Supporting Information for

Solid-phase Synthesis and Circular Dichroism Study of β-ABpeptoids

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Table S1. The optical rotations of (S)- and (R)-forms of the products and intermediates

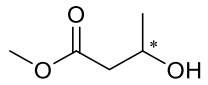
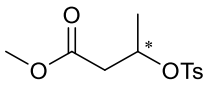
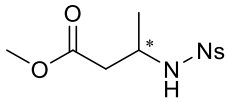
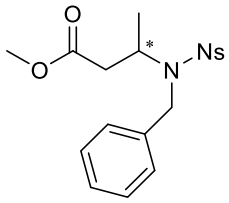
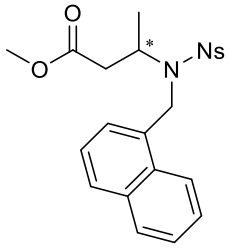
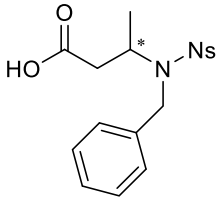
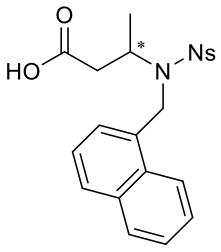
structure	(S)-isomer	(R)-isomer
	+22.9 º (ACN)	-23.3 º (ACN)
	+7.8 º (ACN)	-8.3 º (ACN)
	+14.8 º (ACN)	-16.3 º (ACN)
	+5.4 º (ACN)	-5.8 º (ACN)
	+7.0 º (ACN)	not synthesized
	+8.3 º (ACN/$CHCl_3$ 2:1)	-8.7 º (ACN/$CHCl_3$ 2:1)
	+8.1 º (ACN/$CHCl_3$ 2:1)	not synthesized

Figure S1. Chiral HPLC analyses of compounds **(S)/(R)-5a**, and **(S)/(R)-6a.**

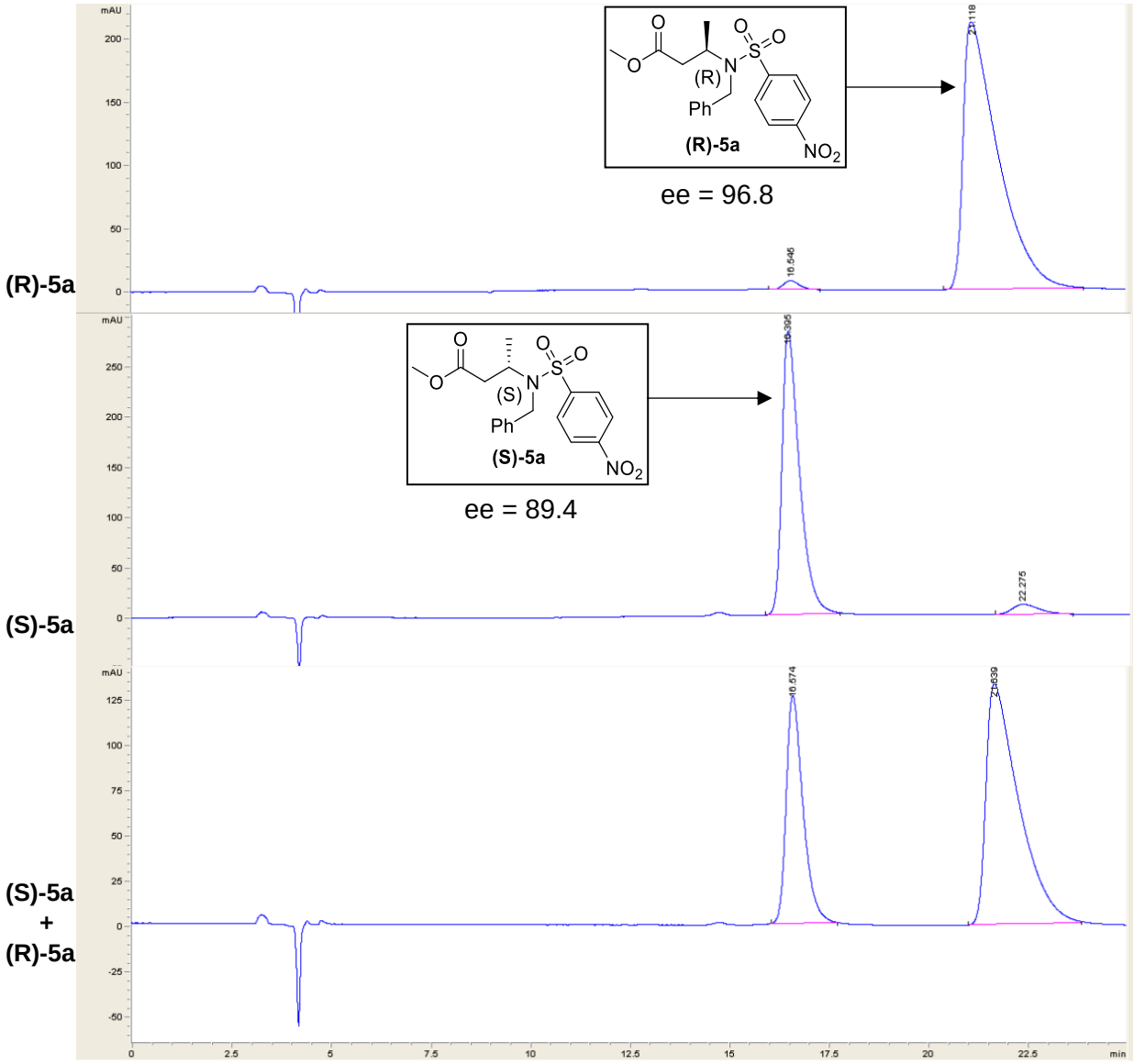


Figure S1, continued

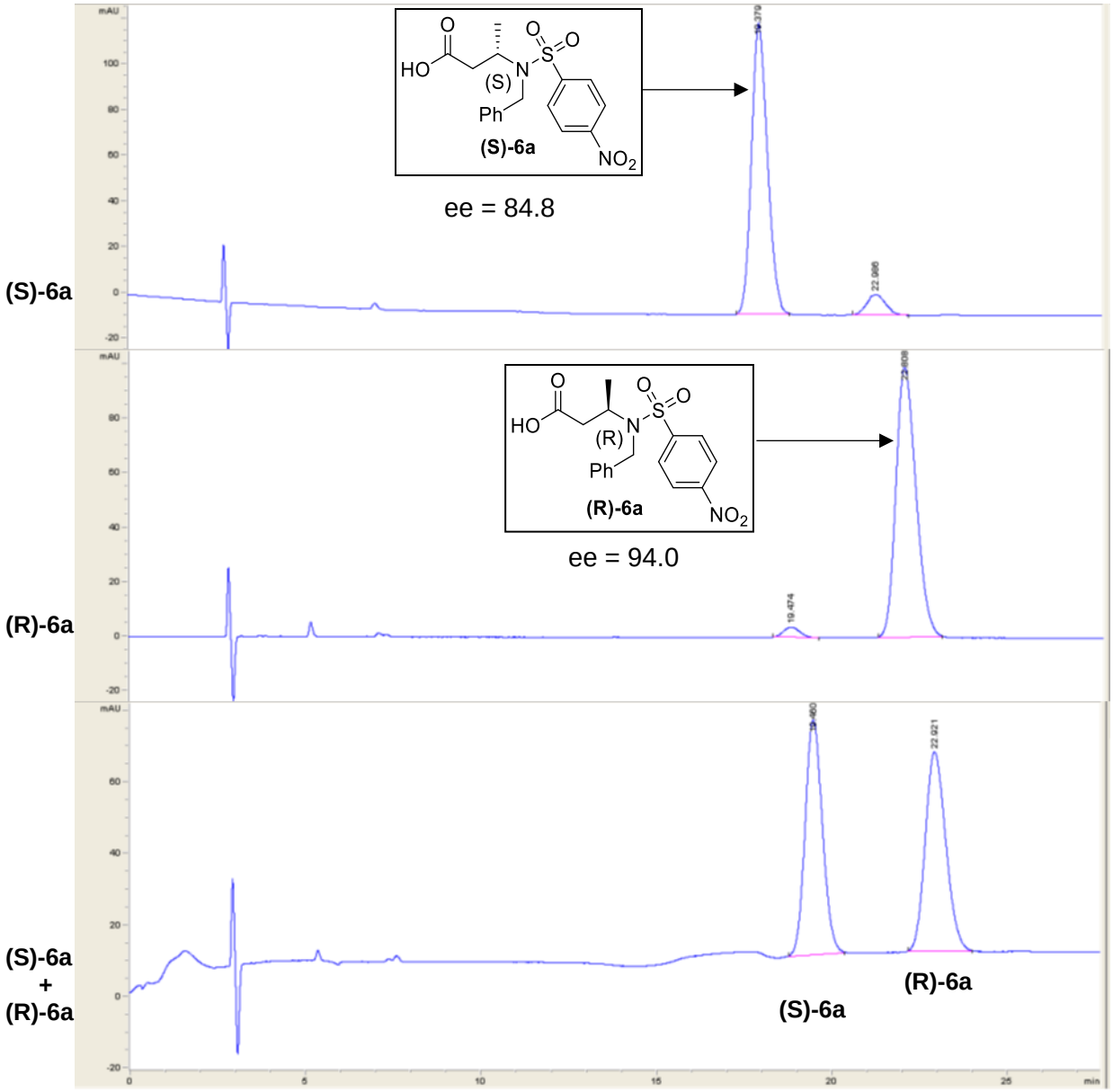
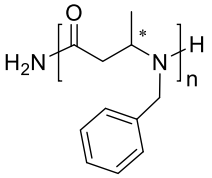
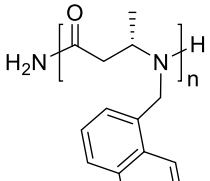


Table S2. Isolated yields and nature of synthesized β-ABpeptoid oligomers



9a-g

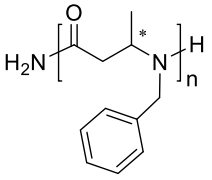


10a-g

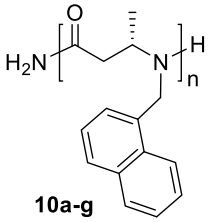
compd. no.	resin Qty. (mg)[a]	isolated product Qty. (mg)[b]	isolated product yield (%)[c]	compound nature
(S)-9a	40	4.8	72%	colorless sticky oil
(S)-9b	40	6.8	70%	colorless sticky semi-solid
(S)-9c	40	8.9	69%	white solid
(S)-9d	30	8.2	68%	white solid
(S)-9e	30	10.4	72%	white solid
(S)-9f	20	7.3	66%	white solid
(S)-9g	20	8.1	63%	white solid
(S)-9e-Ac[d]	40	12.6	64%	white solid
(R)-9a	30	3.5	71%	colorless sticky oil
(R)-9b	30	5.3	73%	colorless sticky semi-solid
(R)-9c	30	6.5	67%	white solid
(R)-9d	20	5.0	62%	white solid
(R)-9e	20	6.1	63%	white solid
(R)-9f	20	6.6	59%	white solid
(R)-9g	20	7.2	57%	white solid
(R)-9e-Ac[d]	20	5.7	58%	white solid
(S)-10a	25	4.9	91%	colorless sticky oil
(S)-10b	25	6.0	78%	white semi-solid
(S)-10c	20	9.1	89%	white solid
(S)-10d	20	7.8	76%	white solid
(S)-10e	20	8.6	70%	white solid
(S)-10f	20	9.6	68%	white solid
(S)-10g	20	10.3	63%	white solid
(S)-10e-Ac[d]	20	7.6	60%	white solid

[a]Initial resin loading was 0.45 mmol/gm. [b]Determined based on 100% initial resin loading of the first residue. [c]Quantities of isolated products are given after lyophilization of purified products. [d]*N*-terminal is acetylated.

Table S3. Synthesized *β*-ABpeptoid Oligomer Sequence, Purity, and Mass Confirmation



9a-g

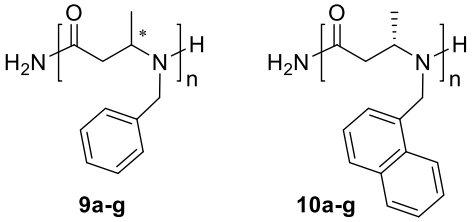


10a-g

compd no.	chain length	% purity[a]	calcd mass	obsd mass[b]
(S)-9a	2	99	367.23	368.2 $[M+H]^+$
(S)-9b	3	98	542.33	543.3 $[M+H]^+$
(S)-9c	4	98	717.43	718.4 $[M+H]^+$
(S)-9d	5	99	892.53	893.4 $[M+H]^+$
(S)-9e	6	97	1067.62	1068.5 $[M+H]^+$
(S)-9f	7	99	1242.72	1243.6 $[M+H]^+$
(S)-9g	8	99	1417.82	1440.9 $[M+Na]^+$[c]
(S)-9e-Ac[d]	6	99	1109.64	1132.5 $[M+H]^+$
(R)-9a	2	97	367.23	368.2 $[M+H]^+$
(R)-9b	3	98	542.33	543.3 $[M+H]^+$
(R)-9c	4	98	717.43	718.4 $[M+H]^+$
(R)-9d	5	97	892.53	893.5 $[M+H]^+$
(R)-9e	6	98	1067.62	1068.6 $[M+H]^+$
(R)-9f	7	99	1242.72	1265.8 $[M+Na]^+$[c]
(R)-9g	8	98	1417.82	1439.8 $[M+H]^+$[c]
(R)-9e-Ac[d]	6	98	1109.64	1133.5 $[M+Na]^+$
(S)-10a	2	99	467.26	468.2 $[M+H]^+$
(S)-10b	3	98	692.37	693.4 $[M+H]^+$
(S)-10c	4	99	917.49	918.4 $[M+H]^+$
(S)-10d	5	99	1142.60	1144.5 $[M+H]^+$
(S)-10e	6	99	1367.72	1368.6 $[M+H]^+$
(S)-10f	7	99	1592.83	1614.8 $[M+Na]^+$[c]
(S)-10g	8	97	1817.95	1839.9 $[M+Na]^+$[c]
(S)-10e-Ac[d]	6	98	1409.73	1431.7 $[M+Na]^+$[c]

[a]Determined by analytical reversed-phase HPLC of purified products. [b]Mass spectrometry data were acquired using ESI techniques. [c]Mass spectrometry data were acquired using MALDI-TOF technique. [d]*N*-terminal is acetylated.

Table S4. HRMS Data for Synthesized *β*-ABpeptoid Oligomers



compd no.	chain length	chemical formula	calcd mass	obsd mass
(S)-9a	2	$C_{22}H_{29}N_3O_2$	368.2338 $[M+H]^+$	368.2339
(S)-9b	3	$C_{33}H_{42}N_4O_3$	543.3335 $[M+H]^+$	543.3332
(S)-9c	4	$C_{44}H_{55}N_5O_4$	718.4332 $[M+H]^+$	718.4334
(S)-9d	5	$C_{55}H_{68}N_6O_5$	893.5329 $[M+H]^+$	893.5332
(S)-9e	6	$C_{66}H_{81}N_7O_6$	1068.6327 $[M+H]^+$	1068.6323
(S)-9f	7	$C_{77}H_{94}N_8O_7$	1243.7324 $[M+H]^+$	1243.7321
(S)-9g	8	$C_{88}H_{107}N_9O_8$	1418.8321 $[M+H]^+$	1418.8329
(S)-9e-Ac[a]	6	$C_{68}H_{83}N_7O_7$	1132.6252 $[M+Na]^+$	1132.6252
(R)-9a	2	$C_{22}H_{29}N_3O_2$	368.2338 $[M+H]^+$	368.2335
(R)-9b	3	$C_{33}H_{42}N_4O_3$	543.3335 $[M+H]^+$	543.3334
(R)-9c	4	$C_{44}H_{55}N_5O_4$	718.4332 $[M+H]^+$	718.4329
(R)-9d	5	$C_{55}H_{68}N_6O_5$	893.5329 $[M+H]^+$	893.5333
(R)-9e	6	$C_{66}H_{81}N_7O_6$	1068.6327 $[M+H]^+$	1068.6330
(R)-9f	7	$C_{77}H_{94}N_8O_7$	1243.7324 $[M+H]^+$	1243.7329
(R)-9g	8	$C_{88}H_{107}N_9O_8$	1418.8321 $[M+H]^+$	1418.8328
(R)-9e-Ac[a]	6	$C_{68}H_{83}N_7O_7$	1132.6252 $[M+Na]^+$	1132.6247
(S)-10a	2	$C_{30}H_{33}N_3O_2$	468.2651 $[M+H]^+$	468.2653
(S)-10b	3	$C_{45}H_{48}N_4O_3$	693.3805 $[M+H]^+$	693.3808
(S)-10c	4	$C_{60}H_{63}N_5O_4$	918.4958 $[M+H]^+$	918.4956
(S)-10d	5	$C_{75}H_{78}N_6O_5$	1143.6112 $[M+H]^+$	1143.6110
(S)-10e	6	$C_{90}H_{93}N_7O_6$	1368.7266 $[M+H]^+$	1368.7272
(S)-10f	7	$C_{105}H_{108}N_8O_7$	1593.8419 $[M+H]^+$	1593.8427
(S)-10g	8	$C_{120}H_{123}N_9O_8$	1818.9573 $[M+H]^+$	1818.9580

[a]*N*-terminal is acetylated. ***Note***- High Resolution Mass Spectrometry (HRMS) data were acquired using Fast Atom Bombardment (FAB^+) ionization techniques.

Figure S2. LC/MS spectra of crude product of nosyl protected dimer **(S)-9a-Ns**

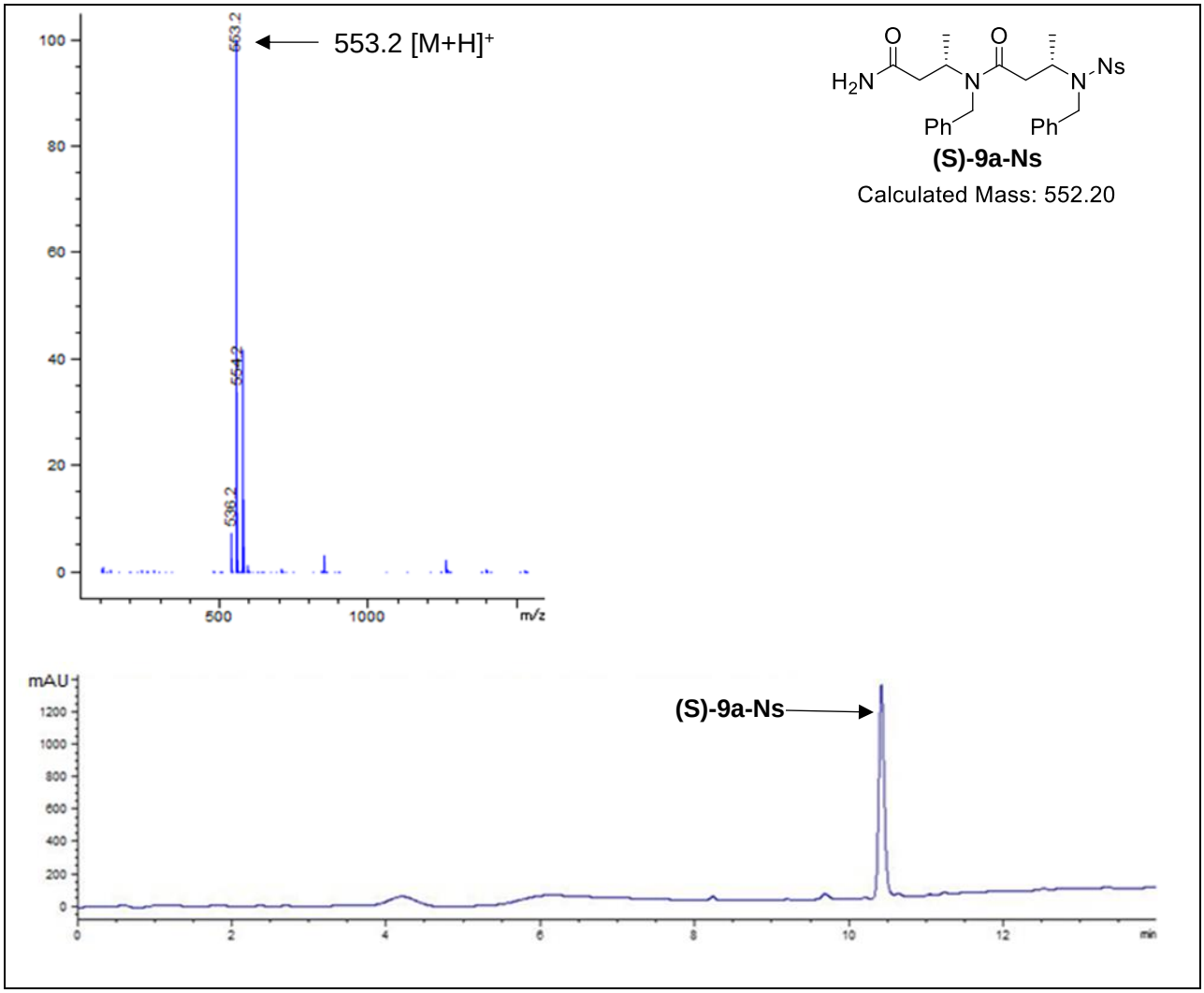
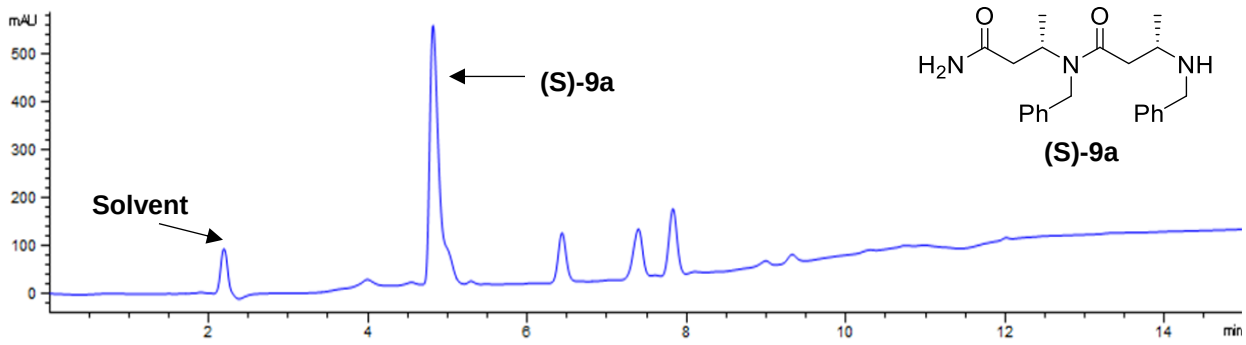


Figure S3. HPLC chromatograms of crude products **(S)-9a-g** and **(S)-10a-g**.



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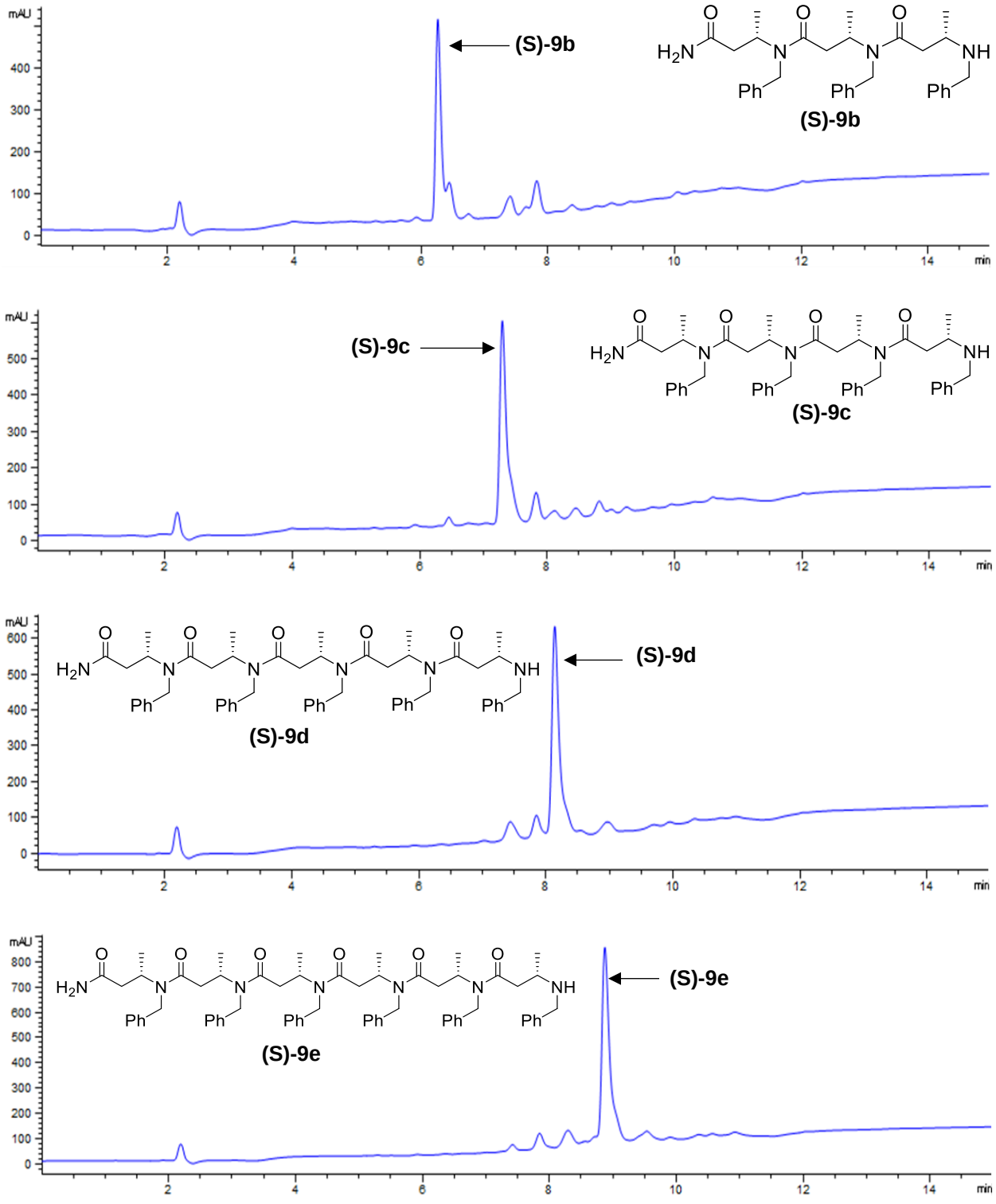

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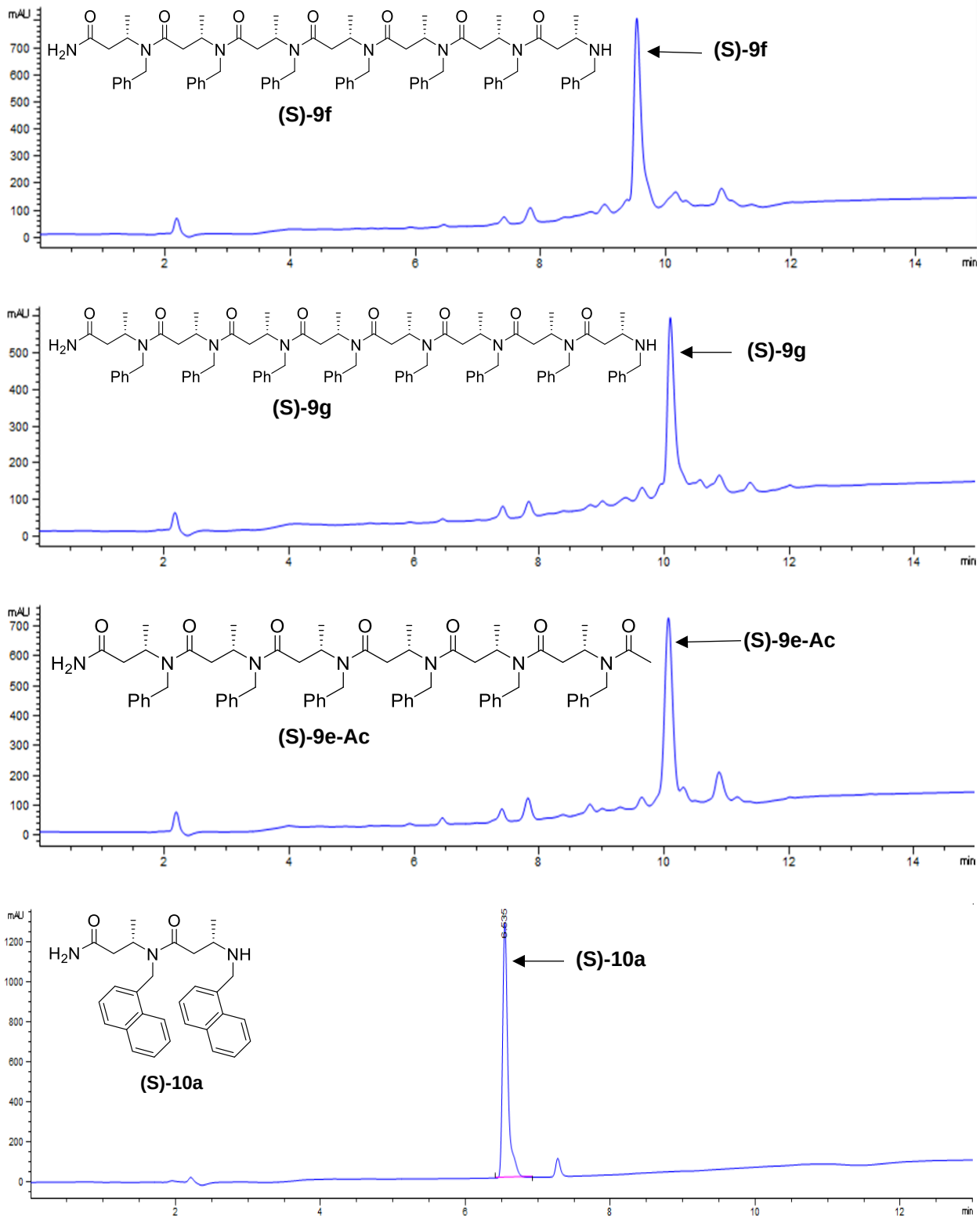

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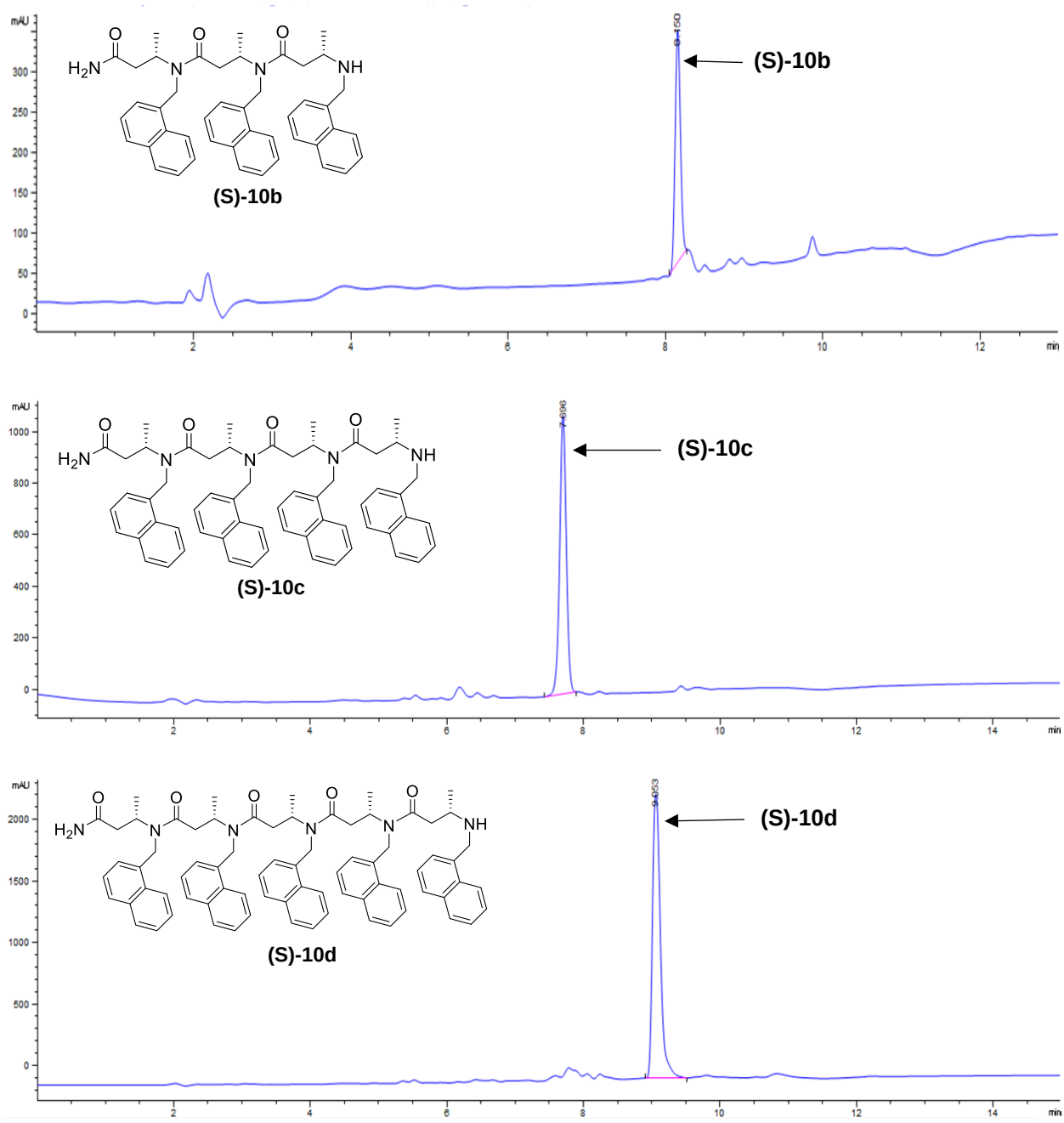

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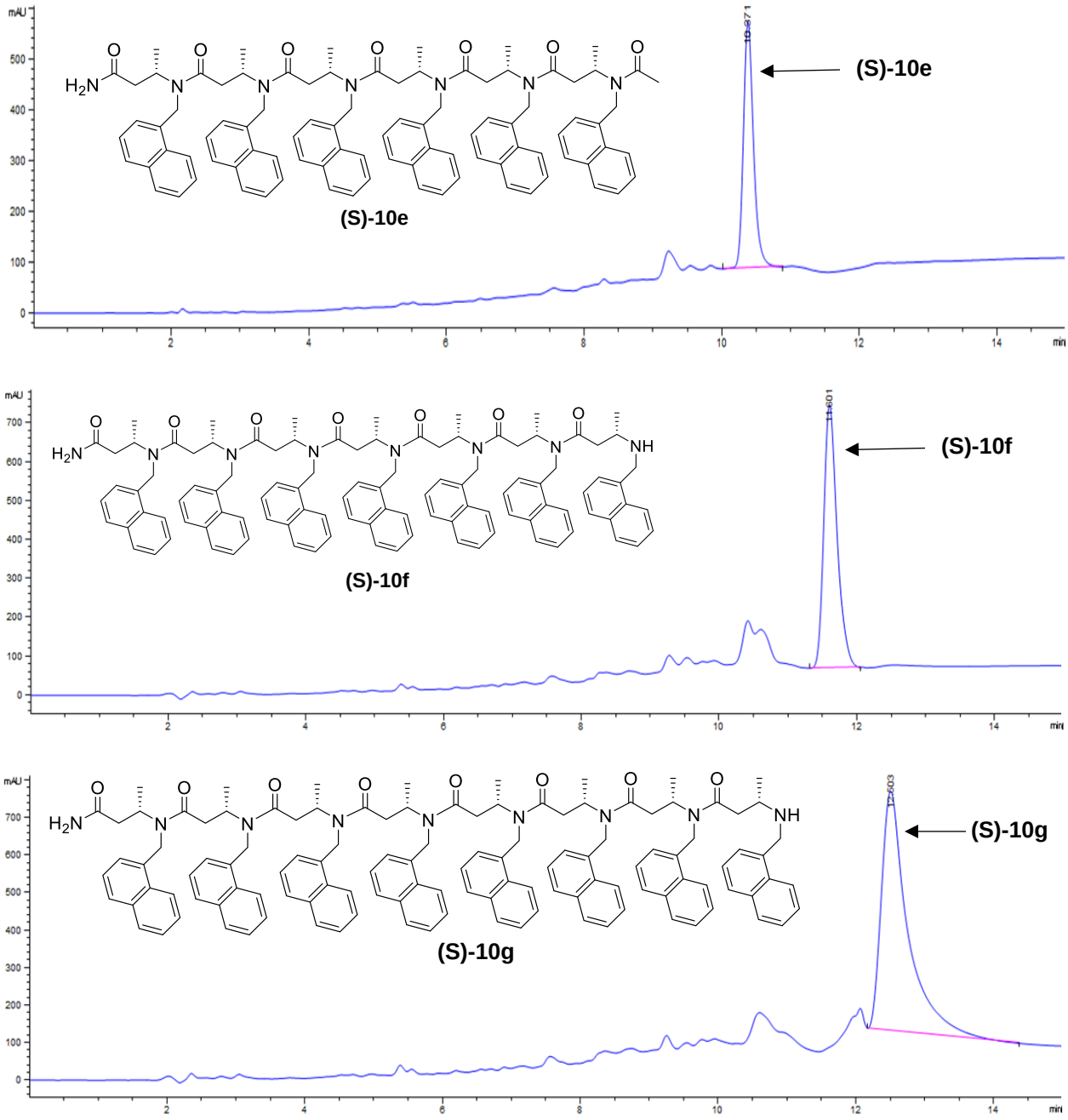
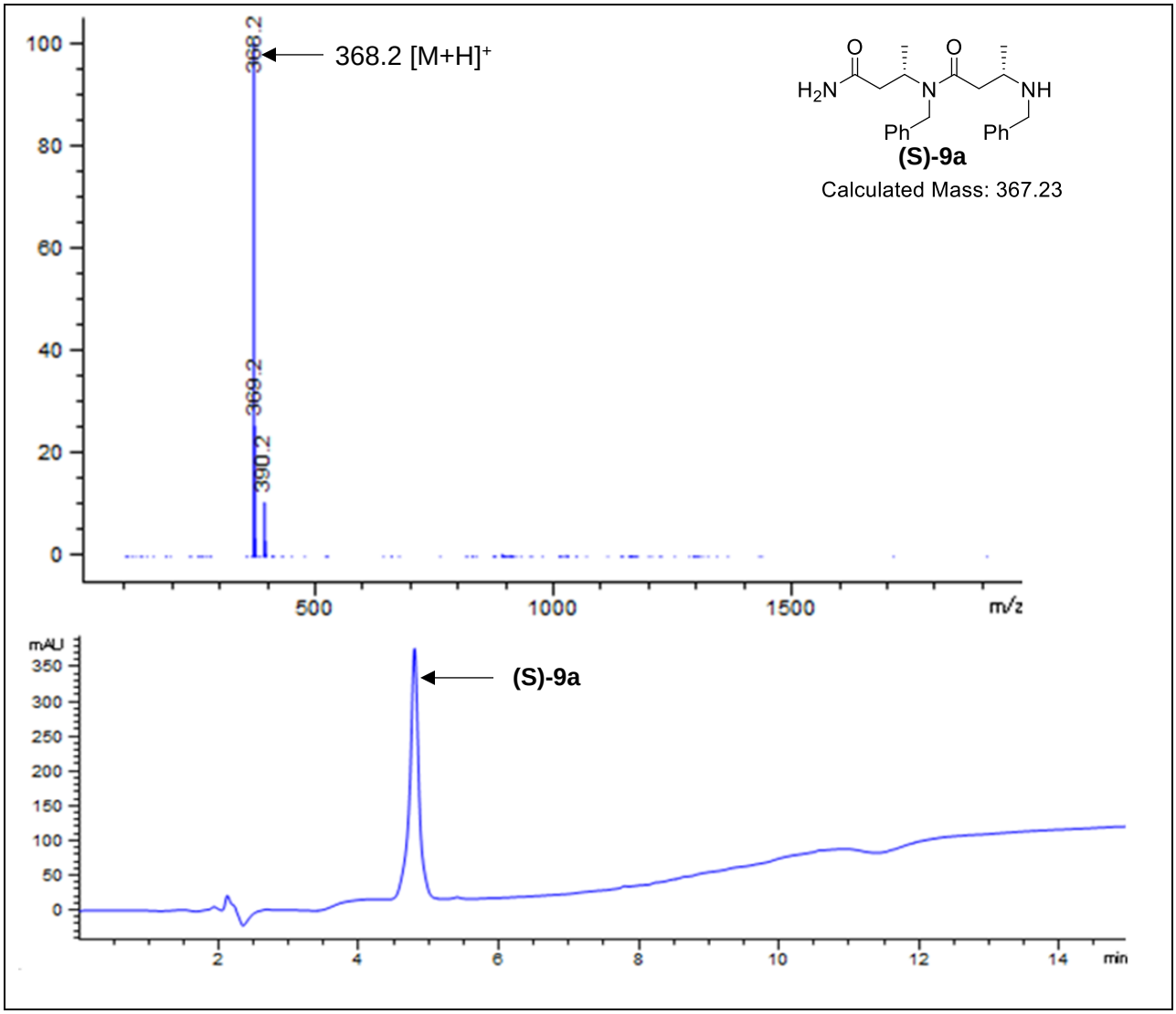


Figure S4. LC/MS spectra of purified oligomers **(S)-** and **(R)-9a-g,** and **(S)-10a-g**.



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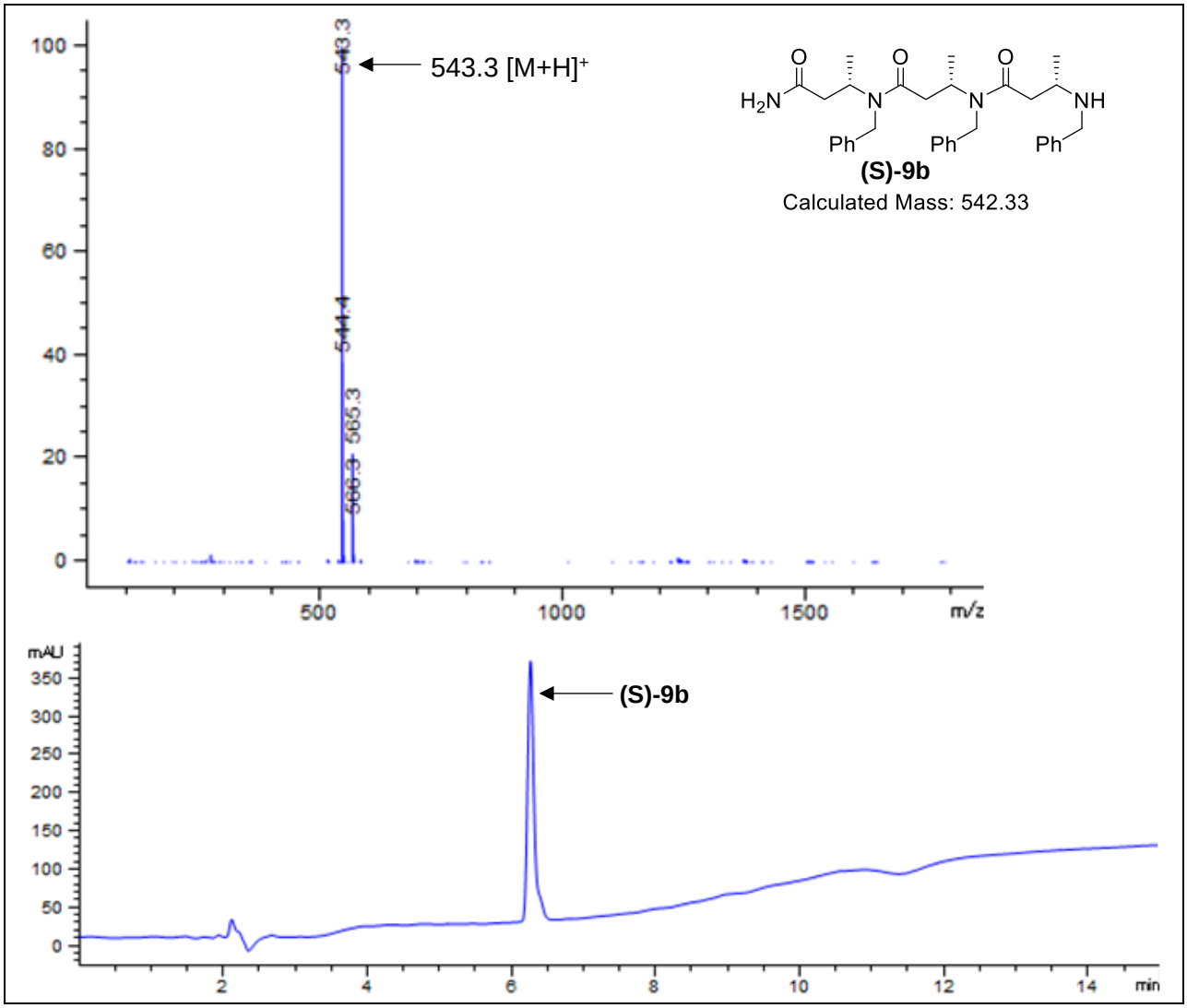

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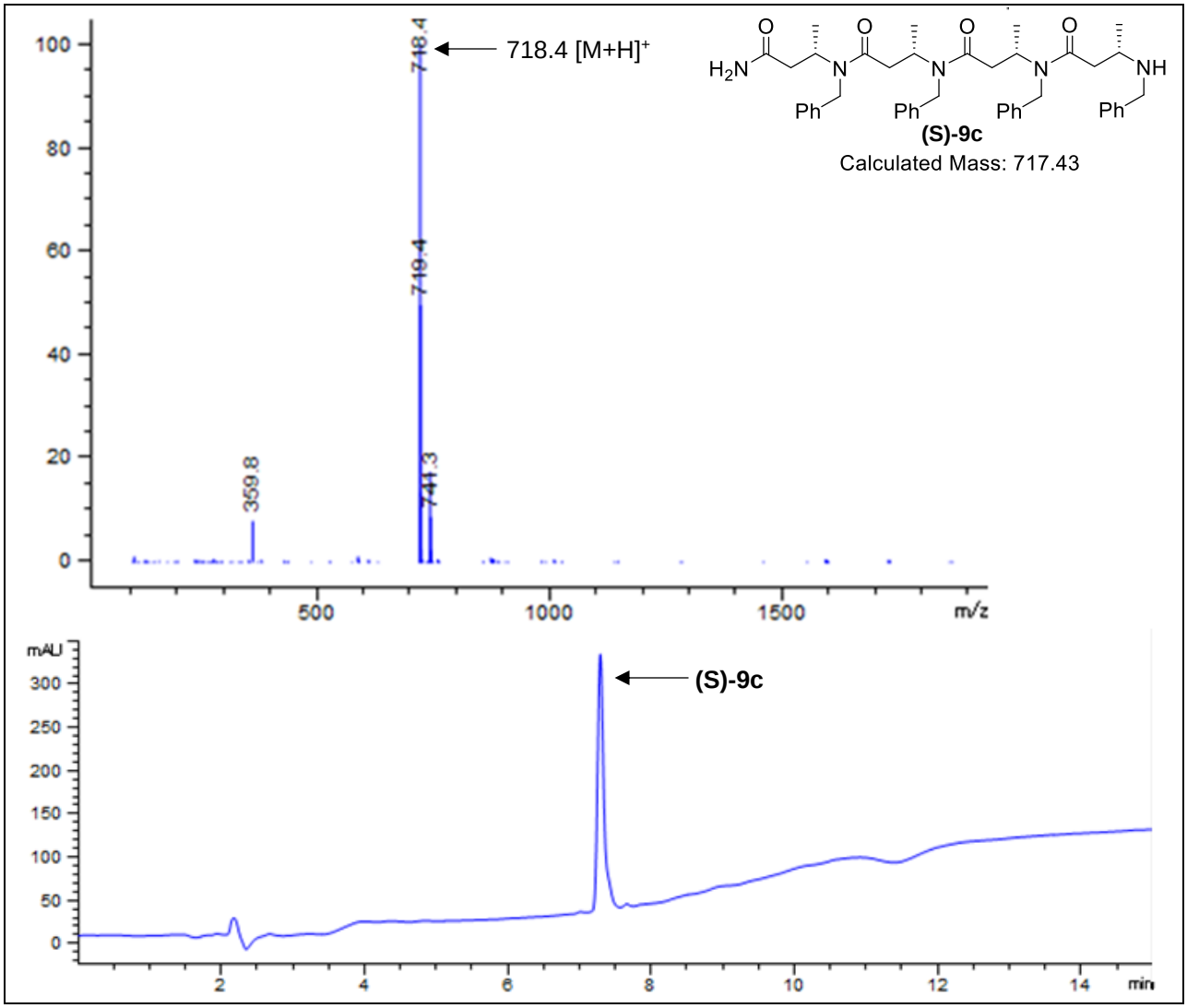

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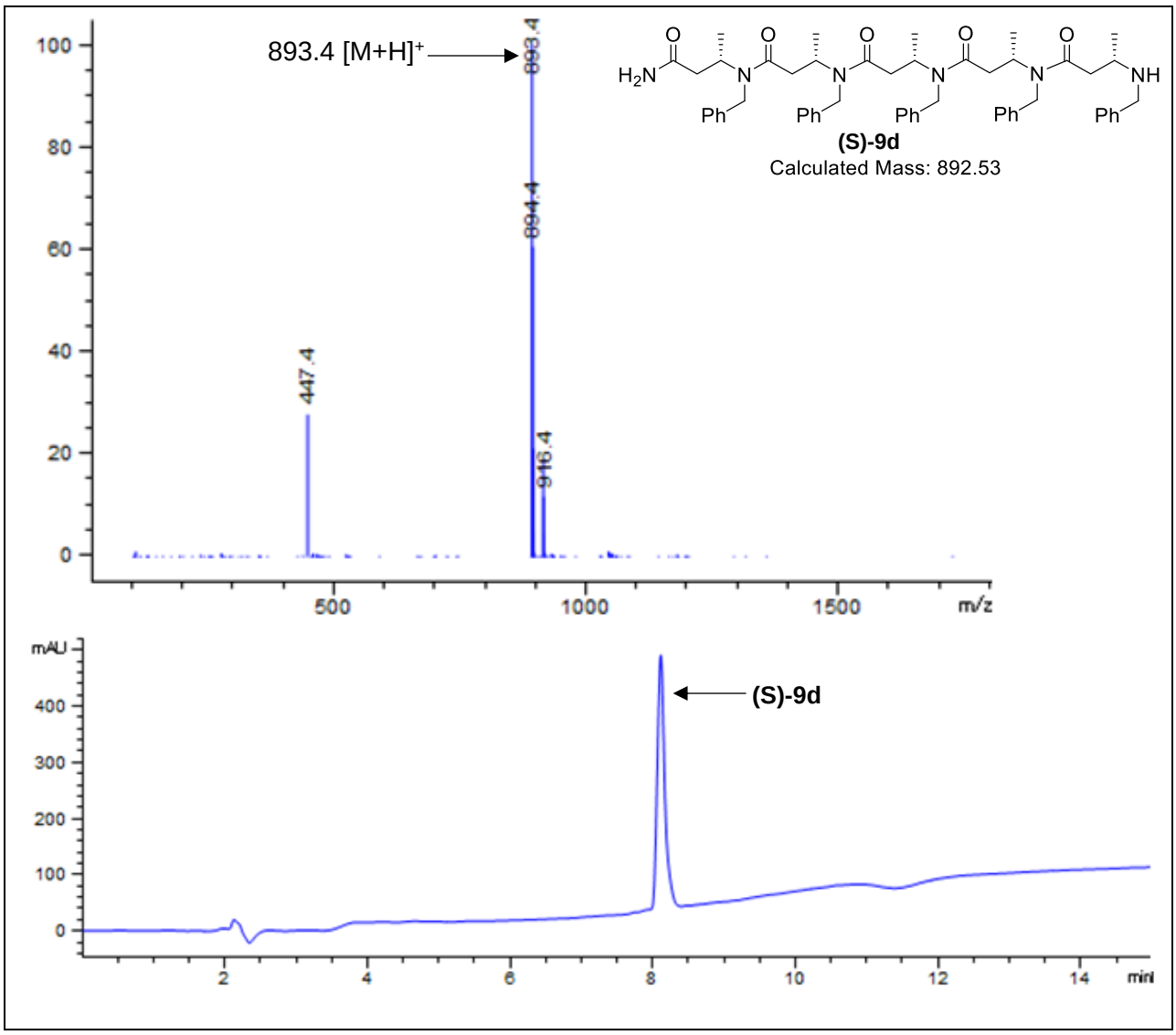

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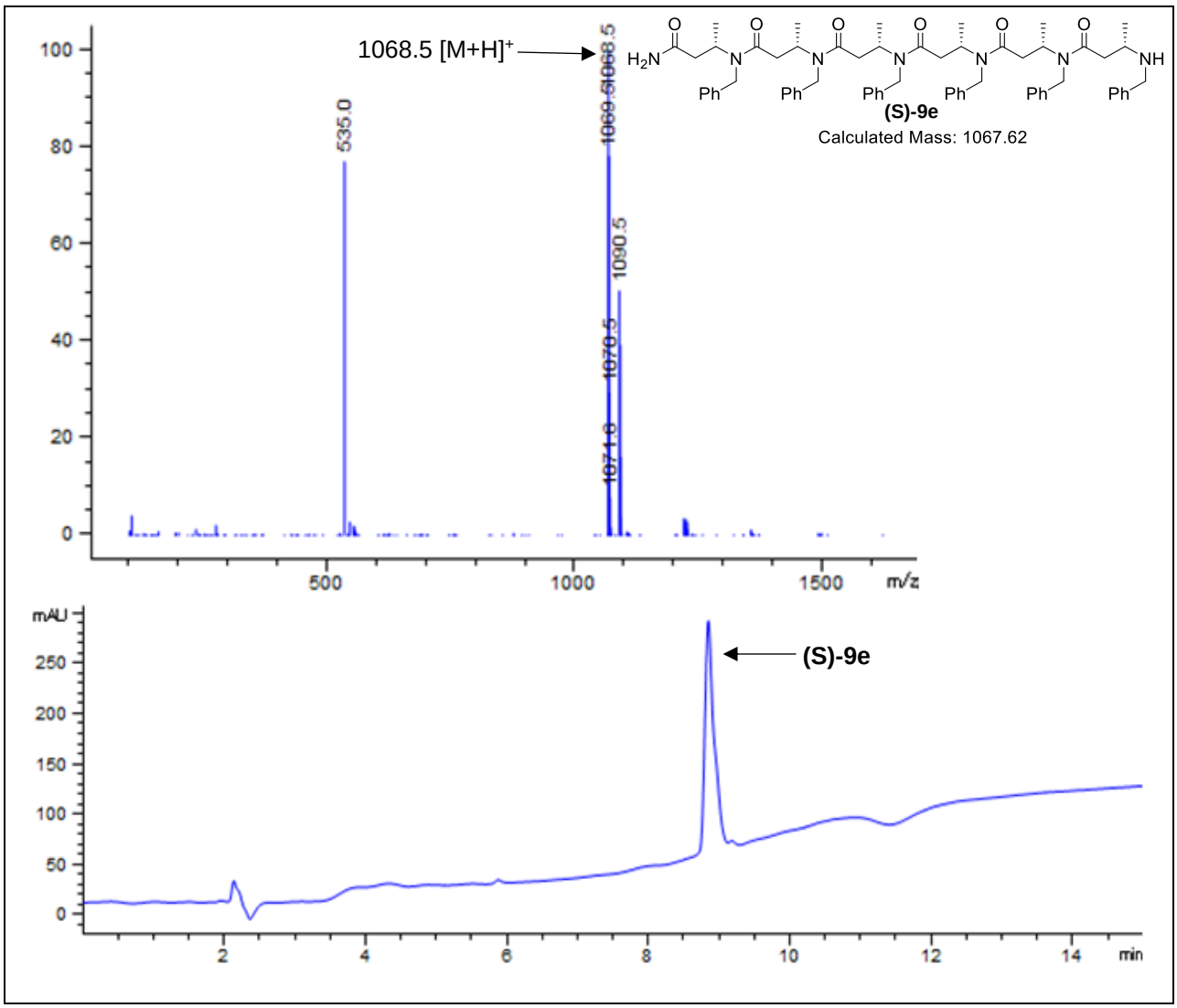

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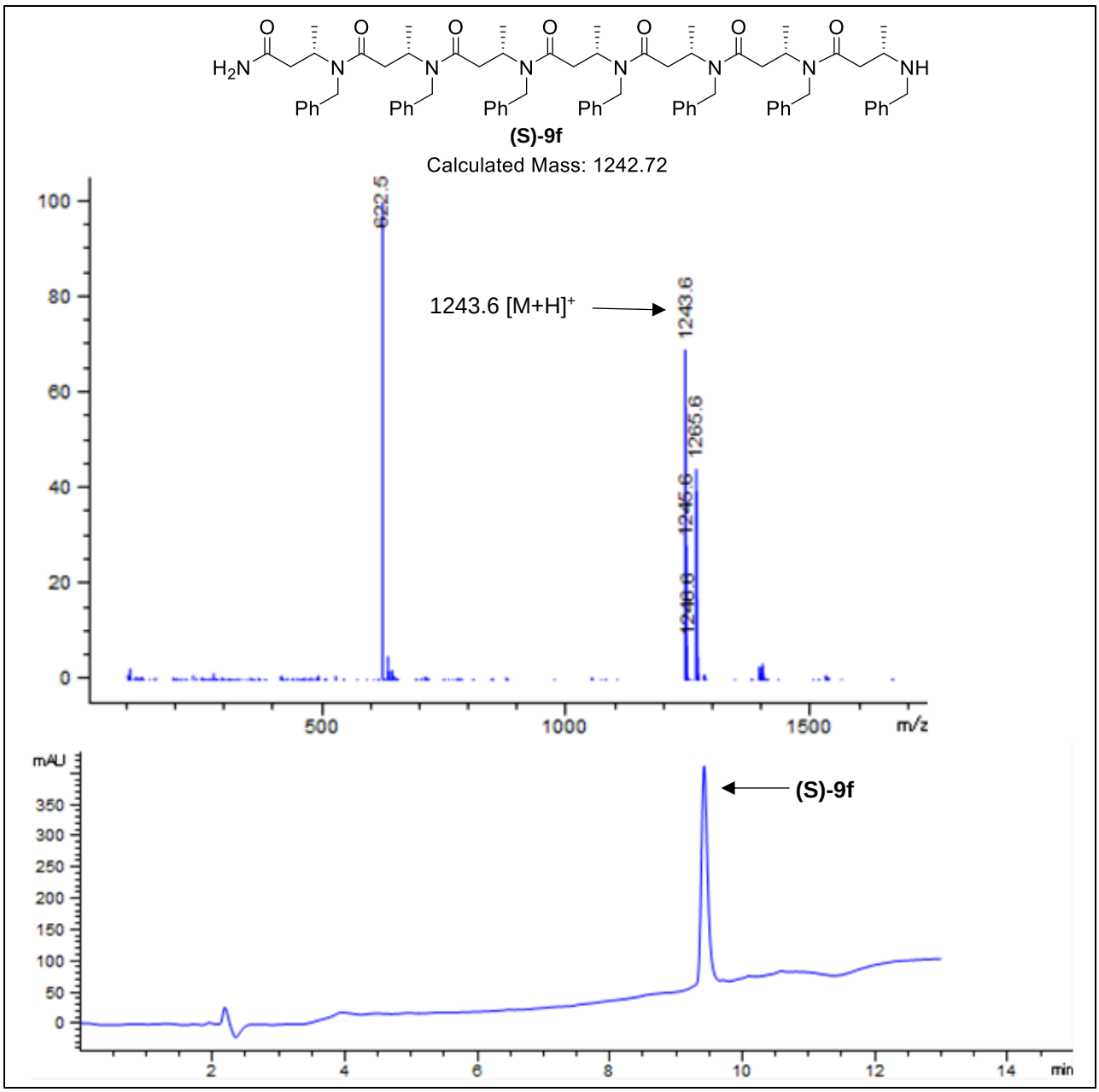

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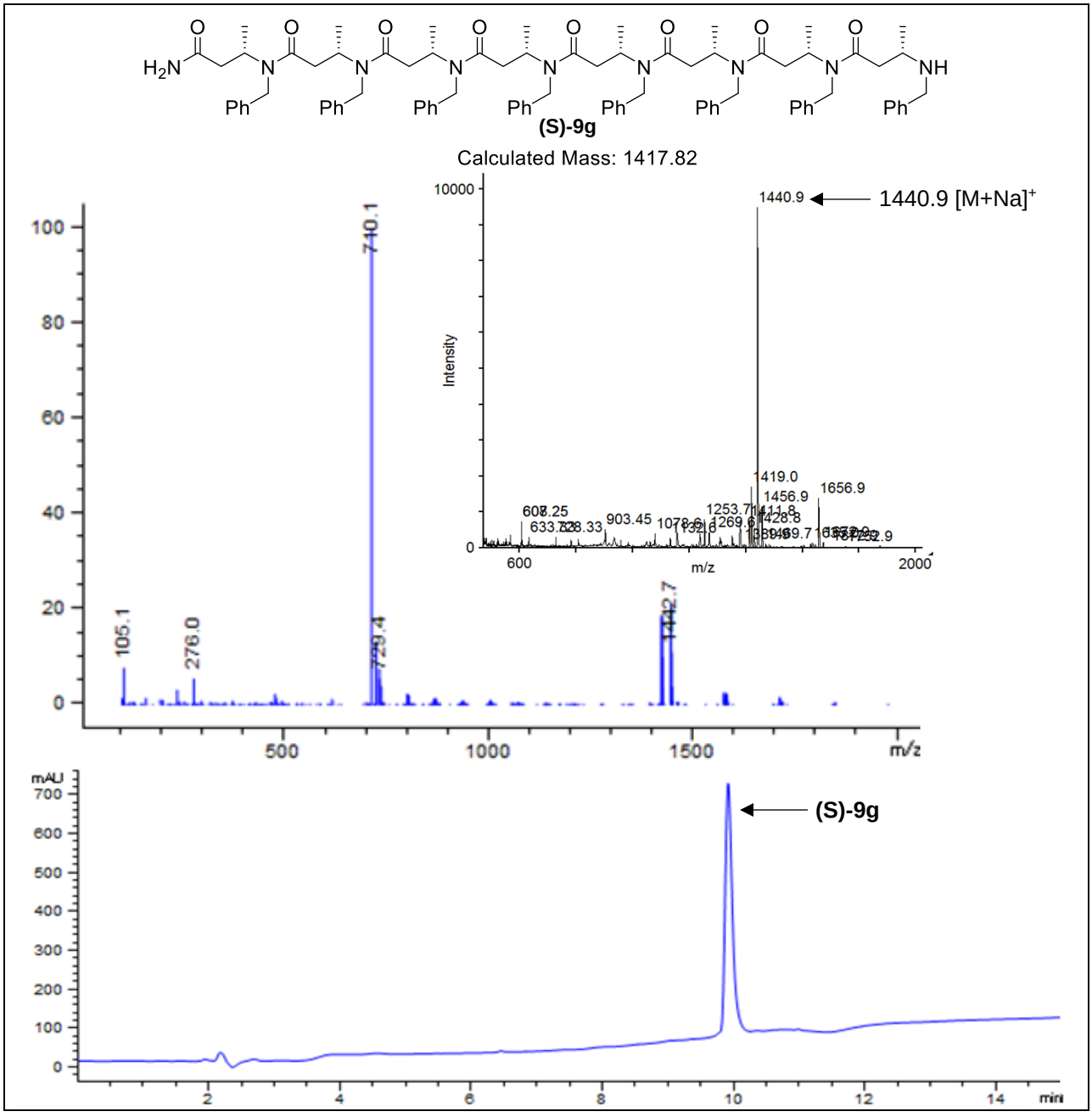

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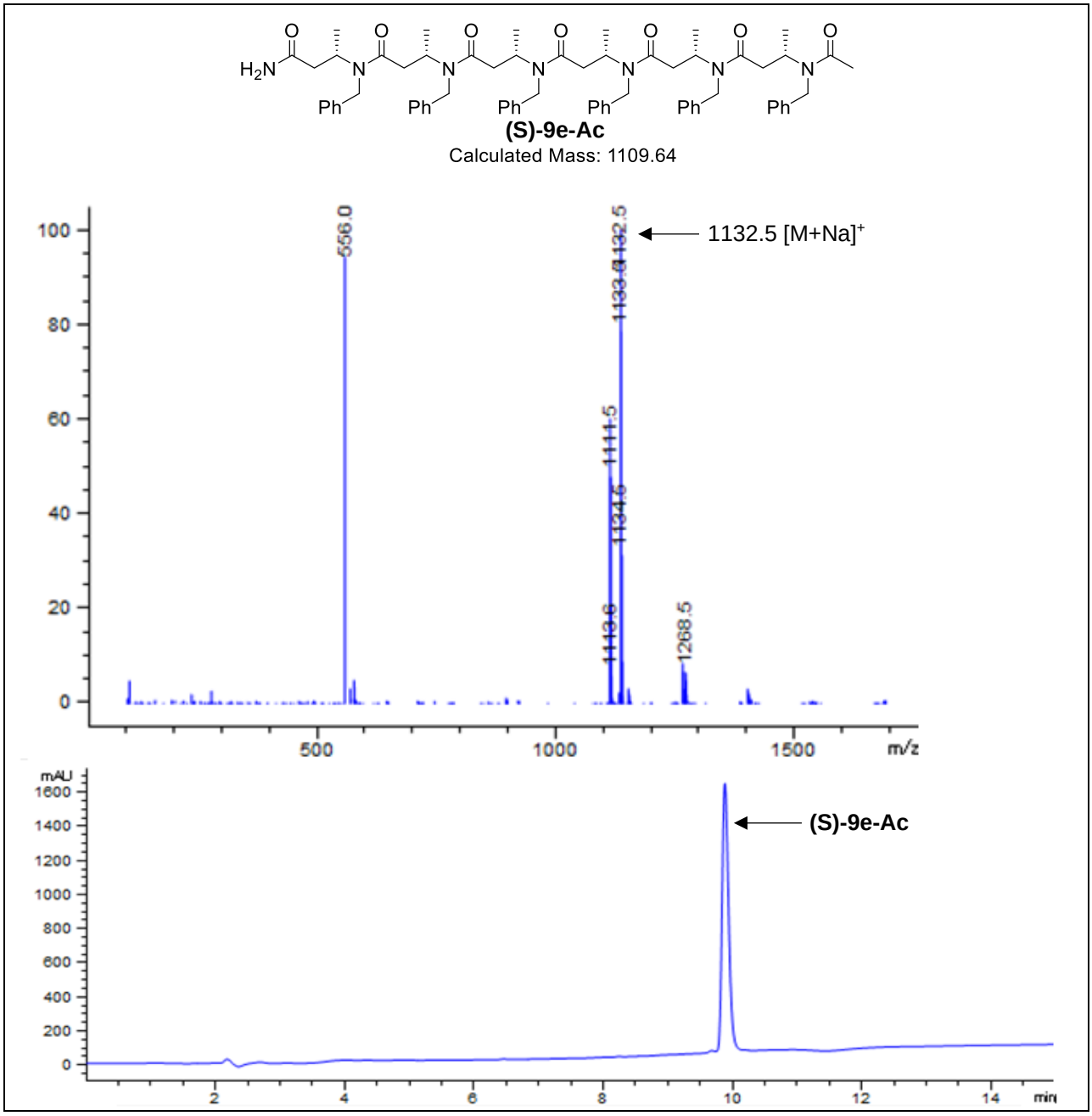

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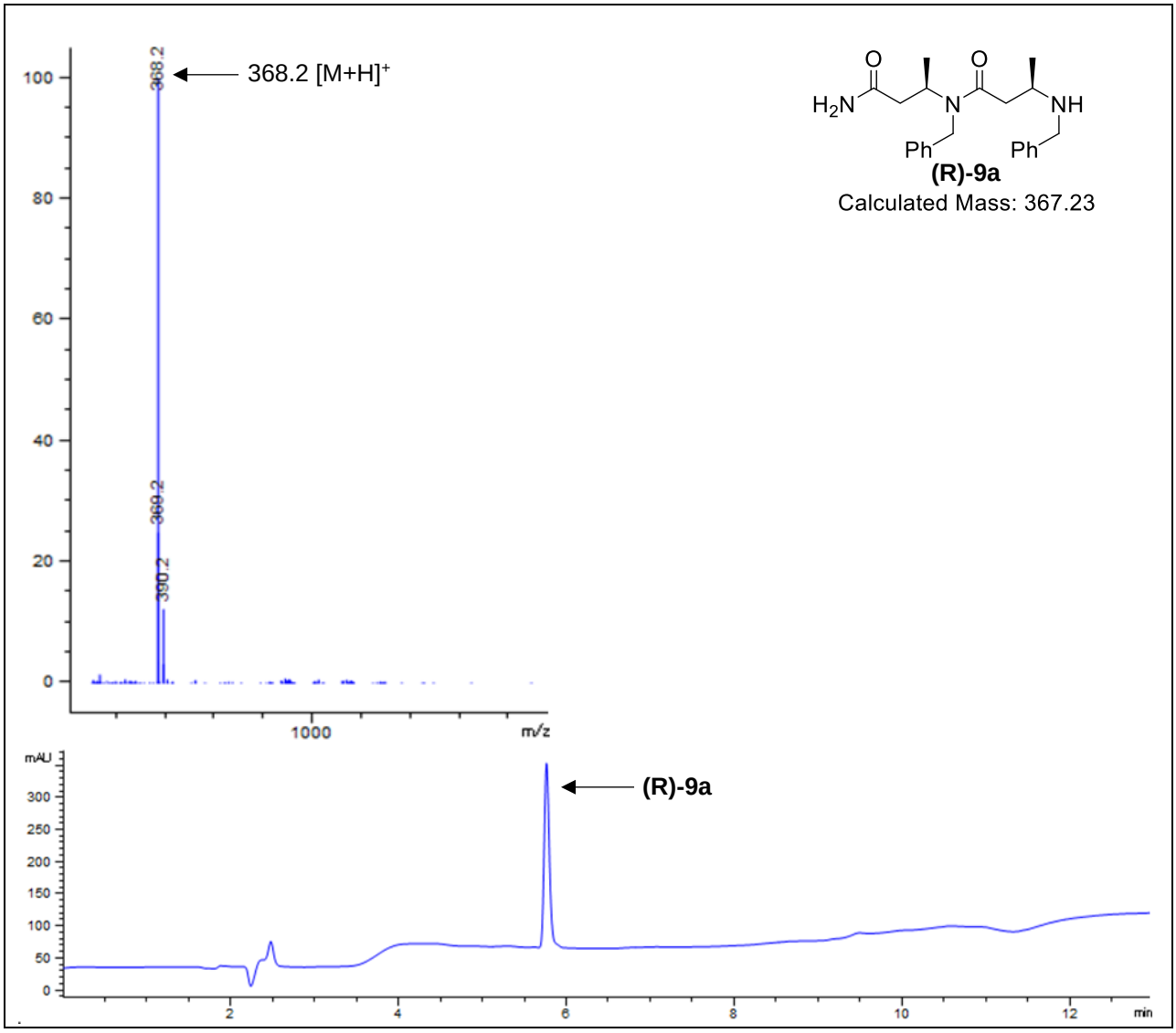

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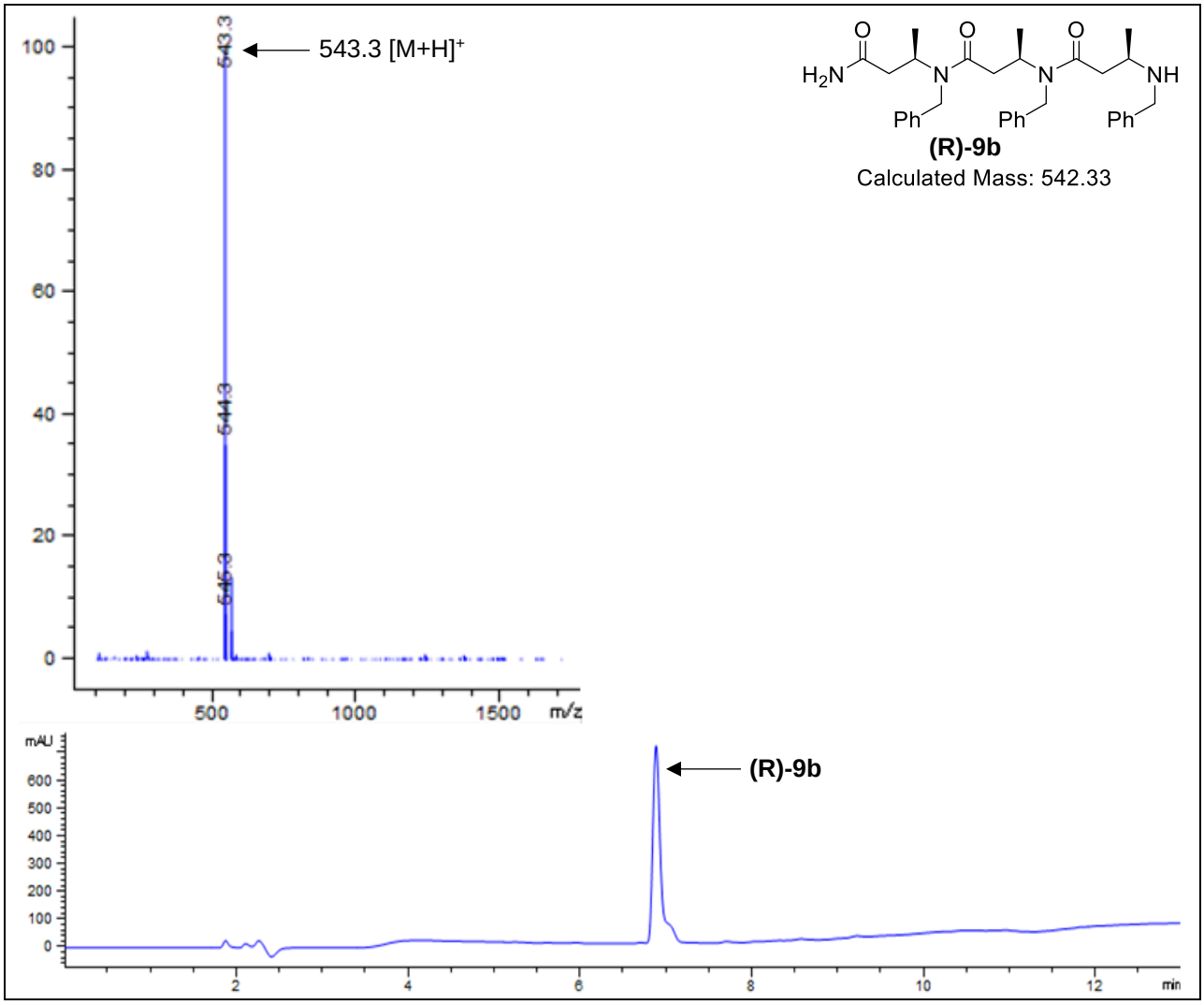

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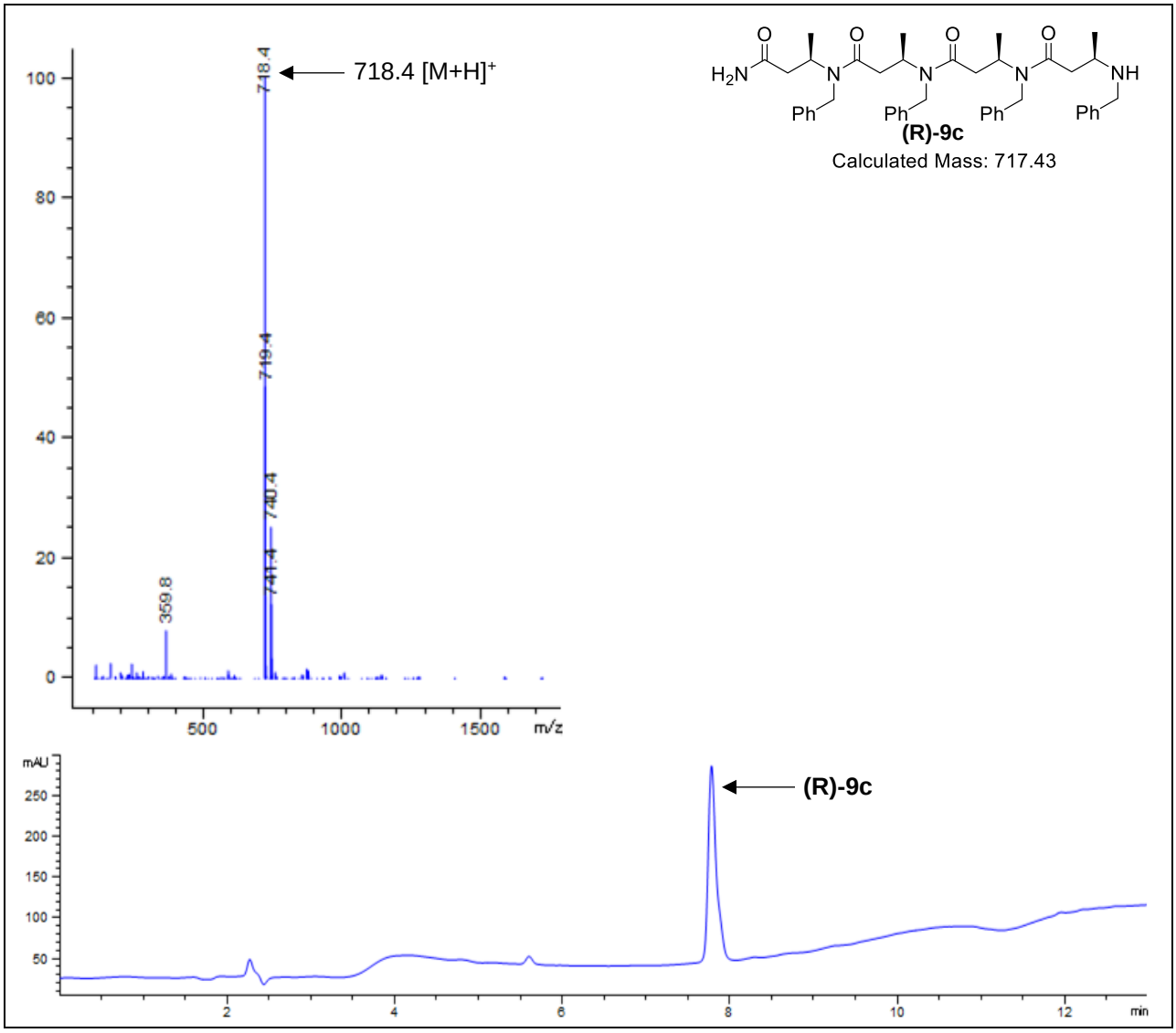

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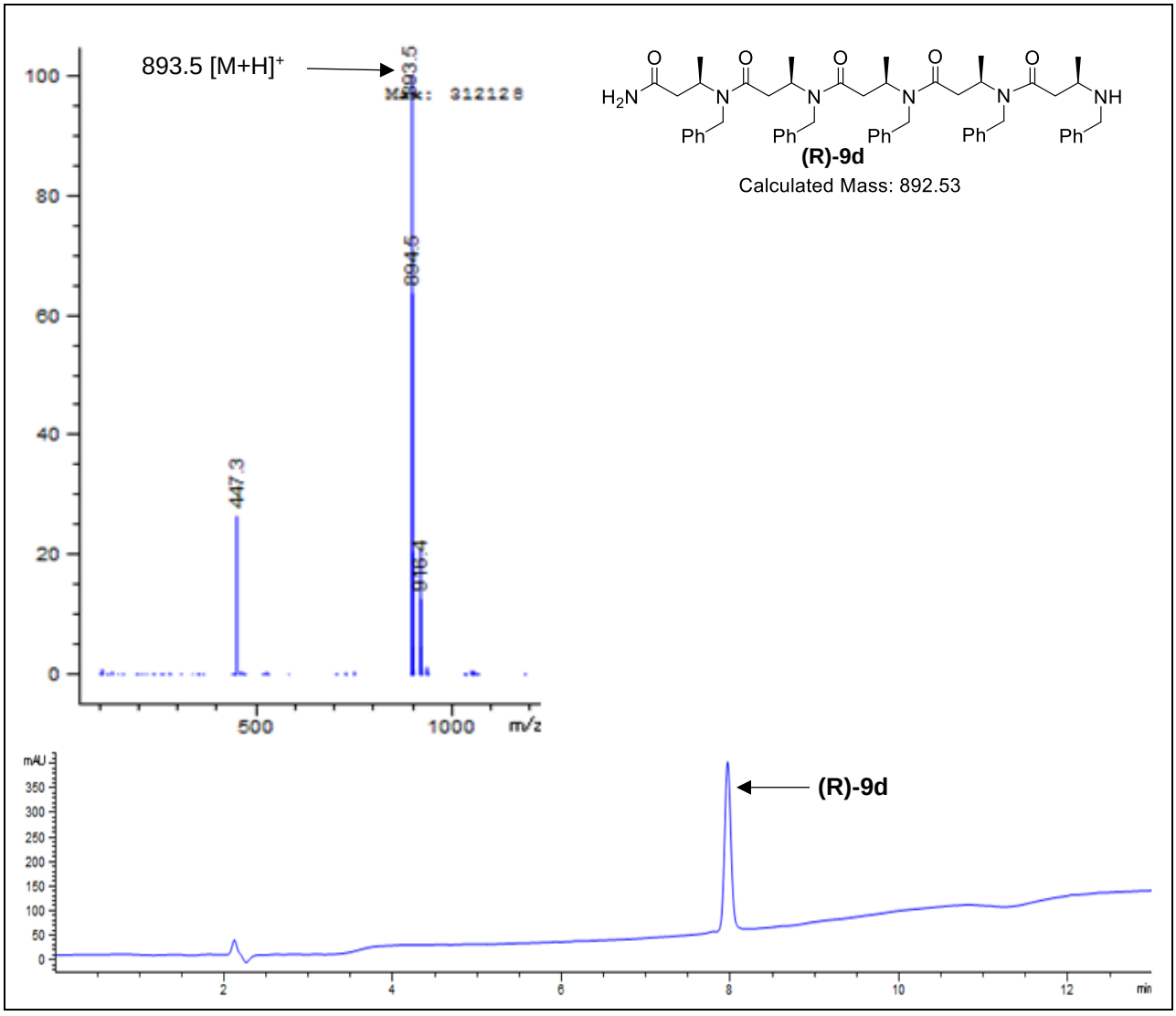

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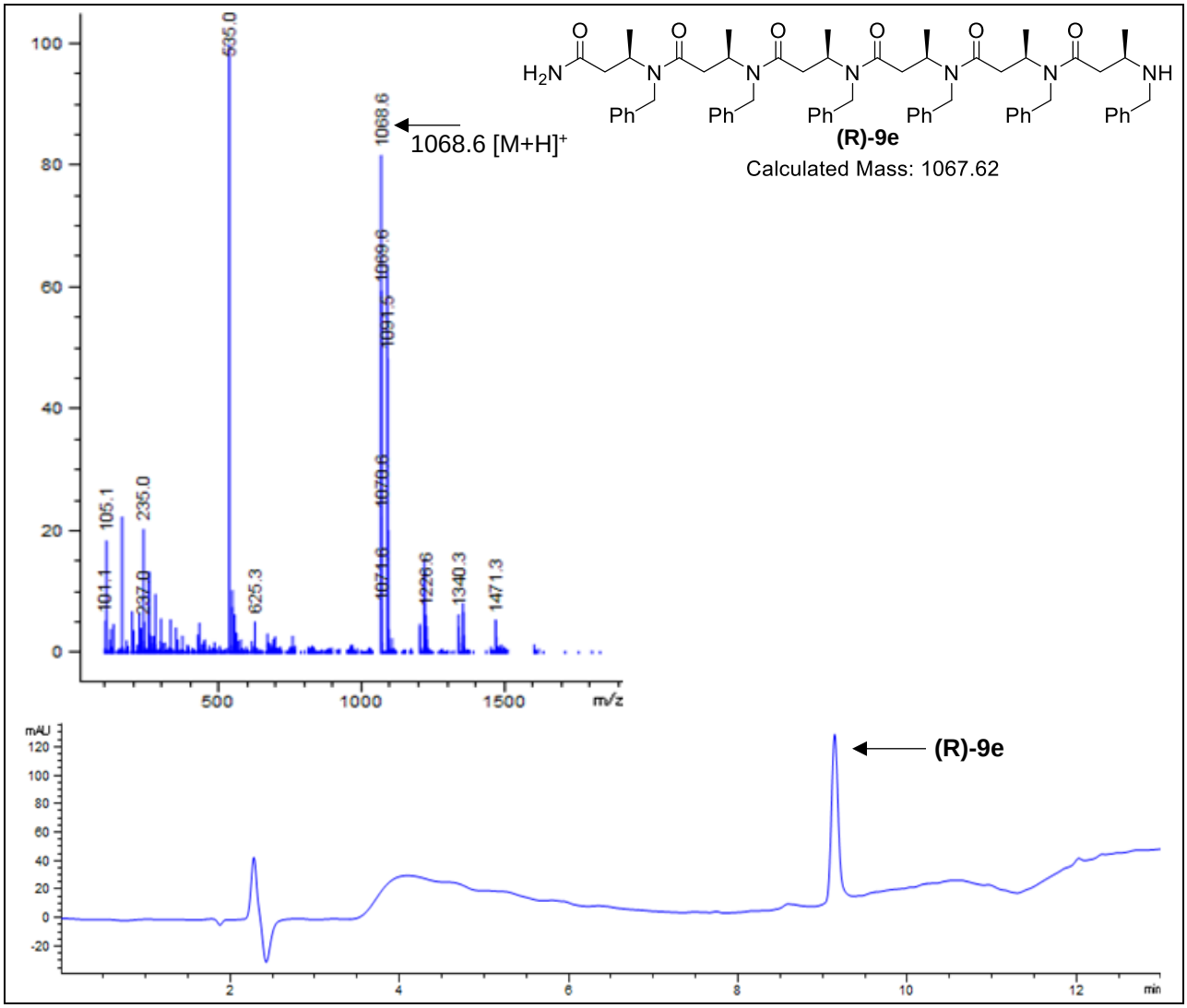

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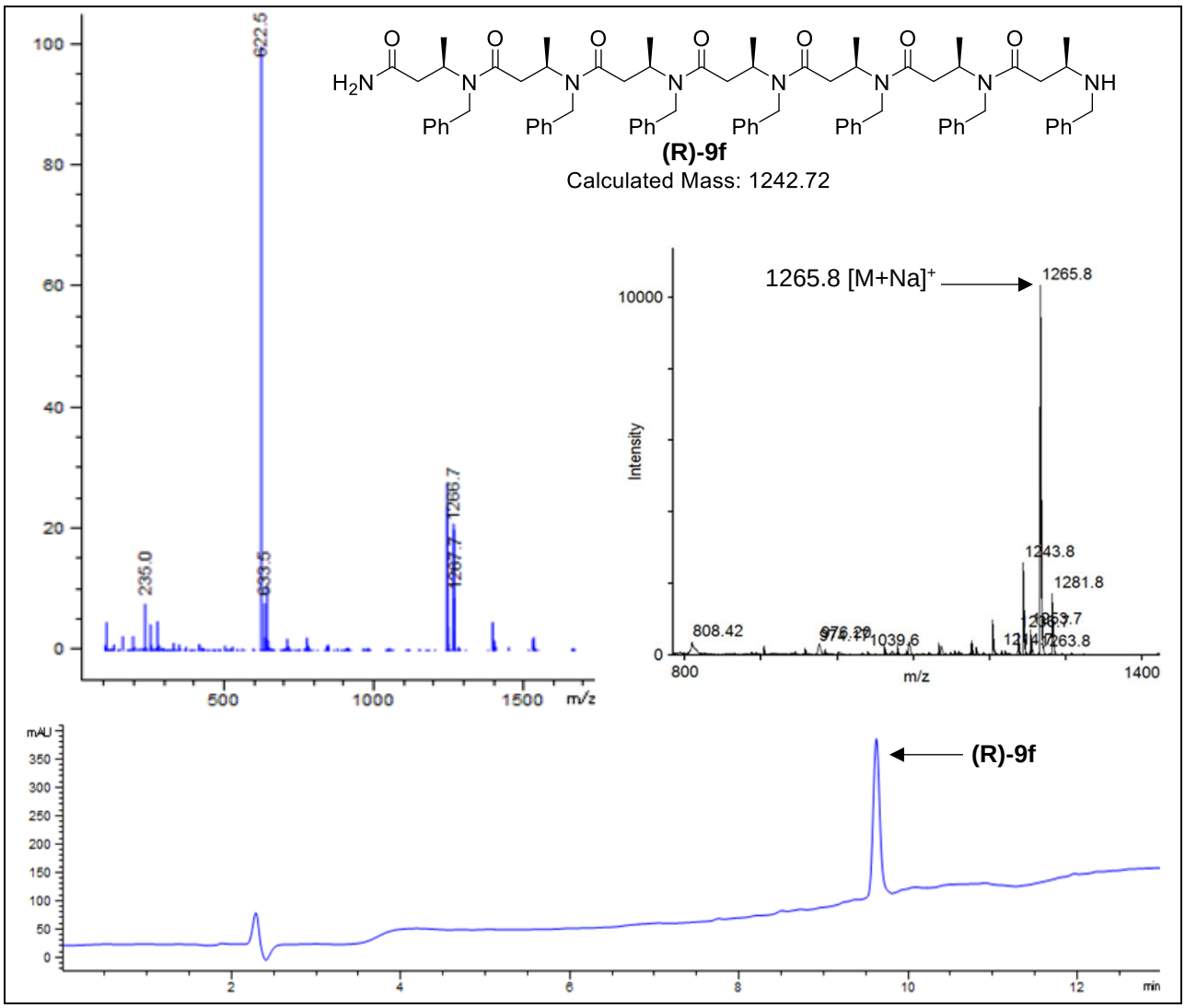

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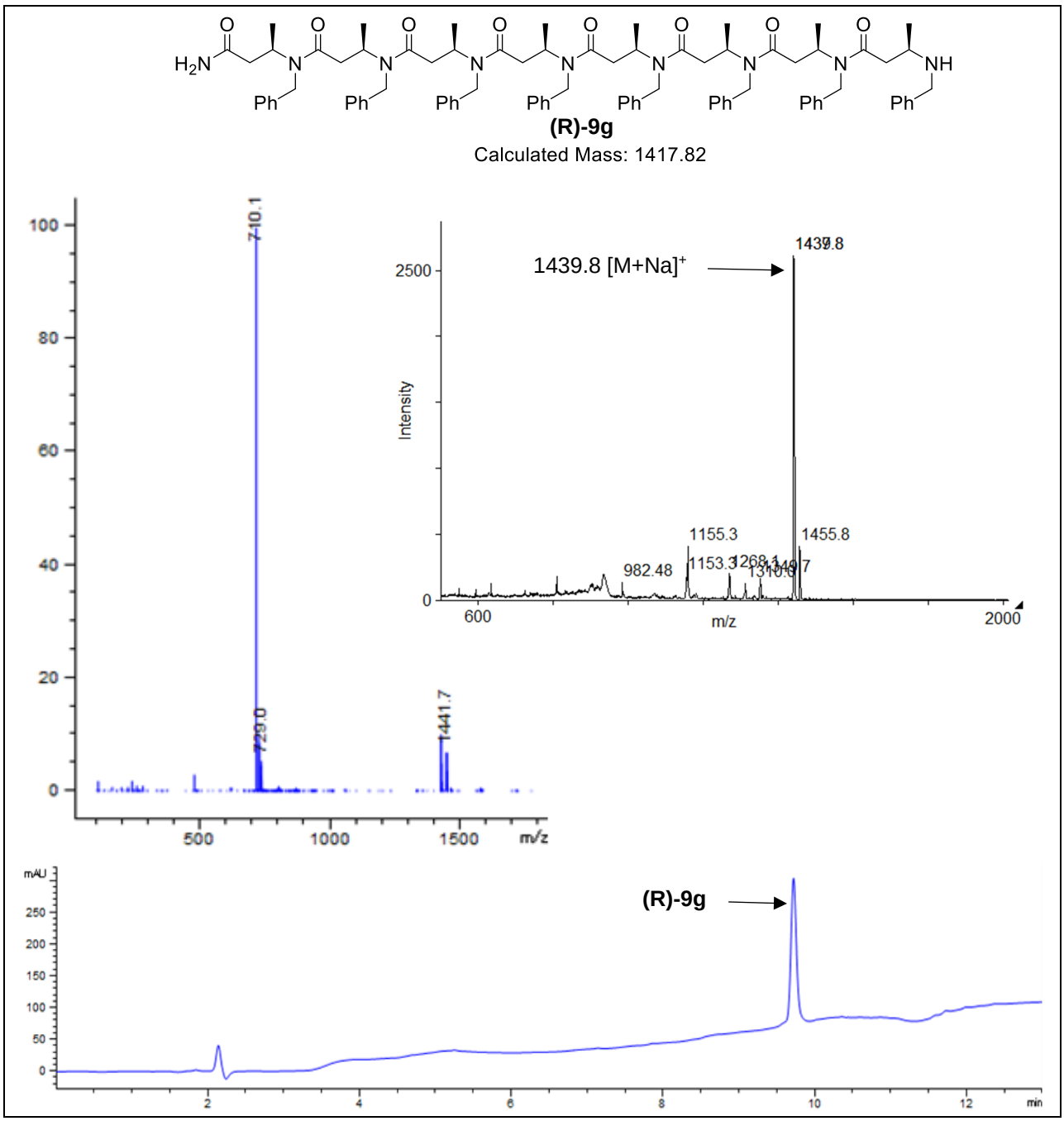

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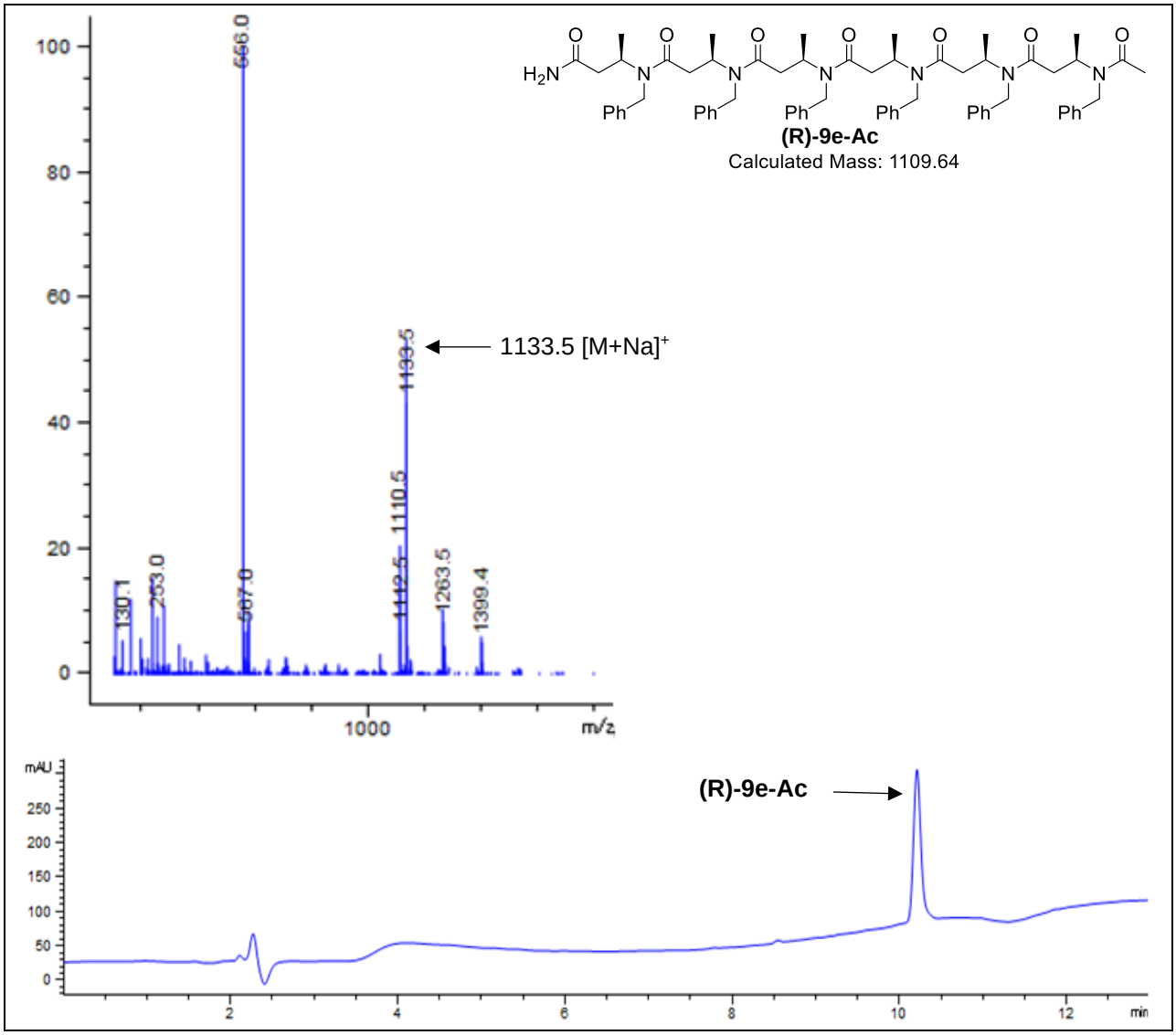

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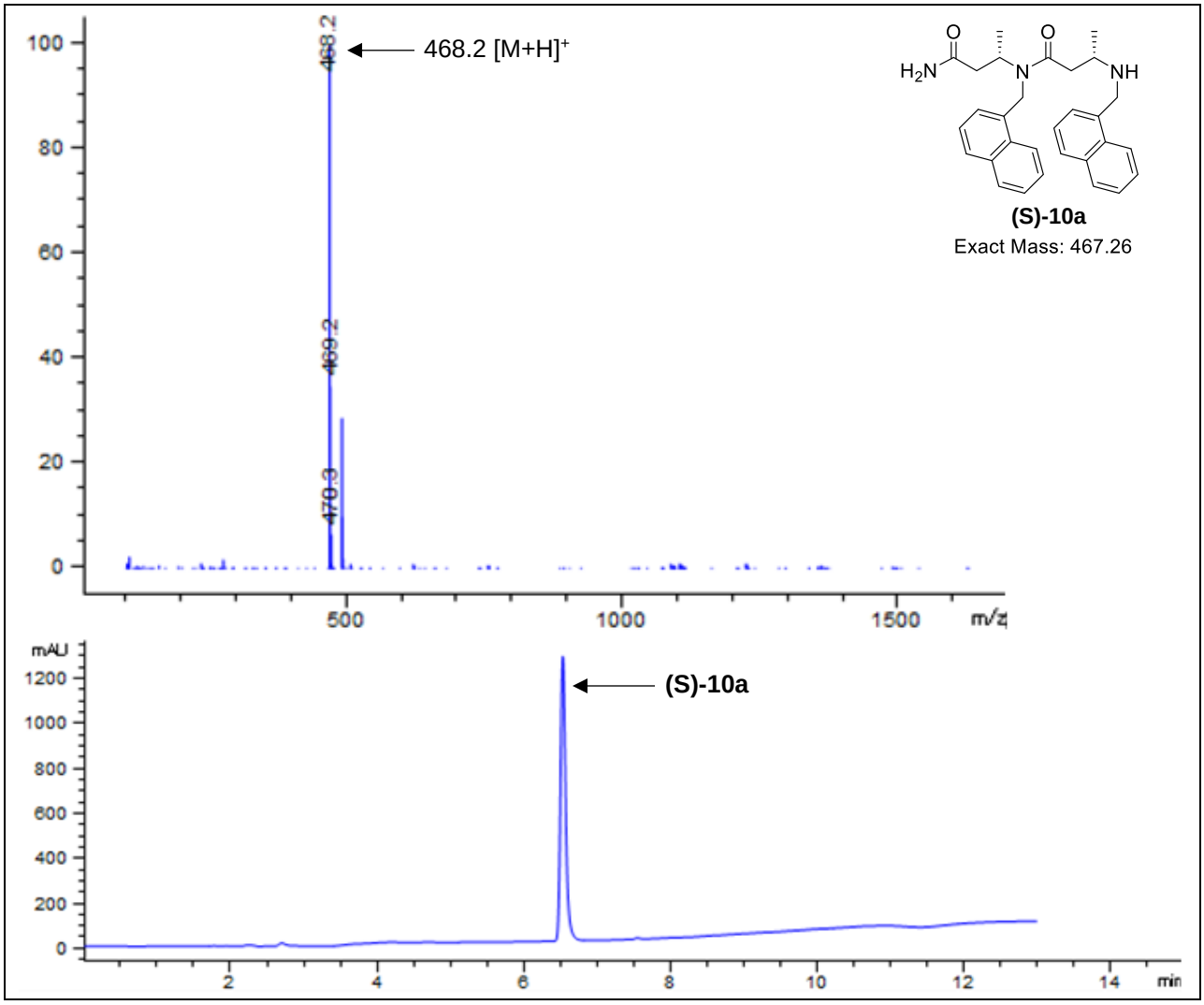

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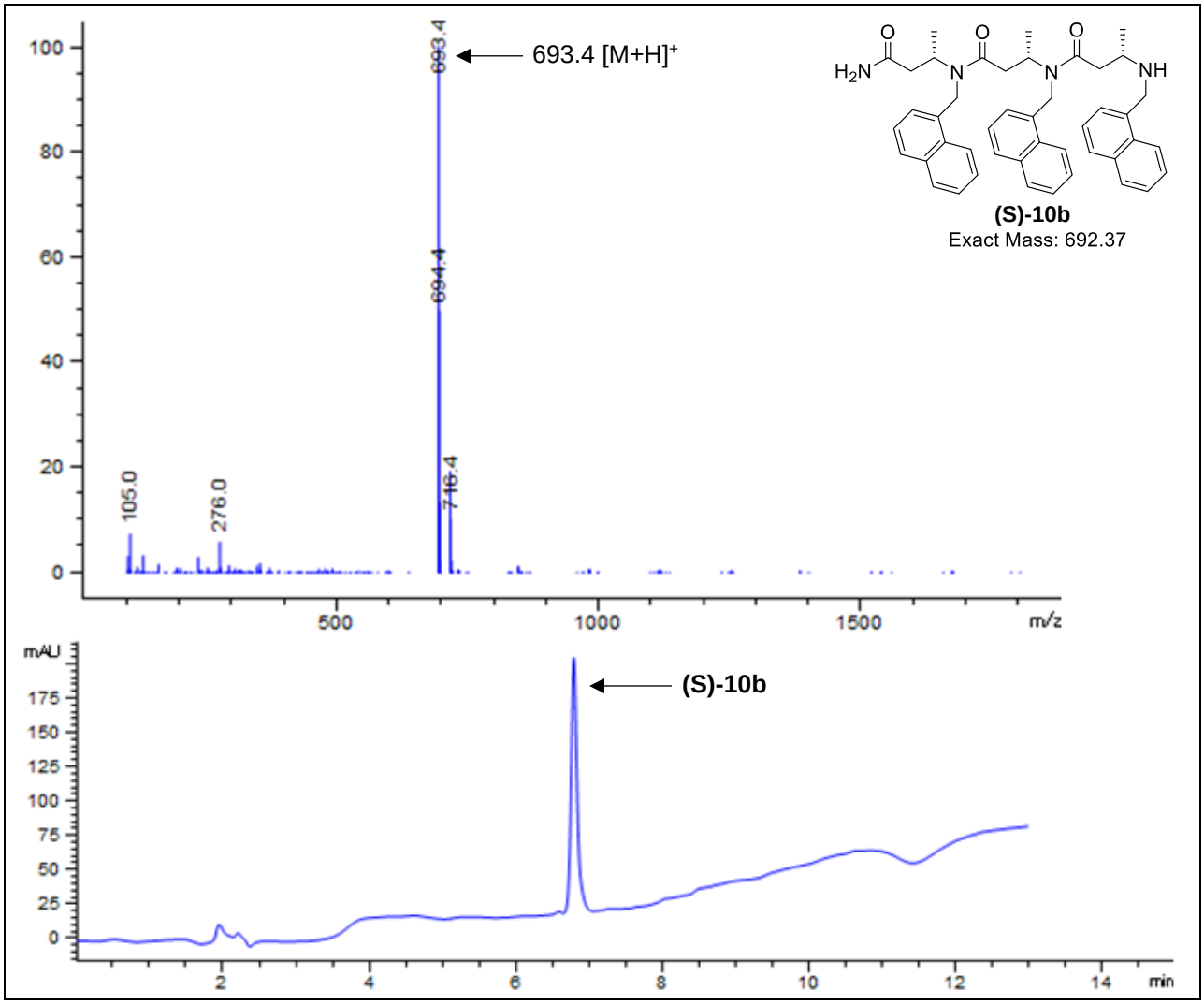
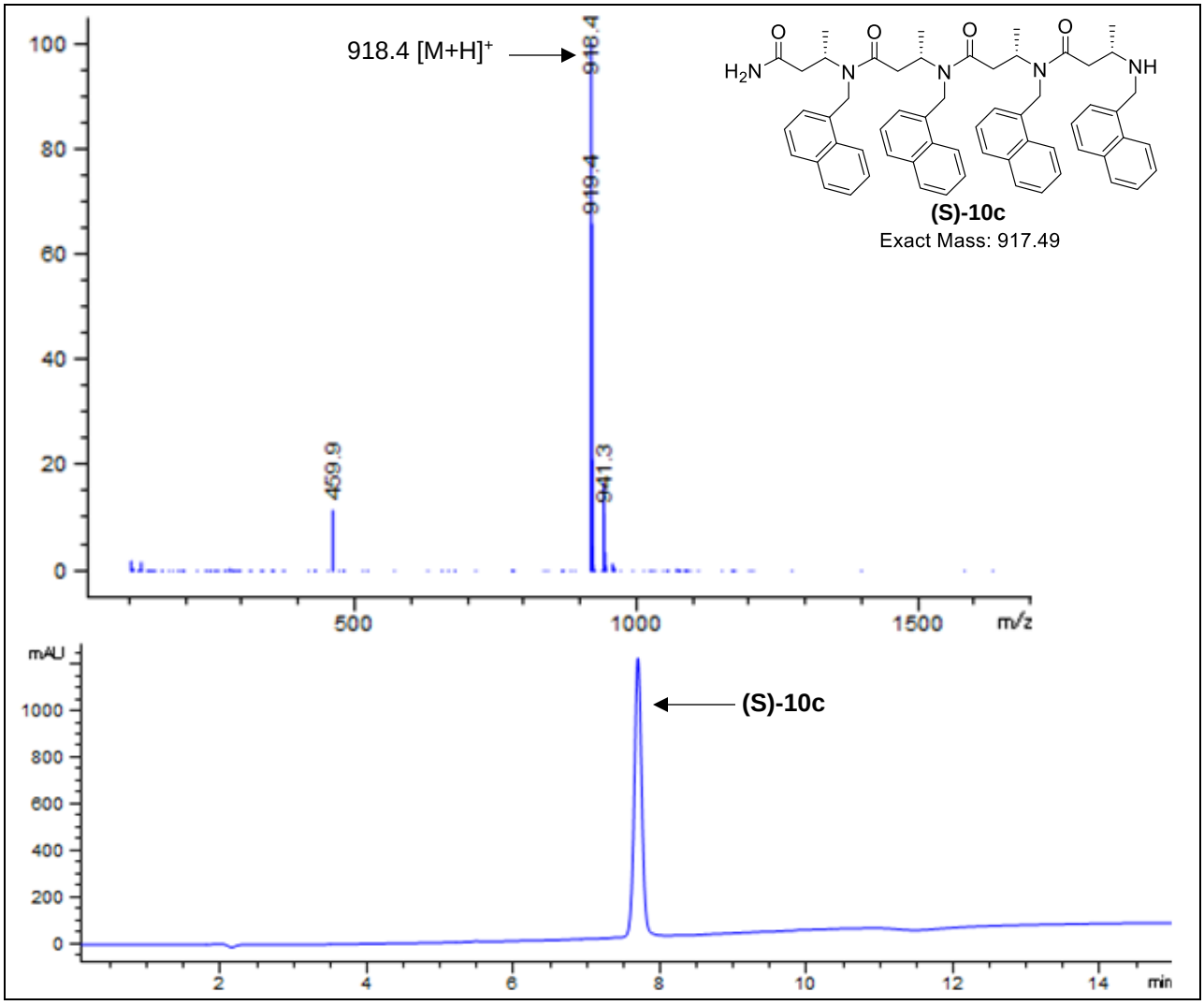


Figure S4, continued



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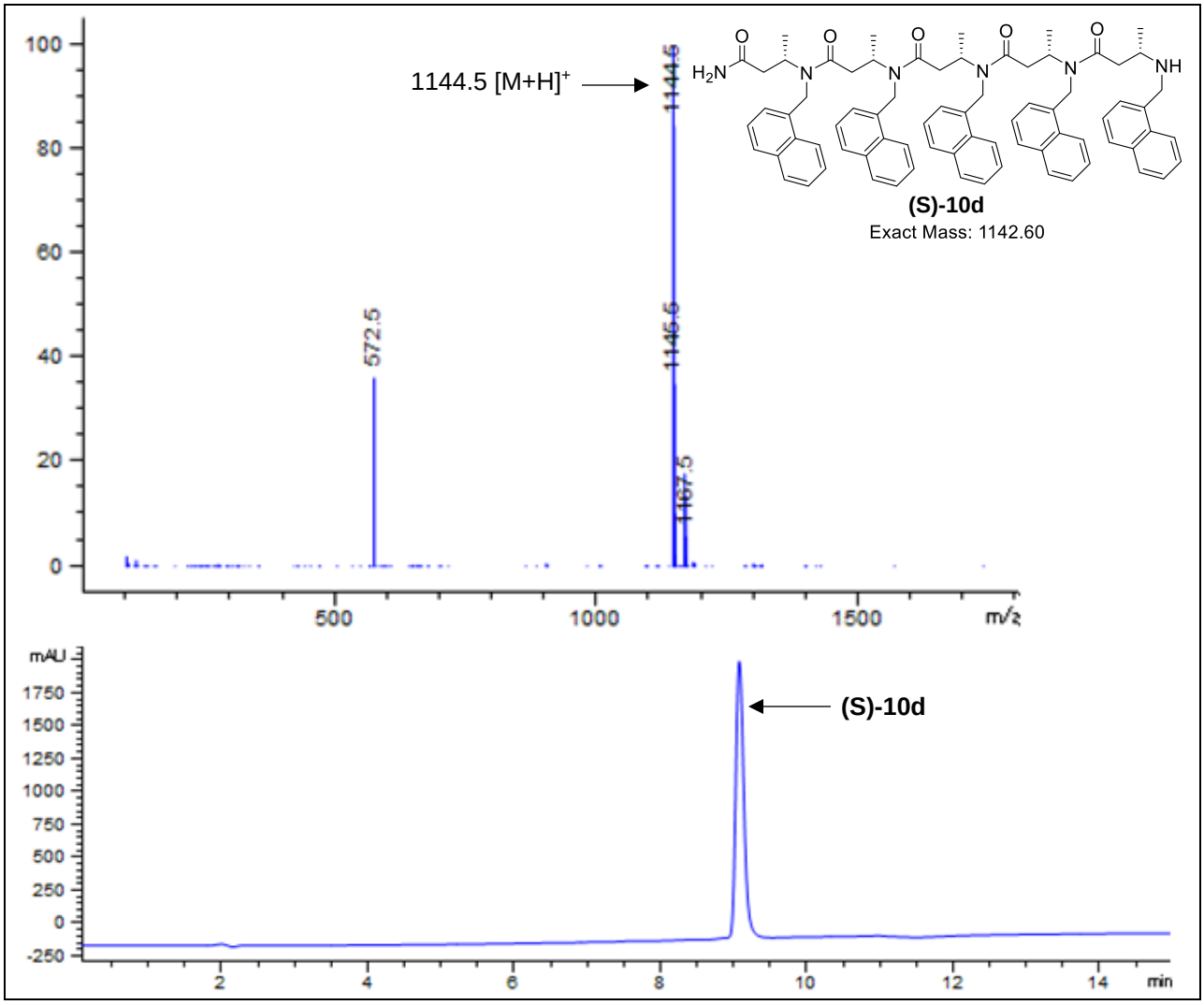


Figure S4, continued

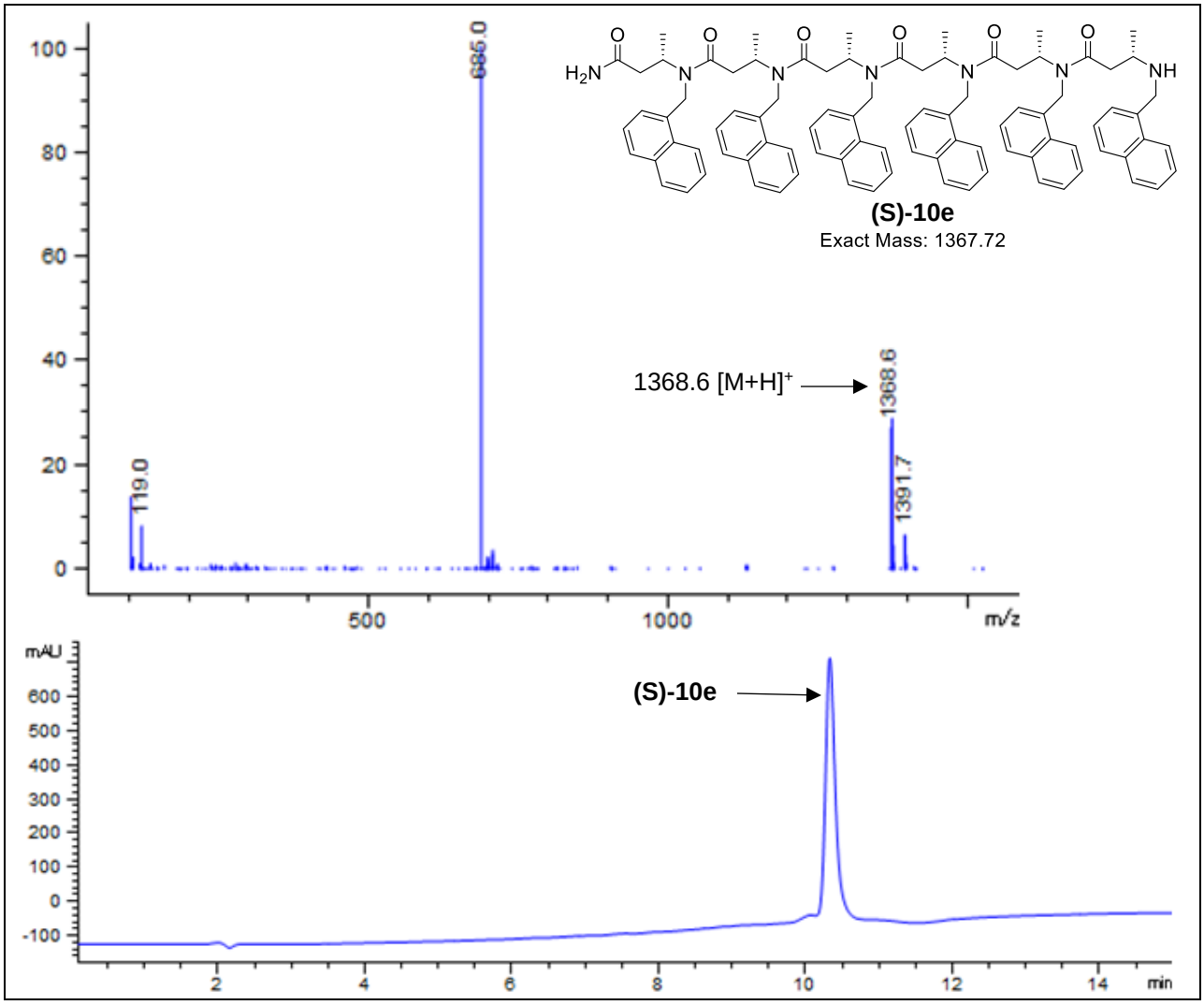
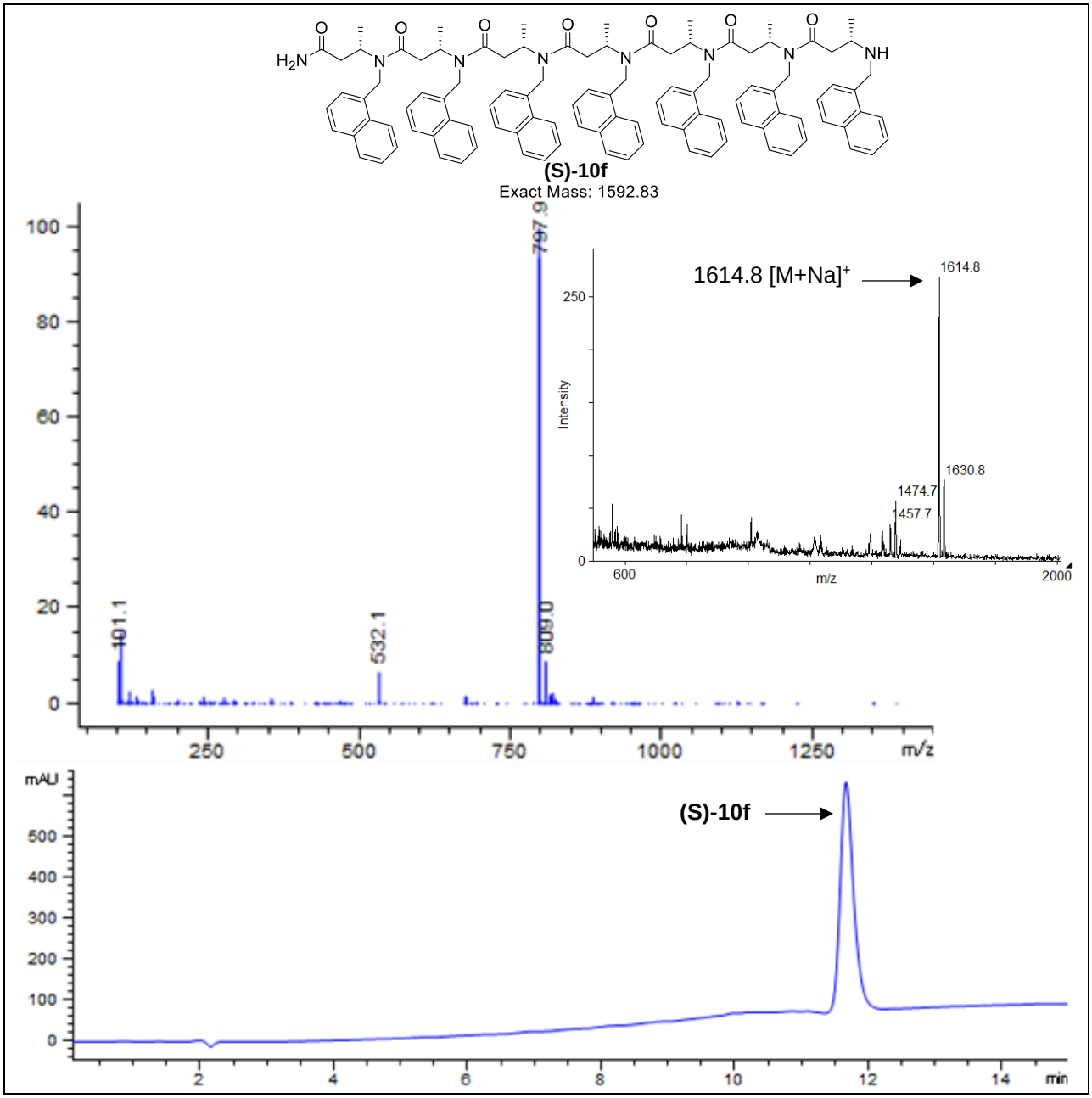


Figure S4, continued



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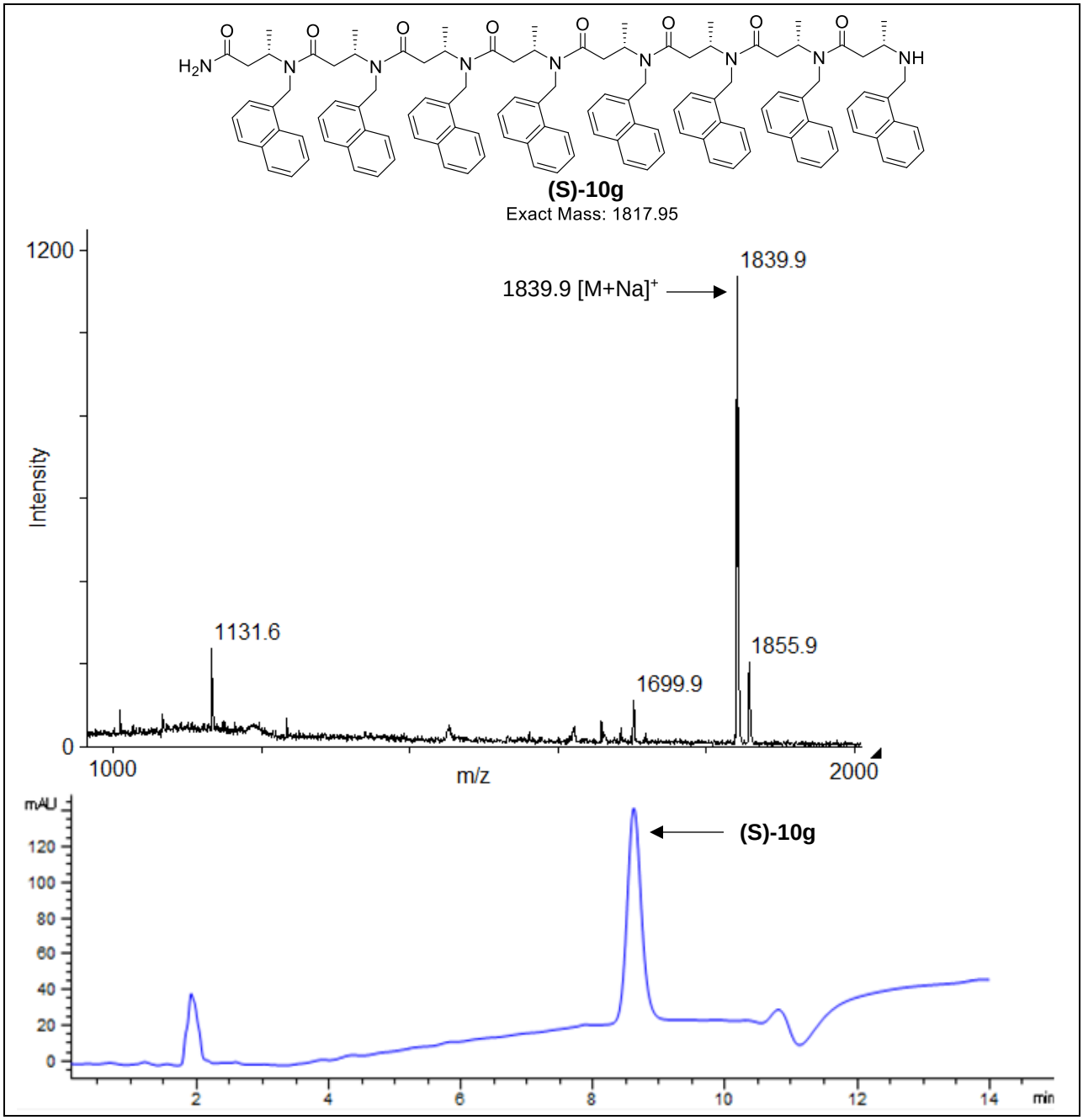


Figure S4, continued

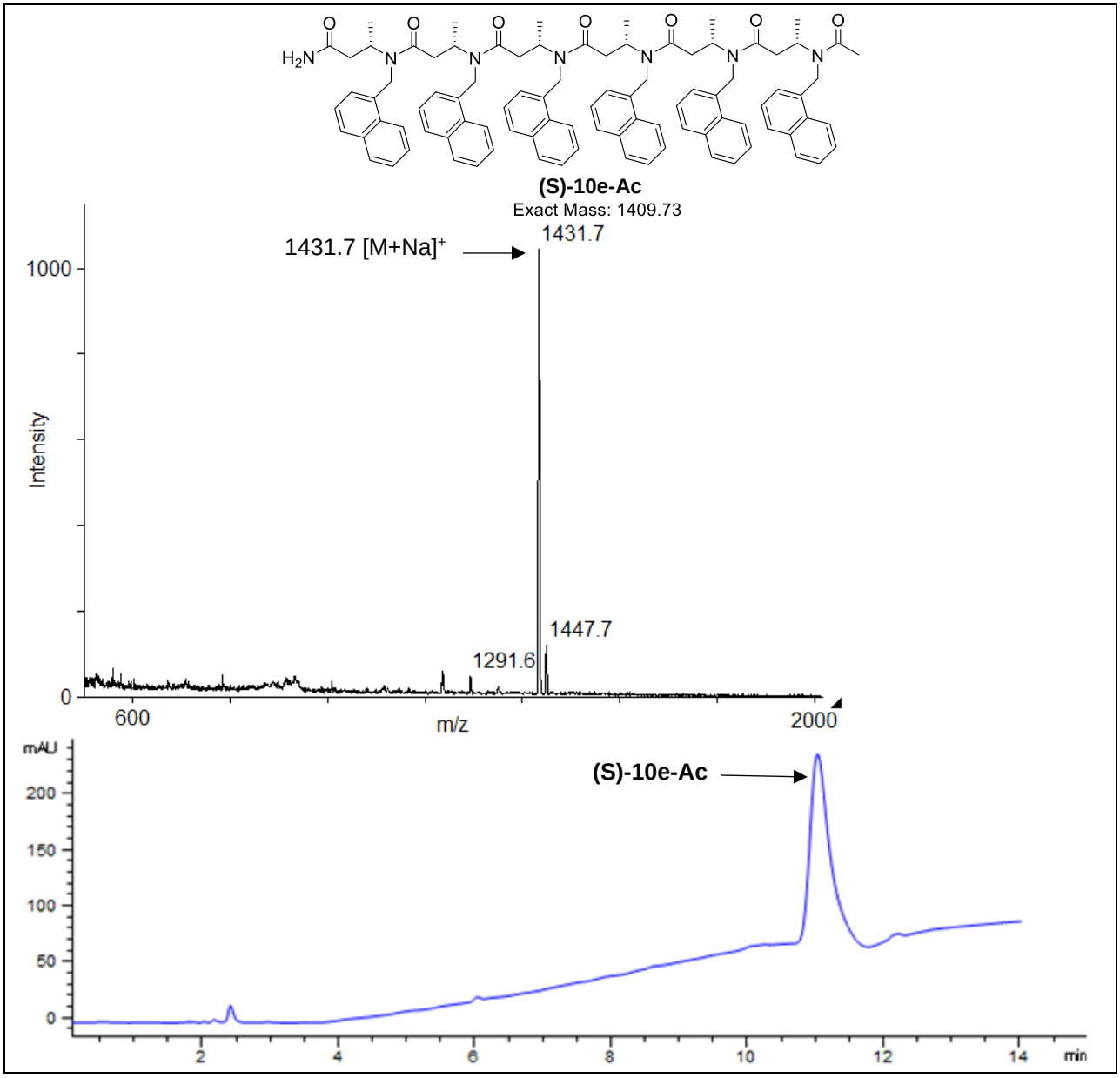


Figure S5. CD data of *N*-benzylated *β*-ABpeptoid oligomers in PBS-ACN (1:3) 60 μM (**a**) oligomers of (R)-form **(R)-9a-g** and, (**b**) (S)-form **(S)-9a-g**.

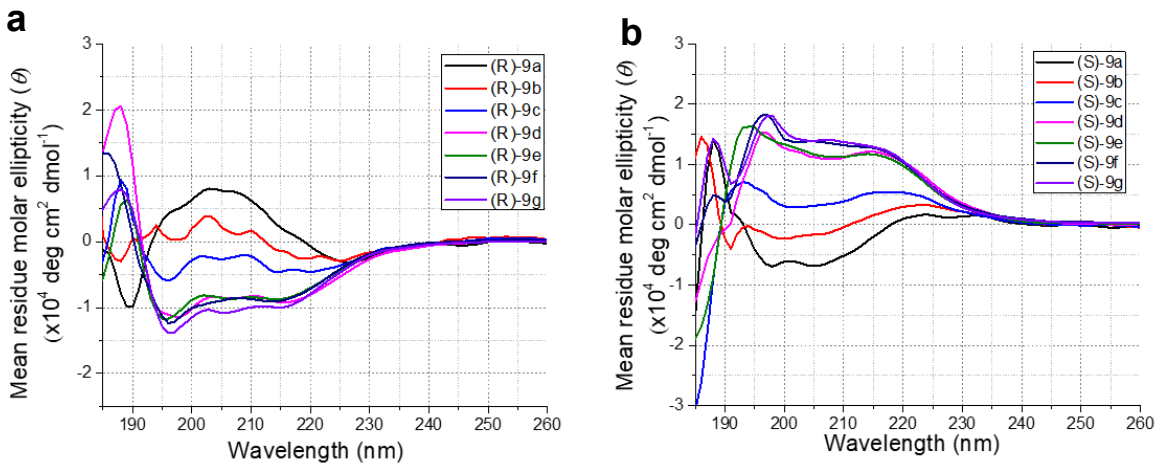


Figure S6. CD data of (S)-form of *N*-benzylated *β*-ABpeptoid oligomers (**S**)-**9a-g** in; **(a)** MeOH (60 μM) and, **(b)** TFE (60 μM).

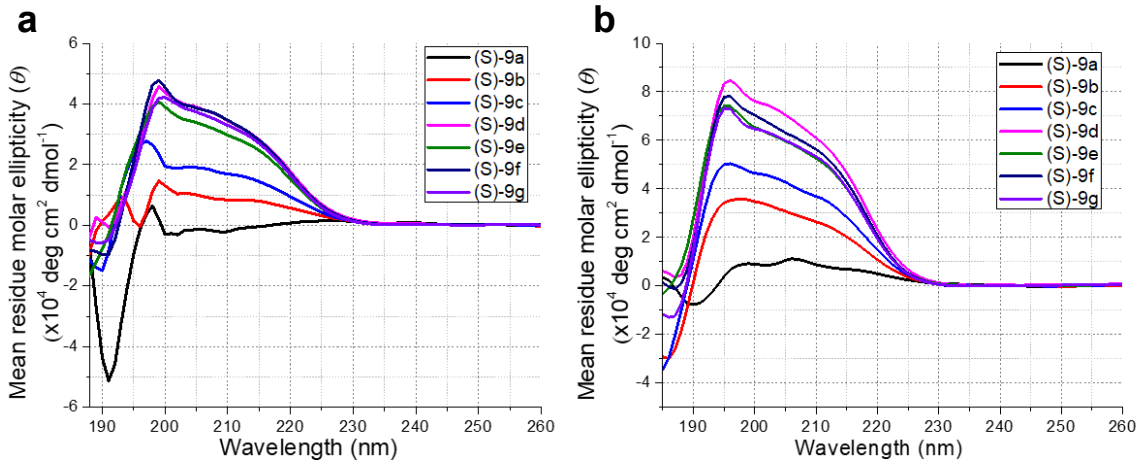


Figure S7. CD data of (S)-form of *N*-napthylmethyl *β*-ABpeptoid oligomers (**S**)-**10a-g** in; **(a)** PBS-ACN (1:3, 60 μM), **(b)** MeOH (60 μM), **(c)** TFE (60 μM).

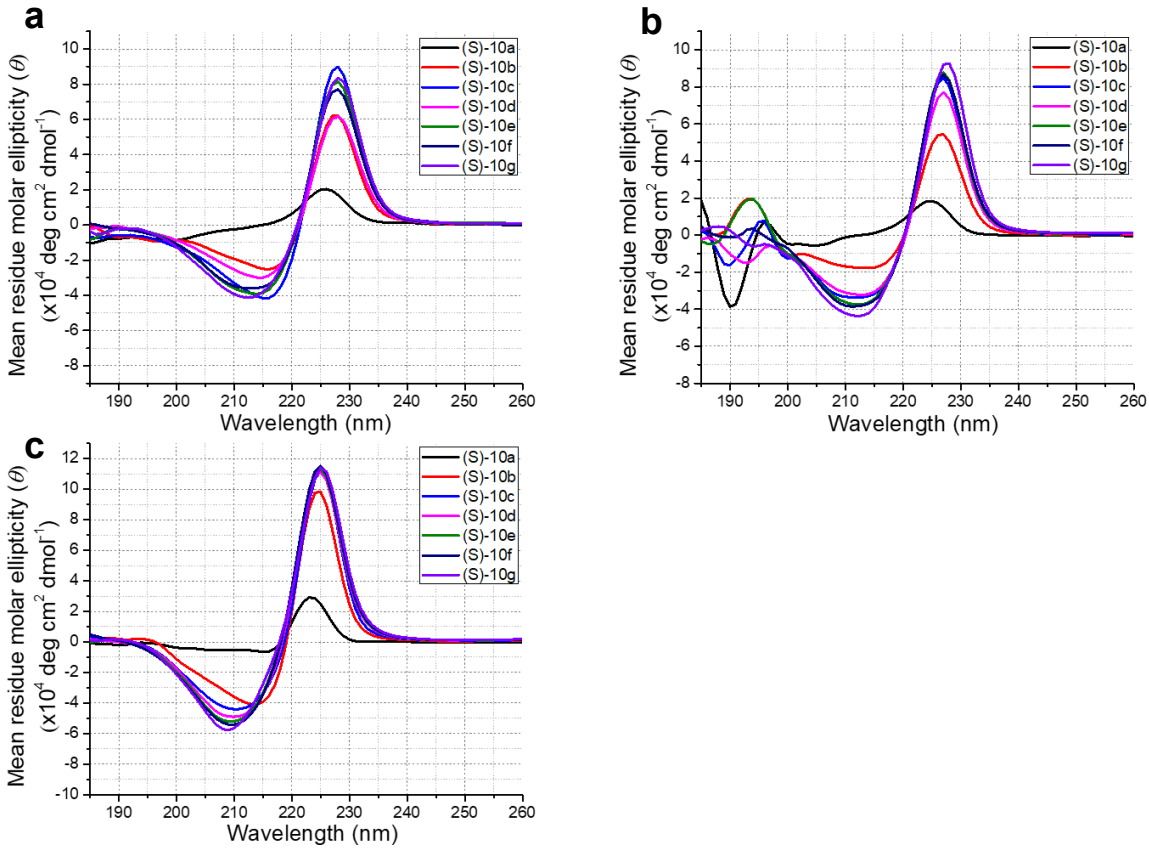


Figure S8. CD spectra (S)-form of *N*-napthylmethyl *β*-ABpeptoid octamer (**S**)-**10g** in ACN (60 μM) measured at 20 °C before and after heating to 70 °C.

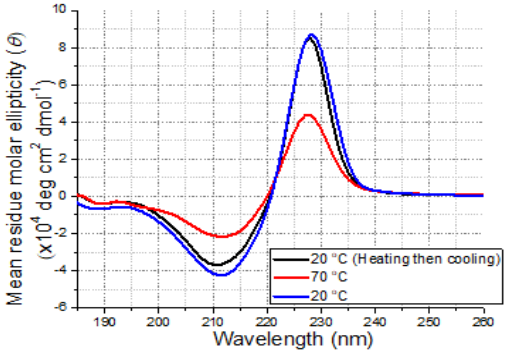


Figure S9. ^{1}H and ^{13}C NMR data of; **(R)-1**, **(S)-4**, **(S)-5a**, **(S)-5b**, **(S)-6a**, and **(S)-6b.**

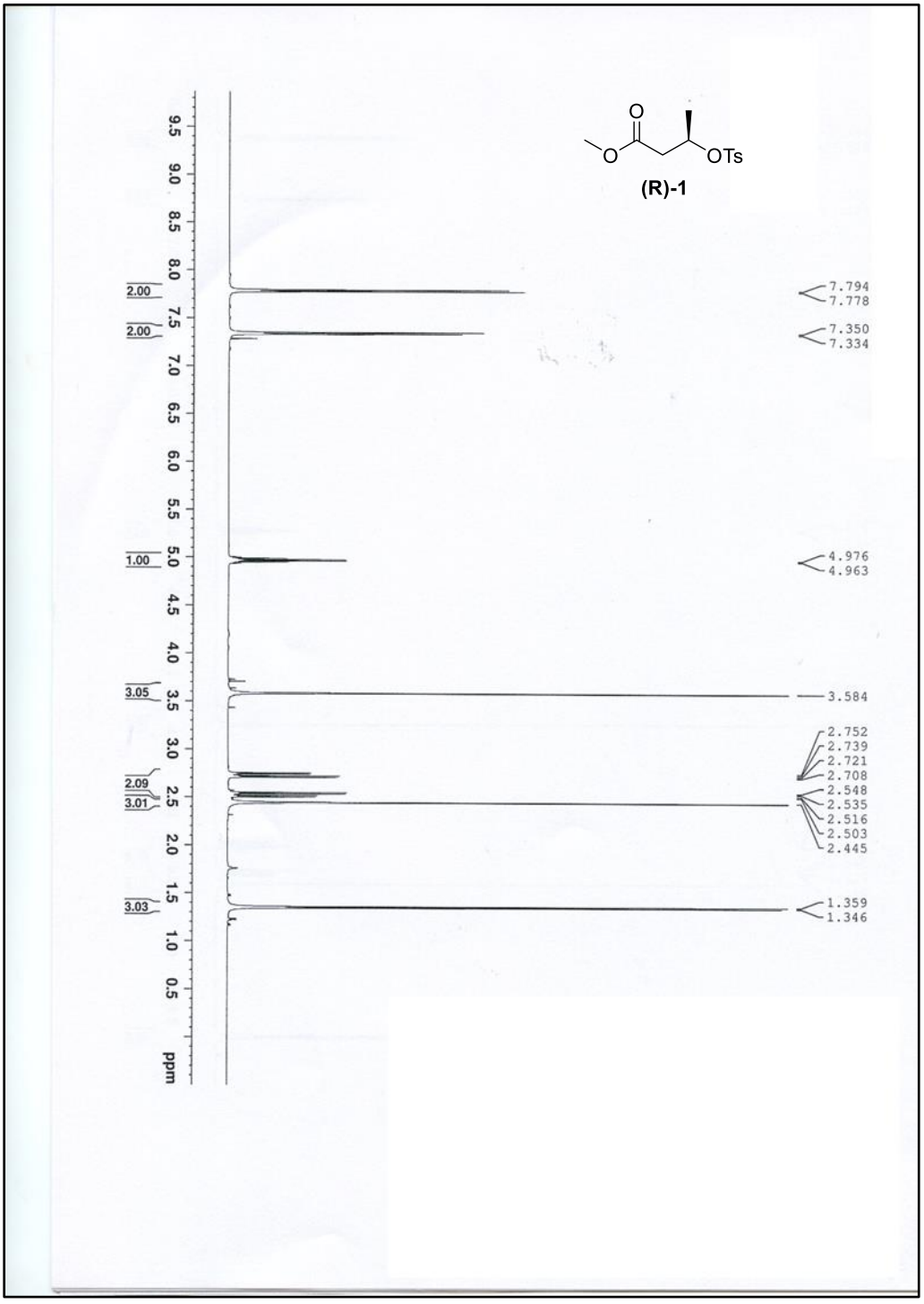


Figure S9, continued

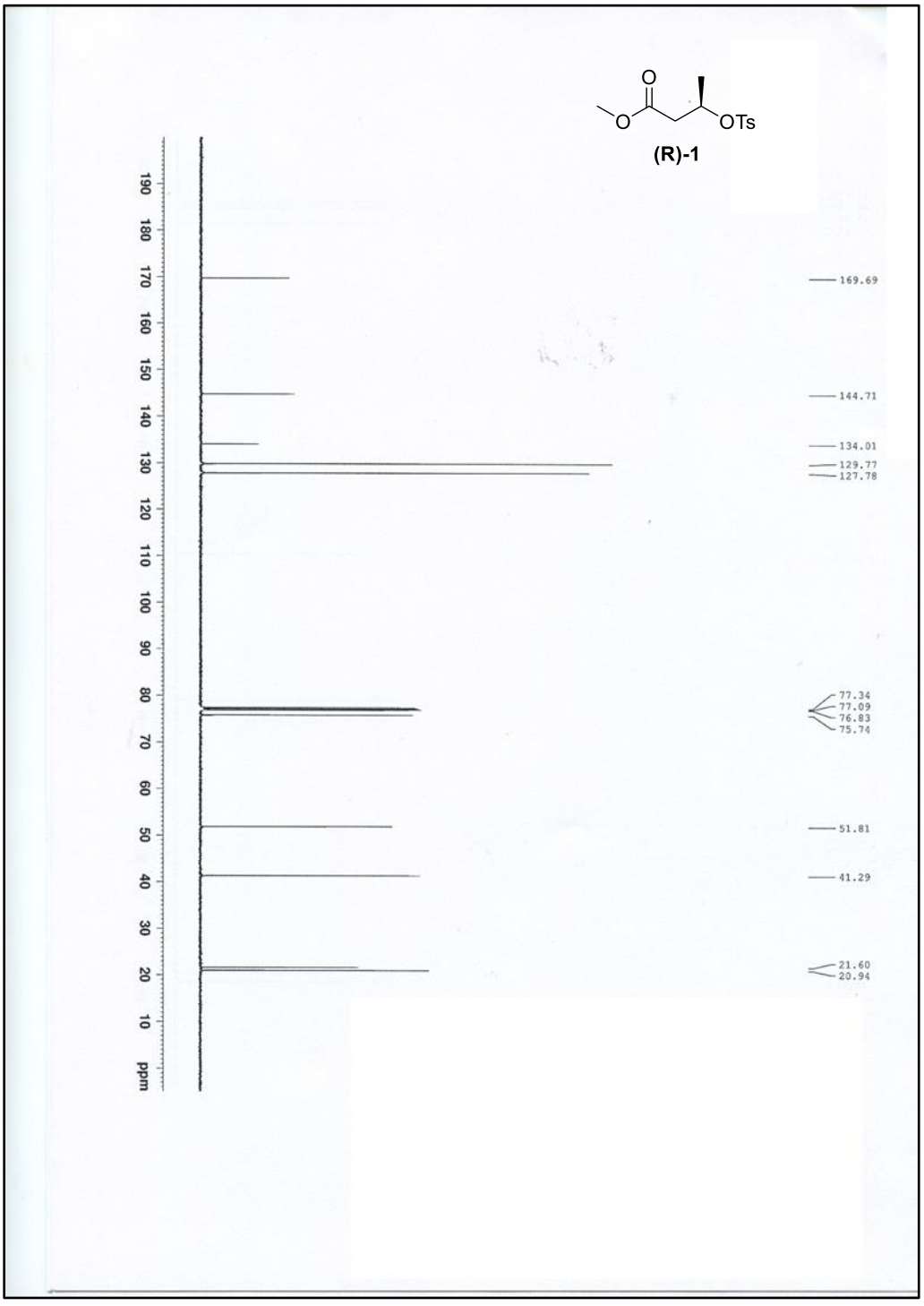


Figure S9, continued

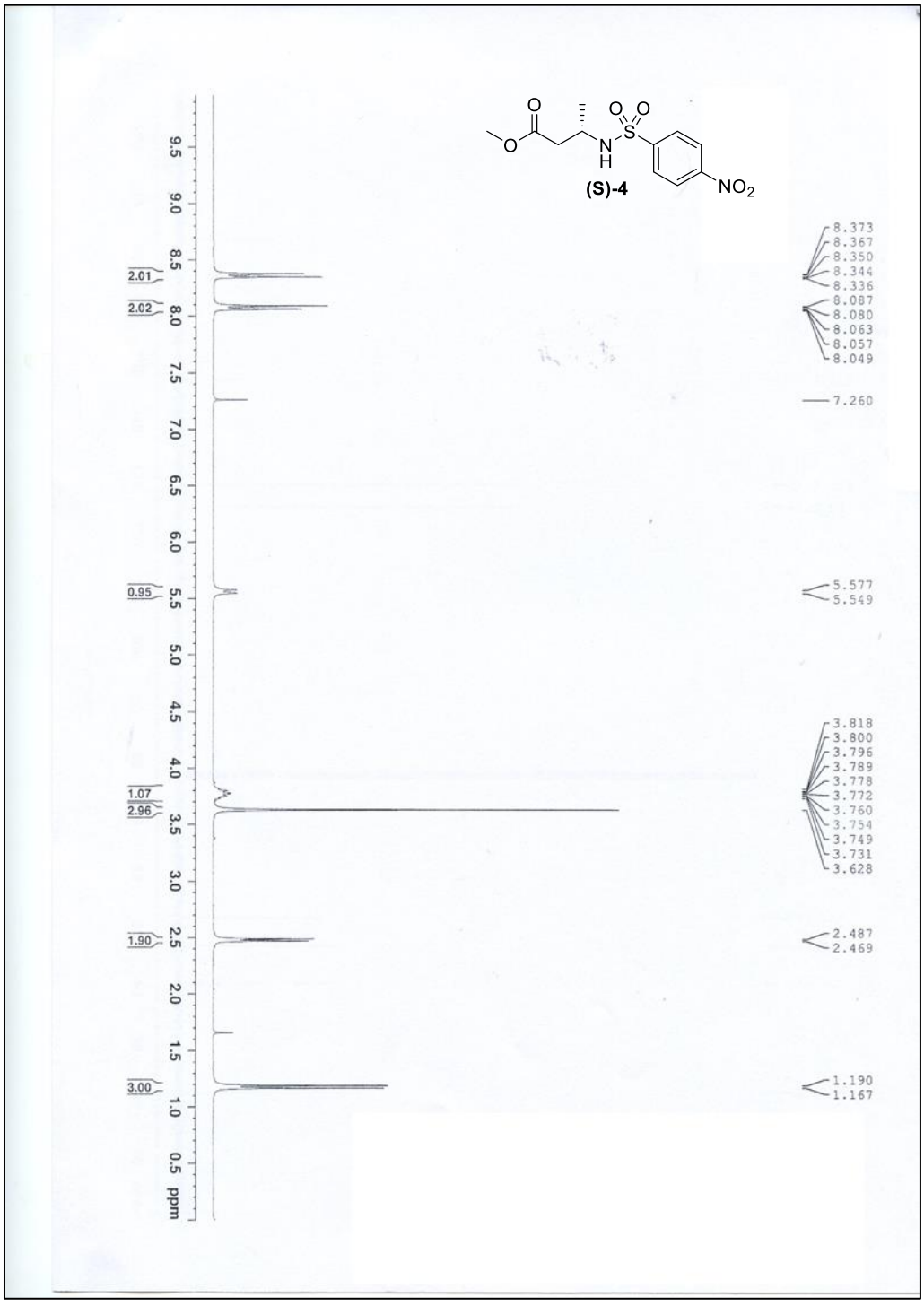


Figure S9, continued

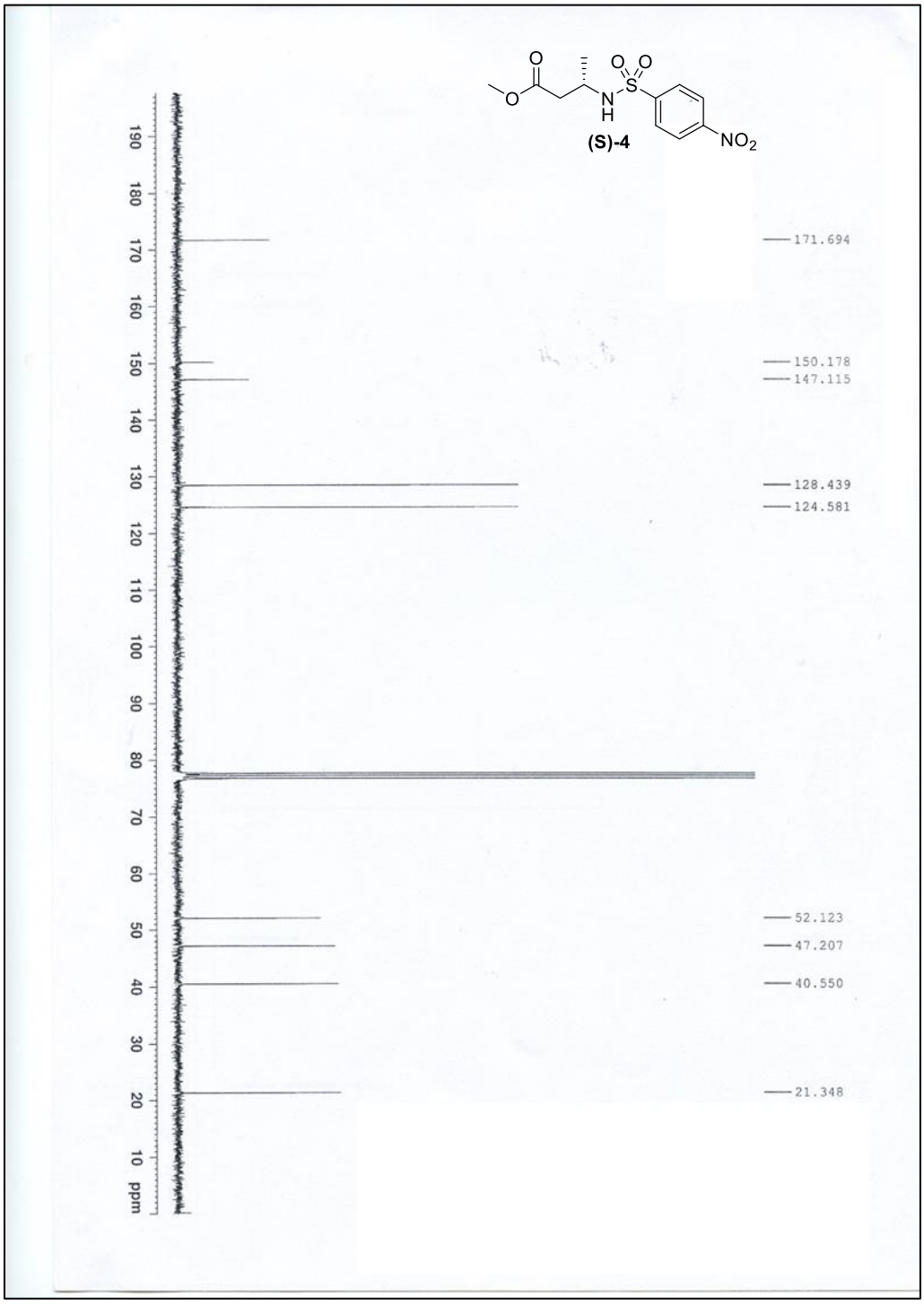


Figure S9, continued

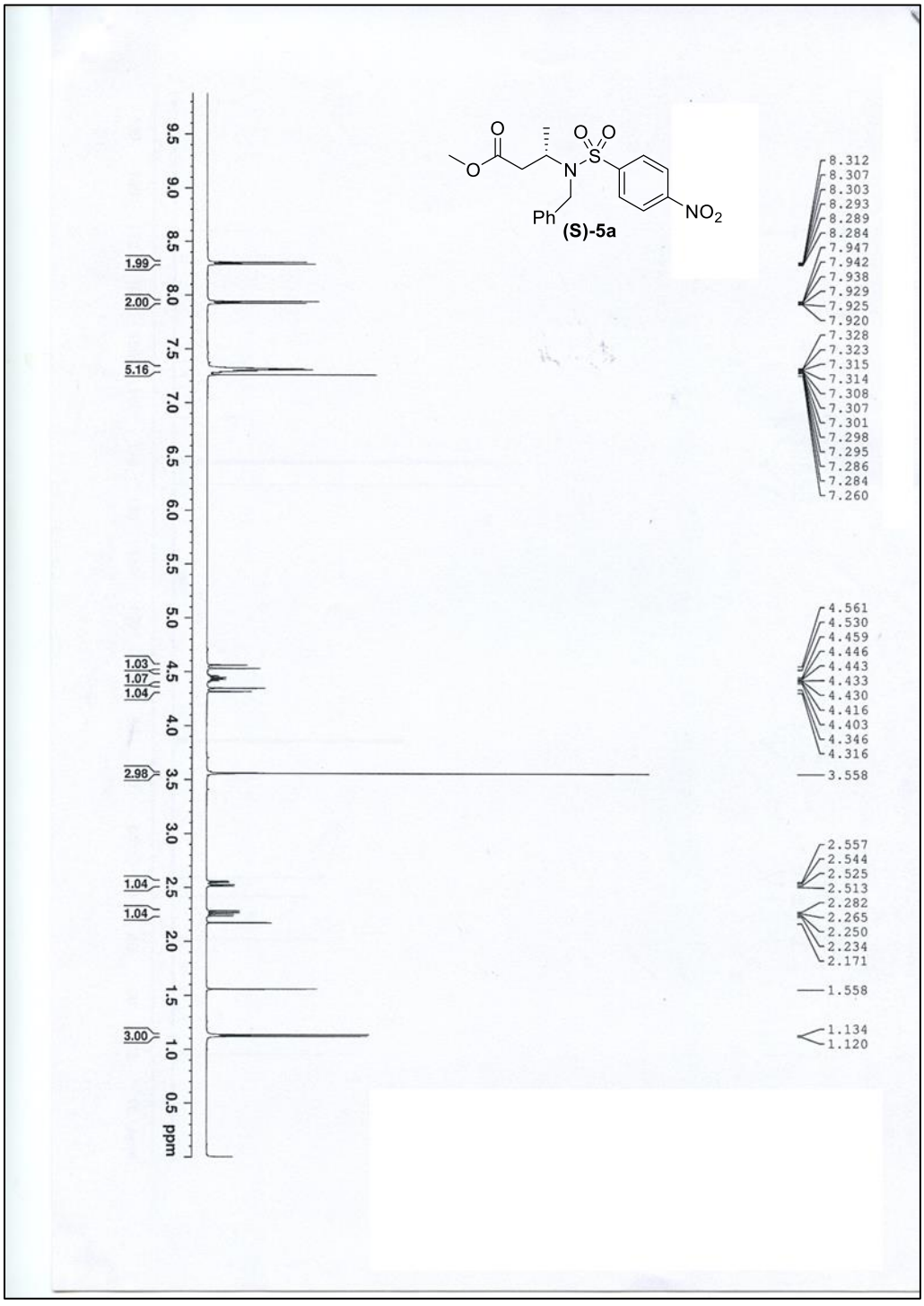


Figure S9, continued

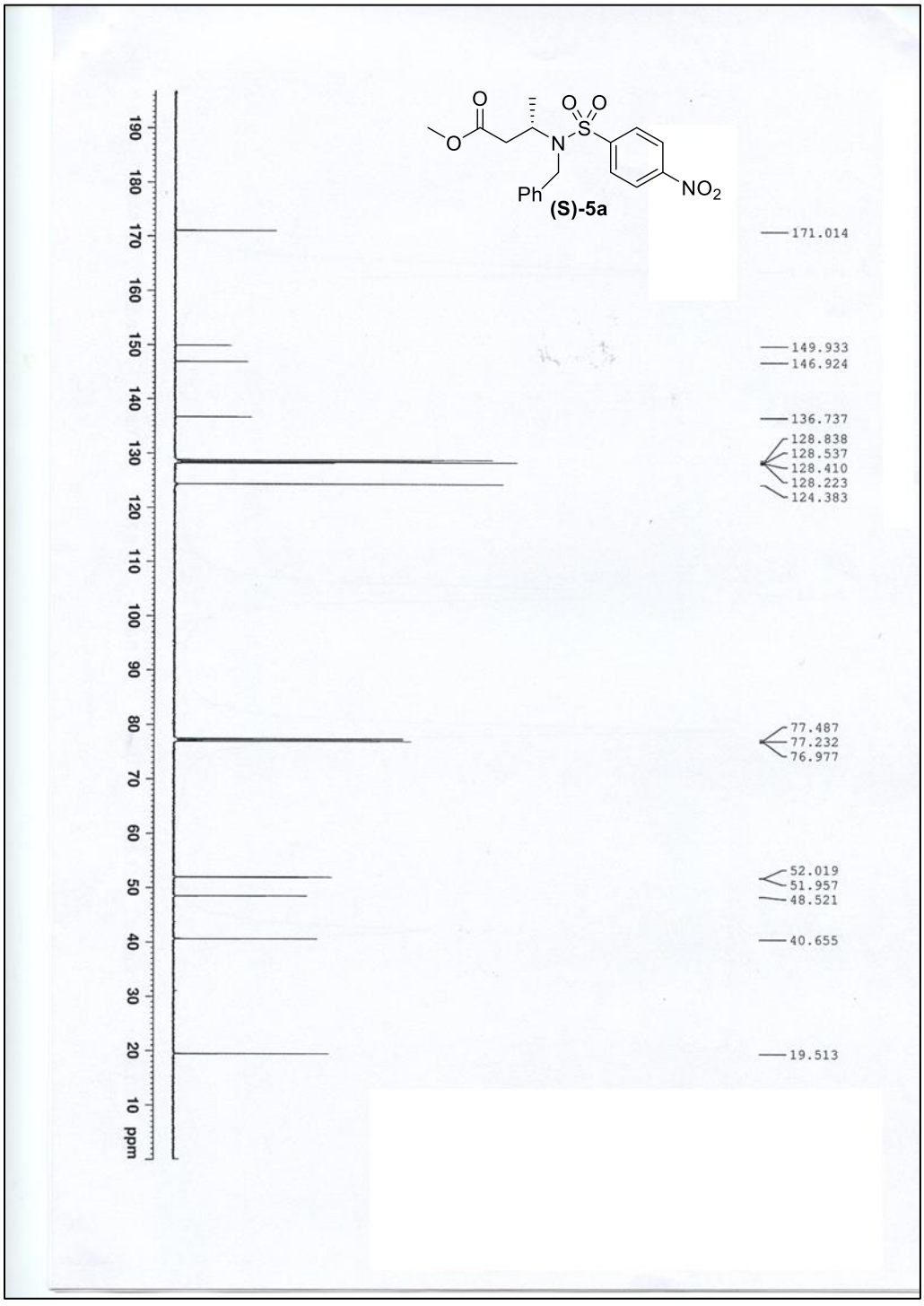


Figure S9, continued

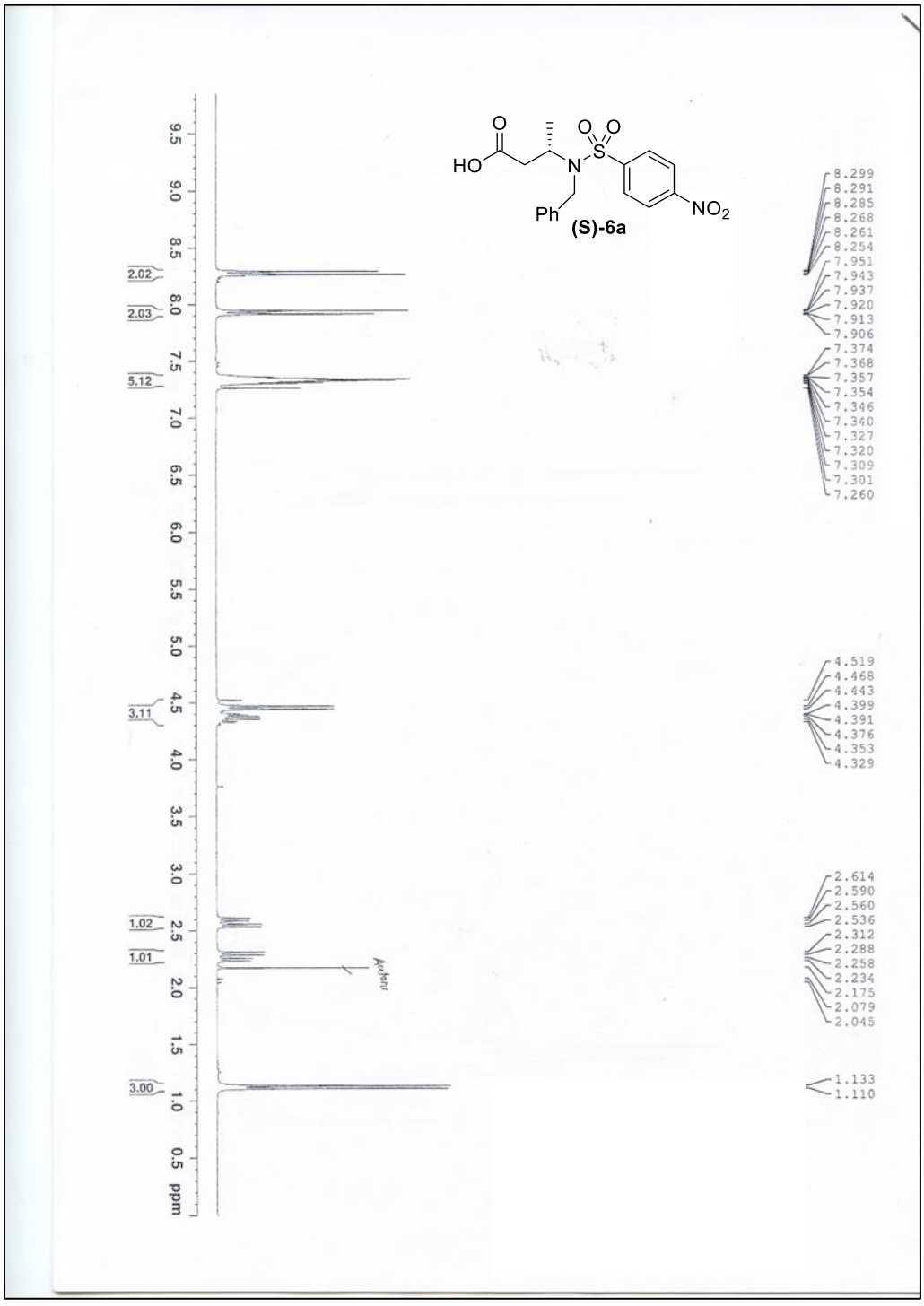
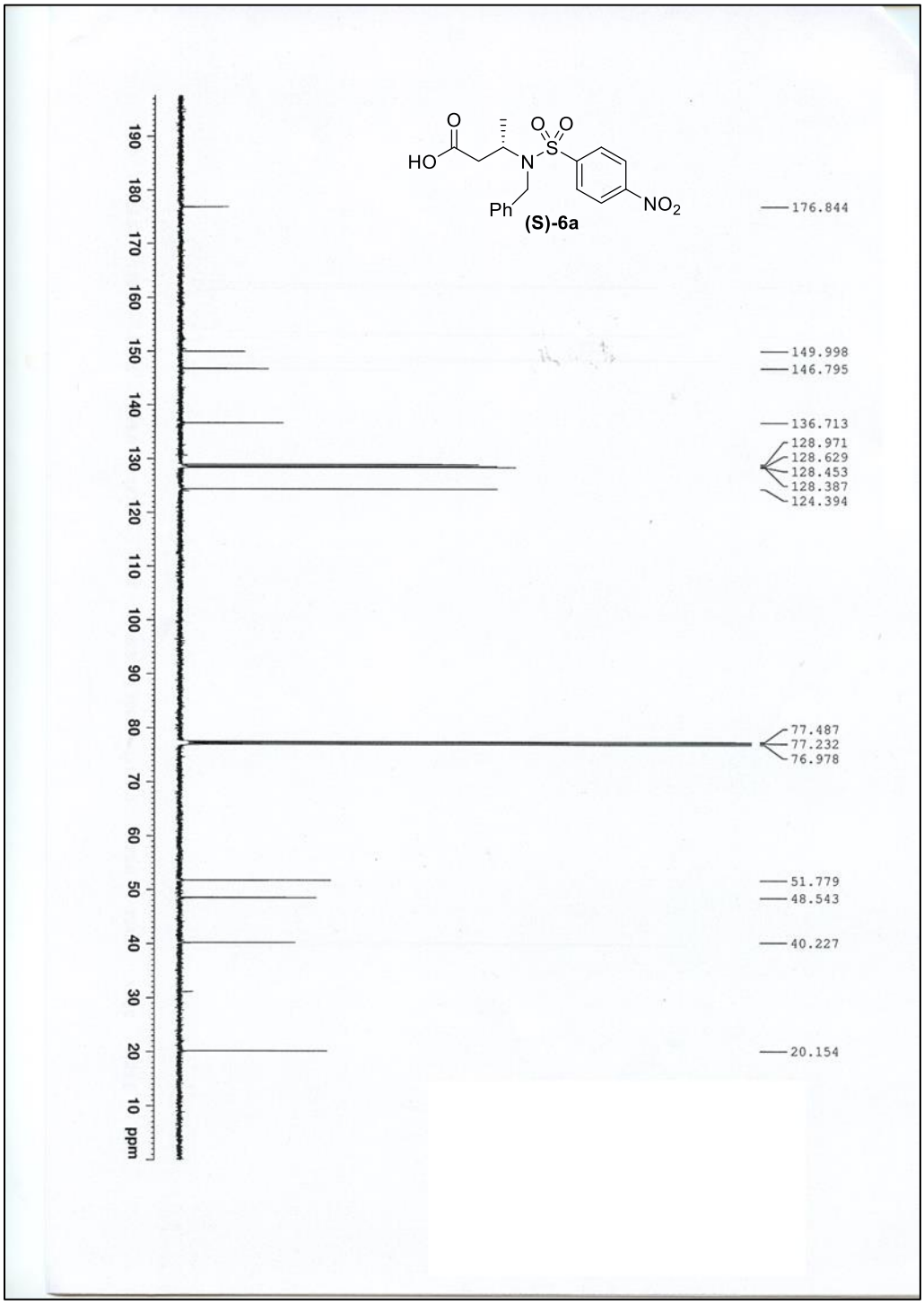


Figure S9, continued



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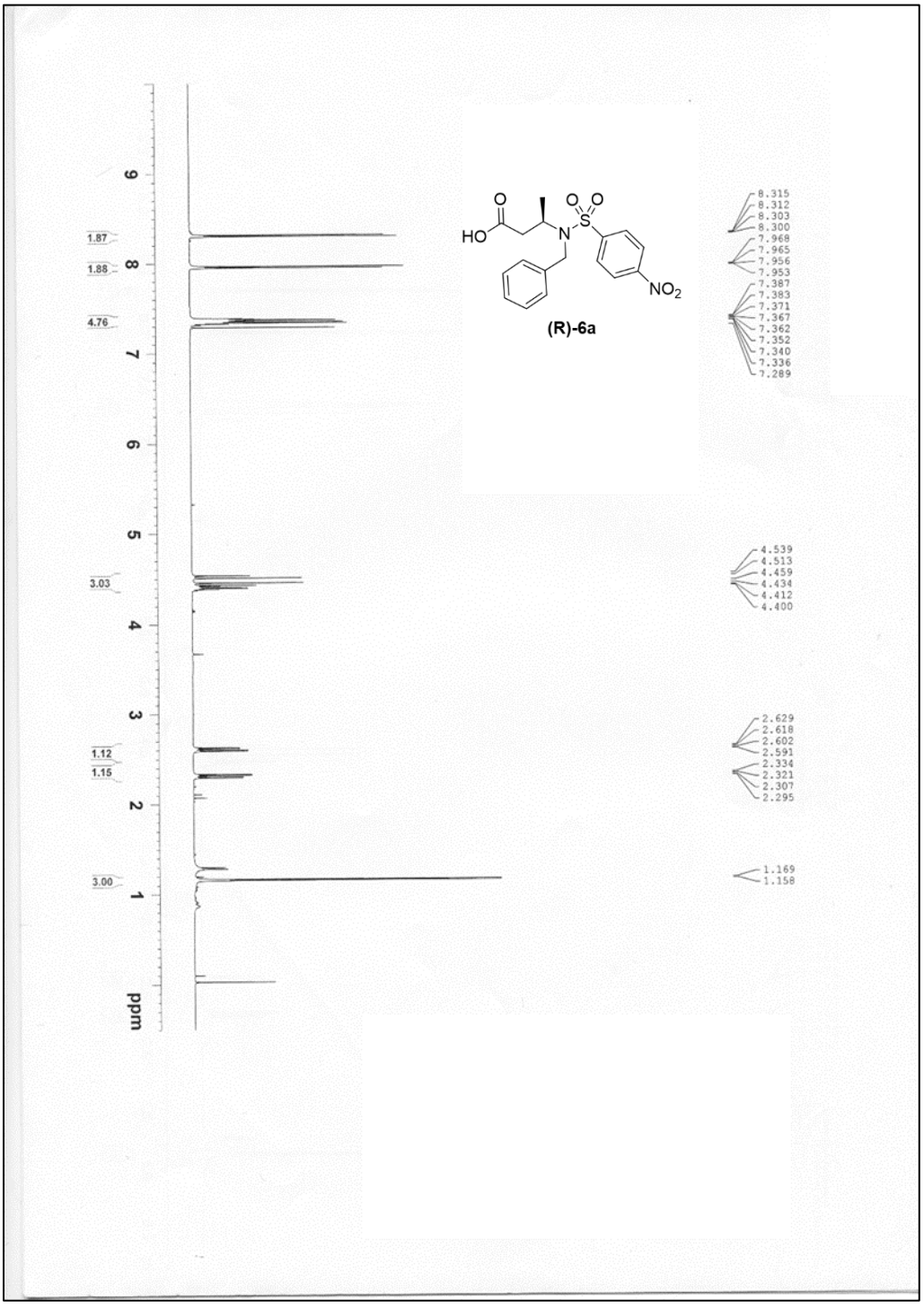


Figure S9, continued

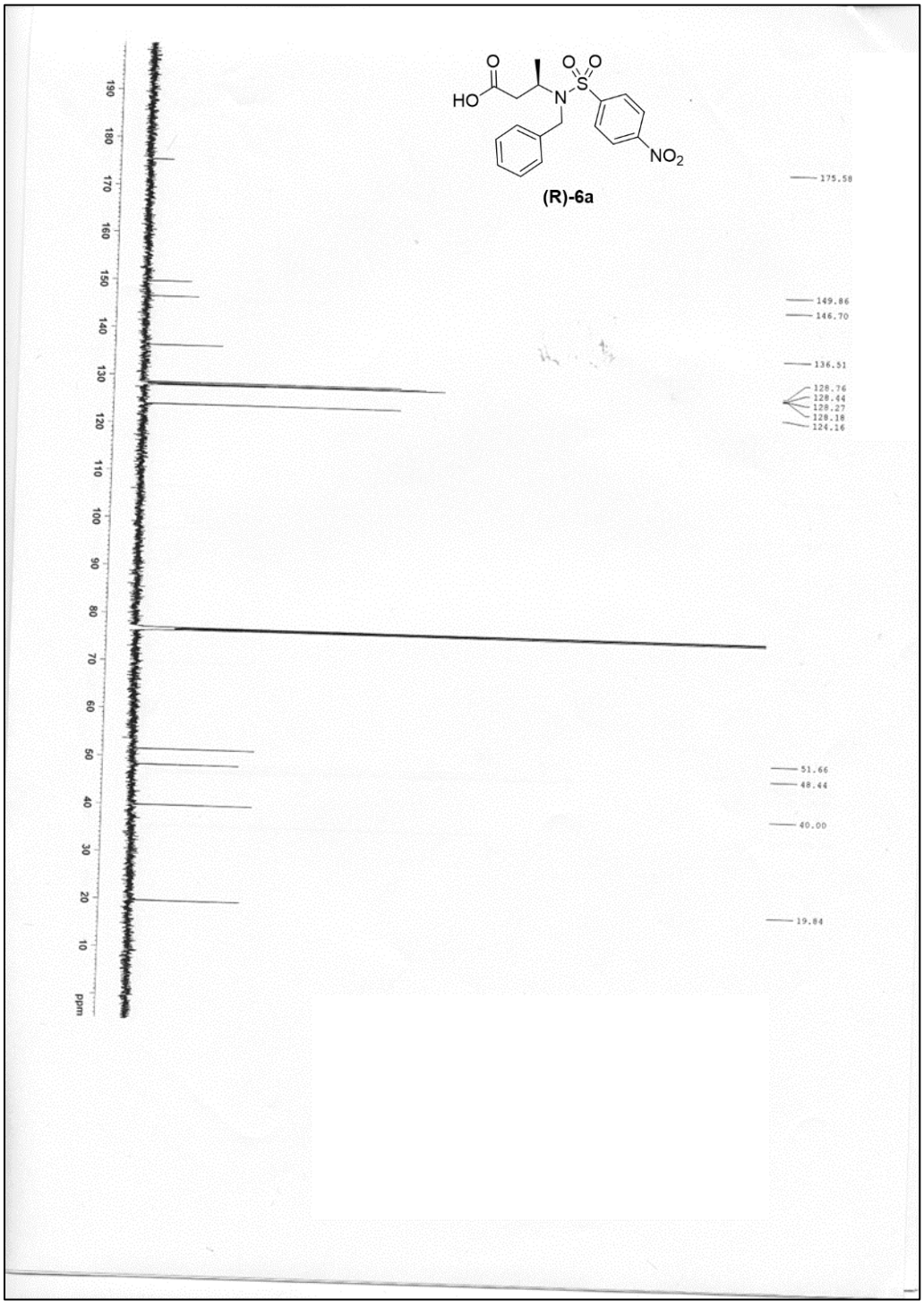


Figure S9, continued

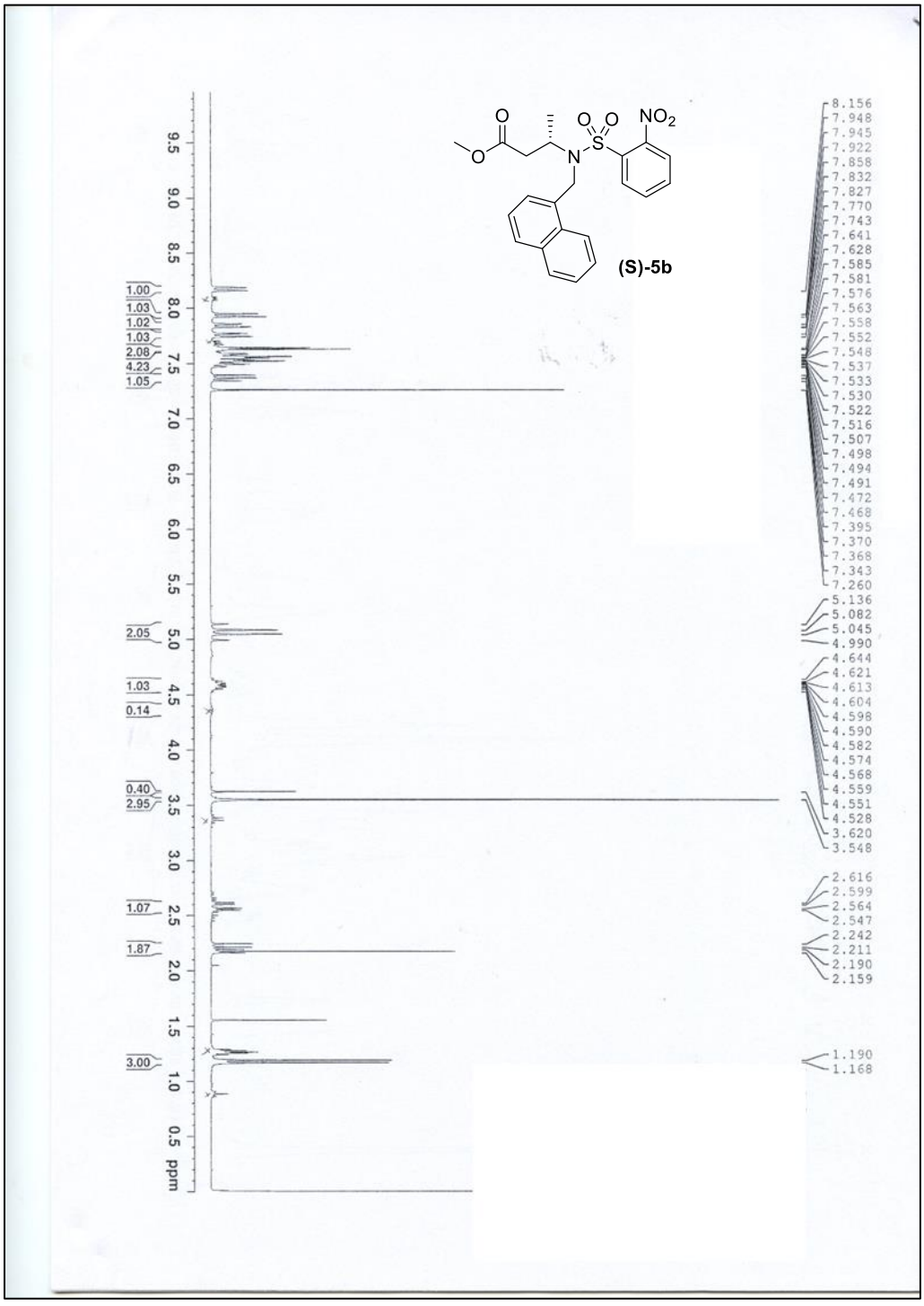


Figure S9, continued

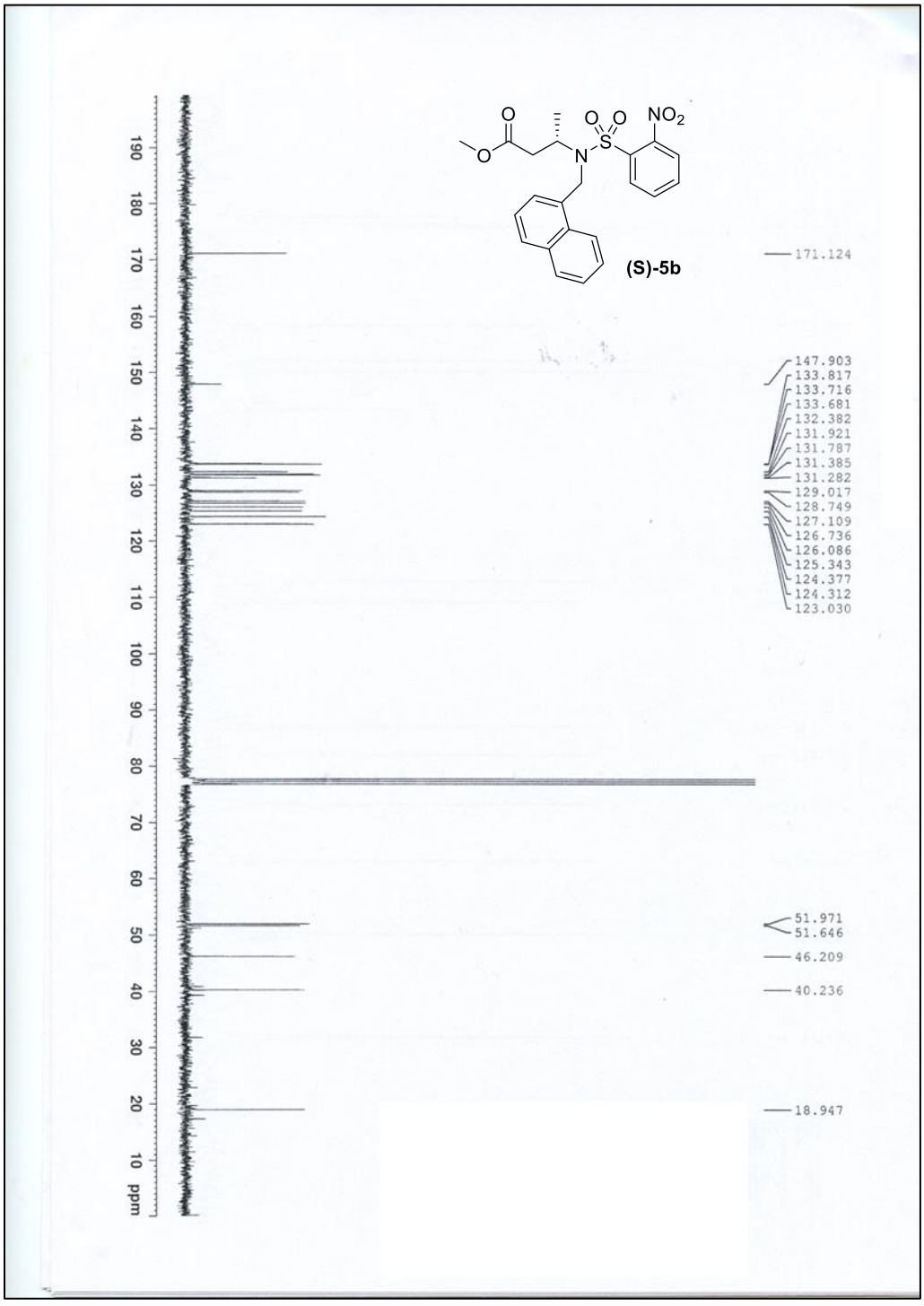


Figure S9, continued

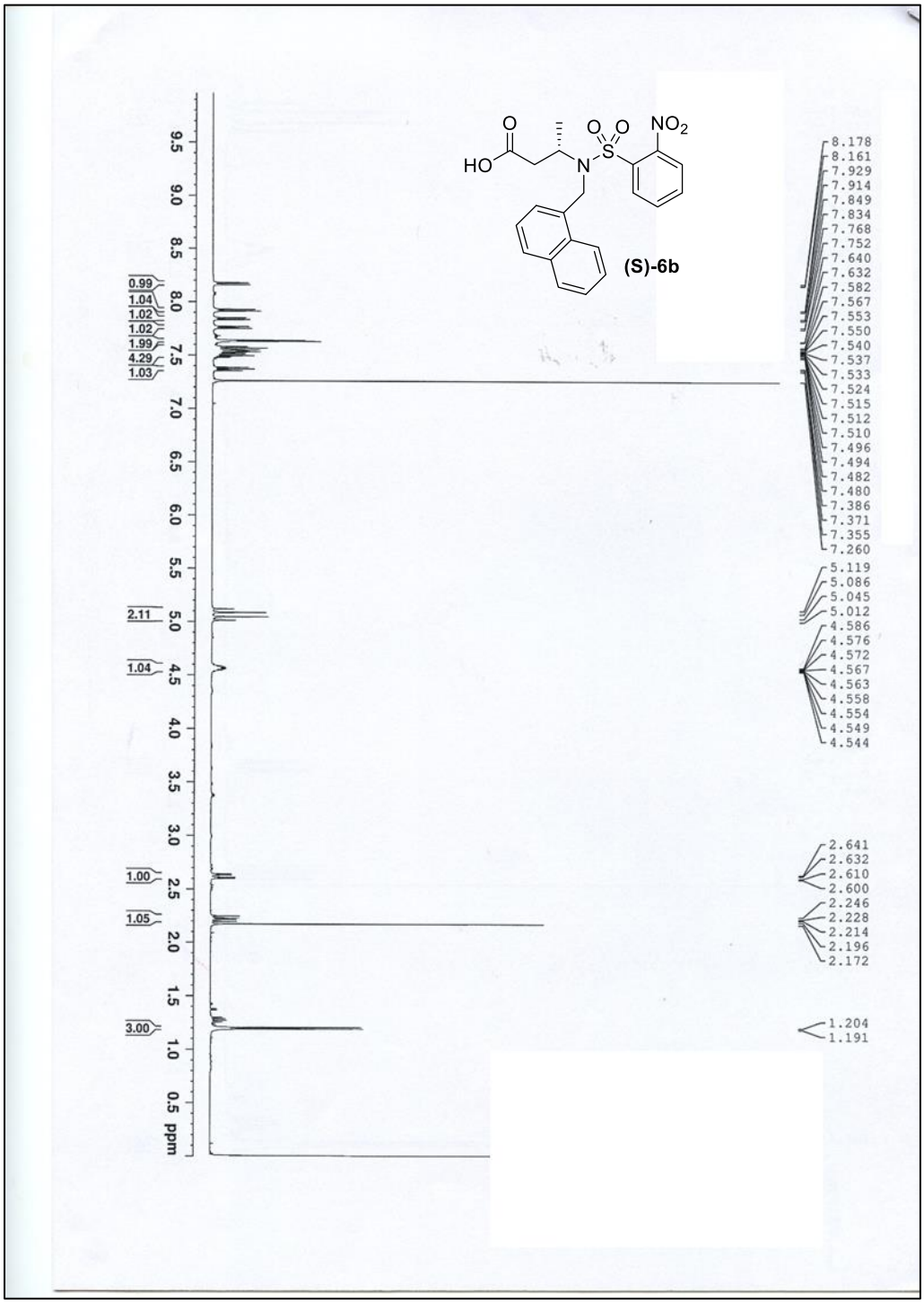


Figure S9, continued

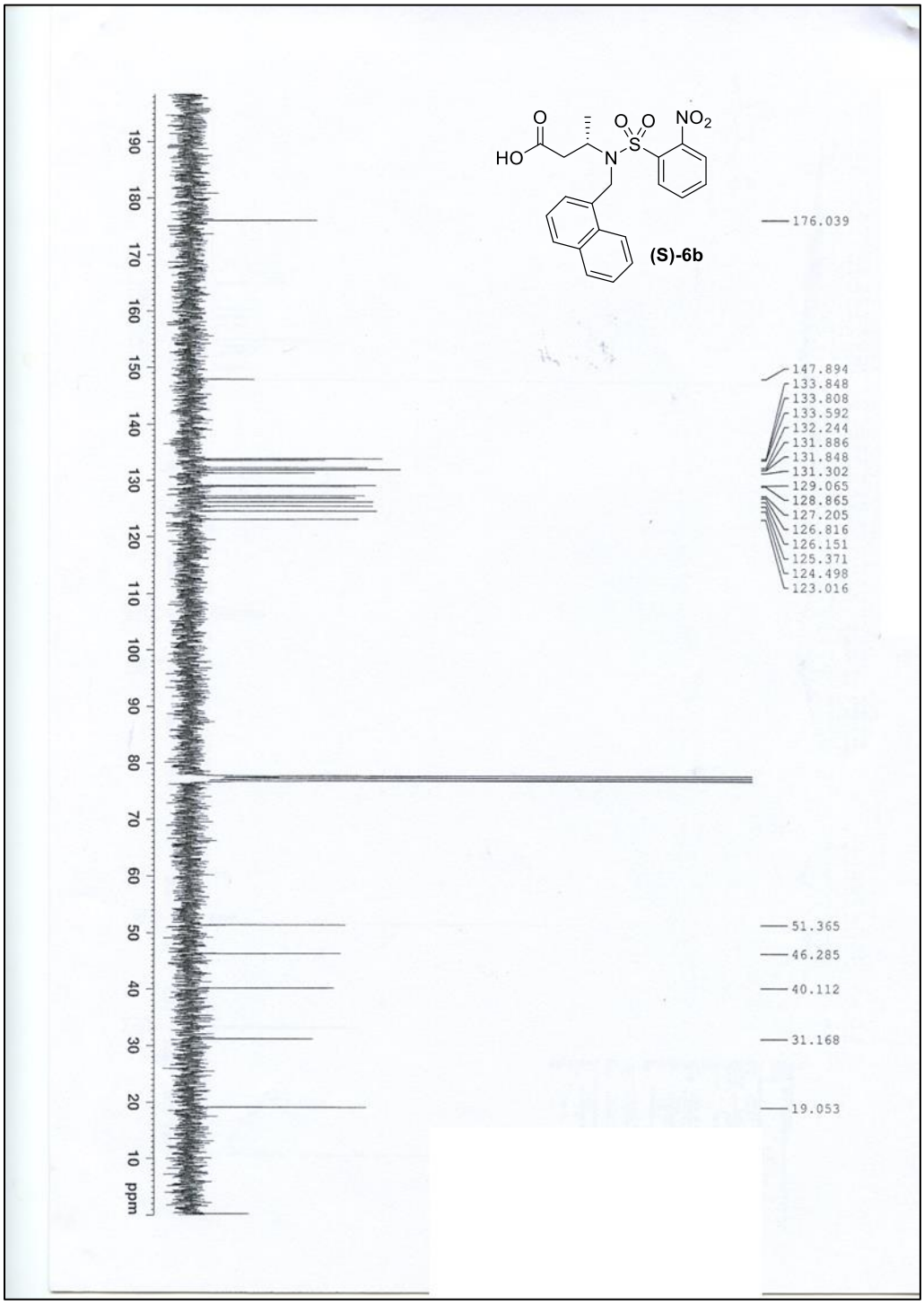


Figure S9, continued

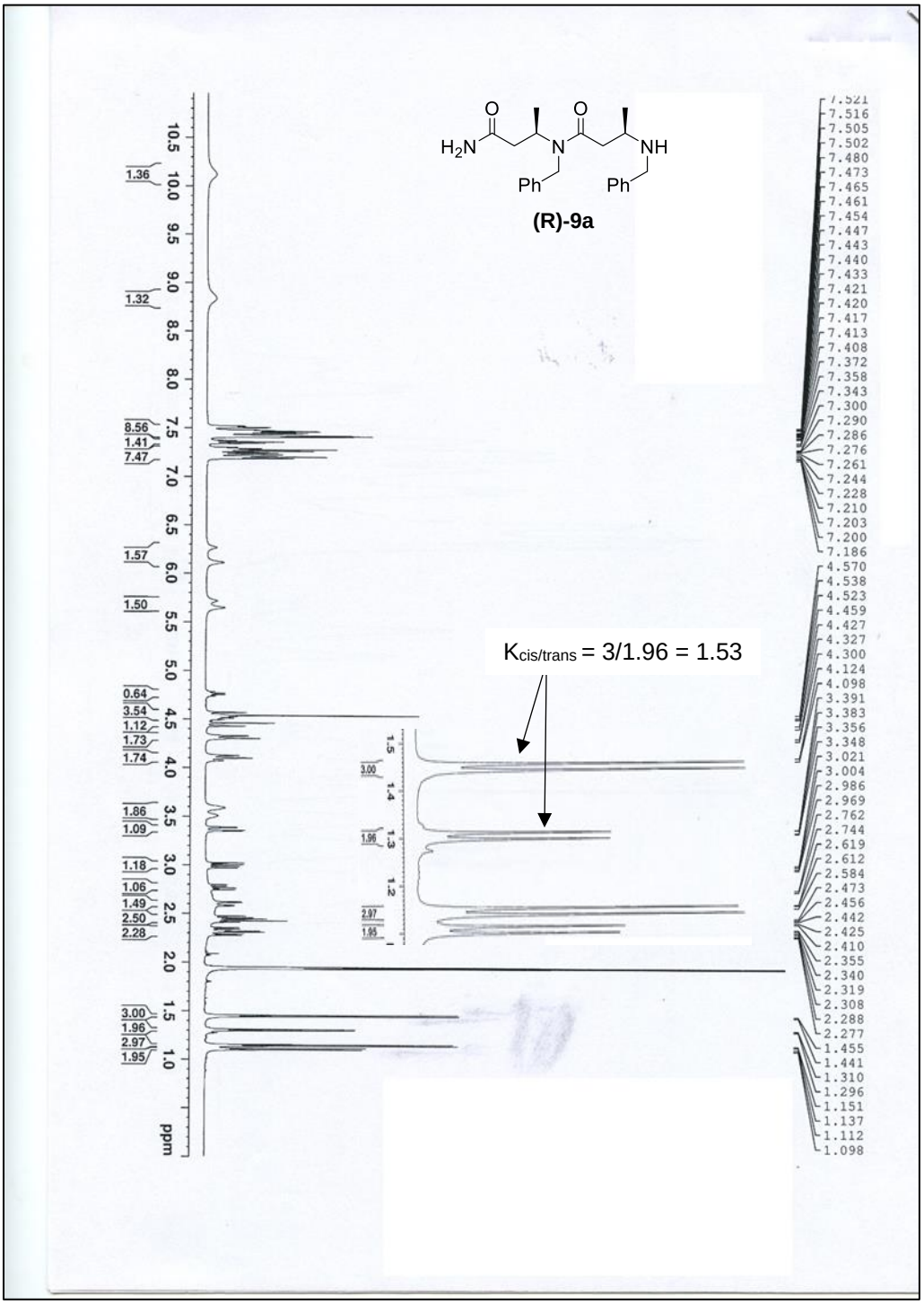


Figure S9, continued

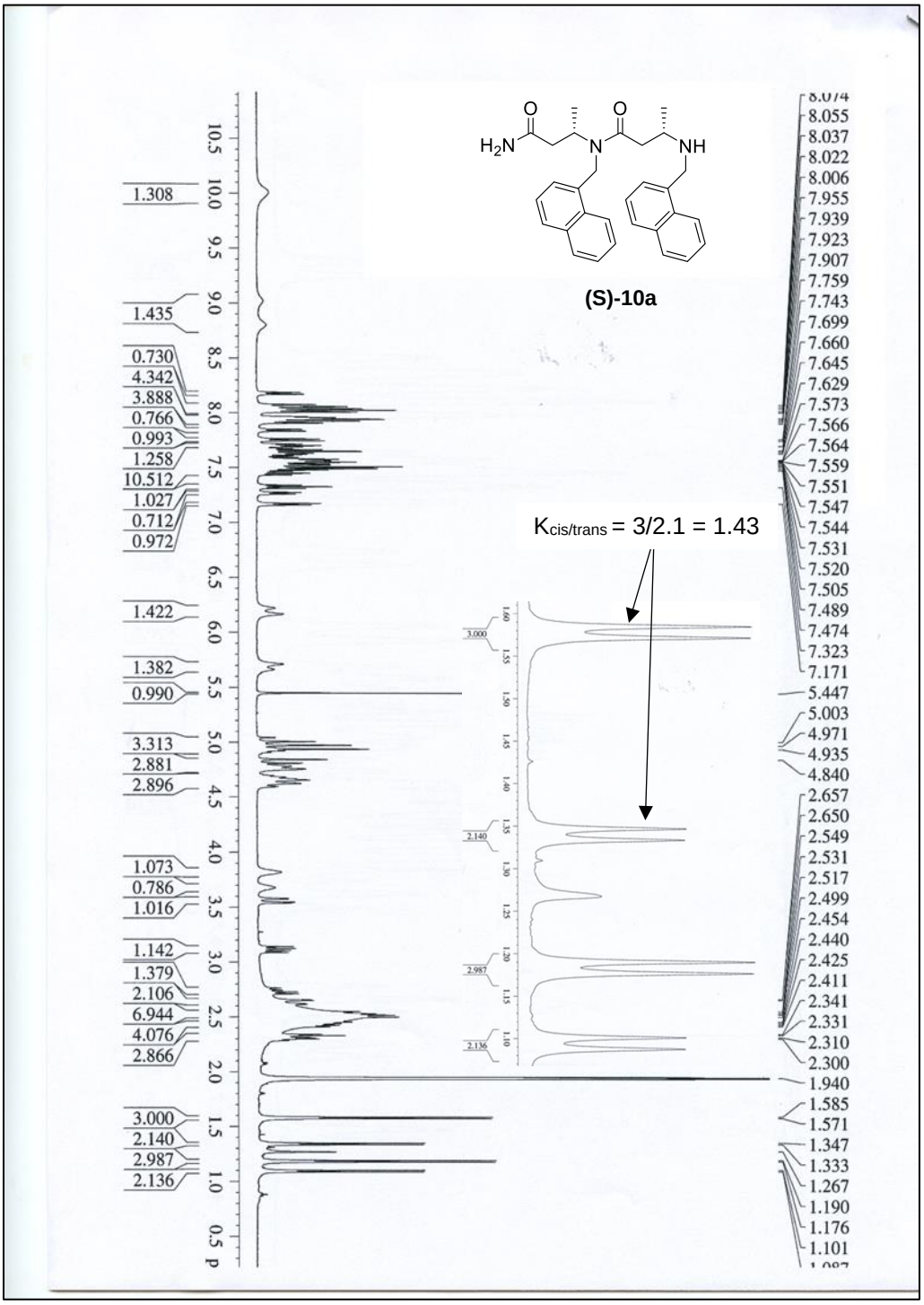
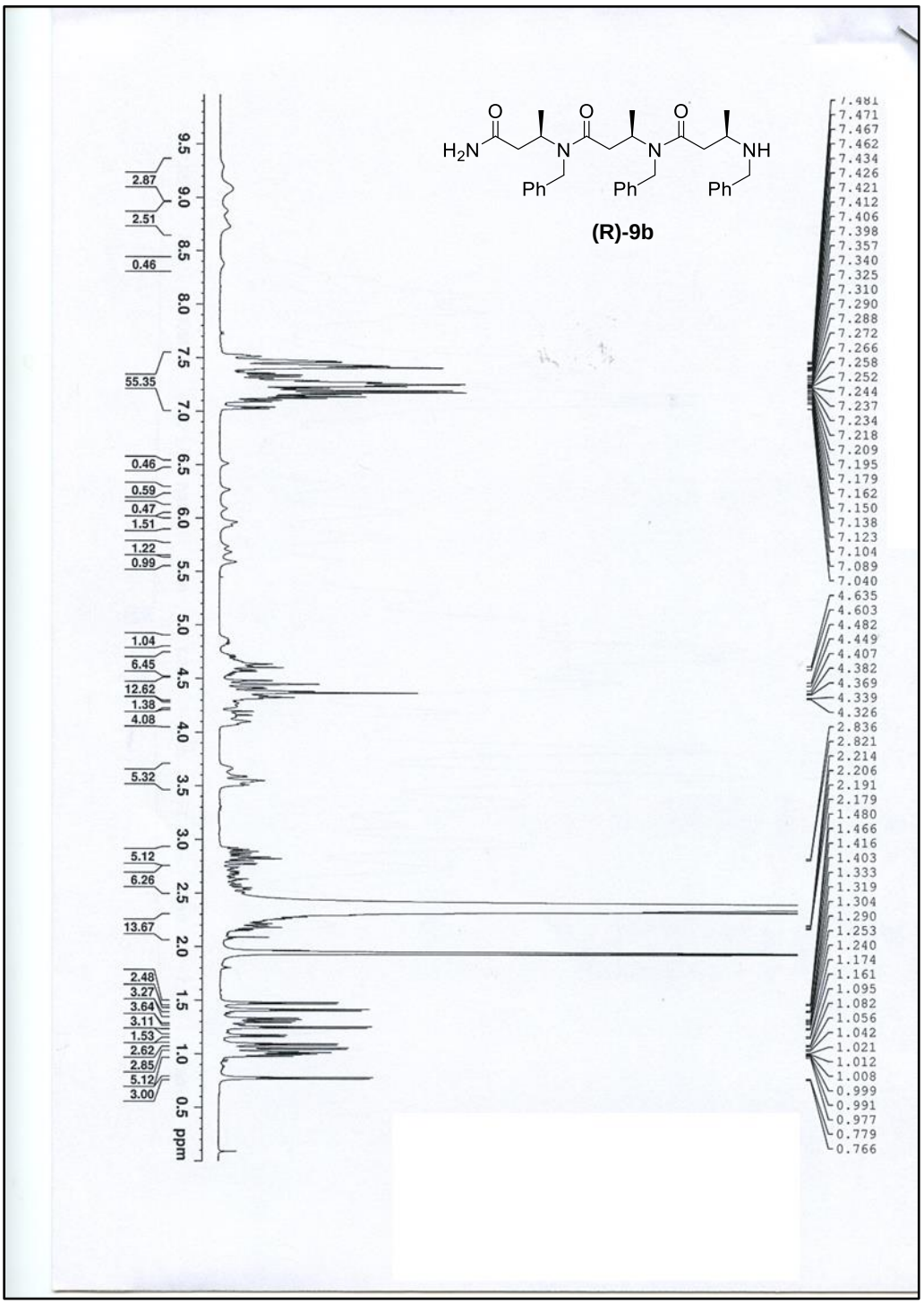


Figure S9, continued



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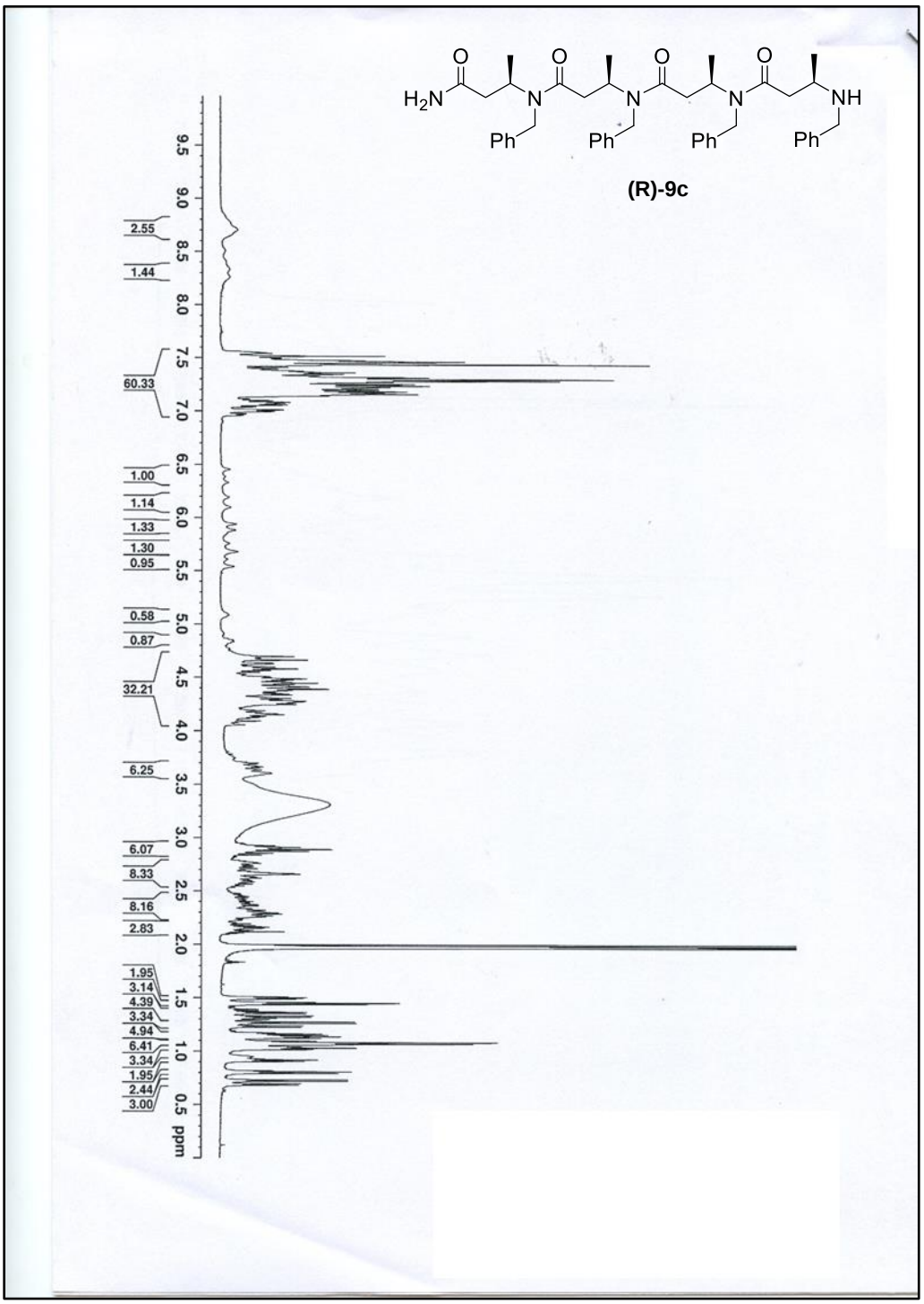

Figure S10. 2D ^{1}H NOESY spectra of (a) 2mer **(R)-9a** (b) 3mer **(R)-9b** and, (c) 4mer **(R)-9c**. [Dotted square regions indicate increase in additional NOEs from 2mer to 4mer, presumably due to the conformationally (cis) dominant ordered arrangement across the amide bond.]

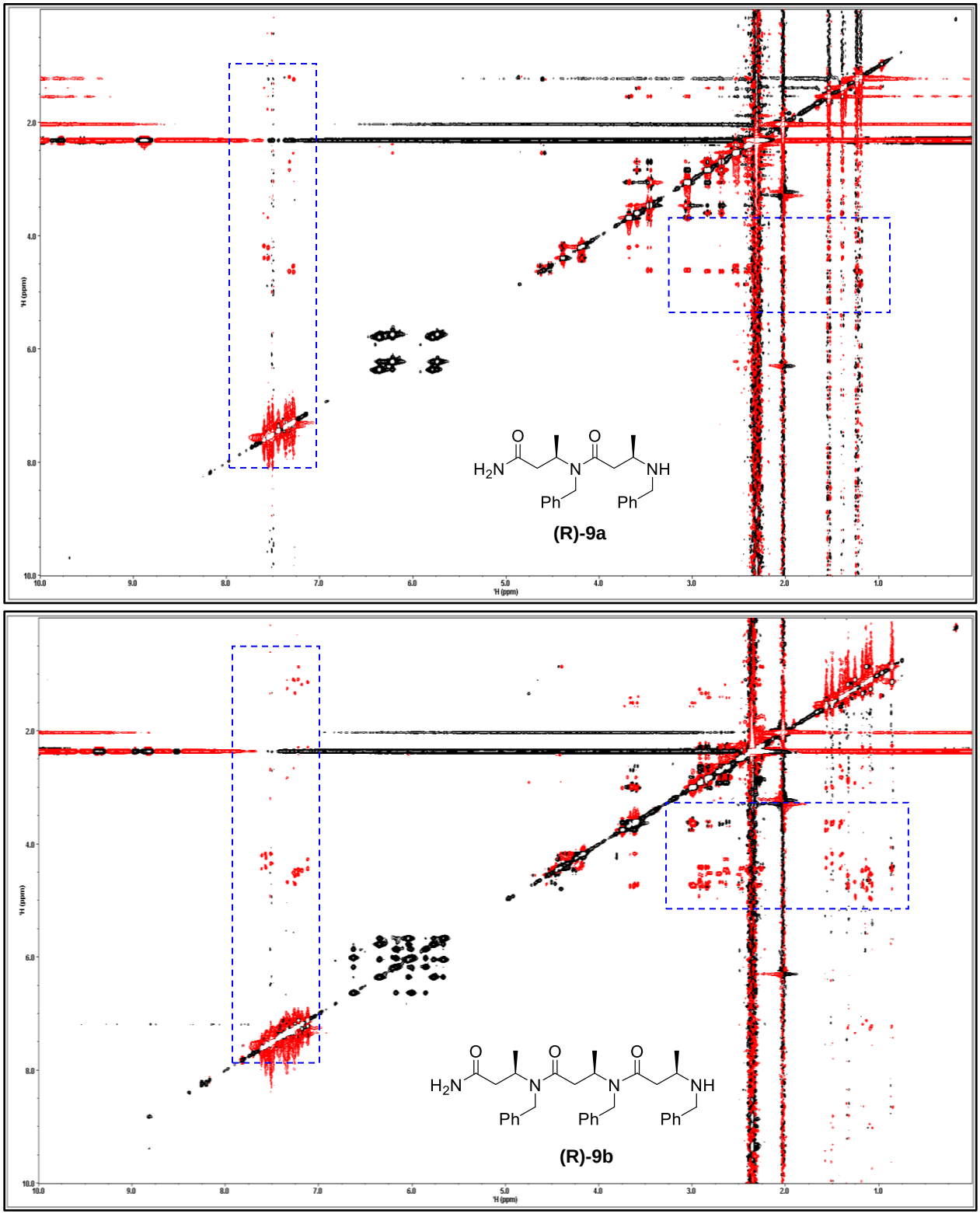

Figure S10, continued