Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry

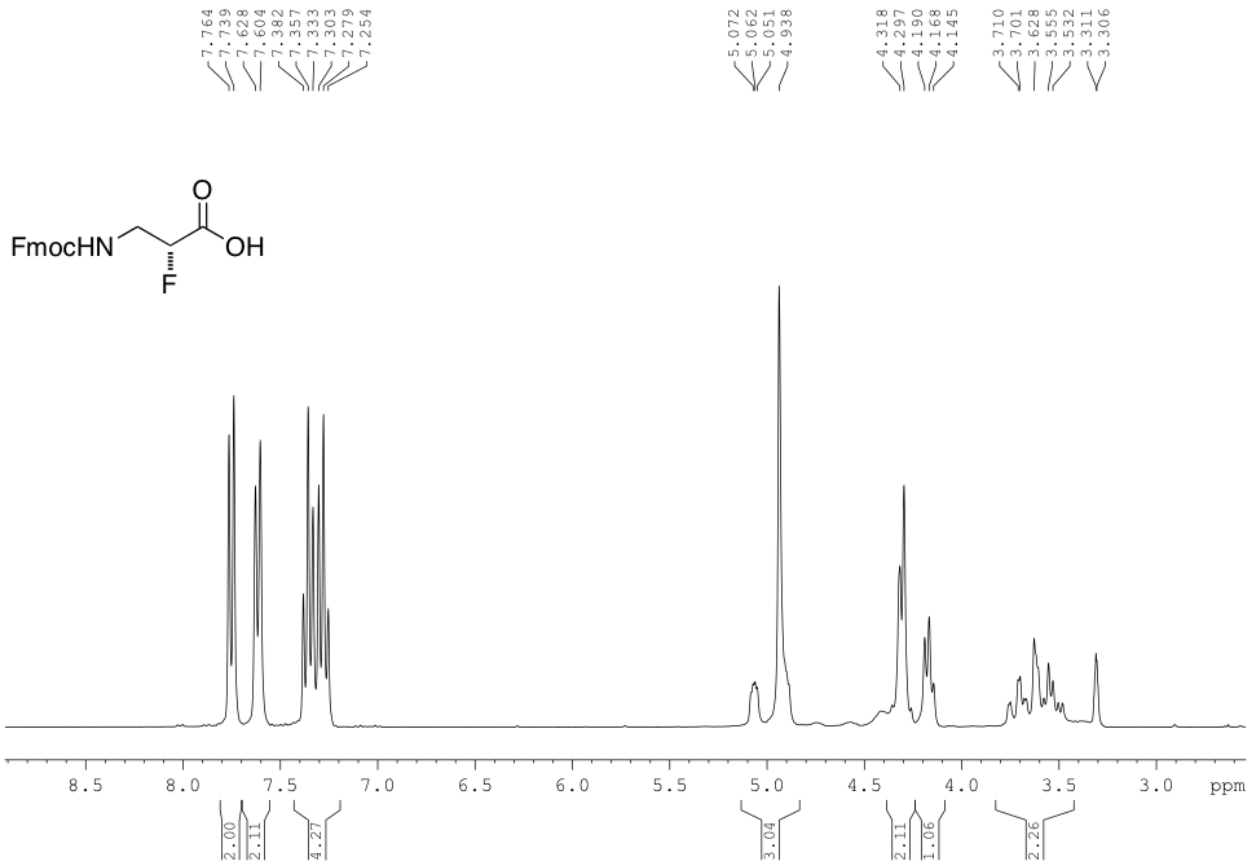
Solid phase synthesis of peptides containing backbone-fluorinated amino acids

Luke Hunter, Sharon Butler and Steve Ludbrook*

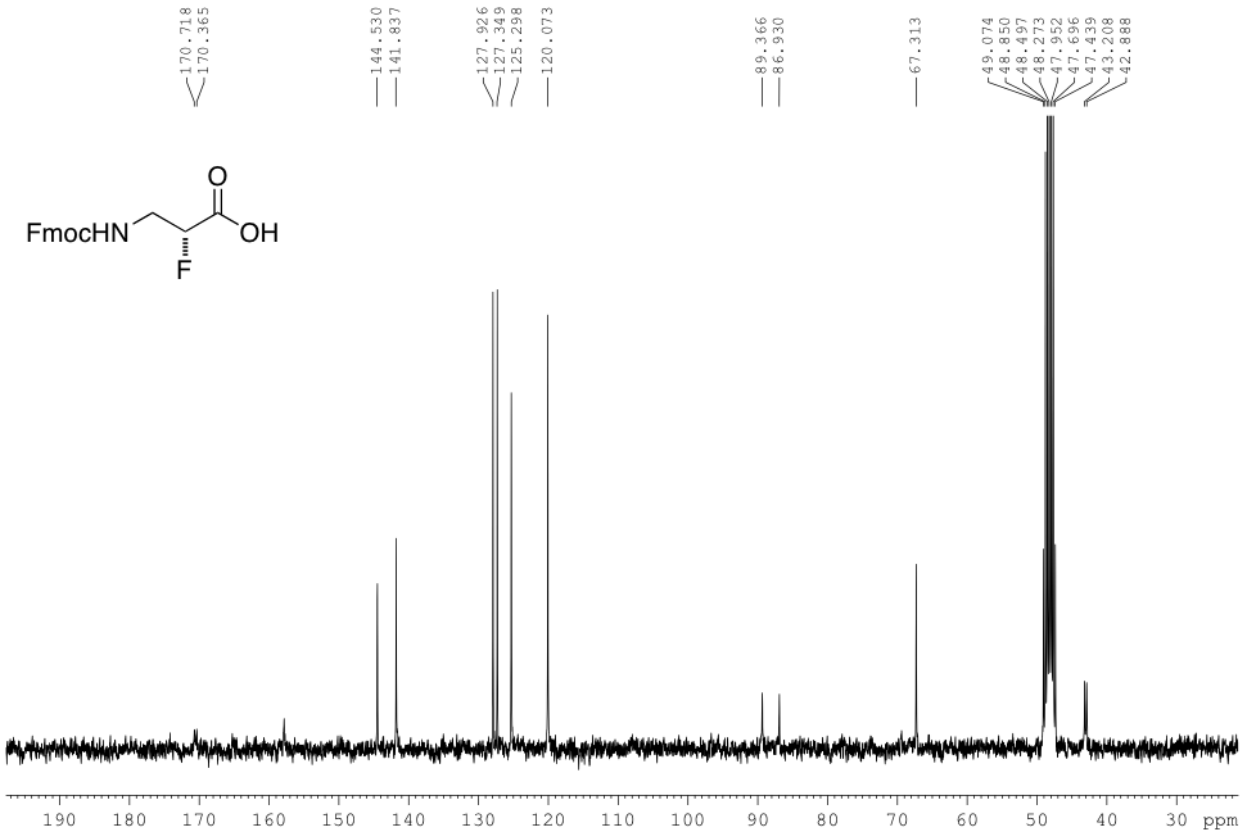
SUPPORTING INFORMATION

Contents

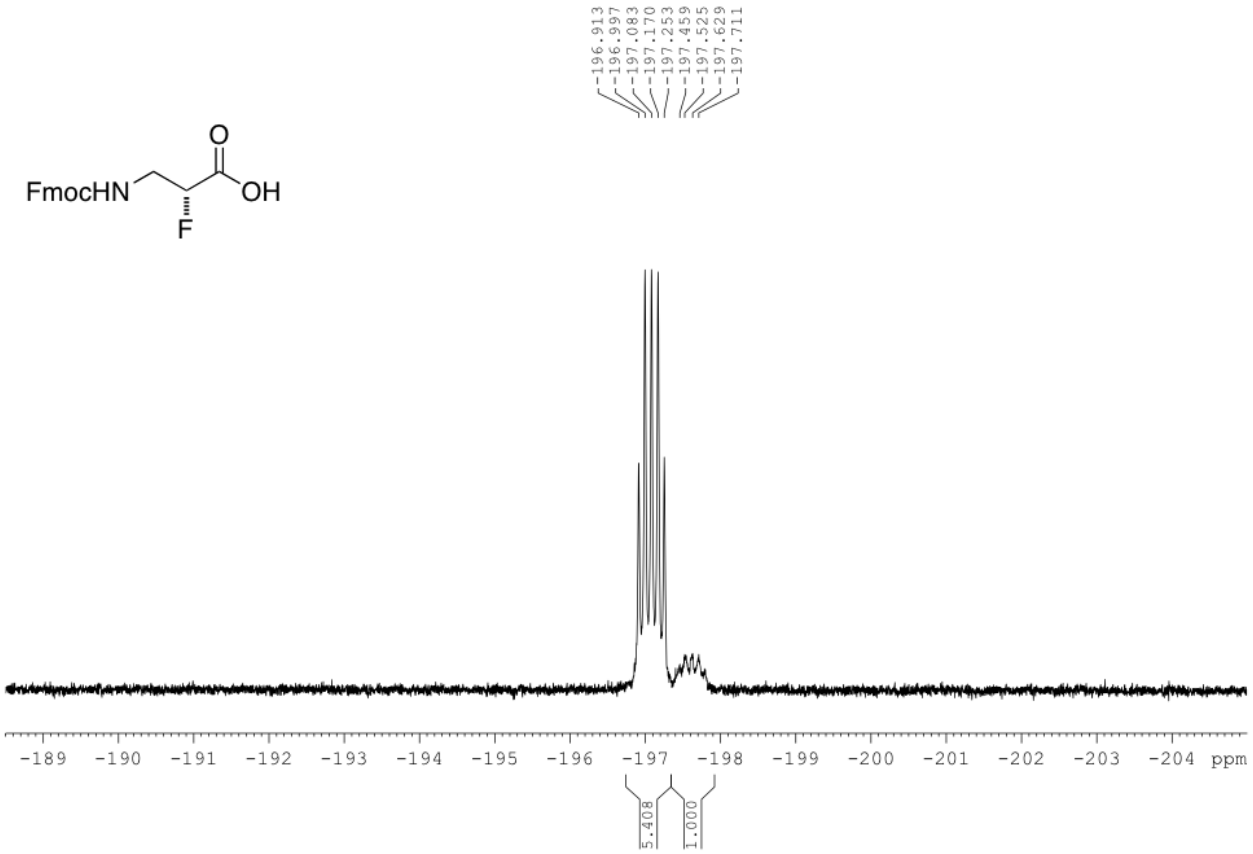
^{1}H NMR (300 MHz, MeOD) of **7**



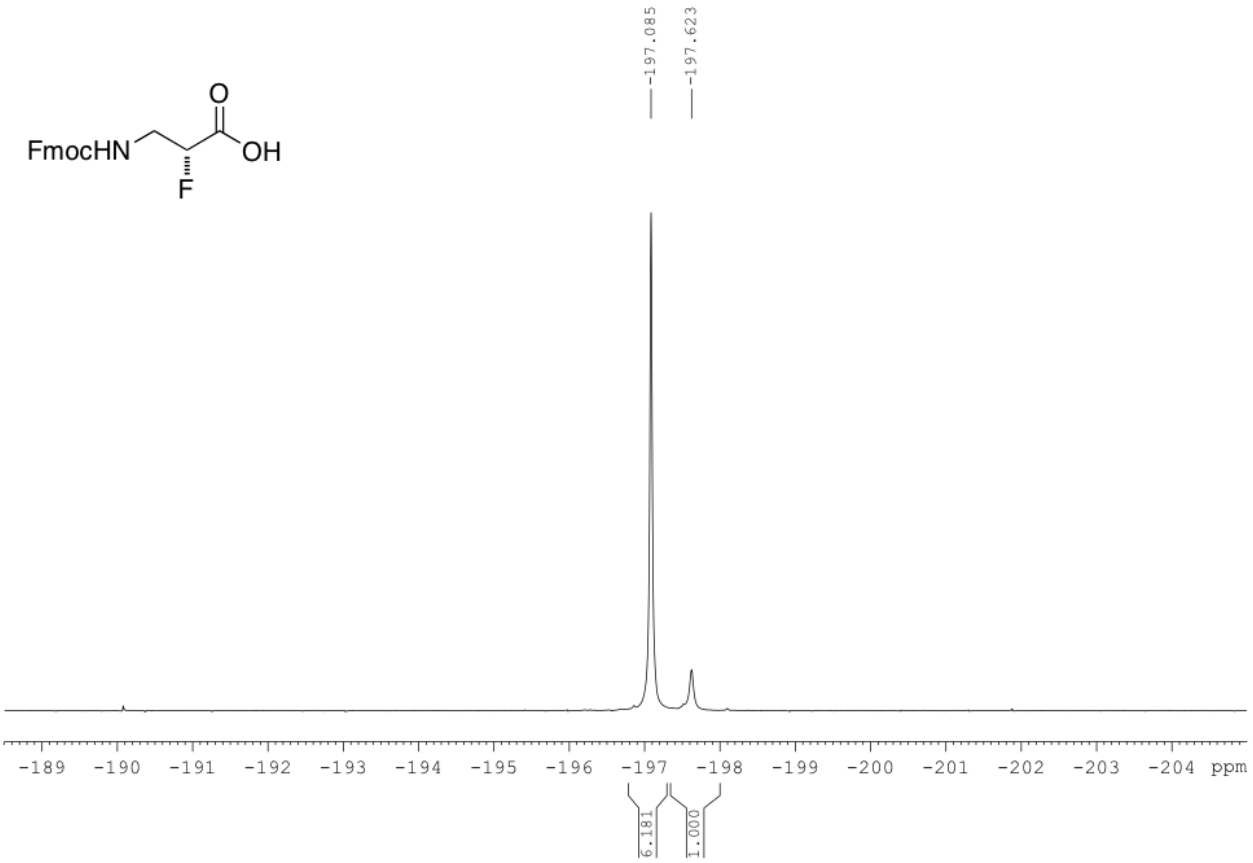
^{13}C {^{1}H} NMR (75 MHz, MeOD) of **7**



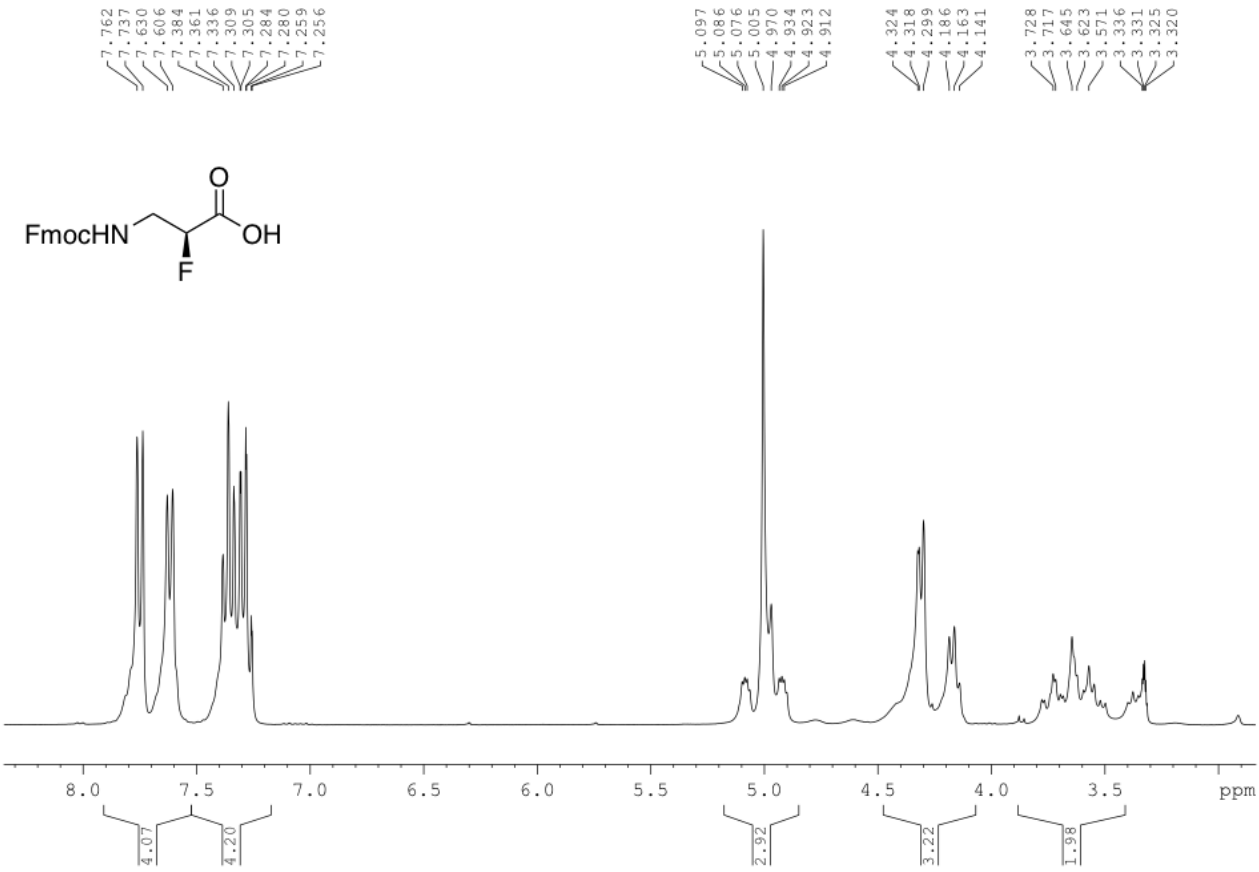
^{19}F NMR (282 MHz, MeOD) of **7**



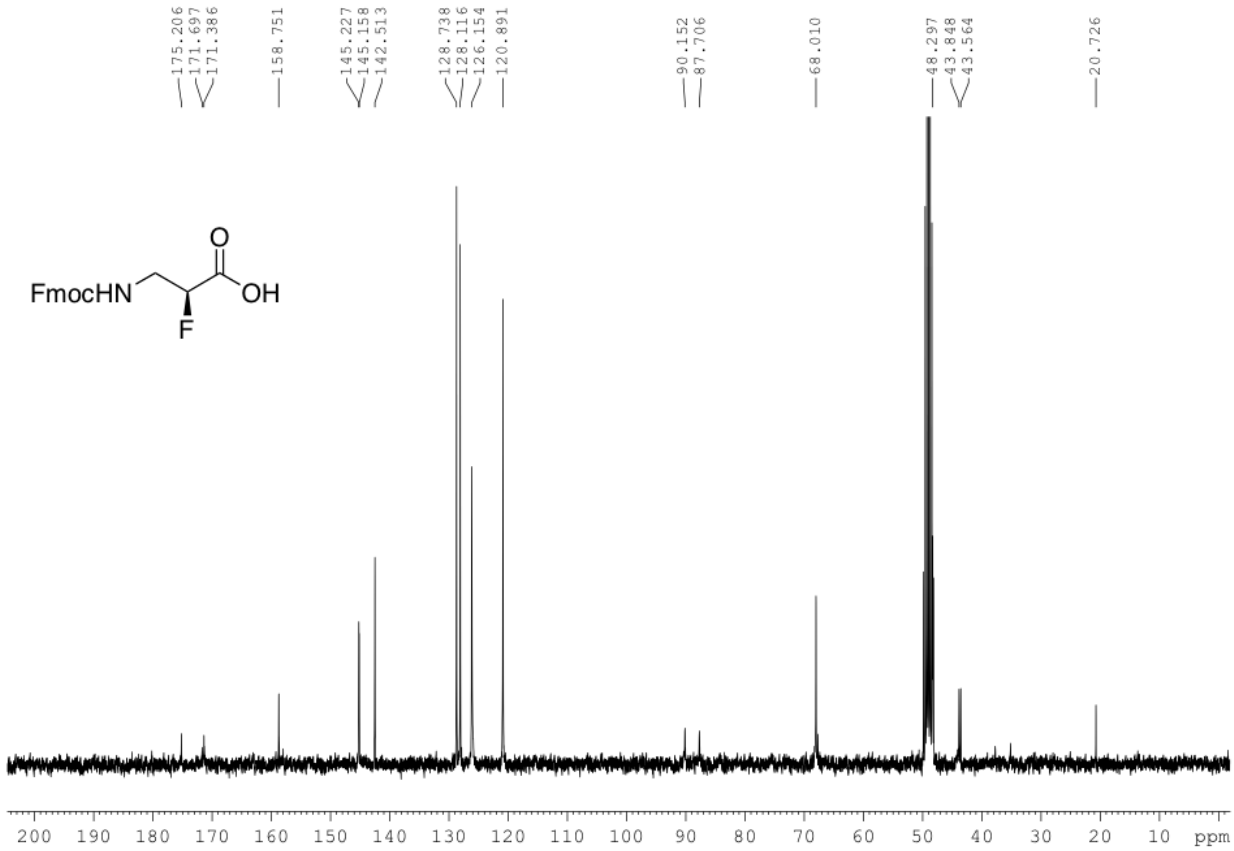
^{19}F {^{1}H} NMR (282 MHz, MeOD) of **7**



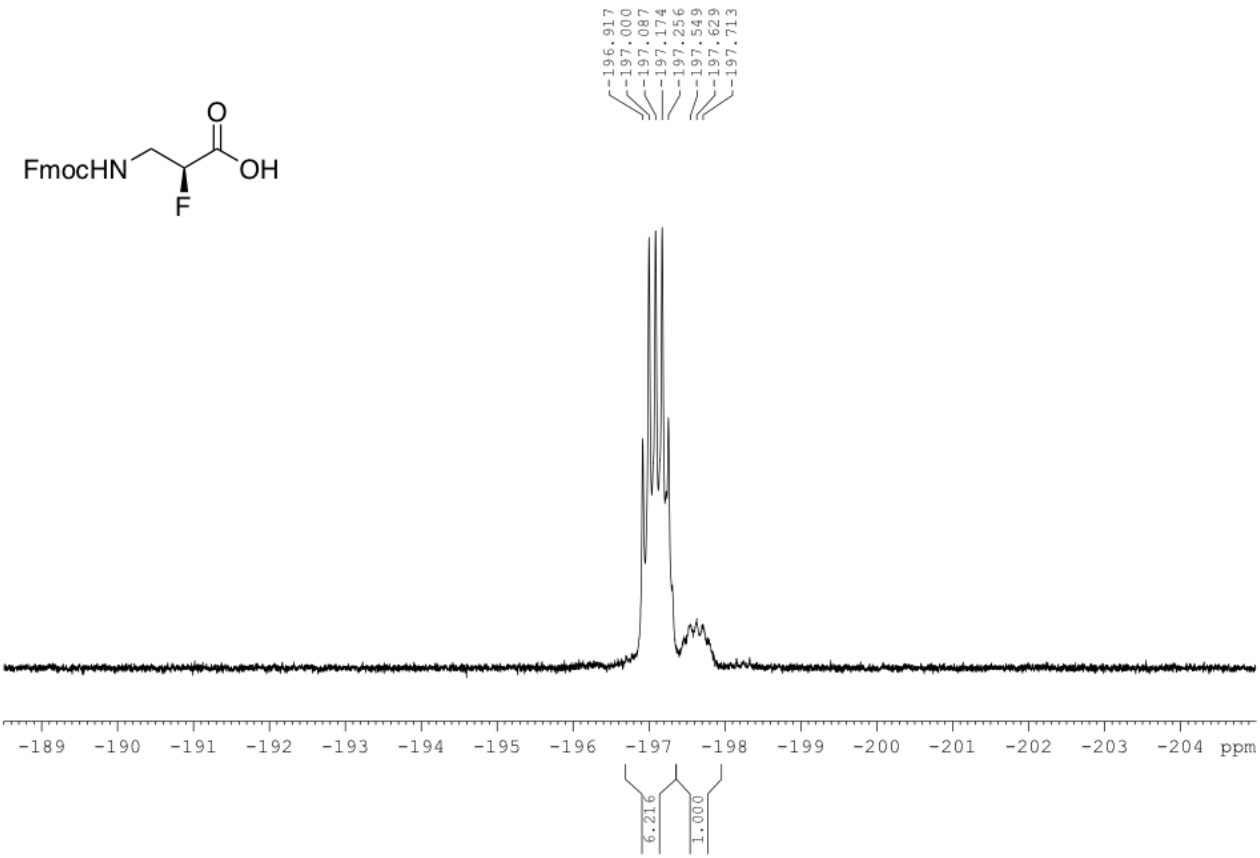
^{1}H NMR (300 MHz, MeOD) of ***ent*-7**



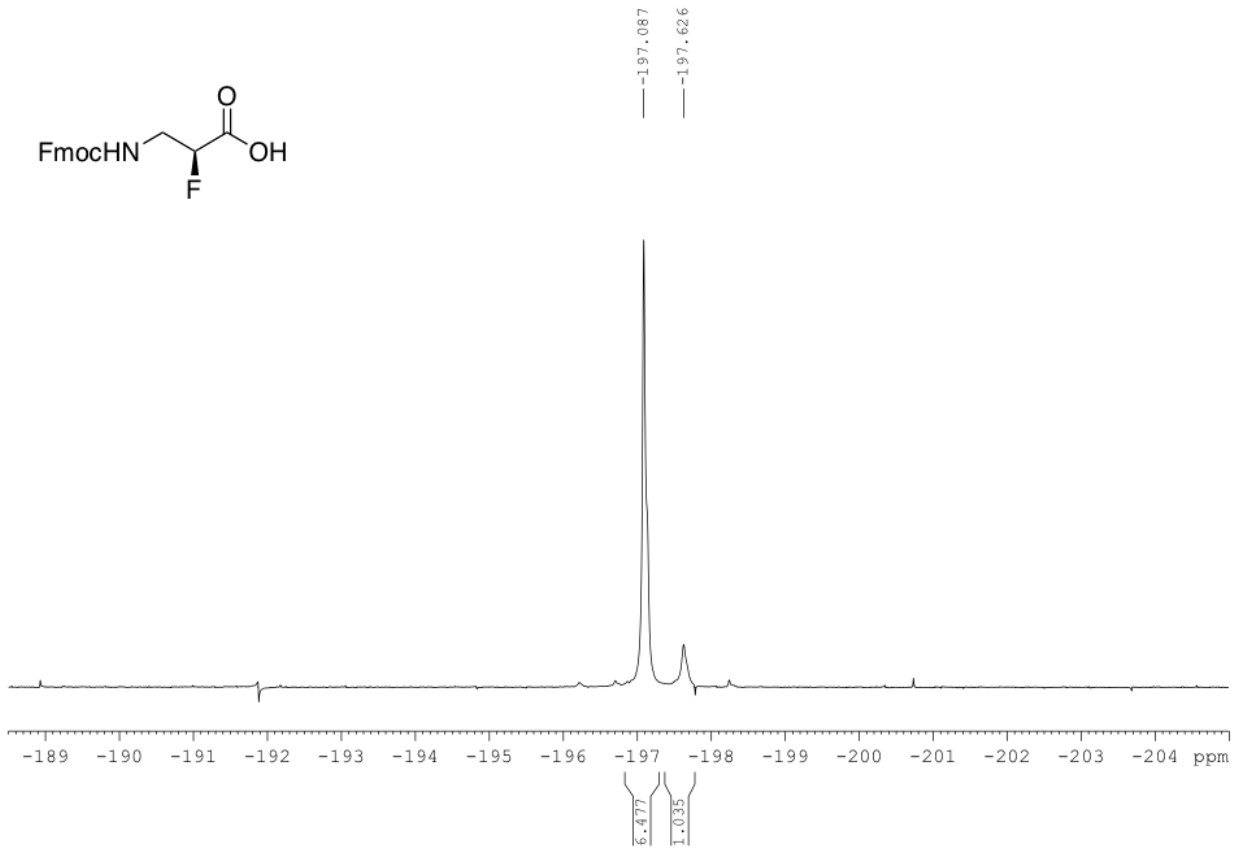
^{13}C {^{1}H} NMR (75 MHz, MeOD) of ***ent*-7**



^{19}F NMR (282 MHz, MeOD) of ***ent*-7**



^{19}F {^{1}H} NMR (282 MHz, MeOD) of ***ent*-7**



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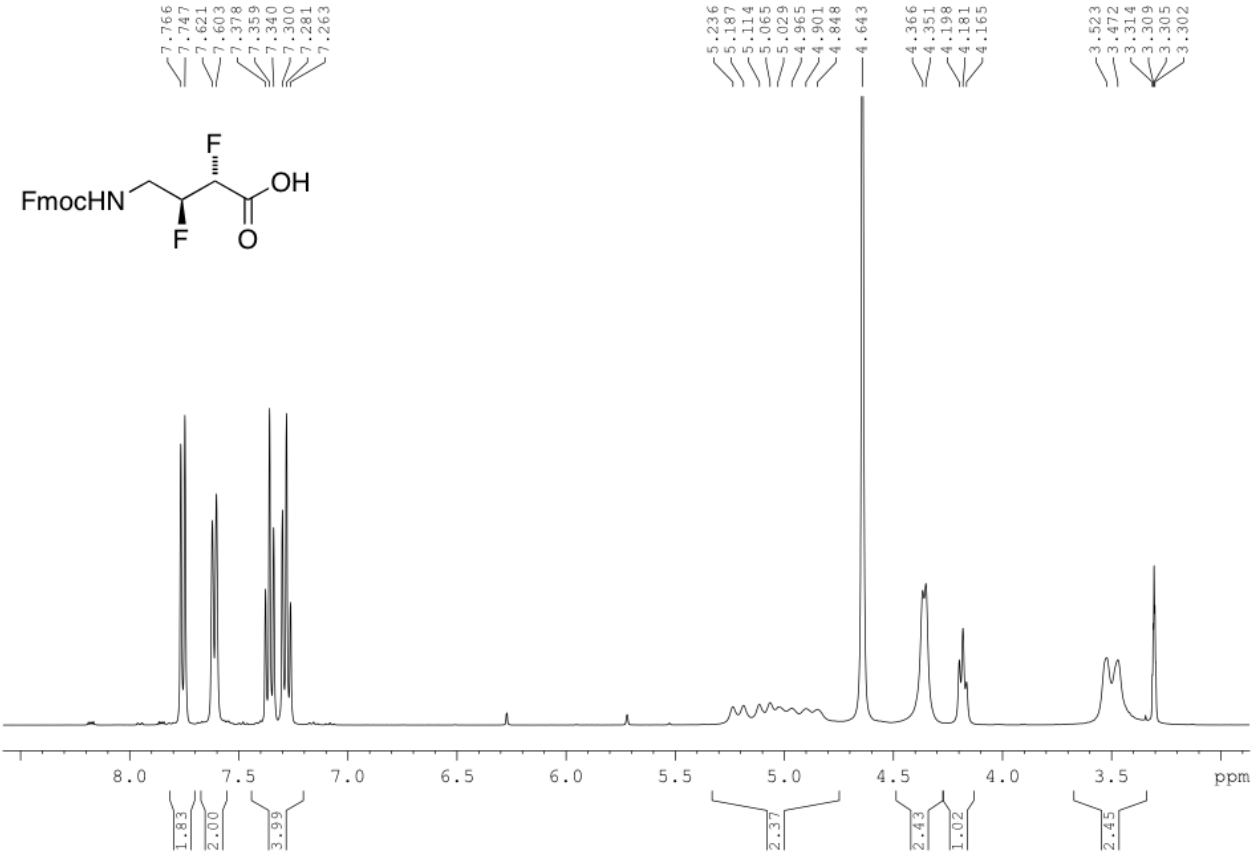

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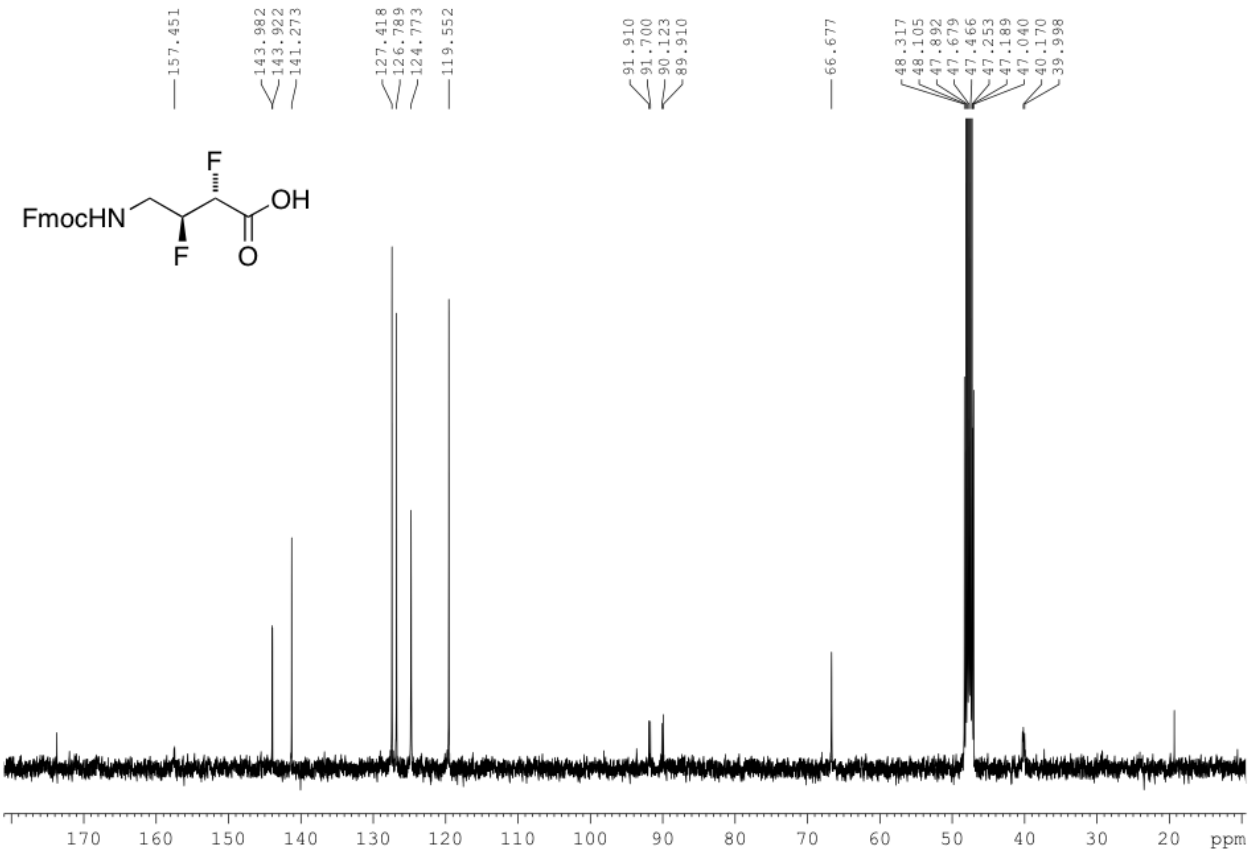
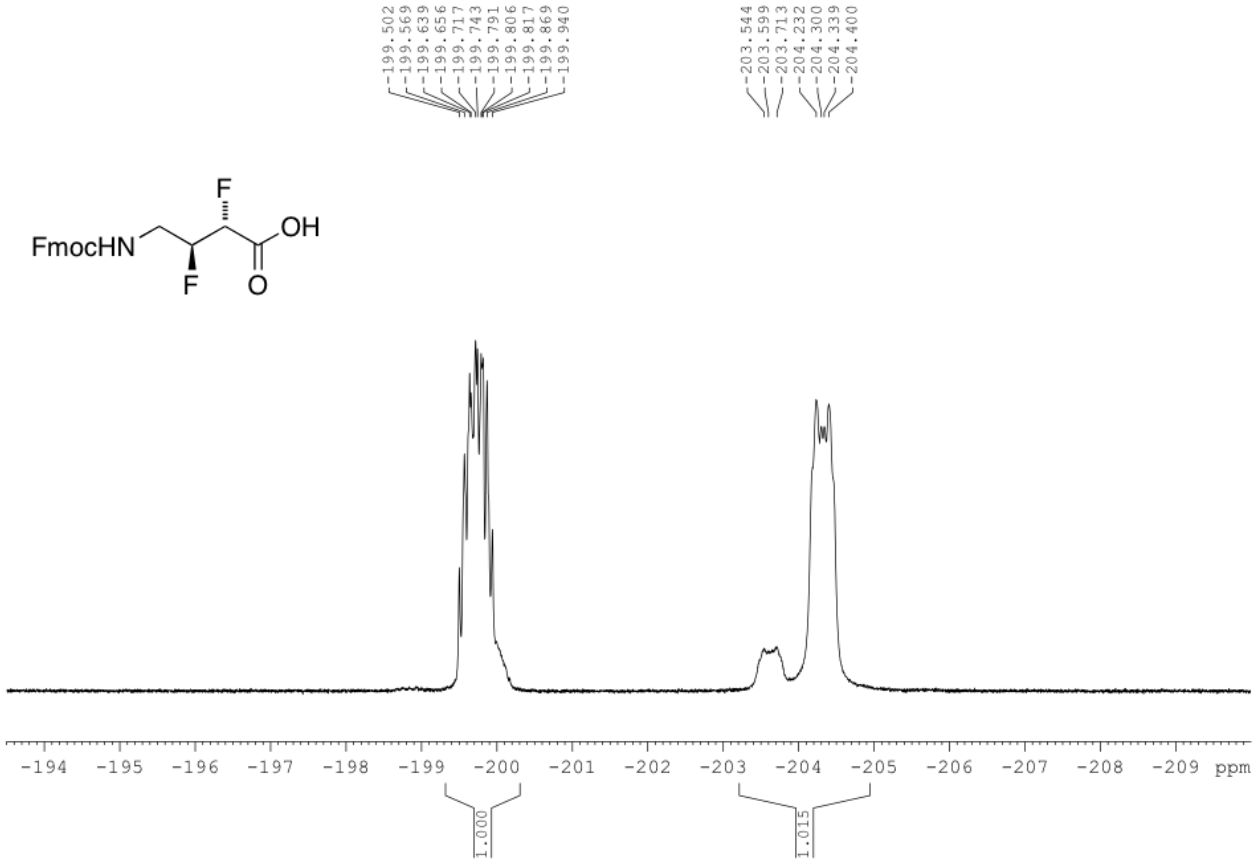


^{19}F NMR (282 MHz, MeOD) of **9**



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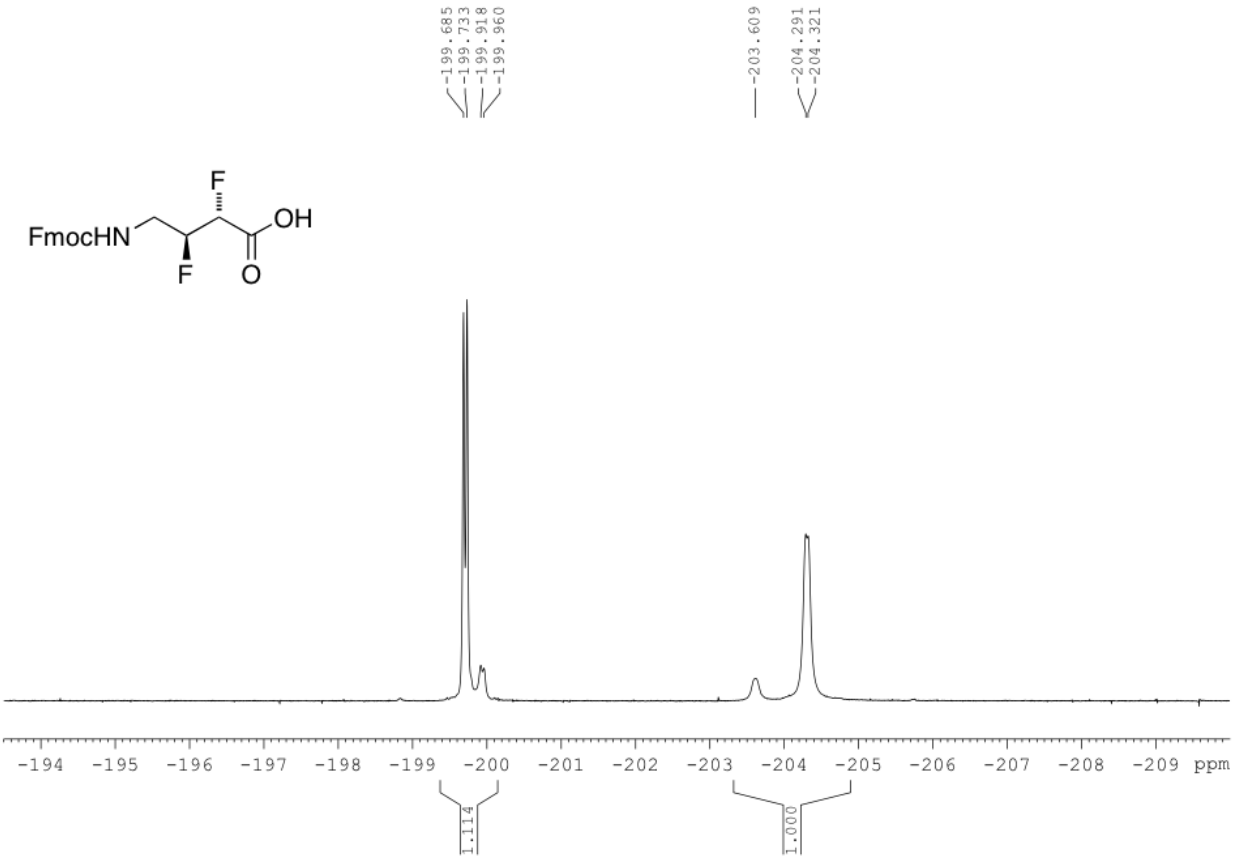
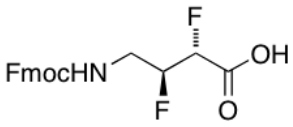
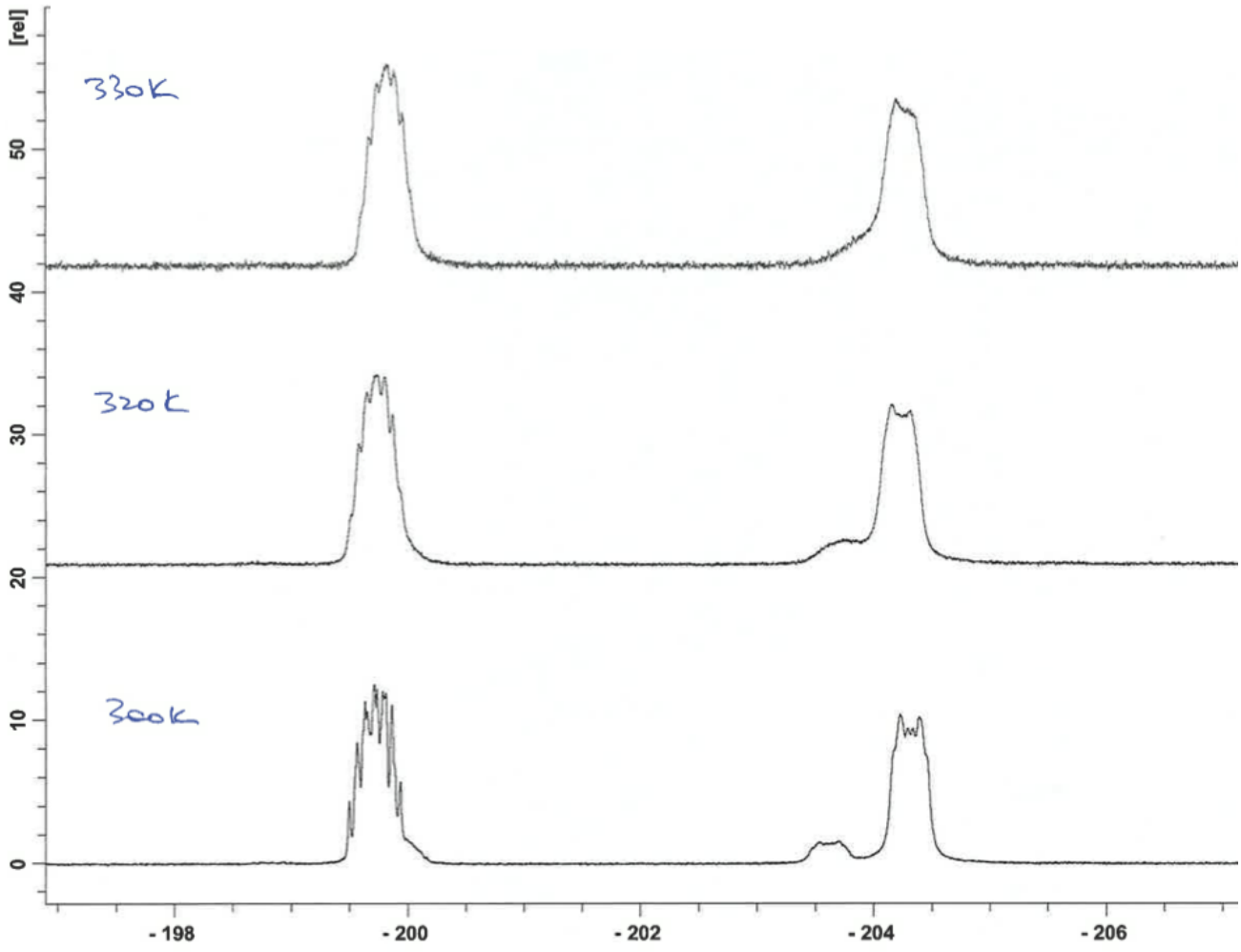
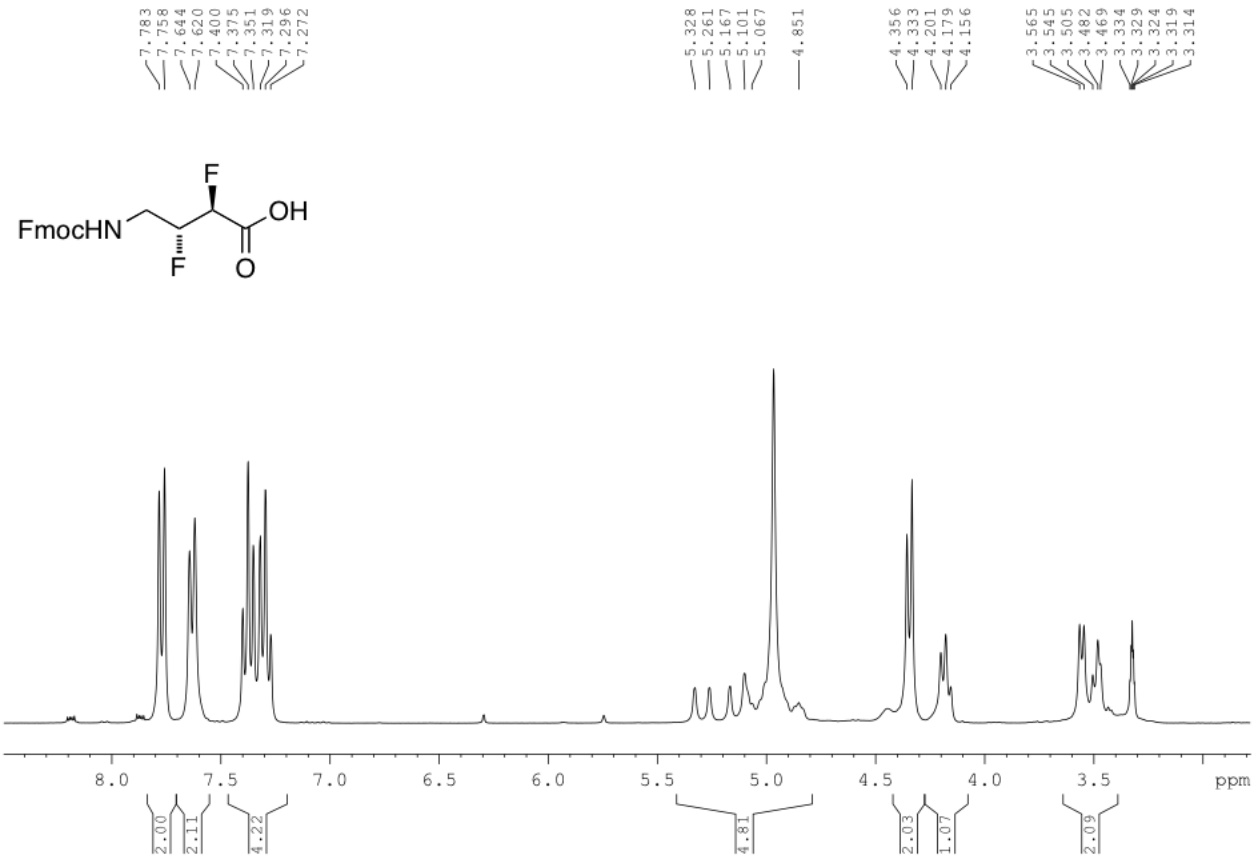


Variable-temperature ^{19}**F NMR** (282 MHz, MeOD) of **9**

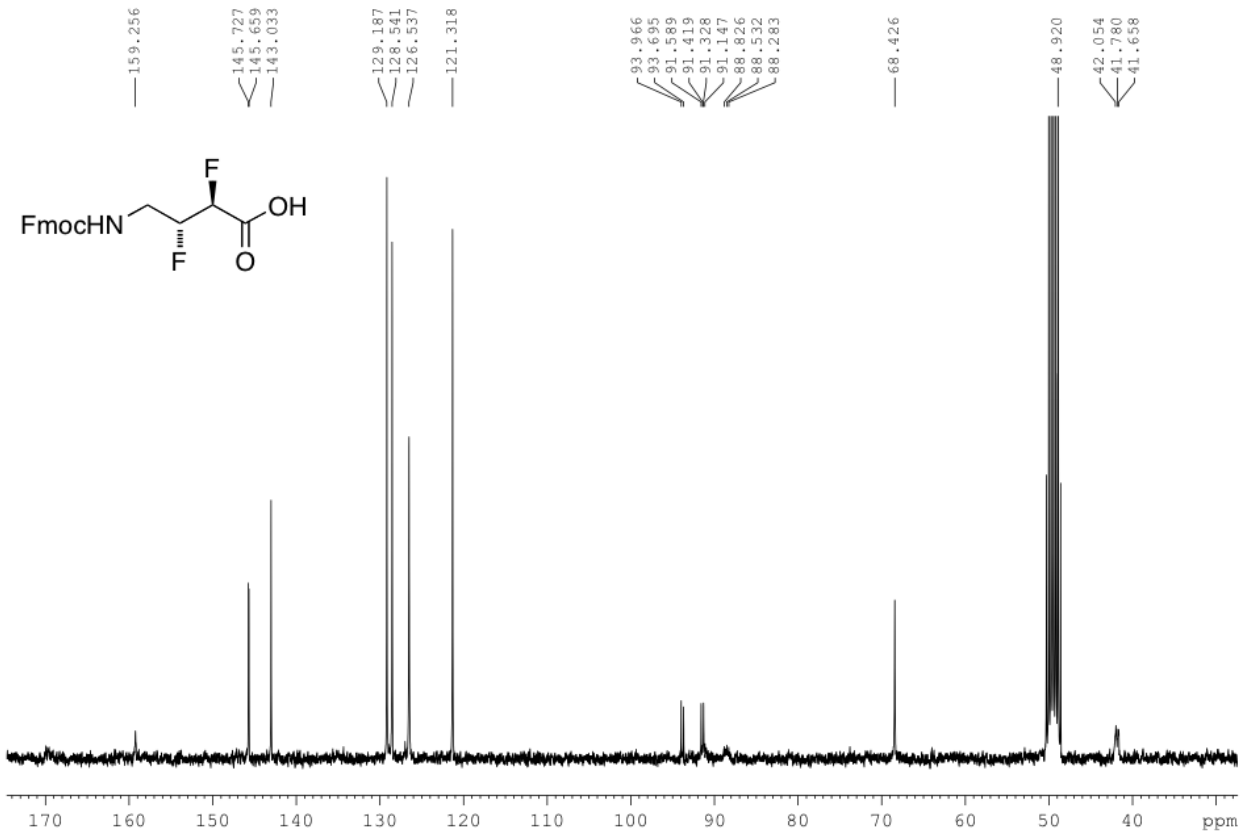




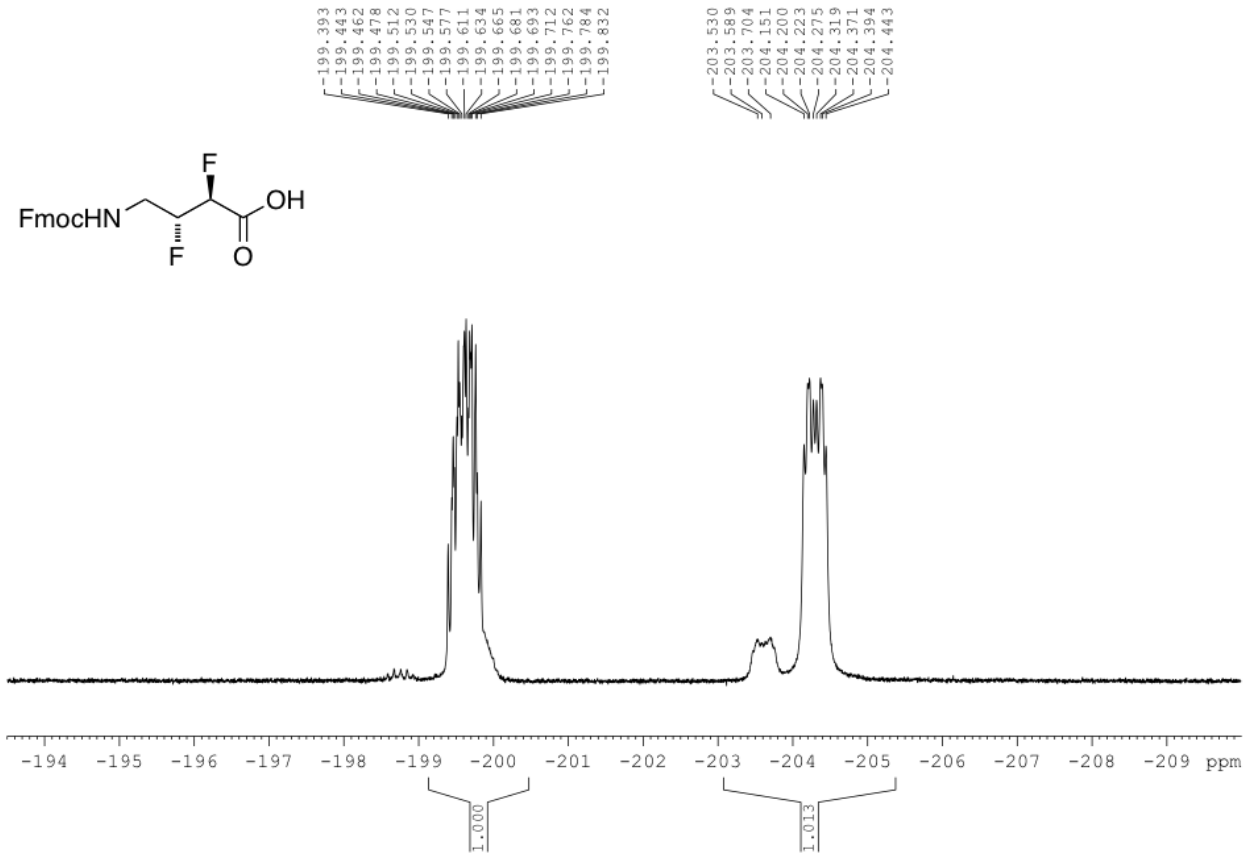
^{1}H NMR (300 MHz, MeOD) of ***ent*-9**



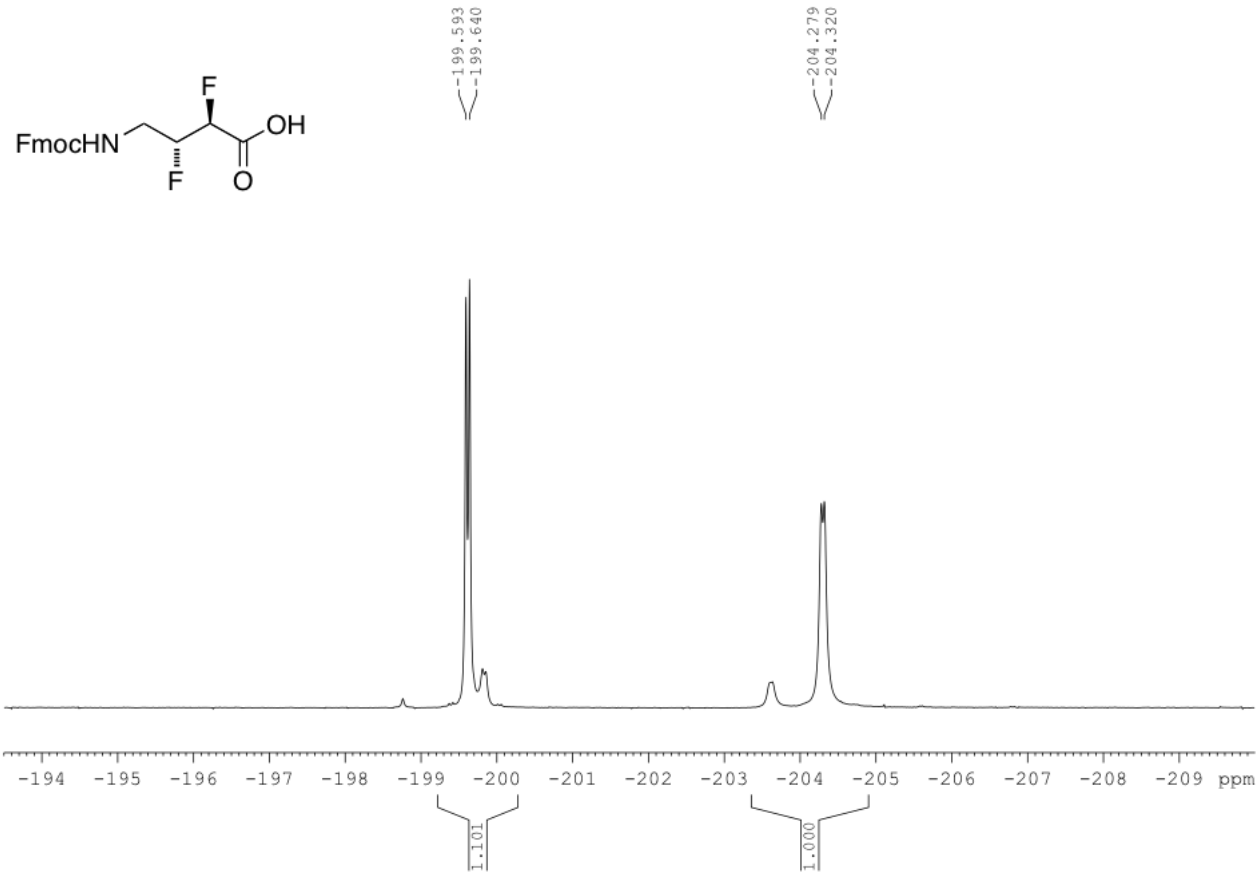
^{13}C {^{1}H} NMR (75 MHz, MeOD) of ***ent*-9**



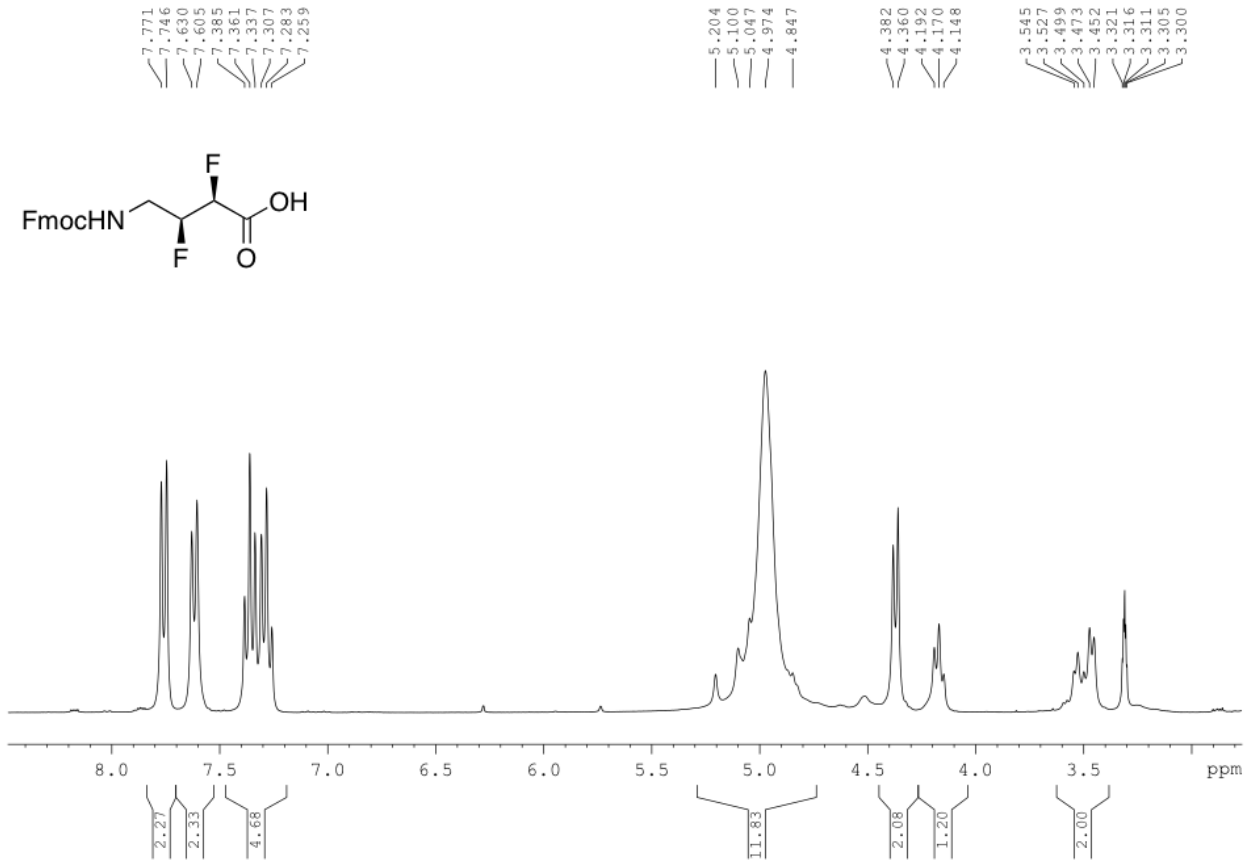
^{19}F NMR (282 MHz, MeOD) of ***ent*-9**



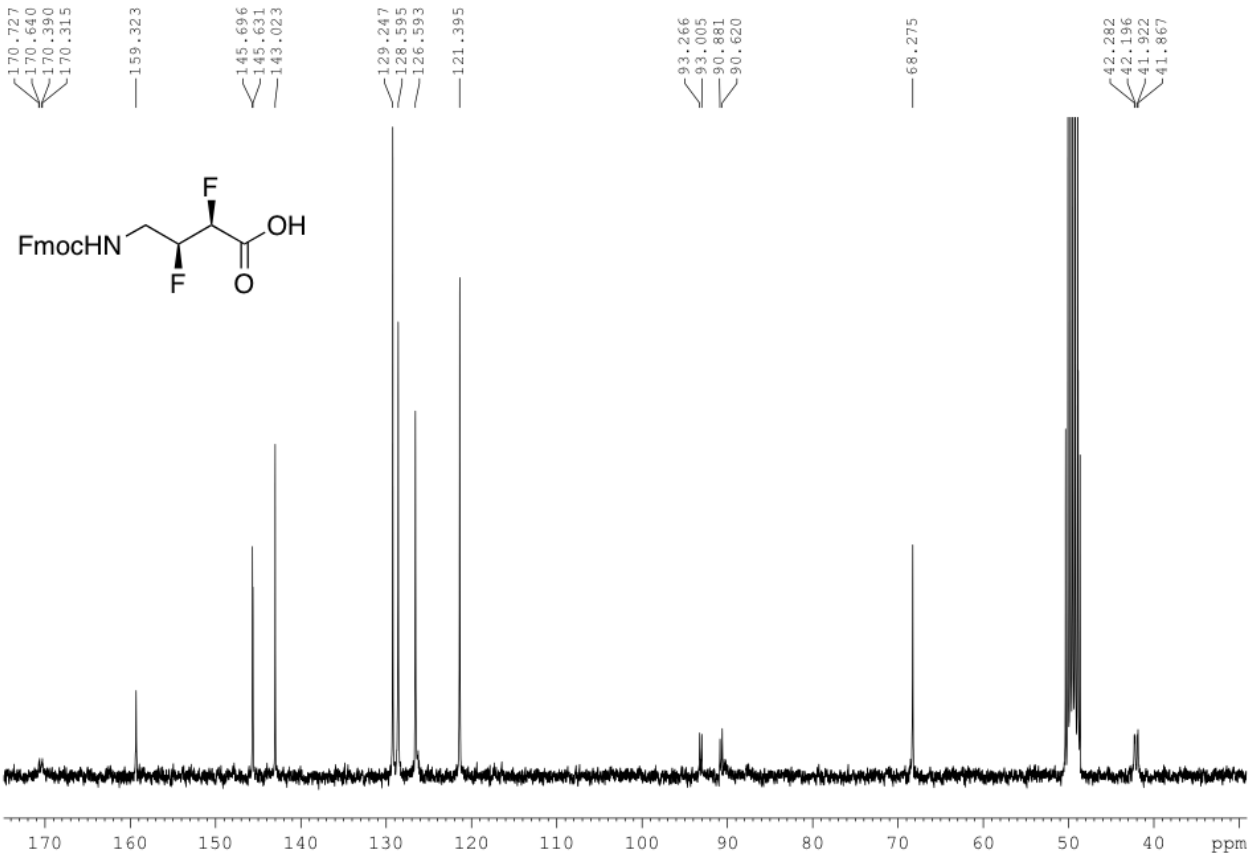
^{19}F {^{1}H} NMR (282 MHz, MeOD) of ***ent*-9**



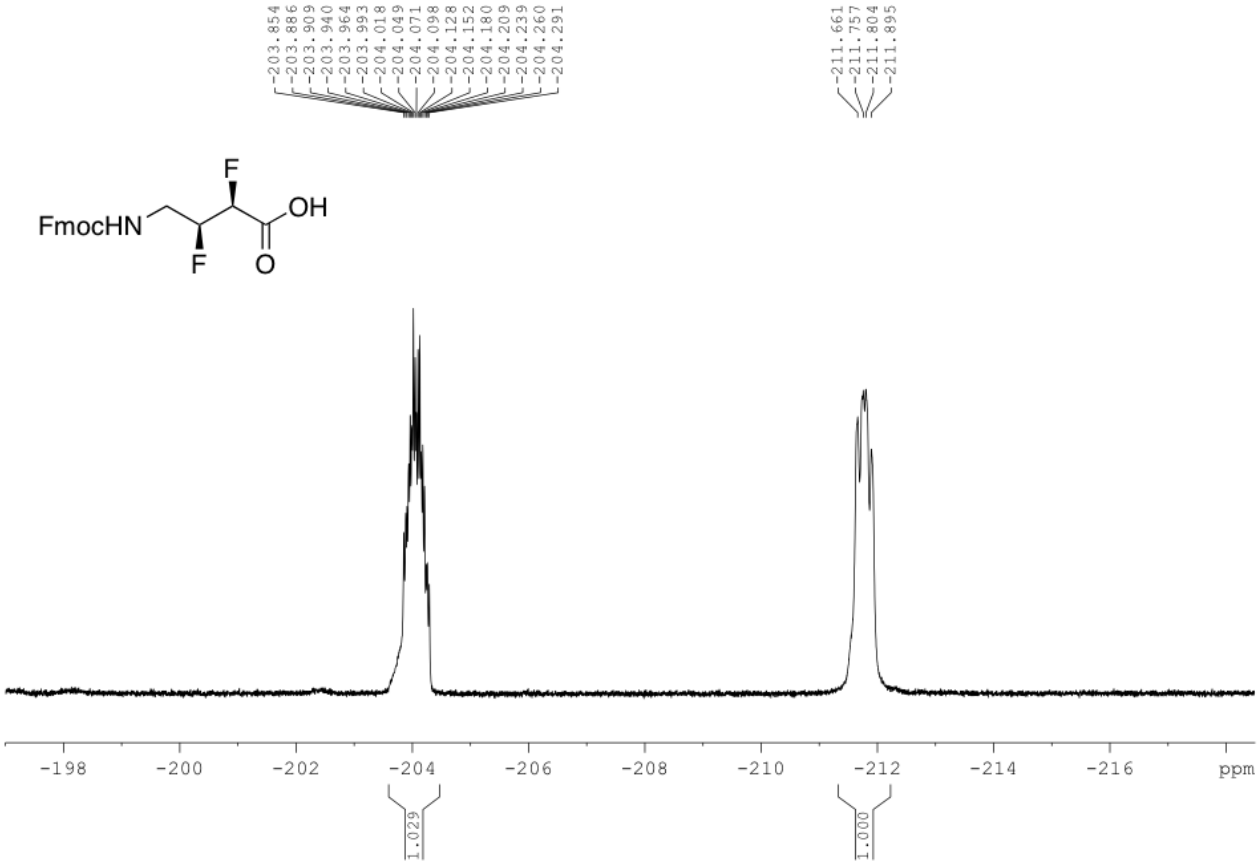
^{1}H NMR (300 MHz, MeOD) of **10**



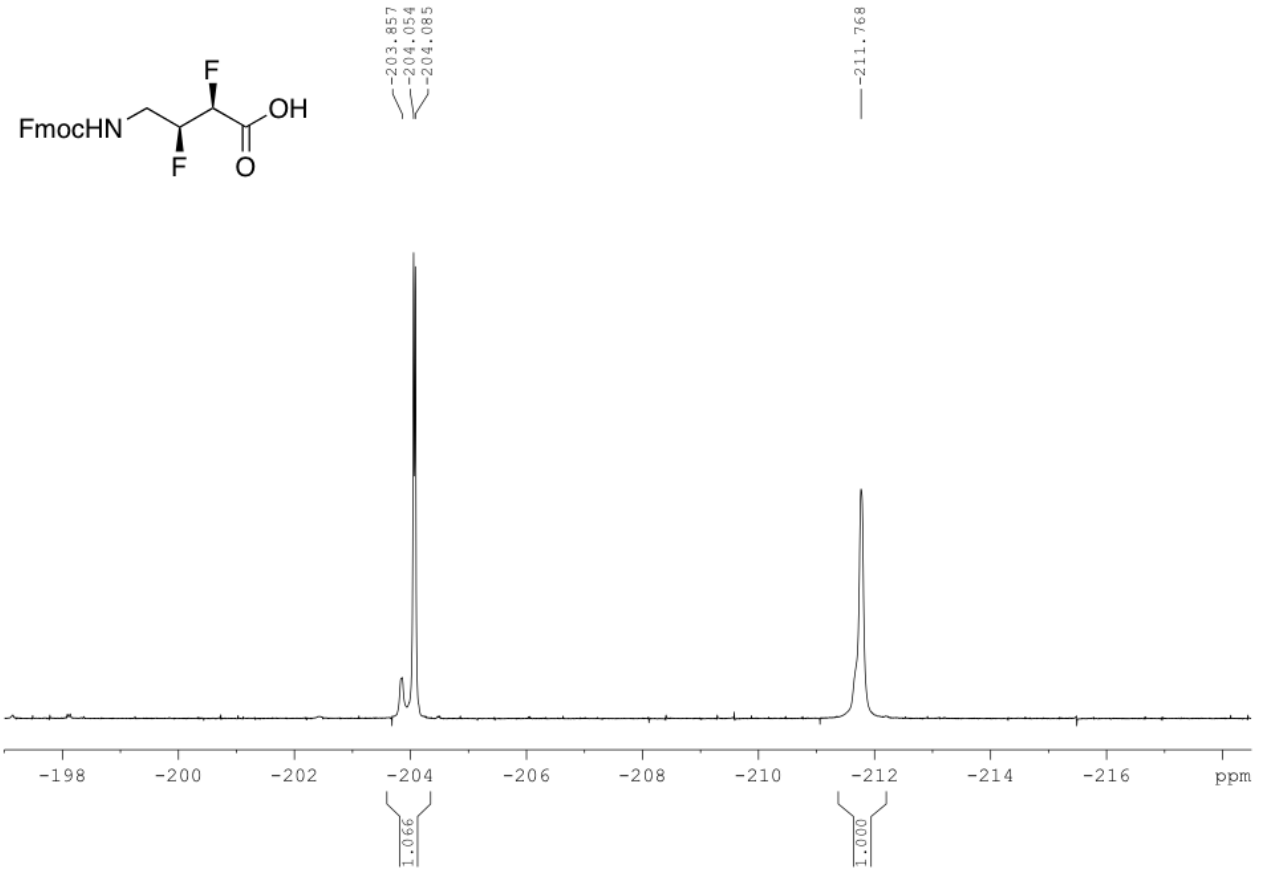
^{13}C {^{1}H} NMR (75 MHz, MeOD) of **10**



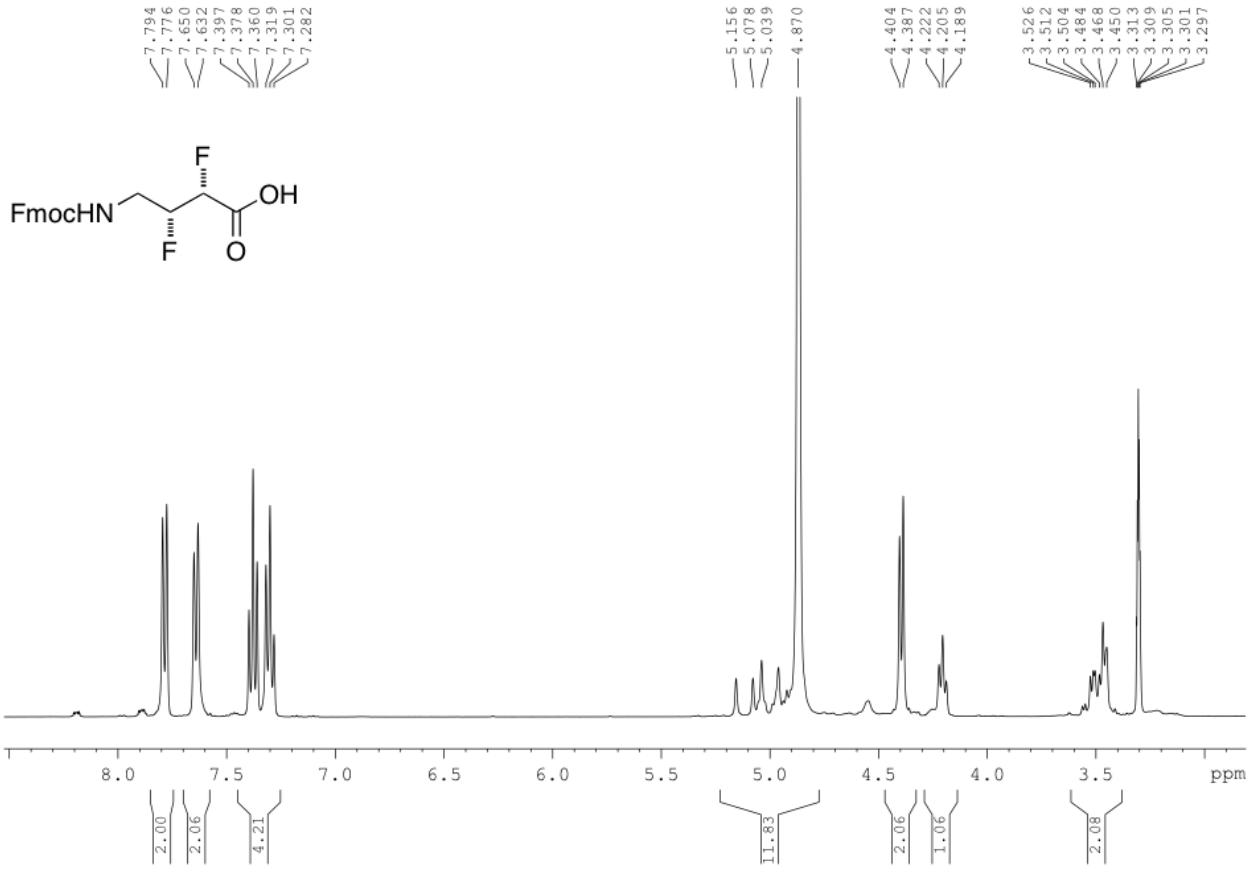
^{19}F NMR (282 MHz, MeOD) of **10**



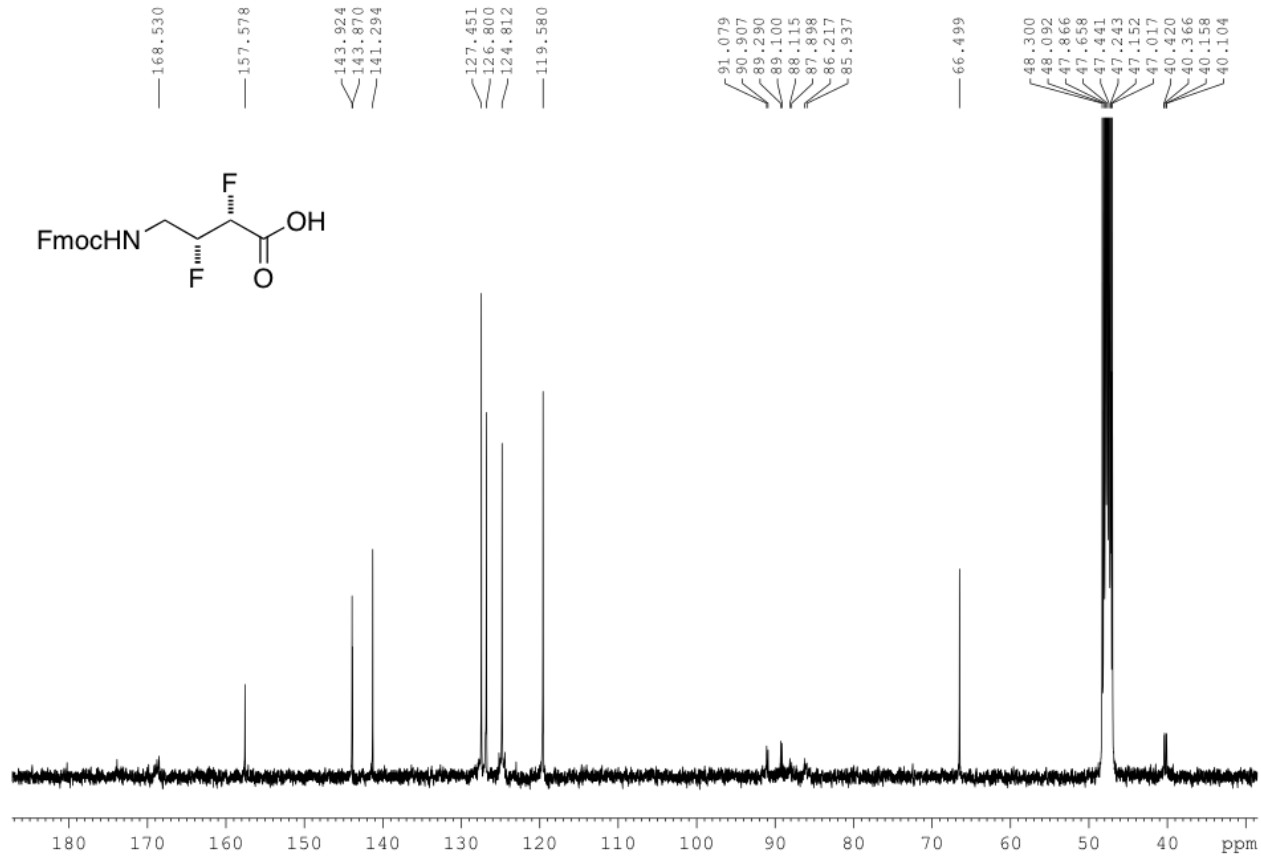
^{19}F {^{1}H} NMR (282 MHz, MeOD) of **10**



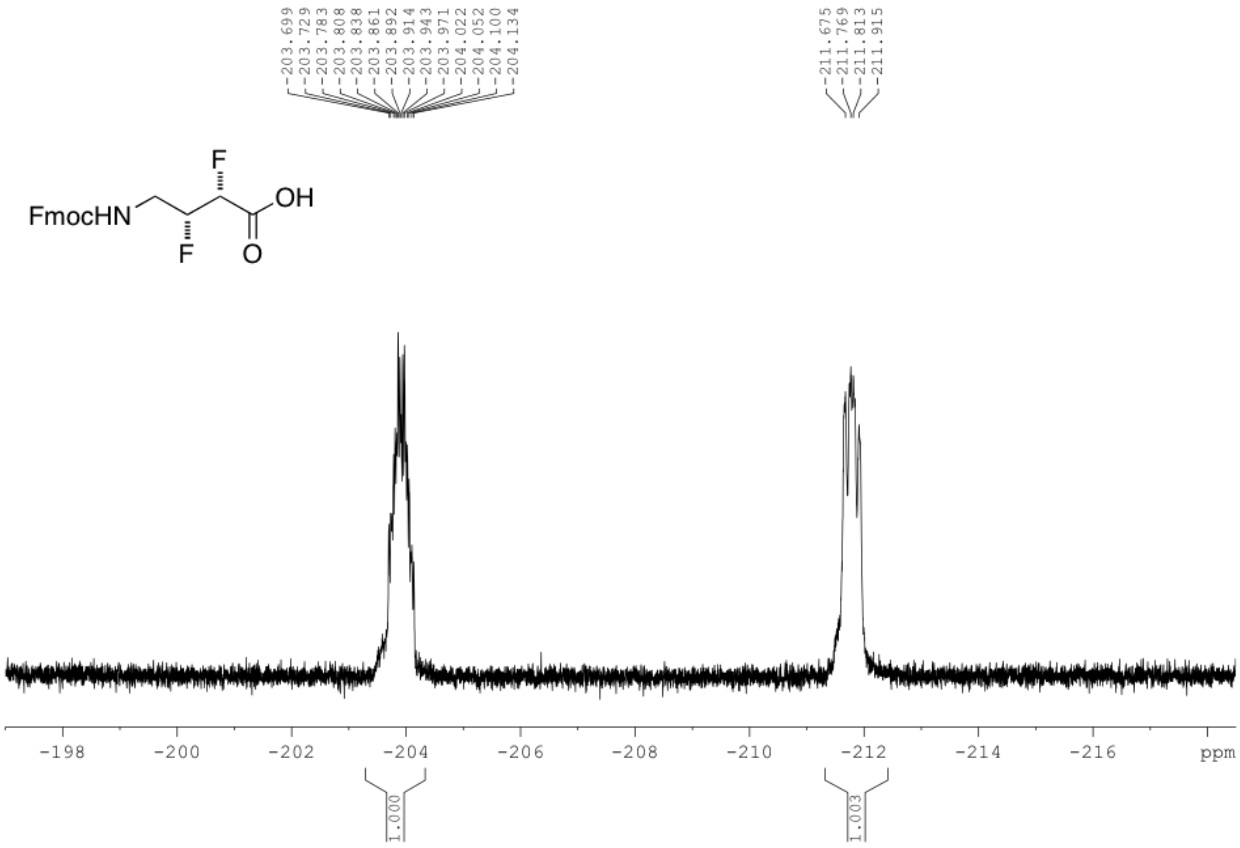
^{1}H NMR (400 MHz, MeOD) of ***ent*-10**



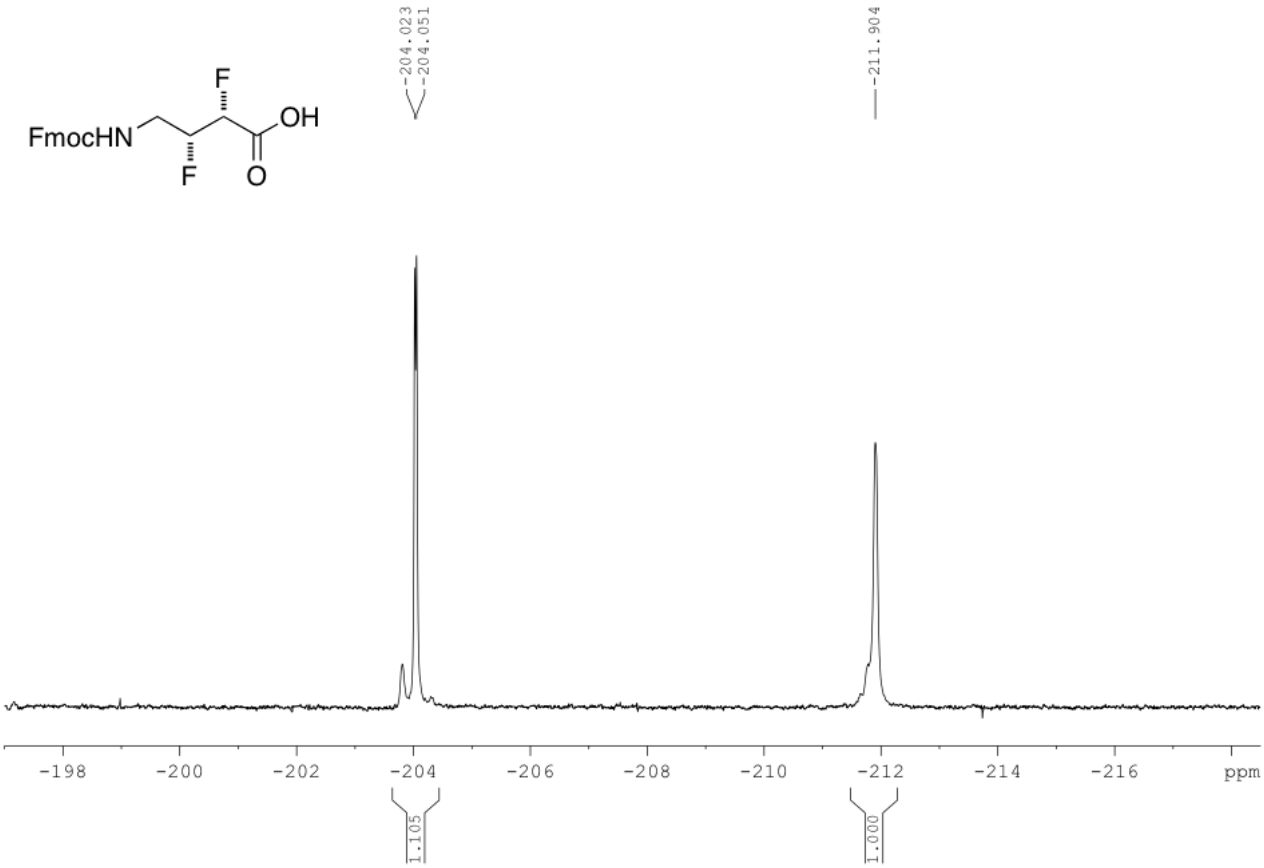
^{13}C {^{1}H} NMR (100 MHz, MeOD) of ***ent*-10**



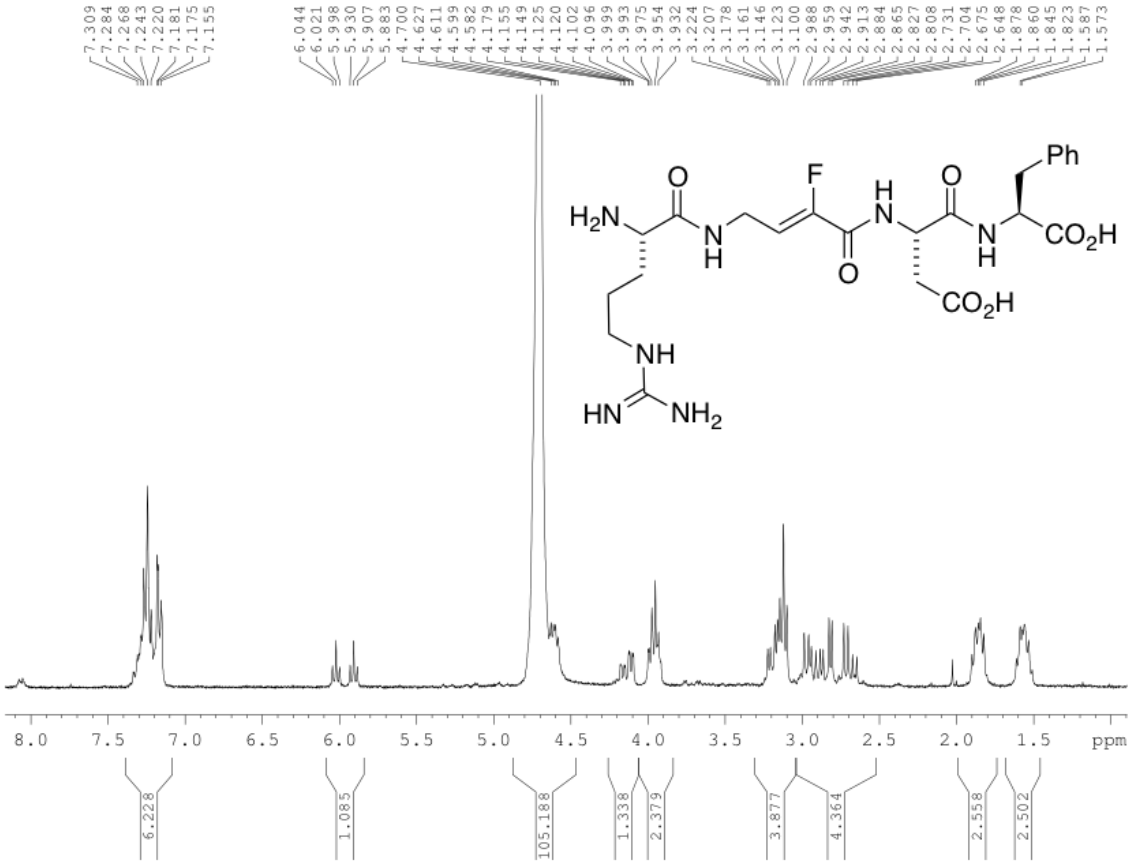
^{19}F NMR (282 MHz, MeOD) of ***ent*-10**



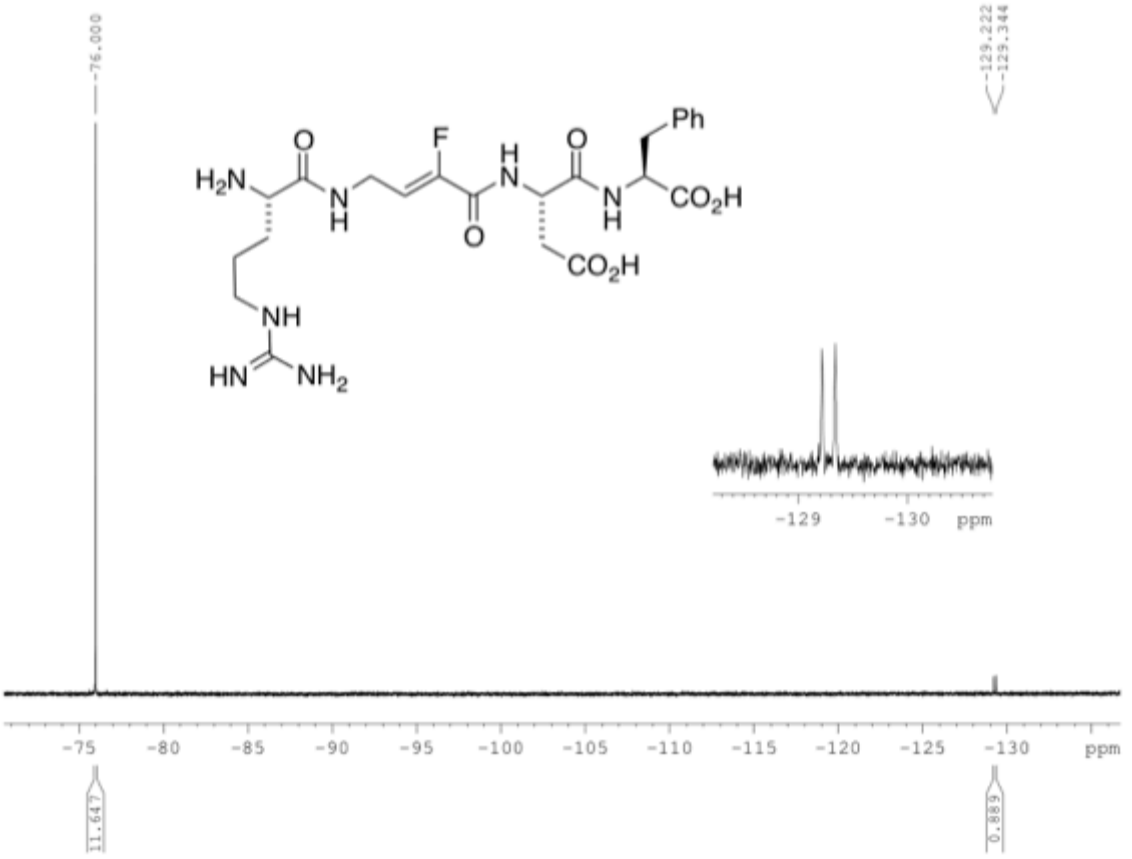
^{19}F {^{1}H} NMR (282 MHz, MeOD) of ***ent*-10**



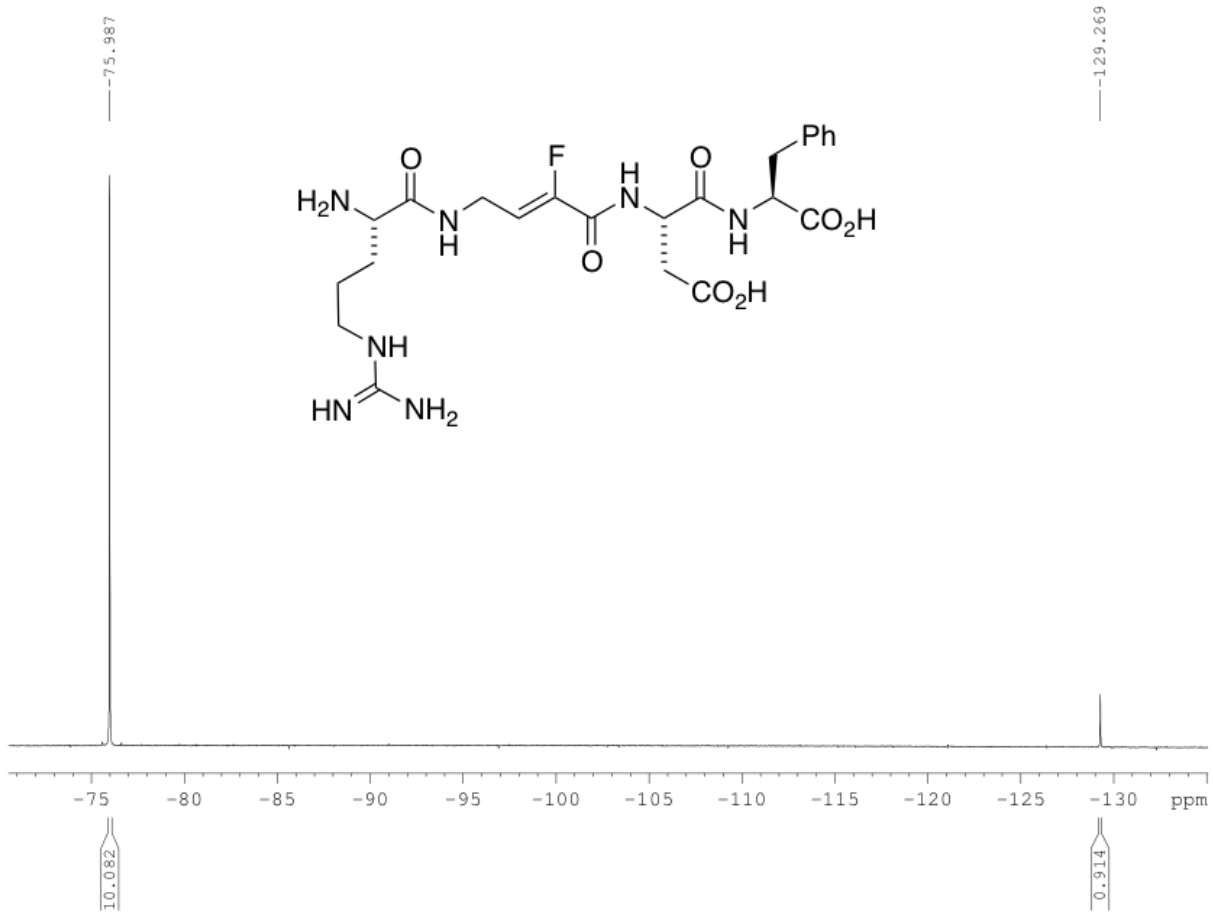
^{1}H NMR (300 MHz, D_2O) of **13**



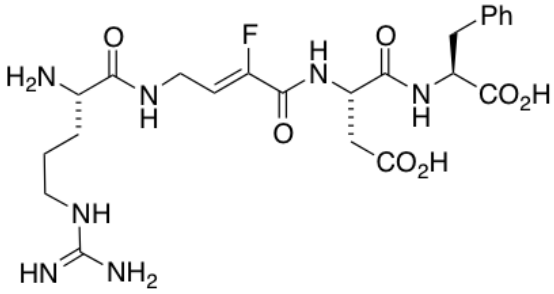
^{19}F NMR (282 MHz, D_2O) of **13**

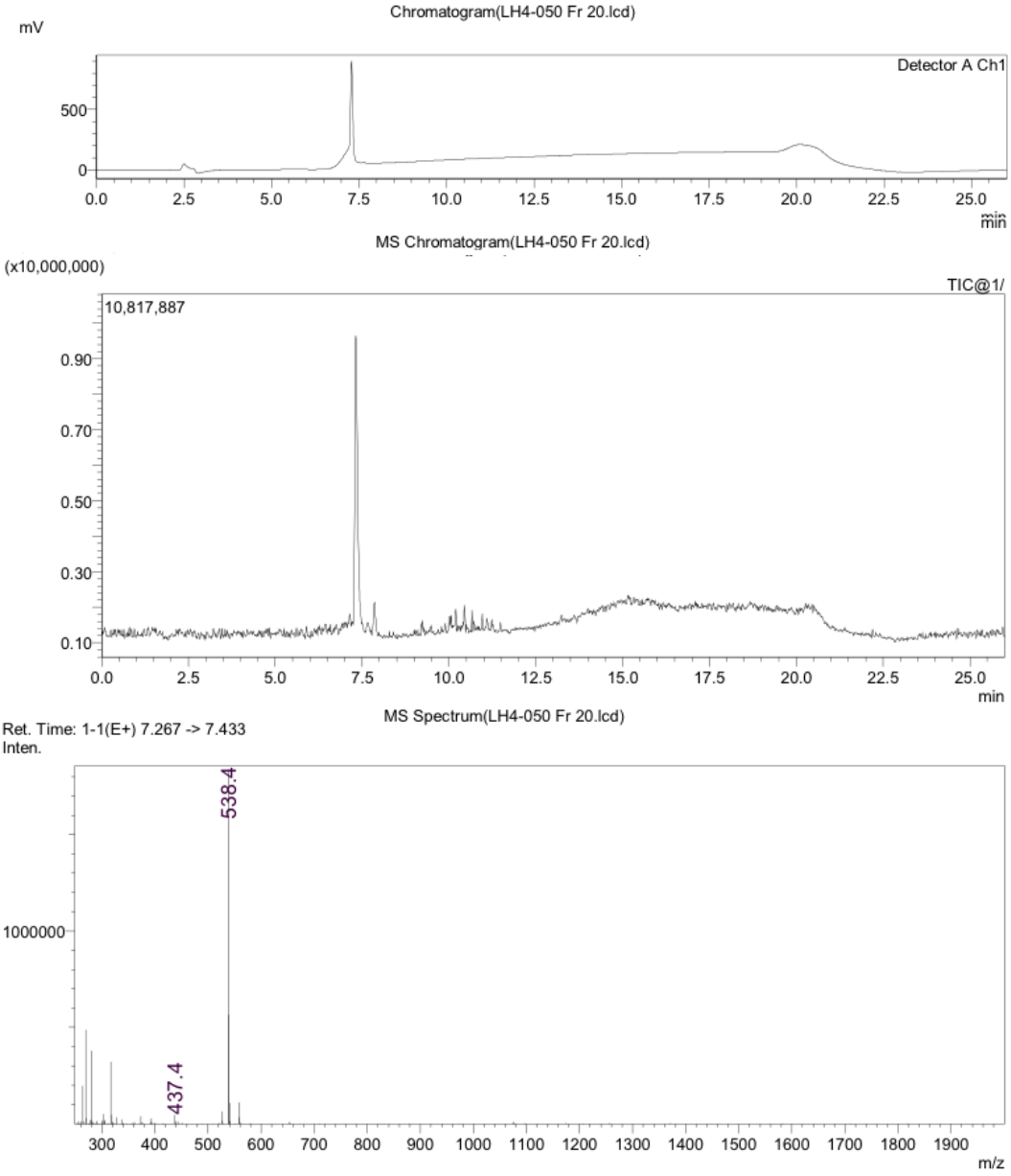


^{19}F {^{1}H} NMR (282 MHz, D_2O) of **13**

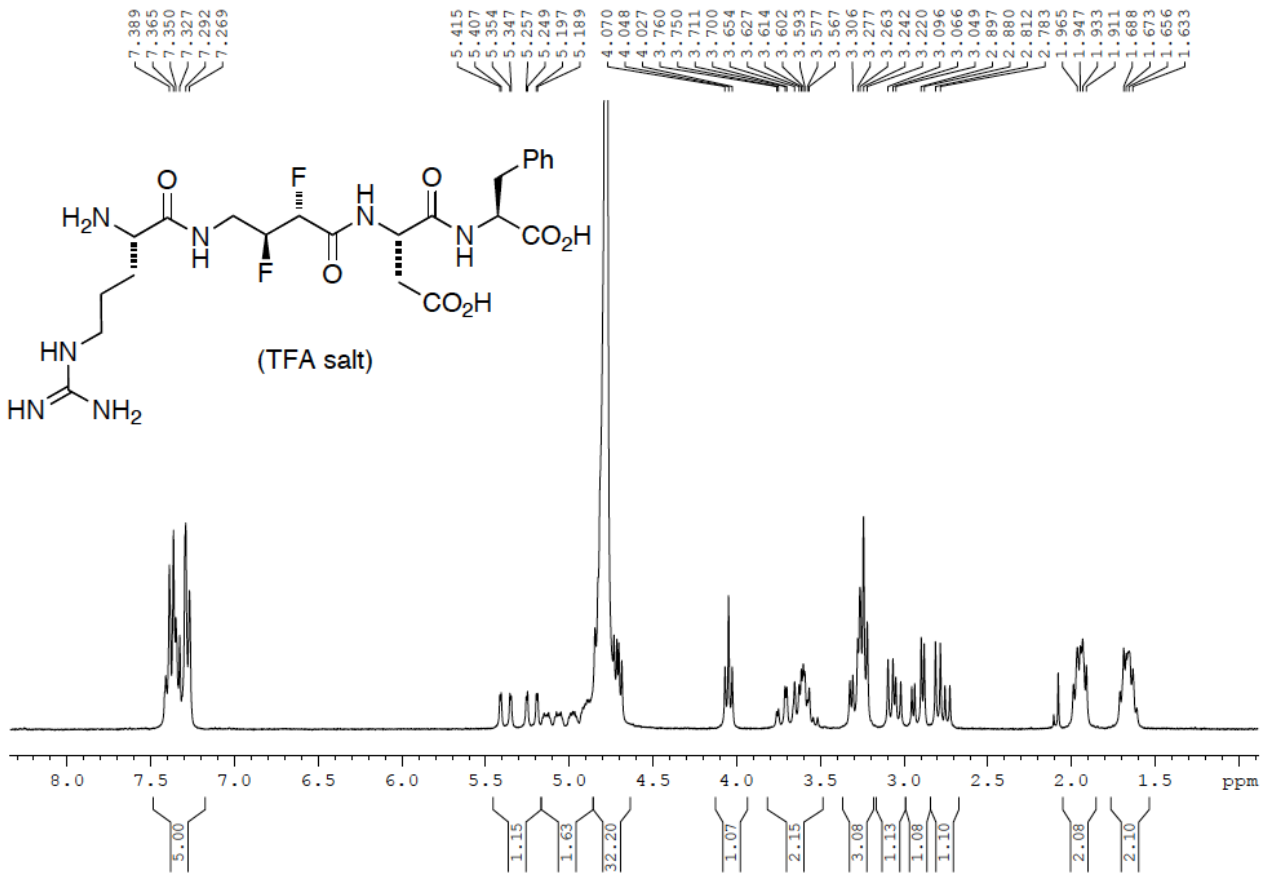


LC-MS (0→25% acetonitrile/water over 30 min) of **13**

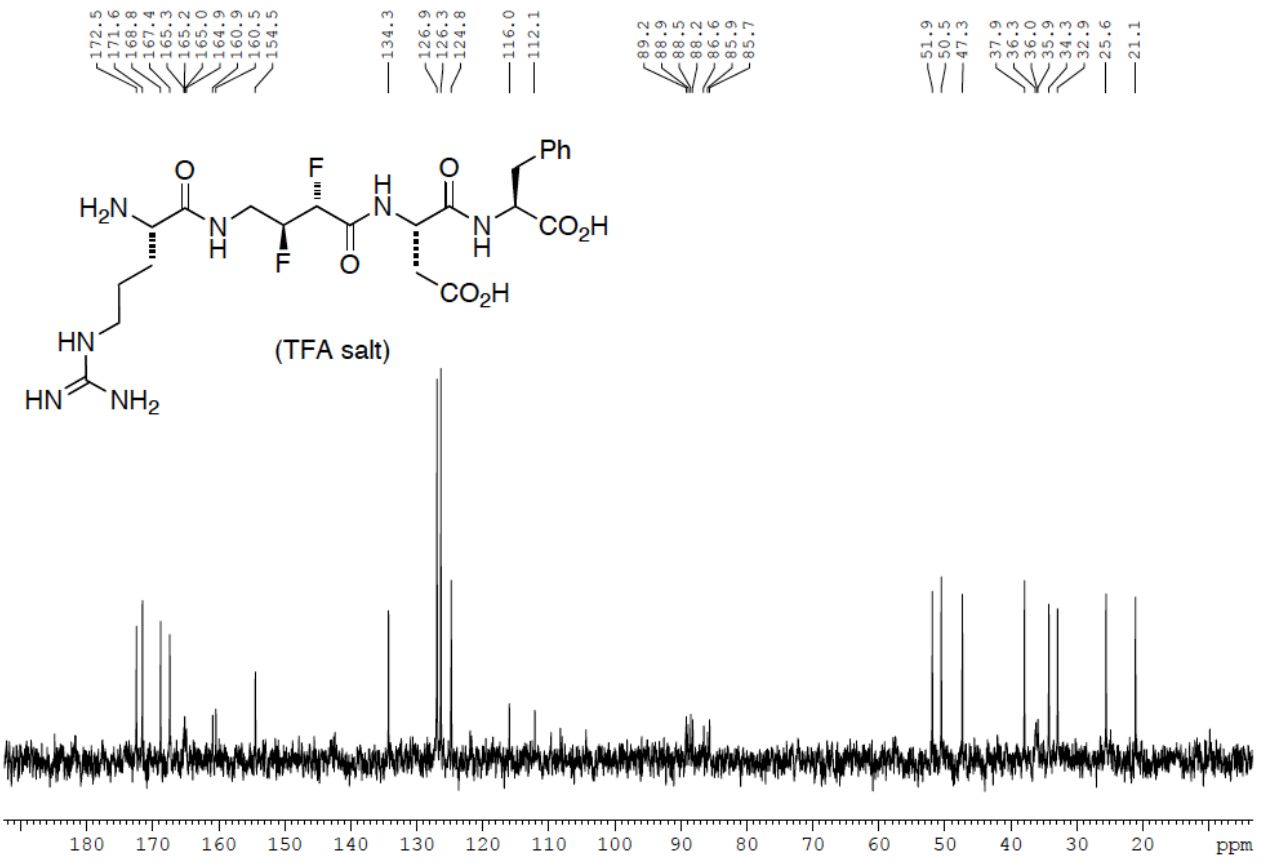




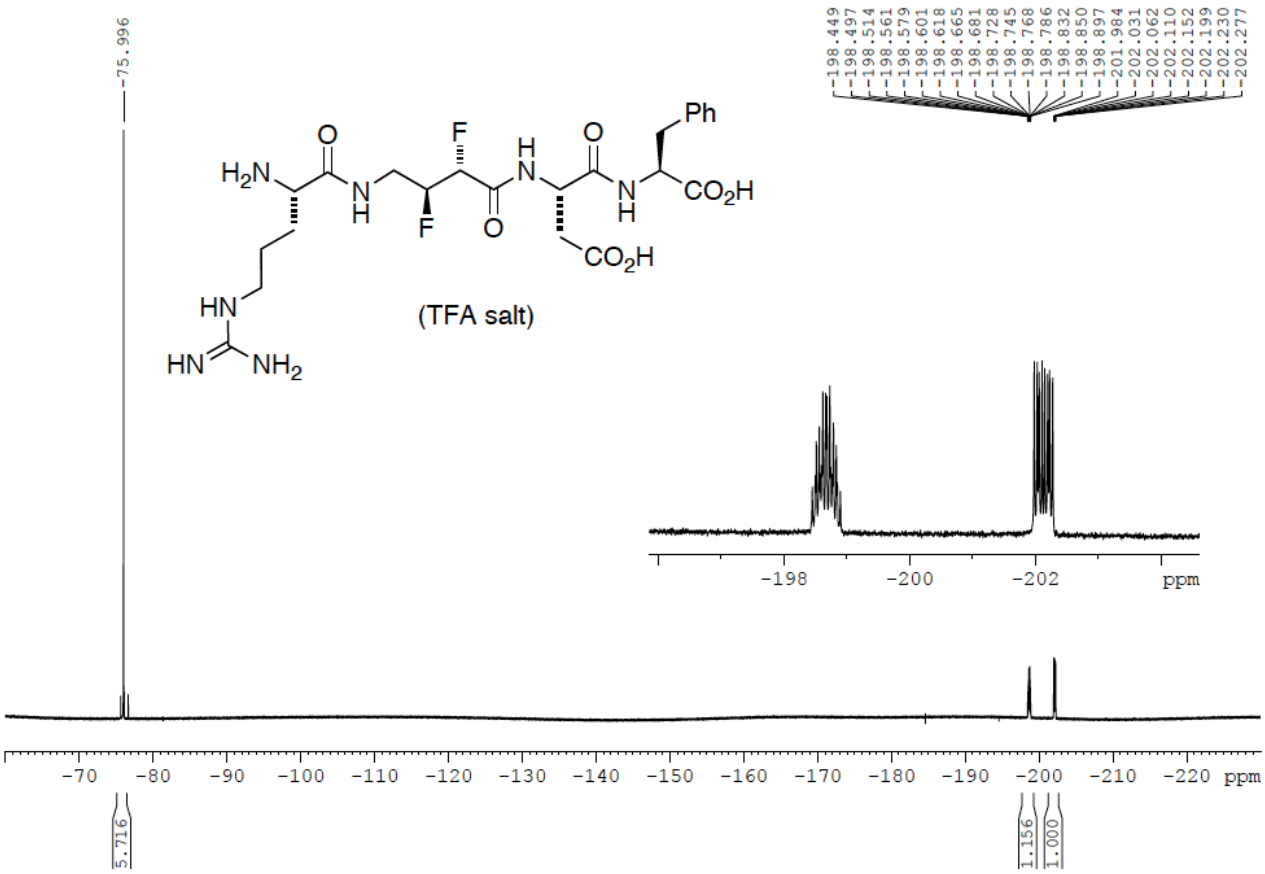
^{1}H NMR (200 MHz, D_2O) of **14**



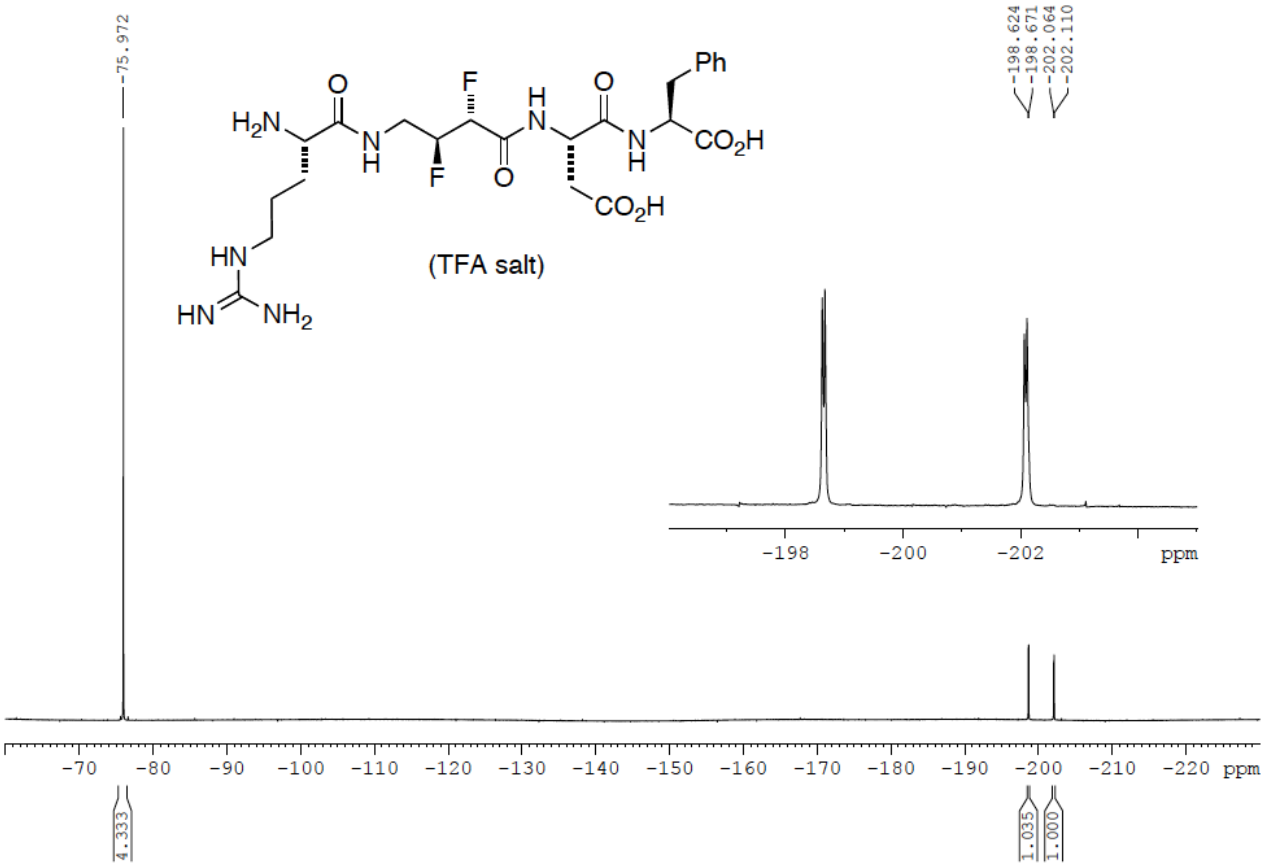
^{13}C {^{1}H} NMR (75 MHz, D_2O) of **14**



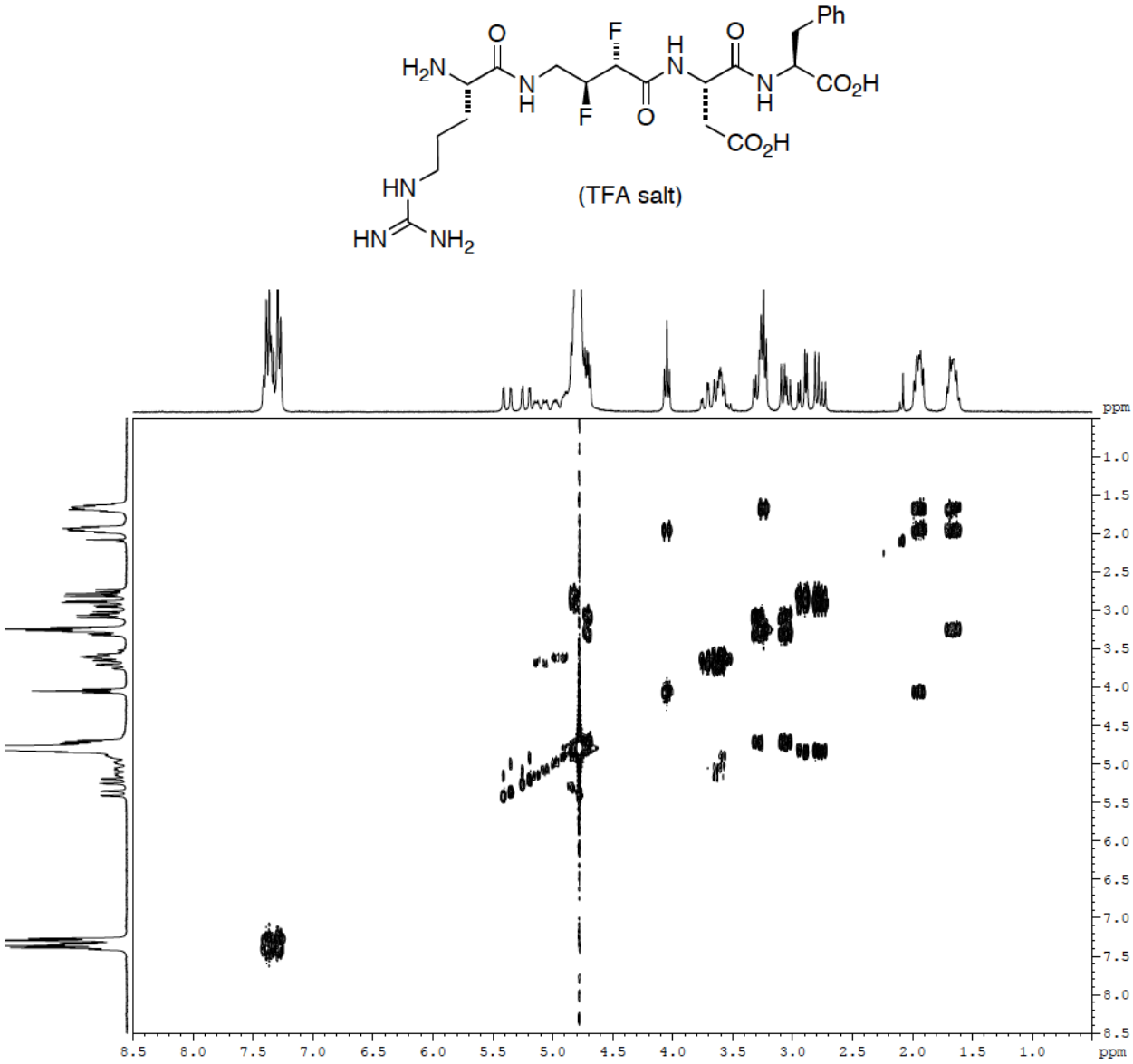
^{19}F NMR (282 MHz, D_2O) of **14**



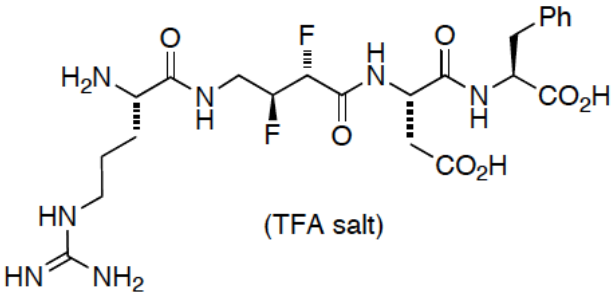
^{19}F {^{1}H} NMR (282 MHz, D_2O) of **14**

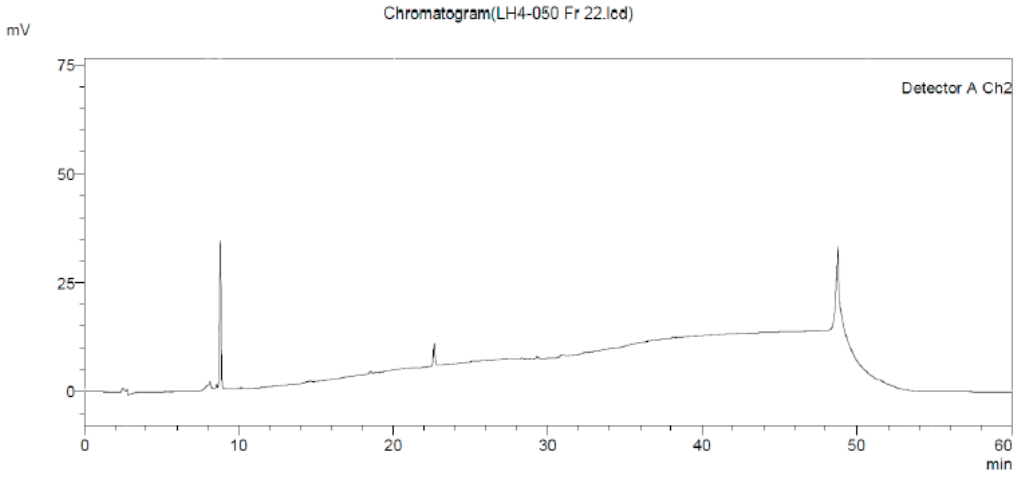


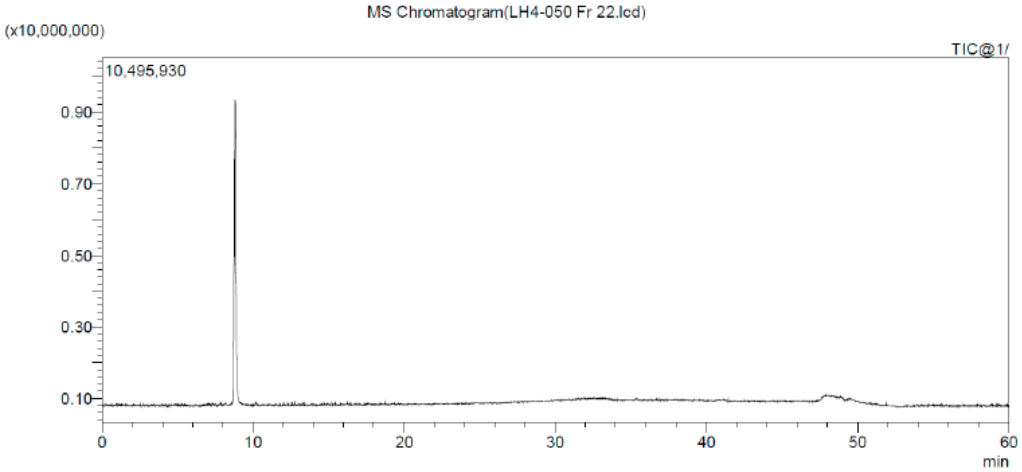
^{1}H-^{1}H COSY (300 MHz, D_2O) of **14**

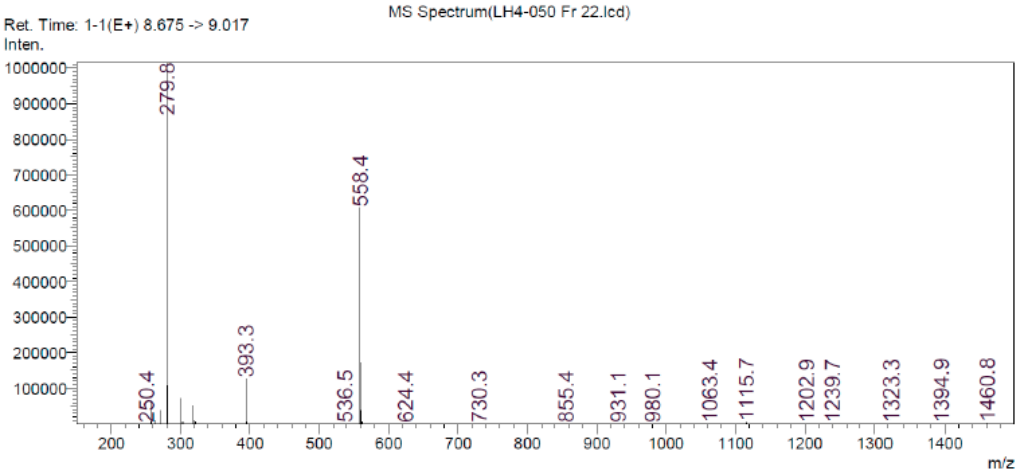


LC-MS (0→25% actetonitrile/water over 30 min) of **14**

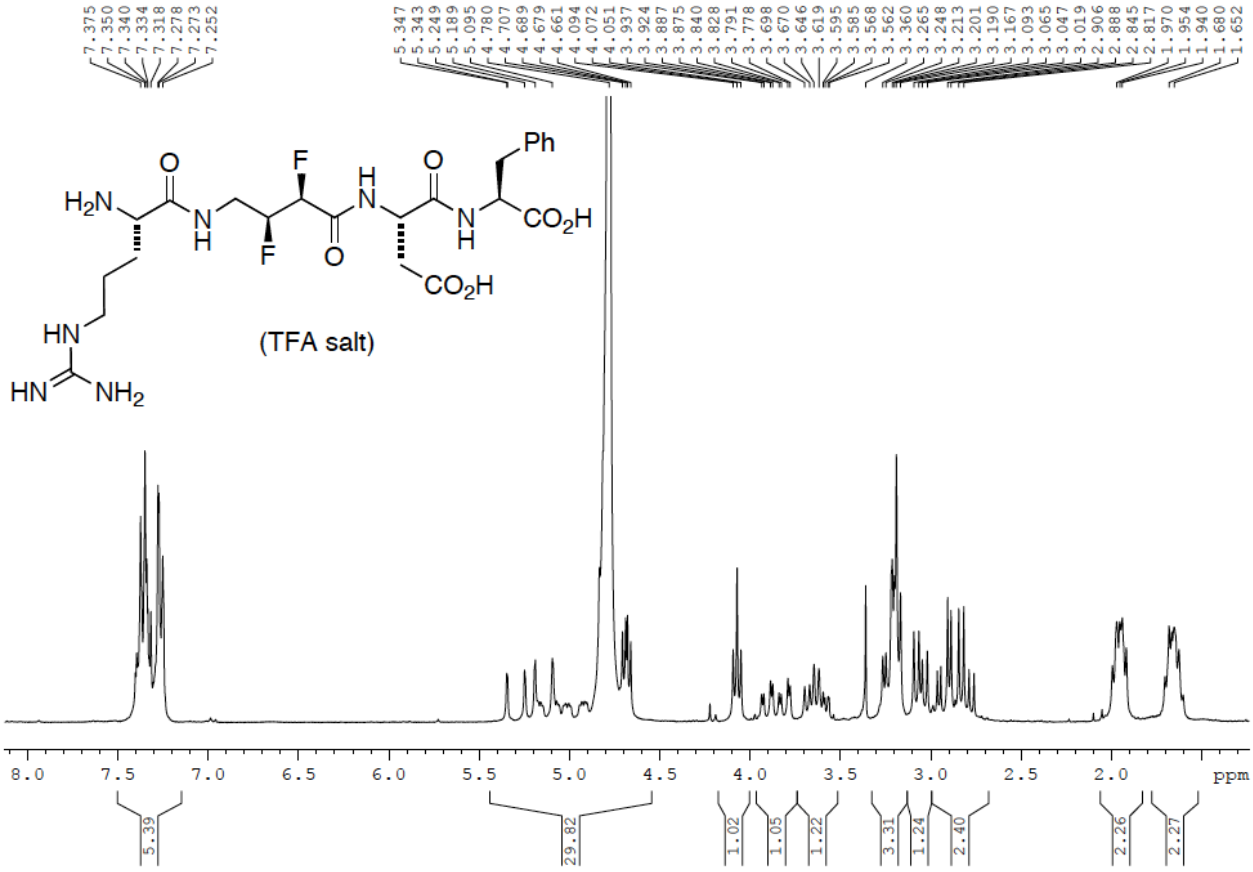




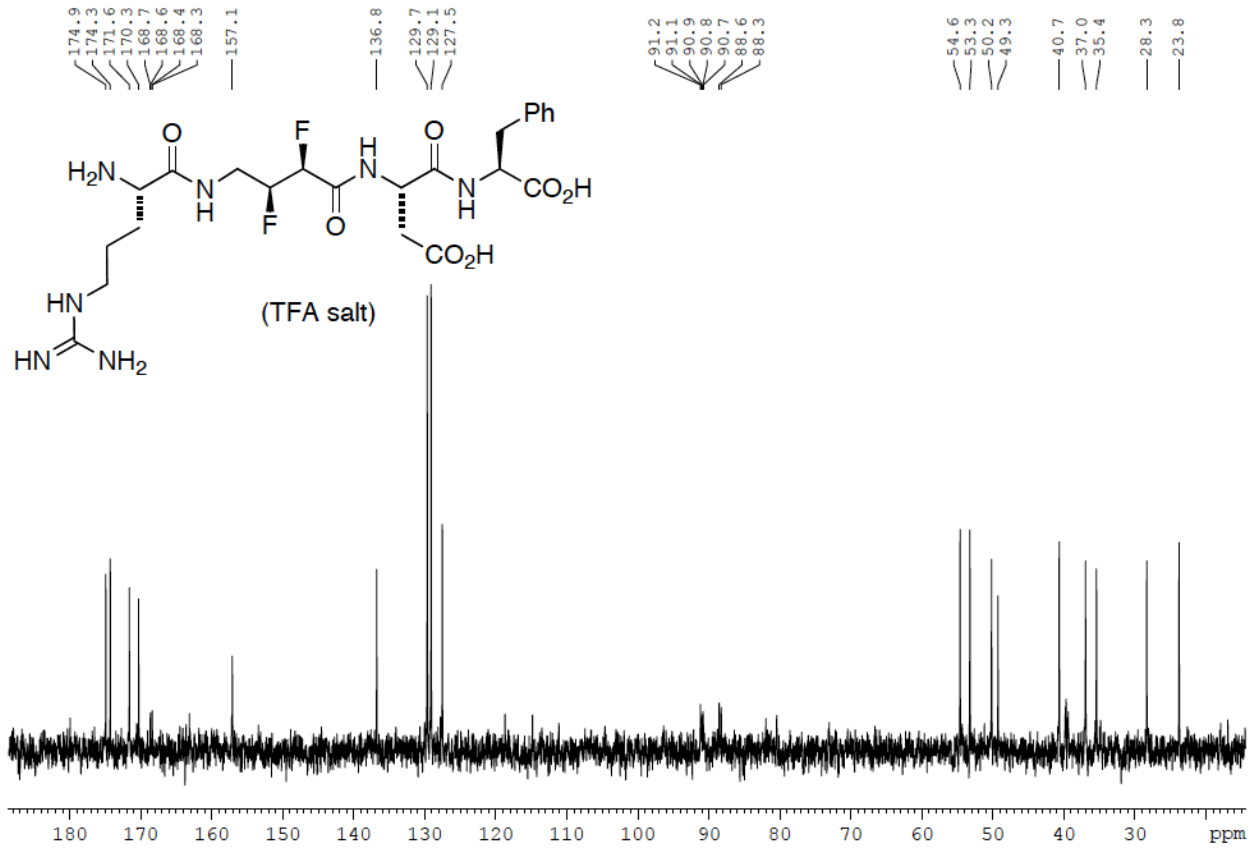




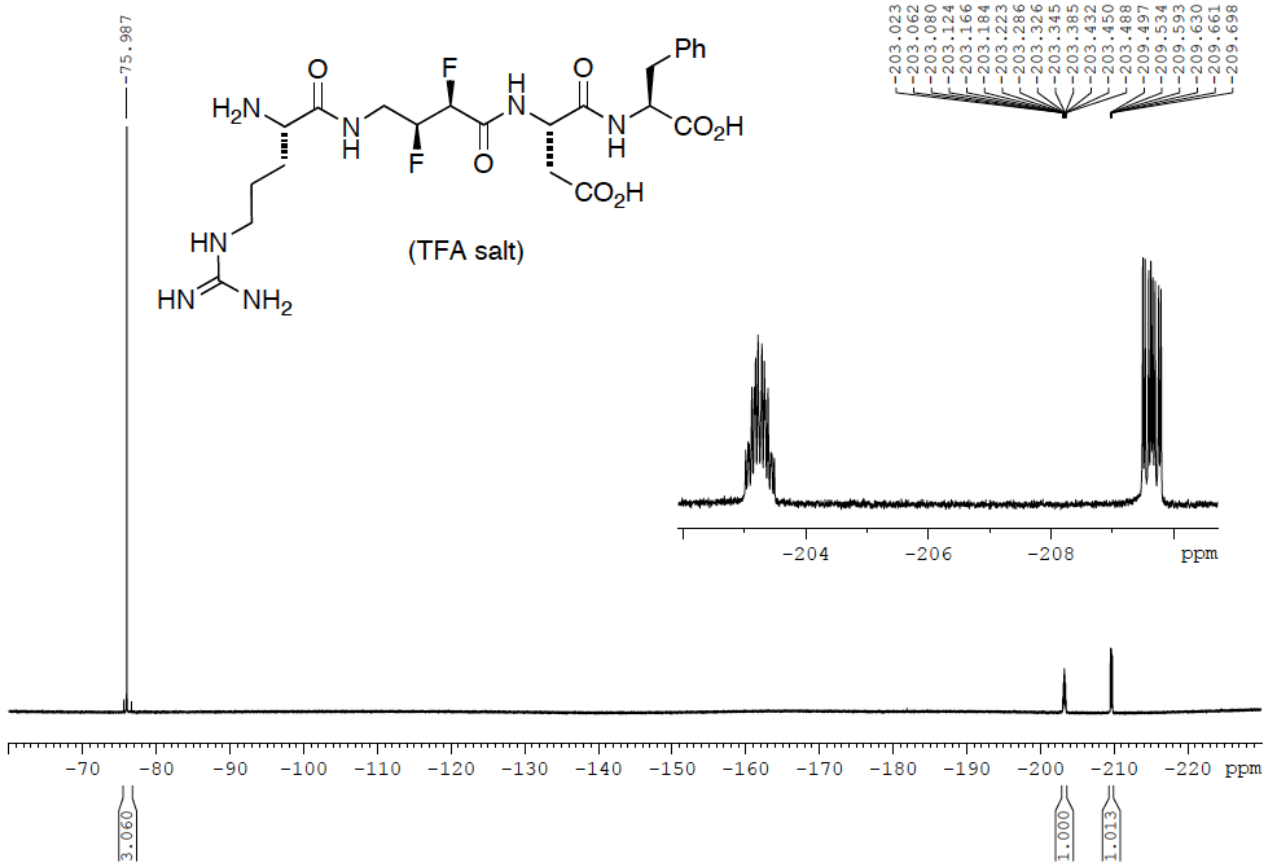
^{1}H NMR (300 MHz, D_2O) of **15**



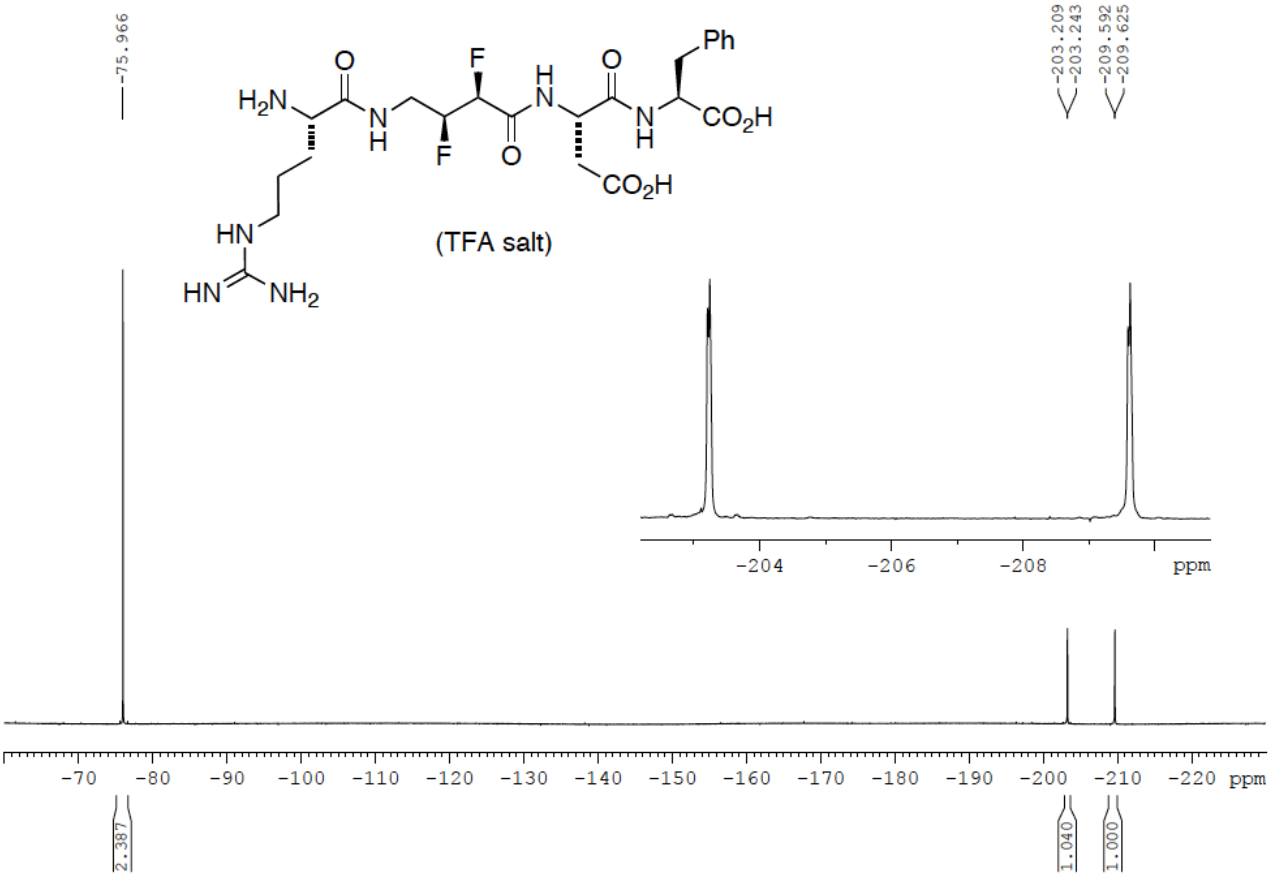
^{13}C {^{1}H} NMR (75 MHz, D_2O) of **15**



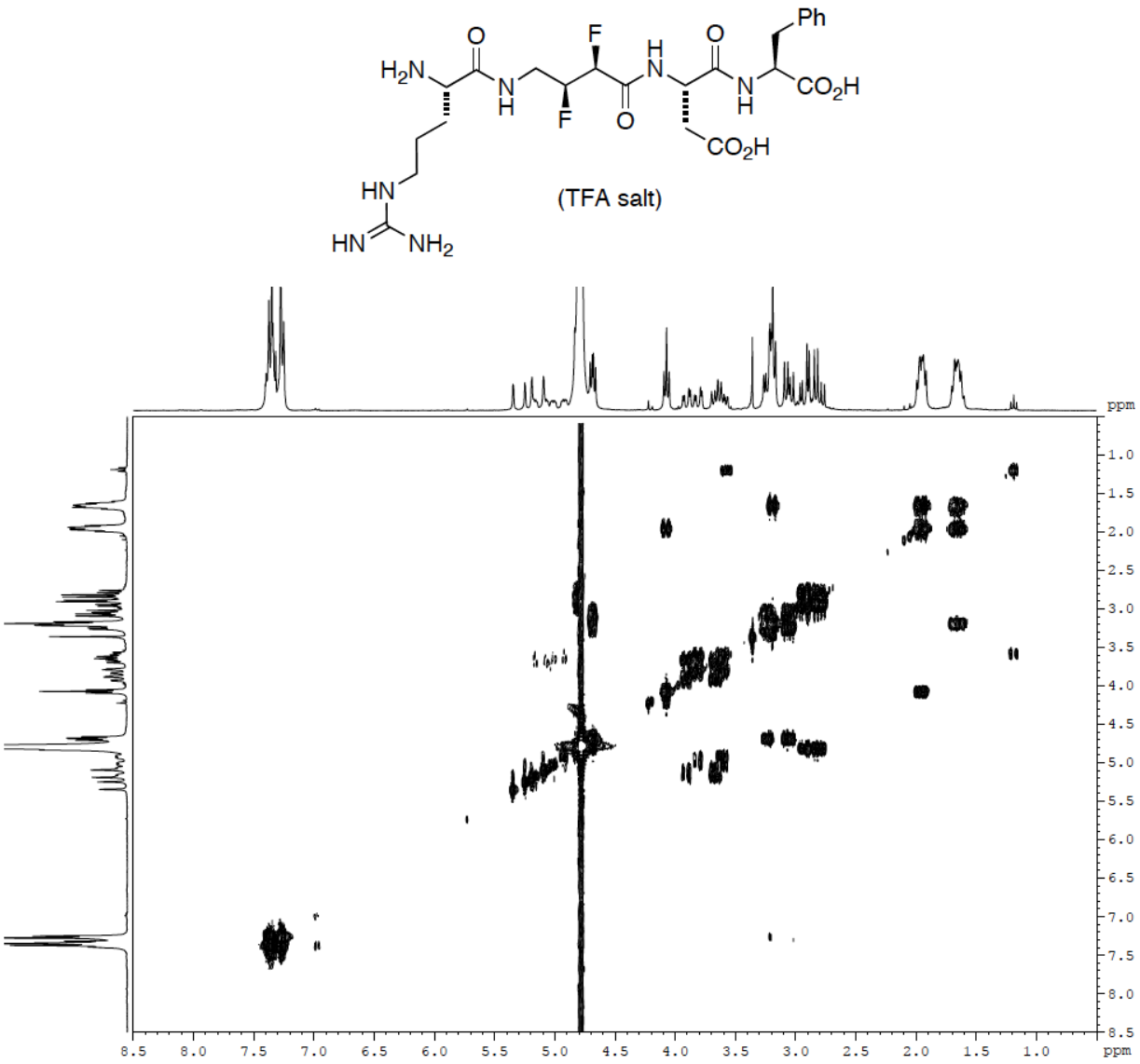
^{19}F NMR (282 MHz, D_2O) of **15**



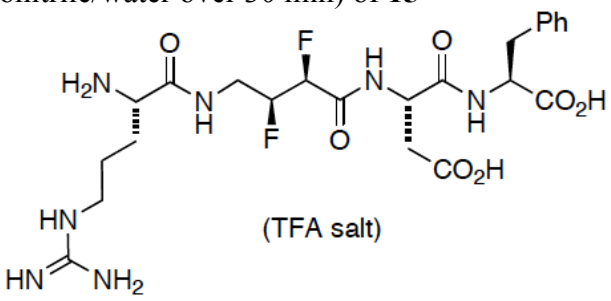
^{19}F {^{1}H} NMR (282 MHz, D_2O) of **15**

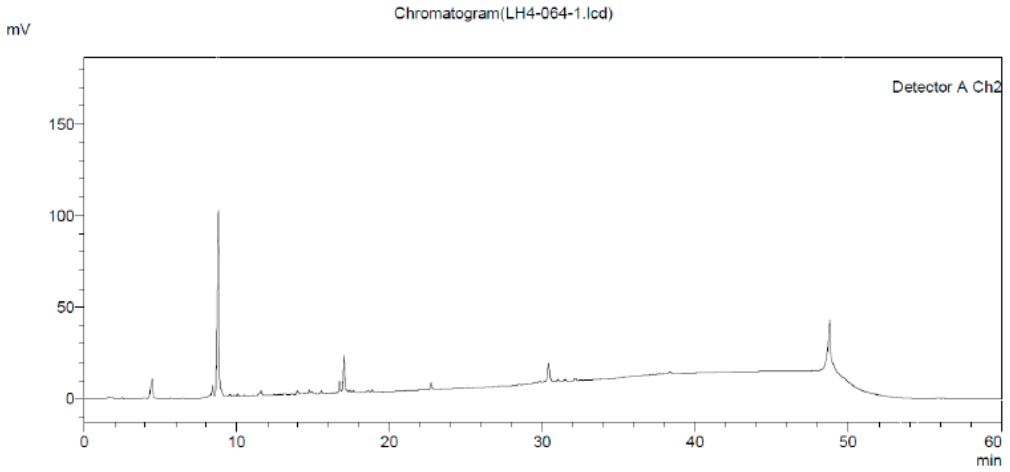


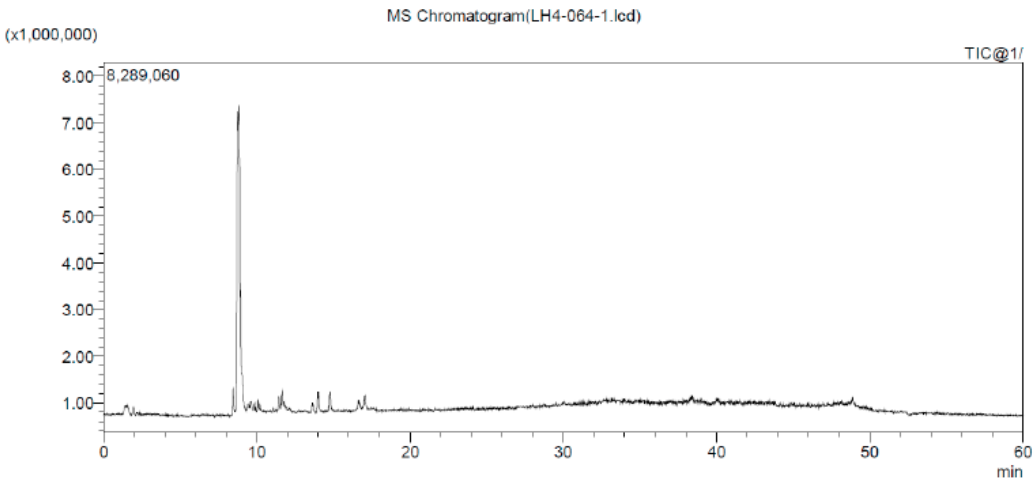
^{1}H-^{1}H COSY (300 MHz, D_2O) of **15**

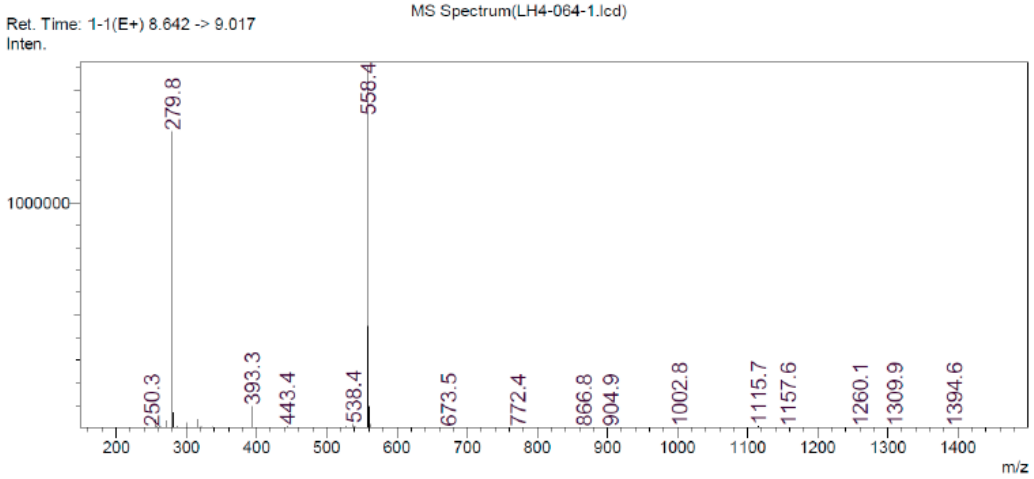


LC-MS (0→25% actetonitrile/water over 30 min) of **15**

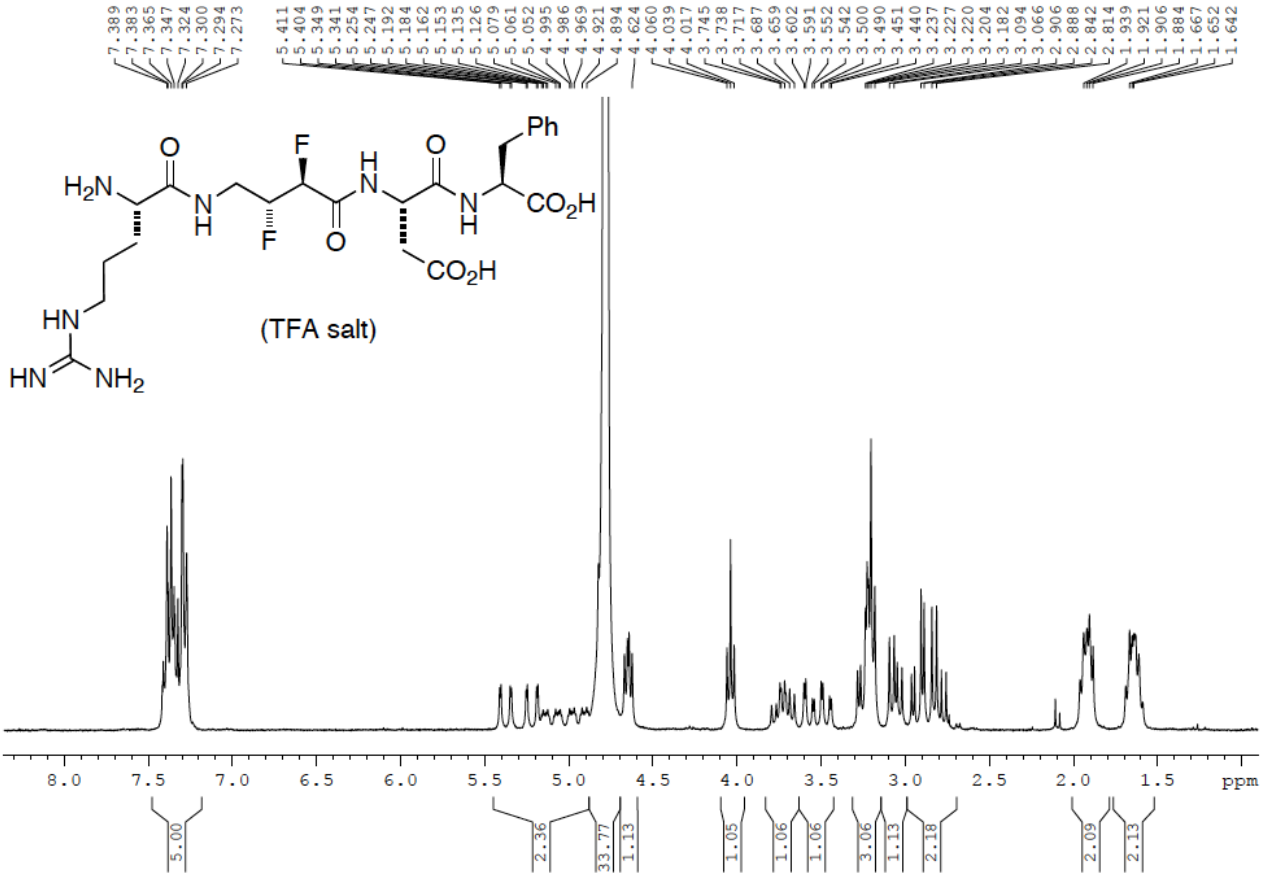




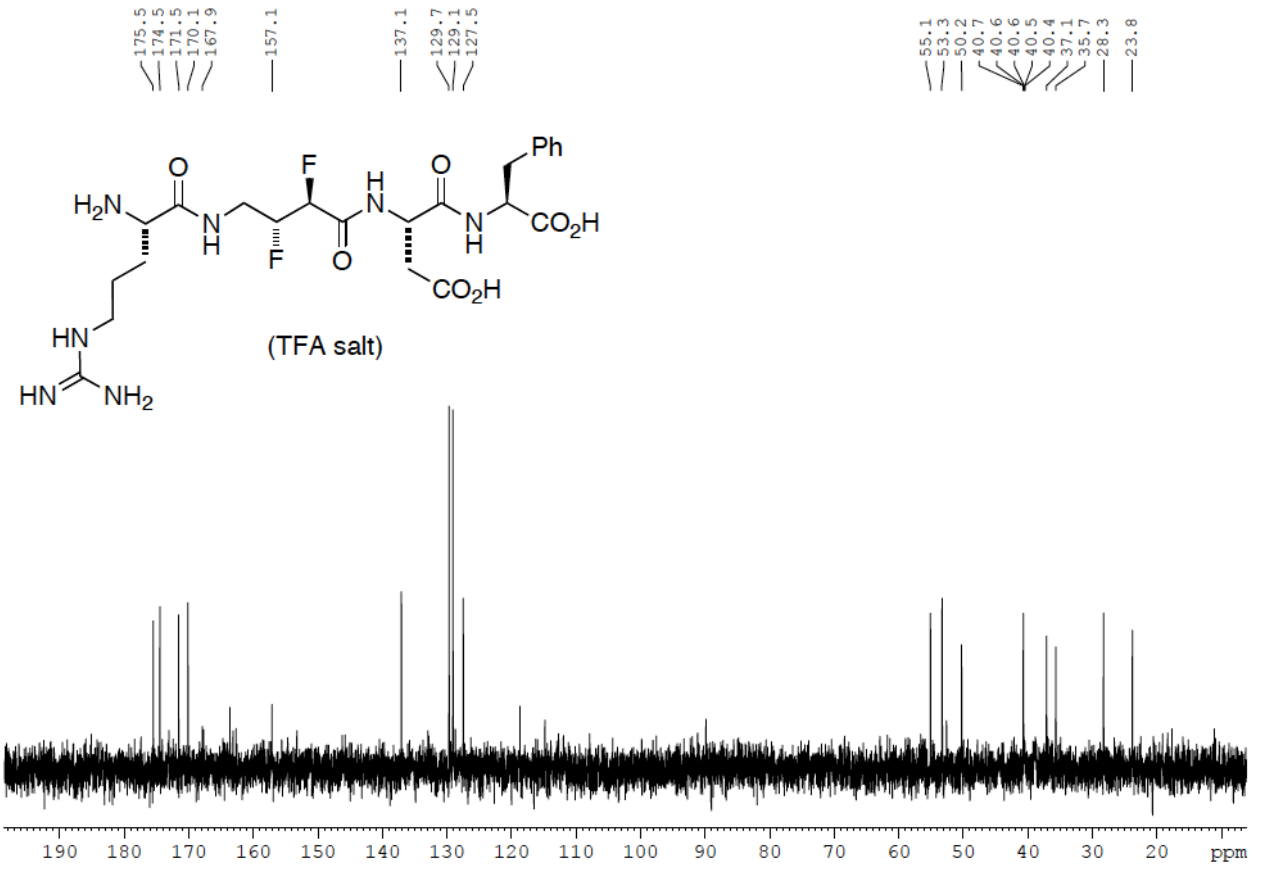




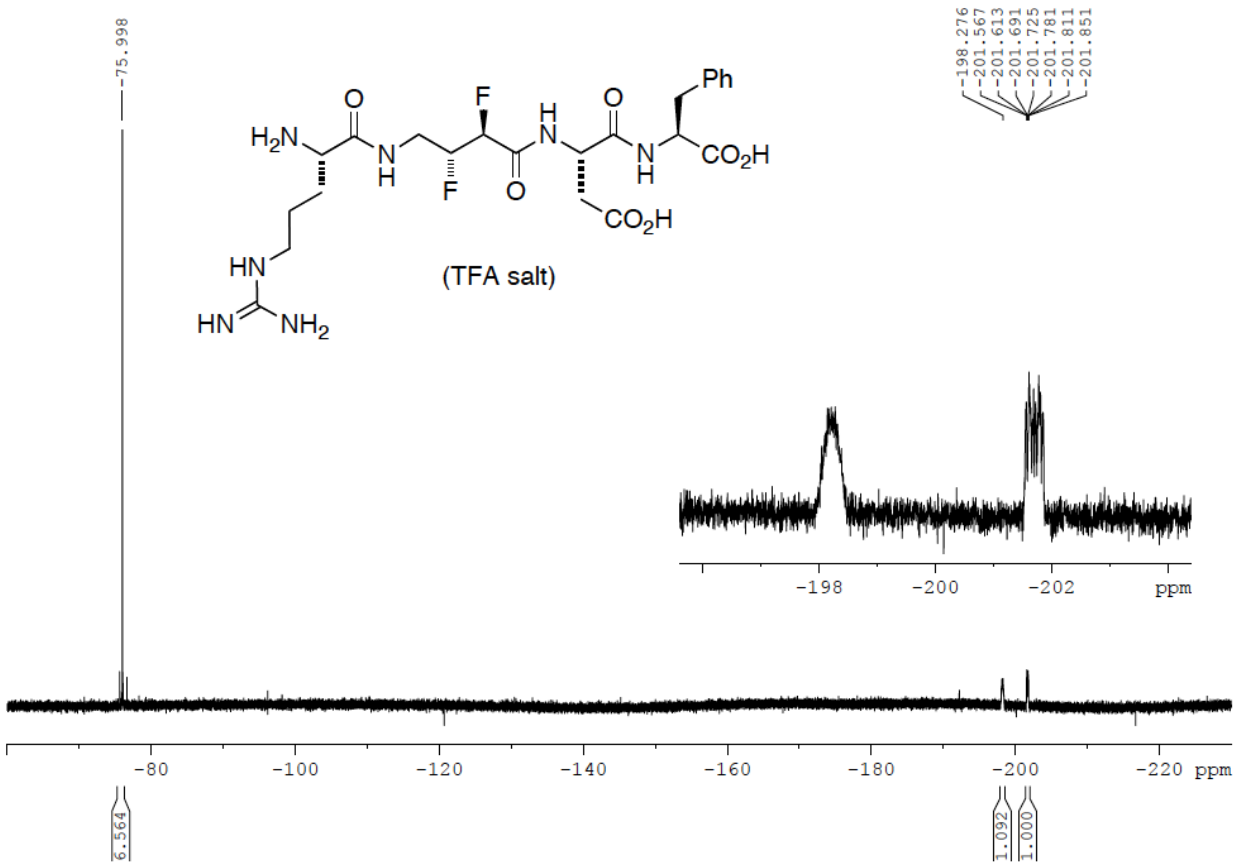
^{1}H NMR (200 MHz, D_2O) of **16**



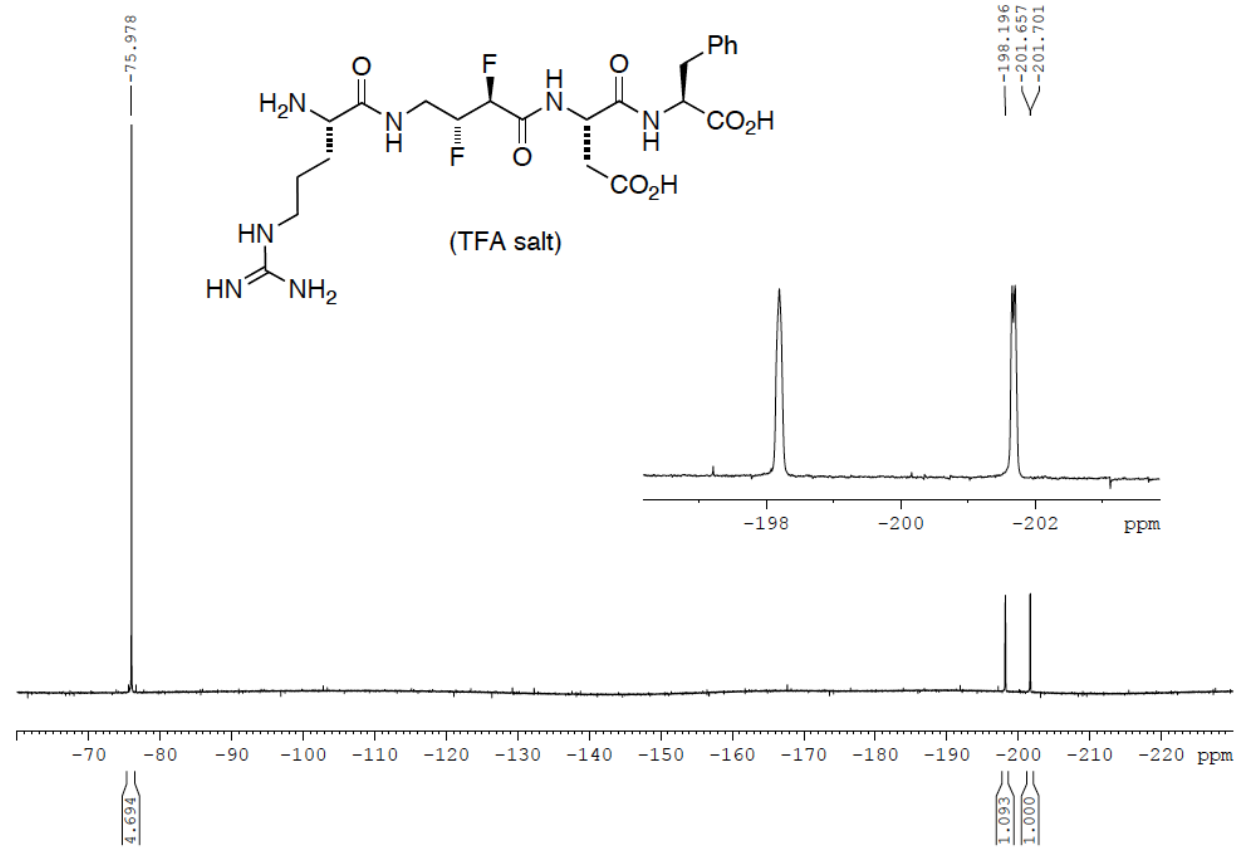
^{13}C {^{1}H} NMR (75 MHz, D_2O) of **16**



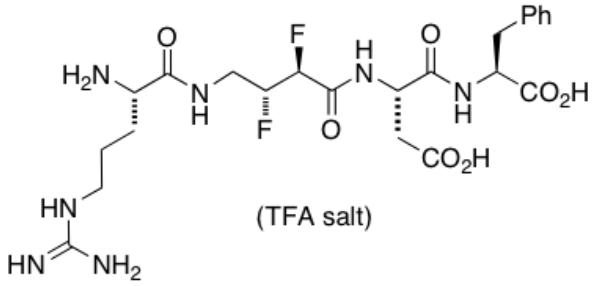
^{19}F NMR (282 MHz, D_2O) of **16**

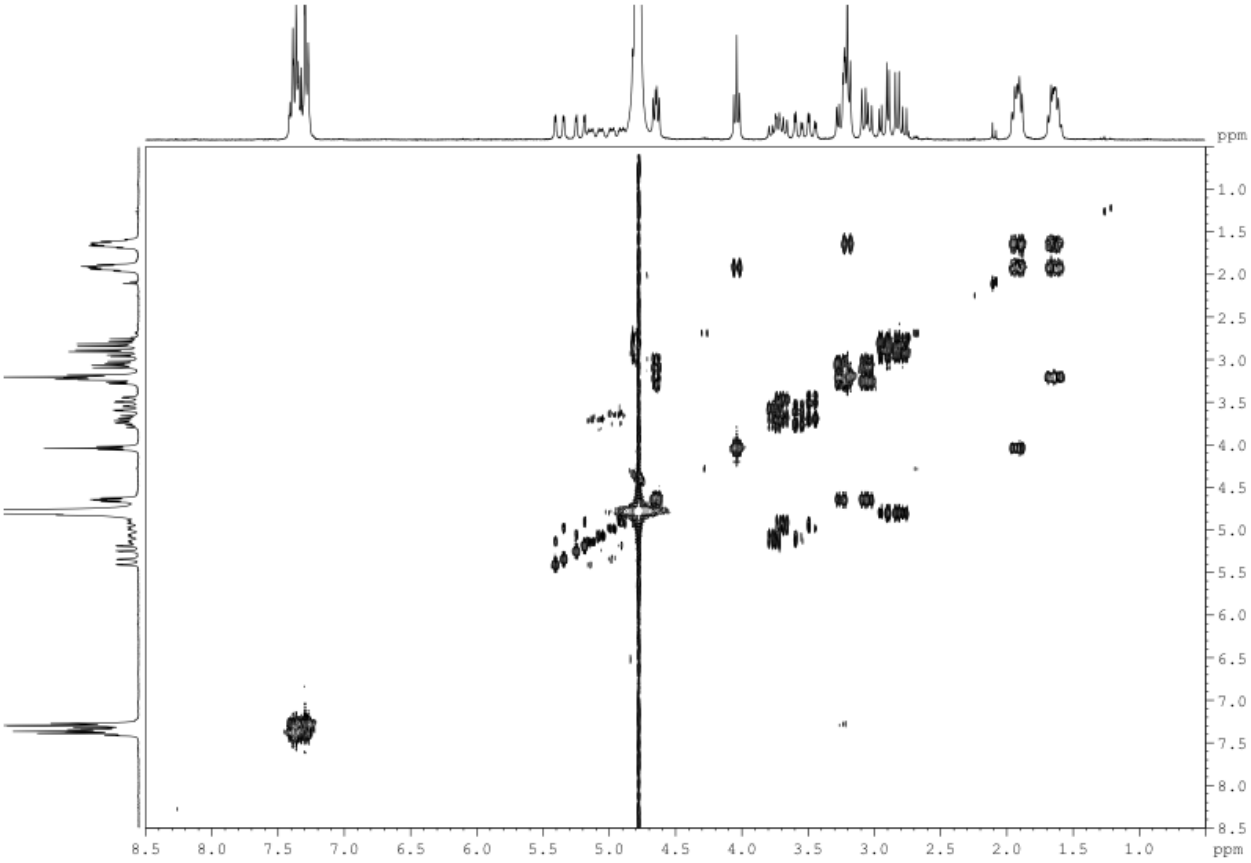


^{19}F {^{1}H} NMR (282 MHz, D_2O) of **16**

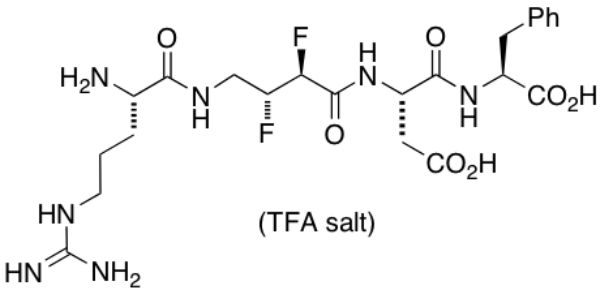


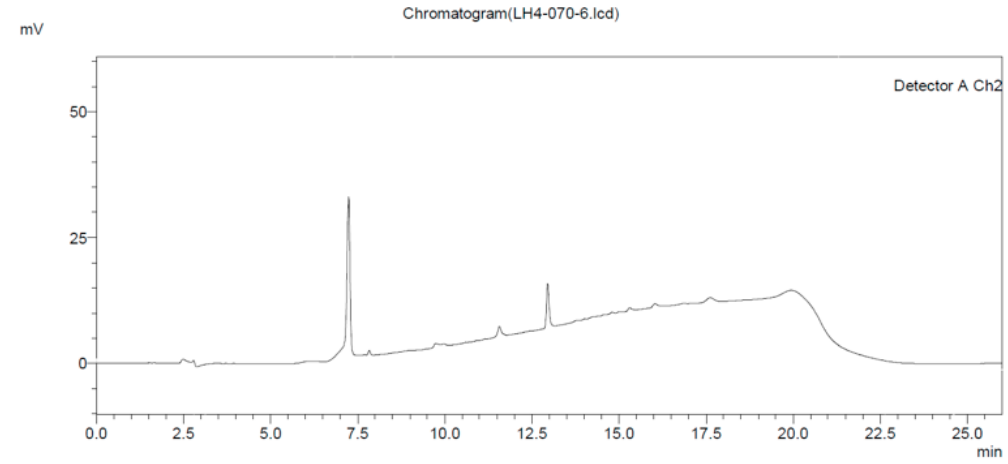
^{1}H-^{1}H COSY (300 MHz, D_2O) of **16**

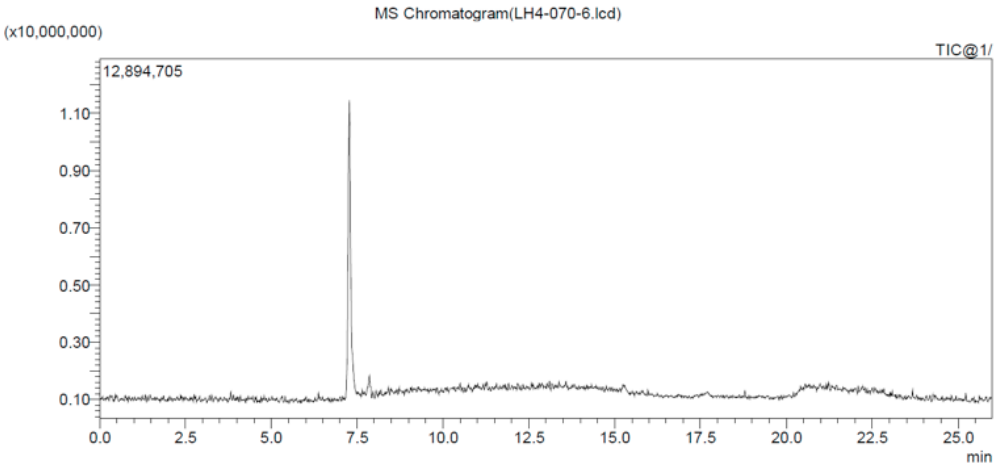


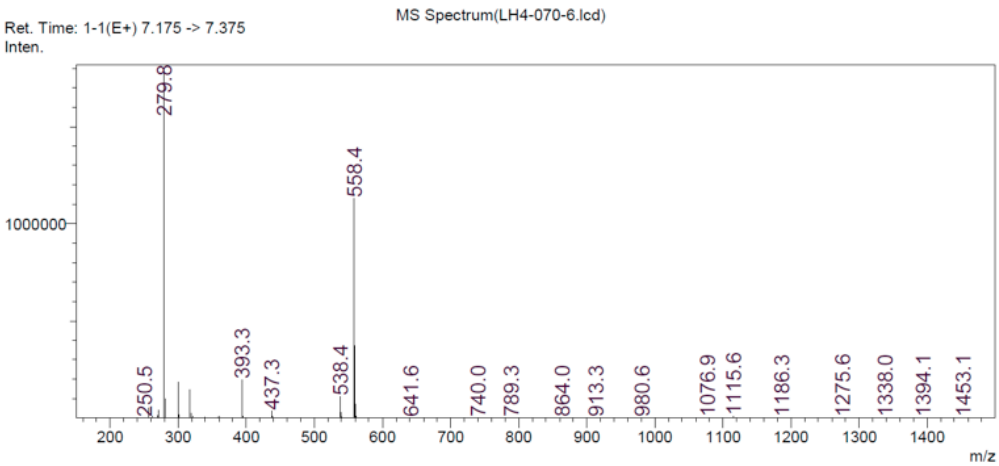


LC-MS (0 →100% acetonitrile/water over 10 min) of **16**

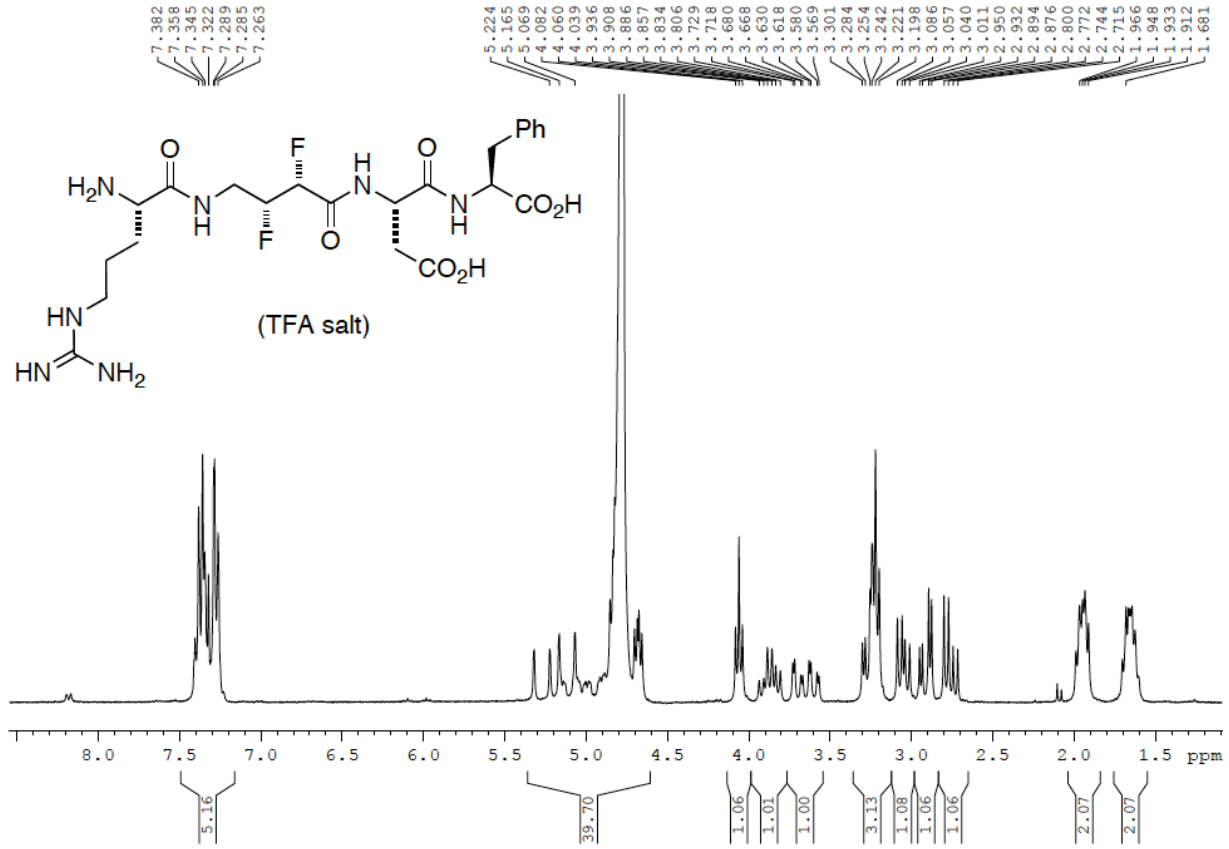




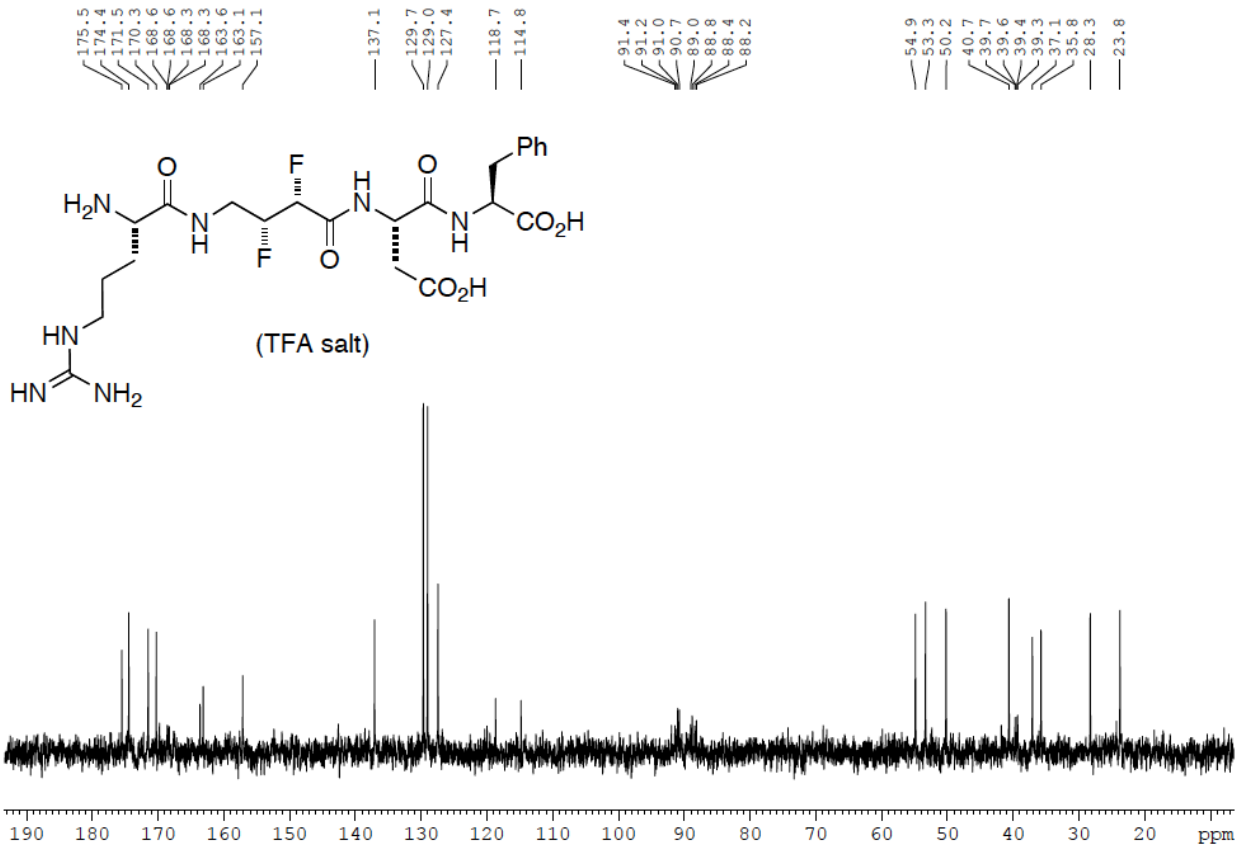




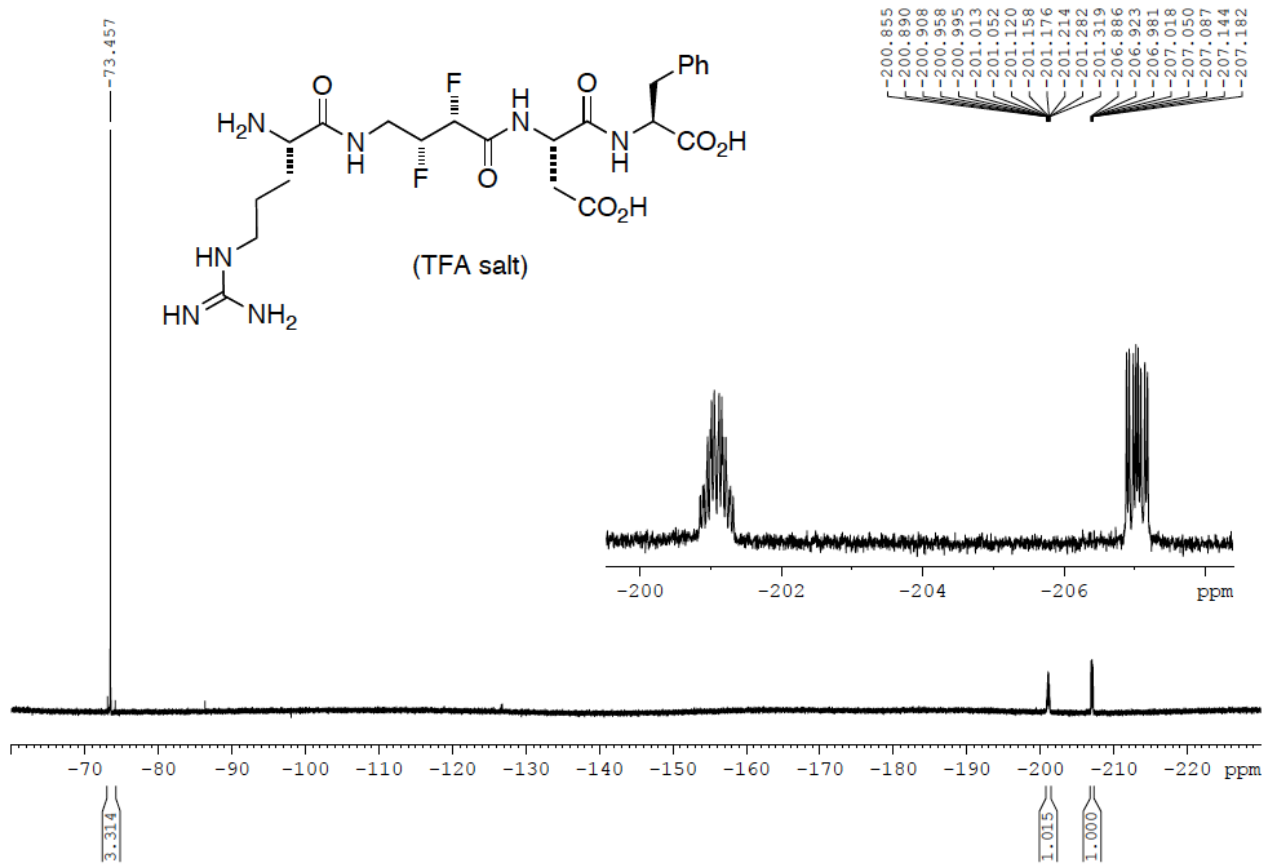
^{1}H NMR (300 MHz, D_2O) of **17**



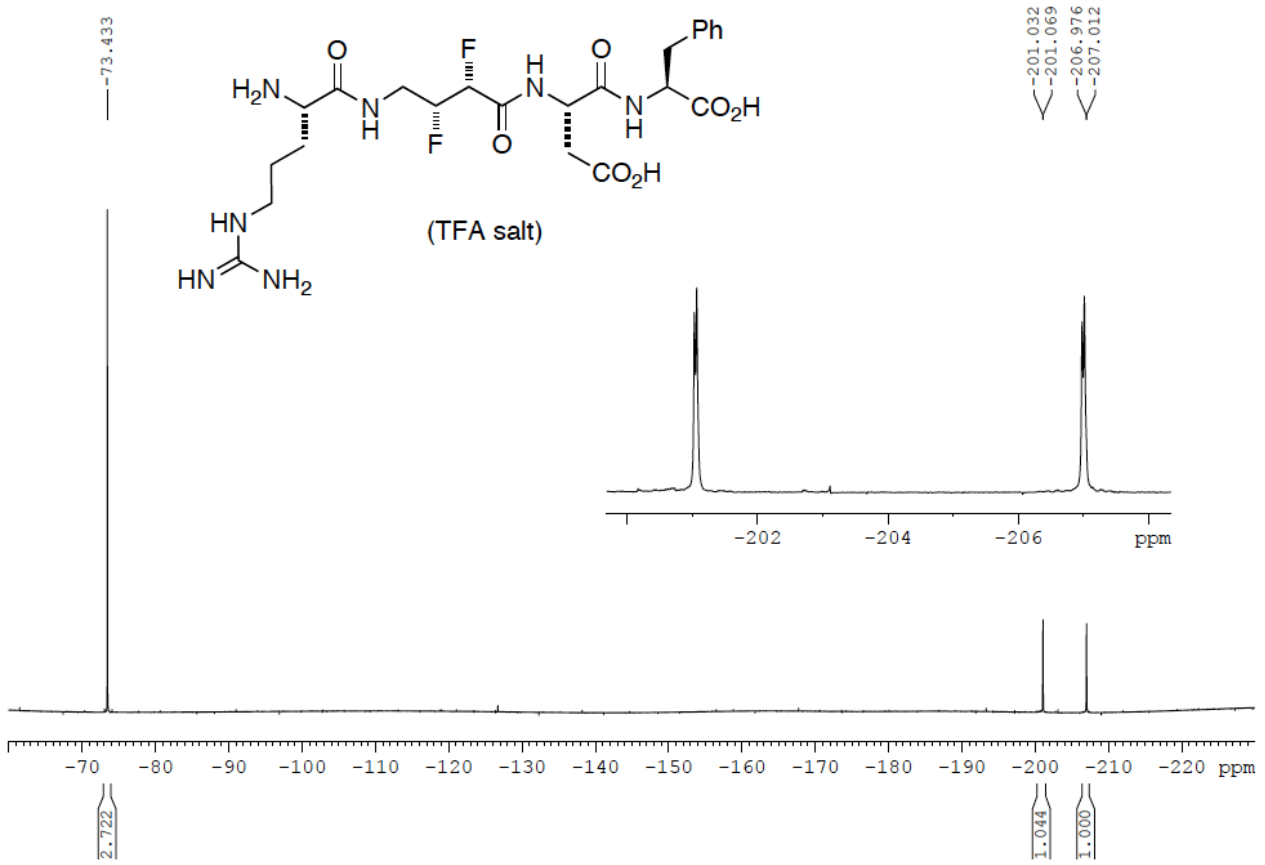
^{13}C {^{1}H} NMR (75 MHz, D_2O) of **17**



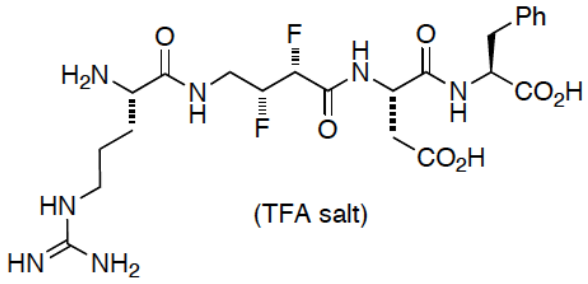
^{19}F NMR (282 MHz, D_2O) of **17**

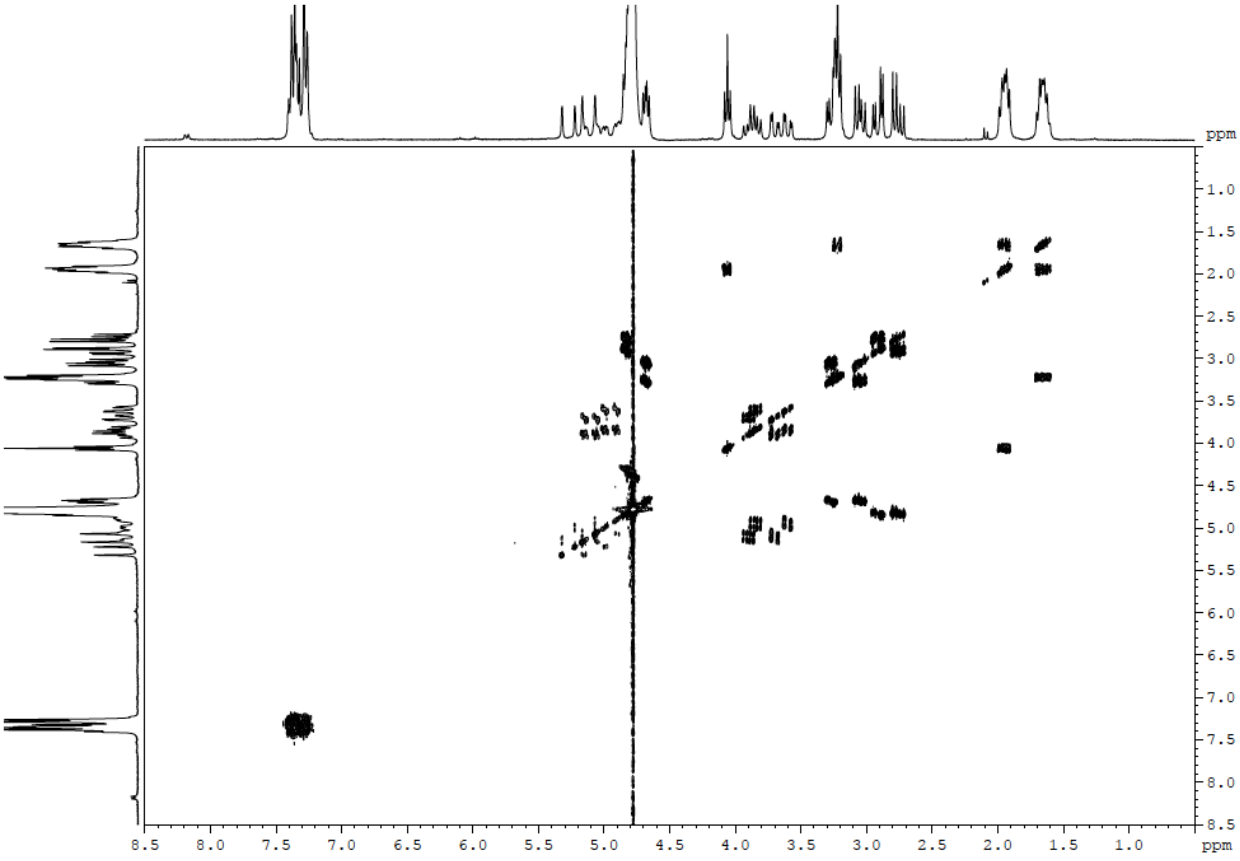


^{19}F {^{1}H} NMR (282 MHz, D_2O) of **17**

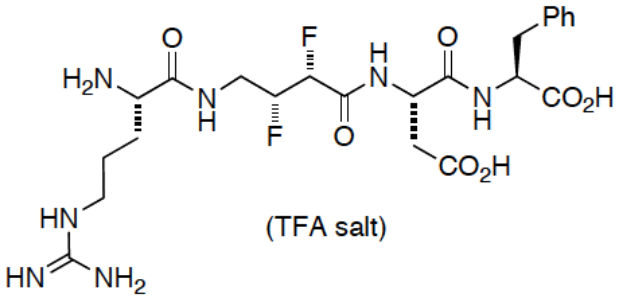


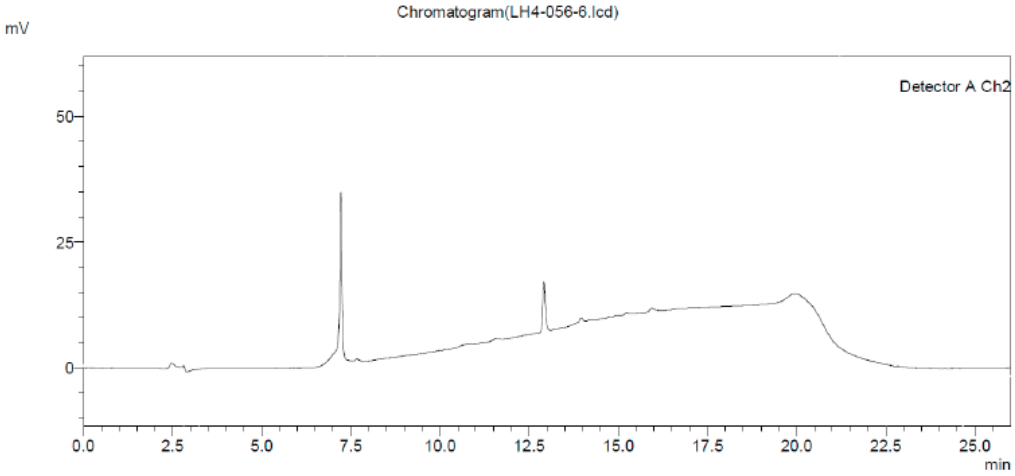
^{1}H-^{1}H COSY (300 MHz, D_2O) of **17**

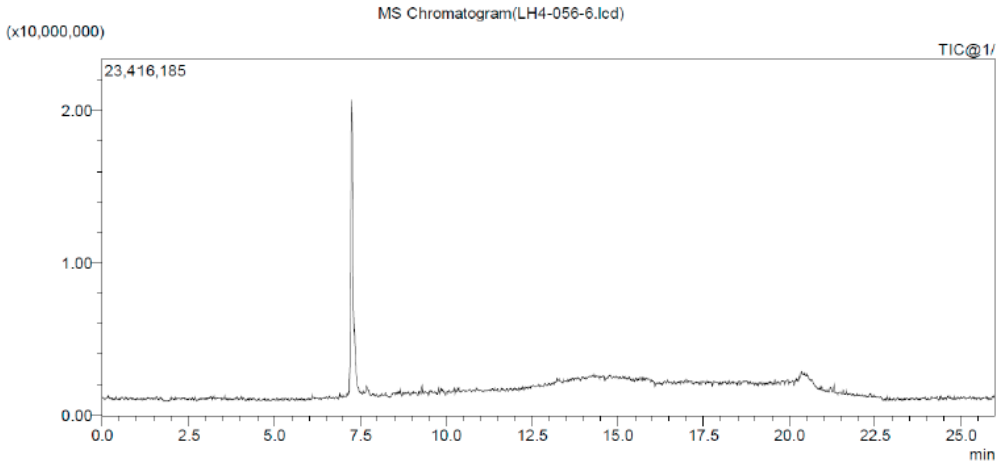


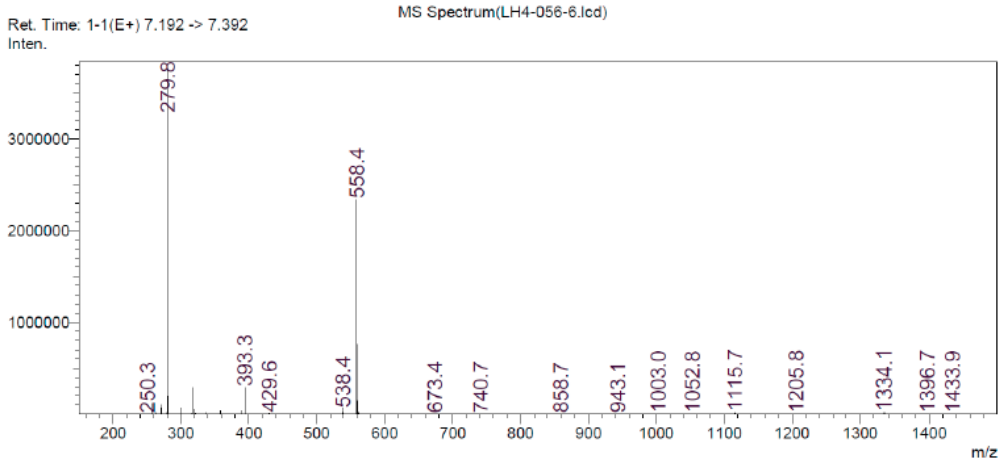


LC-MS (0→25% acetonitrile/water over 30 min) of **17**

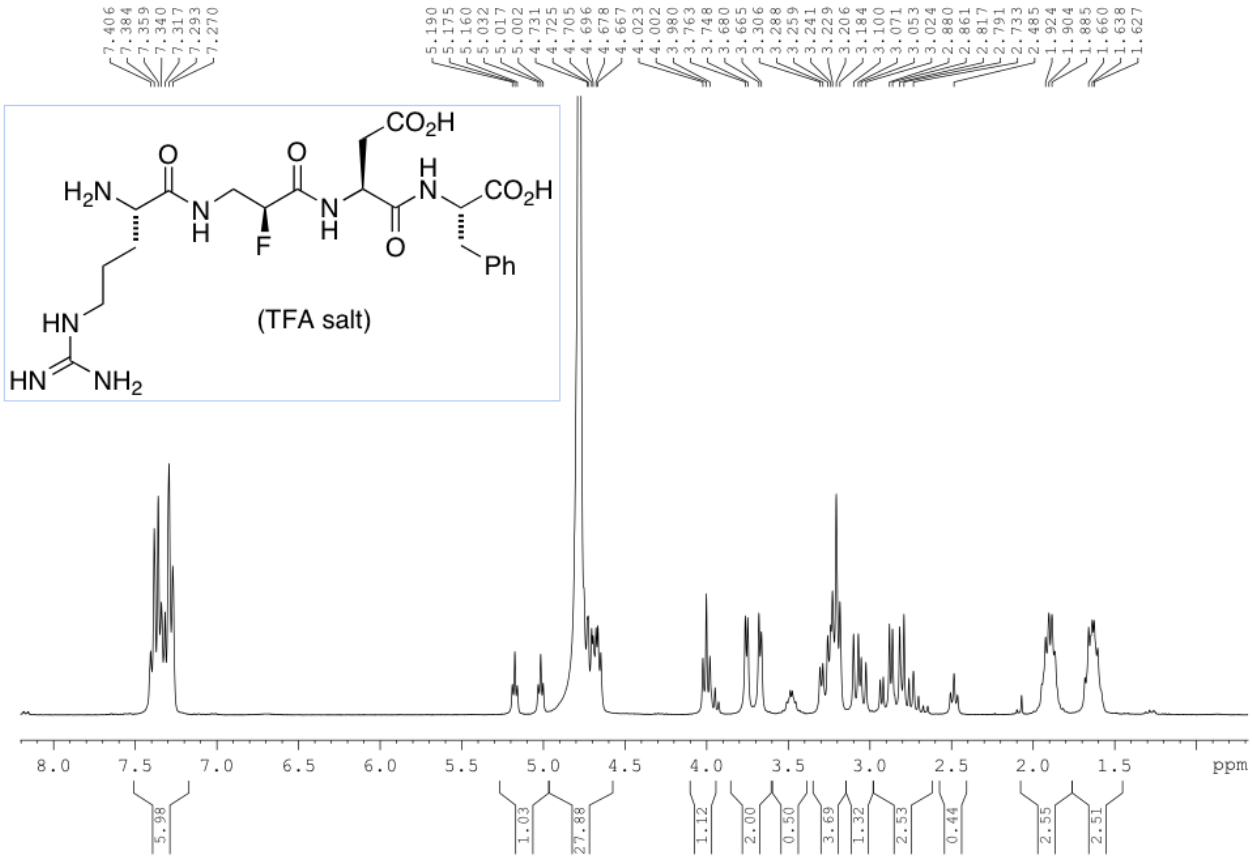




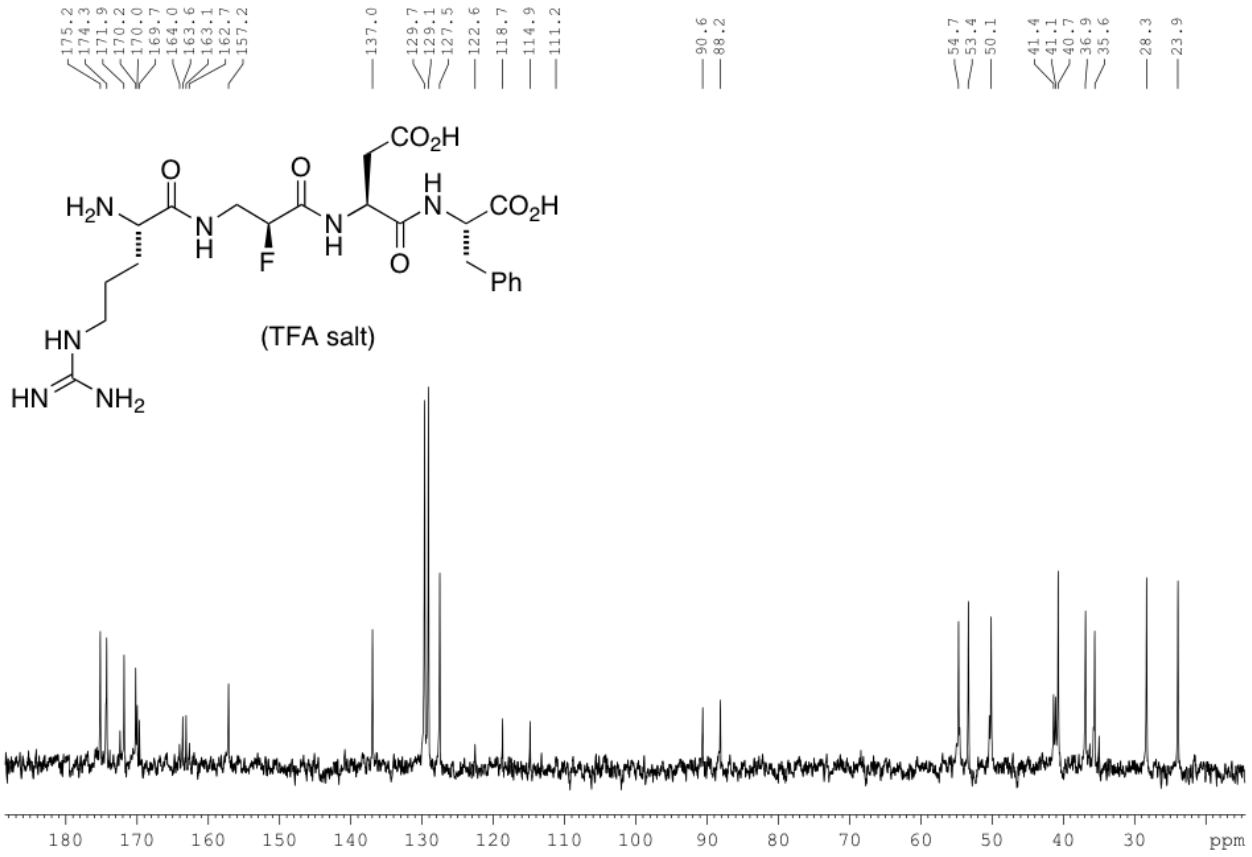




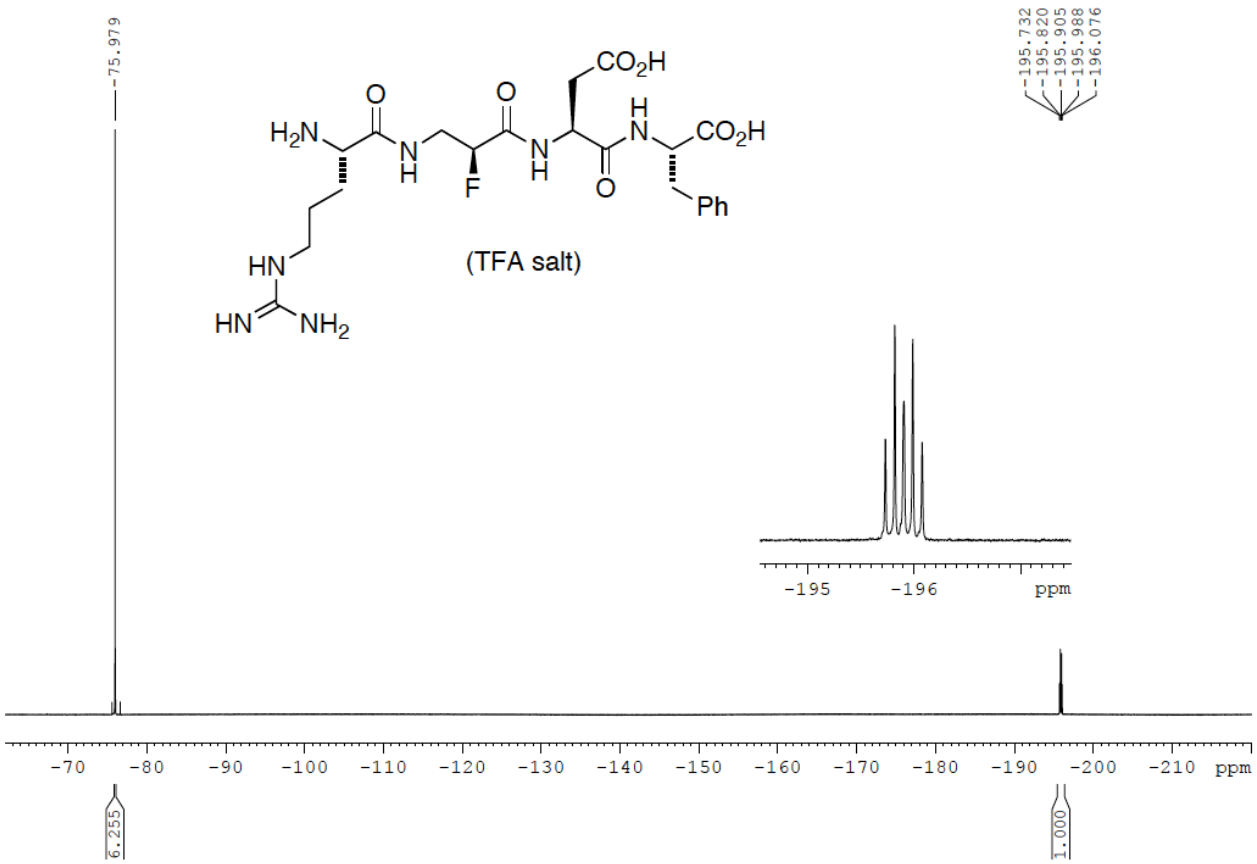
^{1}H NMR (200 MHz, D_2O) of **18**



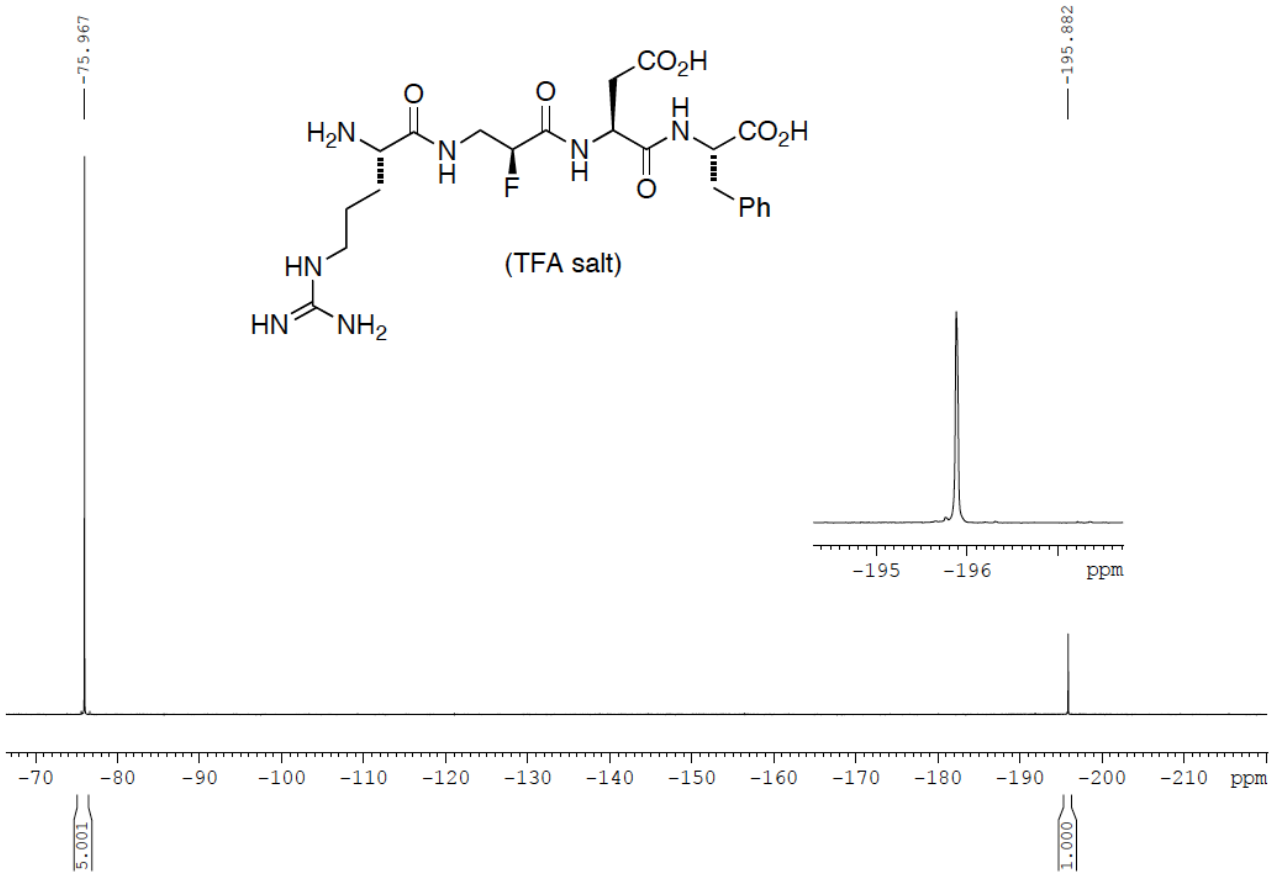
^{13}C {^{1}H} NMR (75 MHz, D_2O) of **18**



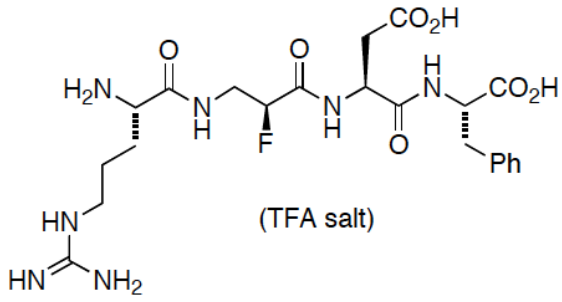
^{19}F NMR (282 MHz, D_2O) of **18**

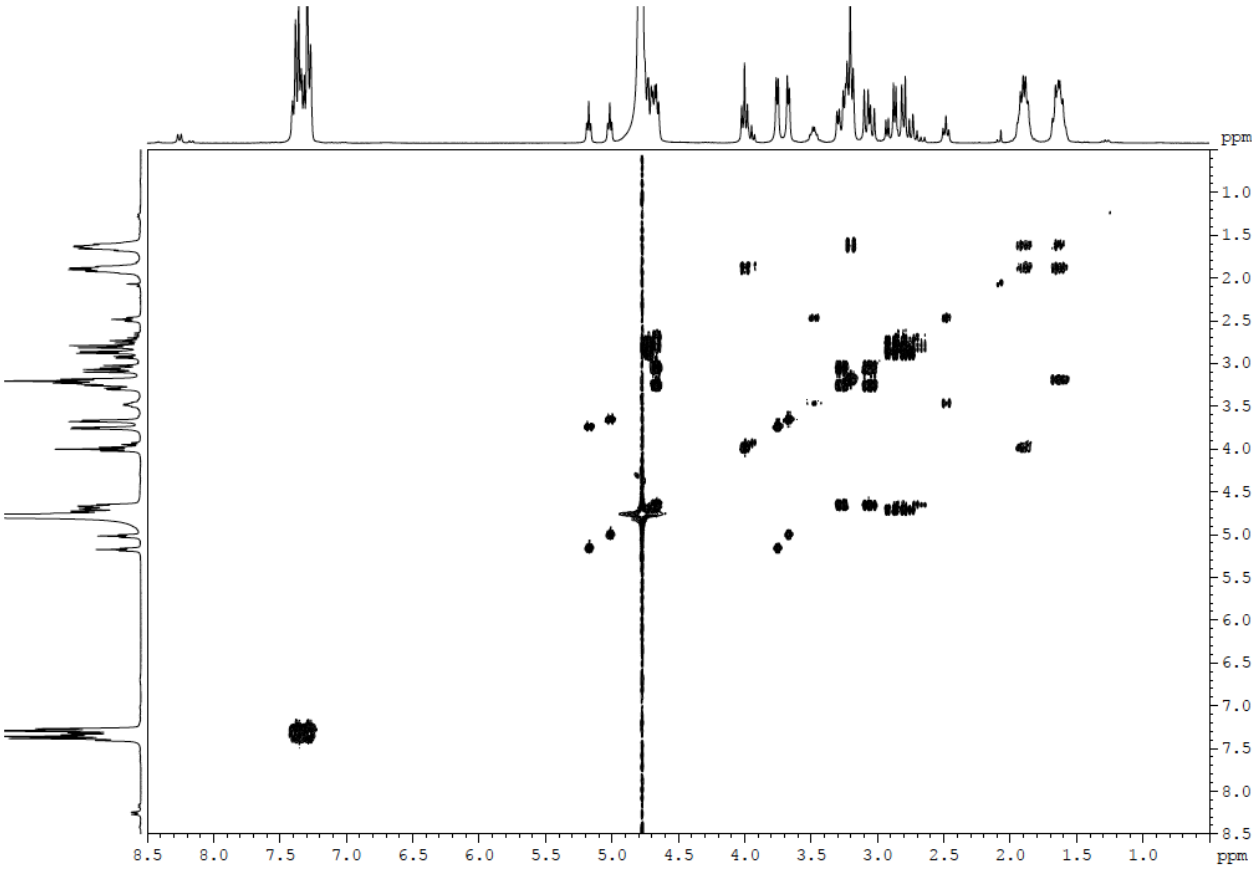


^{19}F {^{1}H} NMR (282 MHz, D_2O) of **18**

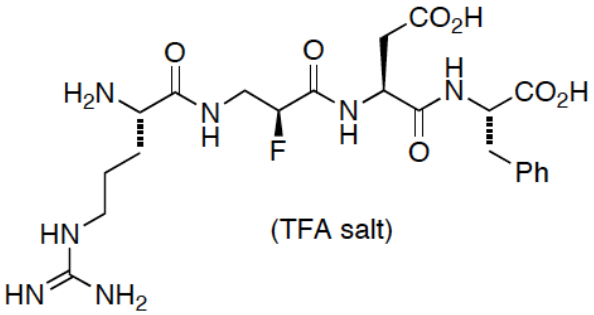


^{1}H-^{1}H COSY (300 MHz, D_2O) of **18**

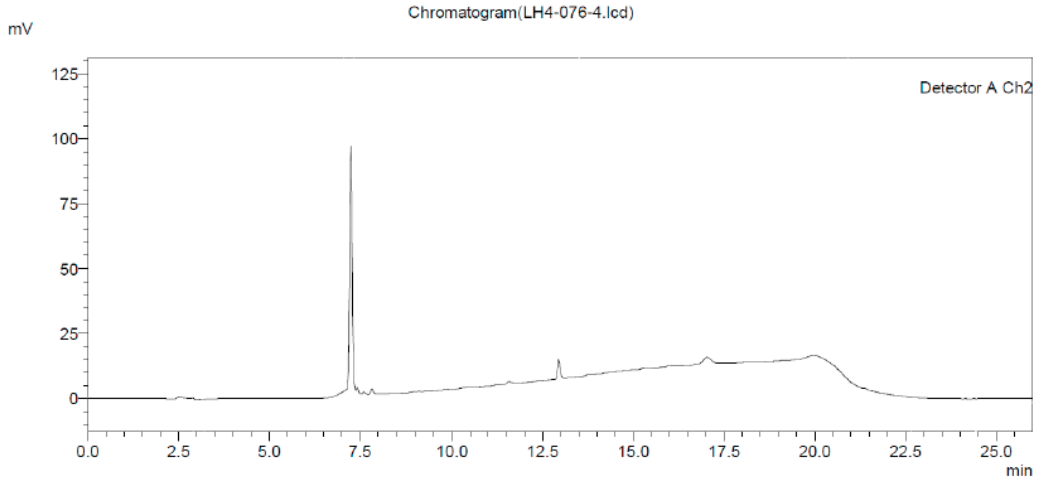


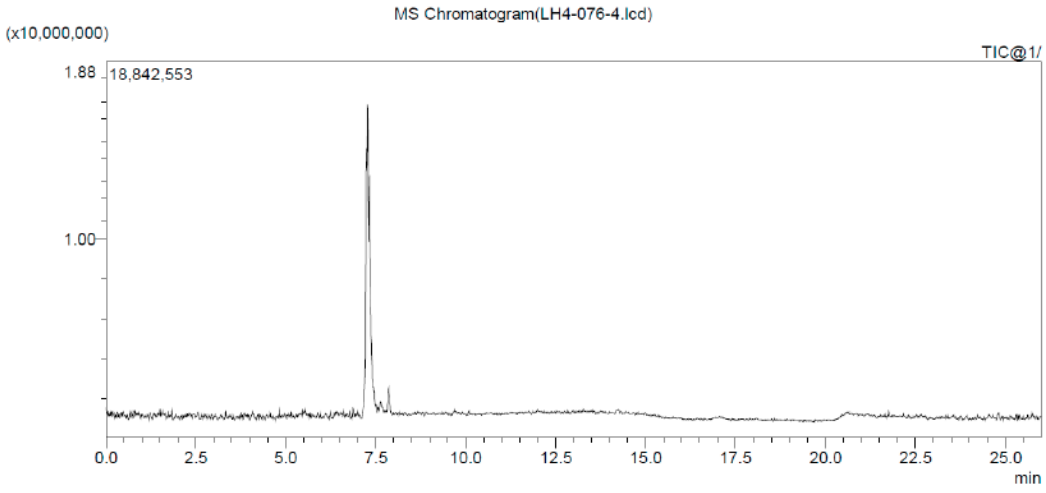


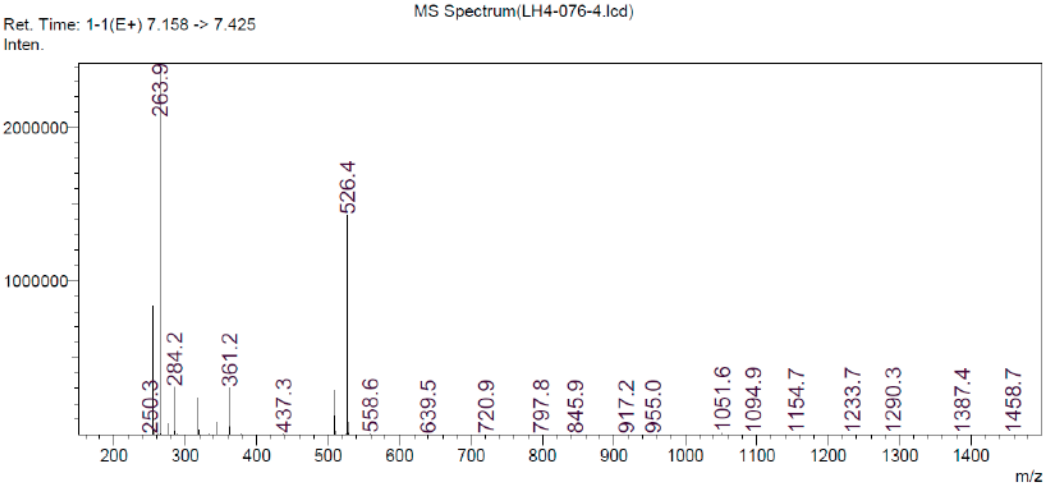
LC-MS (0→100% acetonitrile/water over 10 min) of **18**



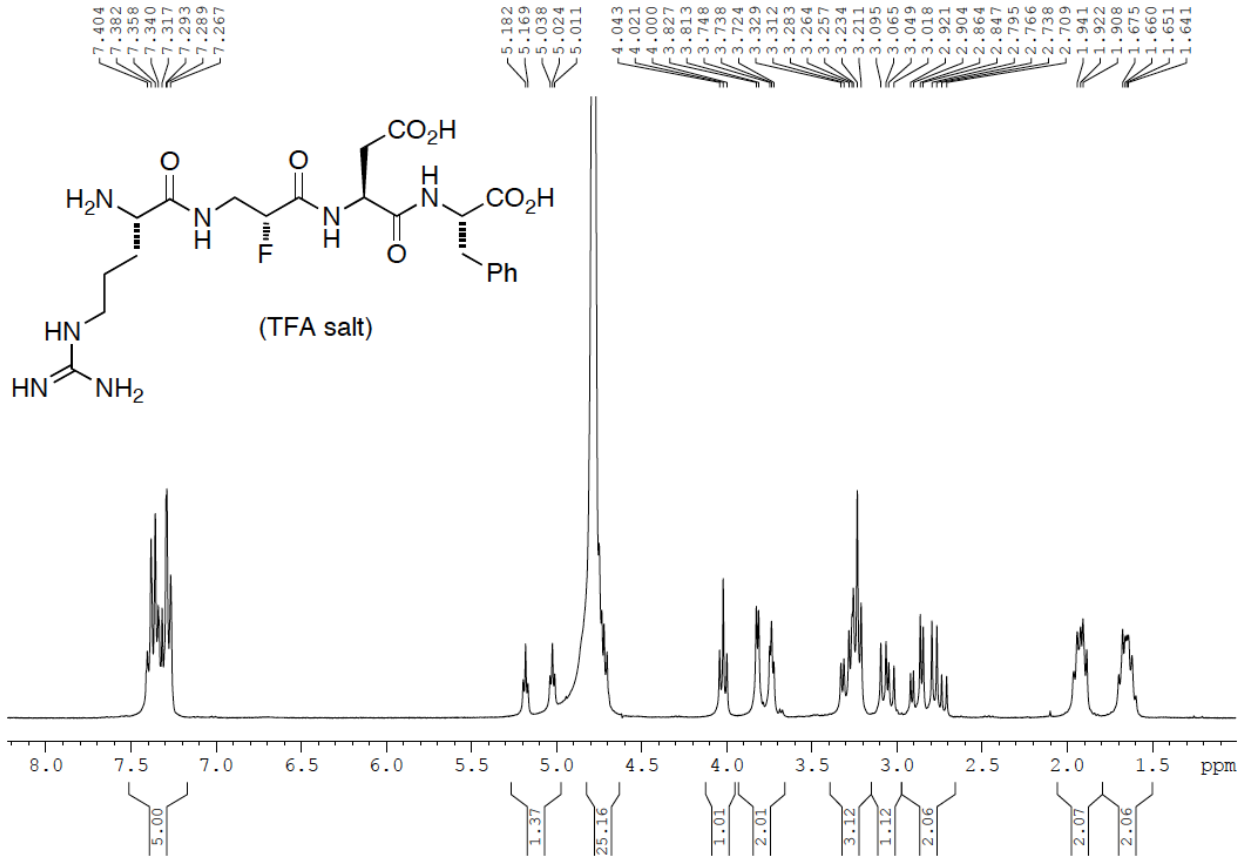
(TFA salt)



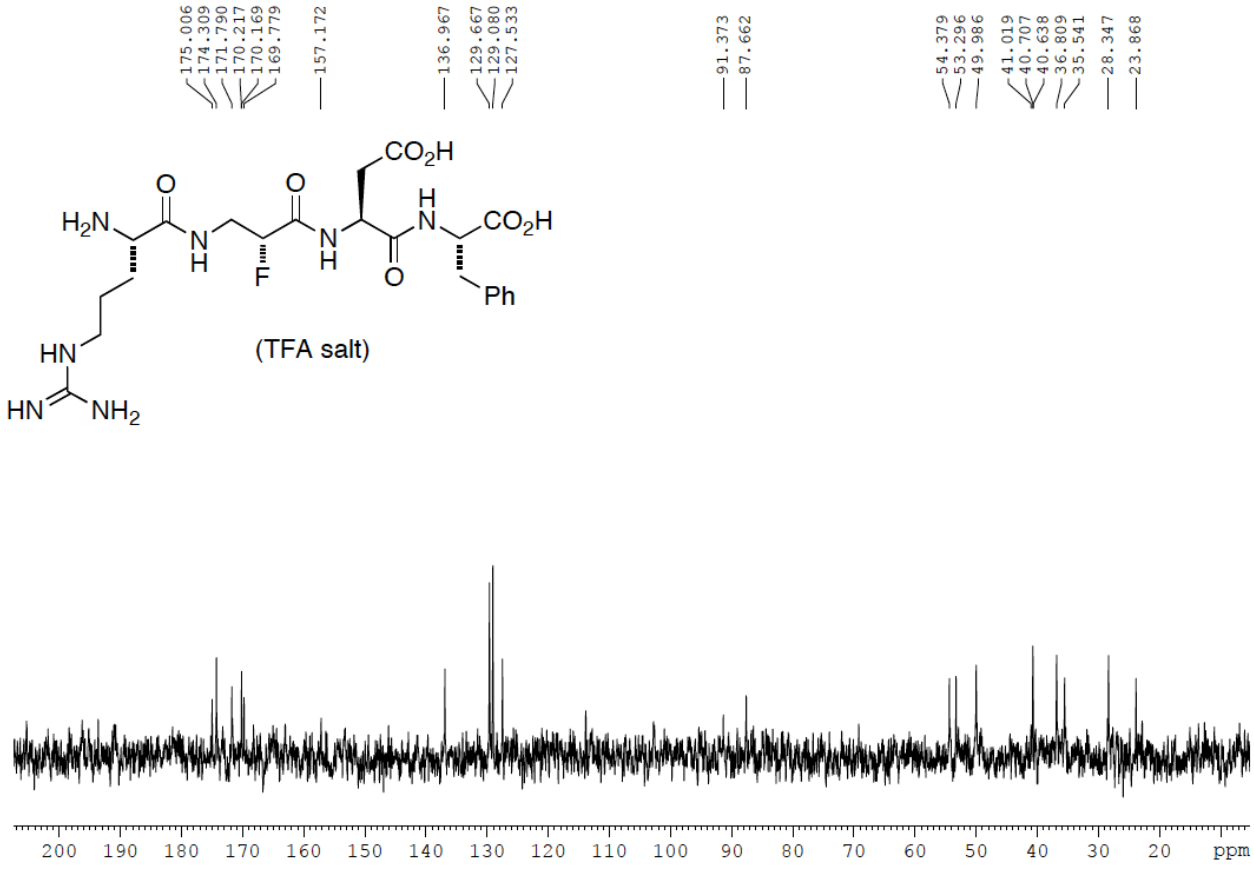




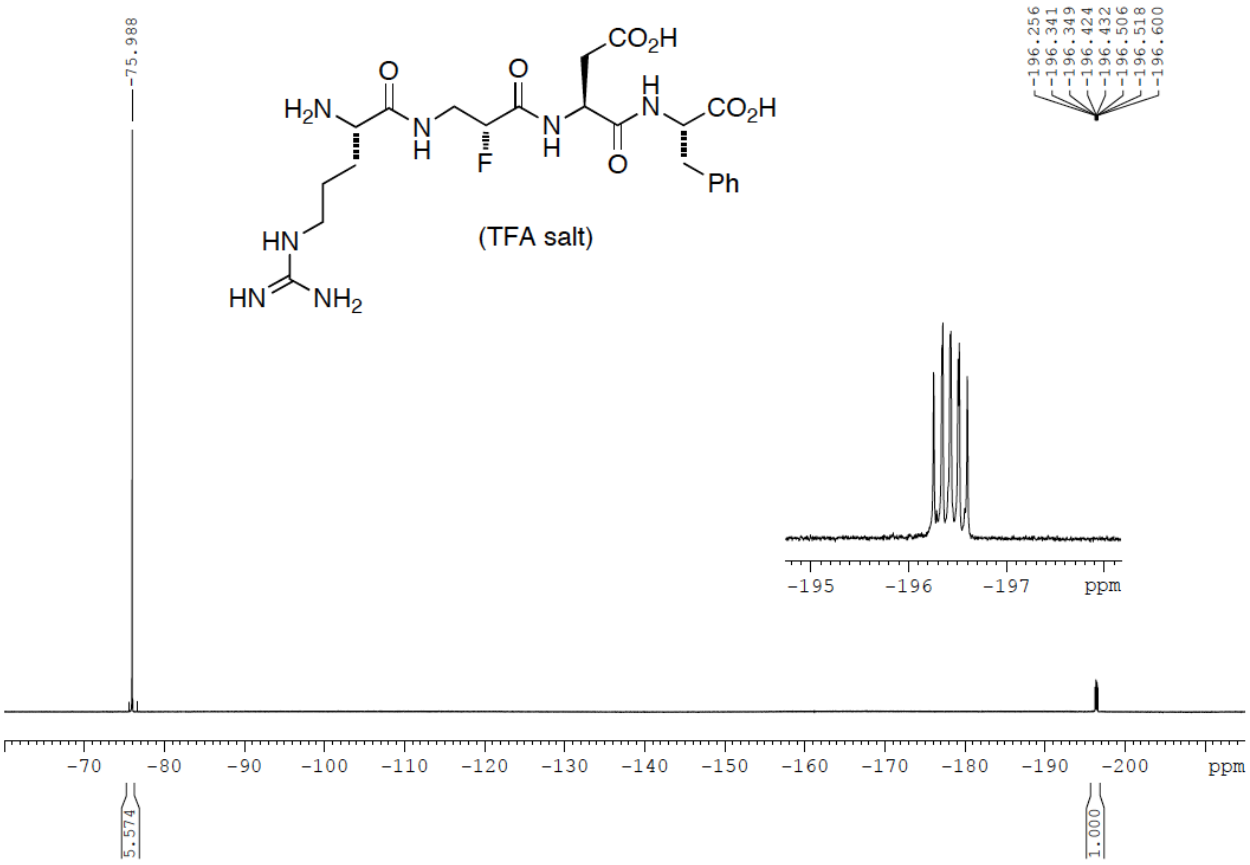
^{1}H NMR (300 MHz, D_2O) of **19**



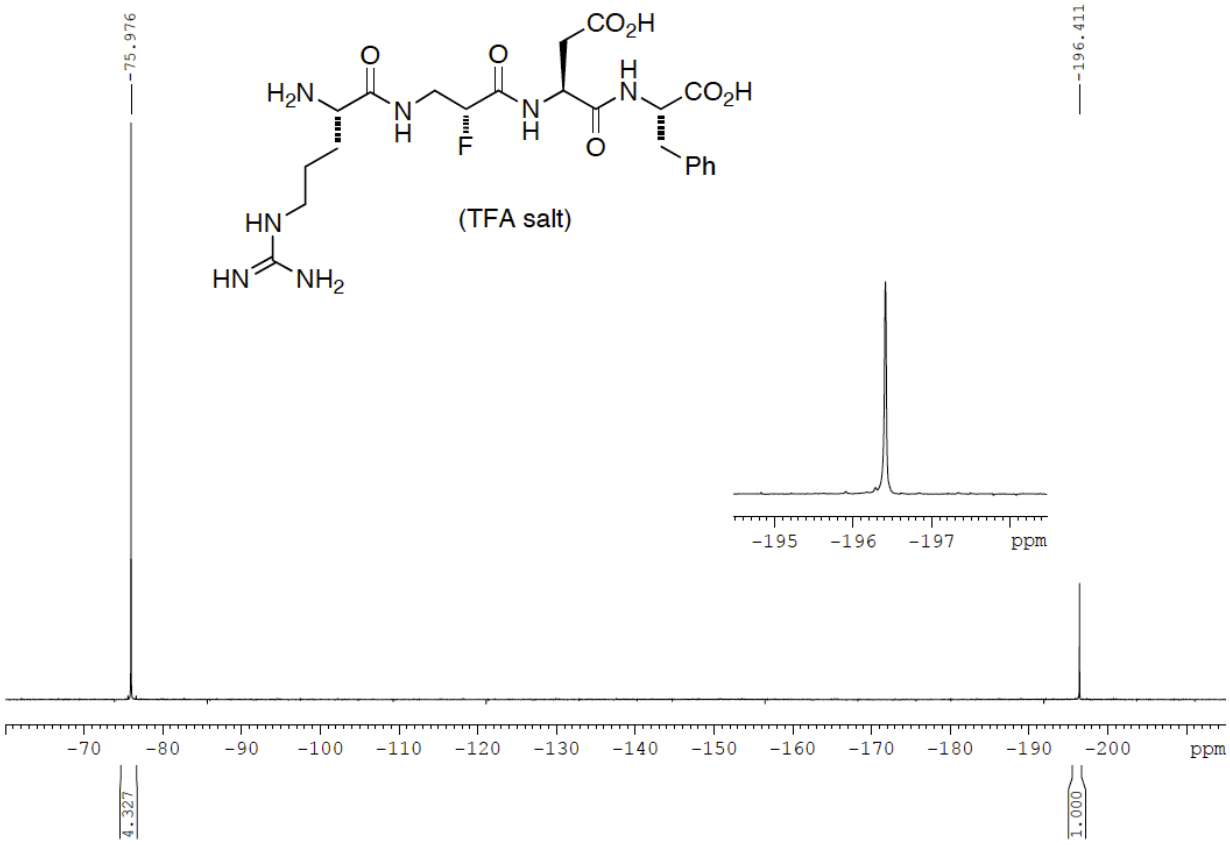
^{13}C {^{1}H} NMR (50 MHz, D_2O) of **19**



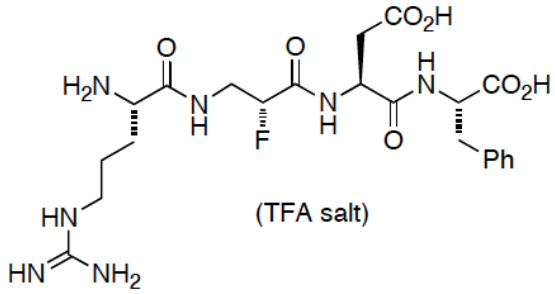
^{19}F NMR (282 MHz, D_2O) of **19**

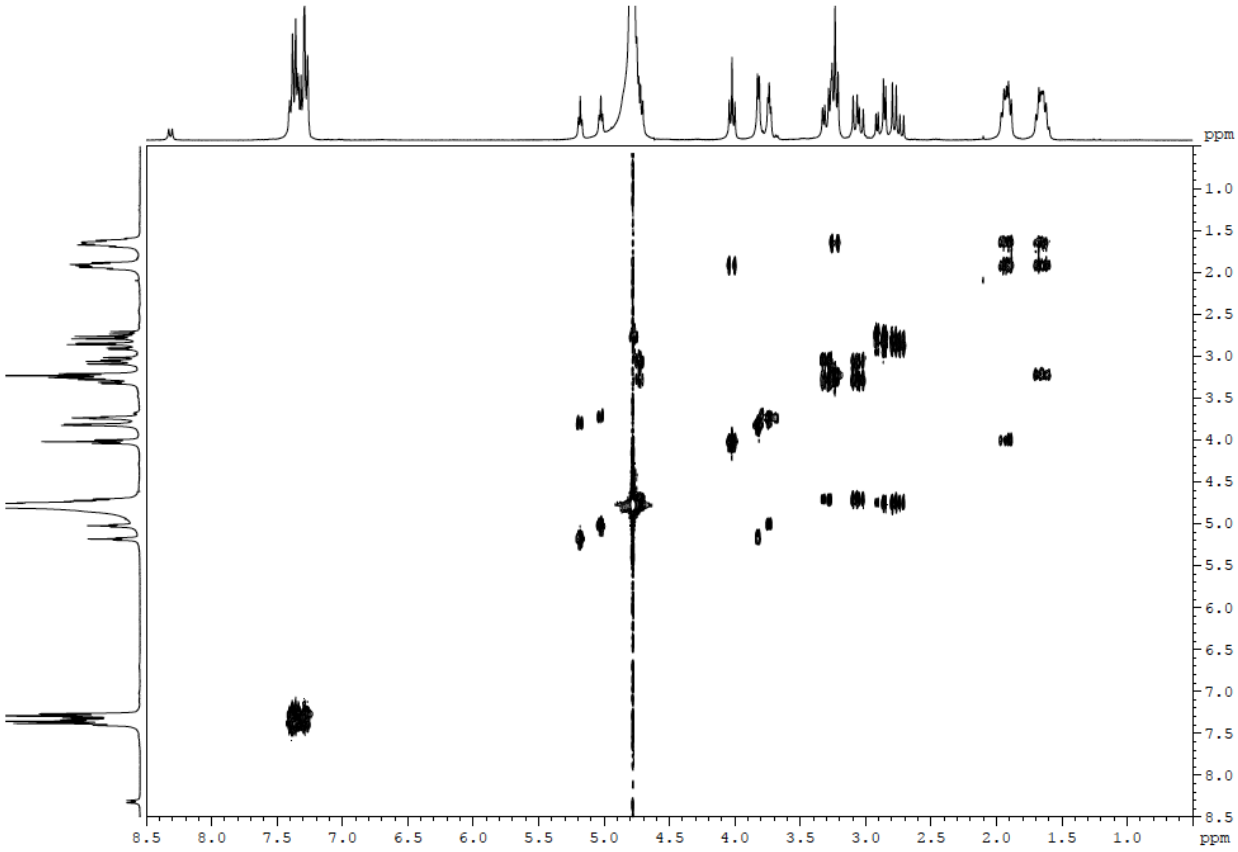


^{19}F {^{1}H} NMR (282 MHz, D_2O) of **19**

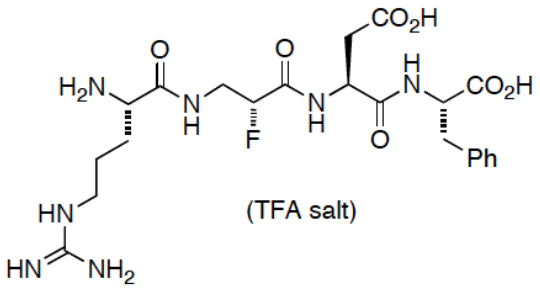


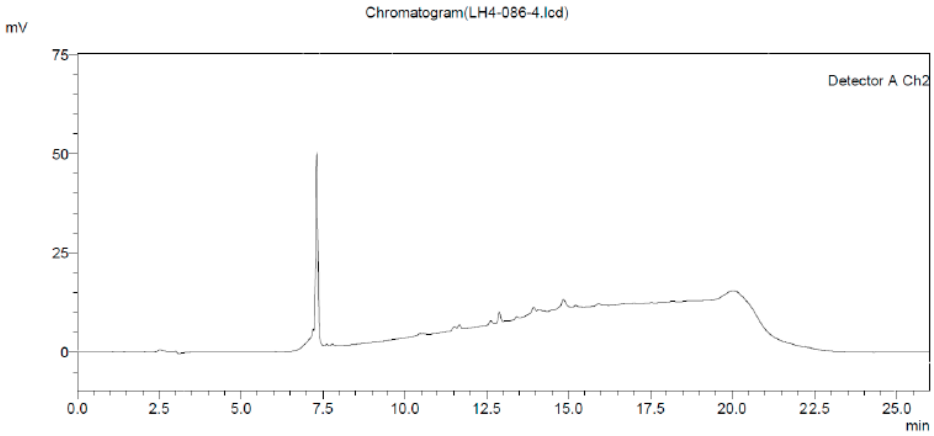
^{1}H-^{1}H COSY (300 MHz, D_2O) of **19**

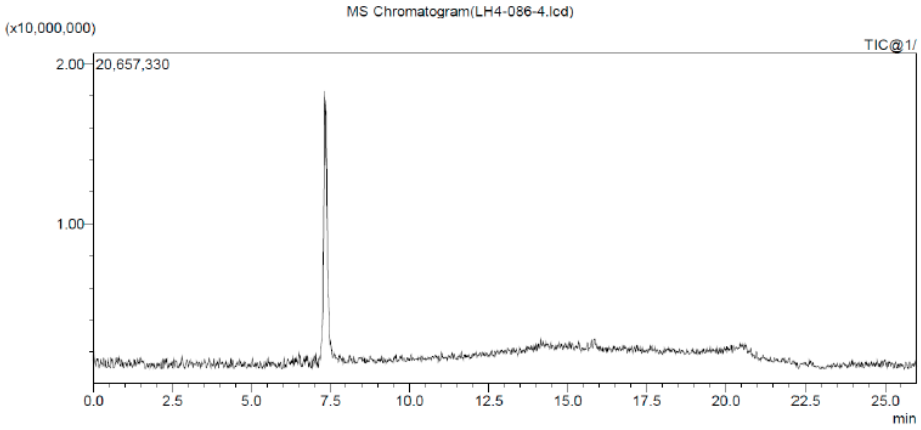


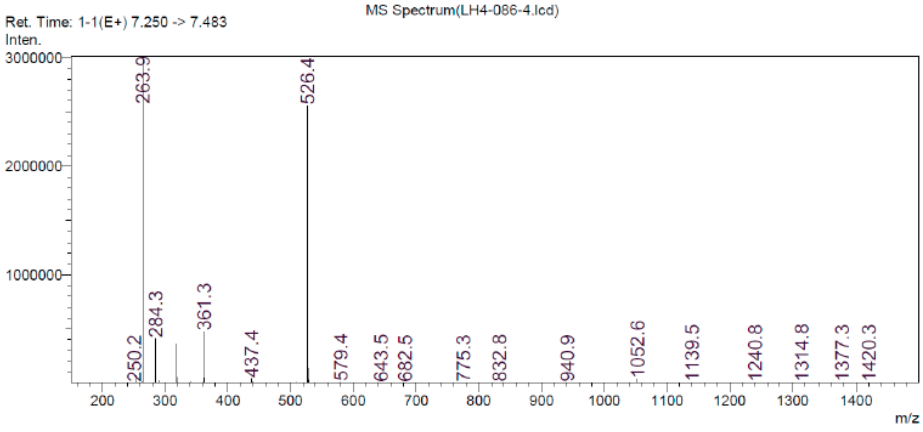


LC-MS (0→100% acetonitrile/water over 10 min) of **19**

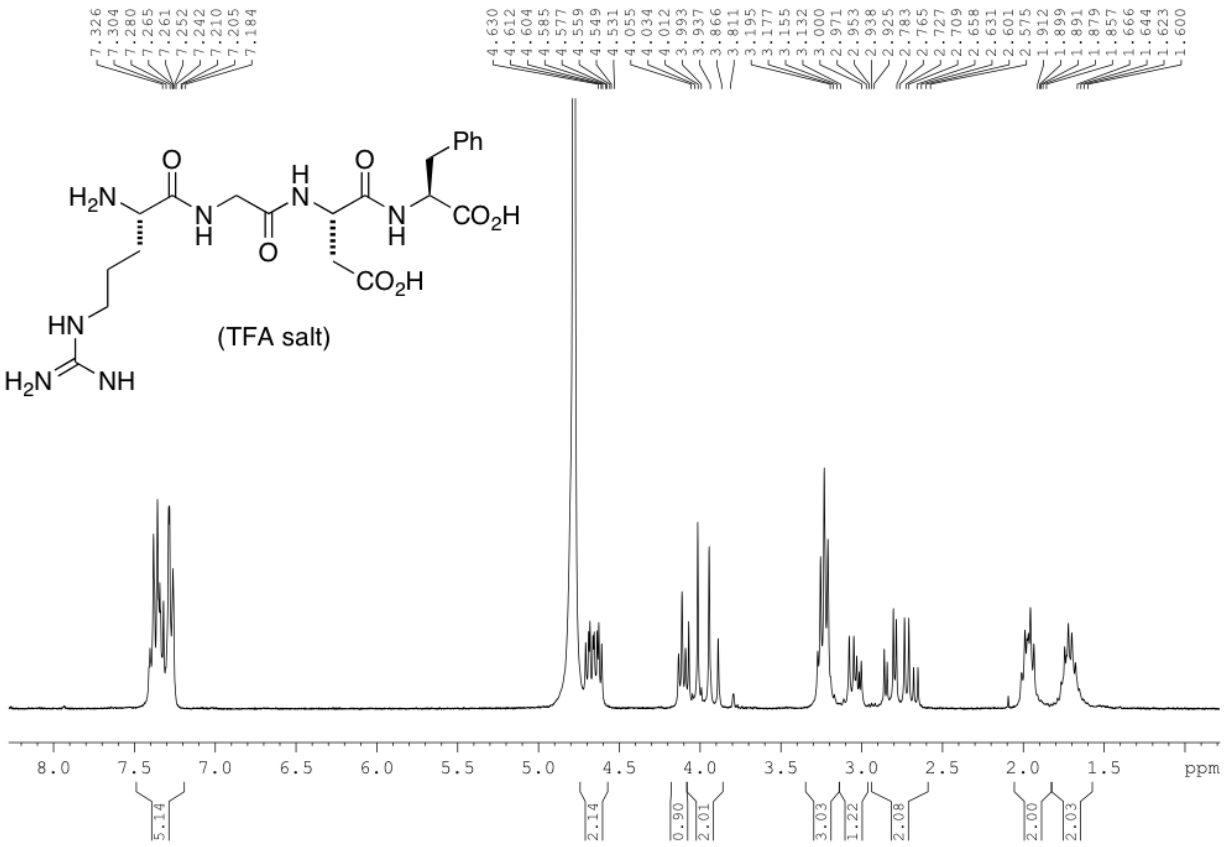




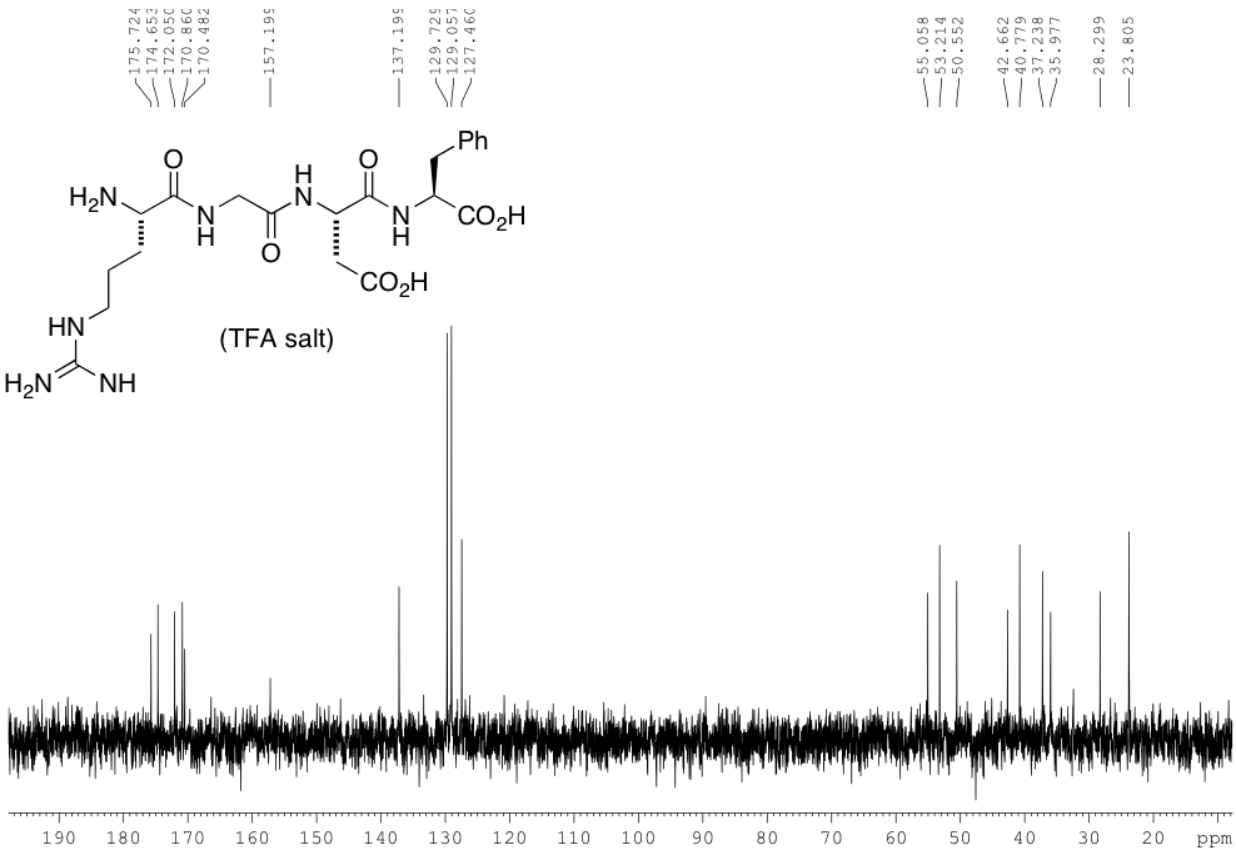




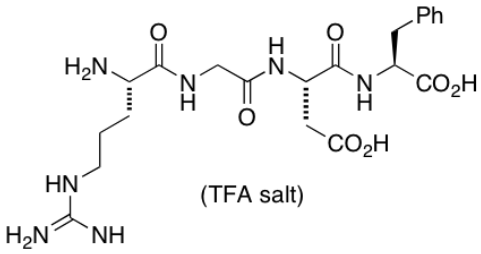
^{1}H NMR (300 MHz, D_2O) of **20**

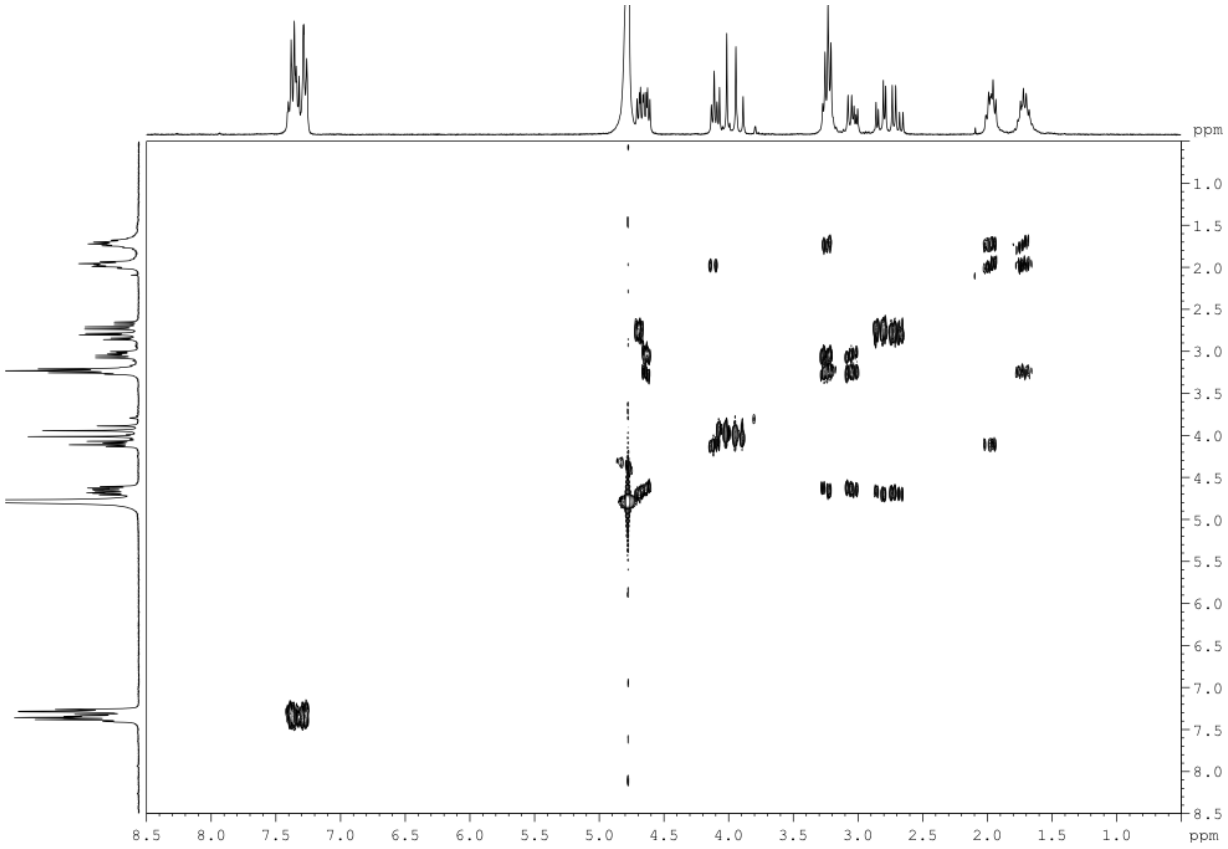


^{13}C {^{1}H} NMR (75 MHz, D_2O) of **20**

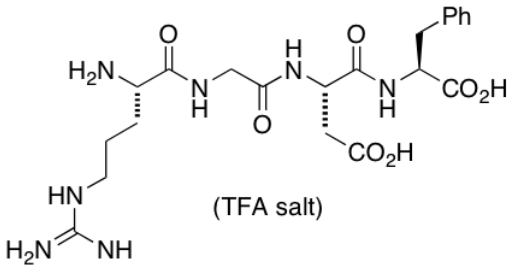


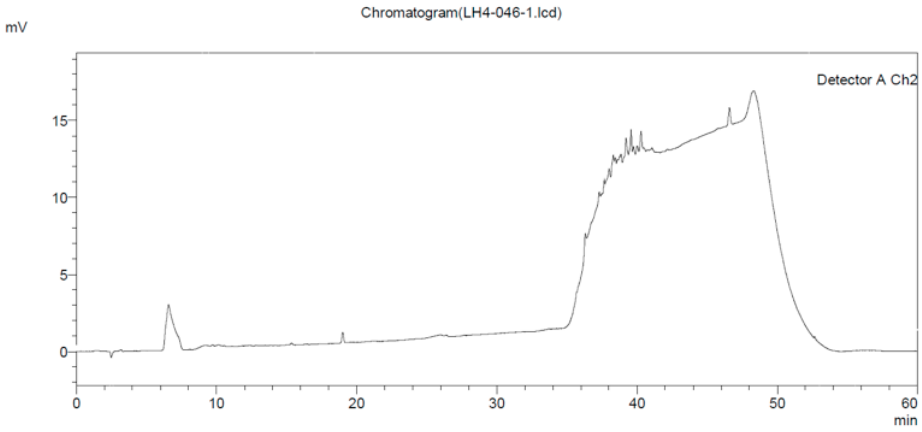
^{1}H-^{1}H COSY (300 MHz, D_2O) of **20**

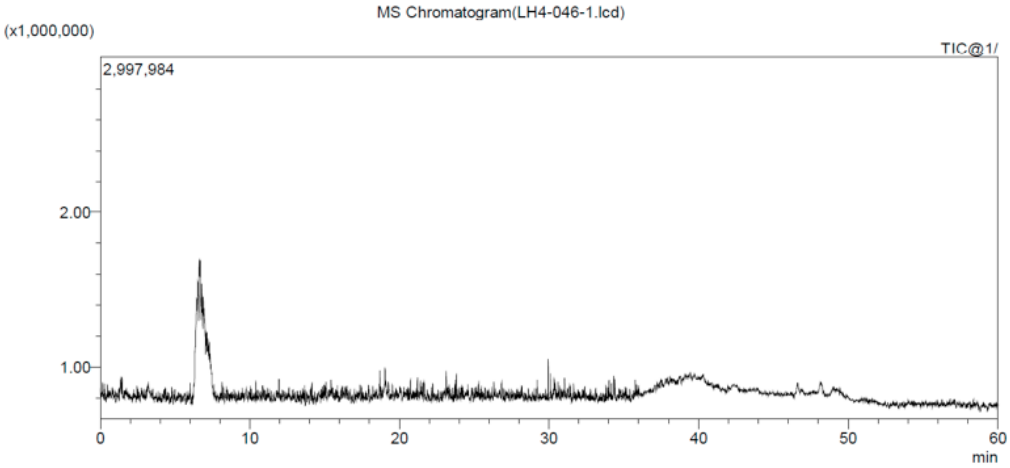




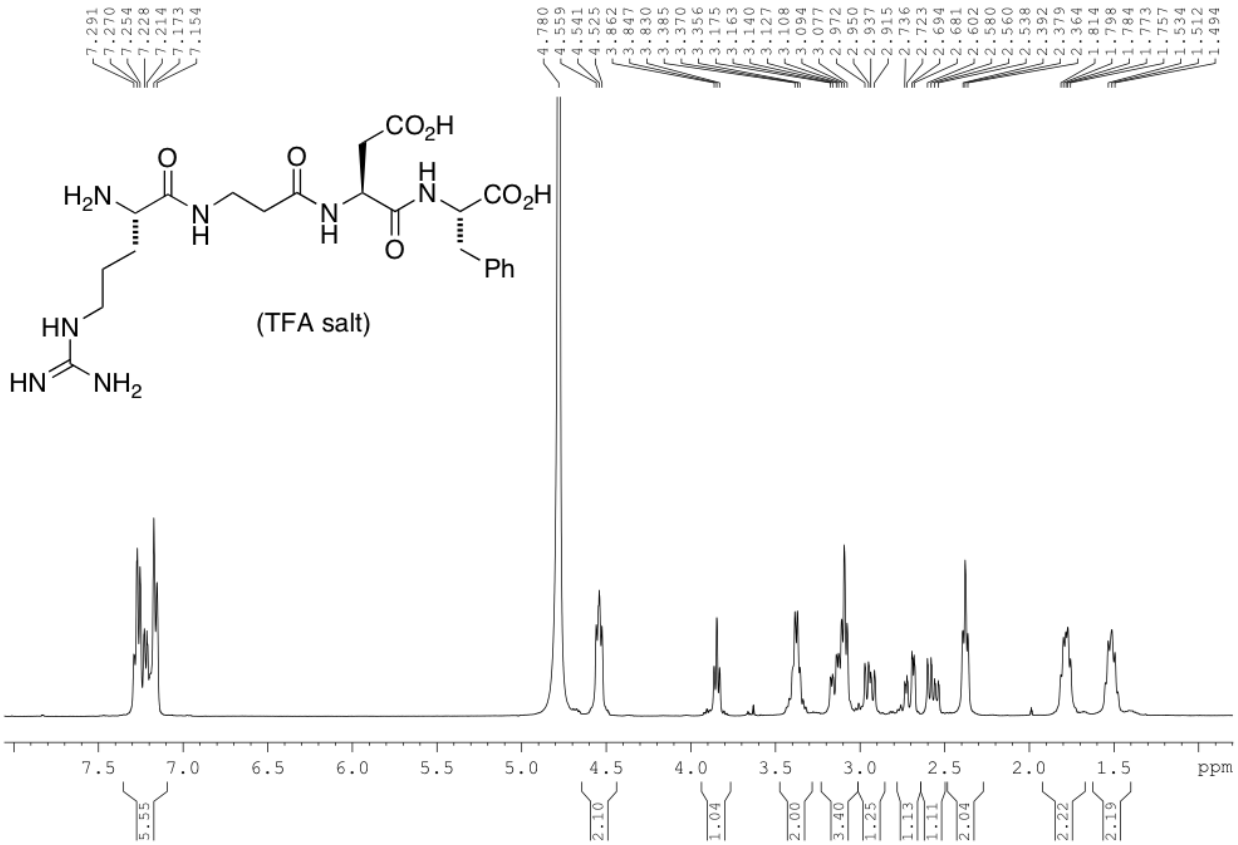
LC-MS (0→25% acetonitrile/water over 30 min) of **20**



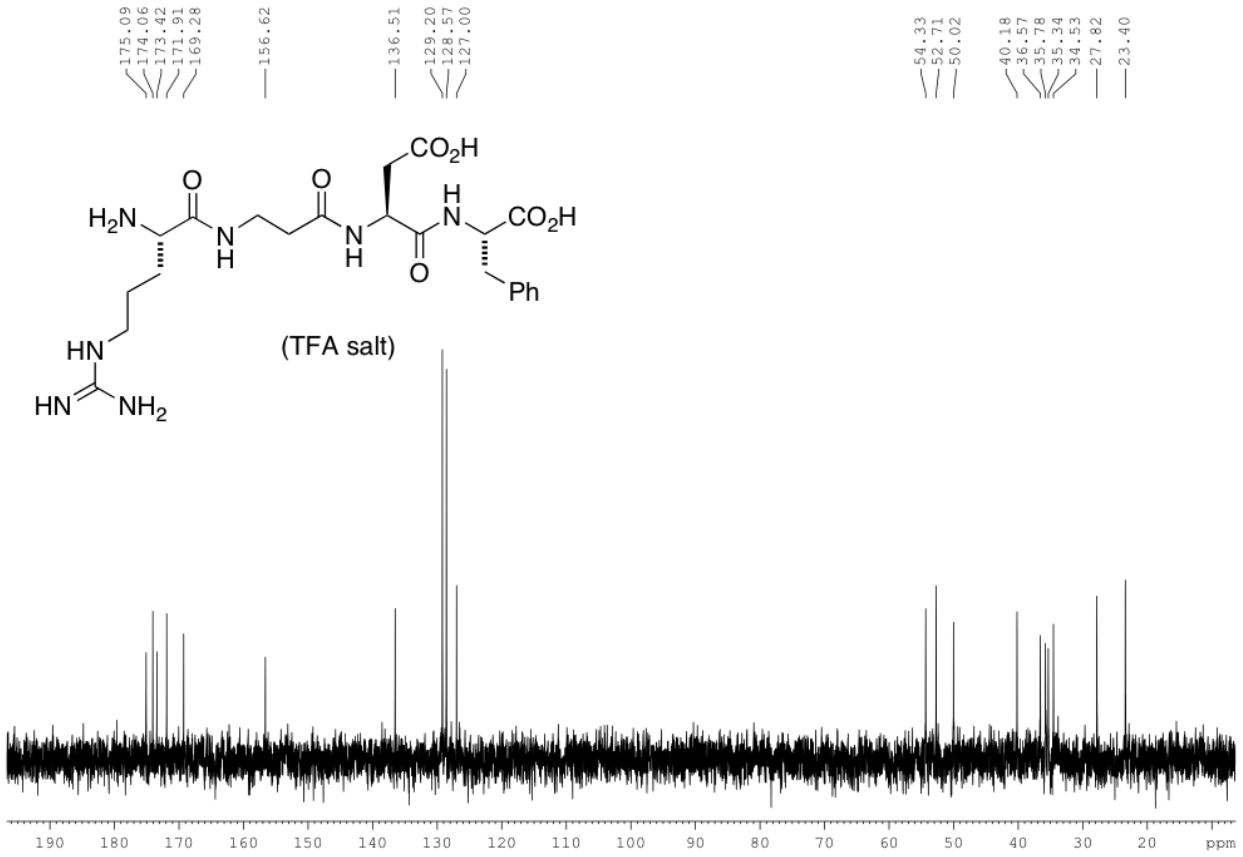




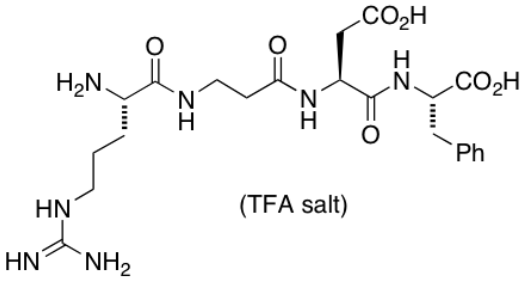
^{1}H NMR (400 MHz, D_2O) of **21**

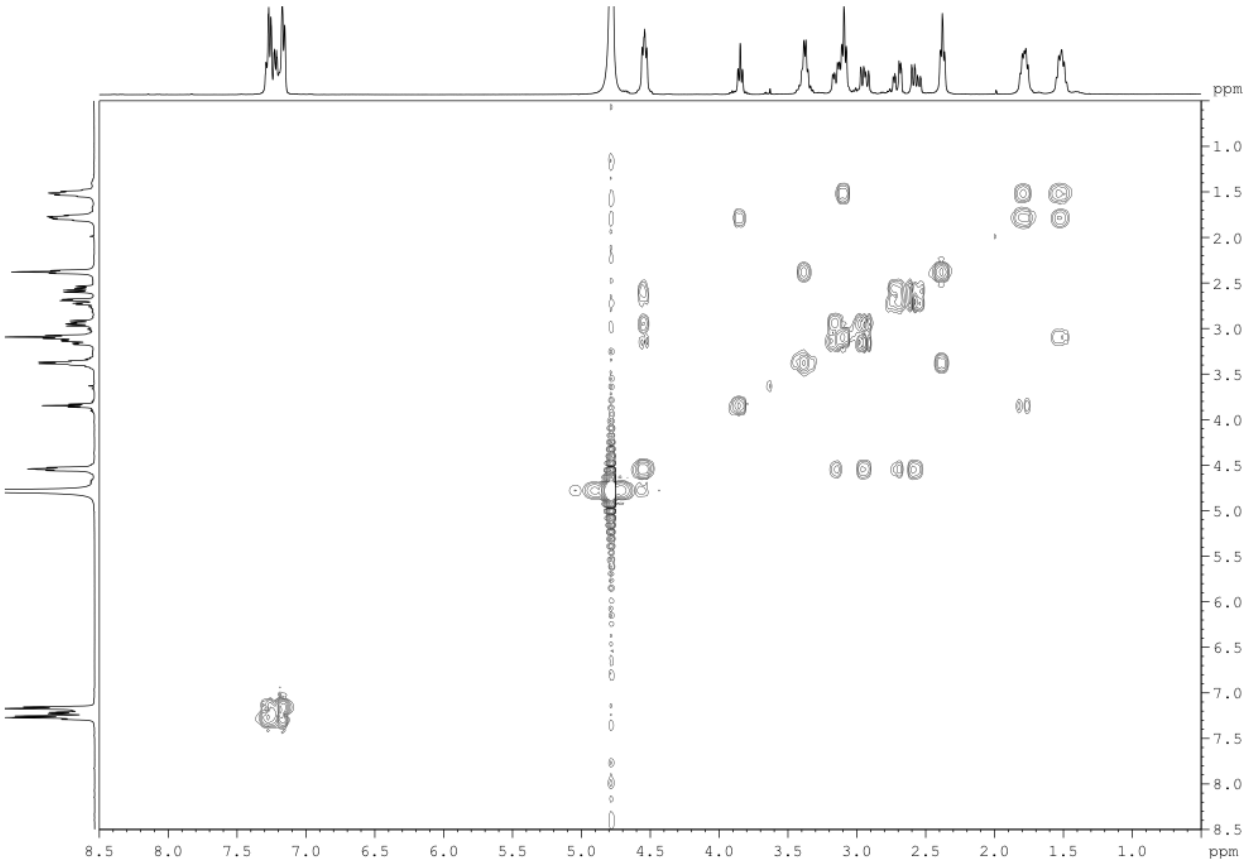


^{13}C {^{1}H} NMR (75 MHz, D_2O) of **21**

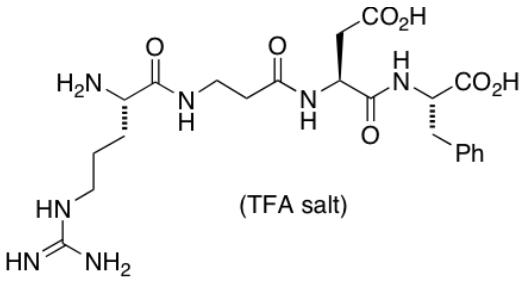


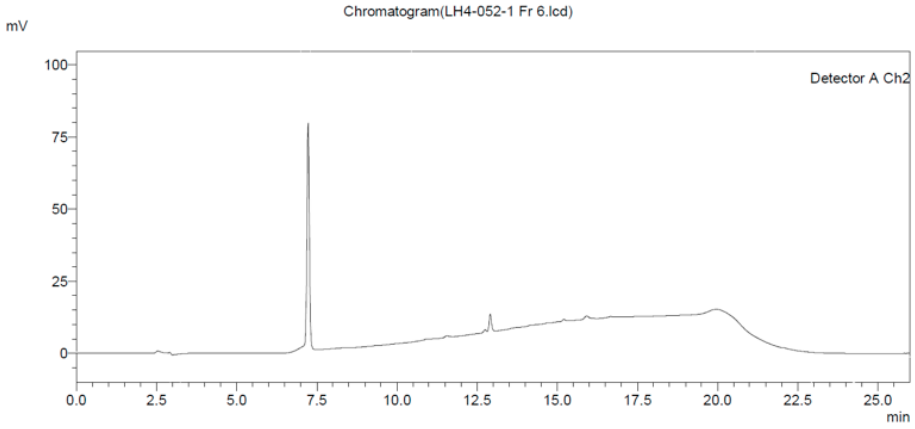
^{1}H-^{1}H COSY (300 MHz, D_2O) of **21**

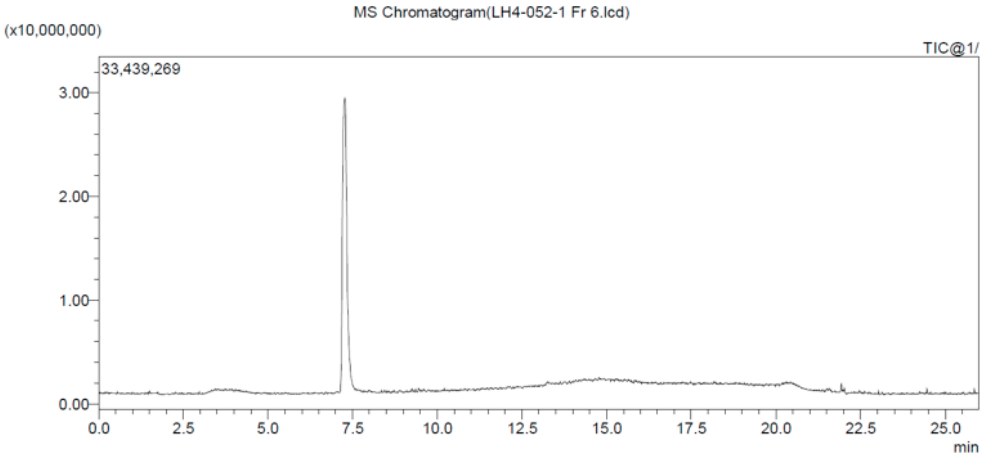


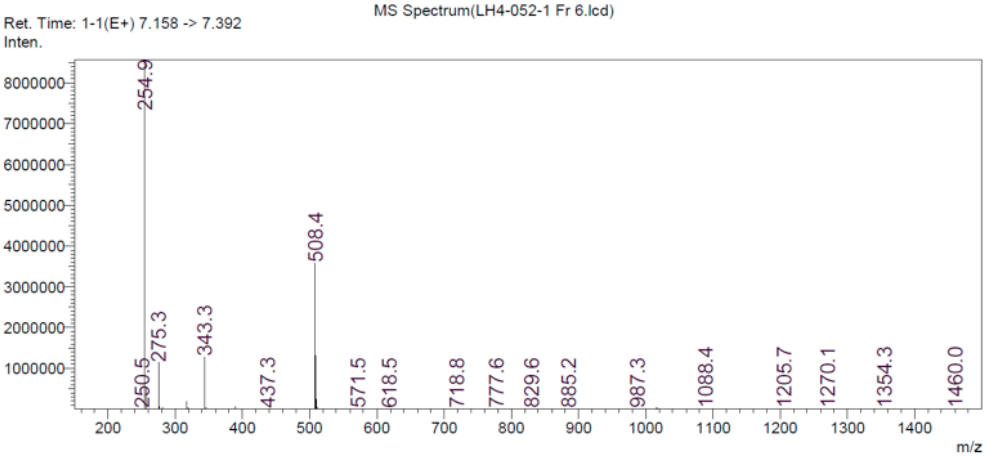


LC-MS (0→100% acetonitrile/water over 10 min) of **21**

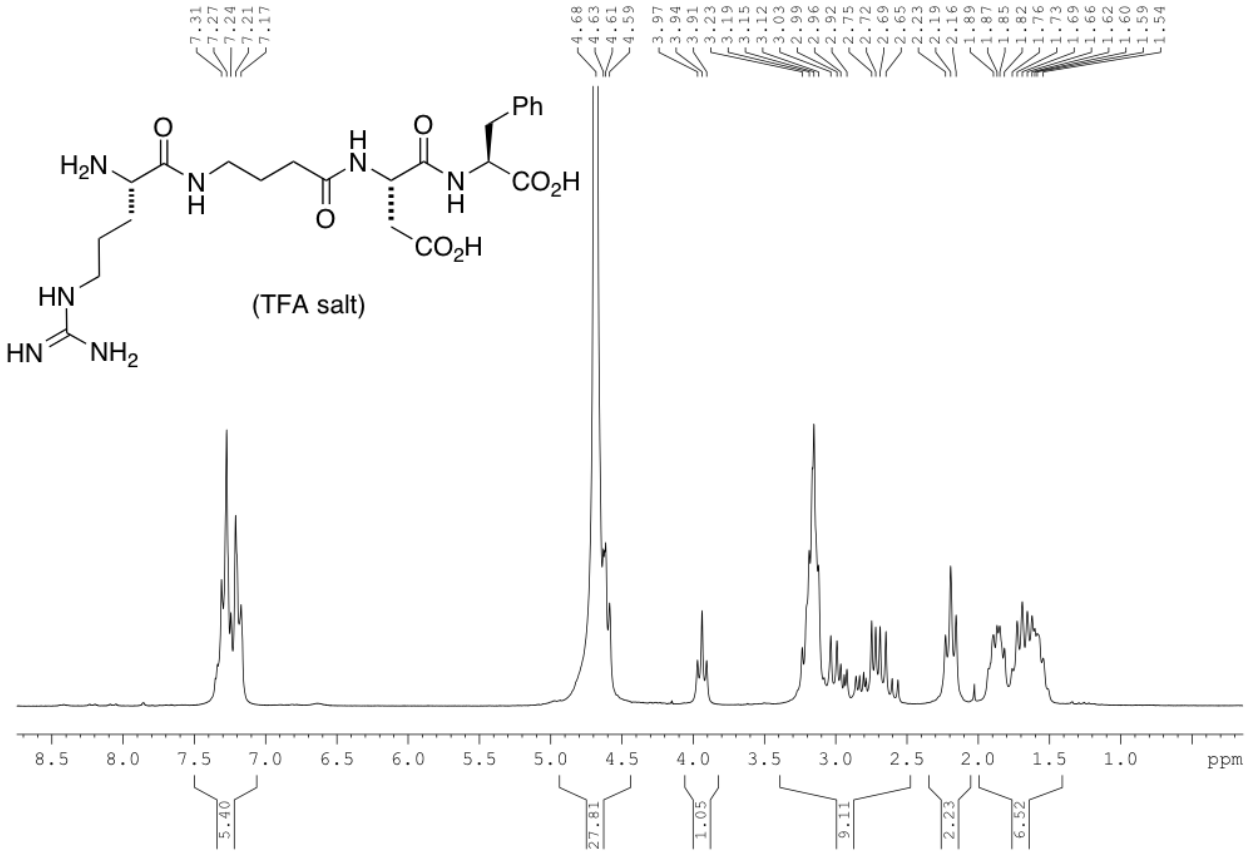




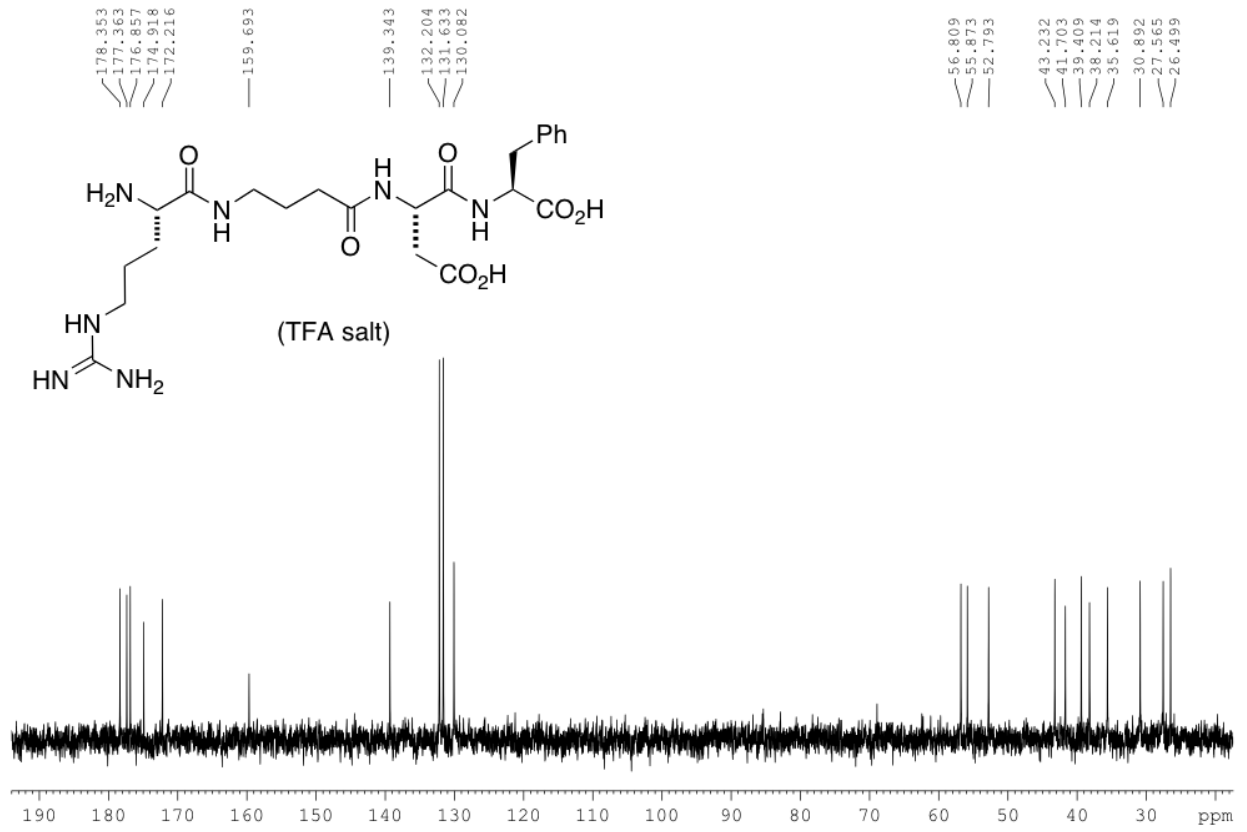




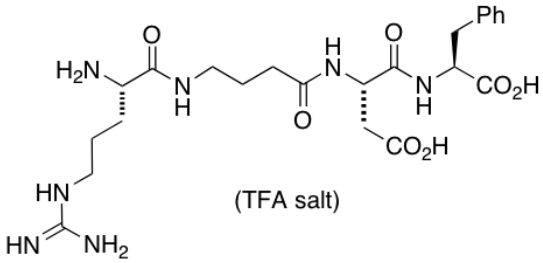
^{1}H NMR (200 MHz, D_2O) of **22**

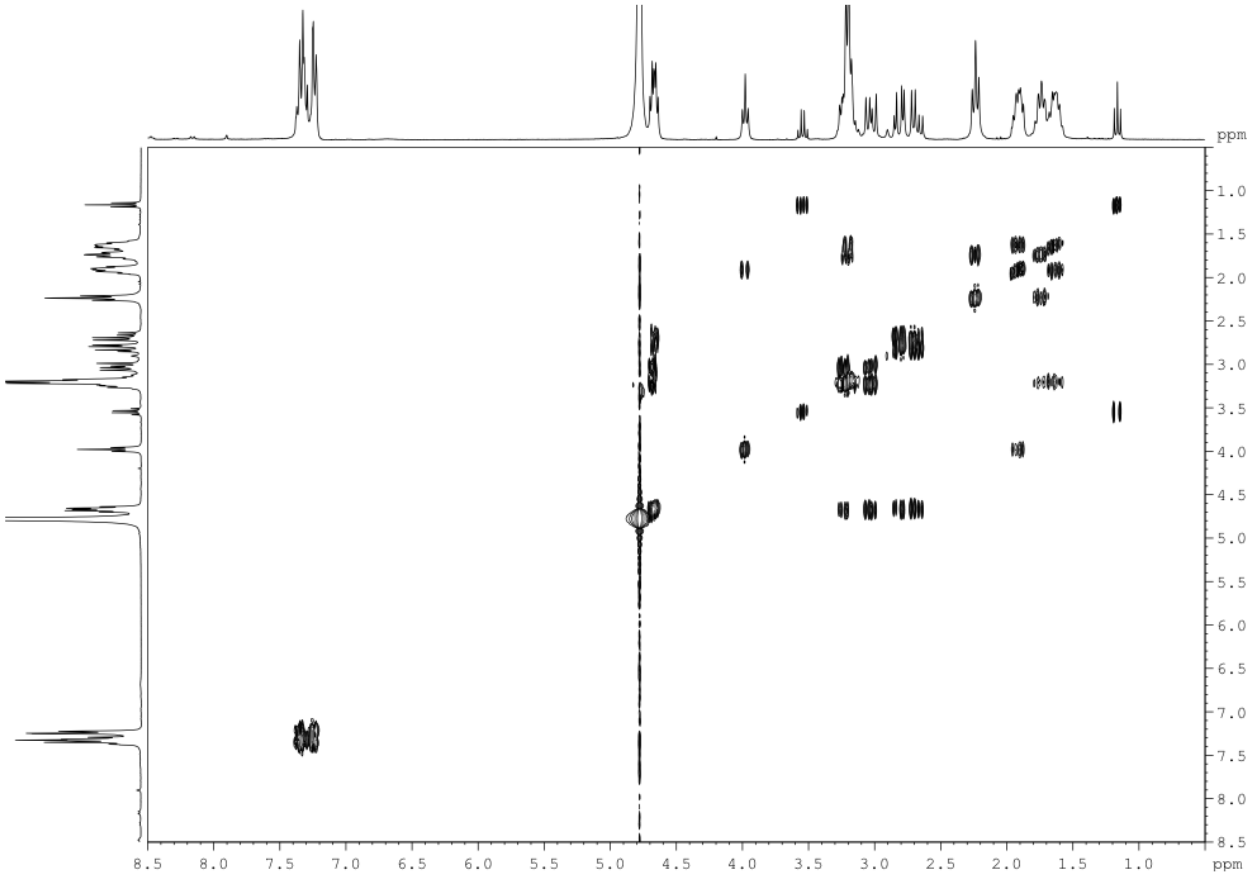


^{13}C {^{1}H} NMR (75 MHz, D_2O) of **22**

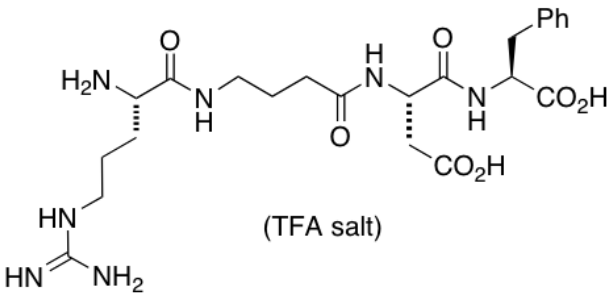


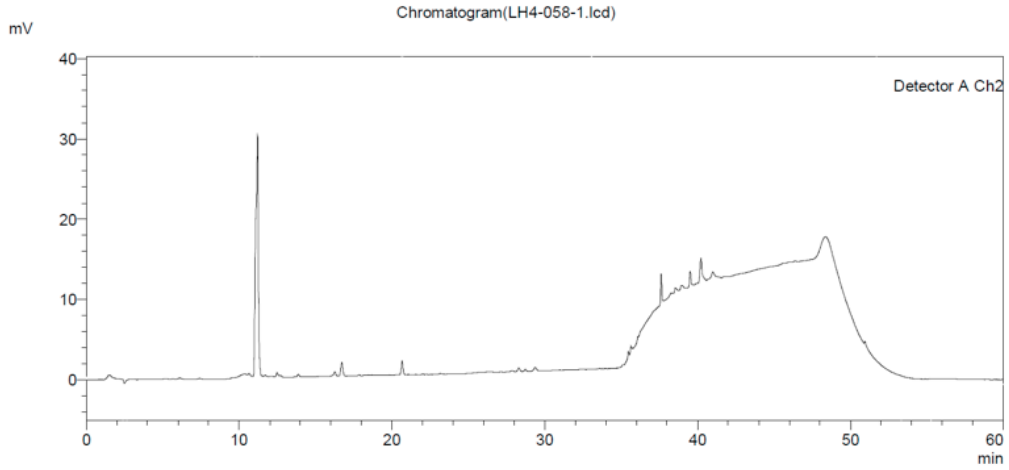
^{1}H-^{1}H COSY (300 MHz, D_2O) of **22**

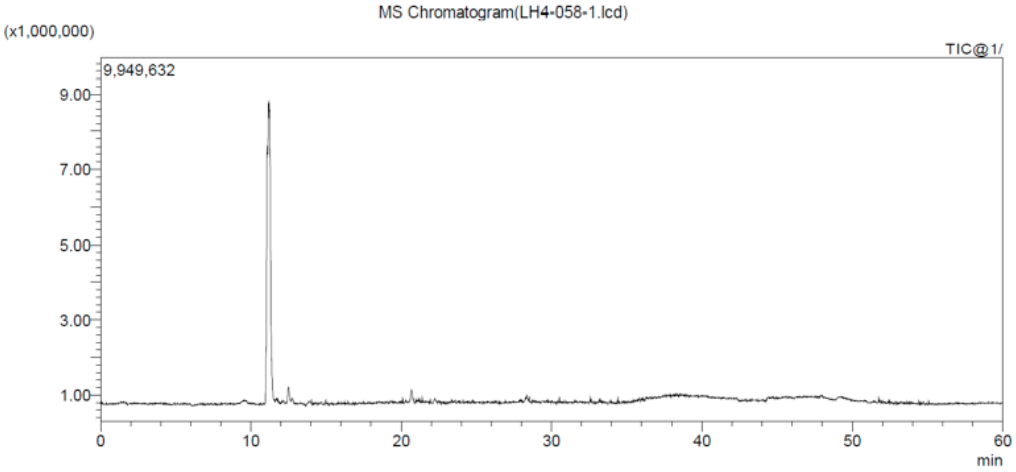


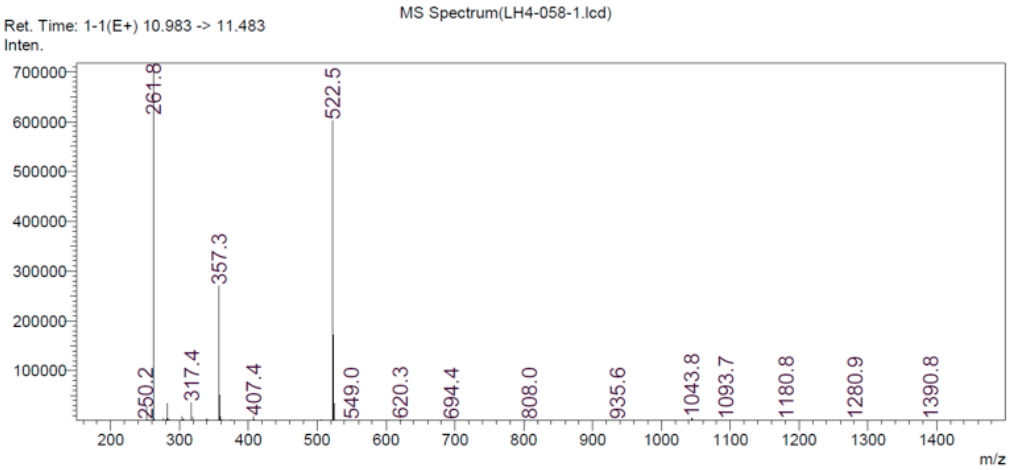


LC-MS (0→25% acetonitrile/water over 30 min) of **22**







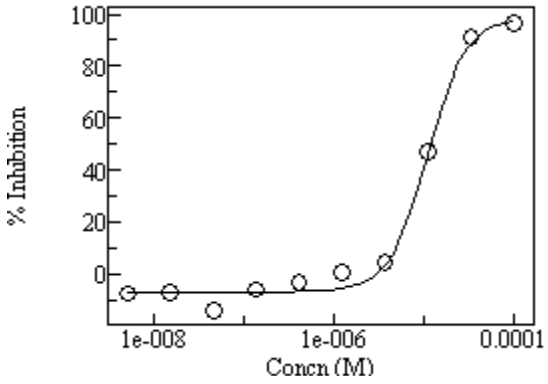


Cell adhesion assay data

[Note: pIC_{50} values for active antagonists are quoted, where $pIC_{50} = 4$ is 100 μM, 5 is 10 μM, 6 is 1 μM etc.]

Peptide	pIC_{50}			
	$\alpha_V\beta_3$	$\alpha_V\beta_6$	$\alpha_V\beta_5$	$\alpha_5\beta_1$
14	<4	<4	<4	<4
15	<4	<4	<4	<4
16	<4	<4	<4	<4
17	<4	<4	<4	<4
18	<4	<4	<4	<4
19	<4	<4	<4	<4
20	4.97 [a]	<4	4.59 [b]	<4
21	<4	<4	<4	<4
22	<4	<4	<4	<4

[a] Activity of **20** at $\alpha_V\beta_3$:



[b] Activity of **20** at $\alpha_V\beta_5$: