous grinding for 10 minutes by hand, a deep greenish brown gummy mass was observed as a crude product. 10 mL of ethyl alcohol and 0.5 mL of water were poured into the vessel. The resulting precipitate was filtered off and washed with ethyl alcohol by several times to get the pure 1,4-bis(propoxy)pillar[6]arene (**2b**) as white solid. Yield 82%, mp. 121-123 ºC (Lit. 119-121 ºC); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 6.66 (s, 12H, phenyl protons), 3.89 (s, 12H, methylene bridges), 3.76 (t, J = 6.4 Hz, 24H, –OCH_2CH$_2$CH$_3$), 1.74-1-68 (m, 24H, –OCH$_2$CH_2CH$_3$), 0.96 (t, J = 7.36 Hz, 36H, methyl protons); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 150.71, 128.07, 114.80 (C of phenyl), 70.42 (C of oxymethylene groups), 30.90 (C of methylene bridge), 22.89 (C of methylene groups), 10.64 (C of methyl groups). ESI-HRMS: Calcd. for $C_{78}H_{108}O_{12}$ m/z = 1236.78, found 1254.82 $[M+NH_4]^+$. Anal. Calcd for $C_{78}H_{108}O_{12}$: C, 75.69; H, 8.80; Found: C, 75.77; H, 8.68.

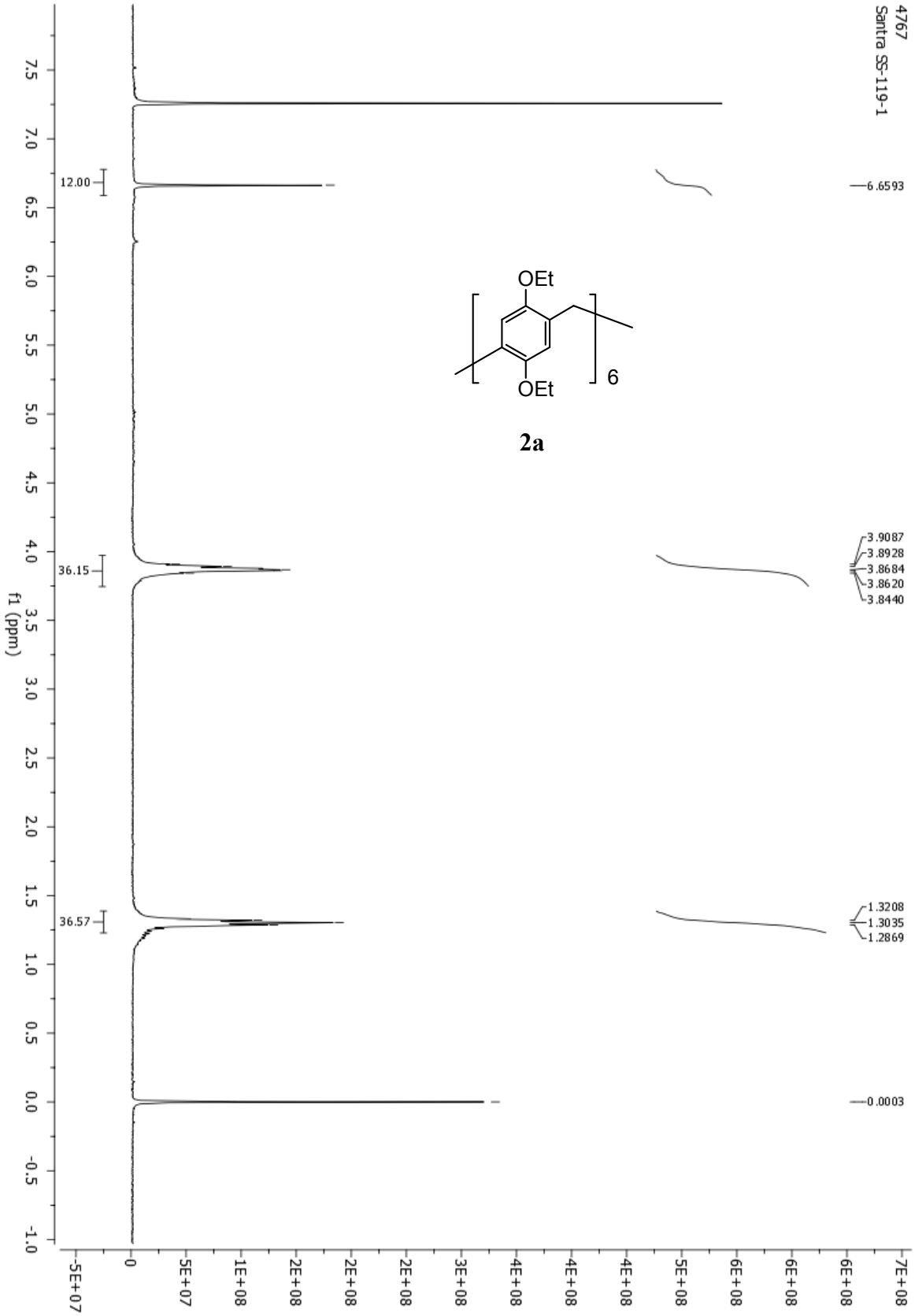
Typical procedure for the synthesis of 1,4-bis(butoxy)pillar[6]arene (2c):[2] A mixture of 1,4-dibutoxy benzene (1.11 g, 5 mmol), paraformaldehyde (0.46 g, 15 mmol) and 98% H_2SO_4 (30 µL, 10 mol%) was ground in a mortar and pestle at room temperature. Continuous grinding for 10 minutes by hand, a deep brown gummy mass was observed as a crude product. 10 mL of ethyl alcohol and 0.5 mL of water were poured into the vessel. The resulting precipitate was filtered off and washed with ethyl alcohol by several times to get the pure 1,4-bis(butoxy)pillar[6]arene (**2c**) as white solid. Yield 83%, mp. 90-92 ºC (Lit. 89-91 ºC); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 6.67 (s, 12H, phenyl protons), 3.87 (s, 12H, methylene bridges), 3.81 (t, J = 8.96 Hz, 24H, –OCH_2CH$_2$–), 1.70-1.66 (m, 24H, –OCH$_2$CH_2–), 1.45-1.39 (m, 24H, –OCH$_2$CH$_2$CH_2–), 0.90 (t, J = 7.36 Hz, 36H, methyl protons); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 150.71, 128.08, 114.75 (C of phenyl), 68.56 (C of oxymethylene groups), 31.77 (C of methylene groups), 30.91 (C of methylene bridge), 19.37 (C of methylene groups), 13.89 (C of methyl groups). ESI-HRMS: Calcd. for $C_{90}H_{132}O_{12}$ m/z = 1404.97, found 1423.01 $[M+NH_4]^+$. Anal. Calcd for $C_{90}H_{132}O_{12}$: C, 76.88; H, 9.46; Found: C, 77.07; H, 9.22.

1,4-Bis(2-ethylhexyloxy)pillar[6]arene (2d): A mixture of 1,4-di-(2-ethylhexyloxy)benzene (1.67 g, 5 mmol), paraformaldehyde (0.46 g, 15 mmol) and 98% H_2SO_4 (30 µL, 10 mol%) was ground in a mortar and pestle at room temperature. Continuous grinding for 10 minutes by hand,

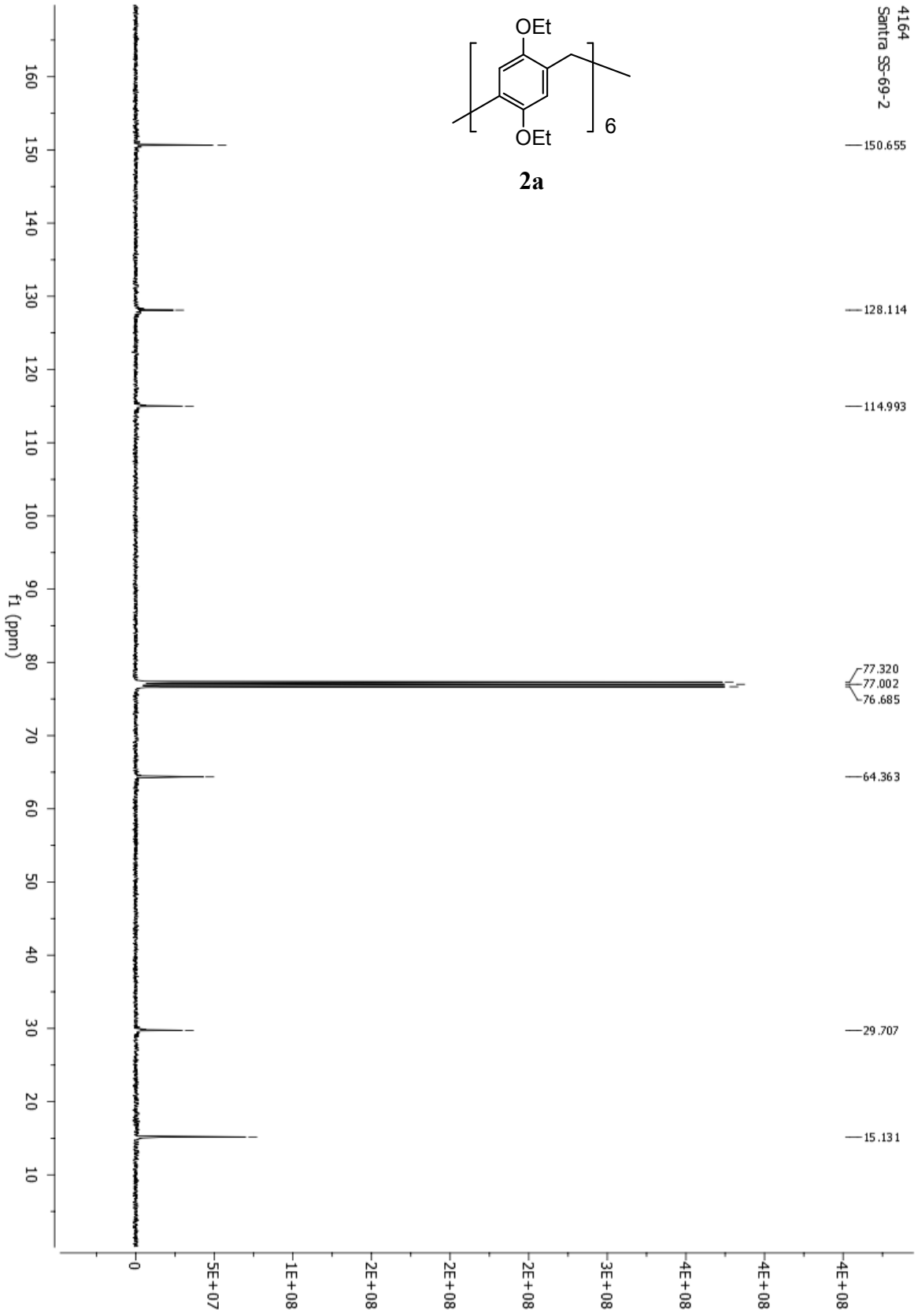
a deep brown gummy mass was observed as a crude product. 10 mL of ethyl alcohol and 0.5 mL of water were poured into the vessel. The resulting precipitate was filtered off and washed with ethyl alcohol by several times to get the pure 1,4-bis(2-ethylhexyloxy)pillar[6]arene (**2d**) as colorless sticky mass. Yield 80%; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 6.66 (s, 12H, phenyl protons), 3.90 (s, 12H, methylene bridges), 3.69 (d, *J* = 4.8 Hz, 24H, –OC*H*$_2$CH(CH_2CH_3)CH_2–), 1.62-1.17 (m, 108H, –OCH_2C*H*(C*H*$_2$$CH_3$)C*H*$_2$C*H*$_2$C*H*$_2$–), 0.89-0.80 (m, 72H, methyl protons); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 153.46, 129.89, 115.39 (C of phenyl), 71.25 (C of oxymethylene groups), 39.48, 31.89 (C of methylene groups), 30.54 (C of methylene bridges), 28.82, 23.86, 22.83 (C of methylene groups), 14.08, 10.87 (C of methyl groups). Anal. Calcd. For $C_{138}H_{228}O_{12}$: C, 79.71; H, 11.05%; Found: C, 79.65; H, 11.01%. MALDI-TOF Calcd. For $C_{138}H_{228}O_{12}$: m/z = 2078.54; Found: m/z = 2078.5 $[M]^+$, m/z = 2096.6 $[M+NH_4]^+$, m/z = 2117.5 $[M+K]^+$.

Typical procedure for the synthesis of 1,4-bis(heptyloxy)pillar[6]arene (2e): A mixture of 1,4-diheptyloxy benzene (1.53 g, 5 mmol), paraformaldehyde (0.46 g, 15 mmol) and 98% H_2SO_4 (30 µL, 10 mol%) was ground in a mortar and pestle at room temperature. Continuous grinding for 10 minutes by hand, a deep brown gummy mass was observed as a crude product. 10 mL of ethyl alcohol and 0.5 mL of water were poured into the vessel. The resulting precipitate was filtered off and washed with ethyl alcohol by several times to get the pure 1,4-bis(heptyloxy)pillar[6]arene (**2e**) as white solid. Yield 78%, mp. 105-107 ºC; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 6.69 (s, 12H, phenyl protons), 3.89 (s, 12H, methylene bridges), 3.82 (t, *J* = 6.4 Hz, 24H, –OC*H*$_2$$CH_2$–), 1.86-1.79 (m, 24H, –$OCH_2$C*H*$_2$$CH_2CH_2$–), 1.37-1.29 (m, 96H, –$OCH_2CH_2$C*H*$_2$C*H*$_2$C*H*$_2$C*H*$_2$–), 0.89 (t, *J* = 6.6 Hz, 36H, methyl protons); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 150.71, 128.04, 114.73 (C of phenyl), 68.88 (C of oxymethylene groups), 31.84, 29.74 (C of methylene groups), 29.22 (C of methylene bridges), 29.17, 26.21, 22.64 (C of methylene groups), 14.07 (C of methyl groups). Anal. Calcd. For $C_{126}H_{204}O_{12}$: C, 79.19; H, 10.76%; Found: C, 79.13; H, 10.70%. MALDI-TOF Calcd. For $C_{126}H_{204}O_{12}$: *m/z* = 1910.54; Found: *m/z* = 1911.9 $[M+H]^+$

1,4-Bis(ethoxy)pillar[6]arene (2a):



2a


OEt
OEt
6
4164
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150.655
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77.002
76.685
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15.131
f1 (ppm)
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
5E+07
1E+08
2E+08
2E+08
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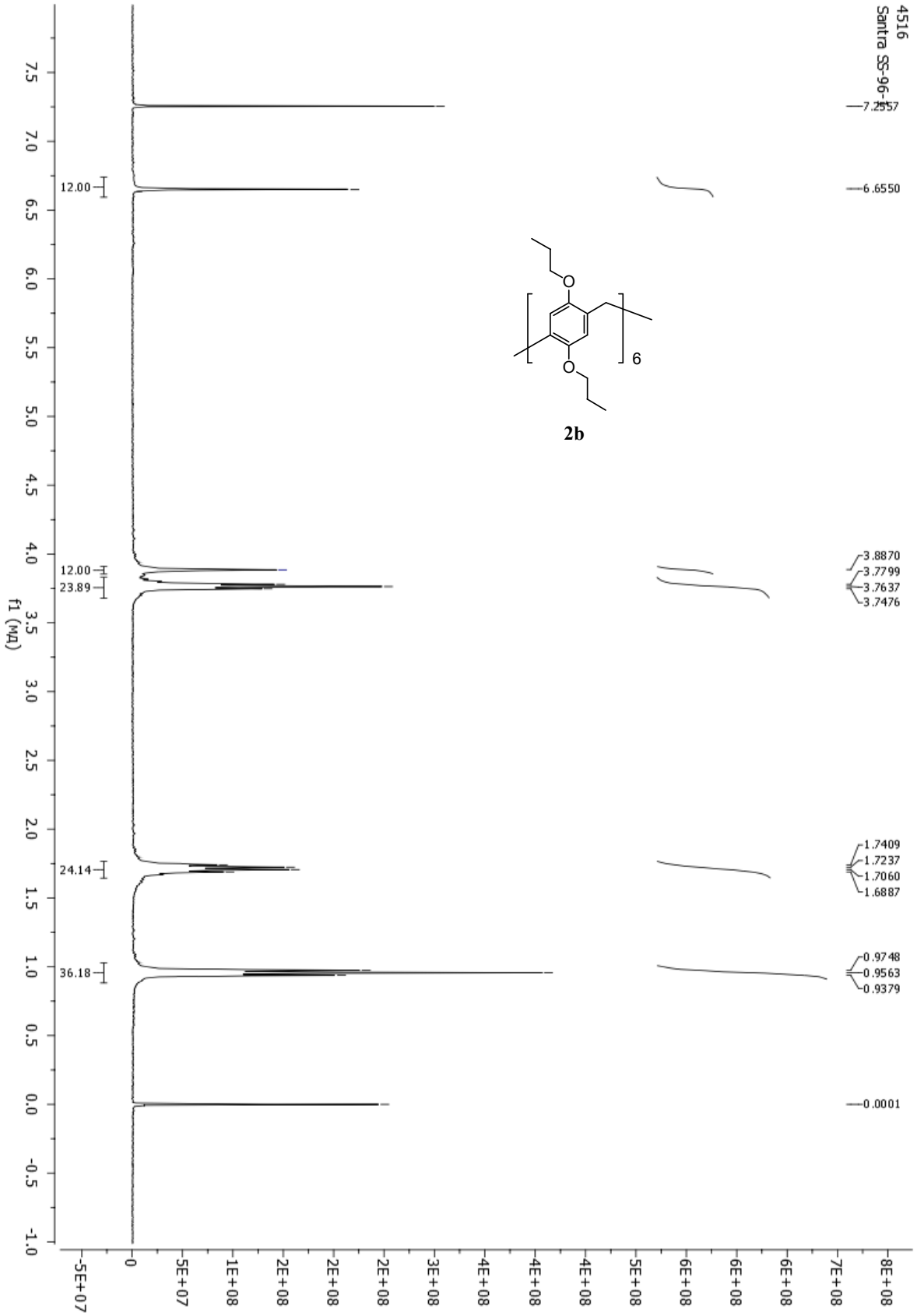
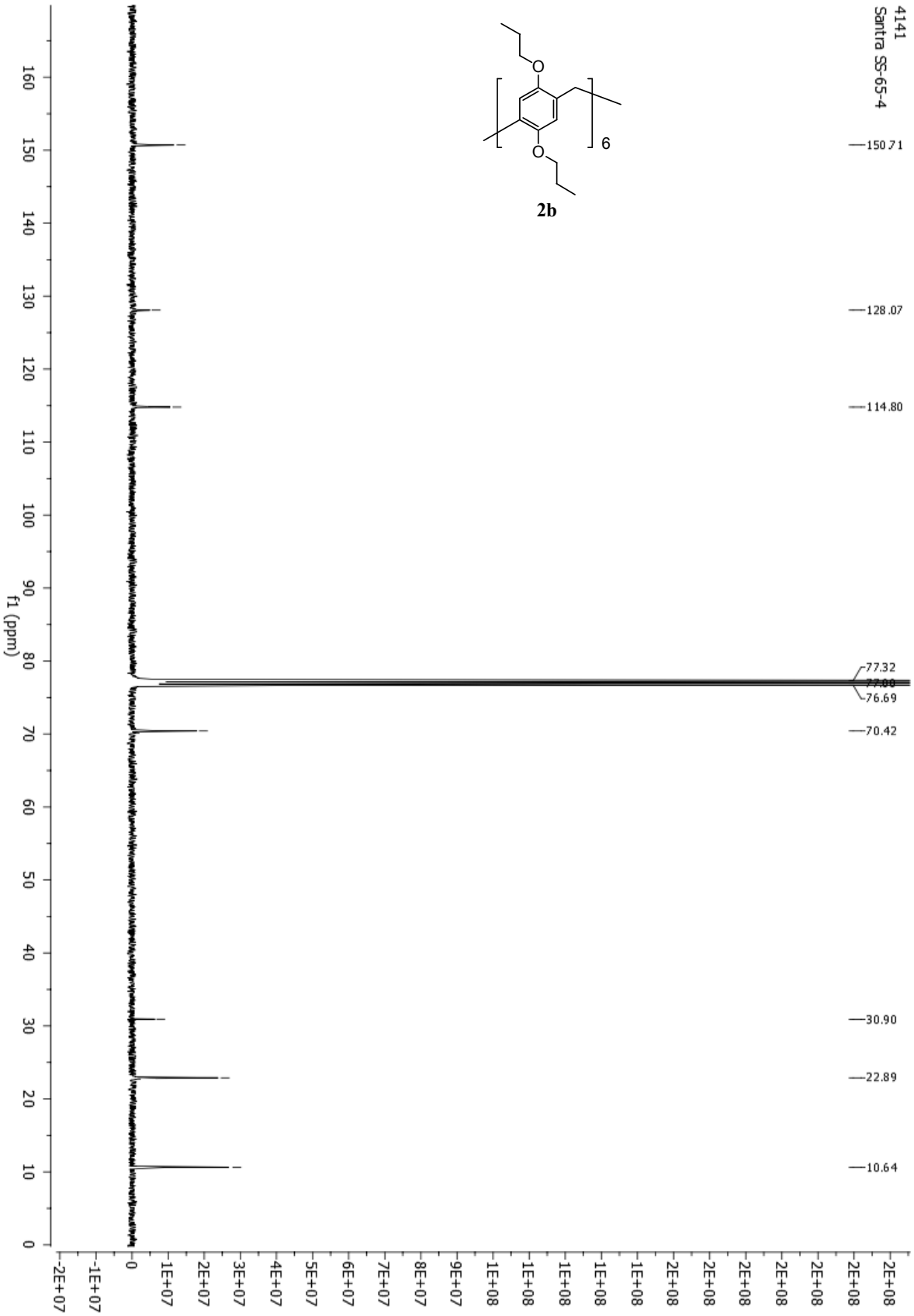
3E+08
4E+08
4E+08
4E+08

2a

1,4-Bis(propoxy)pillar[6]arene (2b):



2b


4141
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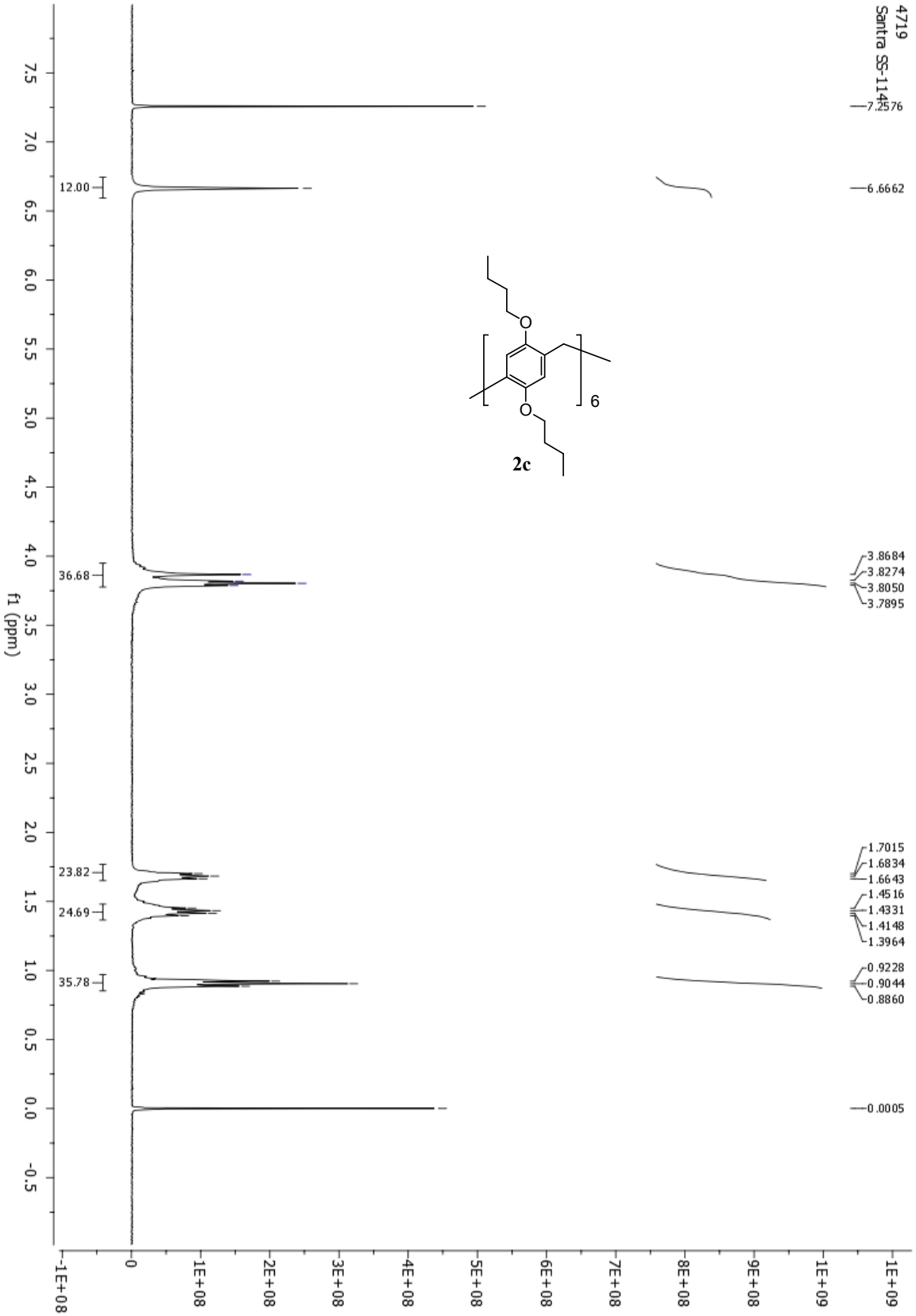
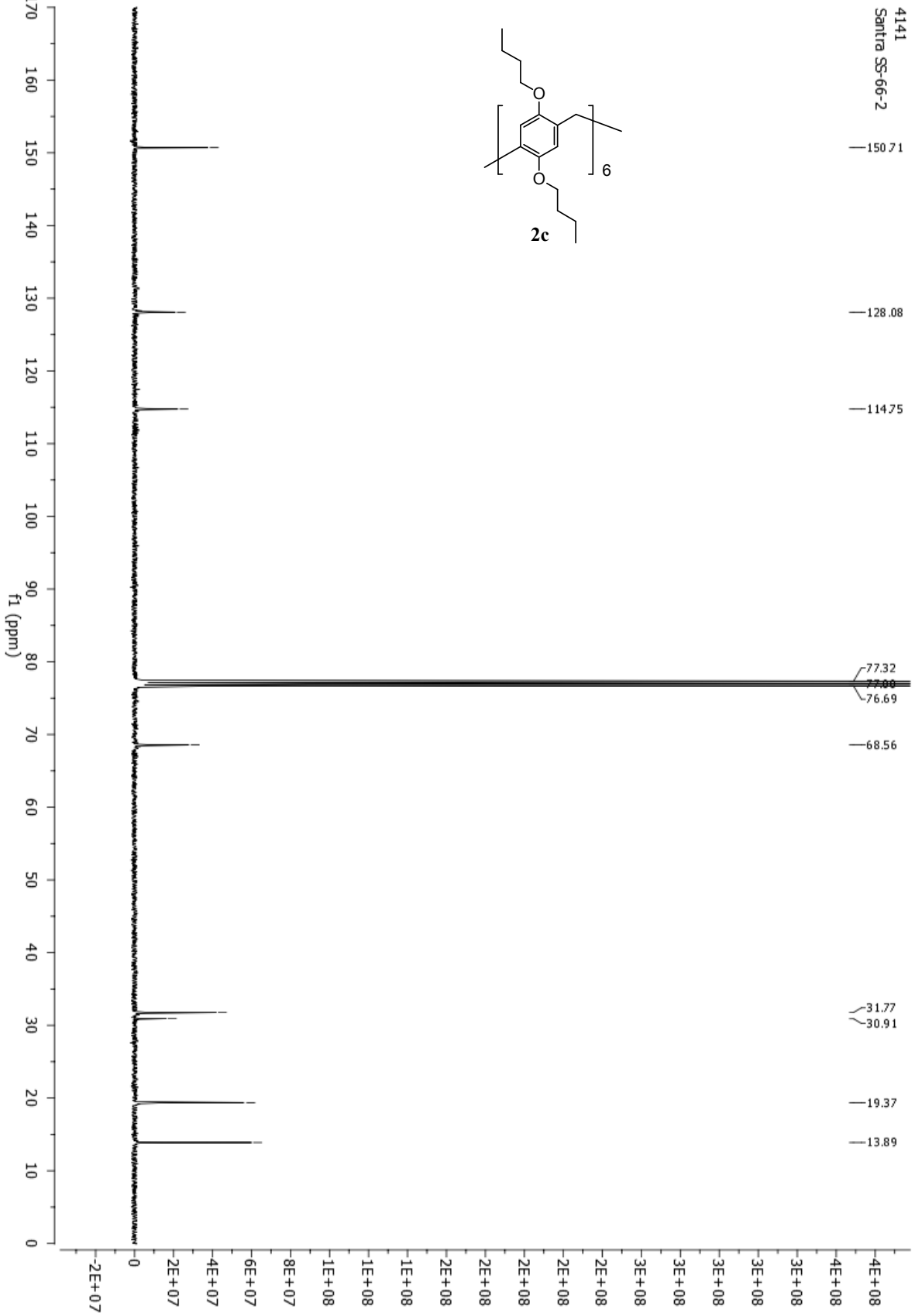
128.07
114.80
77.32
77.00
76.69
70.42
30.90
22.89
10.64
f1 (ppm)
O
O
6

2b

1,4-Bis(butoxy)pillar[6]arene (2c):




4141
Santra SS-66-2
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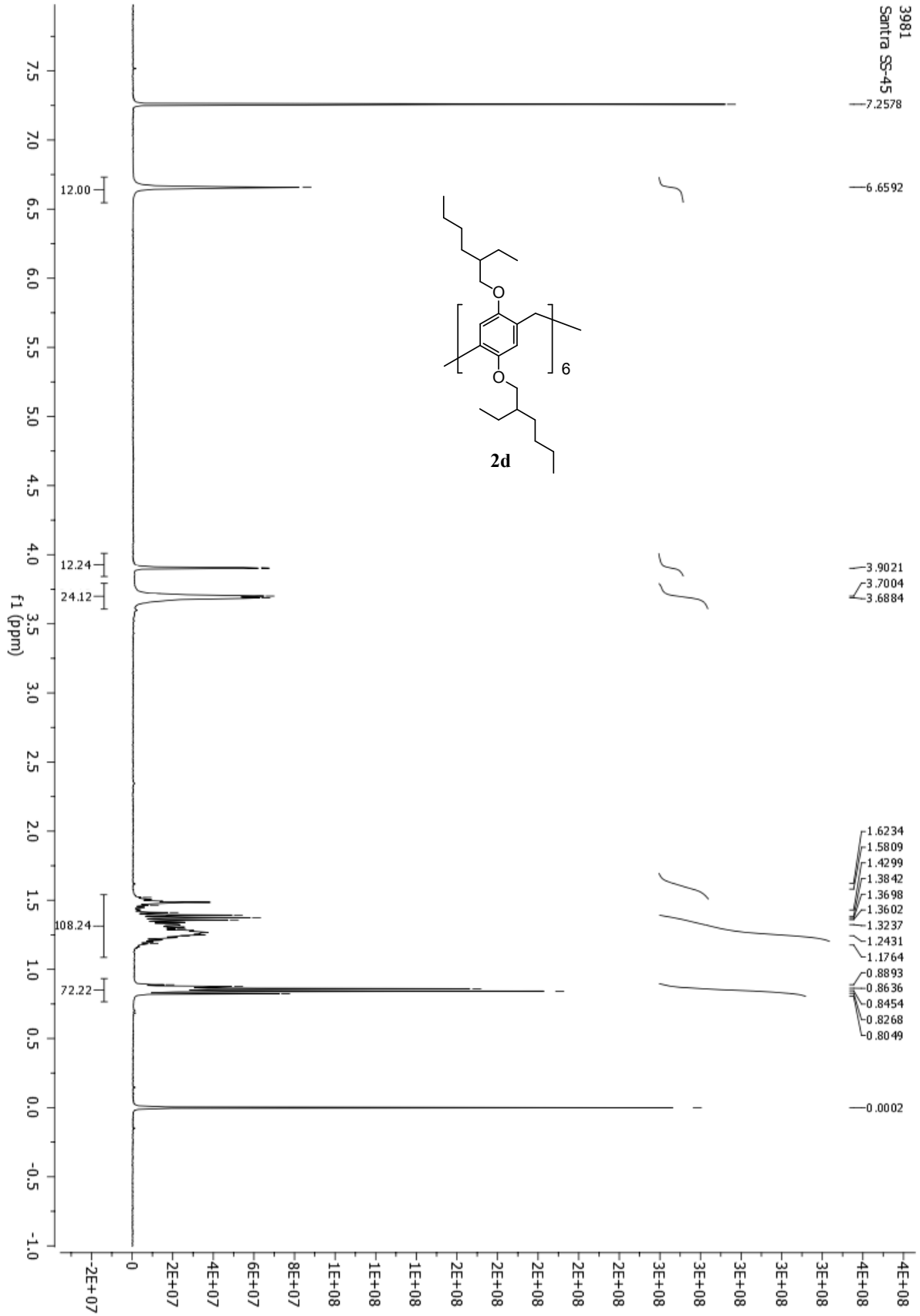
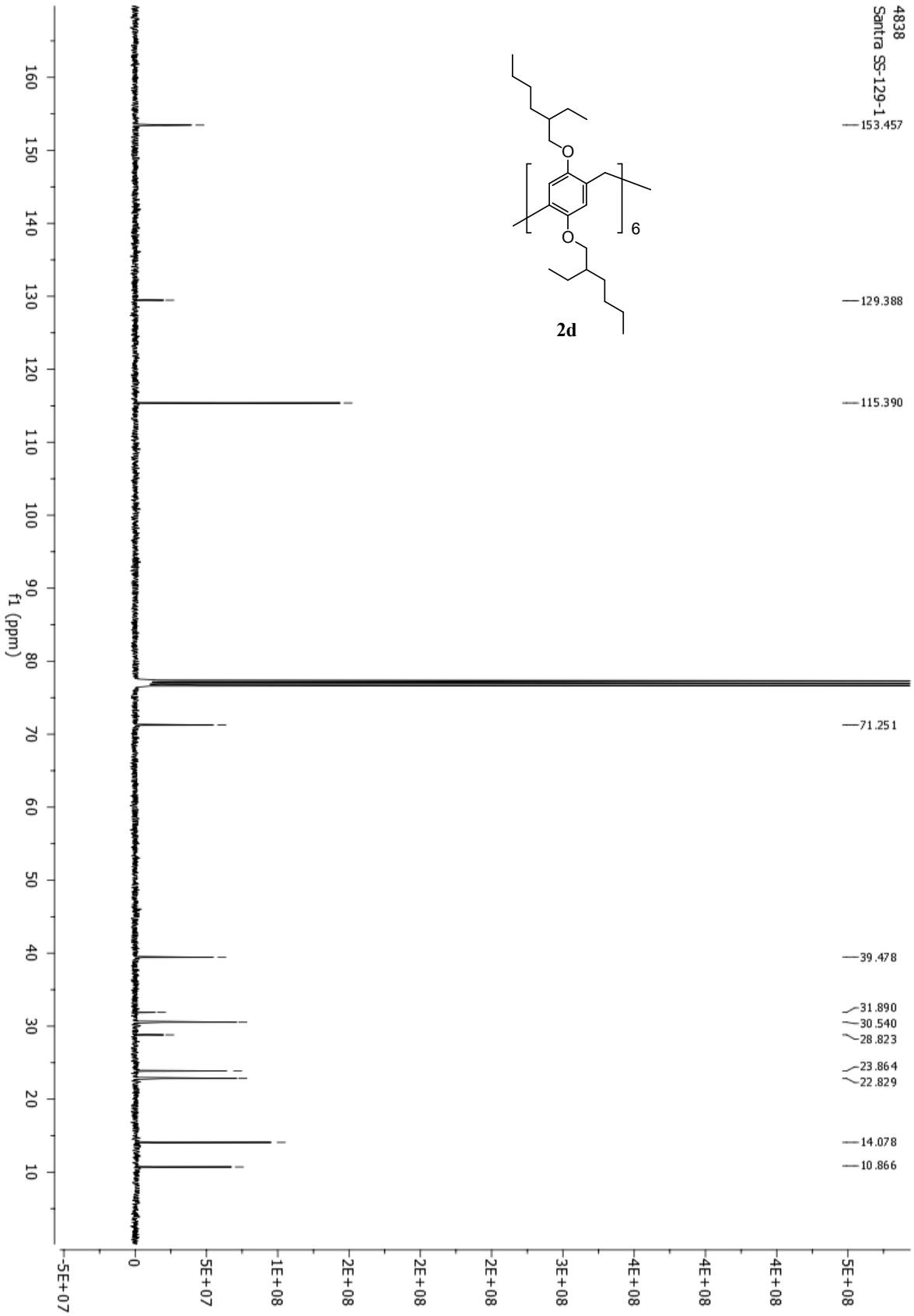
128.08
114.75
77.32
77.00
76.69
68.56
31.77
30.91
19.37
13.89
f1 (ppm)
6
2c

1,4-Bis(2-ethylhexyloxy)pillar[6]arene (2d):



2d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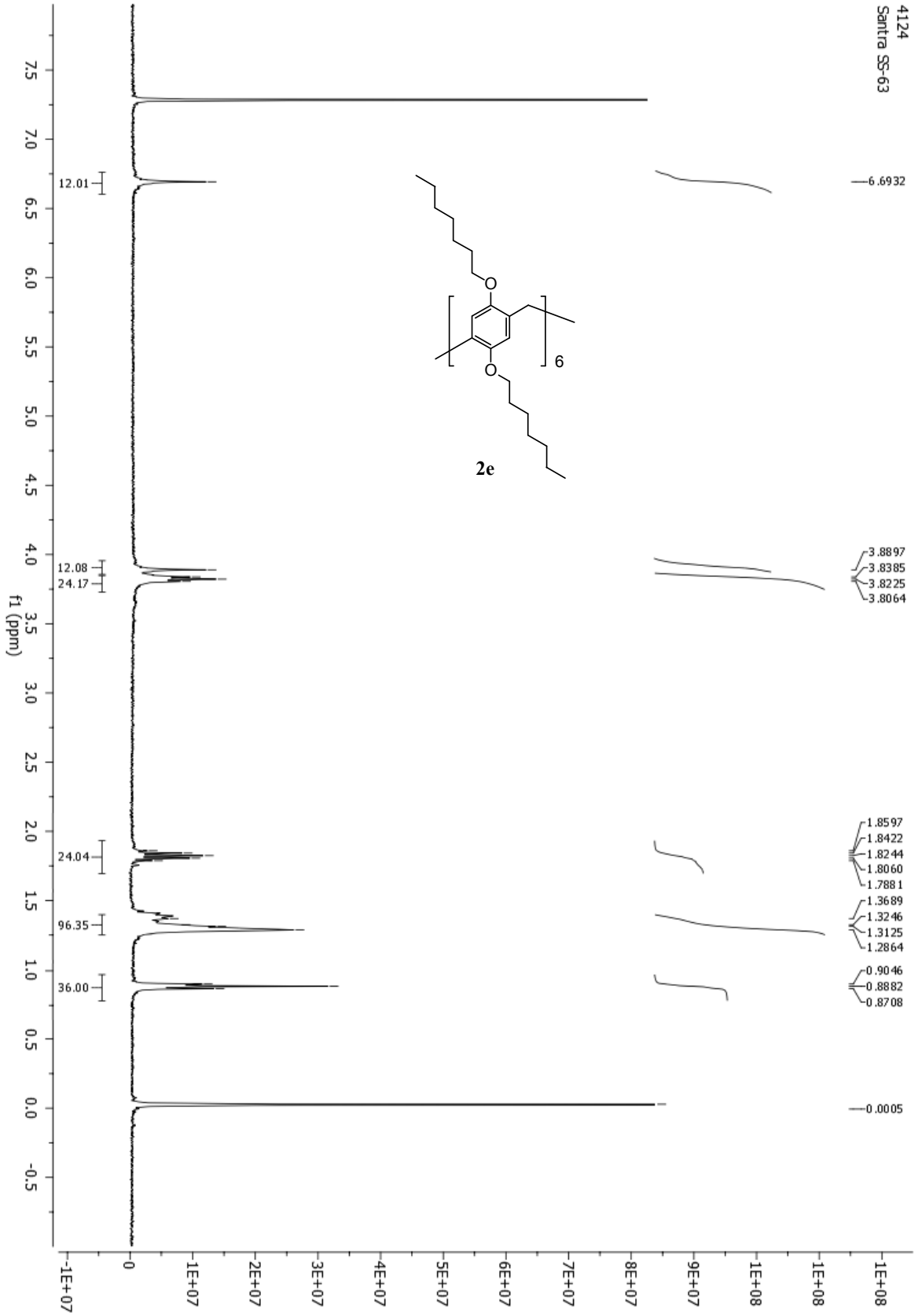
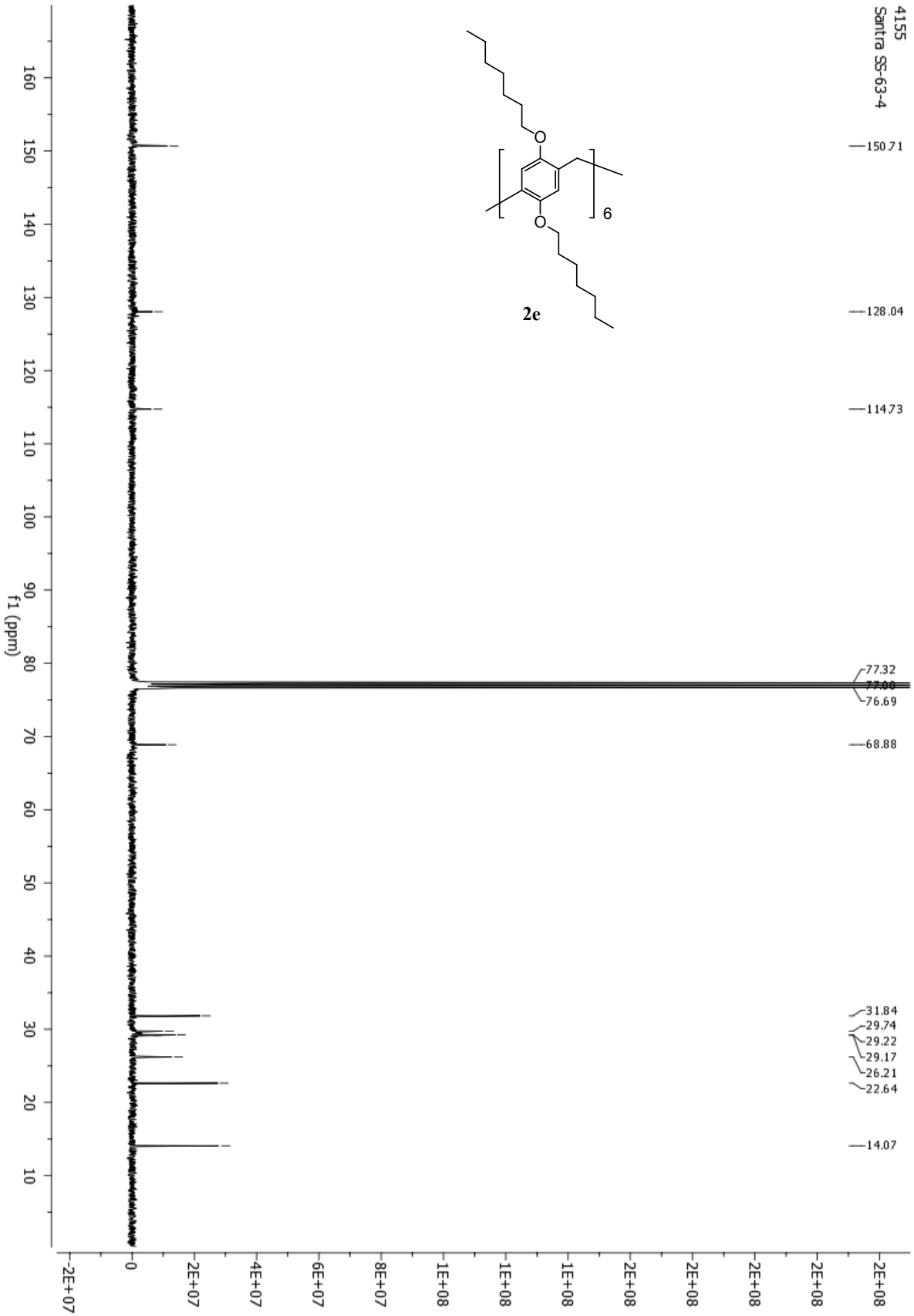
31.890
30.540
28.823
23.864
22.829
14.078
10.866
f1 (ppm)
O
O
6

2d

1,4-Bis(heptyloxy)pillar[6]arene (2e):



2e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114.73
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76.69
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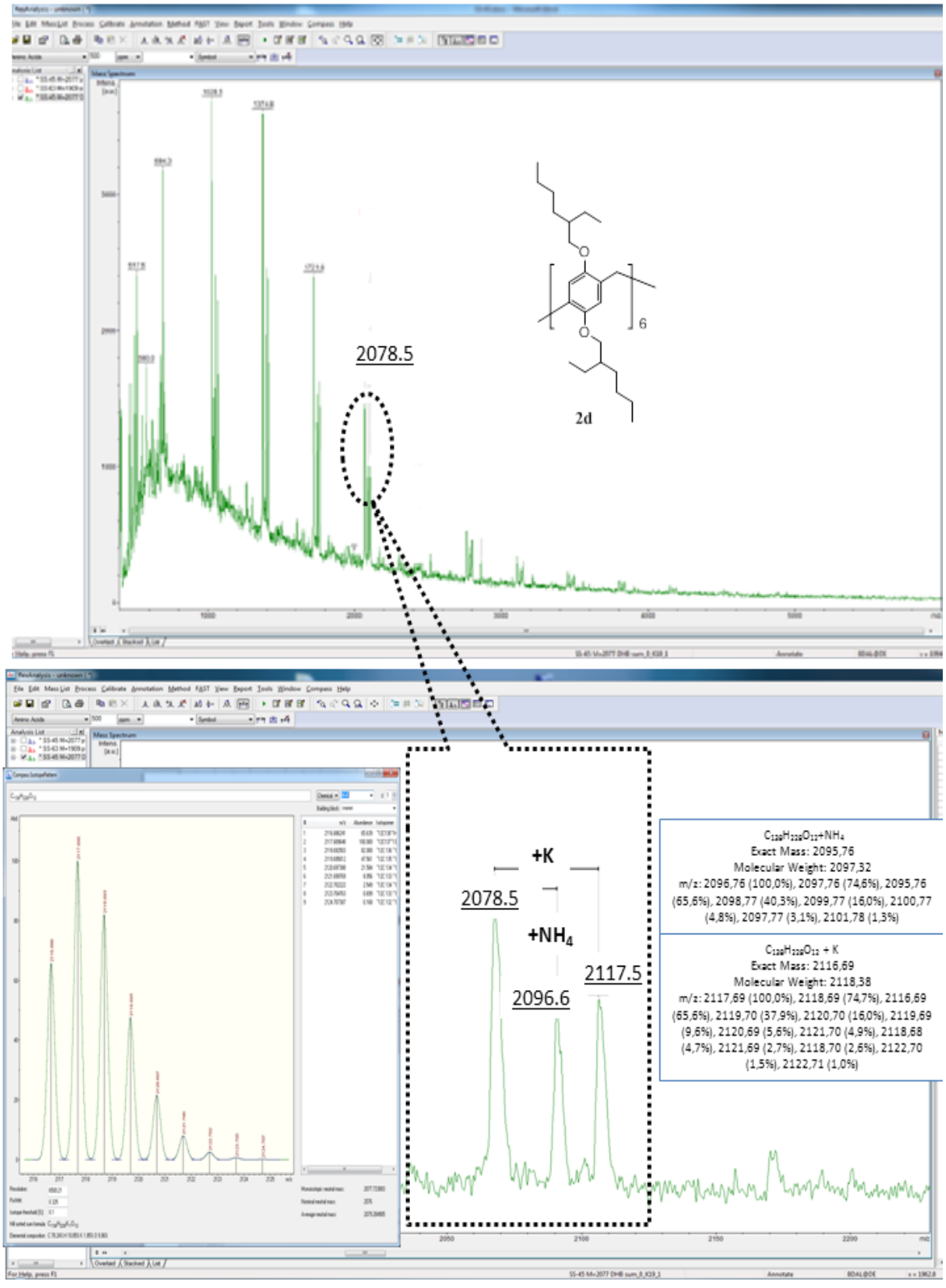
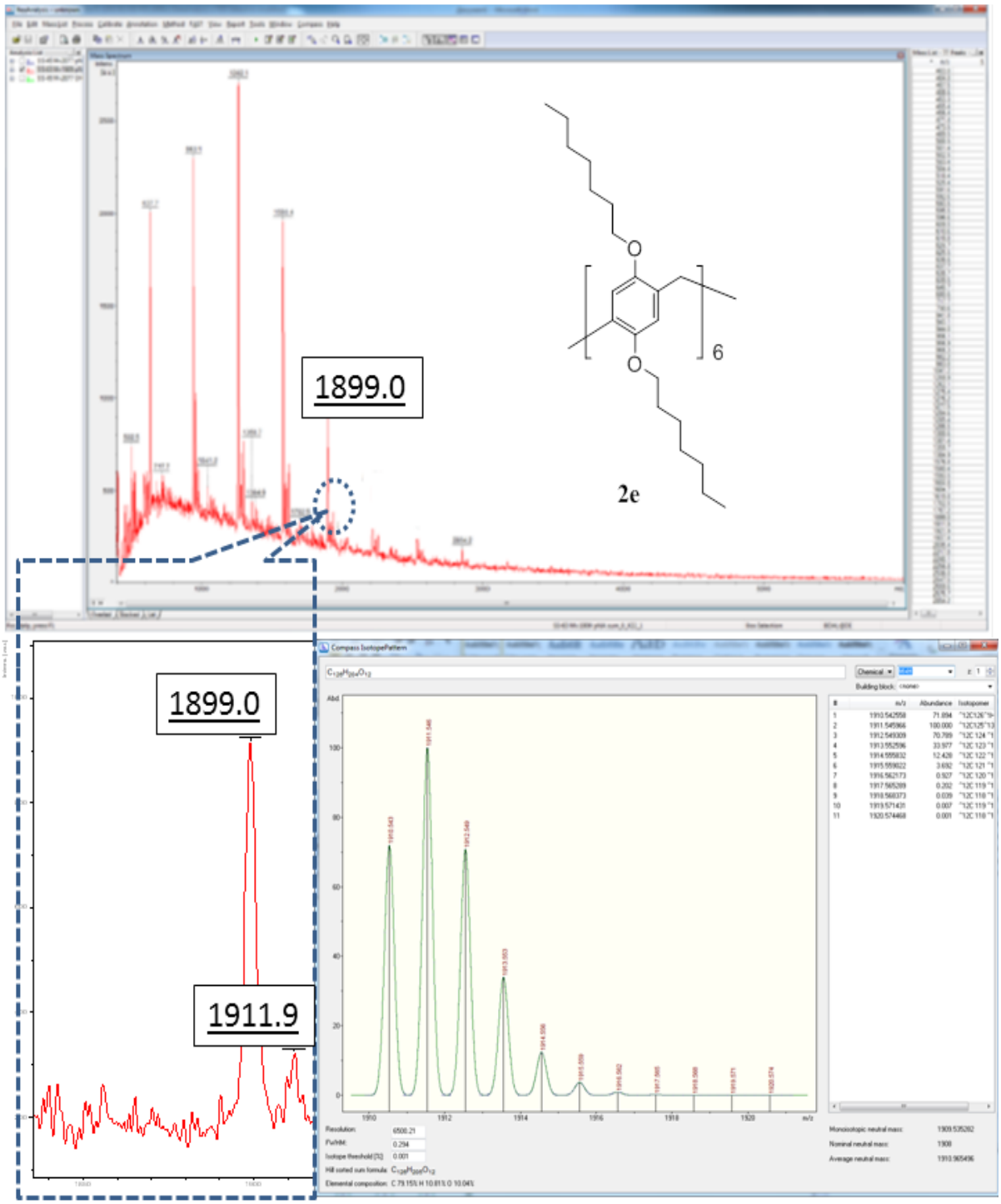
22.64
14.07
f1 (ppm)
O
O
6

2e



$C_{126}H_{228}O_{12}$
Exact Mass: 2077,72
Molecular Weight: 2079,28
m/z: 2078,73 (100,0%), 2079,73 (77,2%), 2077,72 (65,6%), 2080,73 (38,4%), 2081,74 (14,2%), 2082,74 (5,0%), 2080,74 (1,9%), 2081,73 (1,8%), 2083,74 (1,3%)

Fig. 1 MALDI-TOF for **2d**



$C_{126}H_{204}O_{12}H$

Chemical Formula: $C_{126}H_{205}O_{12}$
Exact Mass: 1910,54
Molecular Weight: 1911,97
m/z: 1911,55 (100,0%), 1910,54 (71,9%), 1912,55 (70,8%), 1913,55 (32,4%), 1914,56 (10,7%), 1915,56 (3,6%), 1914,55 (1,7%), 1913,56 (1,6%)
Elemental Analysis: C, 79.15; H, 10.81; O, 10.04

Fig. 2 MALDI-TOF for **2e**

General procedure for the synthesis of 1,4-bis(alkoxy)pillar[5]arenes:

1,4-Bis(alkoxy)pillar[5]arenes have been synthesized by following the reported method.[3] To a solution of 1,4-dialkoxybenzene (10 mmol) in 1,2-dichloroethane (20 mL) was added paraformaldehyde (0.31 g, 10 mmol) under nitrogen atmosphere. Then, boron trifluoride diethyl etherate ($BF_3 \cdot OEt_2$, 1.25 mL, 10 mmol) was added to the solution and the mixture was stirred at 30 °C for 3 h. The solution was poured into methanol and the resulting precipitate was collected by filtration. The solid was dissolved in $CHCl_3$ and the insoluble part was filtered off. The $CHCl_3$ was removed in vacuum, and the residue was crystallized from acetone.

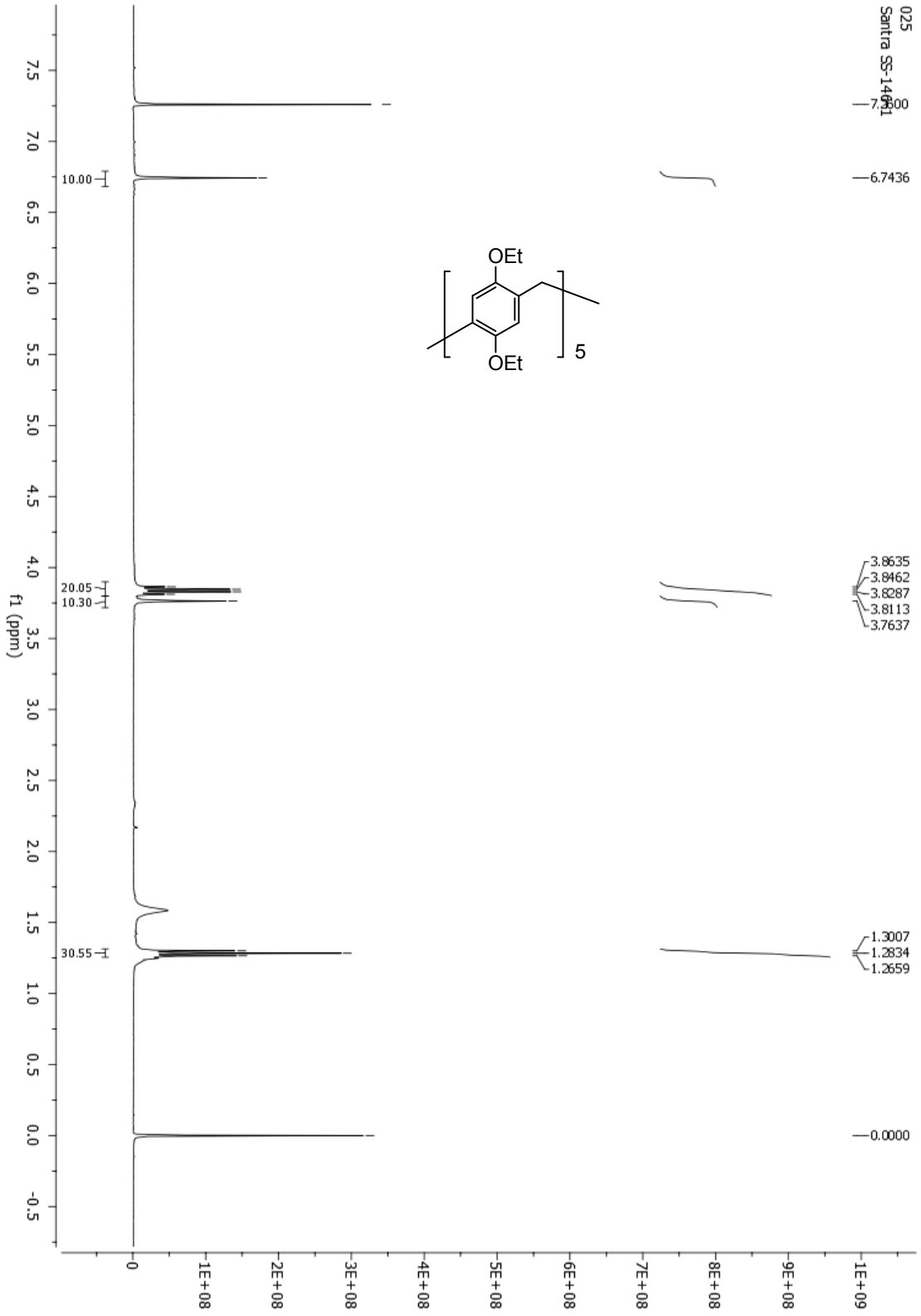
1,4-Bis(ethoxy)pillar[5]arene

White solid, mp. 156-158 °C, yield 15 %. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 6.74 (s, 10H, phenyl protons), 3.83 (q, *J* = 6.92 Hz, 20H, -O*C*$\underline{H_2}$-), 3.76 (s, 10H, methylene bridges), 1.28 (t, *J* = 6.92 Hz, 30H, methyl protons). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 149.66, 128.38, 114.54 (C of phenyl), 63.53 (C of methylene groups), 29.54 (C of methylene bridge), 15.20 (C of methyl groups). ESI-HRMS calcd for $C_{55}H_{71}O_{10}$ $[M]^+$ 891.50468, found 908.5307 $[M+NH_4]^+$. Anal. Calcd for $C_{55}H_{70}O_{10}$: C, 74.13; H, 7.92. Found: C, 74.04; H, 8.11. HPLC Retention time = 21.5 min.

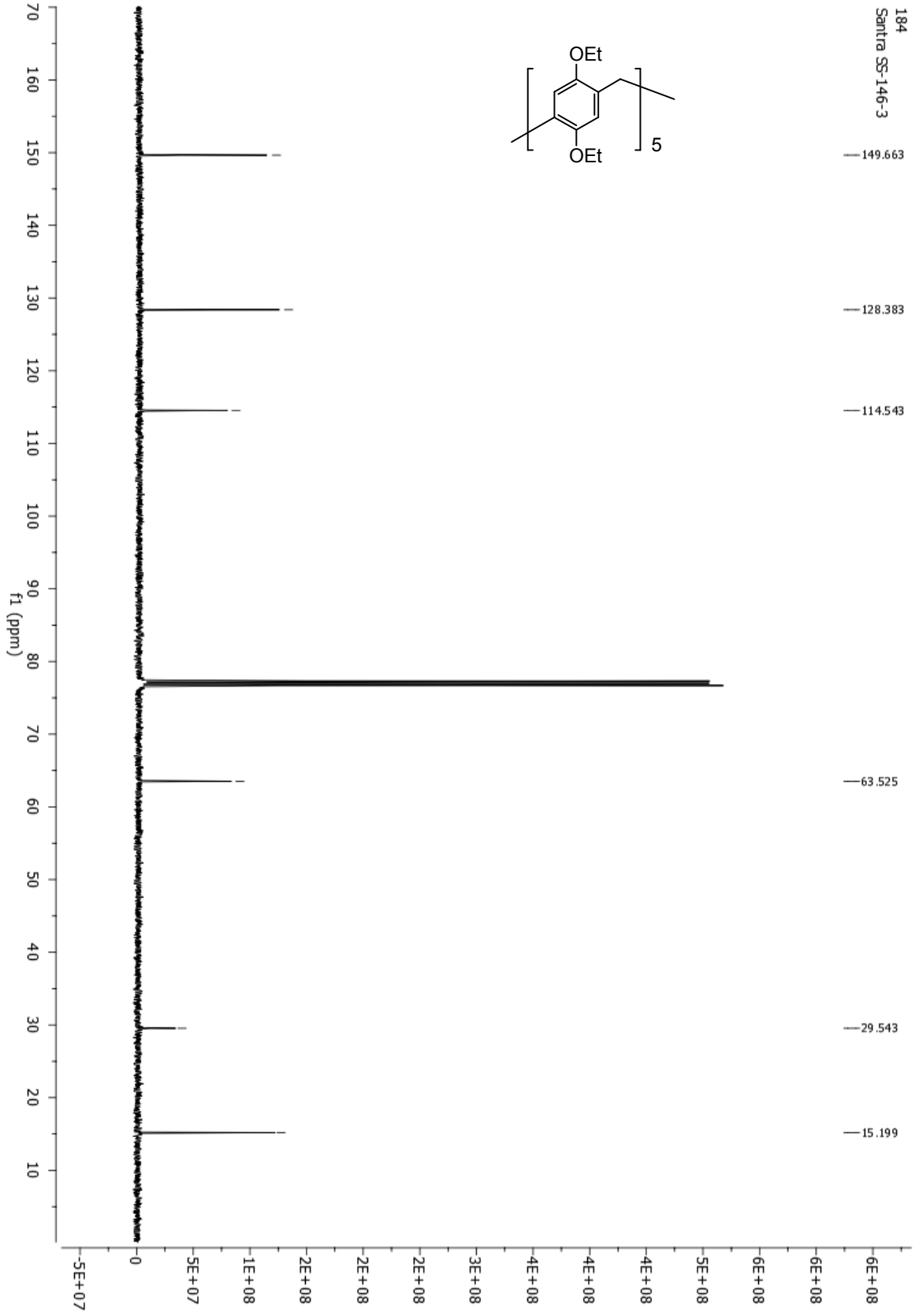
1,4-Bis(Butyloxy)pillar[5]arene

White solid, mp. 129-131 °C, yield 8%. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 6.84 (s, 10H, phenyl protons), 3.85 (t, *J* = 6.54 Hz, 20H, -OC*H*$_2$CH$_2$-), 3.75 (s, 10H, methylene bridges), 1.81-1.74 (m, 20H, -OCH$_2$C*H*$_2$-), 1.56-1.50 (m, 20H, -OCH$_2$CH$_2$C*H*$_2$-), 0.97 (t, *J* = 7.36 Hz, 30H, methyl protons). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 149.78, 128.17, 114.66 (C of phenyl), 67.92, 32.04 (C of methylene groups), 29.36 (C of methylene bridge), 19.52 (C of methylene groups), 14.02 (C of methyl groups). Anal. Calcd for $C_{75}H_{110}O_{10}$: C, 76.88; H, 9.46%. Found: C, 76.76; H, 9.17%.

^{1}H NMR spectra of 1,4-bis(ethoxy)pillar[5]arene synthesized by the reported method.[3]



^{13}C NMR spectra of 1,4-bis(ethoxy)pillar[5]arene synthesized by the reported method.[3]



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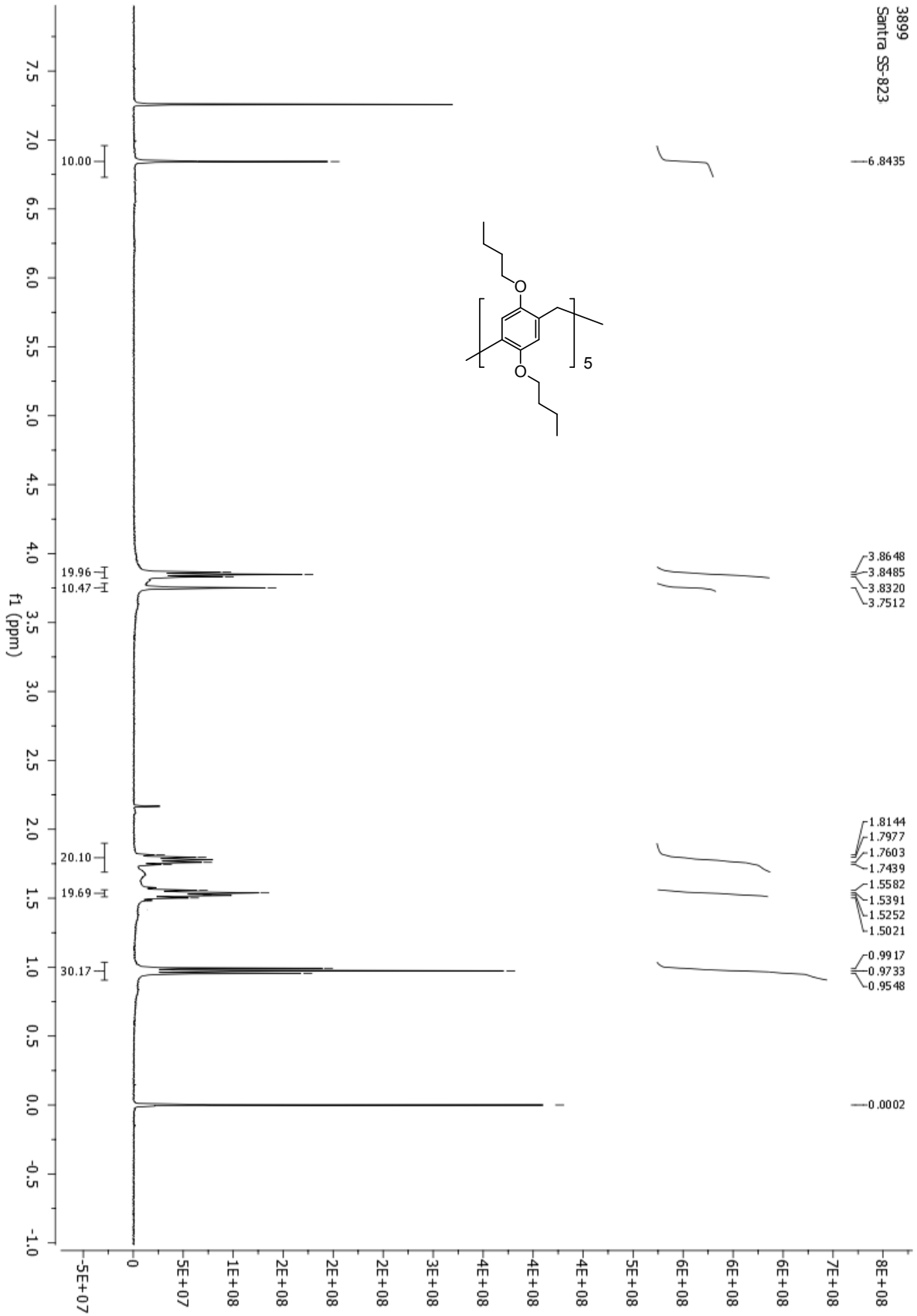
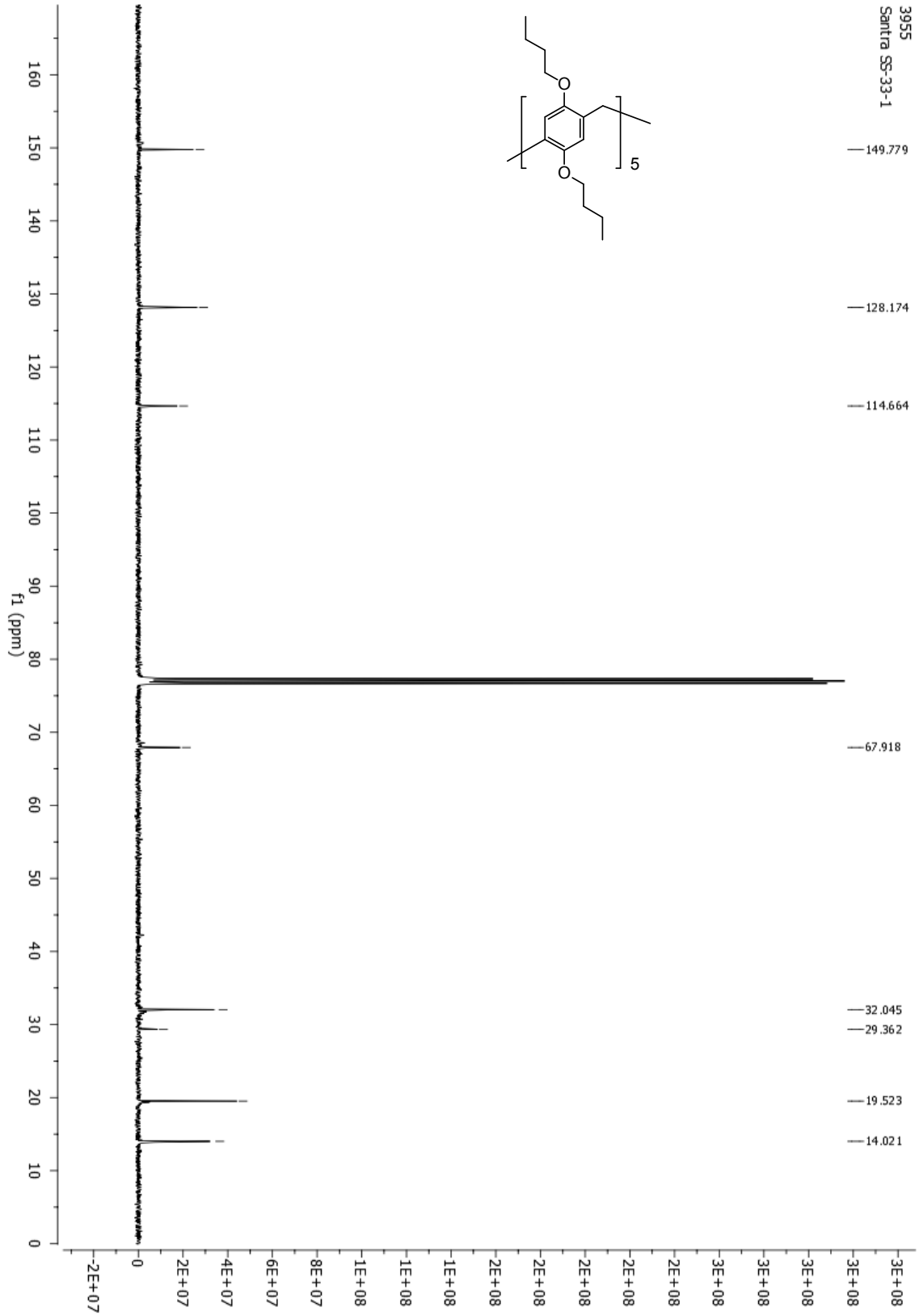


^{13}C NMR spectra of 1,4-bis(butoxy)pillar[5]arene synthesized by the reported method.[3]



^{1}H NMR Spectra of 1,4-bis(butoxy)pillar[6]arene and 1,4-bis(butoxy)pillar[5]arene in one sample: NMR run for a mixture of pillar[5]arene & pillar[6]arene.

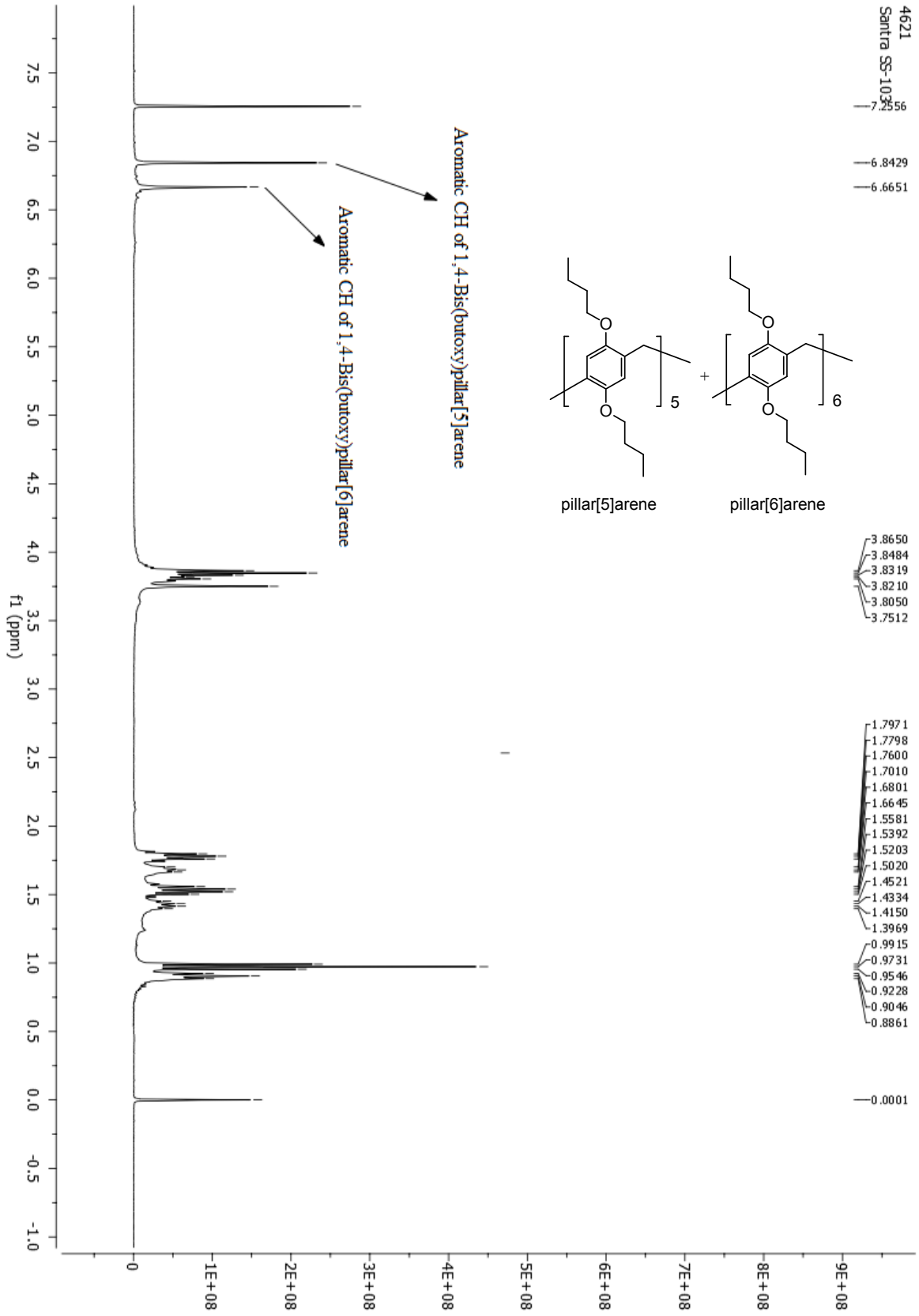


Table 1. ^{1}H NMR data of 1,4-Bis(butoxy) pillar[6]arene (**2c**) and 1,4-Bis(butoxy) pillar[5]arene:

Compound	Aromatic CH	Bridge -CH_2-	OCH_2–	–$OCH_2C\underline{H_2}$–	–$OCH_2CH_2C\underline{H_2}$–	–CH_3
1,4-Bis(butoxy) pillar[6]arene **(2c)**	6.67 s, (12H)	3.87 (s, 12H)	3.80 (t, *J* = 8.96 Hz, 24H)	1.70-1.66 (m, 24H)	1.45-1.39 (m, 24H)	0.90 (t, *J* = 7.36 Hz, 36H
1,4-Bis(butoxy) pillar[5]arene	6.84 s, (10H)	3.75 (s, 10H)	3.85 t, *J* = 6.52 Hz, (20H)	1.81-1.74 (m, 20H)	1.55-1.50 (m, 20H)	0.97 (t, *J* = 7.40 Hz, 30H)

Table 2. ^{13}C NMR data of 1,4-Bis(butoxy) pillar[6]arene (**2c**) and 1,4-Bis(butoxy) pillar[5]arene:

Compound	C of Phenyl	C of oxymethylene groups	C of methylene groups	C of methylene bridge	C of methylene groups	C of methyl groups
1,4-Bis(butoxy) pillar[6]arene **(2c)**	150.71, 128.08, 114.75	68.56	31.77	30.91	19.37	13.89
1,4-Bis(butoxy) pillar[5]arene	149.78,128.17, 114.66,	67.92	32.04	29.36	19.52	14.02

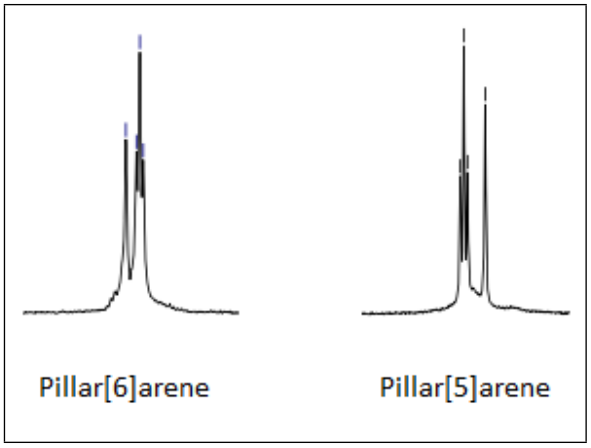


Fig. 3 Spectra pattern of methylene bridges and oxymethylene protons of 1,4-bis(butoxy) pillar[6]arene & 1,4-bis(butoxy) pillar[5]arene

Table 3. Results for using other Lewis or Brønsted acids as catalysts for the synthesis of **2a** [a]

Entry	Catalyst (mol%)	Yields (%)
1	$FeCl_3$ (20)	28
2	$MgCl_2$ (20)	0
3	$BF_3{\cdot}OEt_2$ (20)	15
4	$BF_3{\cdot}OMe_2$ (20)	10
5	$ZnCl_2$ (20)	Traces
6	*p*-$CH_3C_6H_4SO_3H$ (*p*-TSA, 20)	Traces

[a] Reaction conditions: 5 mmol of 1,4-diethoxybenzene, 15 mmol of paraformaldehyde in presence of catalyst for 10 min grinding by hand.

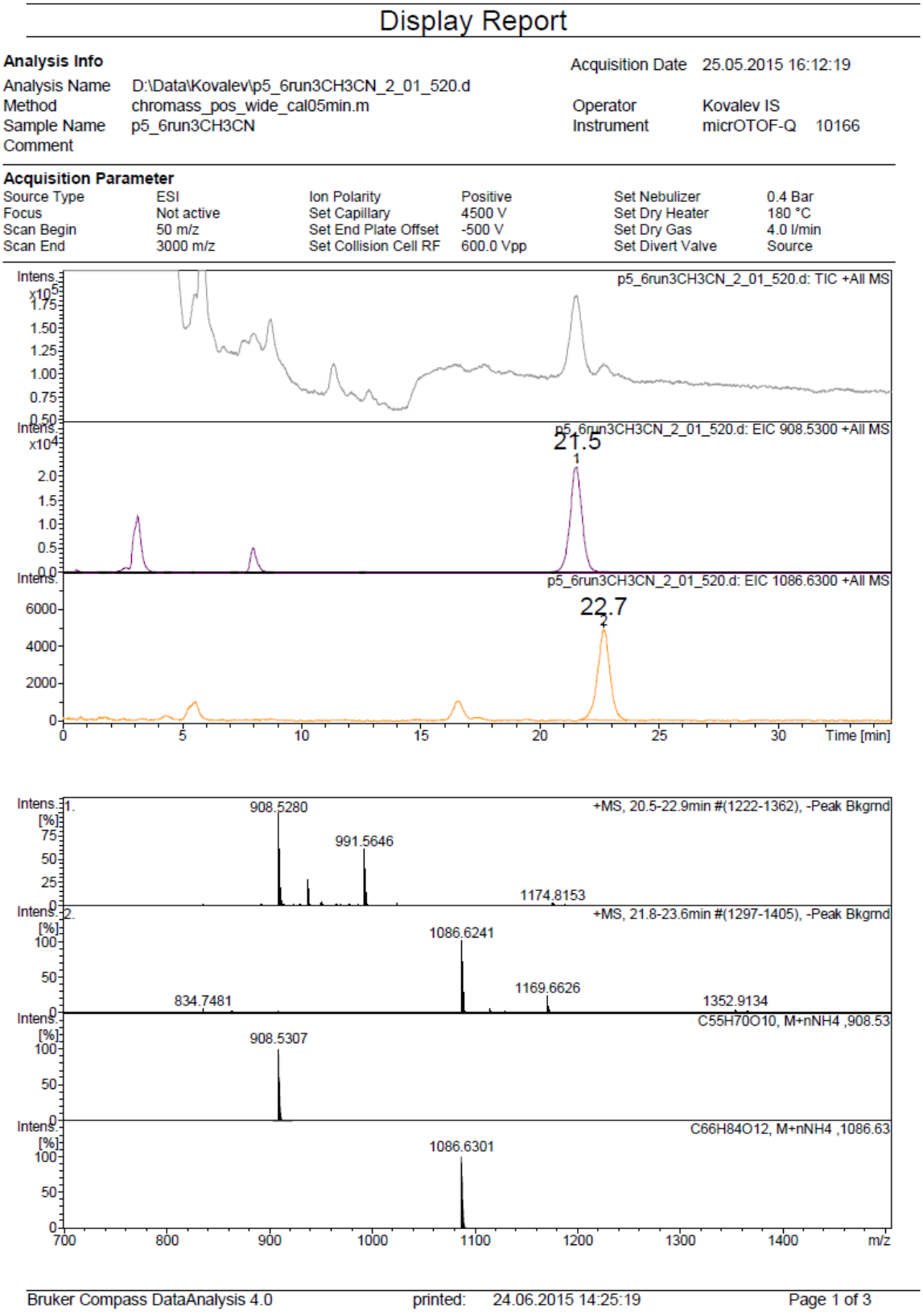


Fig. 4 The data for HPLC analysis for 1,4-bis(ethoxy)-pillar[5]arene (top) and pillar[6]arene **2a** (bottom)

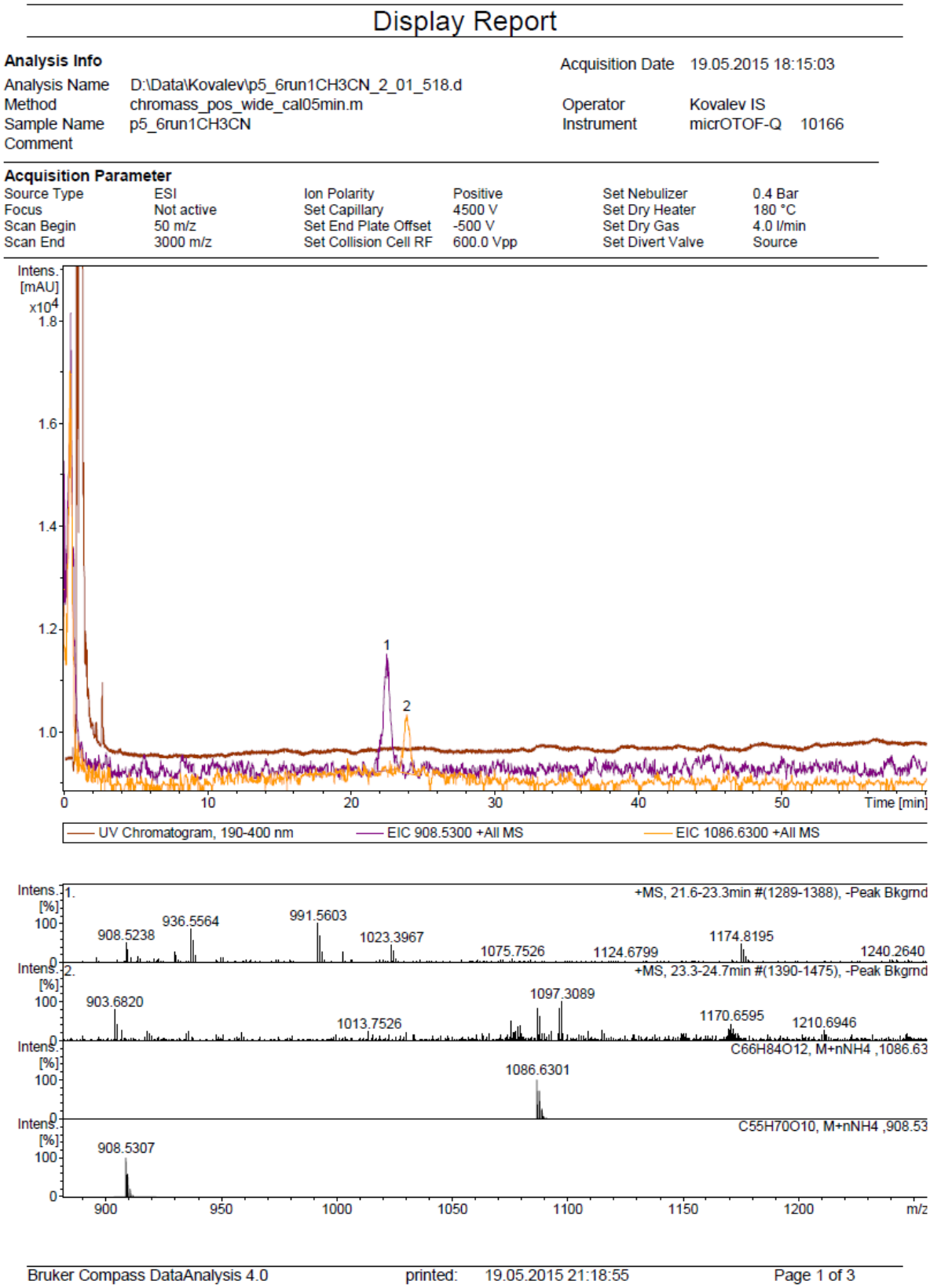


Fig. 5 The data for HPLC analysis for the mixture of 1,4-bis(ethoxy)-pillar[5]arene and pillar[6]arene **2a**

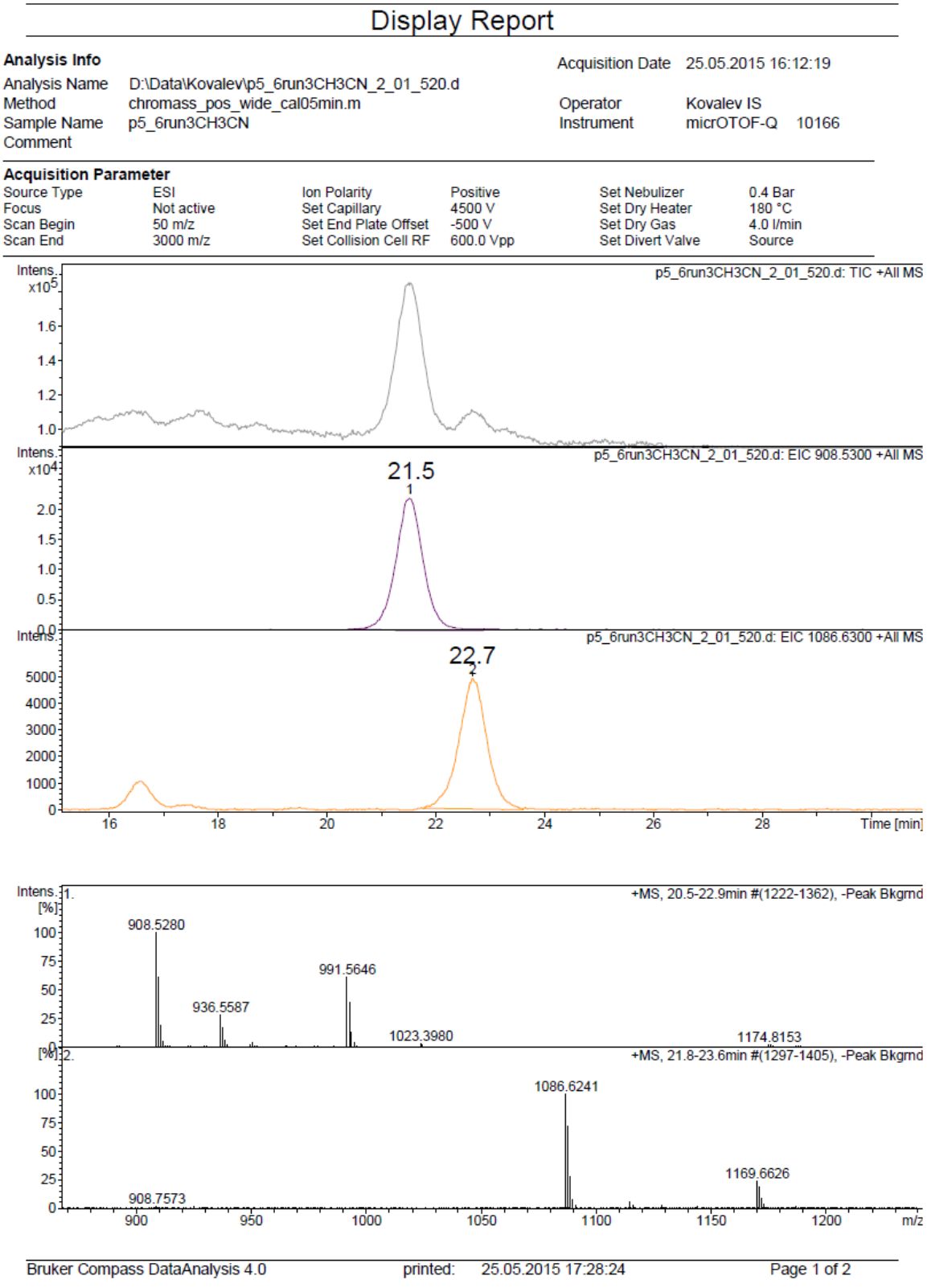


Fig. 6 The data for HPLC analysis for the mixture of 1,4-bis(ethoxy)-pillar[5]arene and pillar[6]arene **2a**

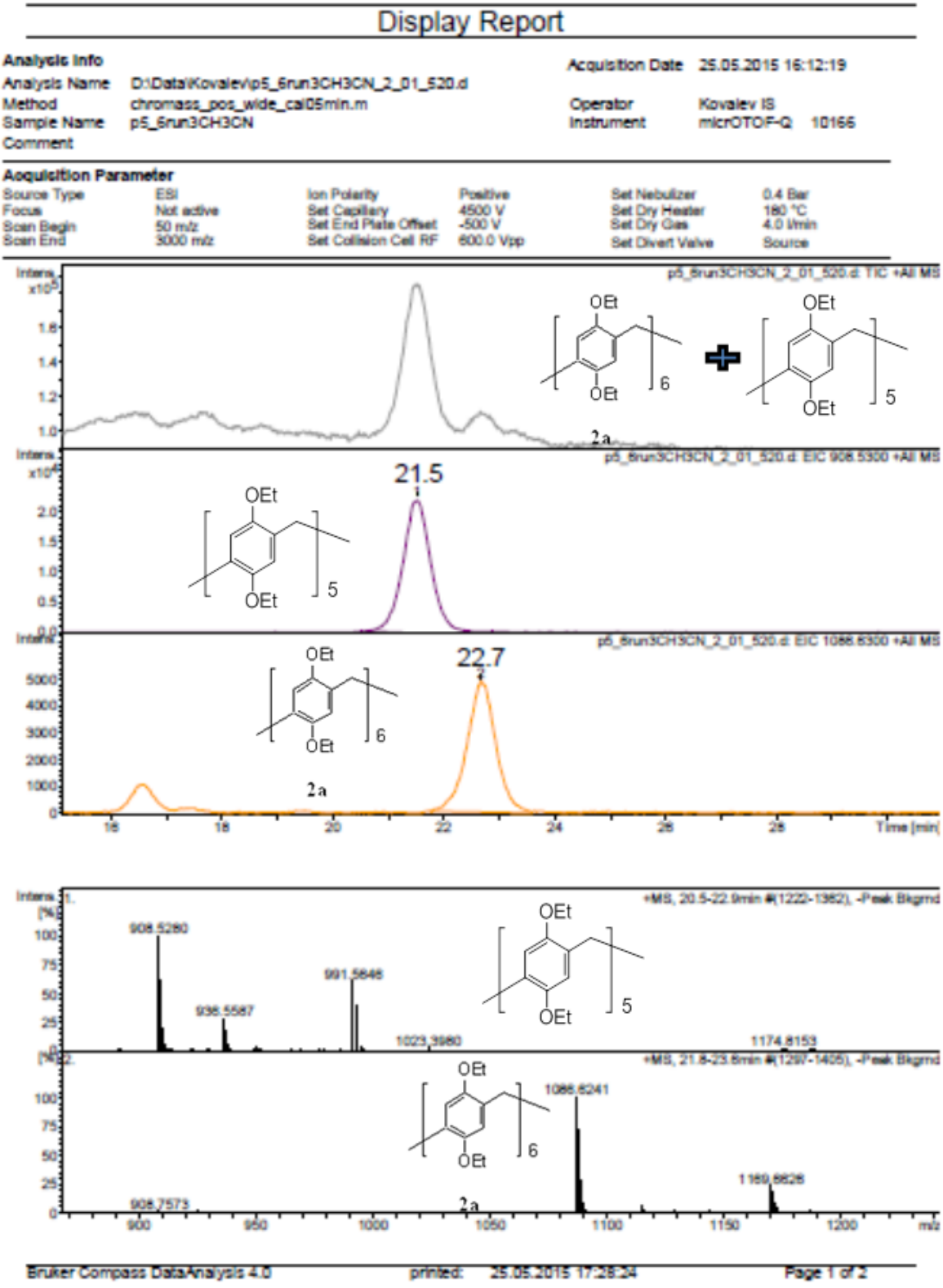


Fig. 7 The data for HPLC analysis for the mixture of 1,4-bis(ethoxy)-pillar[5]arene and pillar[6]arene **2a**

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