

Supporting Information

Synthesis and migrastatic activity of cytochalasin analogues lacking a macrocyclic moiety

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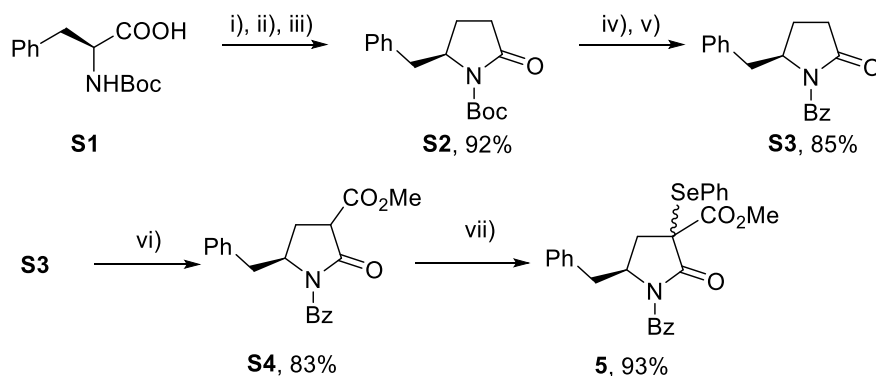
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Synthesis of compound 5

General remarks

Visualization methods for TLC: Compounds were visualized by irradiation with UV light (254 nm) and/or by treatment with a solution of phosphomolybdic acid (5 g), Ce(SO₄)₂·H₂O (2 g), conc. H₂SO₄ (12 mL) and H₂O (188 mL) followed by heating or treatment with a solution of anisaldehyde (9 mL) in EtOH (230 mL), conc. H₂SO₄ (8 mL) and conc. AcOH (3 mL) followed by heating or treatment with a solution of ninhydrin (0.2 g) in EtOH (100 mL), conc. AcOH (0.5 mL) and H₂O (4.5 mL) followed by heating.



Scheme S1: Preparation of selenide **5**. Reaction conditions: i) Meldrum's acid, EDC, DMAP, DCM, 0°C to RT, 3 h; ii) NaBH₄, AcOH, DCM, 0°C to RT; iii) toluene, reflux, 3 h; iv) TFA, DCM, 10°C-RT, 1 h; v) BzCl, py, RT, 4 h; vi) ClCO₂Me, LiHMDS, THF, -78°C, 4.5 h; vii) PhSeCl, LiHMDS, THF, -78°C, 5 h.

The *tert*-butyl (*R*)-2-benzyl-5-oxopyrrolidine-1-carboxylate (**S2**) was synthesized according to the known procedures¹ in three steps starting from (*tert*-butoxycarbonyl)-L-phenylalanine (**S1**, 7.96 g, 30.0 mmol). All three reactions were monitored by TLC (hexanes/EtOAc/AcOH : 7/3/0.5).^{1c}

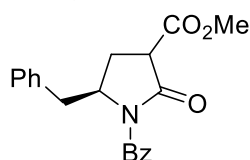
First step was amide coupling of **S1** and Meldrum's acid (4.54 g, 31.5 mmol, recrystallized) proceeded with EDC (8.63 g, 45.0 mmol) and DMAP (5.50 g, 45.0 mmol) in dry DCM (90 mL) at 0°C, then RT for 3 h^{1b,c}, followed by evaporation of a crude extract to ca. 100 mL and reduction by NaBH₄ (3.40 g, 90.0 mmol) and AcOH (17.2 mL, 300 mmol) at 0°C for 1 h, then RT overnight (after 3 h, the reaction is almost complete).^{1a,c} Crude product was refluxed in toluene (150 mL) for 3 h. After evaporation of the solvent, column chromatography of the residue (80 g SiO₂, 0-20% EtOAc in hexanes) furnished the **S2** (7.62 g, 92%), yellow oil. Our physical and spectroscopic data corroborated previously published materials.^{1a}

The (*R*)-1-benzoyl-5-benzylpyrrolidin-2-one (**S3**) was synthesized according to the known procedures^{1d}, TFA (7.6 mL) was added to **S2** (7.62 g, 27.6 mmol) in DCM (30.5 mL) at 10°C dropwise. The reaction mixture was stirred at RT until full conversion (1 h, TLC, hexanes/EtOAc : 4/1). BzCl (6.35 mL, 55.2 mmol) was added dropwise to the crude

¹ a) M. Smrčina, P. Majer, E. Majerová, T. A. Guerassina and M. A. Eissenstat, *Tetrahedron*, 1997, **53**, 12867-12874; b) S. Shankar, N. A. Wani, U. P. Singh and R. Rai, *Chemistry Select*, 2016, **1**, 3675-3678; c) O. Chaloin, F. Cabart, J. Marin, H. Zhang and G. Guichard, *Organic Syntheses*, 2008, **85**, 147-157; d) B. Hao, M. J. Gunaratna, M. Zhang, S. Weerasekara, S. N. Seiwald, V. T. Nguyen, A. Meier and D. H. Hua, *Journal of the American Chemical Society*, 2016, **138**, 16839-16848.

product in pyridine (69 mL) at RT.² After full conversion (4 h, TLC, DCM/MeOH : 20/1), Et₂O (173 mL, 2.5 vol of py) was added, precipitate was filtered, filtrate concentrated and co-distilled with toluene (2×) under reduced pressure. Column chromatography of the residue (80 g SiO₂, 10-25% EtOAc in hexanes) furnished the **S3** (6.55 g, 85%), yellow oil. Our physical and spectroscopic data corroborated previously published materials.³

Methyl (5*R*)-1-benzoyl-5-benzyl-2-oxopyrrolidine-3-carboxylate (**S4**)



This compound was synthesized in analogy with a known procedure⁴ from **S3** (0.57 g, 2.0 mmol) in dry THF (3 mL), using LiHMDS (4.0 mL, 4.0 mmol, 1M in THF) and neat methyl chloroformate (0.31 mL, 4.0 mmol). After full conversion (4 h, TLC, hexanes/EtOAc : 5/1) column chromatography of the residue on silica gel (0-20% EtOAc in hexanes) furnished **S4** (0.57 g, 83%) as a 1.7/1 diastereomeric mixture, yellow oil.

¹H NMR (400 MHz, CDCl₃)

major: δ 7.63–7.58 (m, 2H, 2×Ar), 7.58–7.50 (m, 1H *overlapped*, Ar), 7.47–7.39 (m, 2H *overlapped*, 2×Ar), 7.38–7.21 (m, 5H *overlapped*, Ar), 4.87 (dddd, $J_{5,4a} = 8.6$ Hz, $J_{5,Ph-CHb} = 7.9$ Hz, $J_{5,Ph-CHa} = 3.5$ Hz, $J_{5,4b} = 2.7$ Hz, 1H, H-5), 3.71 (s, 3H, COOCH₃), 3.35 (dd, $J_{3,4a} = 10.0$ Hz, $J_{3,4b} = 9.1$ Hz, 1H, H-3), 3.24 (dd, $J_{gem} = 13.5$ Hz, $J_{Ph-CHa,5} = 3.5$ Hz, 1H, Ph-CHa), 2.98 (dd, $J_{gem} = 13.5$ Hz, $J_{Ph-CHb,5} = 7.9$ Hz, 1H, Ph-CHb), 2.56 (ddd, $J_{gem} = 13.2$ Hz, $J_{4a,3} = 10.0$ Hz, $J_{4a,5} = 8.6$ Hz, 1H, H-4a), 2.34-2.18 (m, 1H *overlapped*, H-4b) ppm.

minor: δ 7.72–7.66 (m, 2H, 2×Ar), 7.58–7.50 (m, 1H *overlapped*, Ar), 7.47–7.39 (m, 2H *overlapped*, 2×Ar), 7.38–7.21 (m, 5H *overlapped*, Ar), 4.62 (dddd, $J_{5,Ph-CHb,5} = 9.8$ Hz, $J_{5,4} = 7.5$ Hz, $J_{5,4} = 7.0$ Hz, $J_{5,Ph-CHb,5} = 3.6$ Hz, 1H, H-5), 3.80 (s, 3H, COOCH₃), 3.53 (dd, $J_{3,4a} = 9.8$ Hz, $J_{3,4b} = 8.4$ Hz, 1H, H-3), 3.48 (dd, $J_{gem} = 13.2$ Hz, $J_{Ph-CHa,5} = 3.6$ Hz, 1H, Ph-CHa), 2.78 (dd, $J_{gem} = 12.9$ Hz, $J_{Ph-CHb,5} = 9.9$ Hz, 1H, Ph-CHb), 2.34-2.18 (m, 2H *overlapped*, H-4a,b) ppm.

¹³C NMR (101 MHz, CDCl₃)

major: δ 170.02 (C=O), 169.50 (C=O), 168.97 (C=O), 136.17 (C), 134.13 (C), 132.12 (CH-Ar), 129.67 (2×CH-Ar), 128.80 (2×CH-Ar), 128.79 (2×CH-Ar), 127.88 (2×CH-Ar), 127.19 (CH-Ar), 56.60 (CH-5), 52.83 (COOCH₃), 48.84 (CH-3), 38.99 (Ph-CH₂), 26.31 (CH₂-4) ppm.

minor: δ 170.93 (C=O), 170.16 (C=O), 170.10 (C=O), 136.39 (C), 144.09 (C), 132.69 (CH-Ar), 129.59 (2×CH-Ar), 129.38 (2×CH-Ar), 128.64 (2×CH-Ar), 128.01 (2×CH-Ar), 126.89 (CH-Ar), 57.24 (CH-5), 52.98 (COOCH₃), 49.12 (CH-3), 39.20 (Ph-CH₂), 26.38 (CH₂-4) ppm.

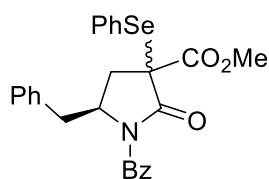
HRMS (ESI) m/z calcd for C₂₀H₁₉O₄NNa⁺ [M+Na]⁺ 360.1206; found 360.1204.

² E. J. Thomas and M. Willis, *Organic & Biomolecular Chemistry*, 2014, **12**, 7537-7550; this approach furnished easily separable mixture and higher yields compared to BzCl/NaH procedure.

³ S. A. Harkin, O. Singh and E. J. Thomas, *Journal of the Chemical Society, Perkin Transactions I*, 1984, 1489-1499.

⁴ X. Long, Y. Ding and J. Deng, *Angewandte Chemie, International Edition*, 2018, **57**, 14221-14224.

Methyl (5S)-1-benzoyl-5-benzyl-2-oxo-3-(phenylselanyl)pyrrolidine-3-carboxylate



LiHMDS (4.5 mL, 4.5 mmol, 1M in THF) was added dropwise to a stirred solution of **S4** (1.71 g, 5.1 mmol) in dry THF (10 mL) at -78°C under argon atmosphere. After 40 min, a solution of PhSeCl (0.86 g, 4.5 mmol) in dry THF (9 mL) was added dropwise and the reaction mixture was stirred at -78°C until full conversion (3-4 h, TLC, hexanes/EtOAc : 5/1). Then, solution of NH_4Cl (10 mL) was added, the resulting mixture was warmed to RT, poured into a solution of NH_4Cl (40 mL) and extracted with EtOAc (2×15 mL). The organics were combined, washed with brine (10 mL), dried (Na_2SO_4) and concentrated under reduced pressure. Column chromatography of the residue on silica gel (12-15% EtOAc in hexanes) furnished the **5** (2.32 g, 93%) as a 1.7/1 diastereomeric mixture, red-brown dense oil.

$^1\text{H NMR}$ (400 MHz, CDCl_3)

major: δ 7.74–7.21 (m, 13H *overlapped*, $13 \times \text{Ar}$), 7.18–7.12 (m, 2H, $2 \times \text{Ar}$), 4.41–4.30 (m, 1H, H-5), 3.76 (s, 3H, COOCH_3), 3.30 (m, 1H *overlapped*, Ph-CHa), 2.68–2.54 (m, 2H *overlapped* Ph-CHb, H-4a), 2.25–2.17 (m, 1H *overlapped*, H-4b) ppm.

minor: 7.74–7.21 (m, 13H *overlapped*, $13 \times \text{Ar}$), 7.12–7.07 (m, 2H, $2 \times \text{Ar}$), 4.63 (dtd, $J_1 = 9.4$ Hz, $J_2 = 7.4$ Hz, $J_3 = 3.4$ Hz, 1H, H-5), 3.66 (s, 3H, COOCH_3), 3.30 (m, 1H *overlapped*, Ph-CHa), 2.73 (dd, $J_{\text{gem}} = 14.4$ Hz, $J_{\text{Ph-CHb,5}} = 7.6$ Hz, 1H, Ph-CHb), 2.68–2.54 (m, 1H *overlapped*, H-4a), 2.25–2.17 (m, 1H *overlapped*, H-4b) ppm.

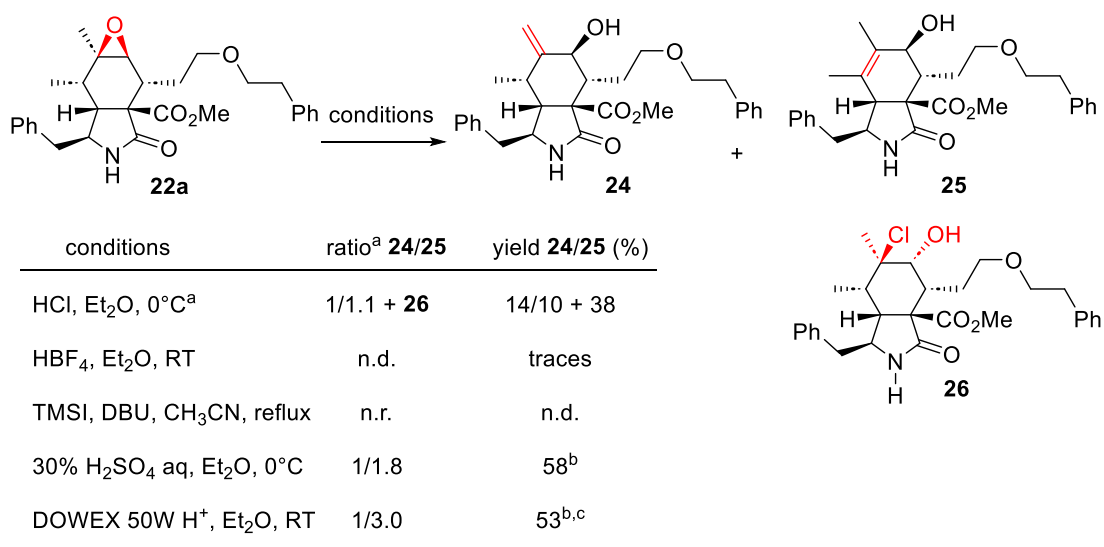
$^{13}\text{C NMR}$ (101 MHz, CDCl_3)

major: δ 170.79 (C=O), 169.73 (C=O), 169.61 (C=O), 137.85 ($2 \times \text{CH-Ar}$), 136.08 (CH-Ar), 134.06 (C *overlapped*), 132.56 (CH-Ar), 130.24 (CH-Ar), 129.56 ($2 \times \text{CH-Ar}$), 129.39 ($2 \times \text{CH-Ar}$), 129.19 ($2 \times \text{CH-Ar}$), 128.62 ($2 \times \text{CH-Ar}$), 127.94 ($2 \times \text{CH-Ar overlapped}$), 126.92 (CH-Ar), 126.29 (C), 55.71 (CH-5), 53.71 (C-3), 53.53 (COOCH_3), 38.46 (Ph- CH_2), 34.21 (CH_2 -4) ppm.

minor: δ 171.18 (C=O), 169.86 (C=O), 169.02 (C=O), 137.89 ($2 \times \text{CH-Ar}$), 135.97 (CH-Ar), 134.06 (C *overlapped*), 132.70 (CH-Ar), 130.10 (CH-Ar), 129.60 ($2 \times \text{CH-Ar}$), 129.42 ($2 \times \text{CH-Ar}$), 129.25 ($2 \times \text{CH-Ar}$), 128.67 ($2 \times \text{CH-Ar}$), 127.94 ($2 \times \text{CH-Ar overlapped}$), 126.96 (CH-Ar), 125.64 (C), 55.92 (CH-5), 54.88 (C-3), 53.59 (COOCH_3), 38.76 (Ph- CH_2), 34.35 (CH_2 -4) ppm.

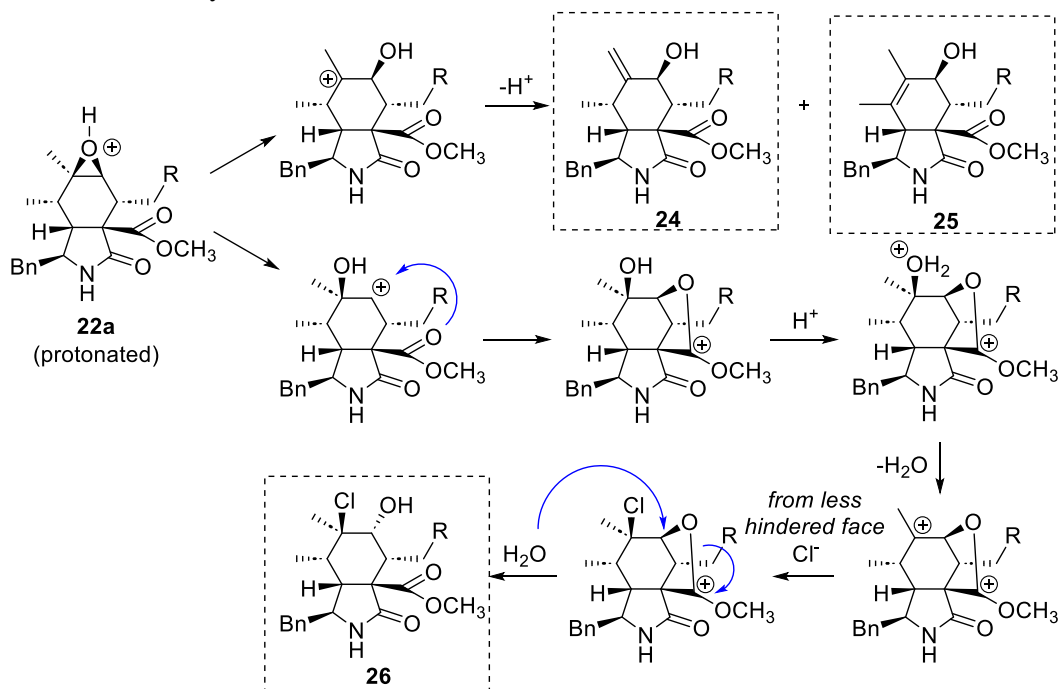
HRMS (ESI) m/z calcd for $\text{C}_{26}\text{H}_{24}\text{O}_4\text{NSe}^+$ $[\text{M}+\text{H}]^+$ 494.0865; found 494.0861.

Scheme S2: Screening of various conditions for epoxide rearrangement.

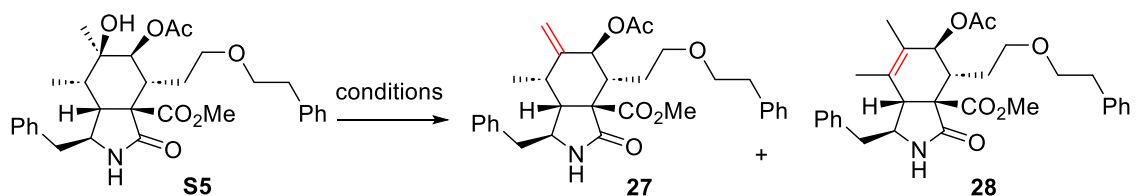


^a Based on ¹H NMR spectra of the crude material. ^b Obtained as a mixture of **24** and **25**. ^c HPLC separation of the mixture furnished compound **25** in 13% yield. n.d. - not determined, n.r. - no reaction

Scheme S3: Proposed mechanism of formation of compounds **24-26** in reaction of **22a** with HCl in diethyl-ether



Scheme S4: Screening of various conditions for elimination reaction.



conditions	ratio ^a 27/28	yield of 27/28 ^b (%)
SOCl ₂ , DMAP, pyridine, 0°C	1.6/1	58
SOCl ₂ , DMAP, pyridine, -40°C	2.0/1	49
SOCl ₂ , DMAP, pyridine, +40°C	1.3/1	50
tripphosgene, DMAP, DCM, RT	2.3/1	15
POCl ₃ , pyridine, DCM, 0-35°C	n.d.	0 ^c

^a Ratio based on ¹H NMR spectra of a crude product. ^b Obtained as a mixture of **27** and **28**. ^c Different product obtained. n.d. - not determined.

Table S1: Spheroid invasion assay with BLM cell line at 10 μ M concentration, 24 h.

compound	DMSO	1	2	17a	17b	17c	17d	17e	17f	17g
rel. spheroid area	1.02	0.26	0.23	1.21	1.14	0.93	1.24	0.96	0.92	1.17
SD	0.28	0.04	0.06	0.11	0.19	0.18	0.26	0.14	0.12	0.28
compound	17f	17g	17h	17i	17j	20	22a	22b	22h	23a
rel. spheroid area	0.92	1.17	1.05	1.14	1.12	0.56	1.11	1.24	0.98	1.01
SD	0.12	0.28	0.21	0.27	0.30	0.25	0.17	0.30	0.39	0.24
compound	23b	23h	24	25	26	27	28	29	30	
rel. spheroid area	1.10	0.93	0.46	1.15	0.88	0.89	0.72	1.05	0.50	
SD	0.21	0.38	0.07	0.30	0.11	0.27	0.15	0.22	0.27	

Figure S1: Representative images of BLM cell spheroid invasion assay with or without inhibitor. Images were taken at 0 h and 24 h. using a Leica TCS SP2 microscope (5x/0.15 dry objective). DMSO (first column) was used as a control. Scale bar 500 μ m.

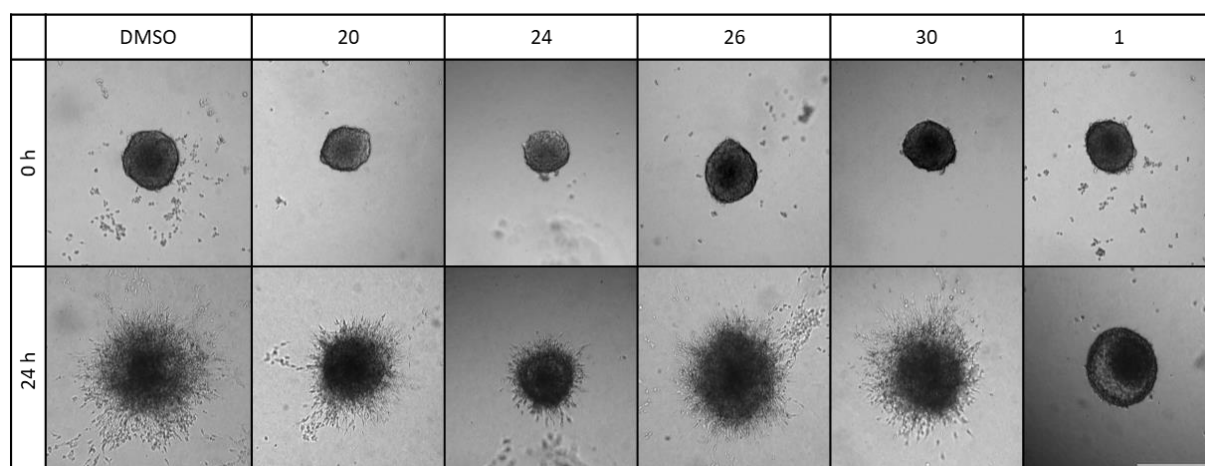
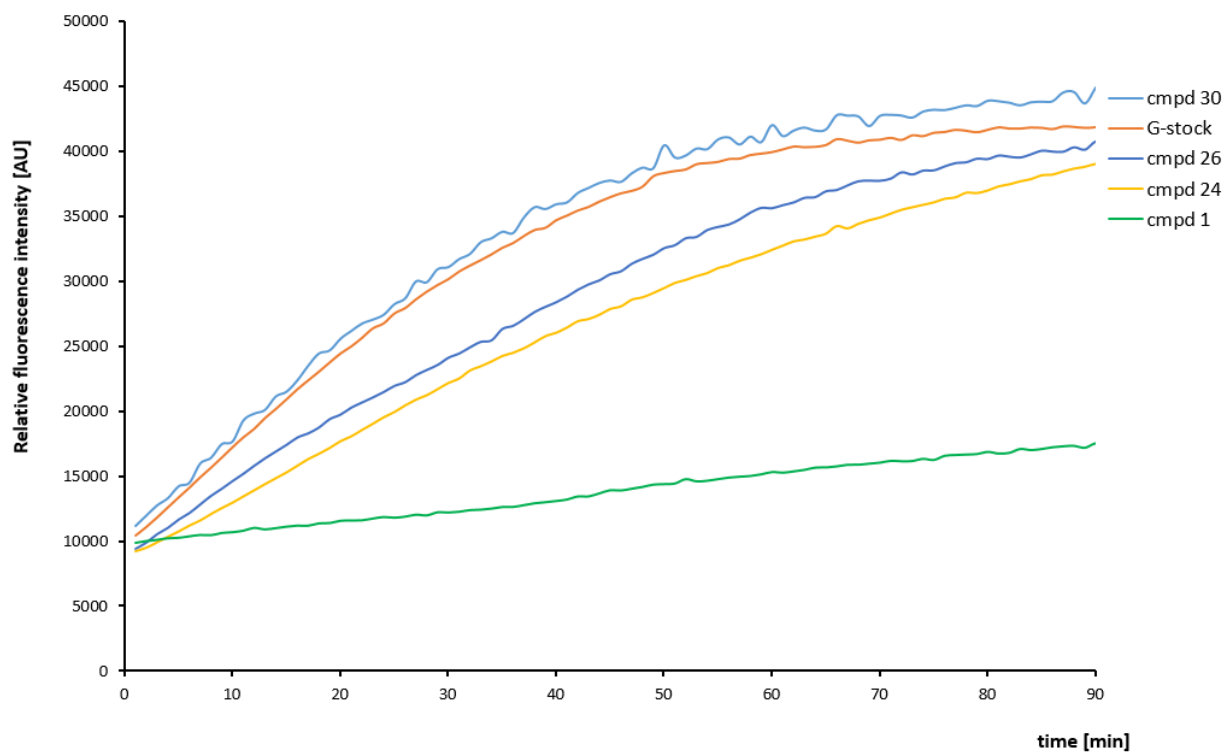


Figure S2: Actin polymerization assay at 10 μM concentration. Representative graph of one measurement.



HPLC purity of final cytochalasin analogues

Separation conditions:

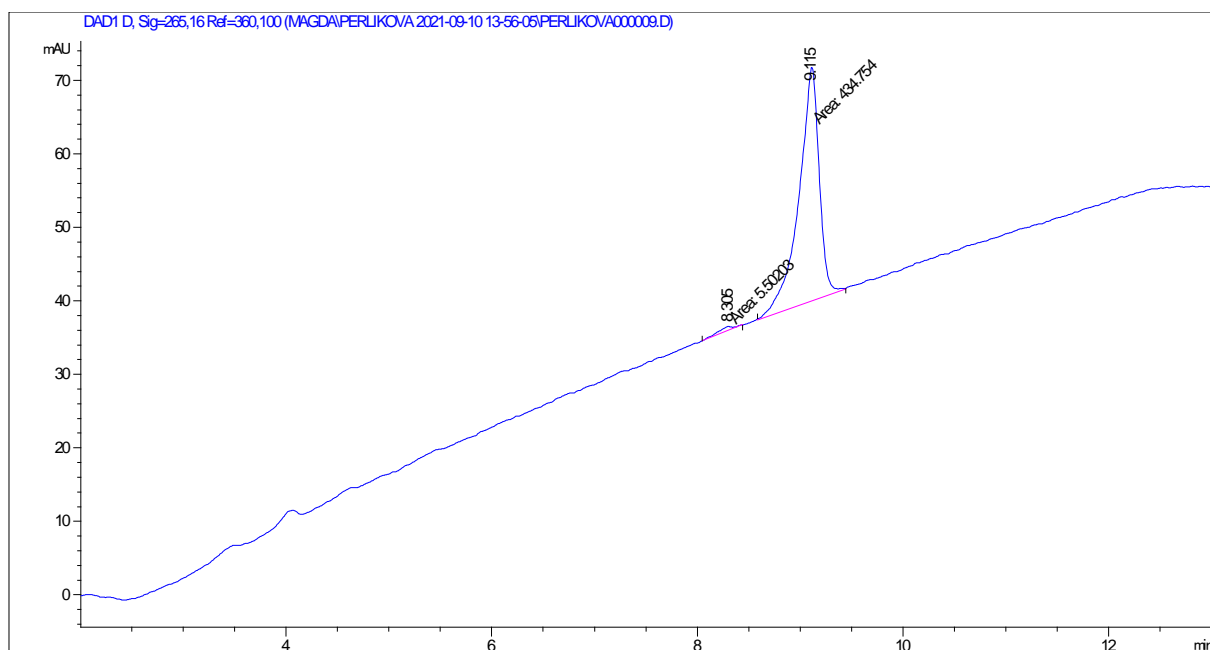
A: column 4x150 mm, 5 μ m silica gel, 60 \AA , V=1 mL/min, EtOAc/hexane – 0. min 20/80, 10. min 80/20, 12.5. min 80/20.

B: column 4x100 mm, 5 μ m C18-RP, 130 \AA , V=0.75 mL/min, MeOH/H₂O – 0. min 60/40, 7. min 95/5, 11. min 95/5, 14. min 60/40, 20. min 60/40.

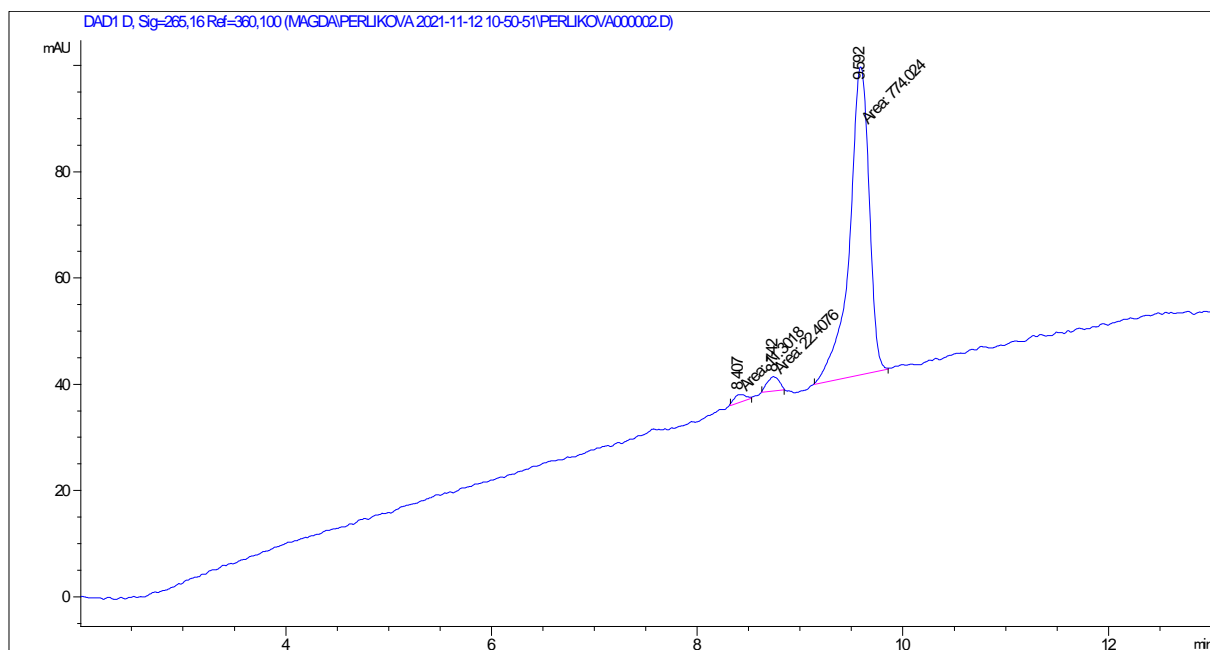
C: column 2.1x100 mm, 1.7 μ m C18-BEH, V=0.5 mL/min, ACN/H₂O – 0. min 0/100, 5. min 100/0, 6. min 100/0.

Sample	Purity (%)	Separation conditions	Retention time (min)
17a	98.8	A	9.12
17b	95.5	A	9.60
17c	95.1	A	8.67
17d	98.3	A	9.79
17e	98.9	A	9.65
17f	98.8	A	8.86
17g	98.0	A	10.04
17h	98.4	A	10.06
17i	98.0	A	9.72
17j	99.0	A	11.38
20	97.1	A	7.60
22a	98.4	A	10.62
22b	96.6	A	10.31
22h	99.9	A	10.57
23a	95.8	C	4.44
23b	99.3	A	10.91
23h	98.9	A	10.98
24	99.3	A	9.98
25	98.0	B	9.14
26	97.6	A	7.21
27	99.9	A	7.07
28	97.9	A	7.77
29	95.8	B	8.29
30	99.9	A	13.54

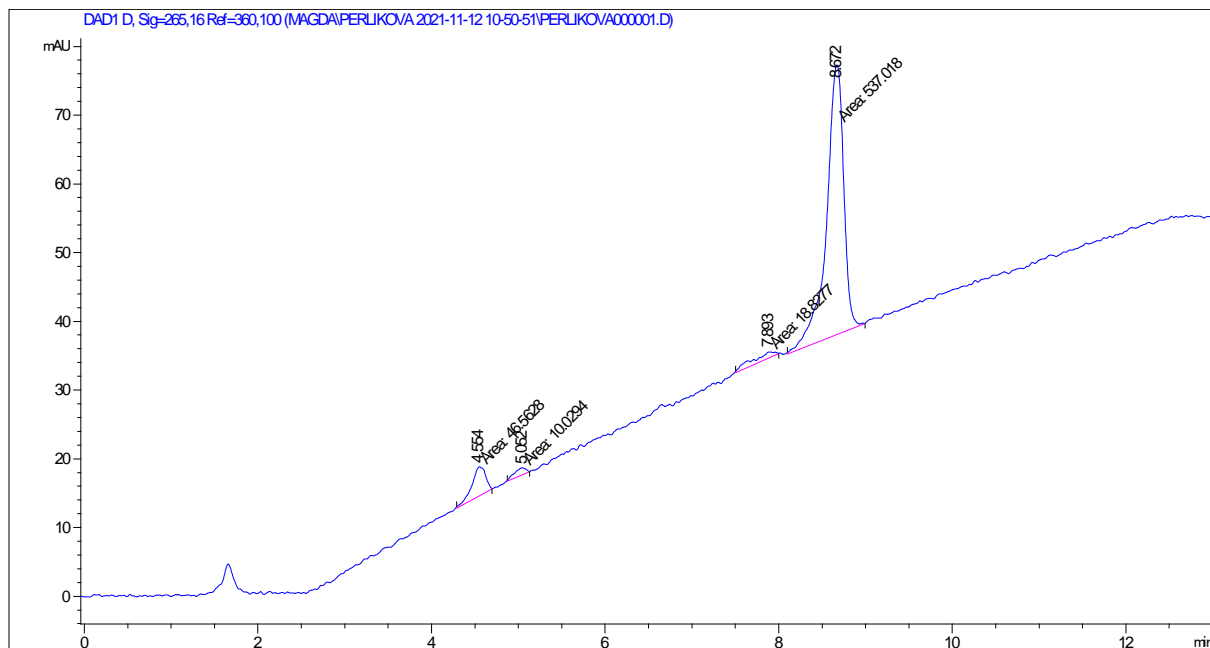
17a – purity 98.8 %, $T_R = 9.12$ min



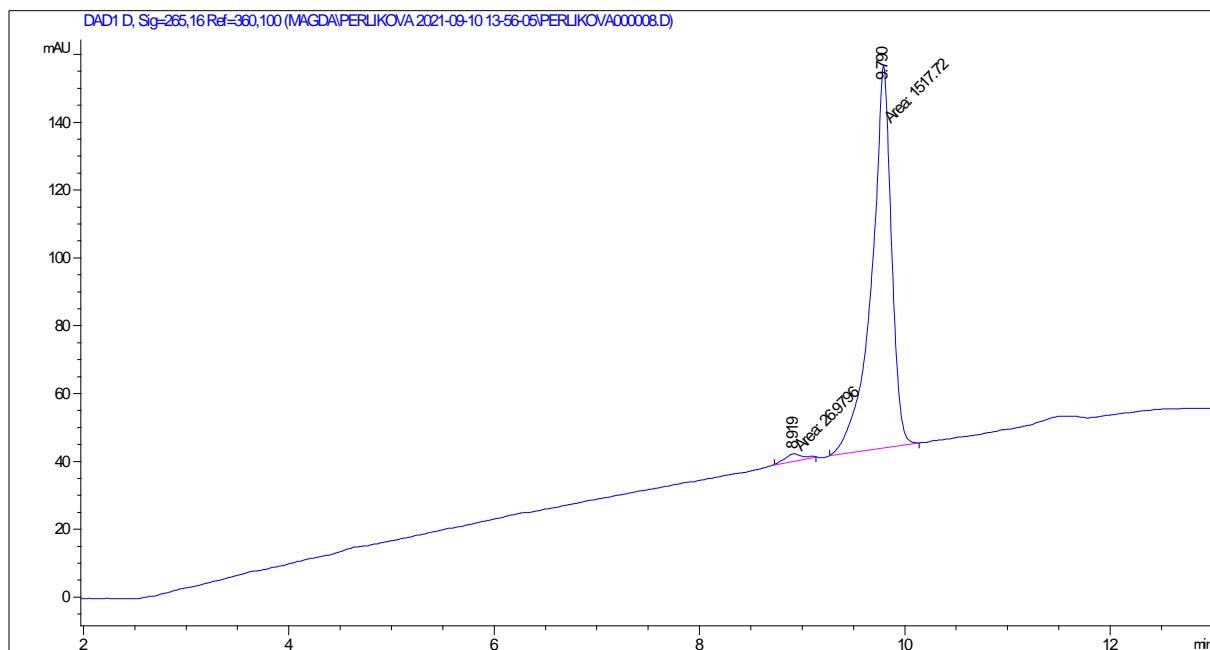
17b – purity 95.5 %, $T_R = 9.60$ min



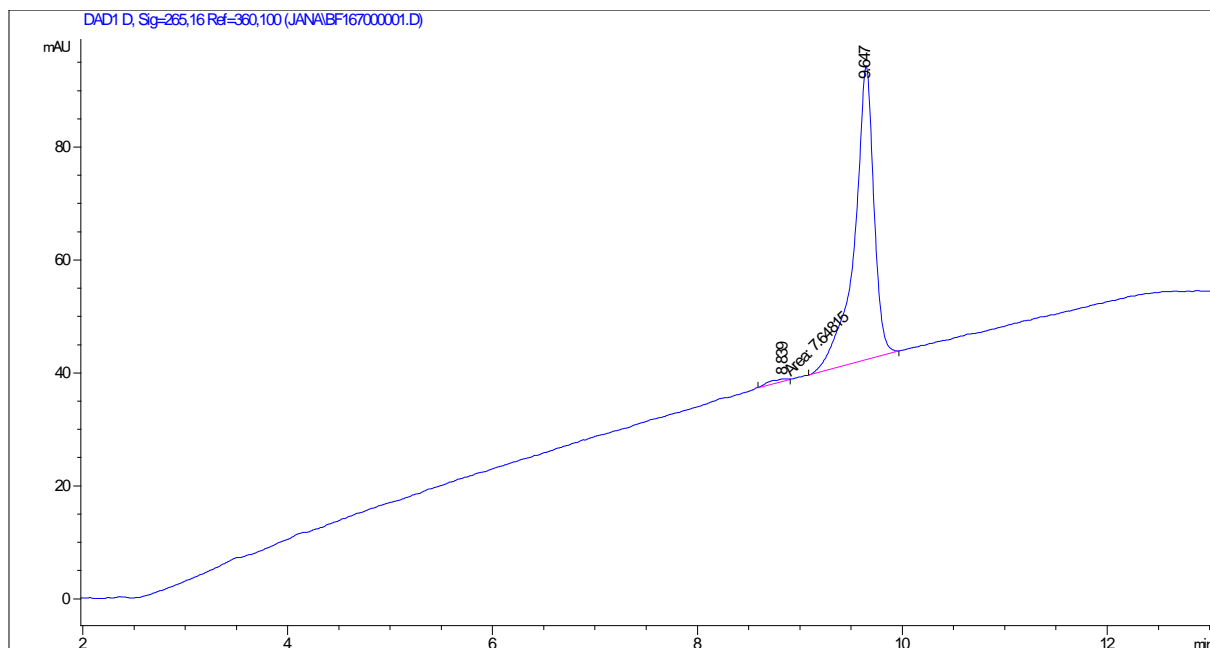
17c – purity 95.1 %, $T_R = 8.67$ min



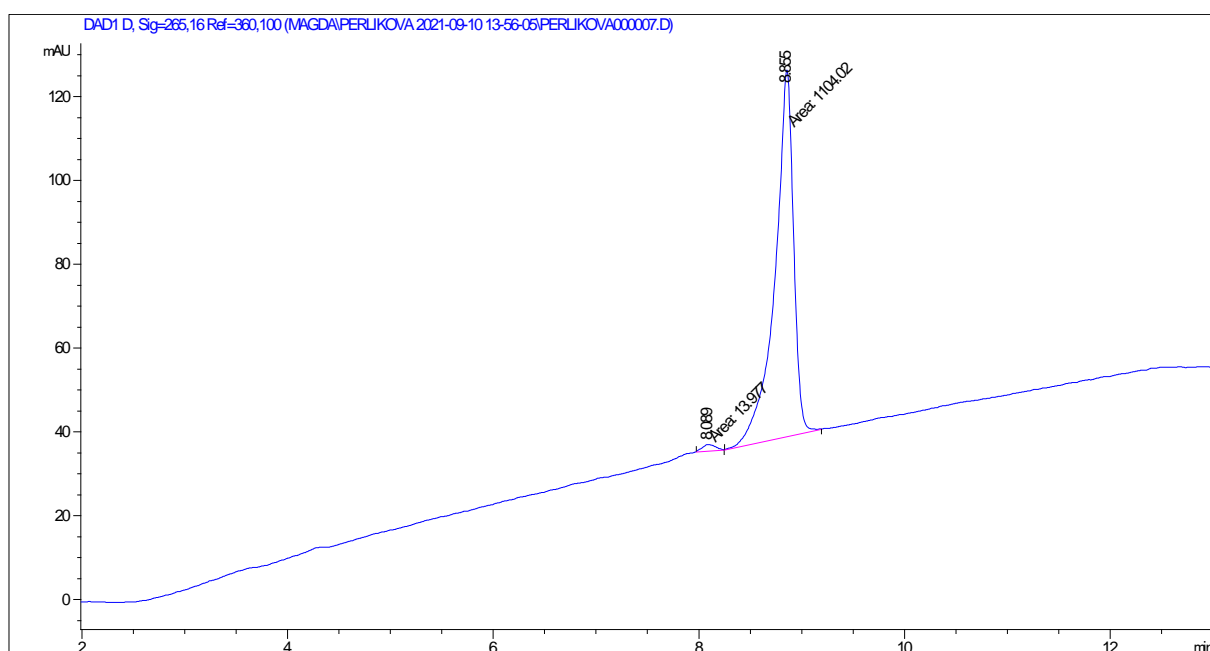
17d – purity 98.3 %, $T_R = 9.79$ min



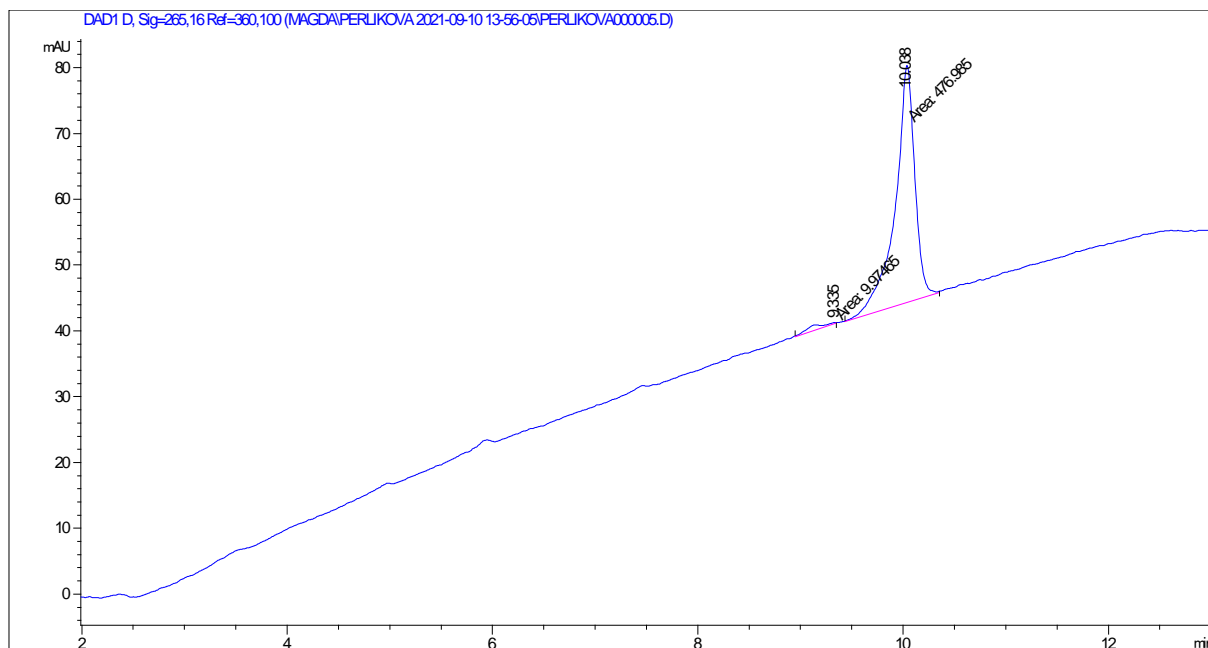
17e – purity 98.9 %, $T_R = 9.65$ min



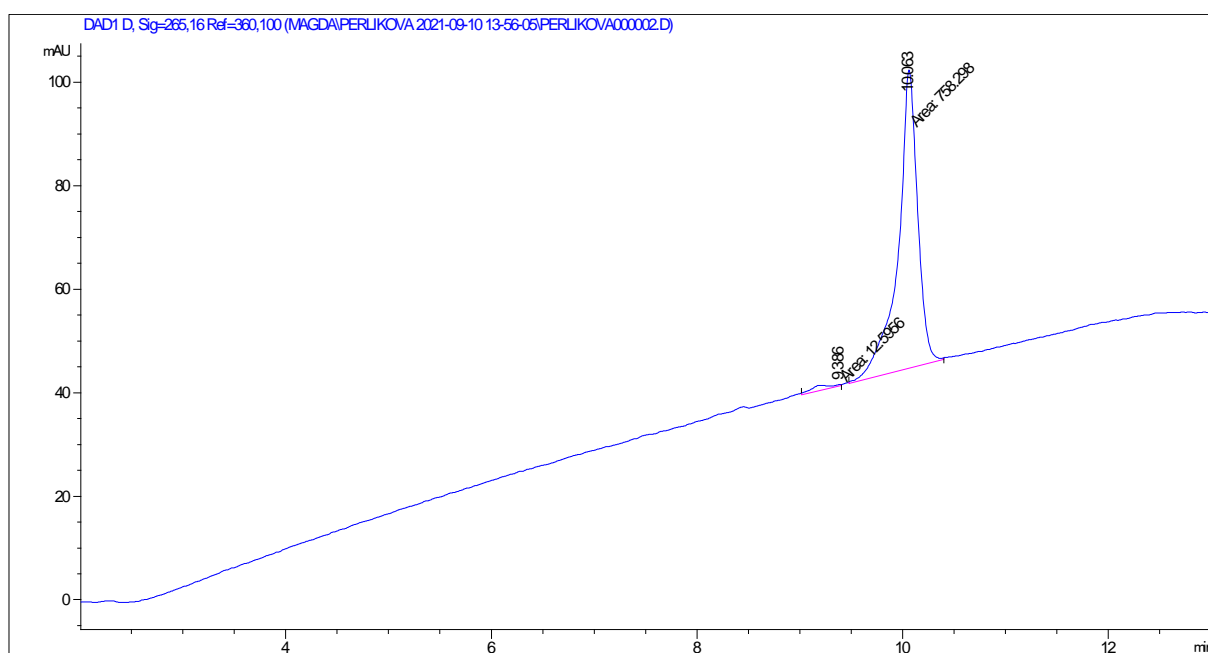
17f – purity 98.8 %, $T_R = 8.86$ min



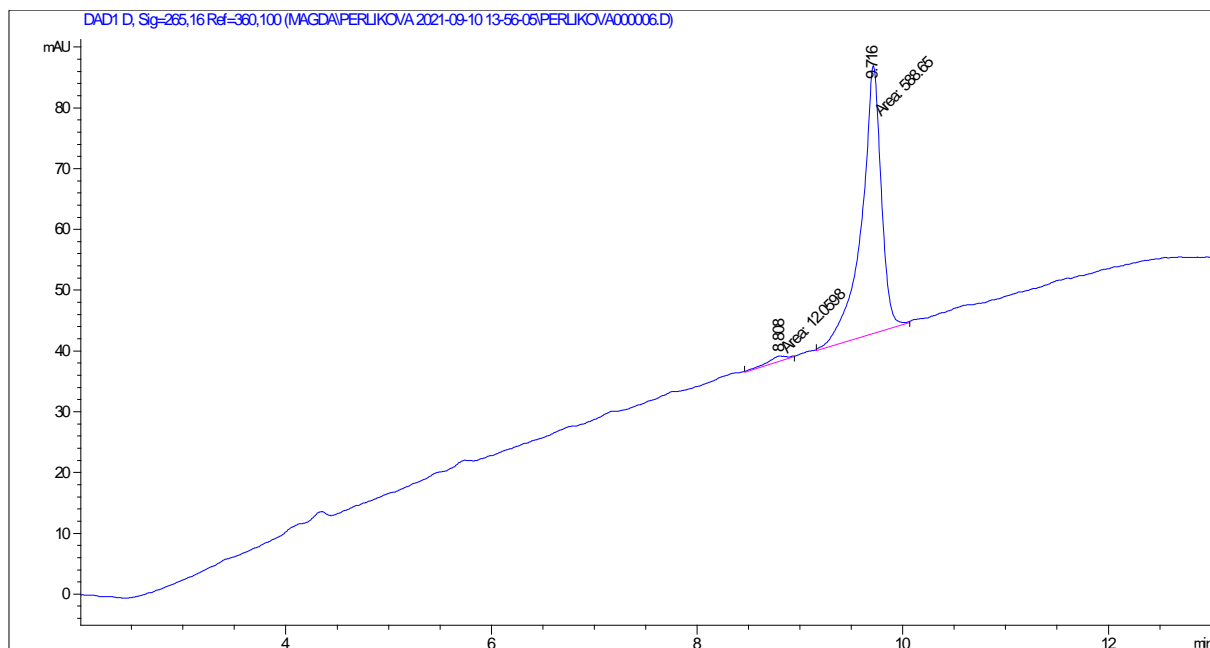
17g – purity 98.0 %, $T_R = 10.04$ min



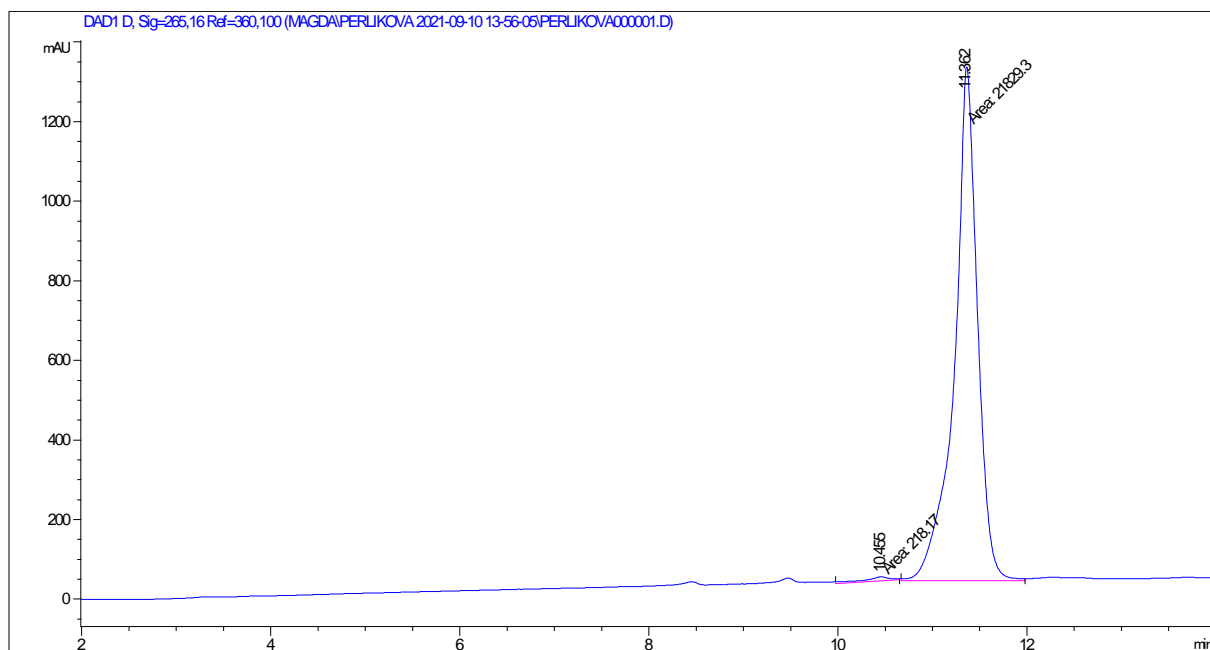
17h – purity 98.4 %, $T_R = 10.06$ min



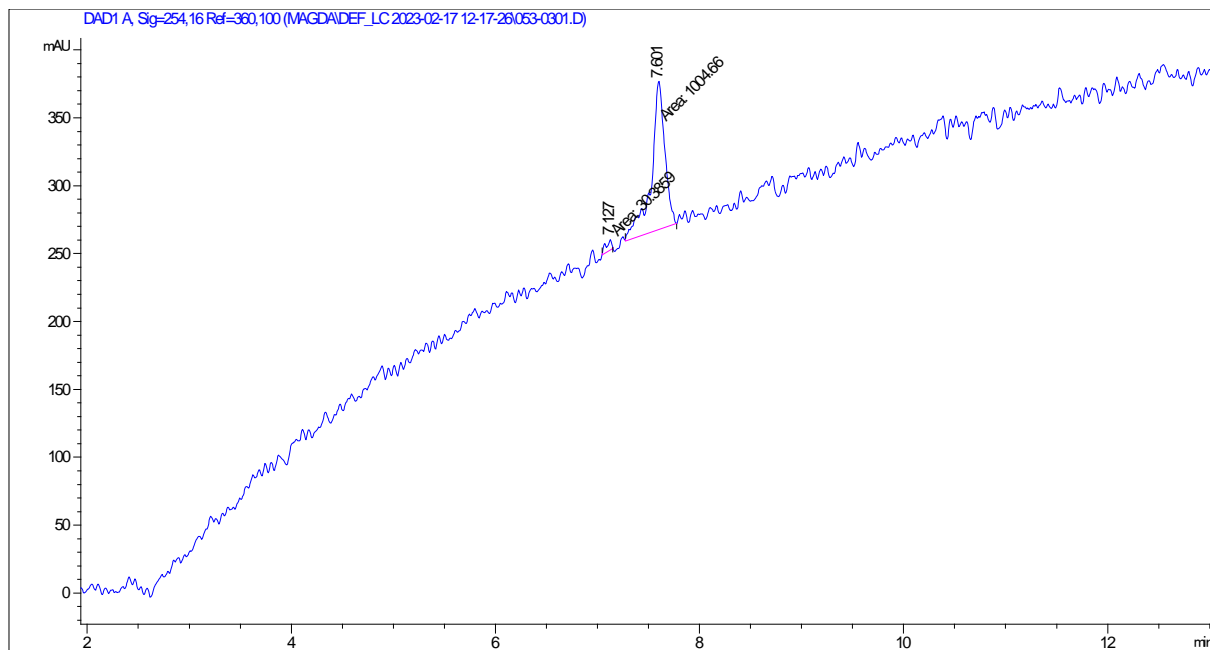
17i – purity 98.0 %, $T_R = 9.72$ min



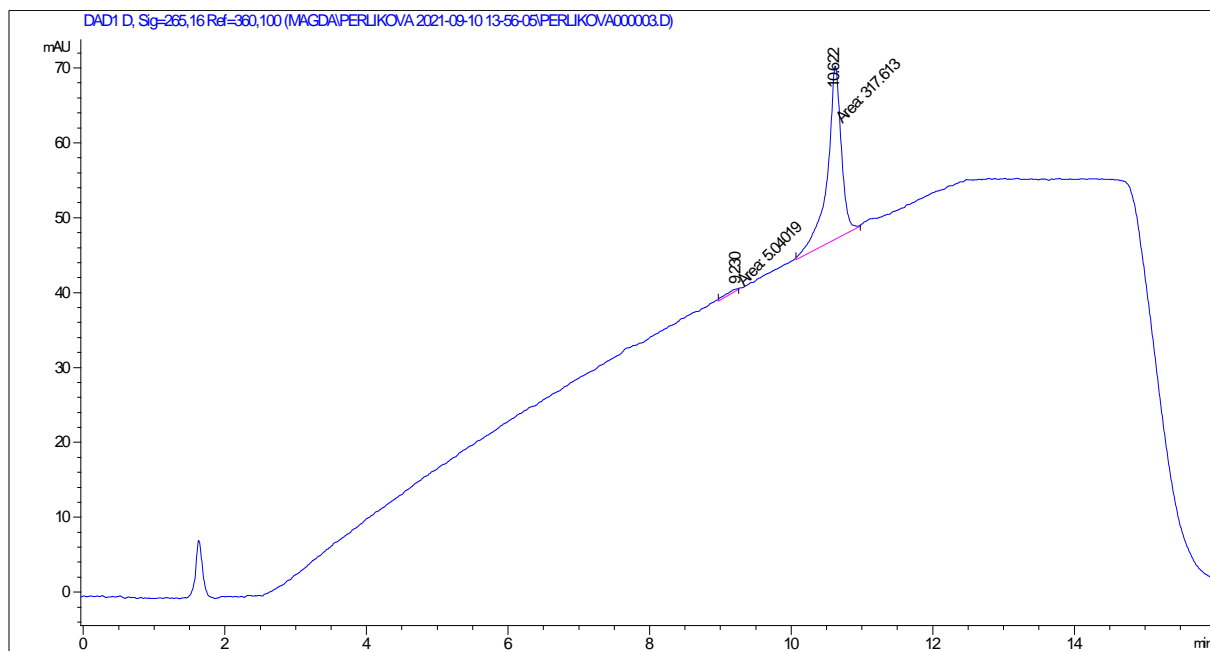
17j – purity 99.0 %, $T_R = 11.38$ min



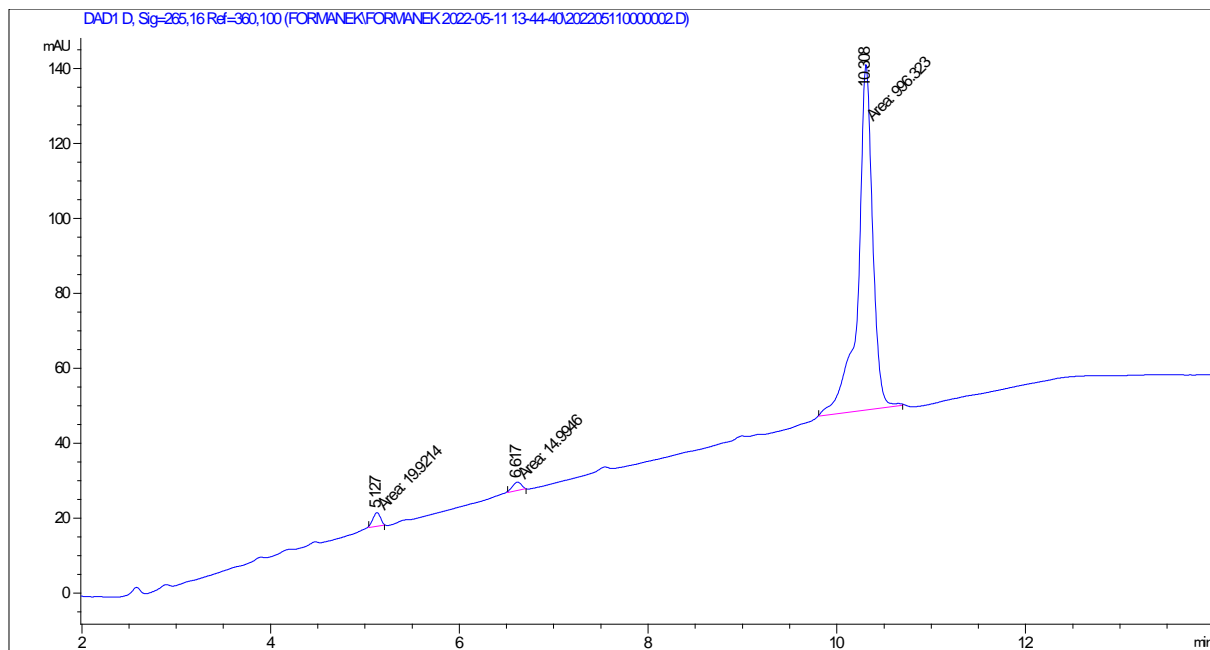
20 – purity 97.1 %, $T_R = 7.60$ min



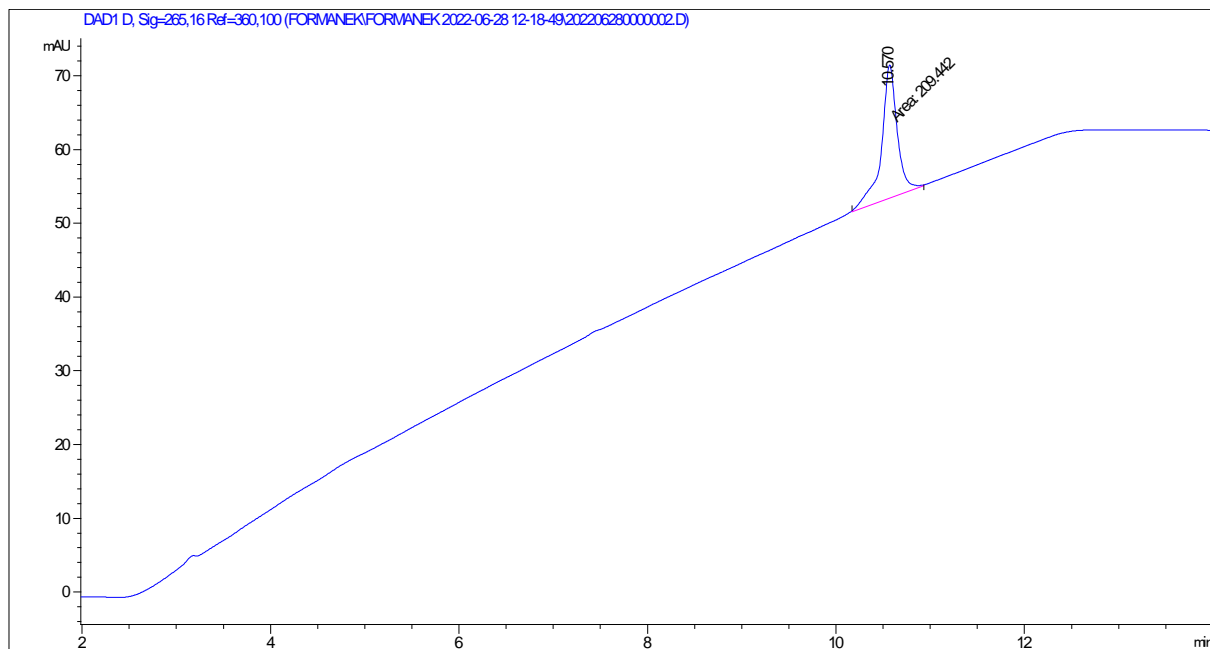
22a – purity 98.4 %, $T_R = 10.62$ min



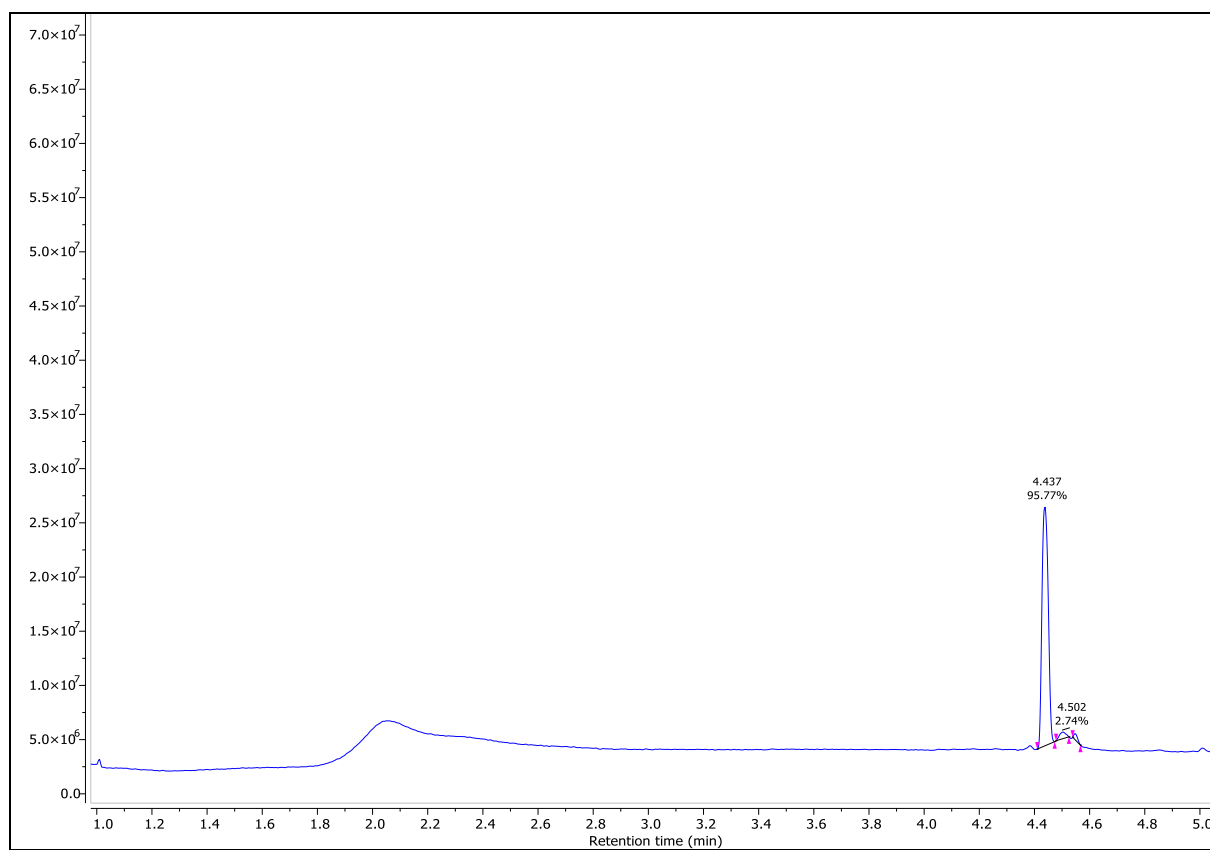
22b – purity 96.6 %, $T_R = 10.31$ min



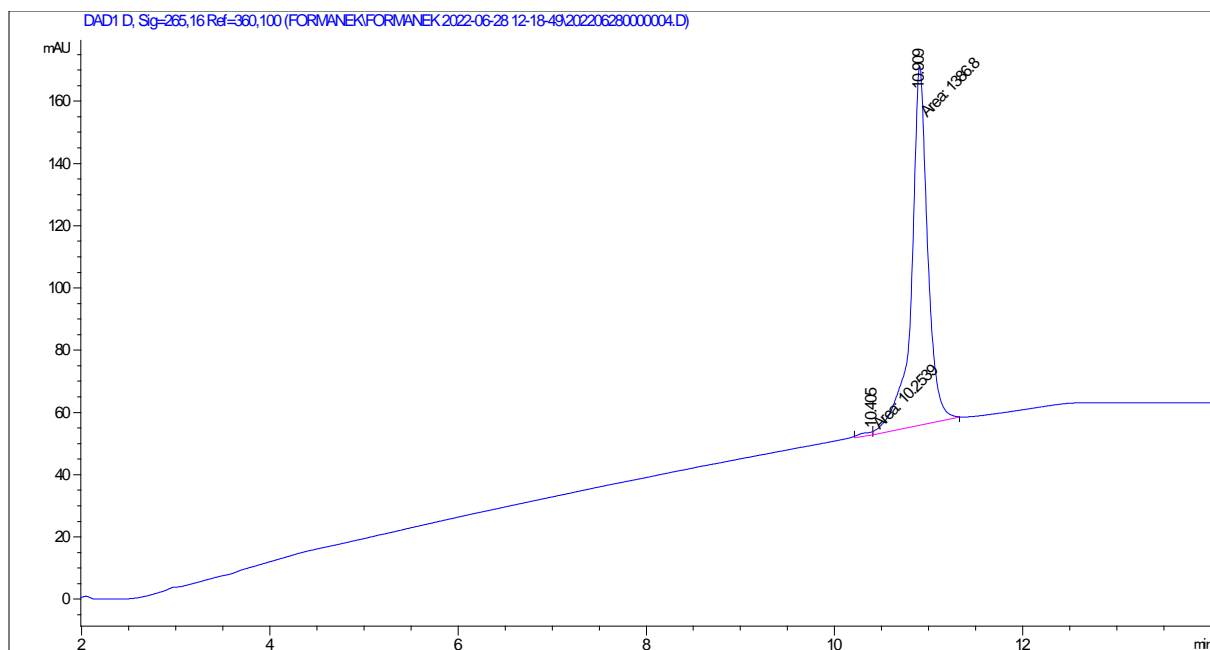
22h – purity 99.9 %, $T_R = 10.57$ min



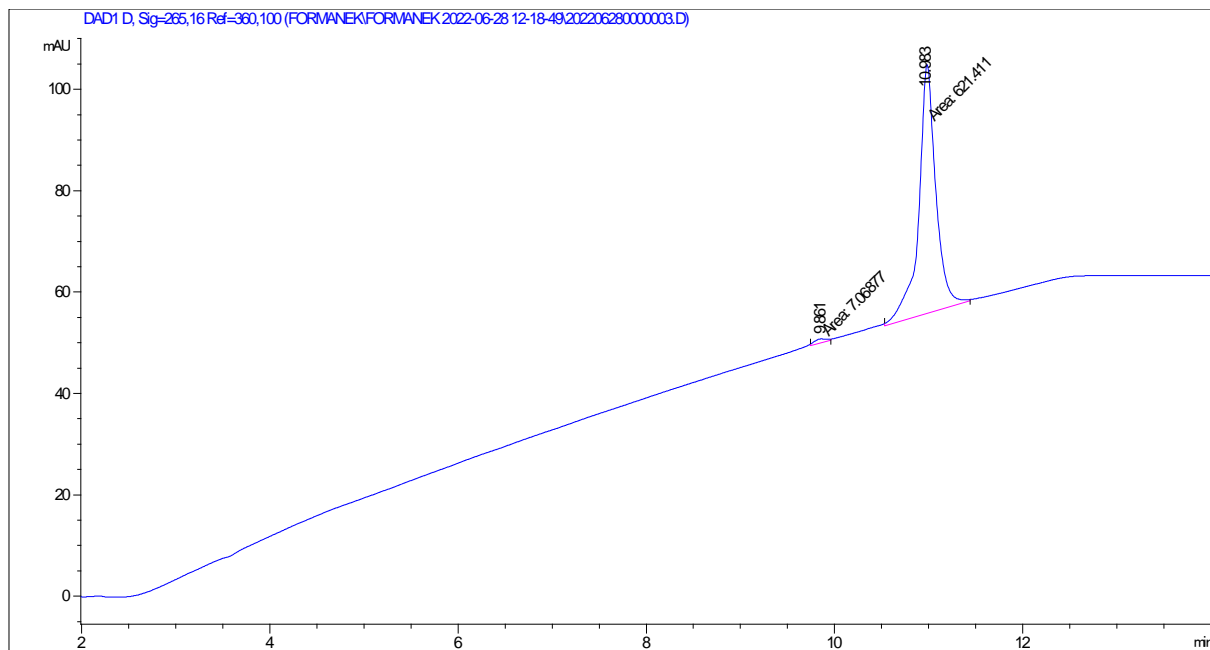
23a – purity 95.8 %, $T_R = 4.44$ min



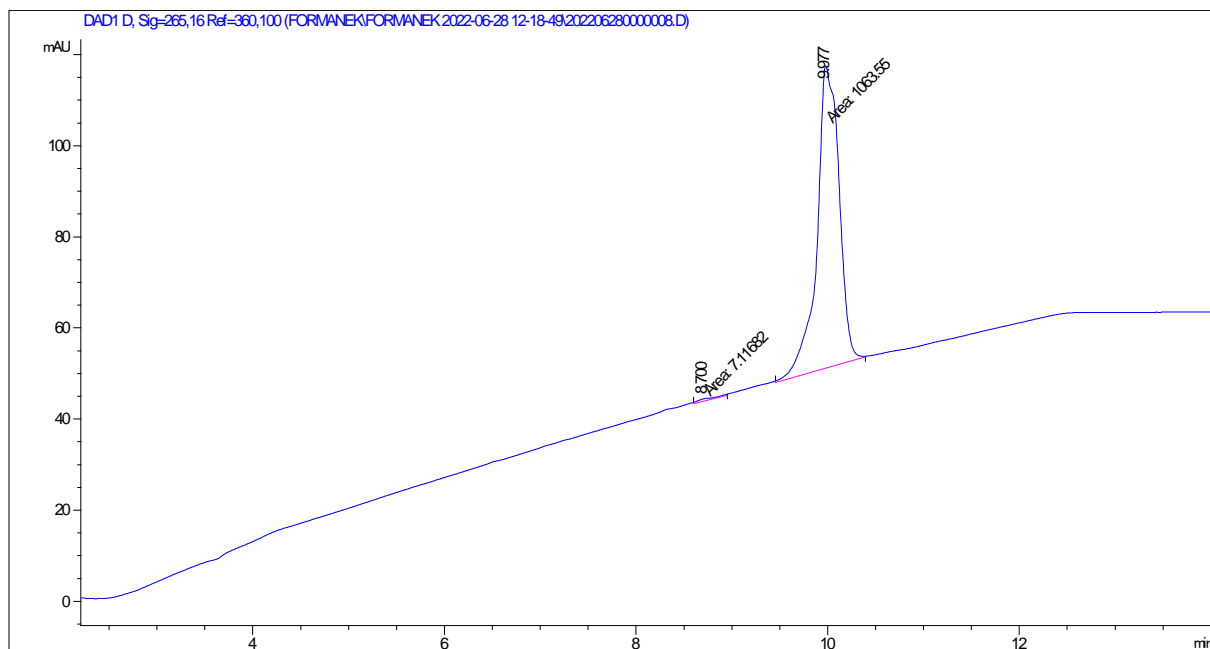
23b – purity 99.3 %, $T_R = 10.91$ min



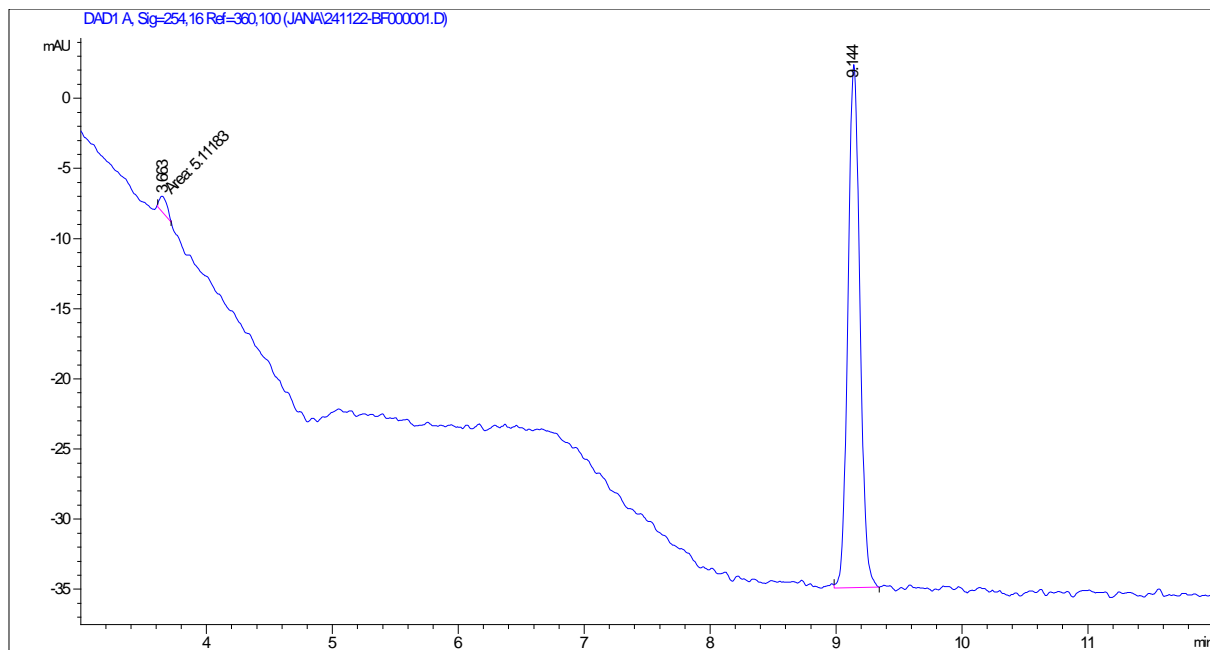
23h – purity 98.9 %, $T_R = 10.98$ min



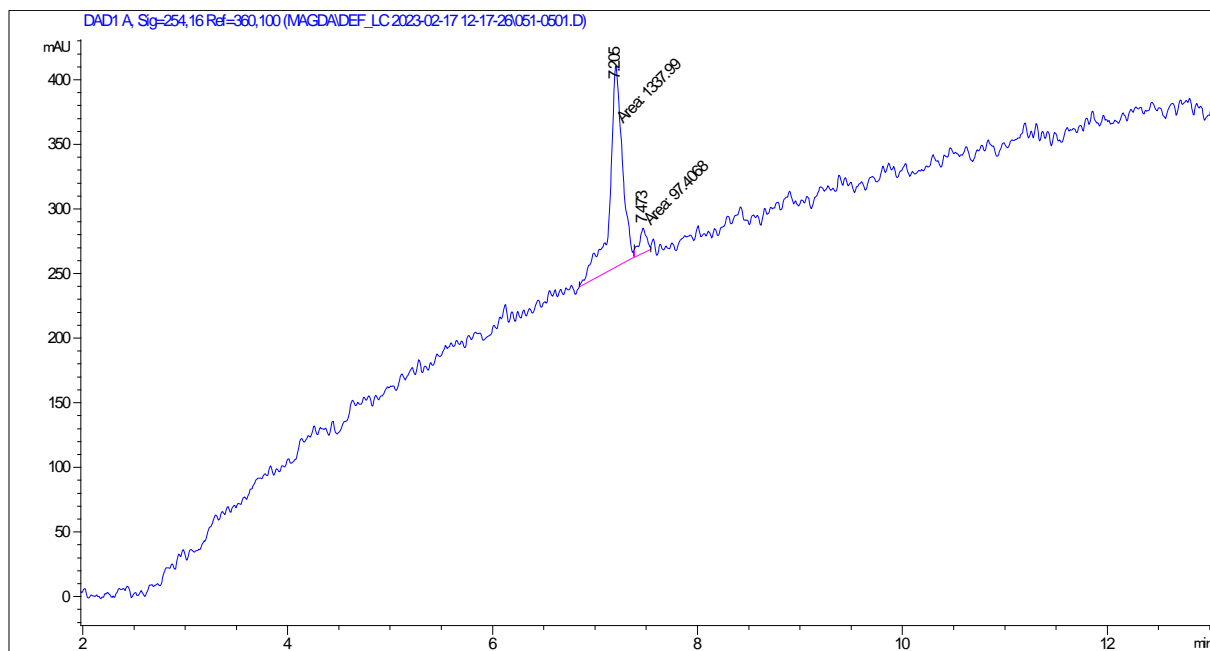
24 – purity 99.3 %, $T_R = 9.98$ min



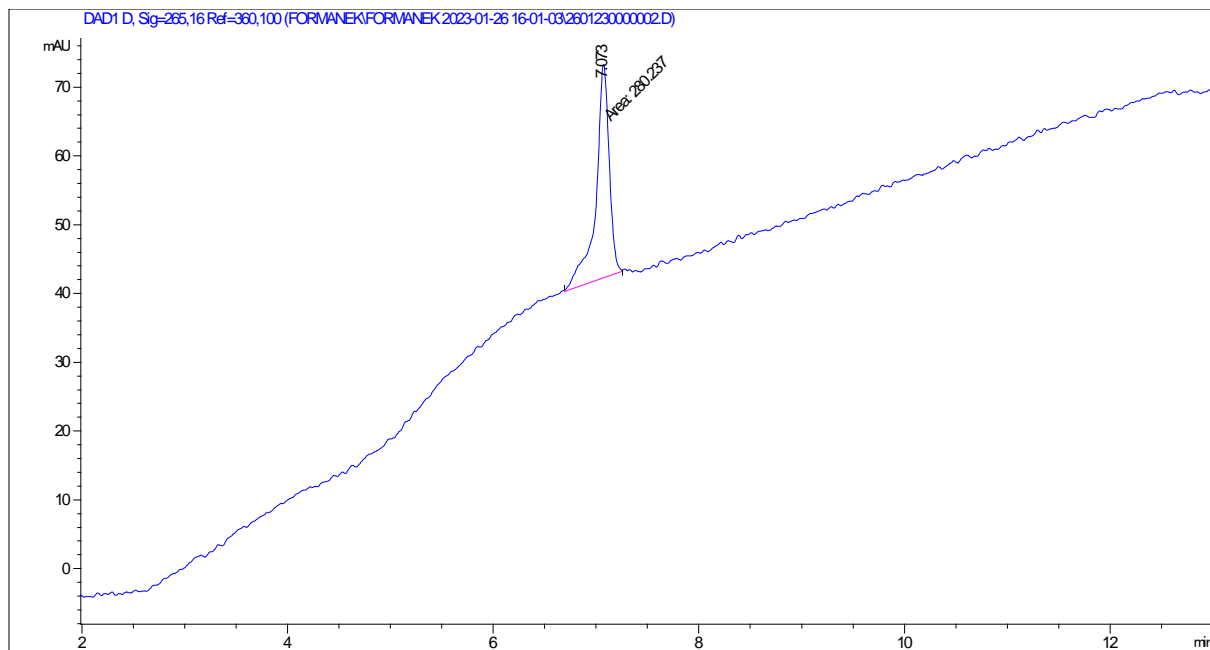
25 – purity 98.0 %, $T_R = 9.14$ min



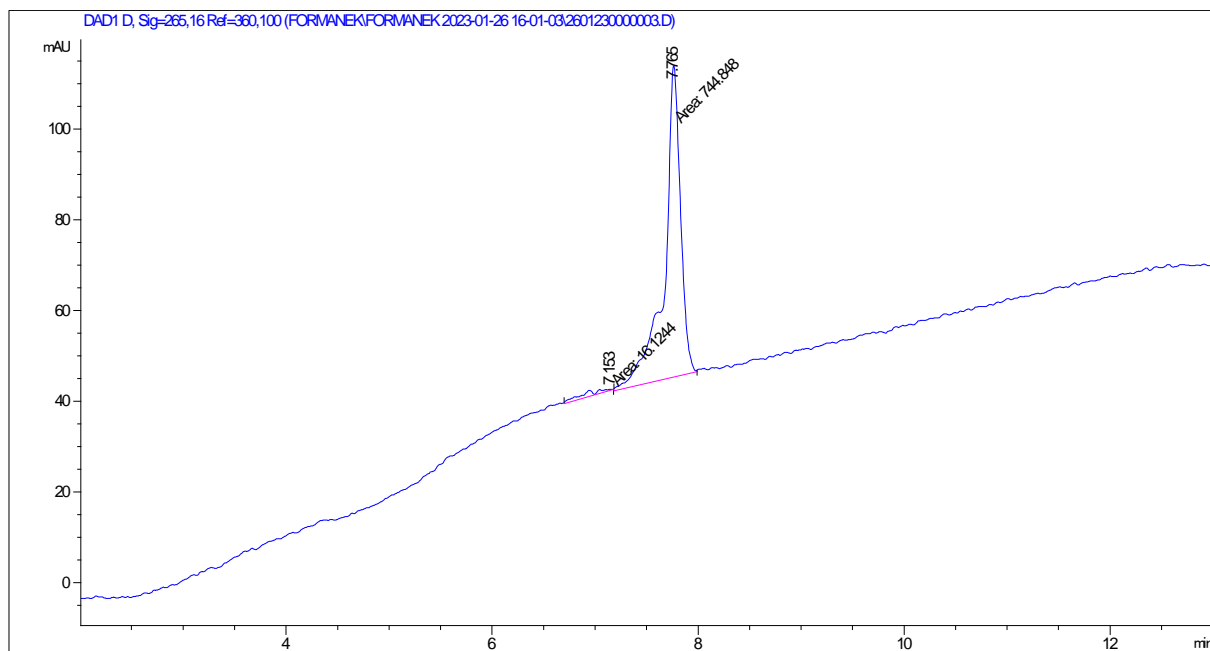
26 – purity 97.6 %, $T_R = 7.21$ min



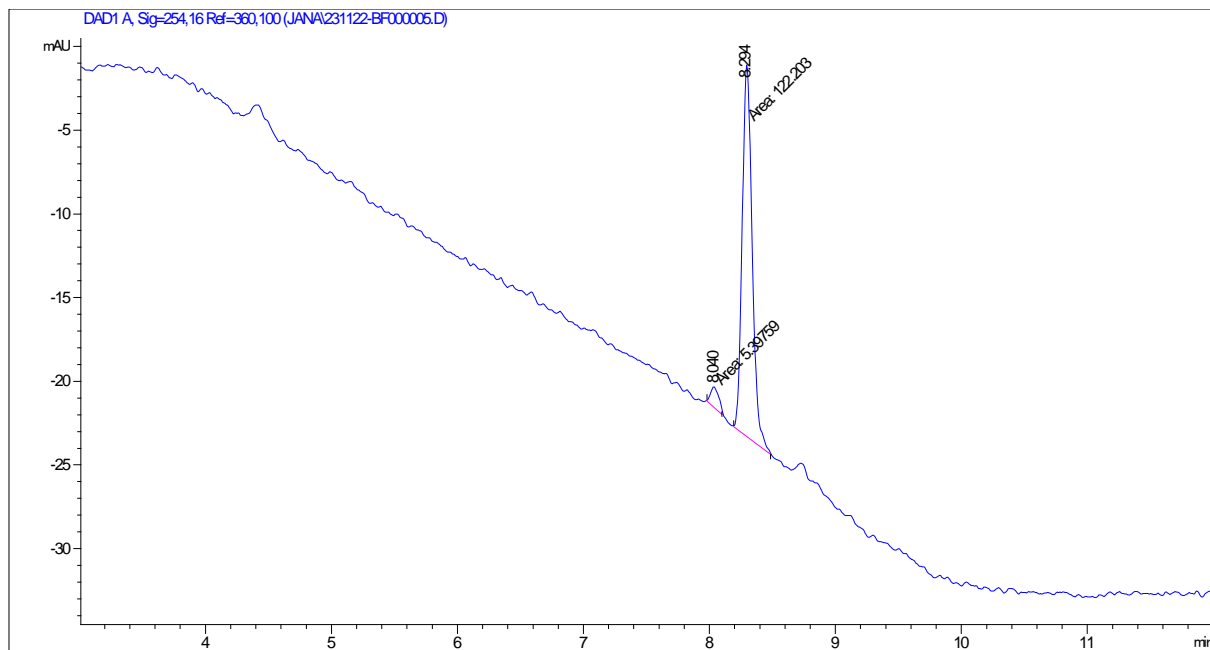
27 – purity 99.9 %, $T_R = 7.07$ min



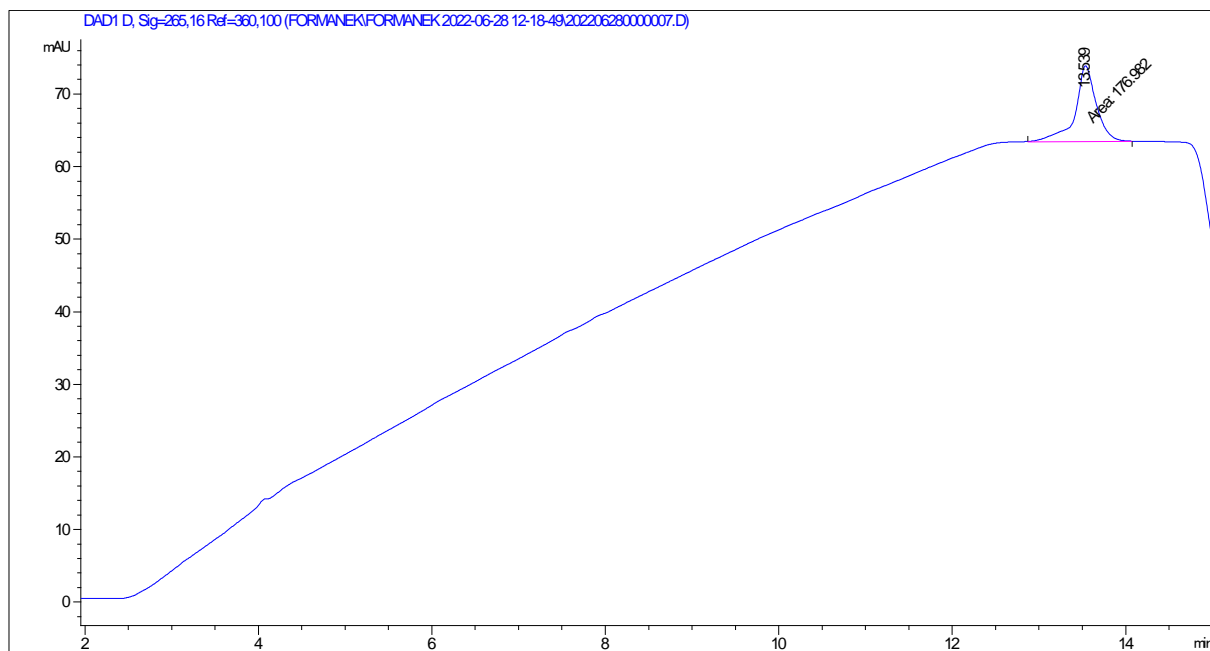
28 – purity 97.9 %, $T_R = 7.77$ min



29 – purity 95.8 %, $T_R = 8.29$ min



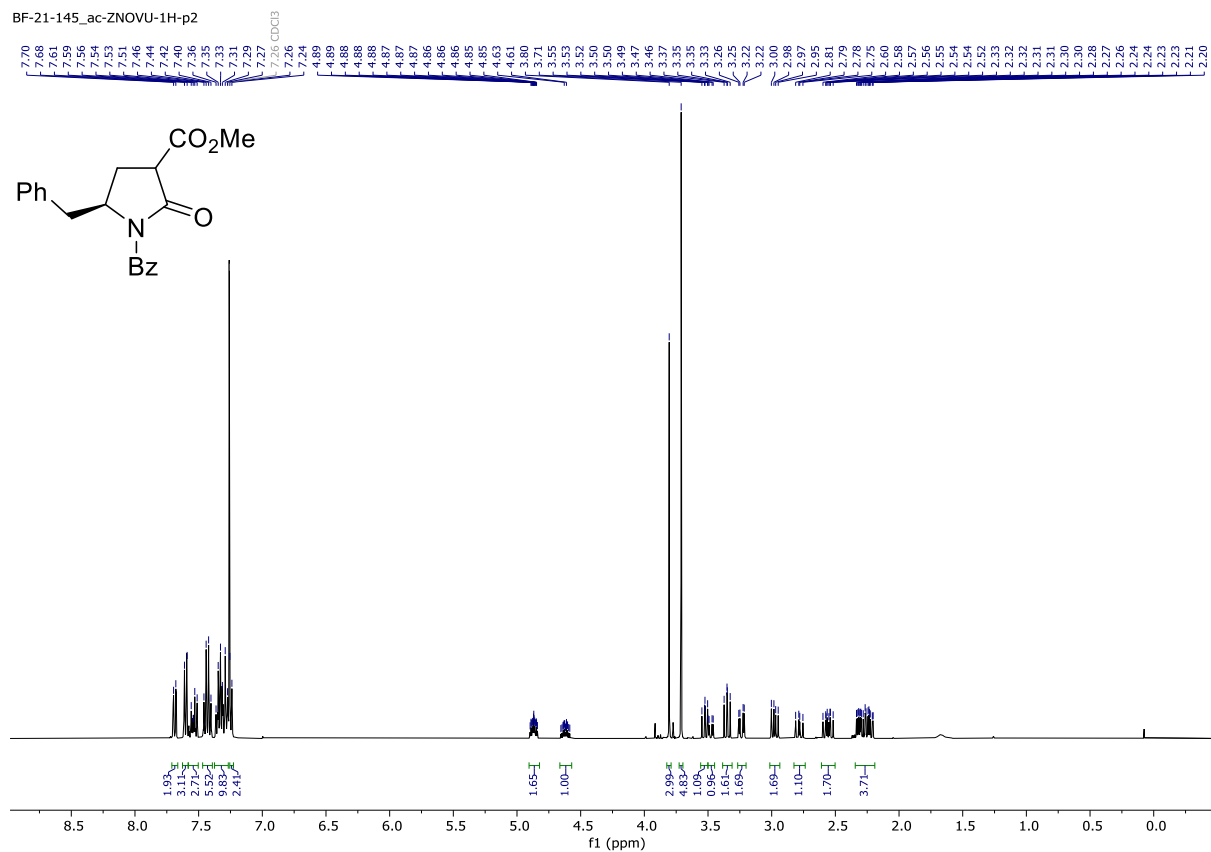
30 – purity 99.9 %, $T_R = 13.54$ min



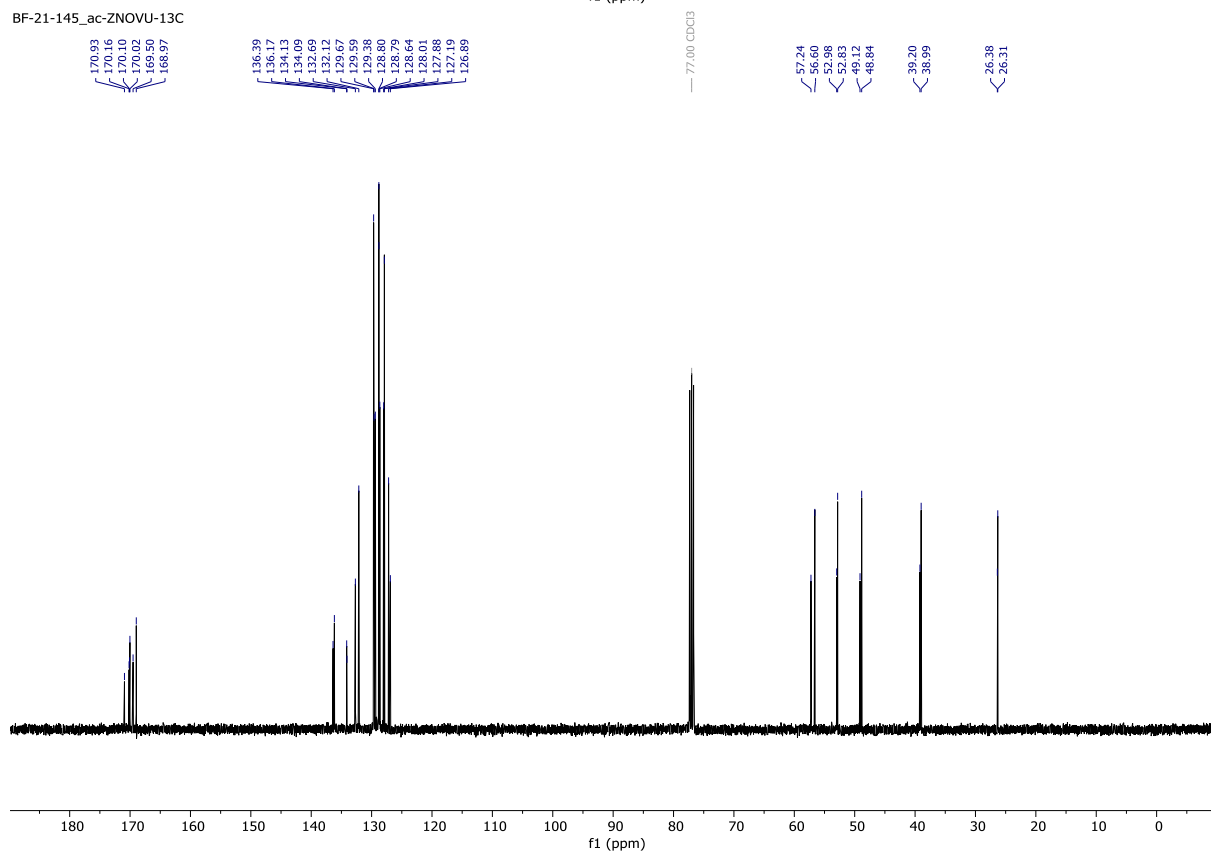
Copies of NMR spectra

S4

BF-21-145_ac-ZNOVU-1H-p2

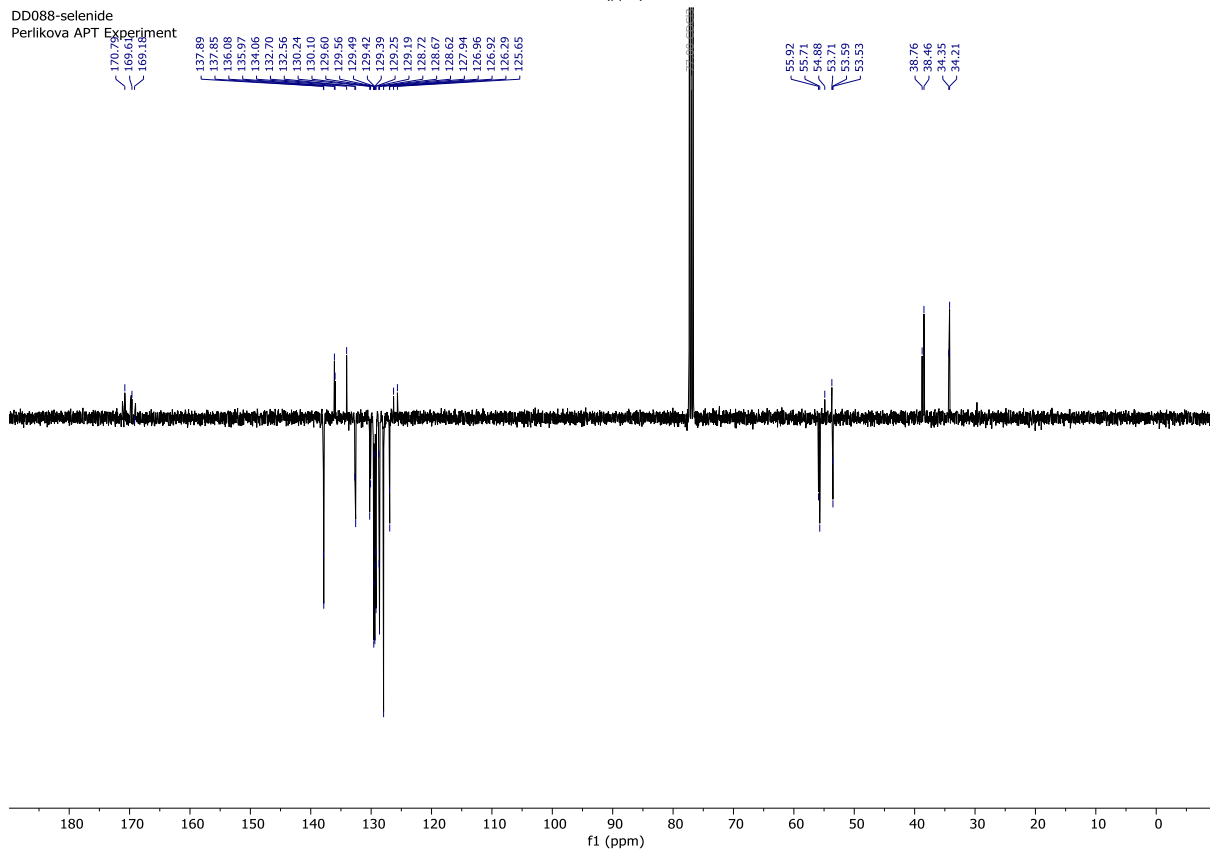
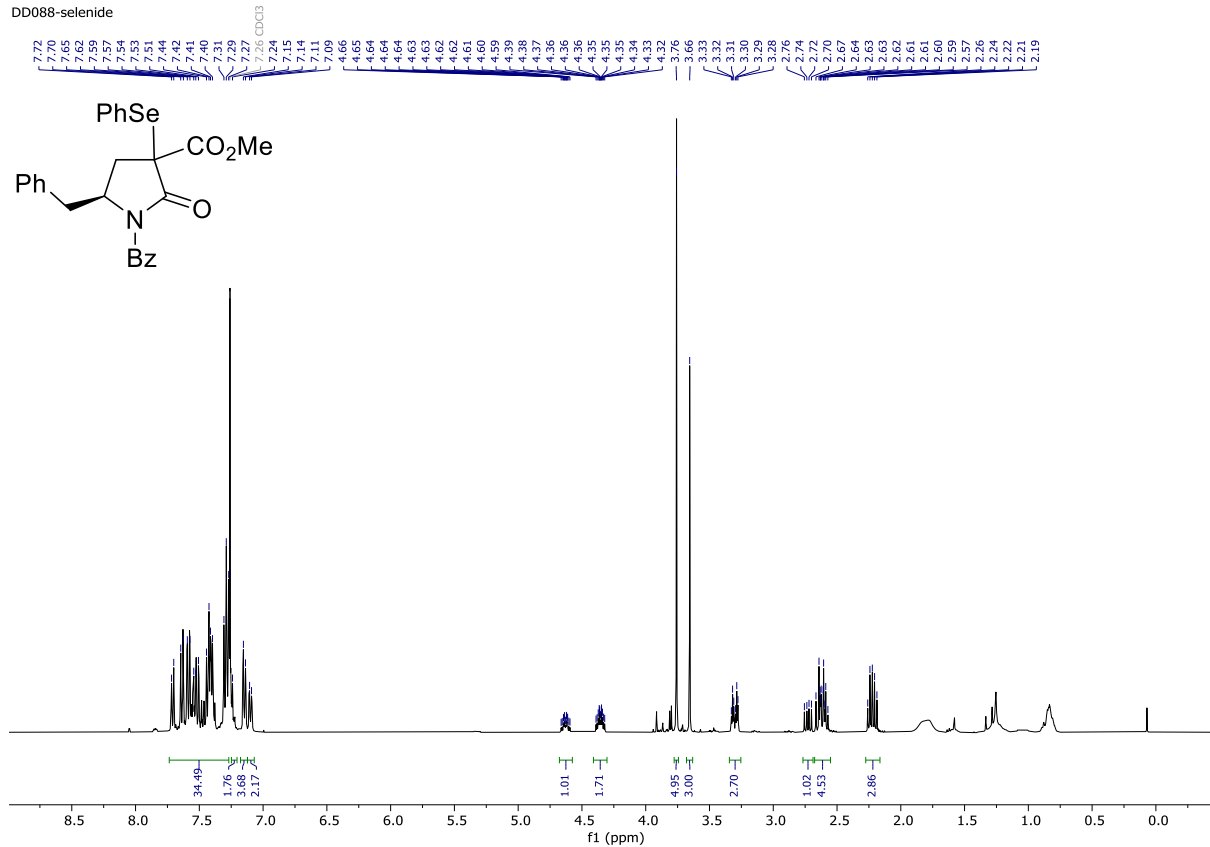


BF-21-145_ac-ZNOVU-13C



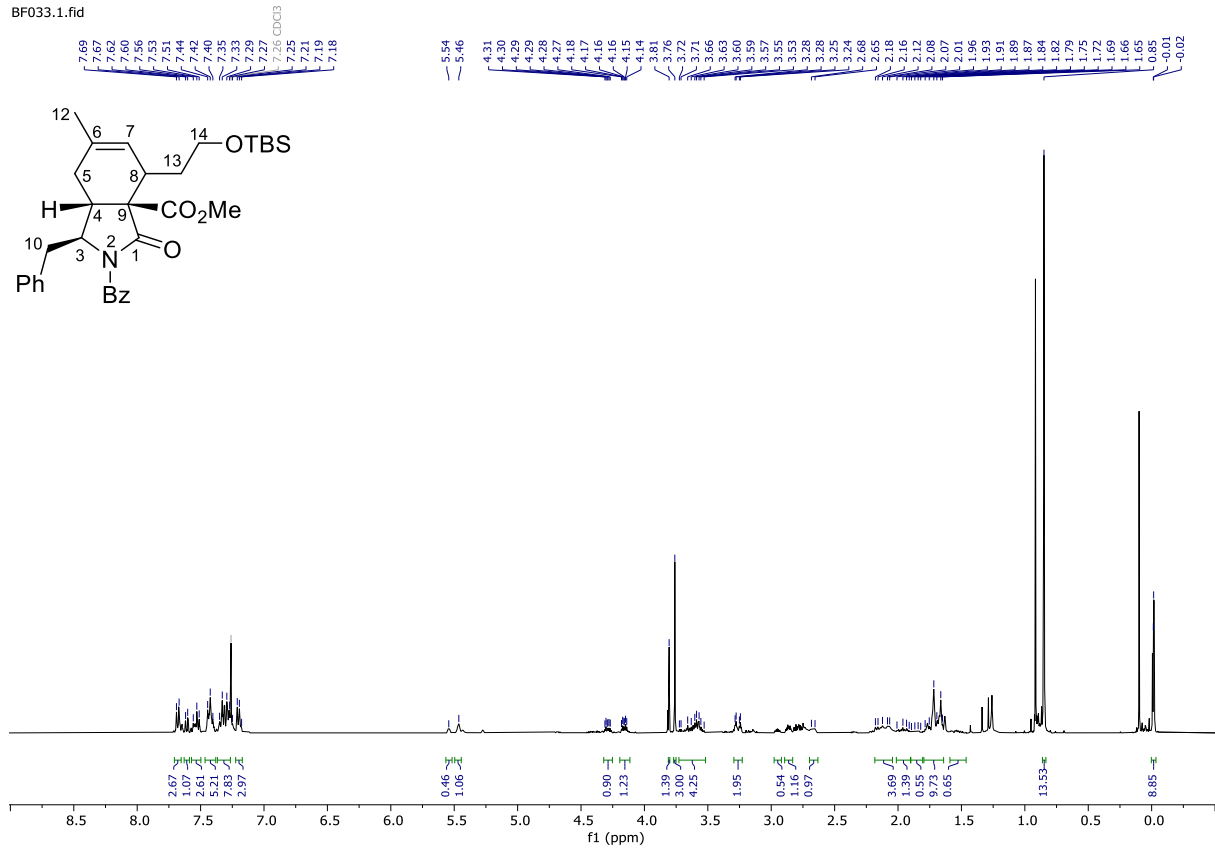
5

DD088-selenide

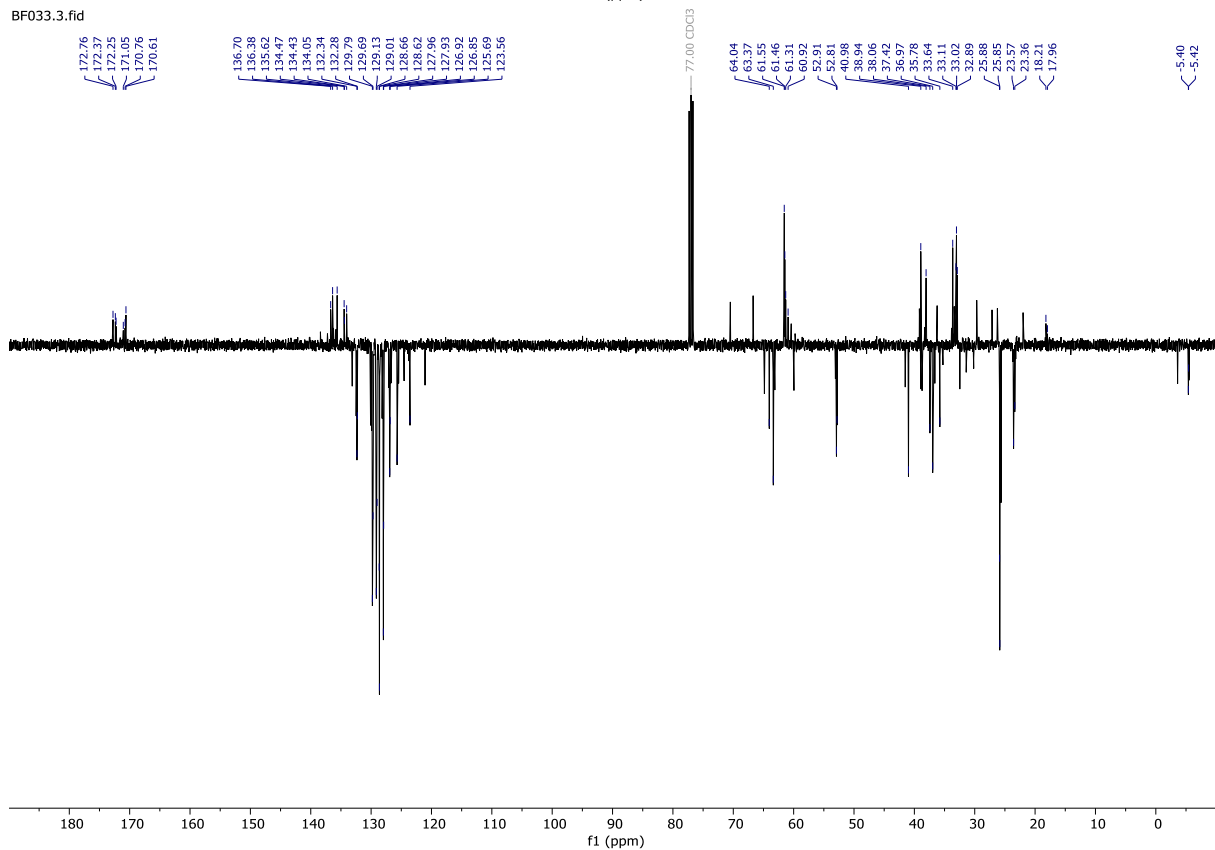


11

BF033.1.fid

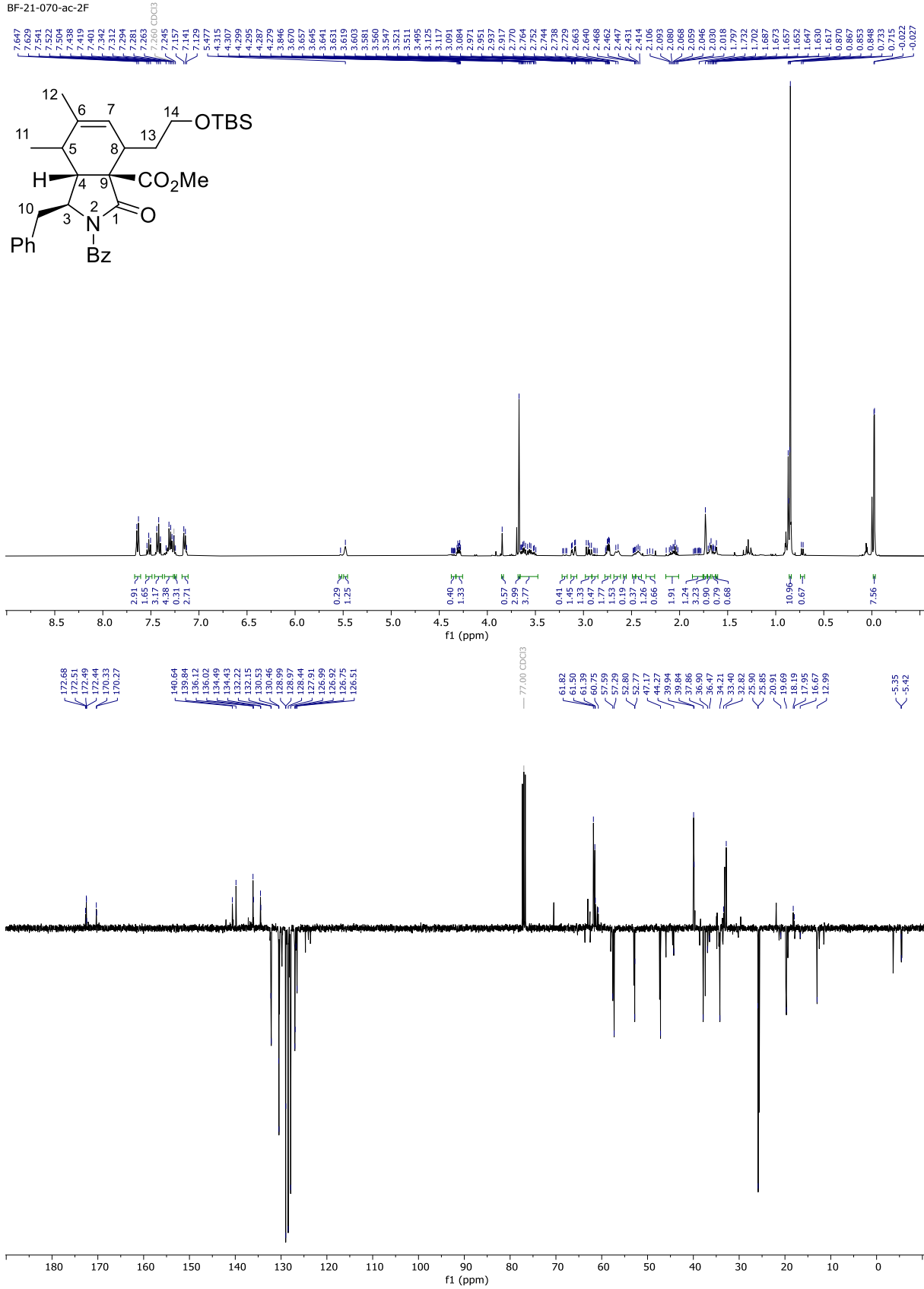


BF033.3.fid



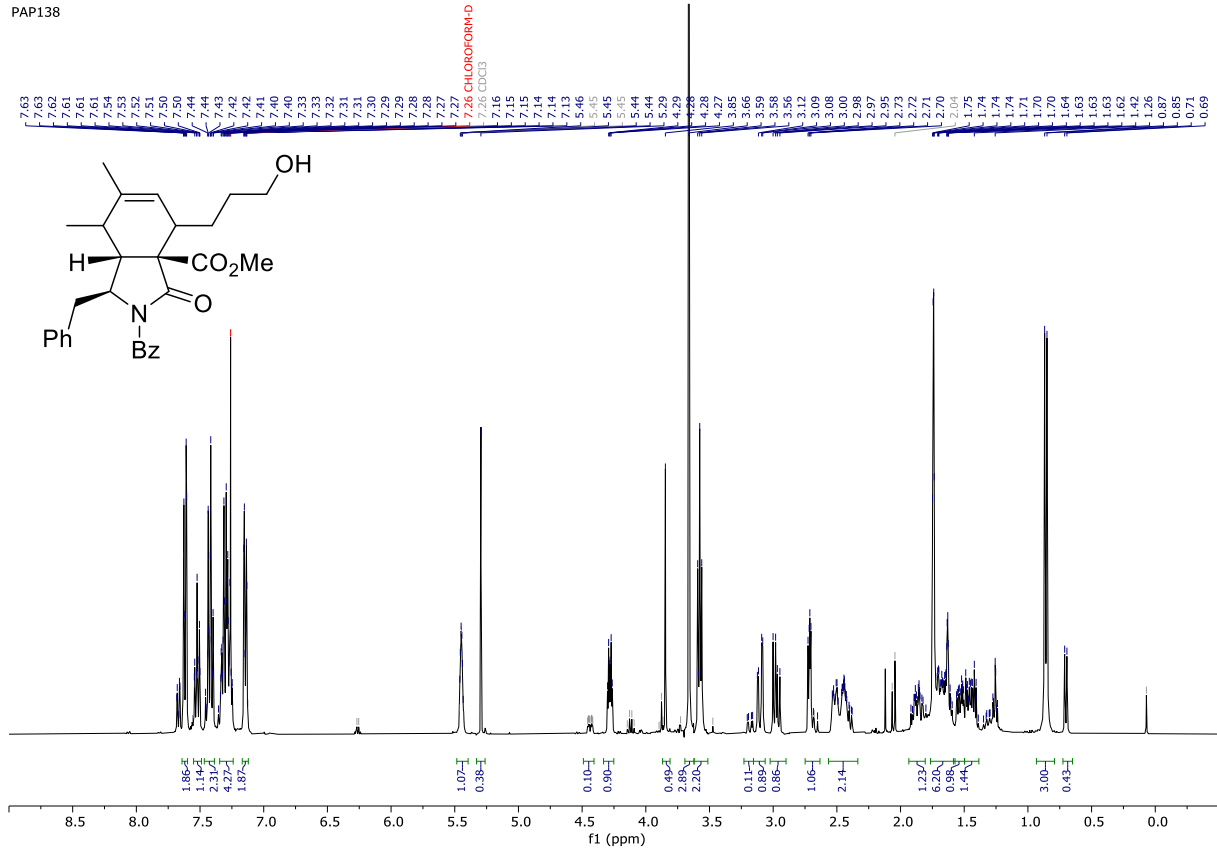
12

BF-21-070-ac-2F

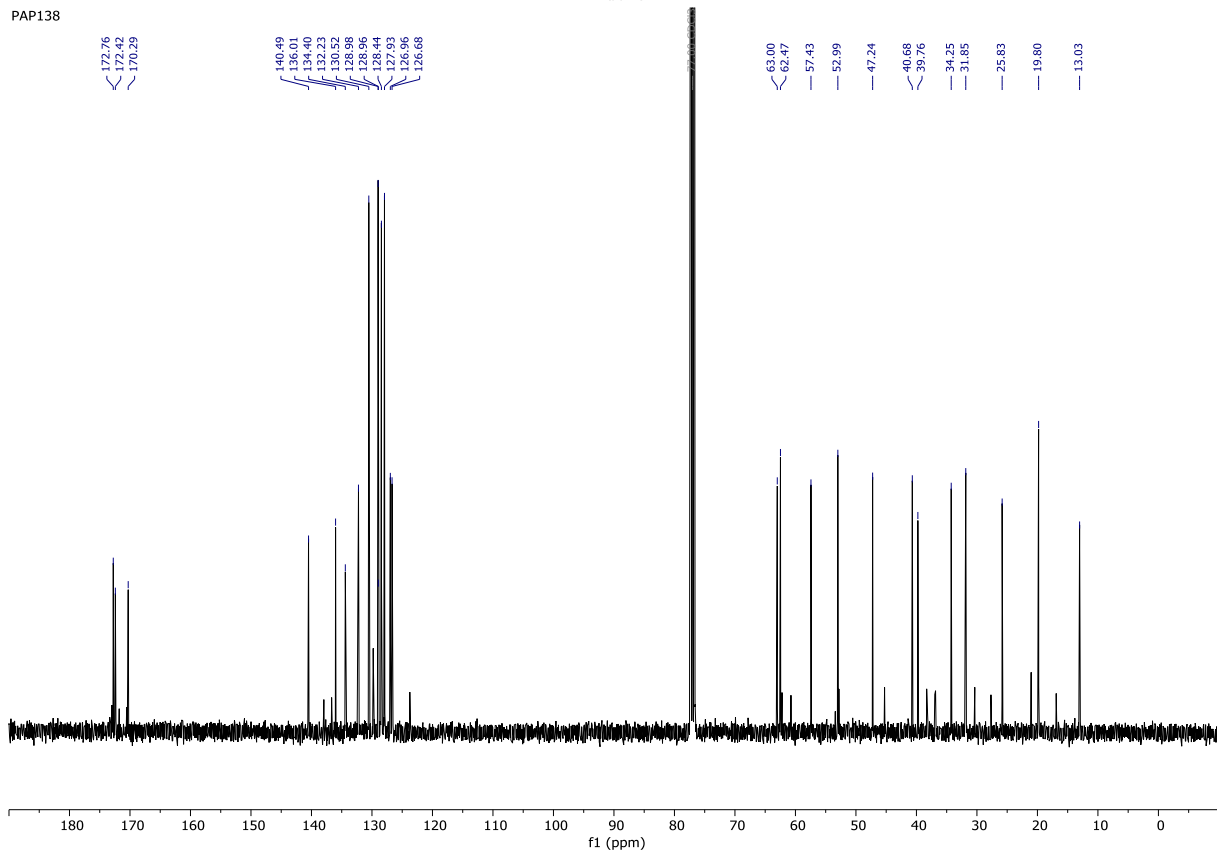


14

PAP138

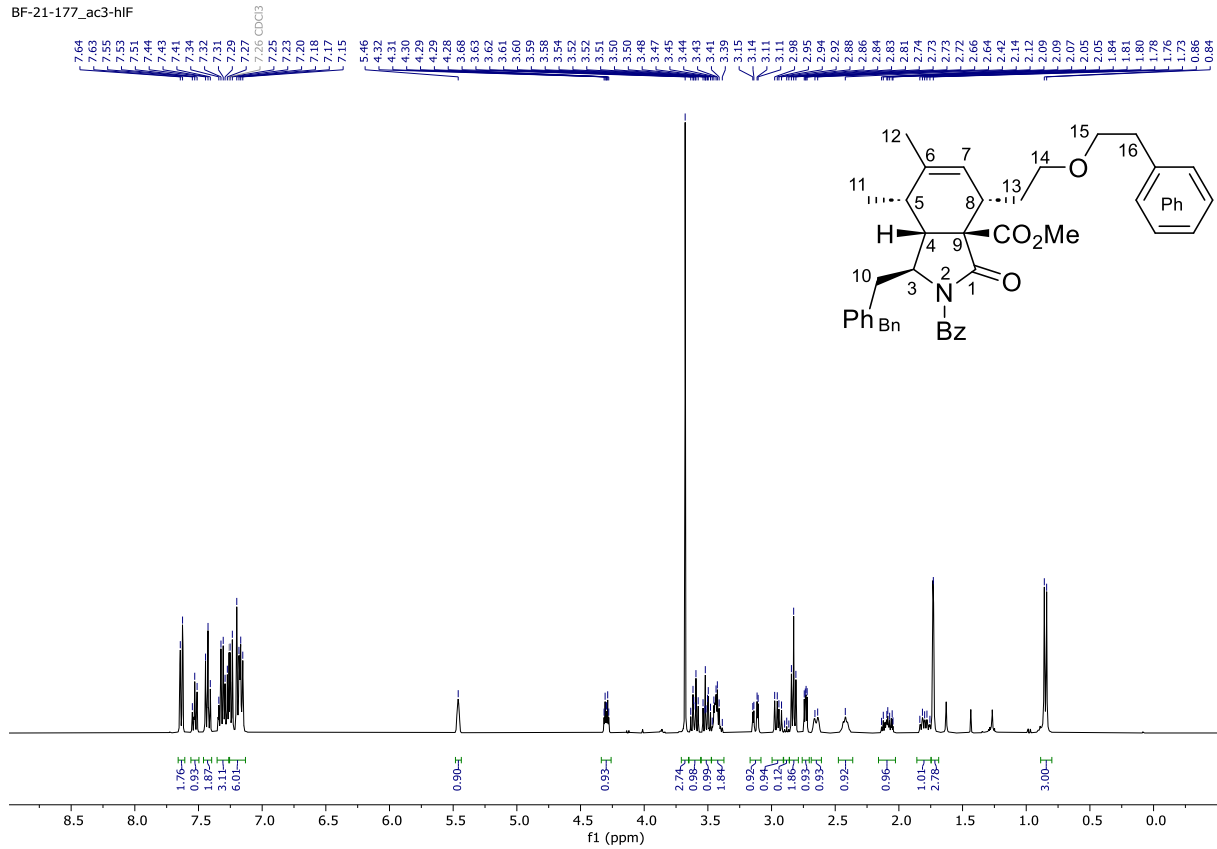


PAP138

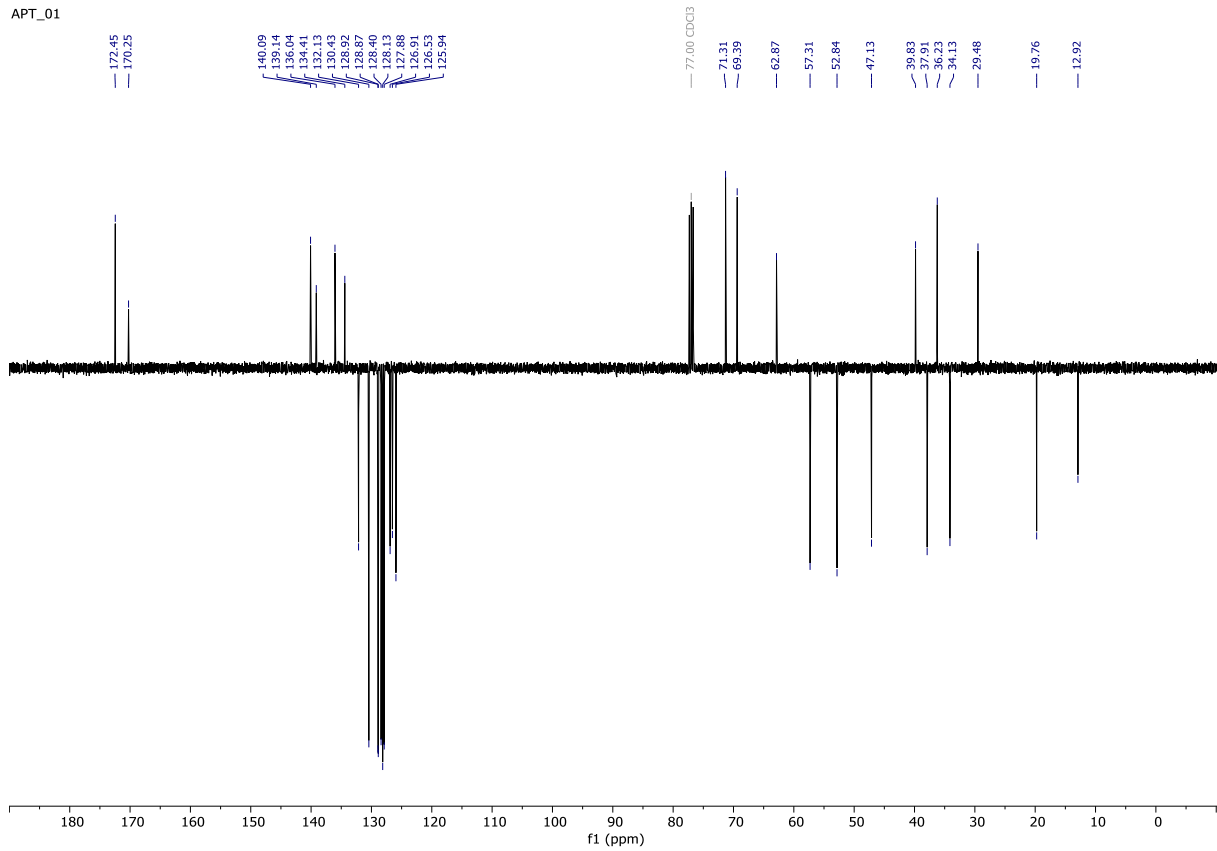


16a

BF-21-177_ac3-hf

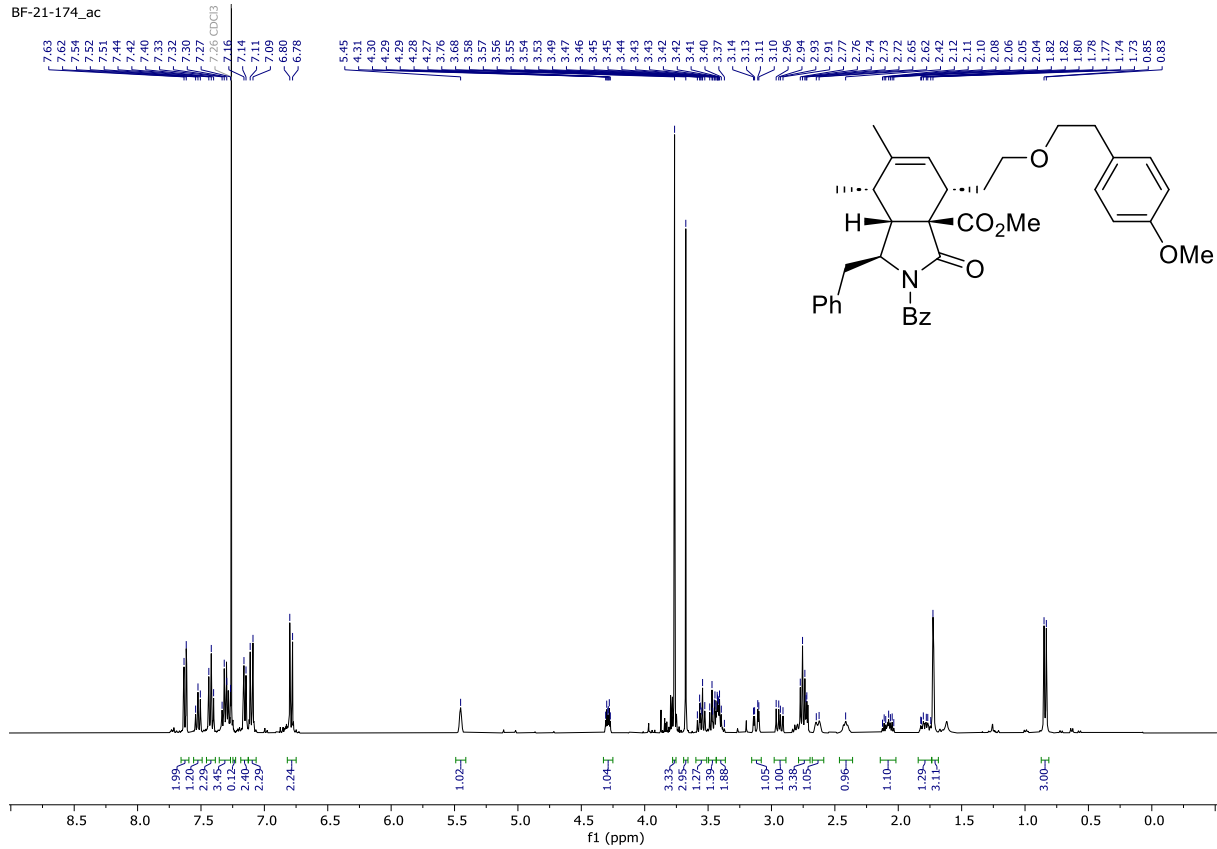


APT_01

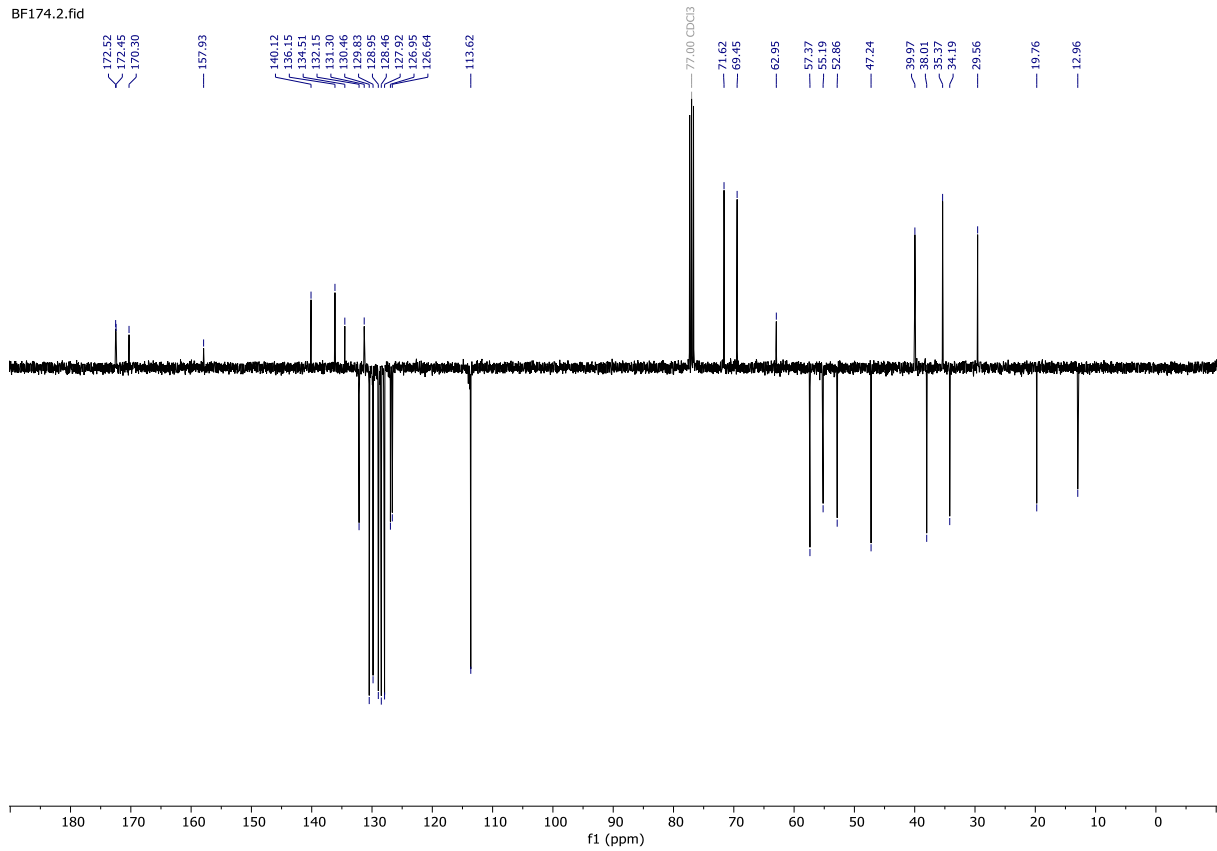


16b

BF-21-174_ac

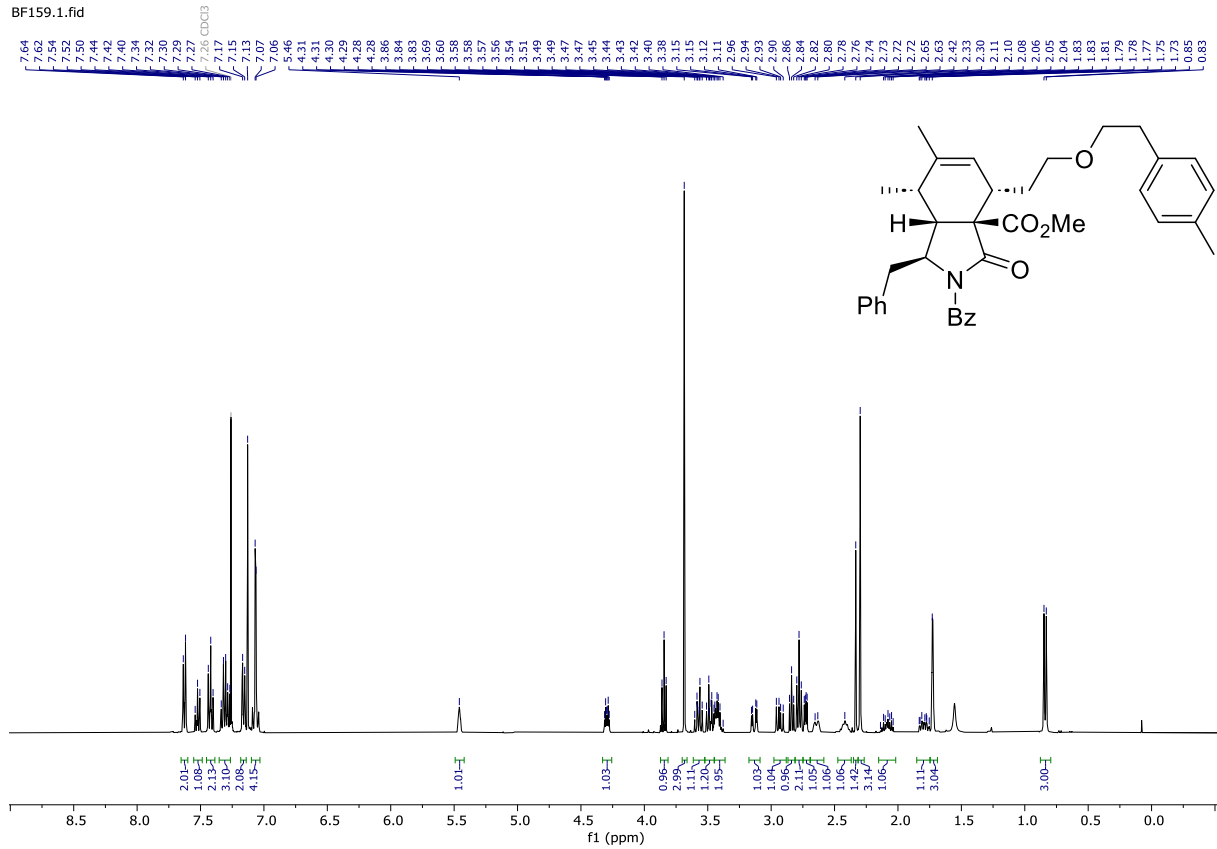


BF174.2.fid

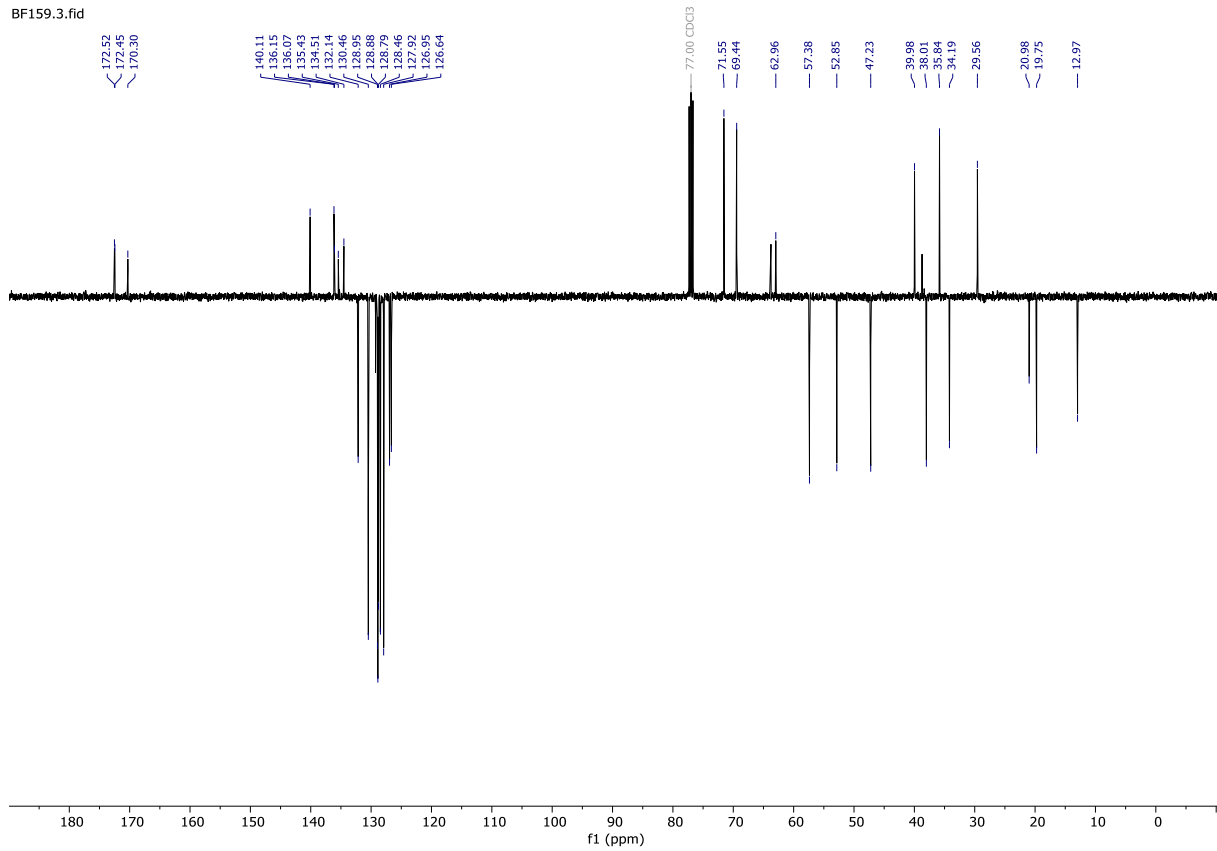


16c

BF159.1.fid

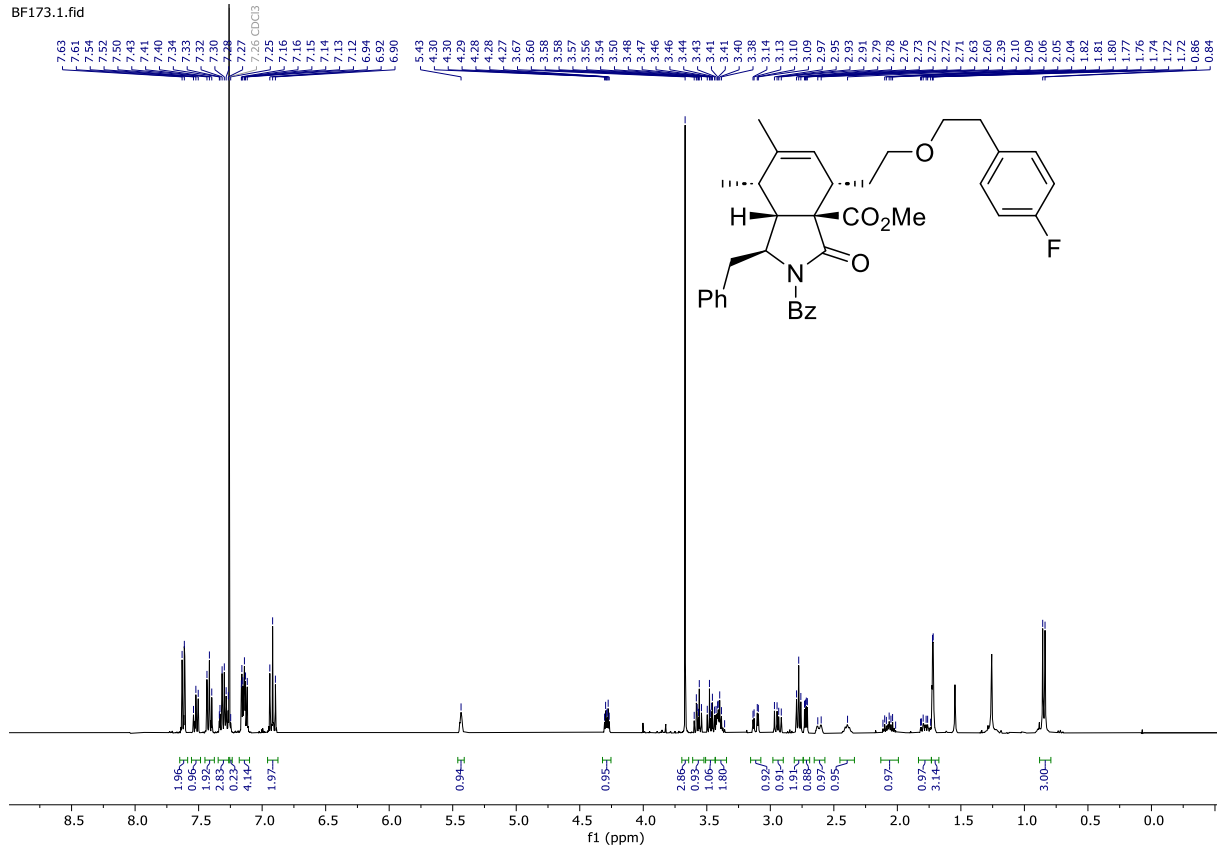


BF159.3.fid

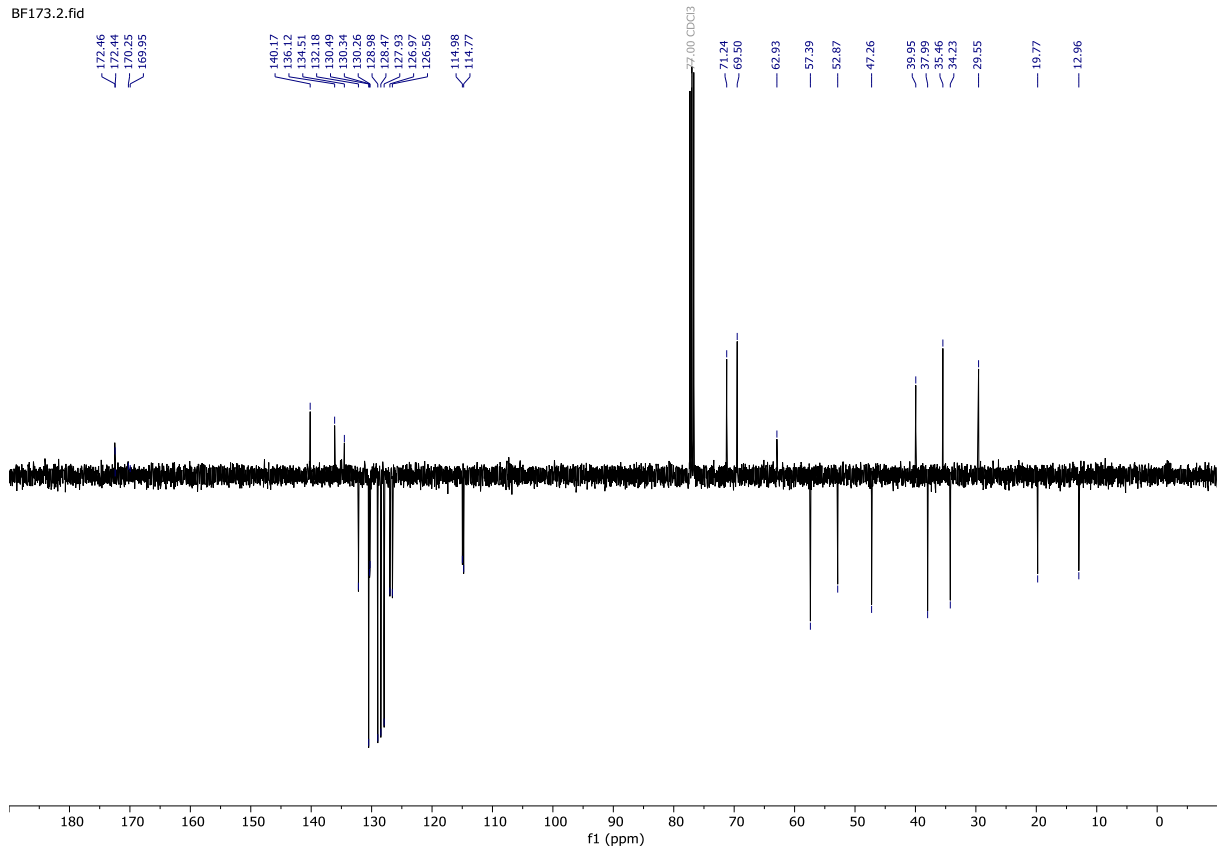


16d

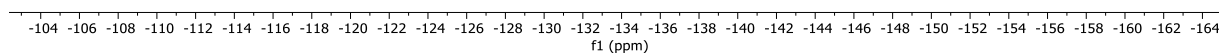
BF173.1.fid

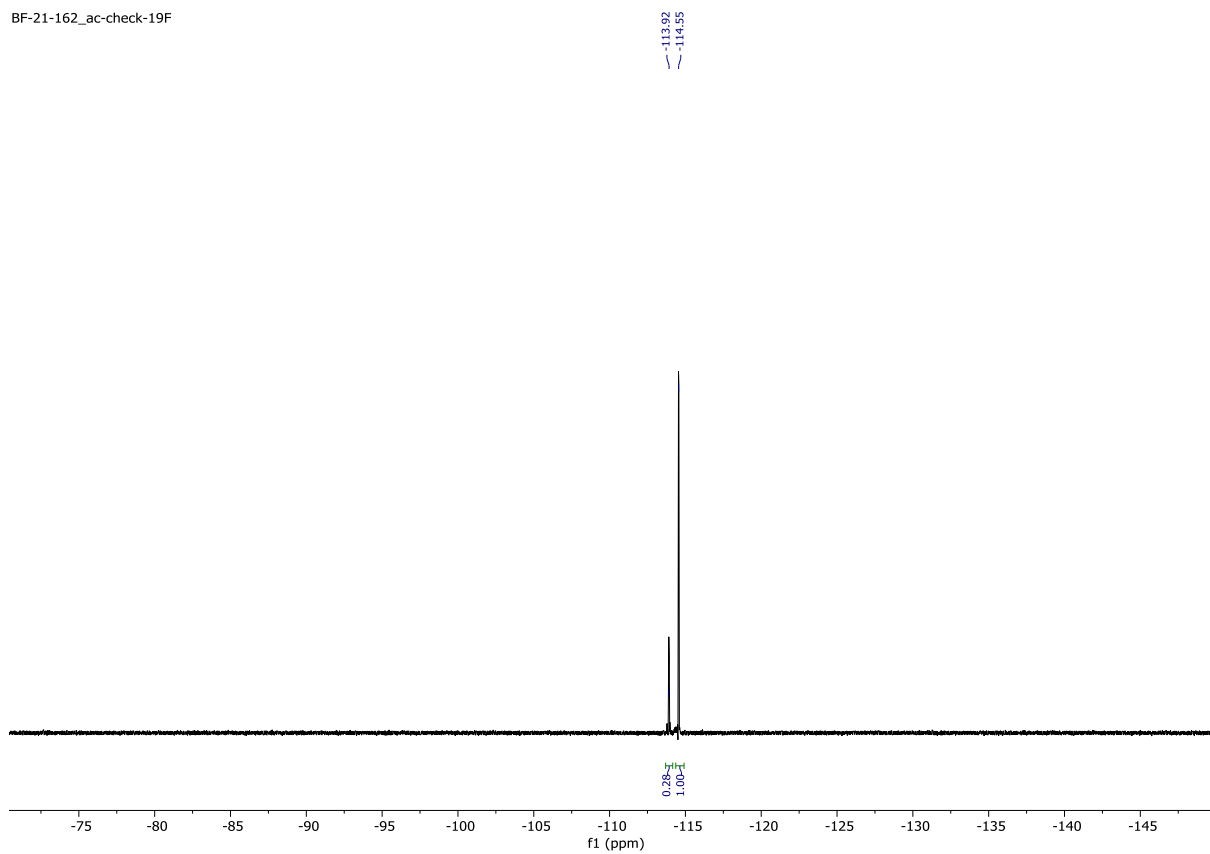


BF173.2.fid



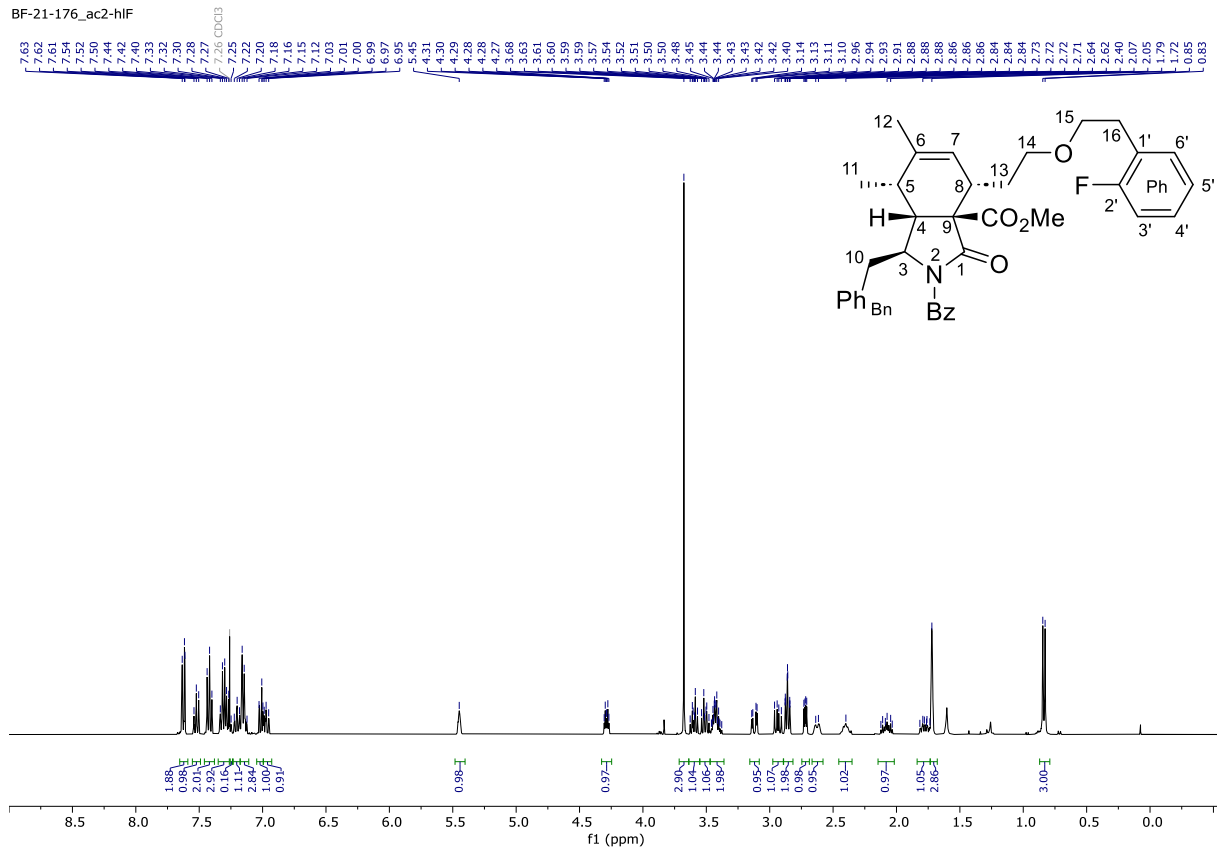
— 144.71



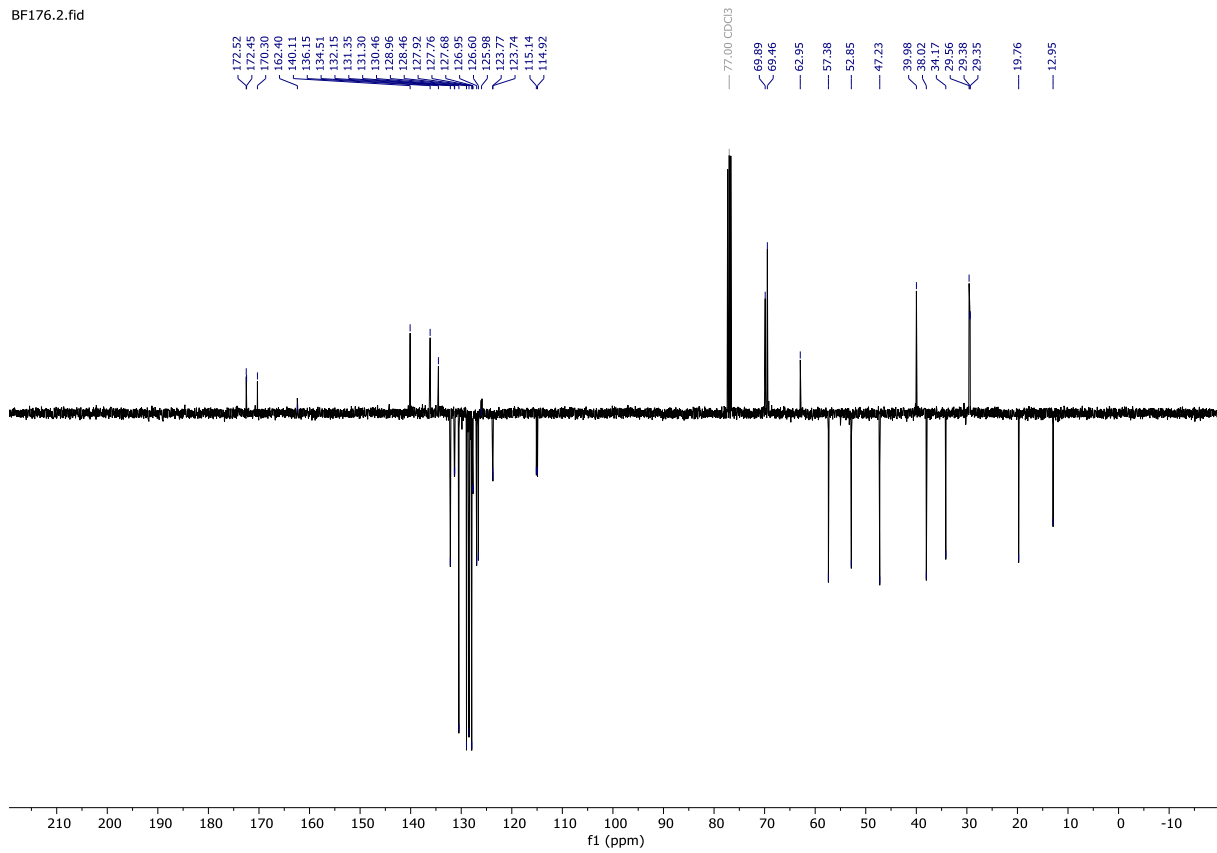


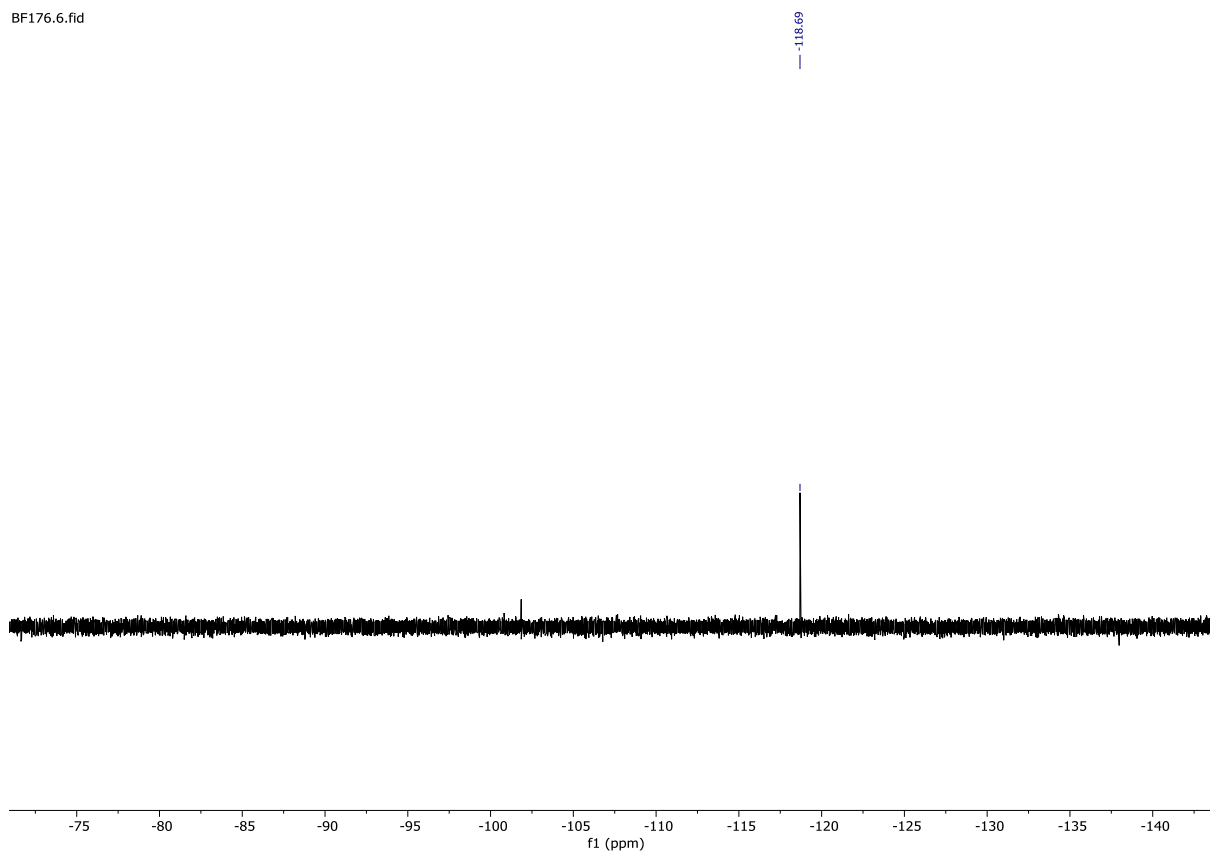
16f

BF-21-176_ac2-h1F



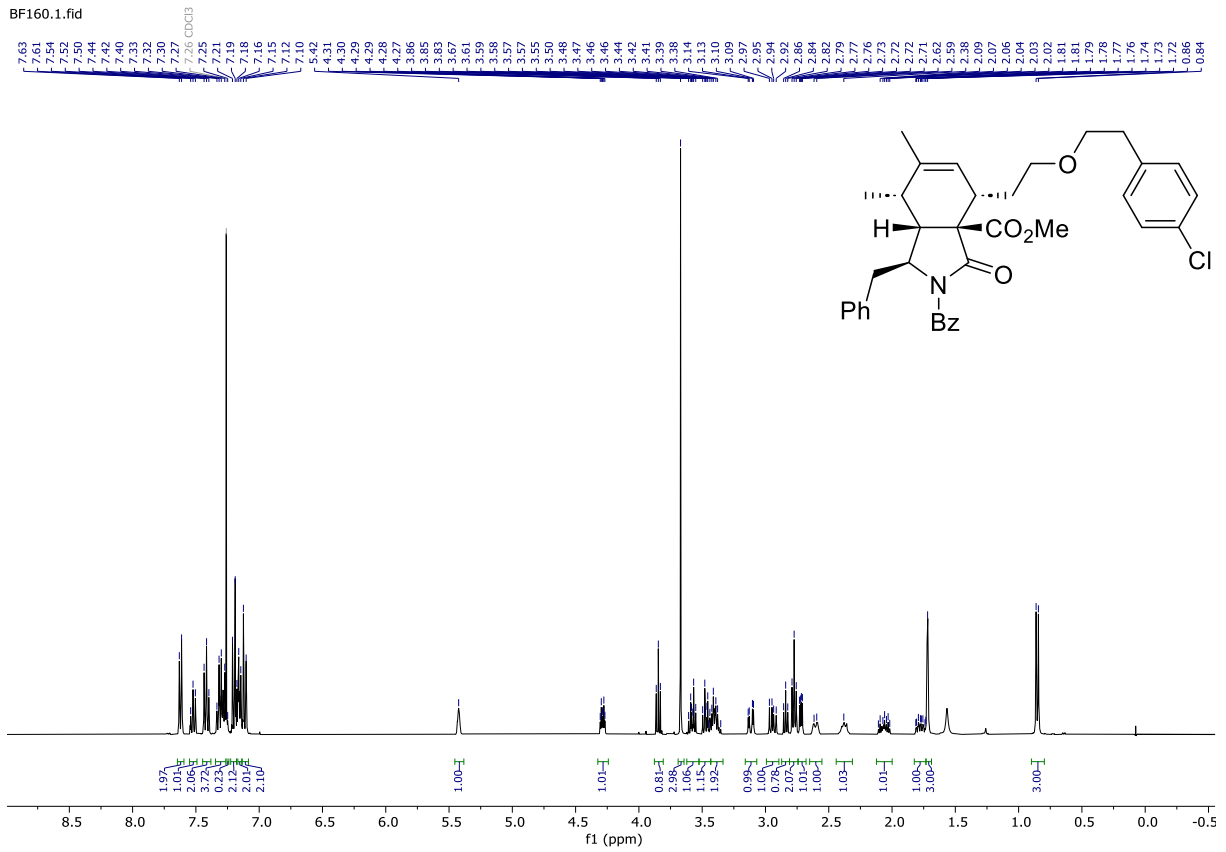
BF176.2.fid



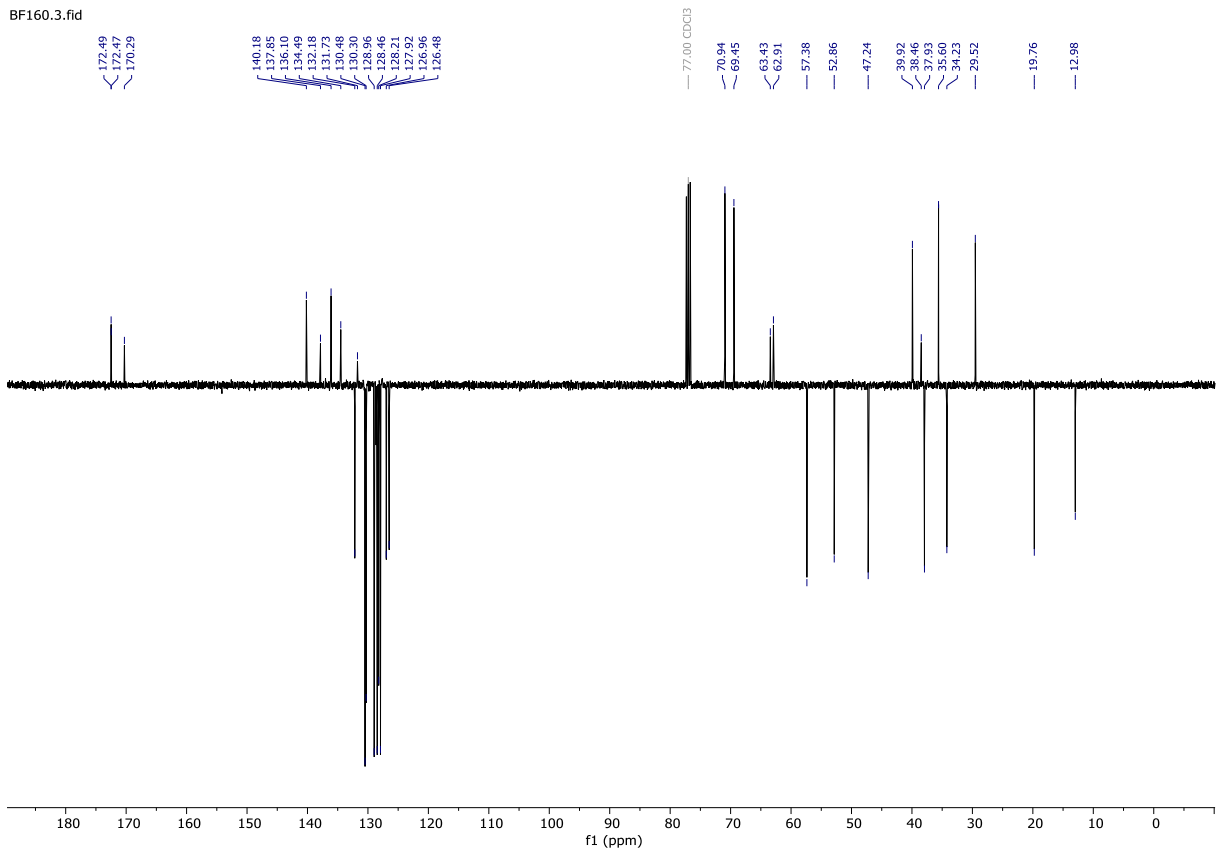


16g

BF160.1.fid

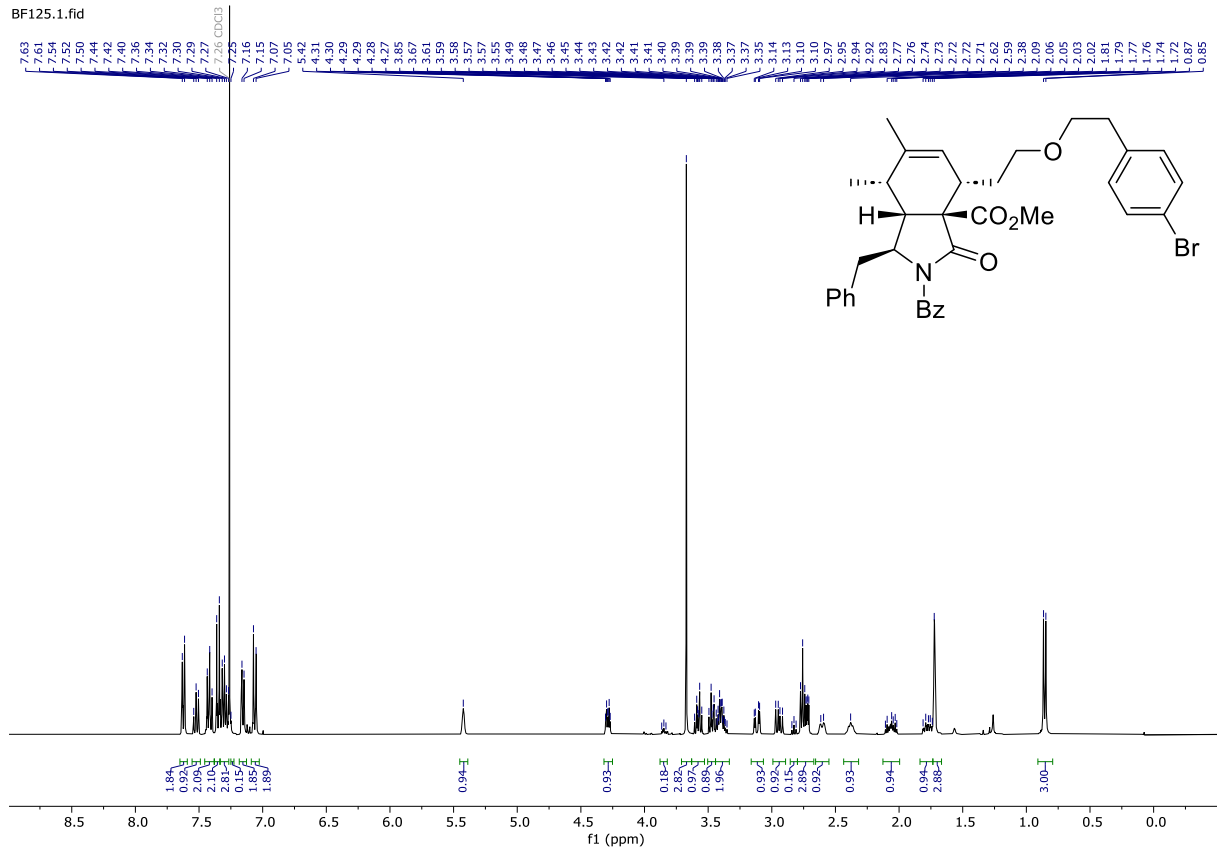


BF160.3.fid

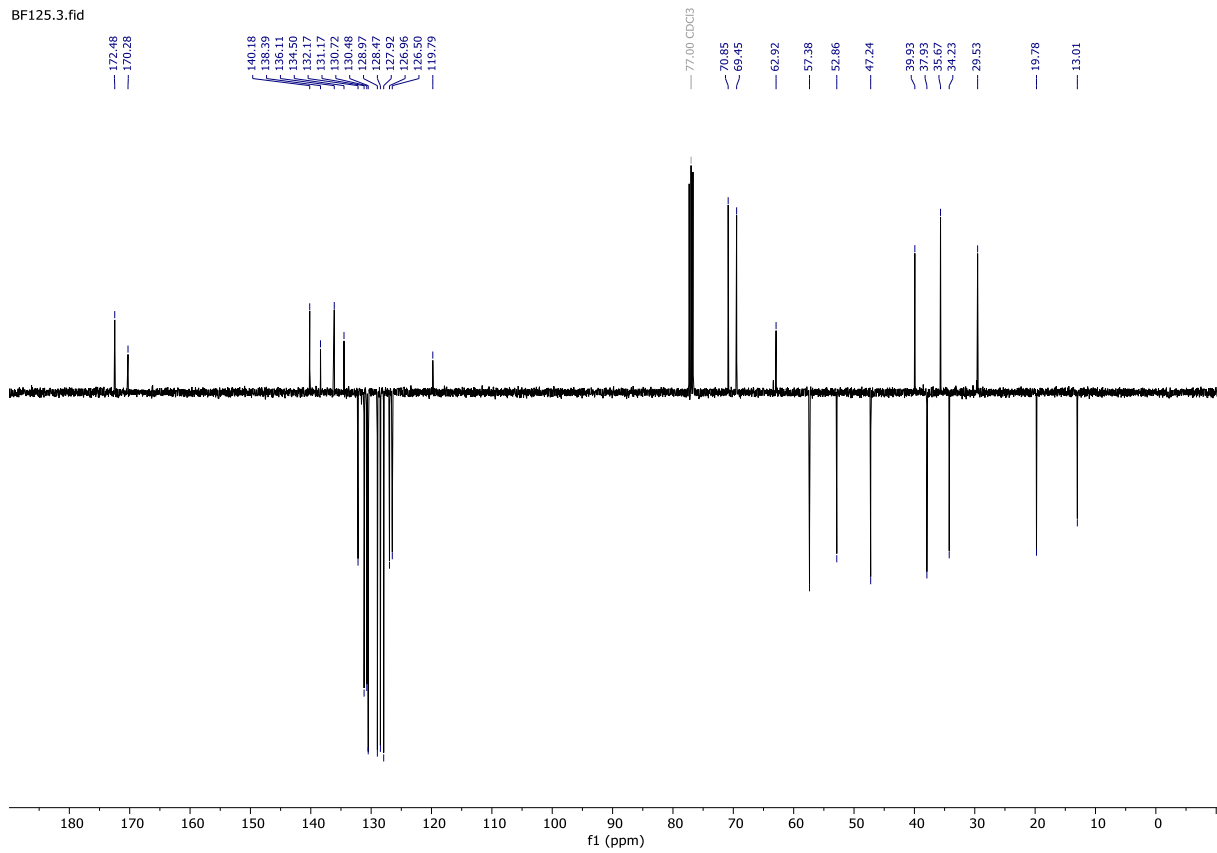


16h

BF125.1.fid

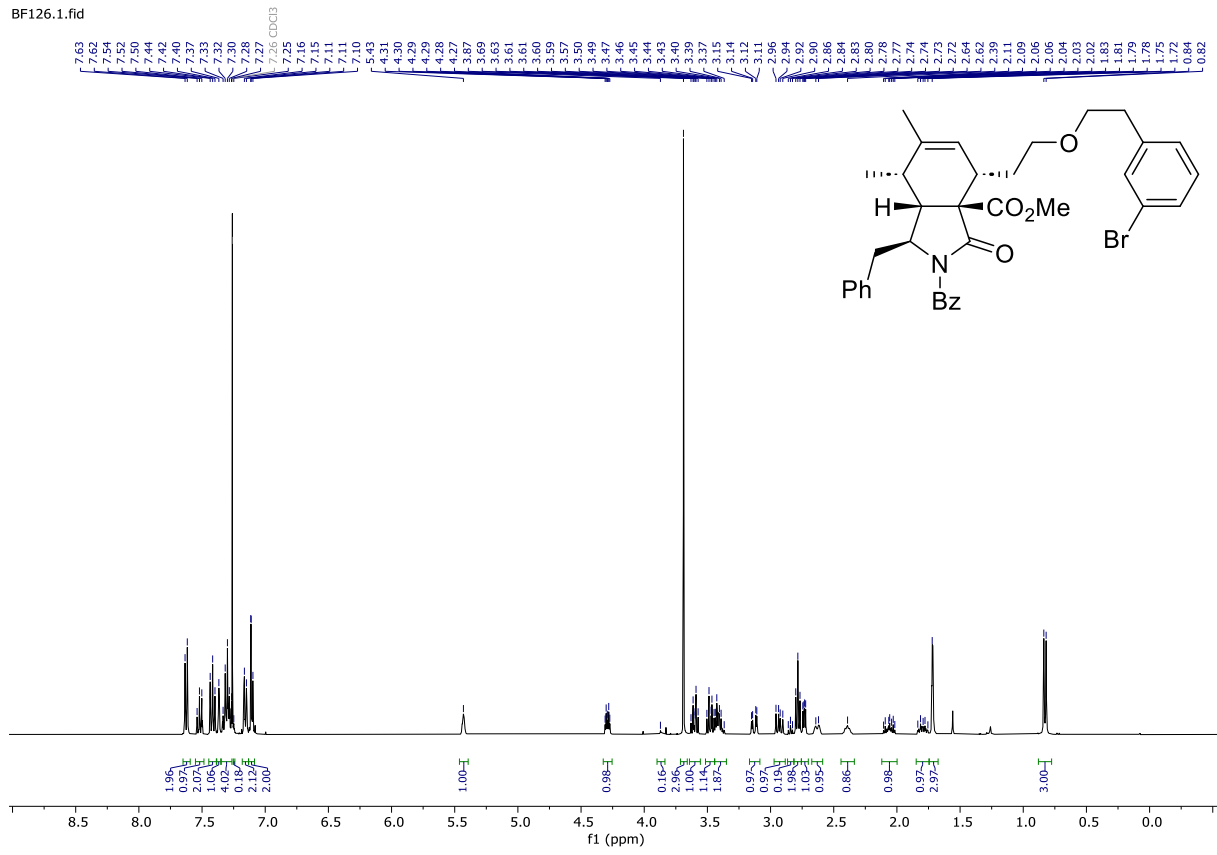


BF125.3.fid

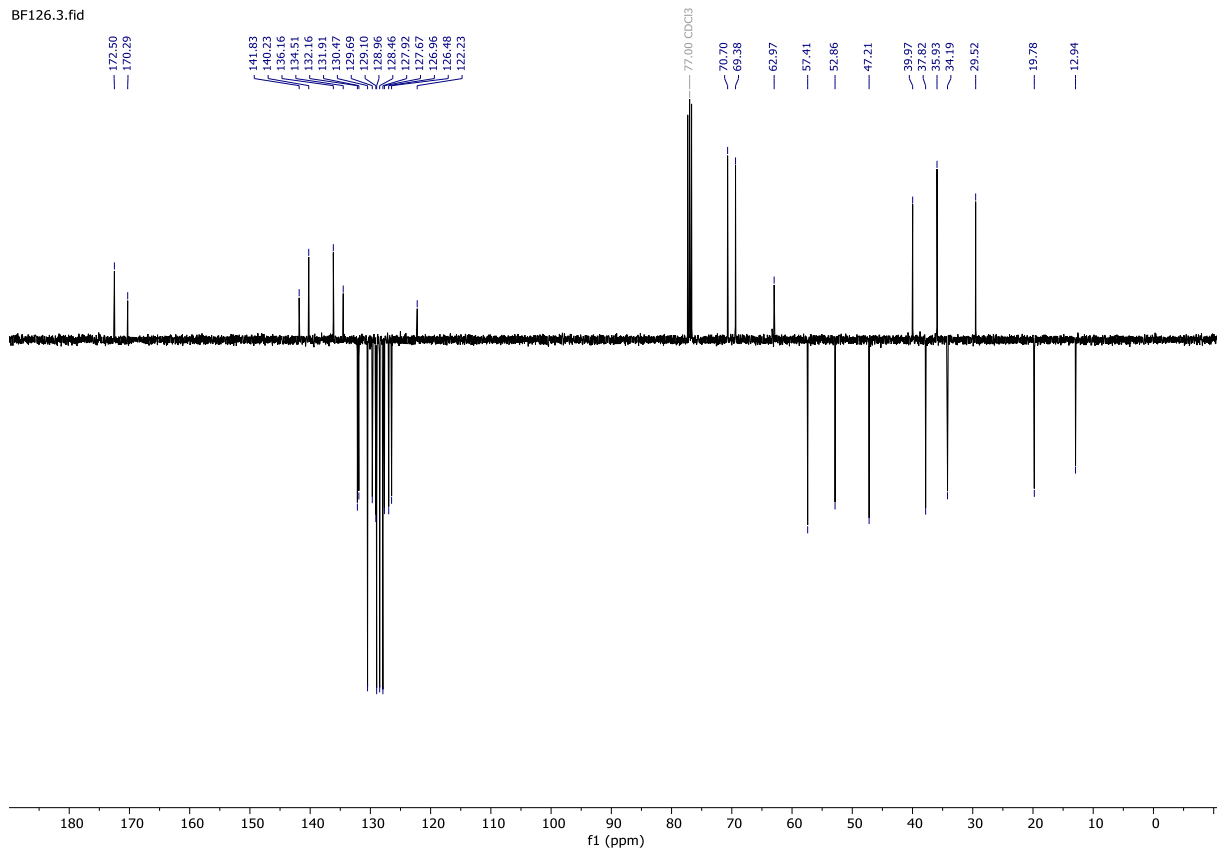


16i

BF126.1.fid

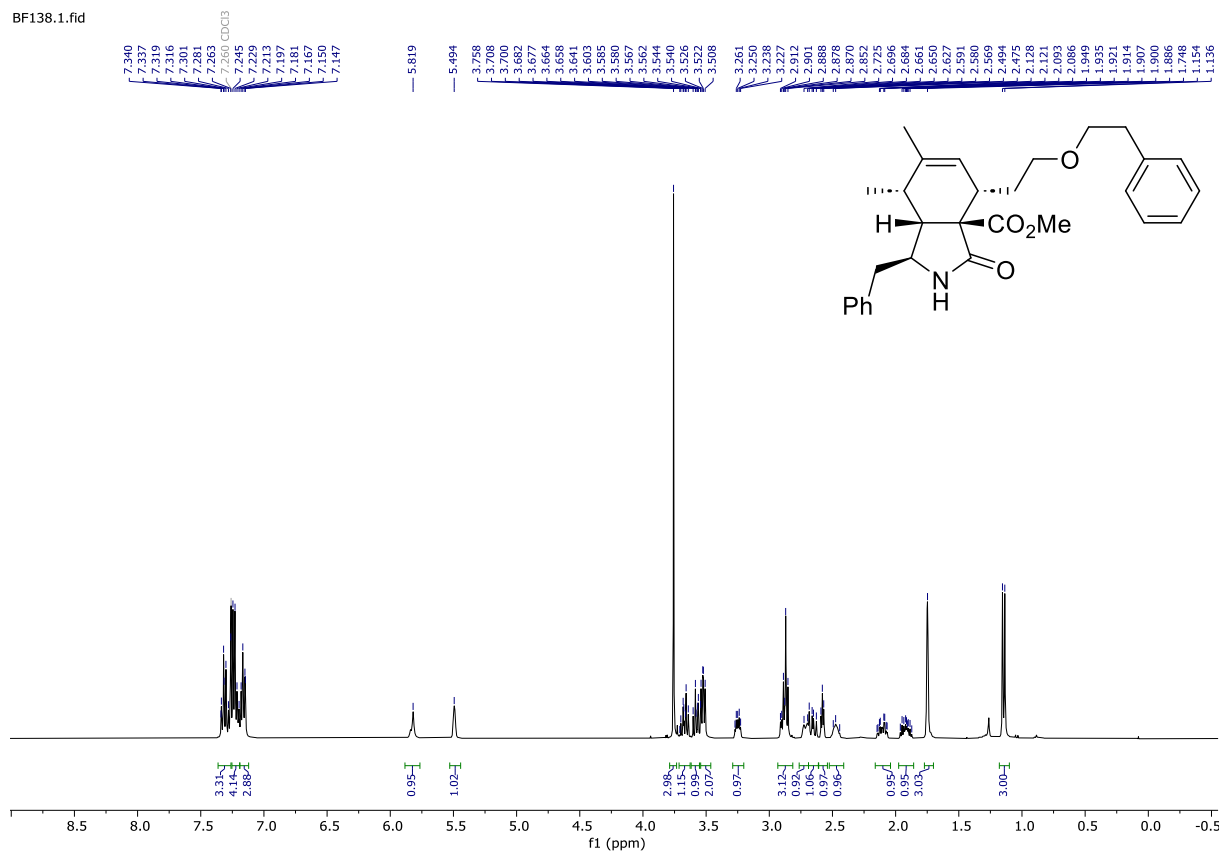


BF126.3.fid

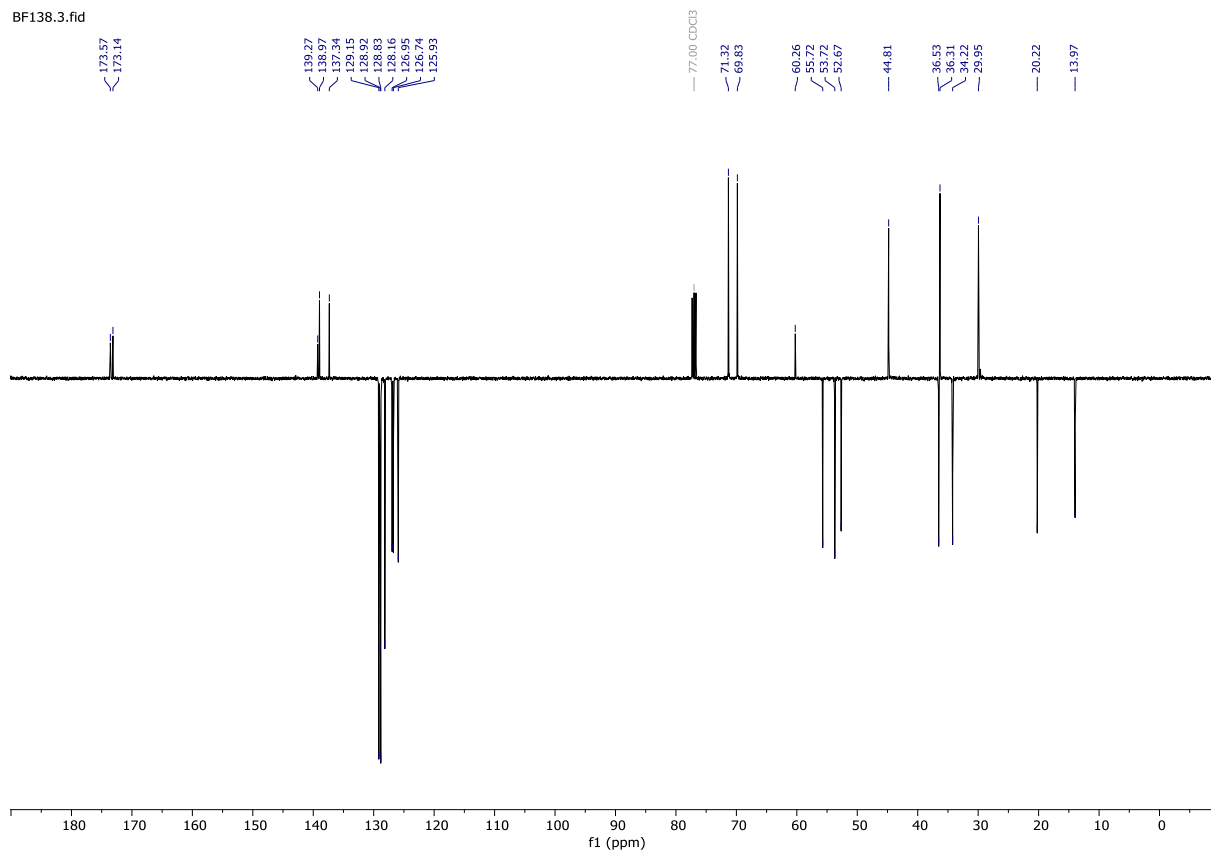


17a

BF138.1.fid

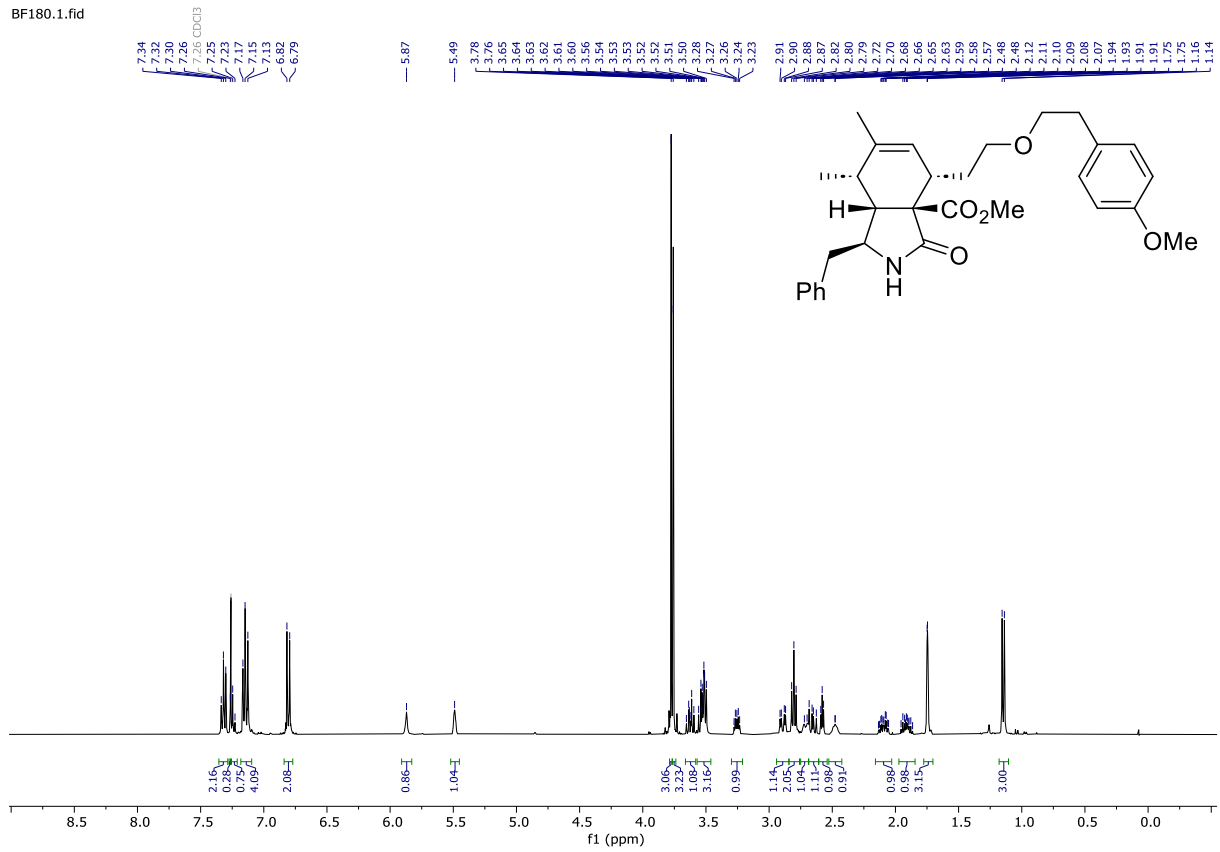


BF138.3.fid

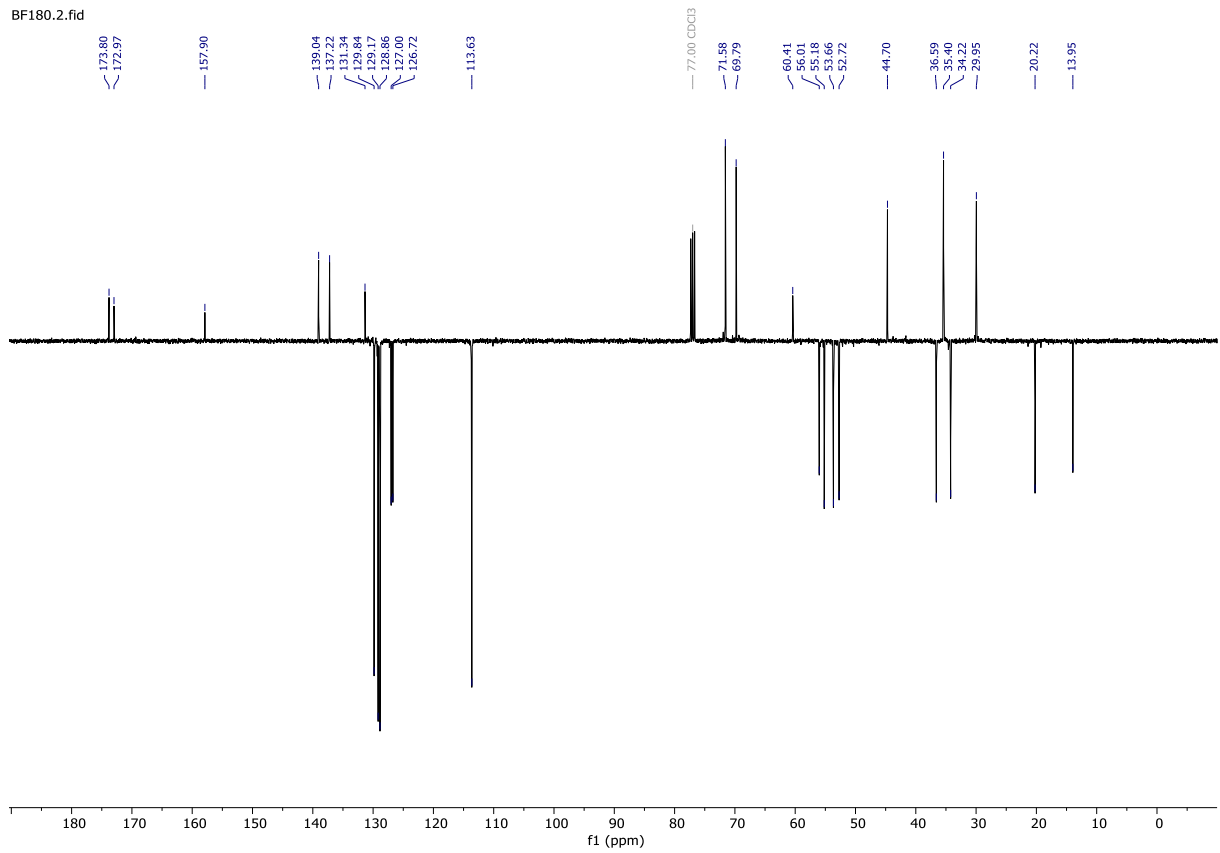


17b

BF180.1.fid

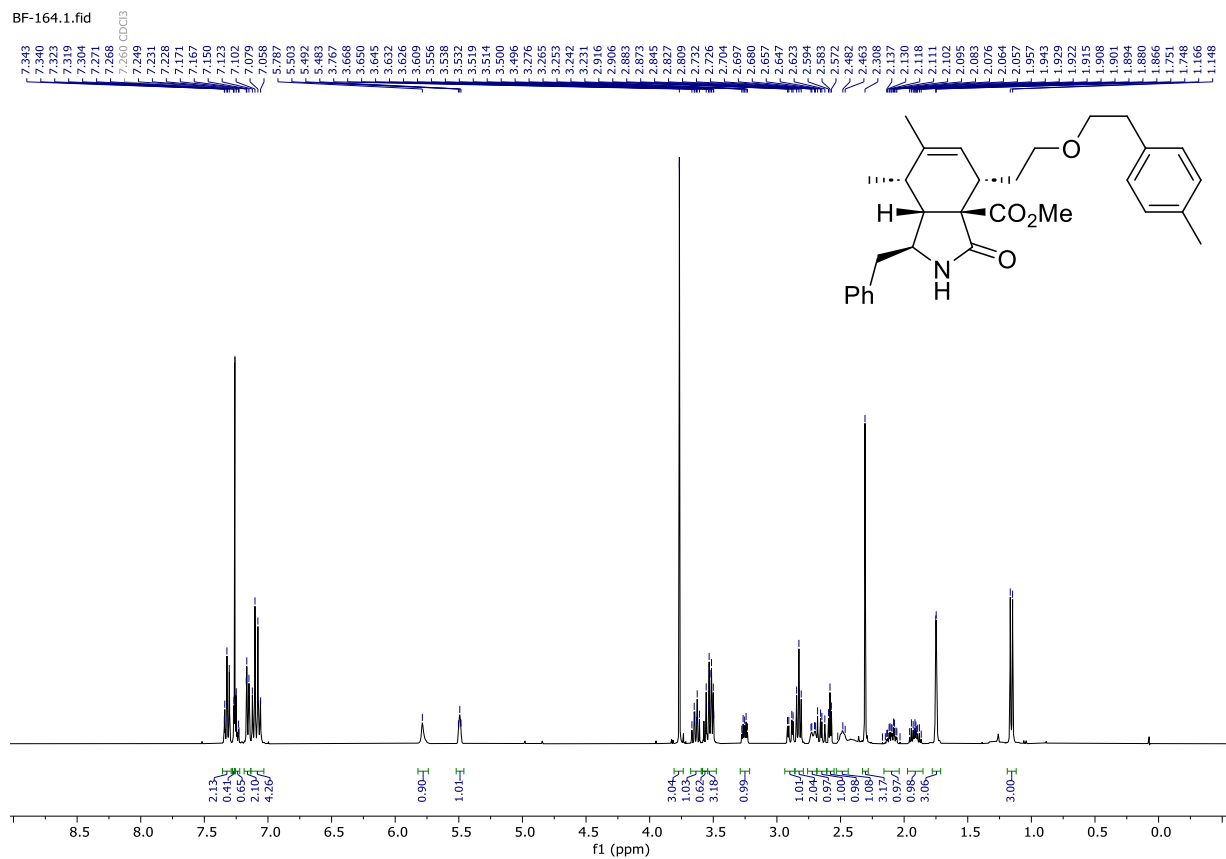


BF180.2.fid

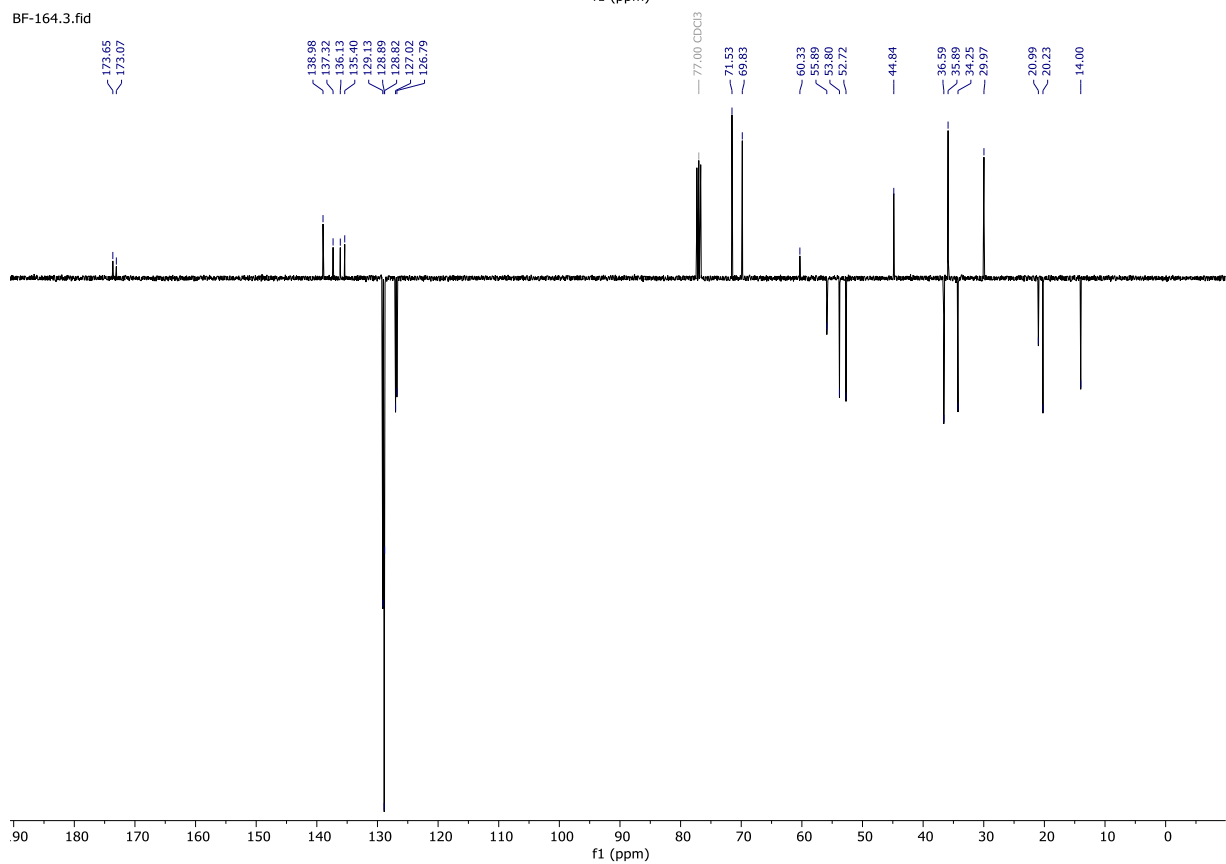


17c

BF-164.1.fid

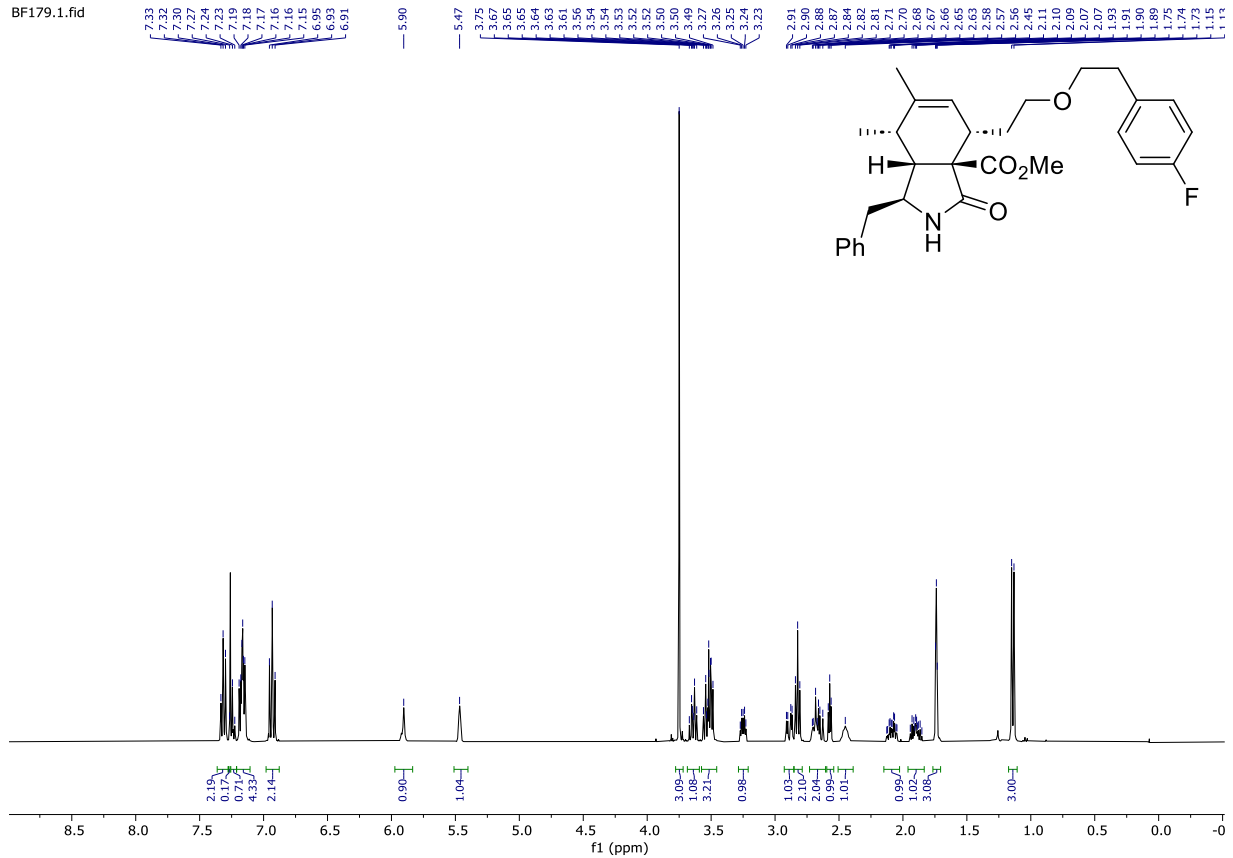


BF-164.3.fid

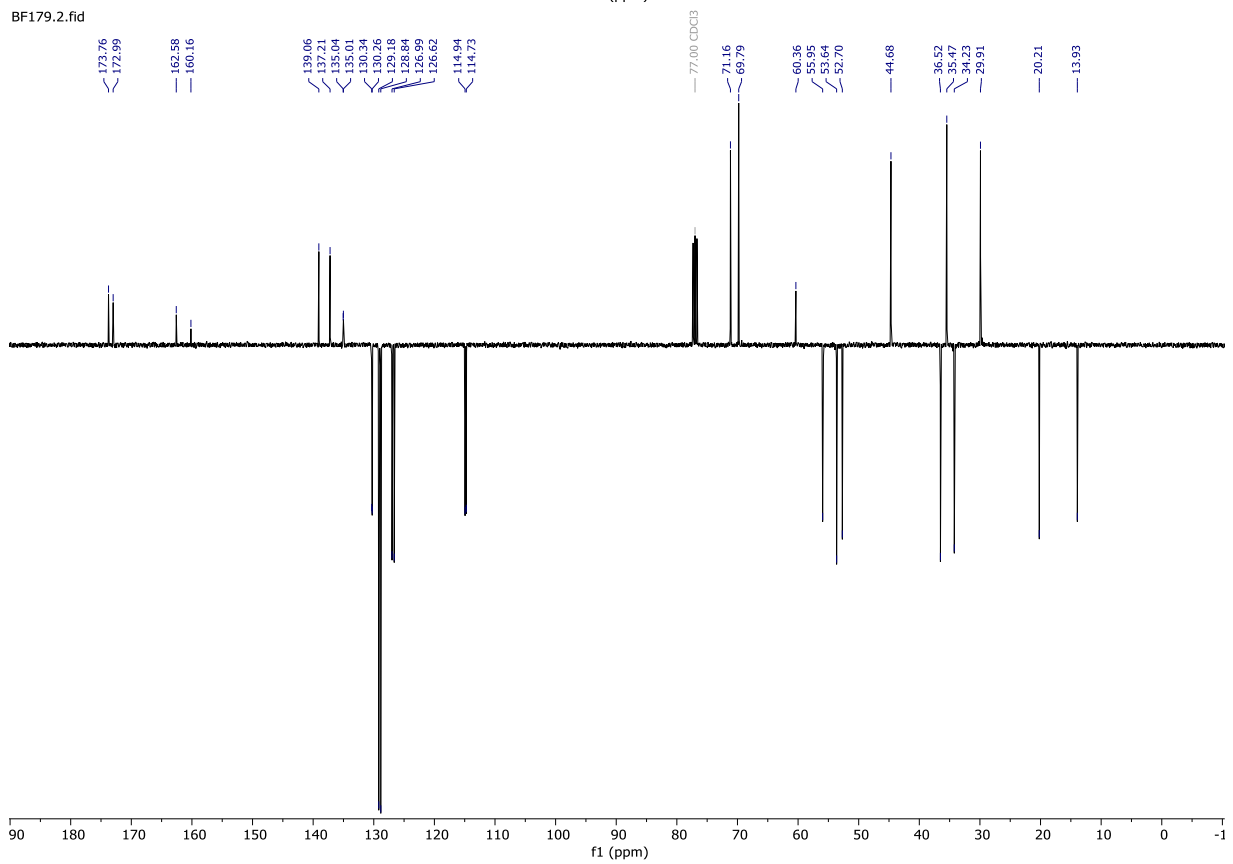


17d

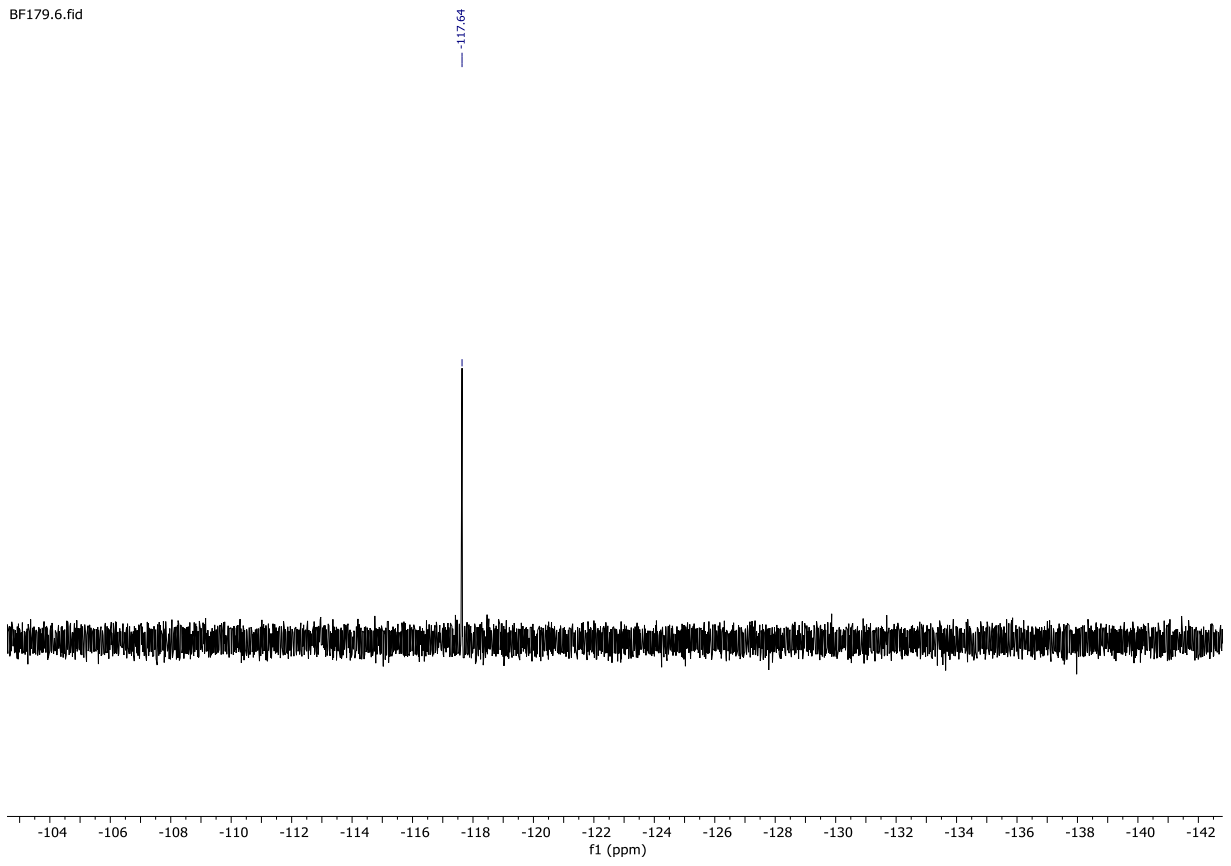
BF179.1.fid



BF179.2.fid

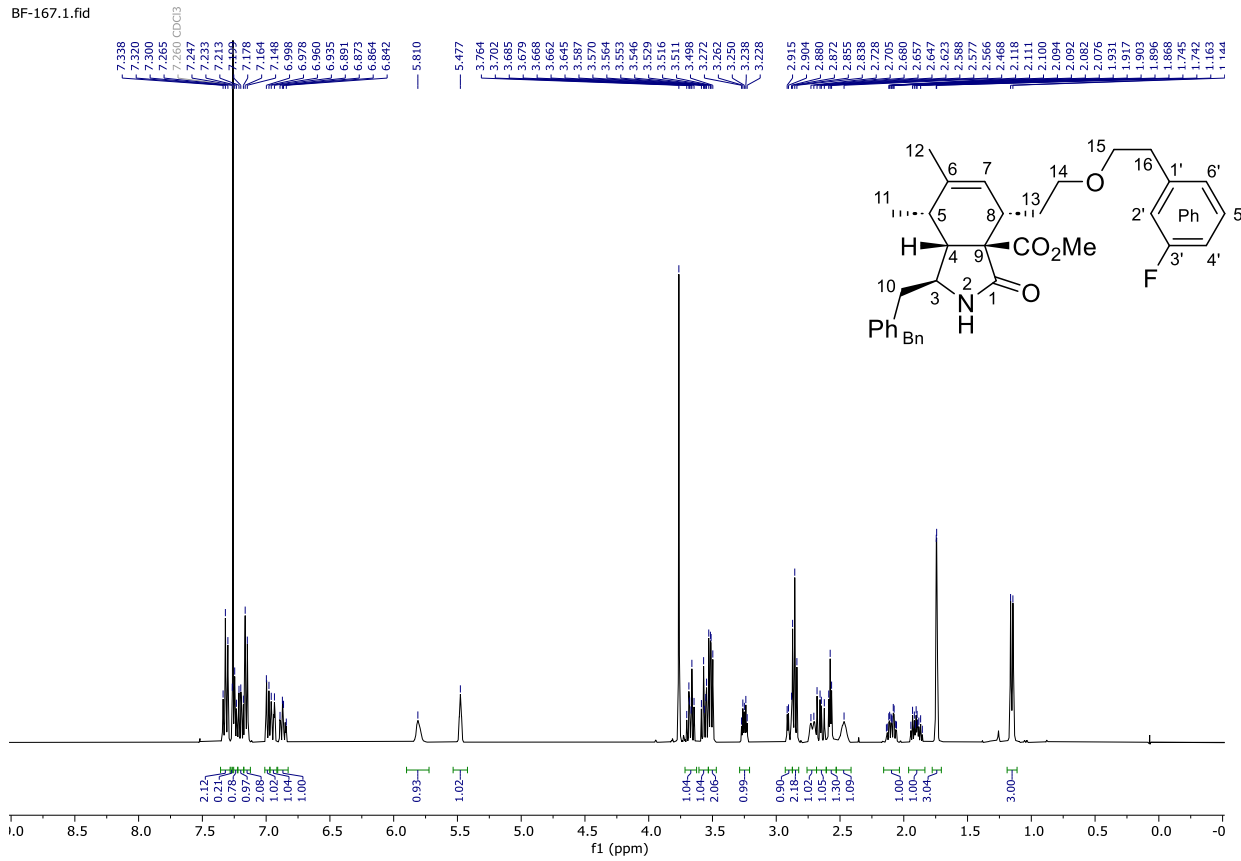


BF179.6.fid

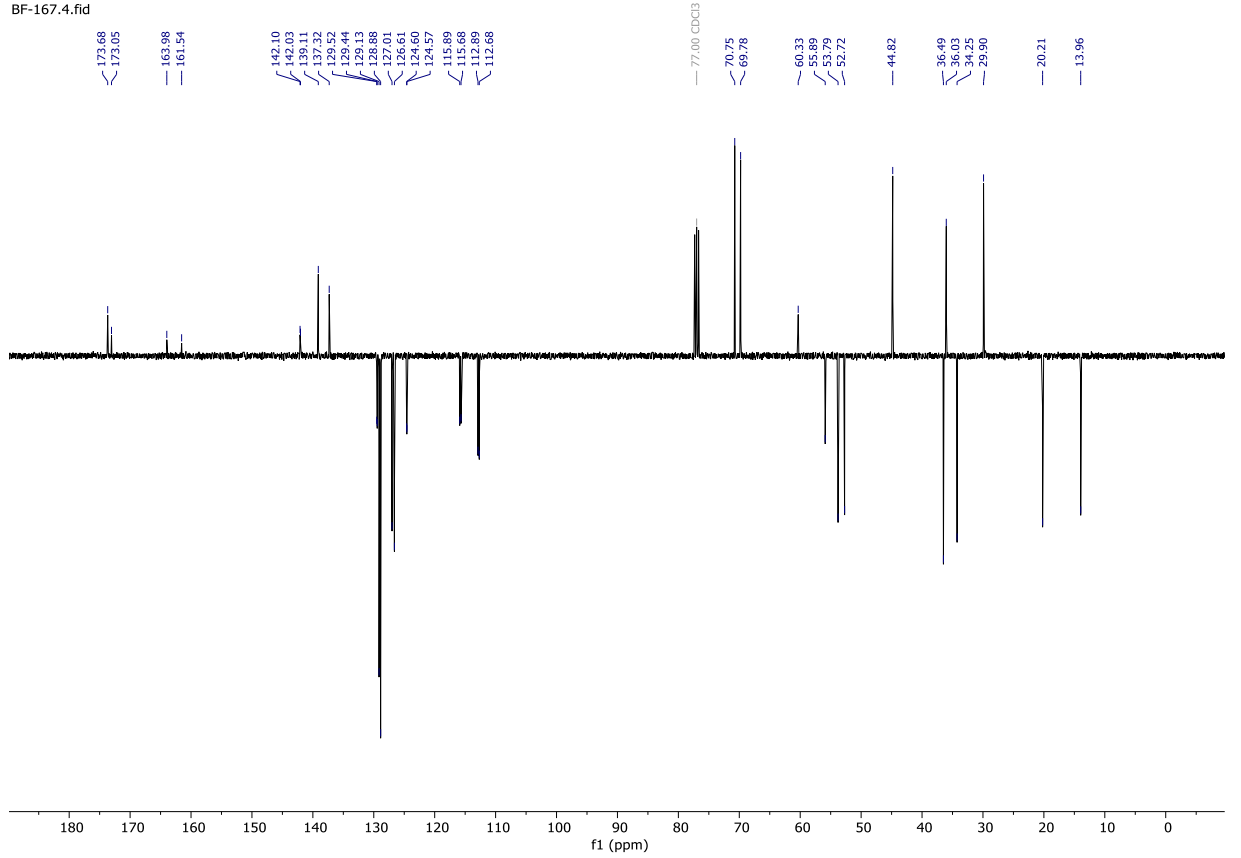


17e

BF-167.1.fid

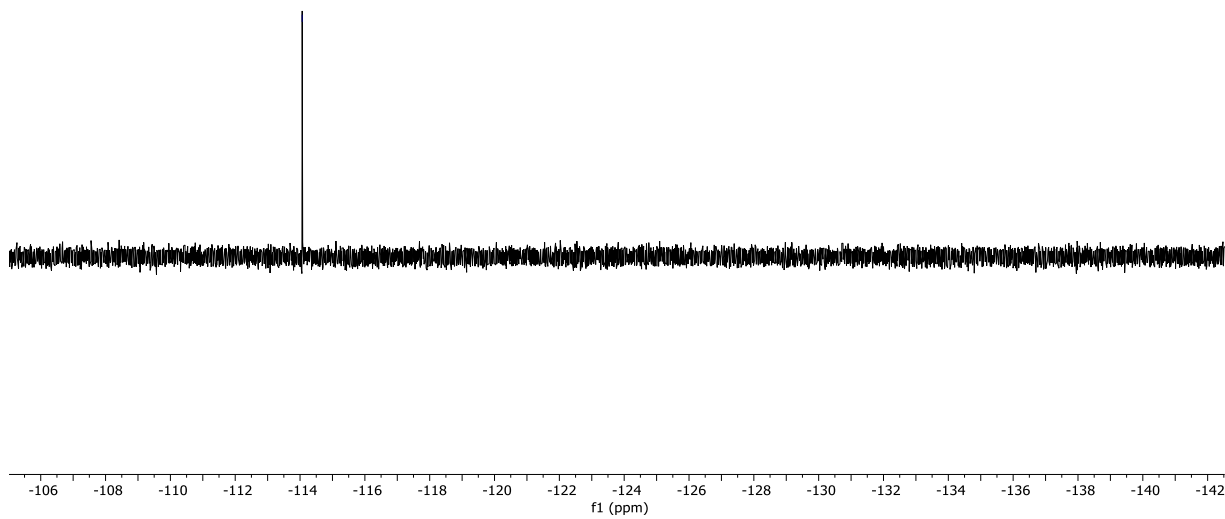


BF-167.4.fid



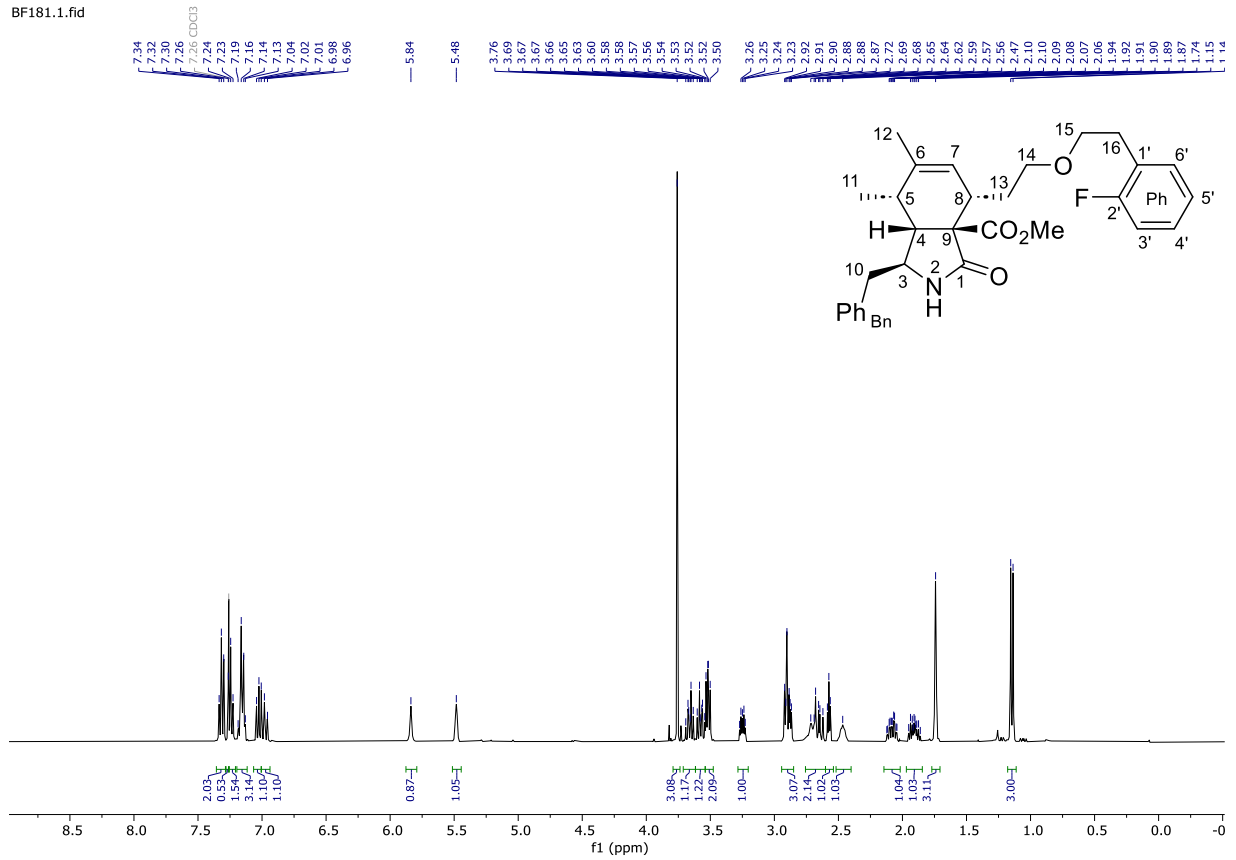
BF-167.3.fid

— 114.07

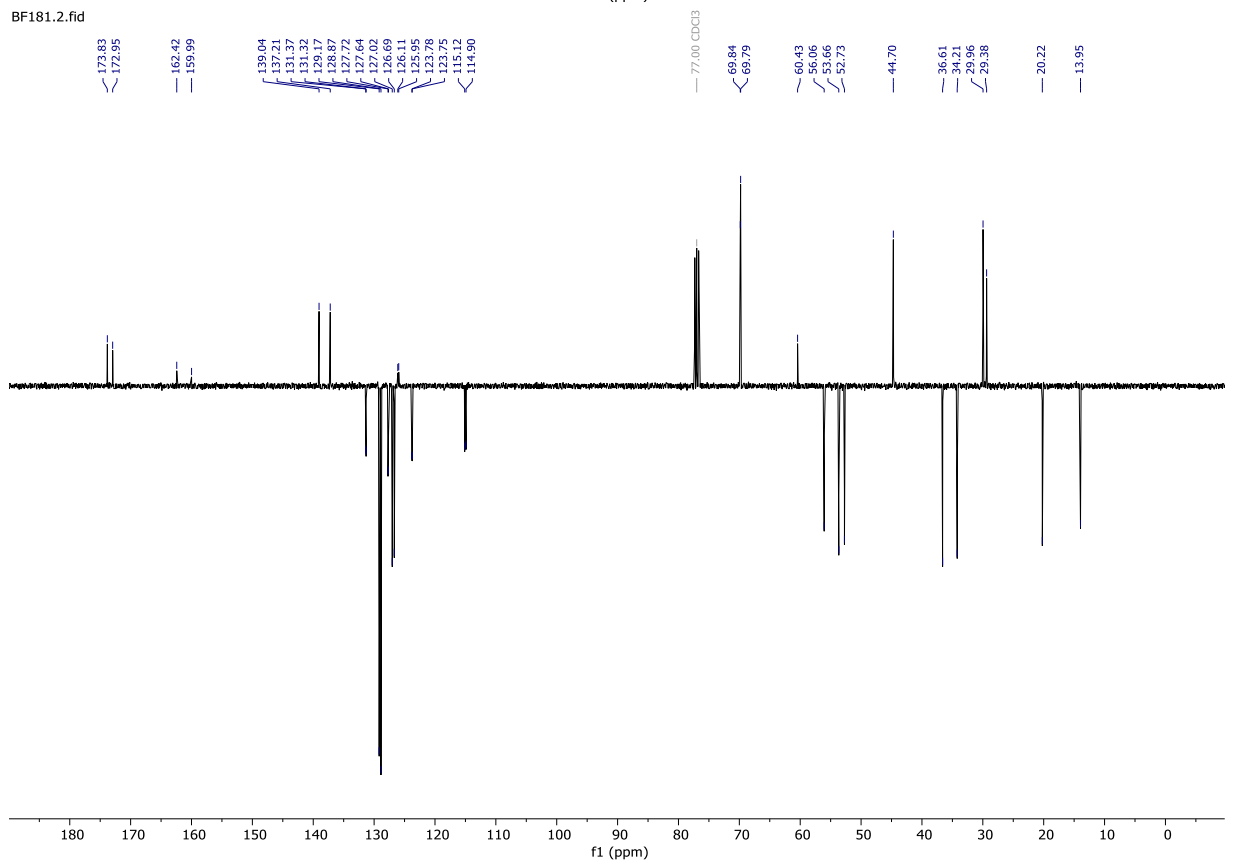


17f

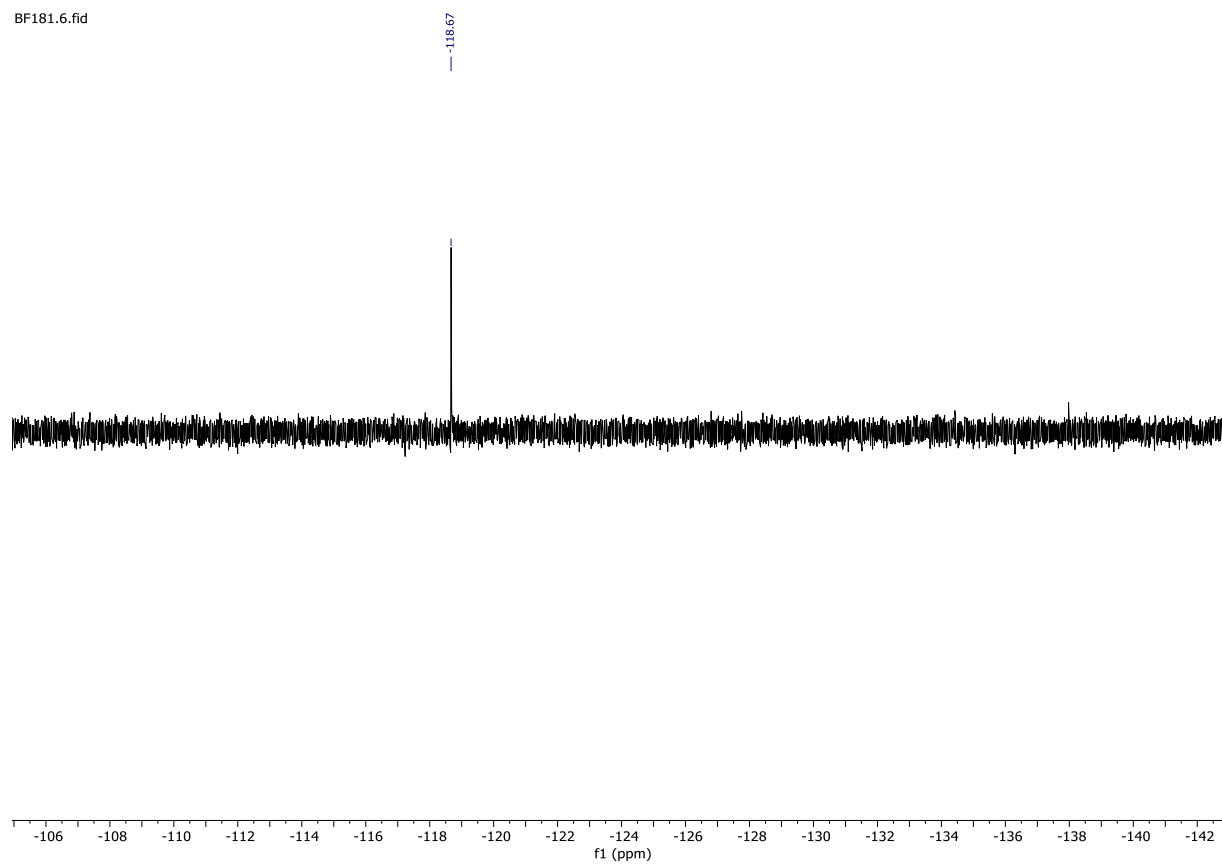
BF181.1.fid



BF181.2.fid

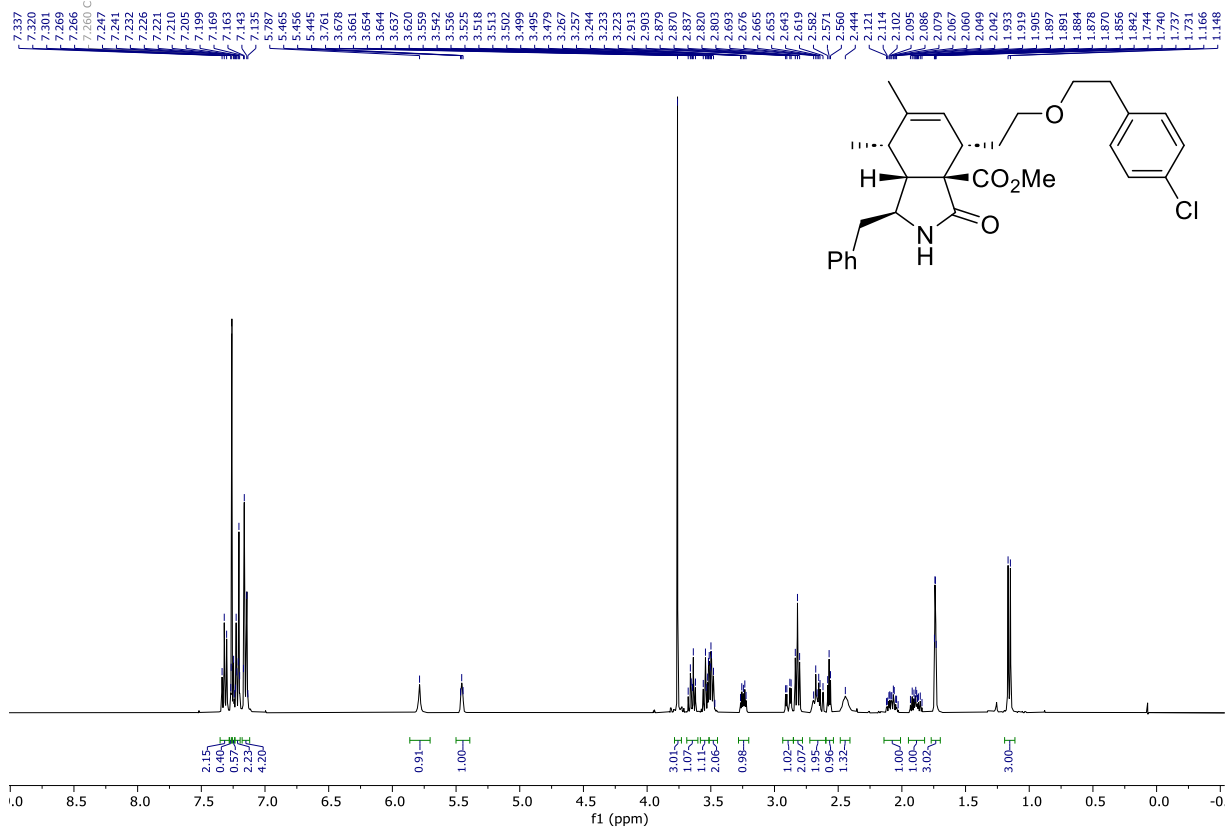


BF181.6.fid

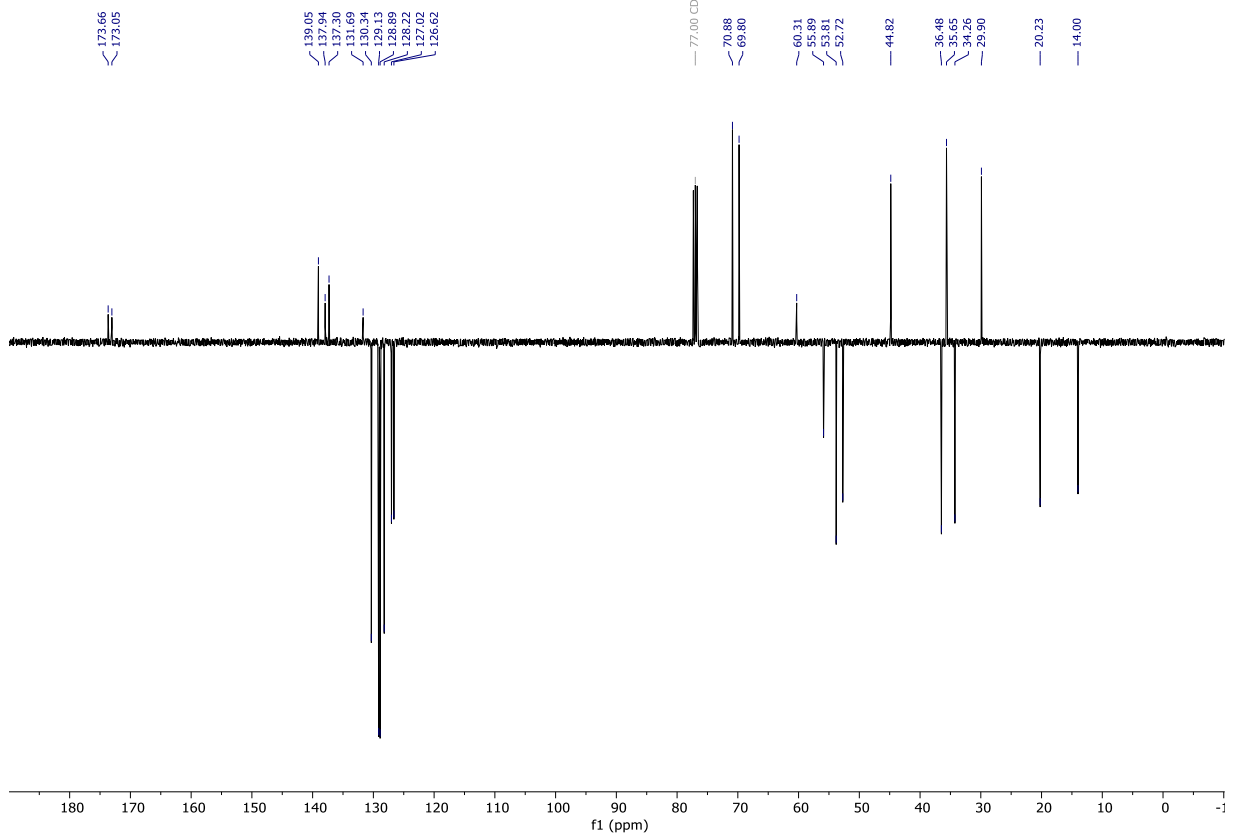


17g

BF-165.1.fid

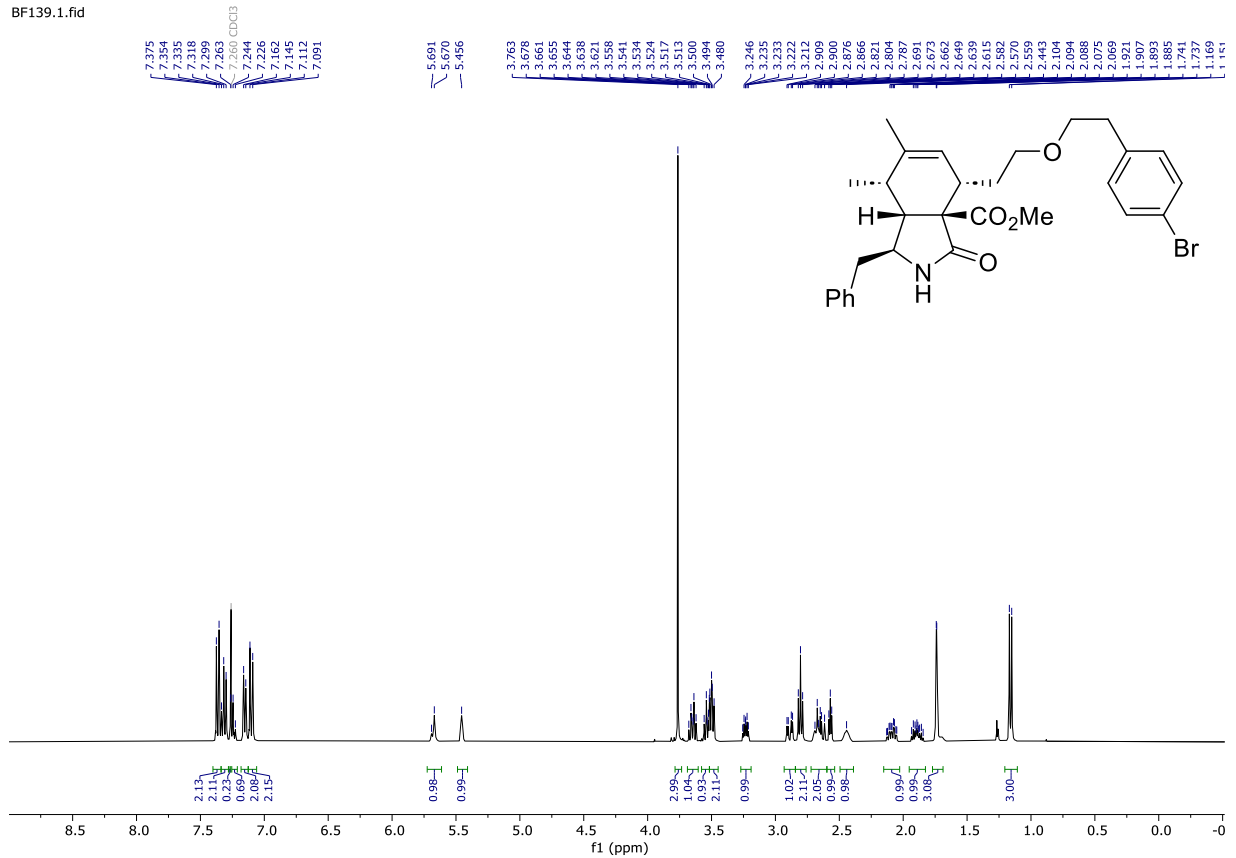


BF-165.3.fid

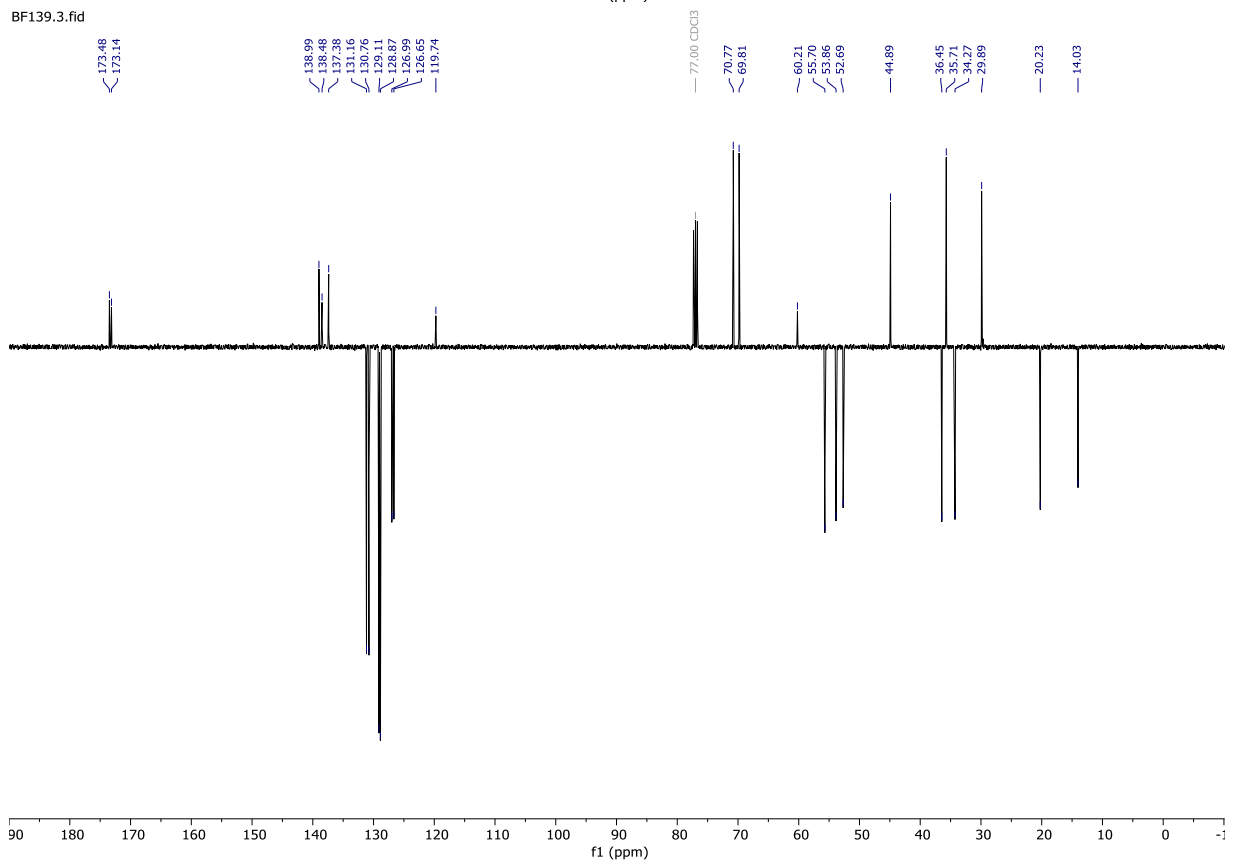


17h

BF139.1.fid

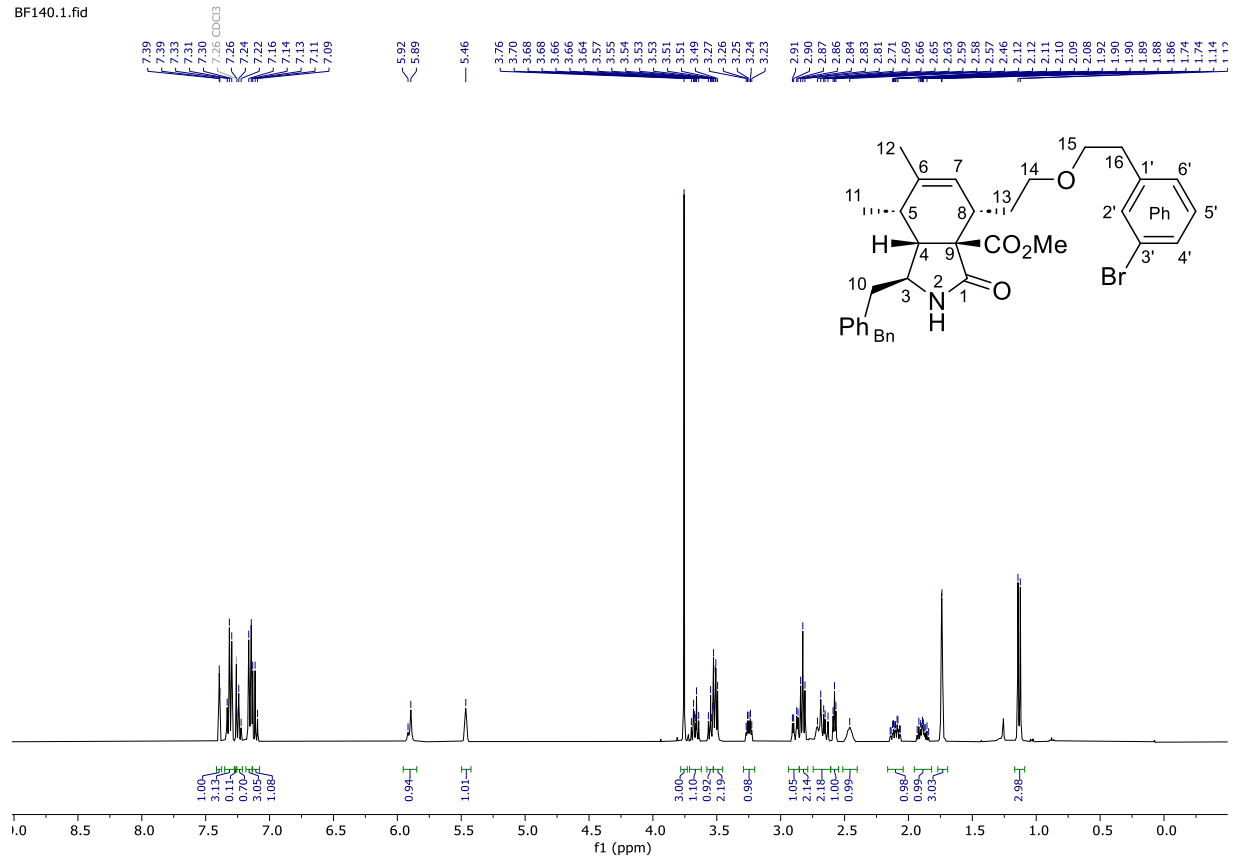


BF139.3.fid

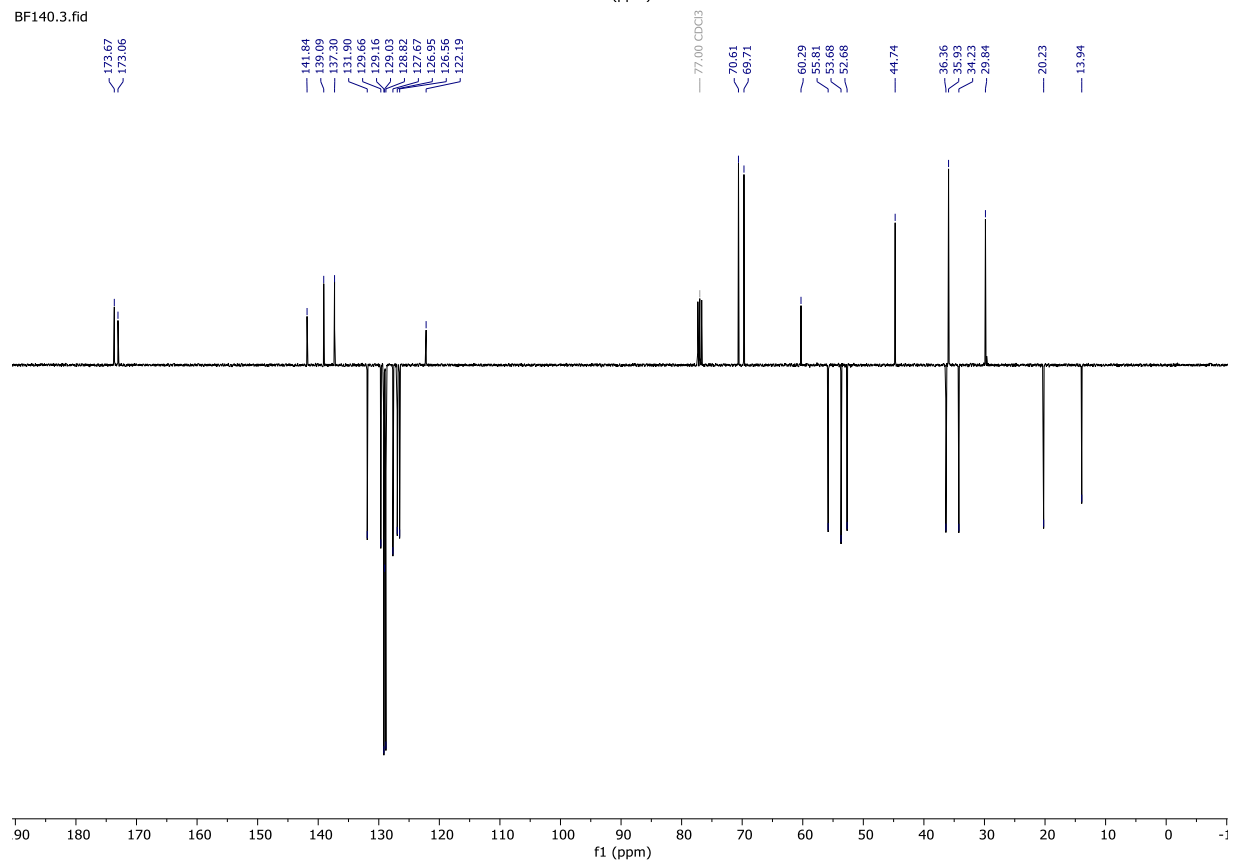


17i

BF140.1.fid

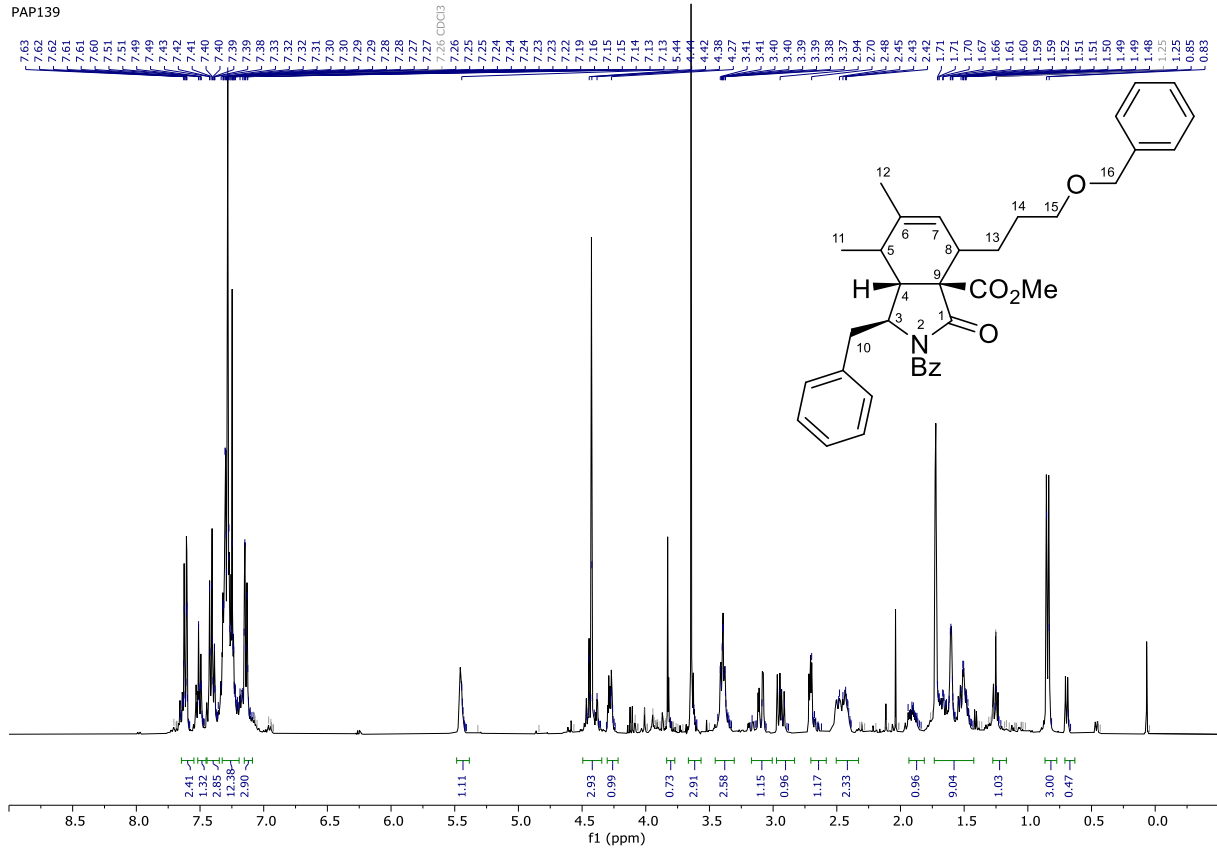


BF140.3.fid



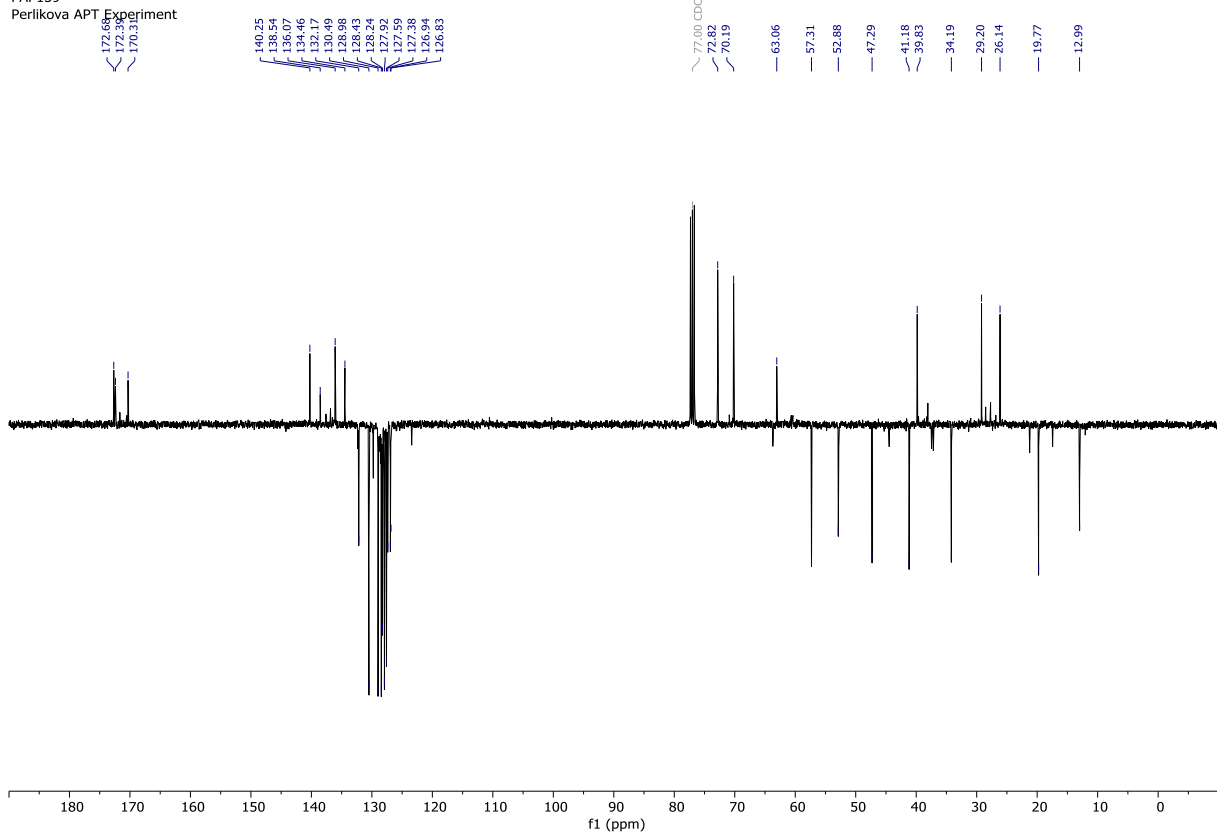
19

PAP139



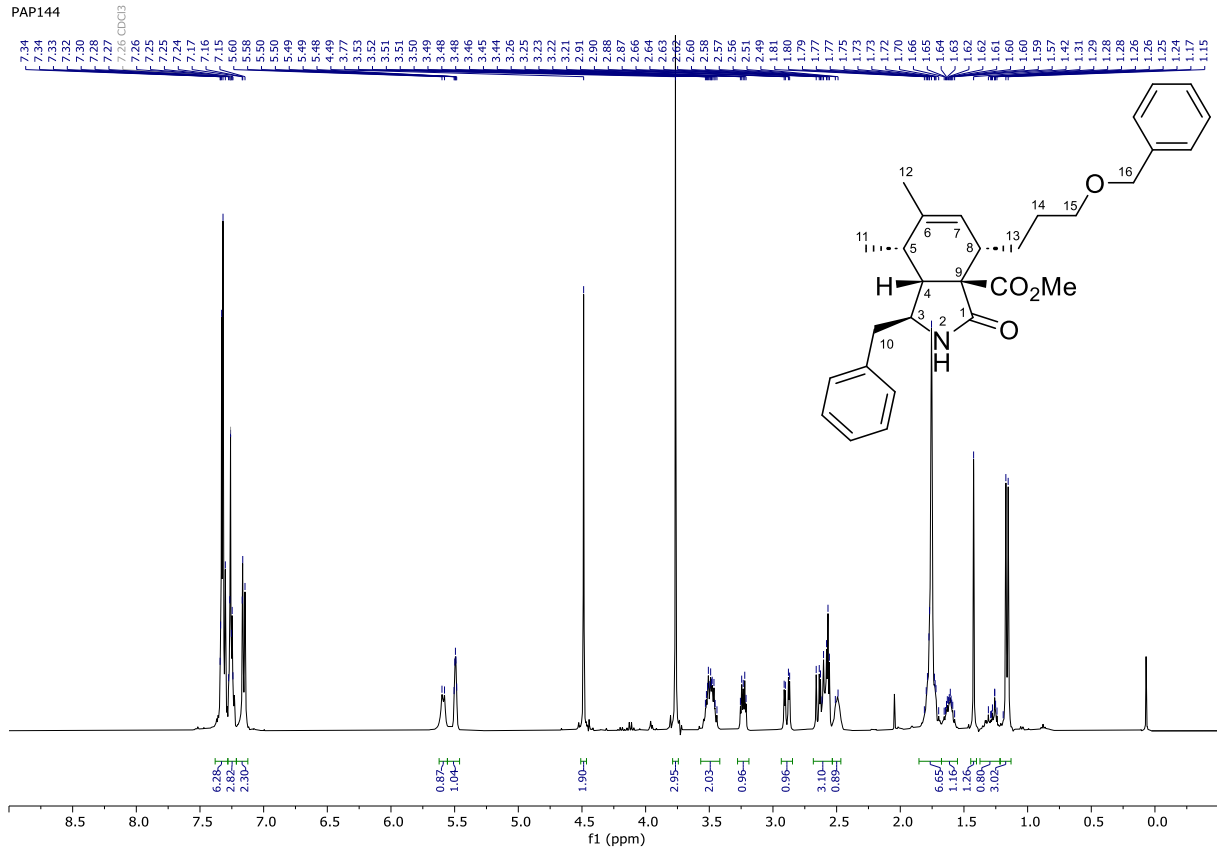
PAP139

Perlikova APT Experiment

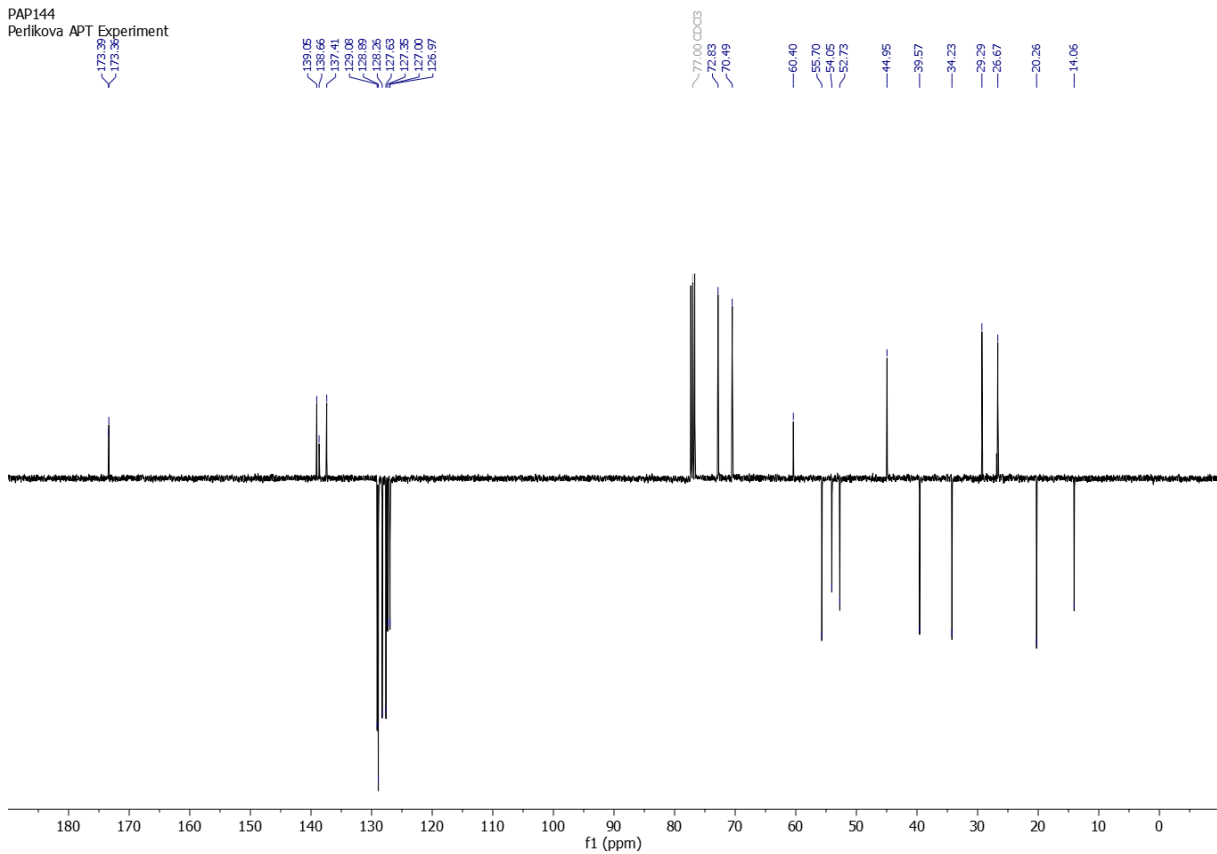


20

PAP144

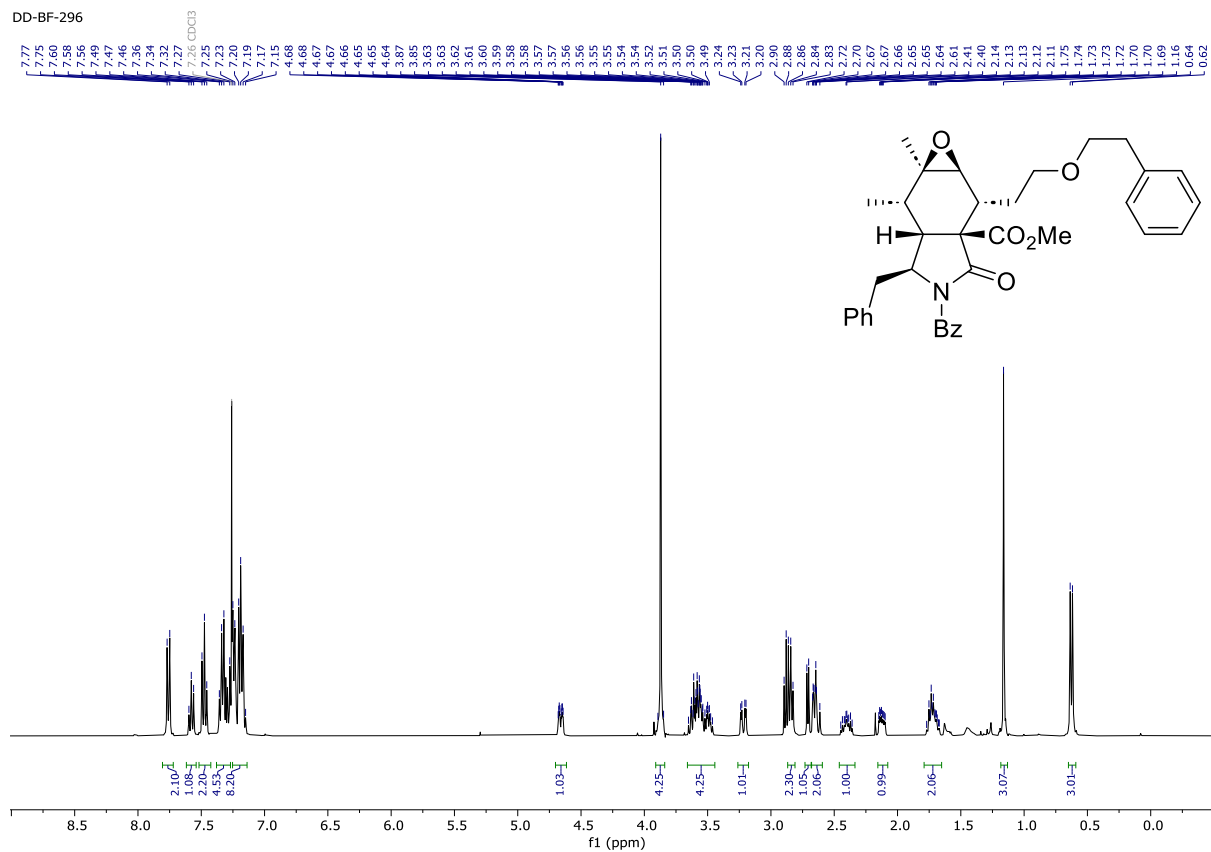


PAP144
Perikova APT Experiment



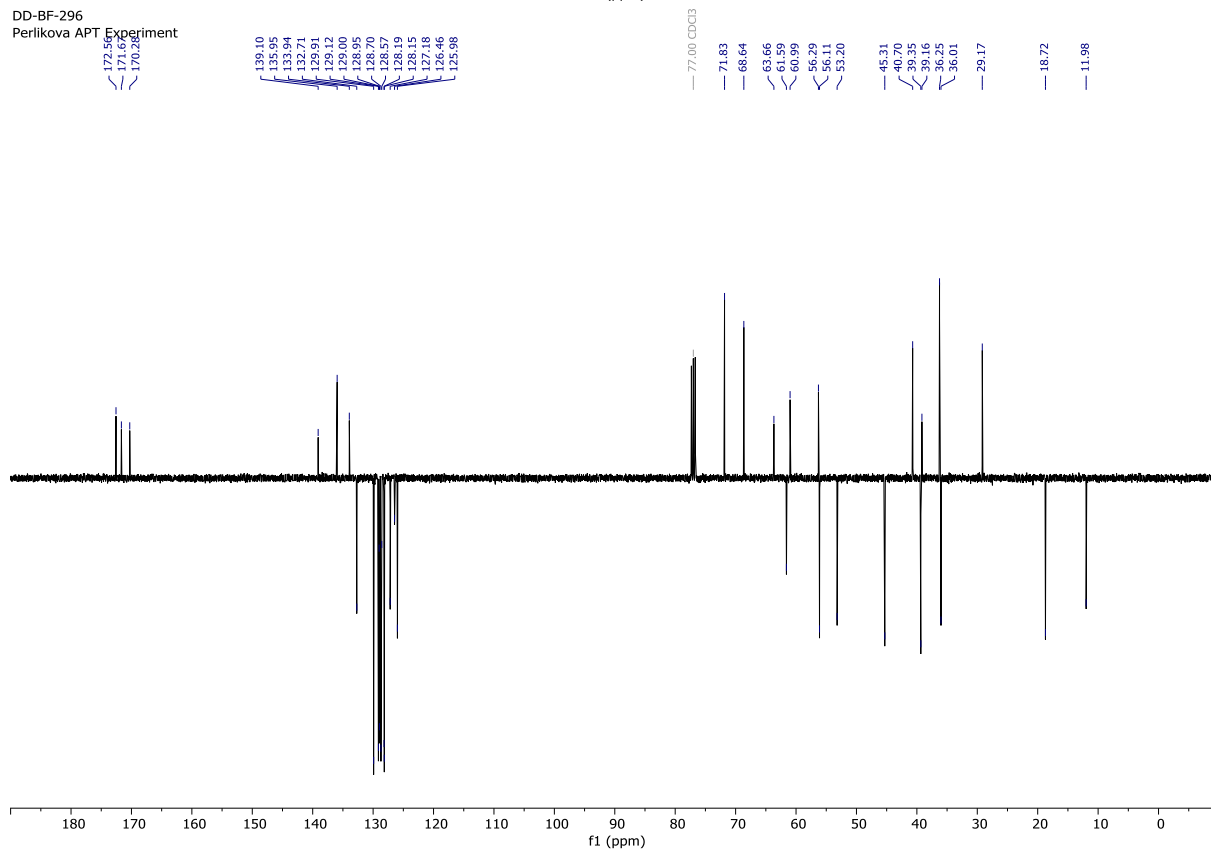
21a

DD-BF-296



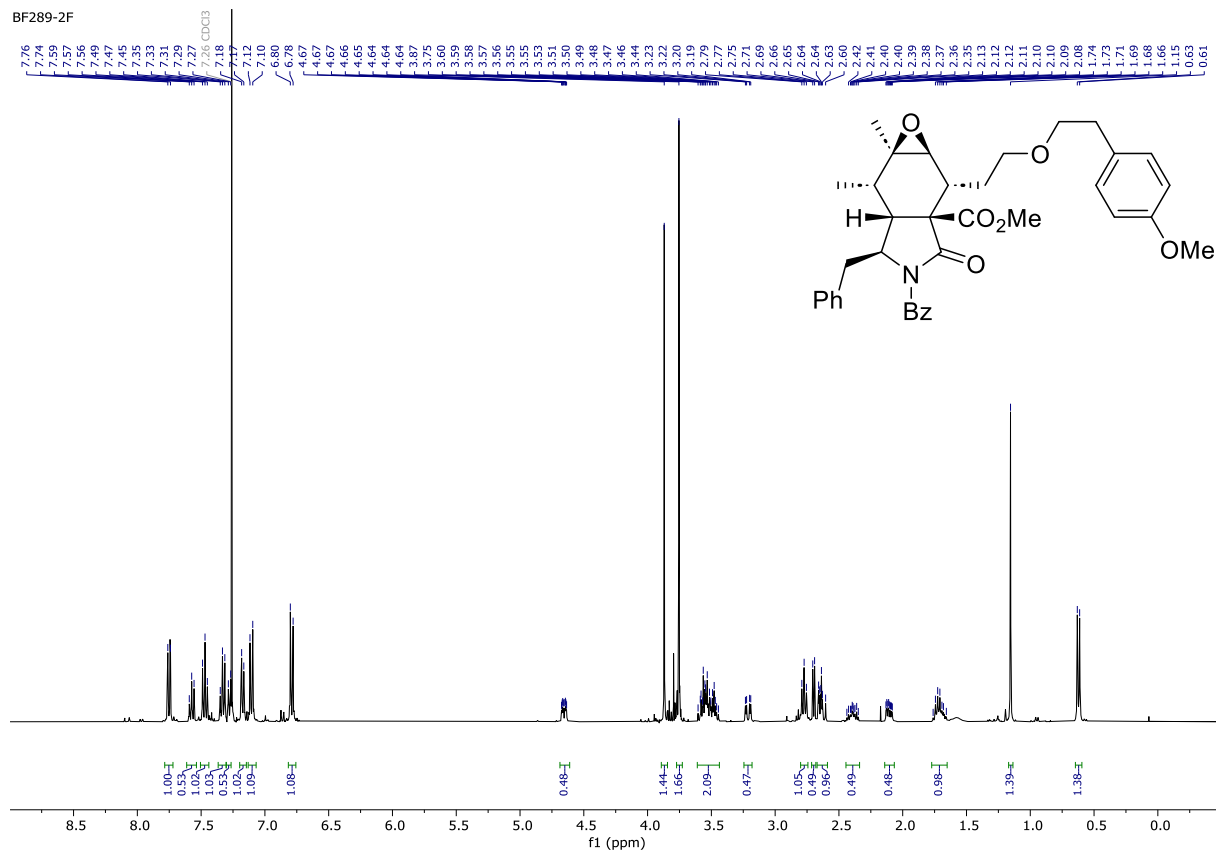
DD-BF-296

Perlikova APT Experiment



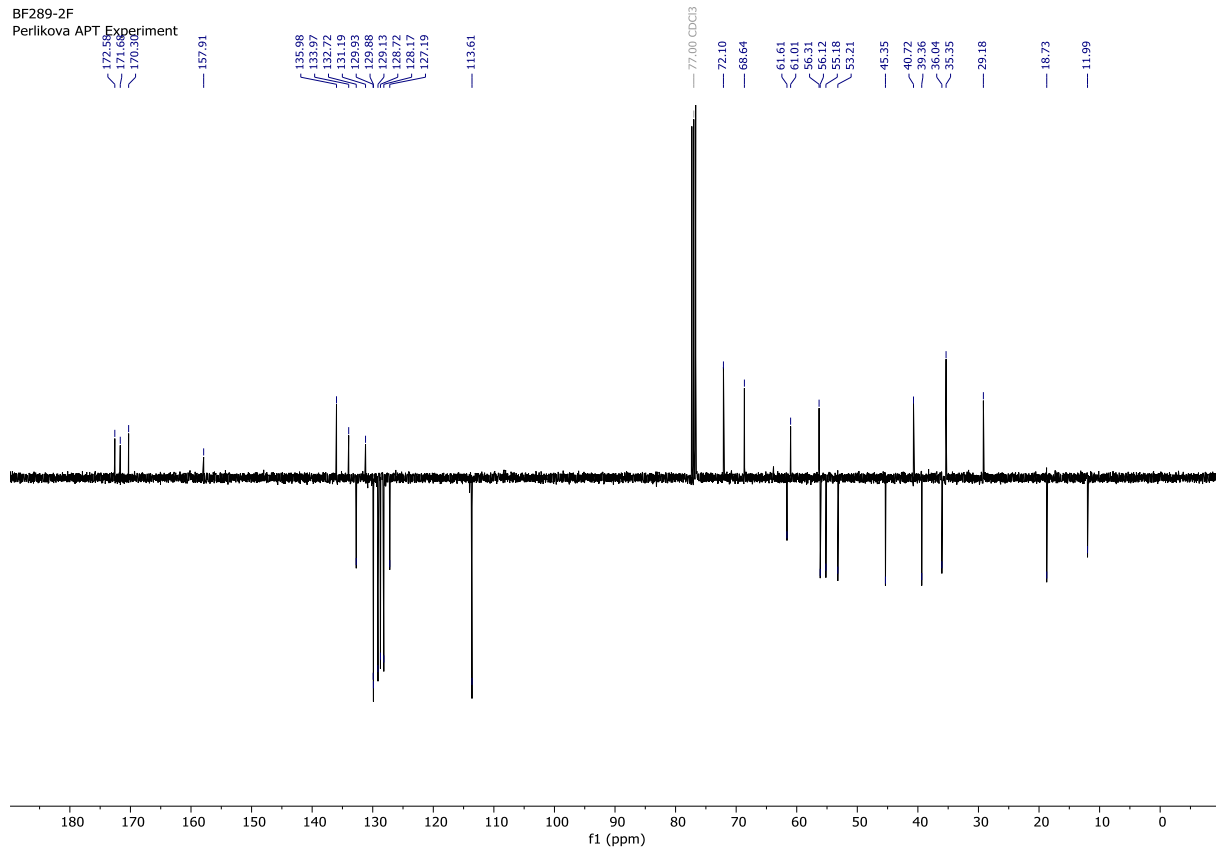
21b

BF289-2F



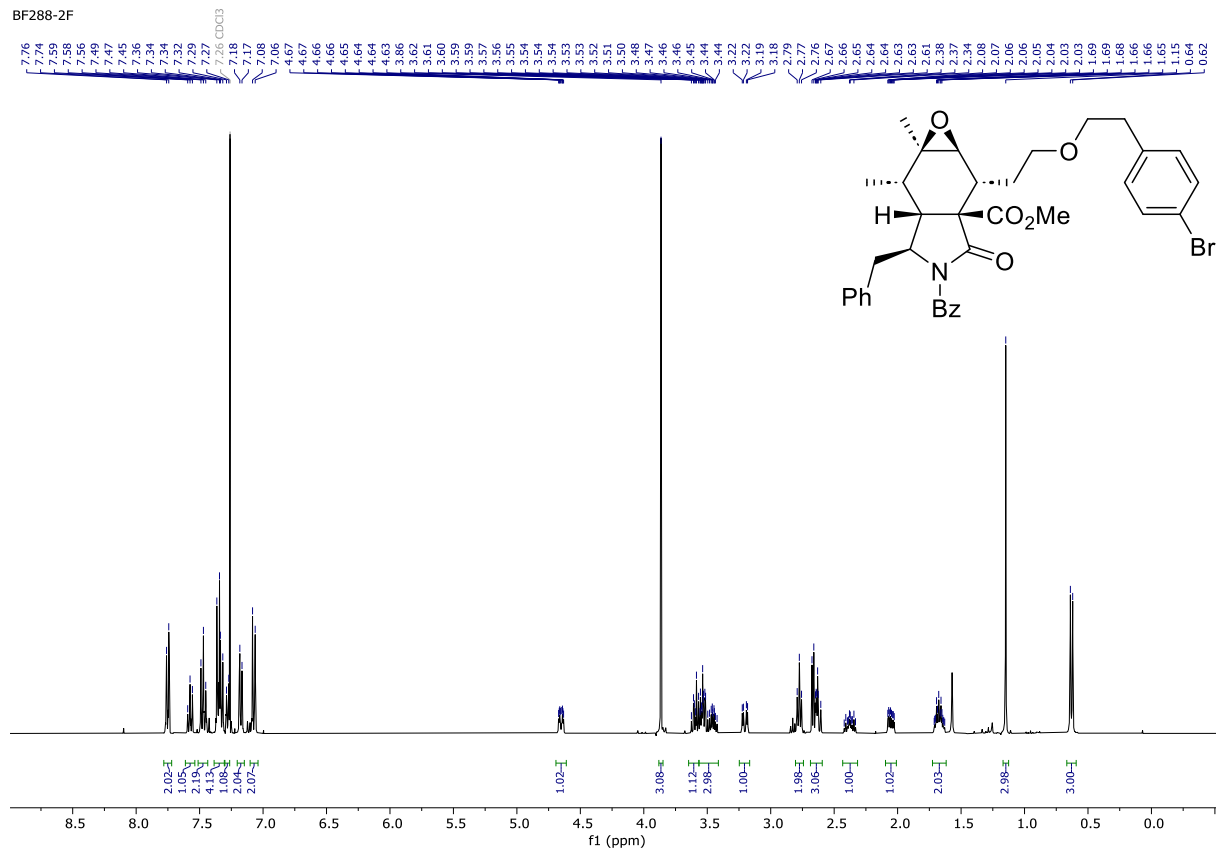
BF289-2F

Perlikova APT Experiment



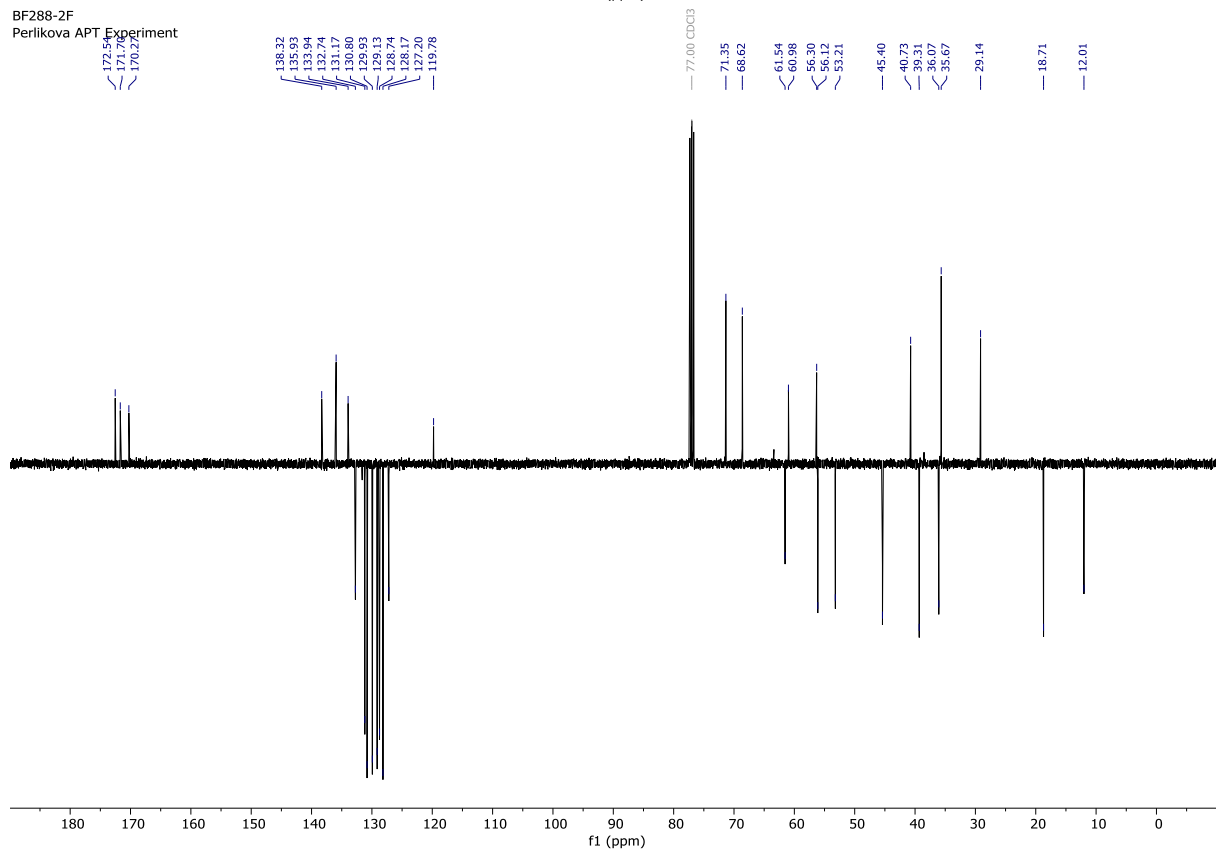
21h

BF288-2F



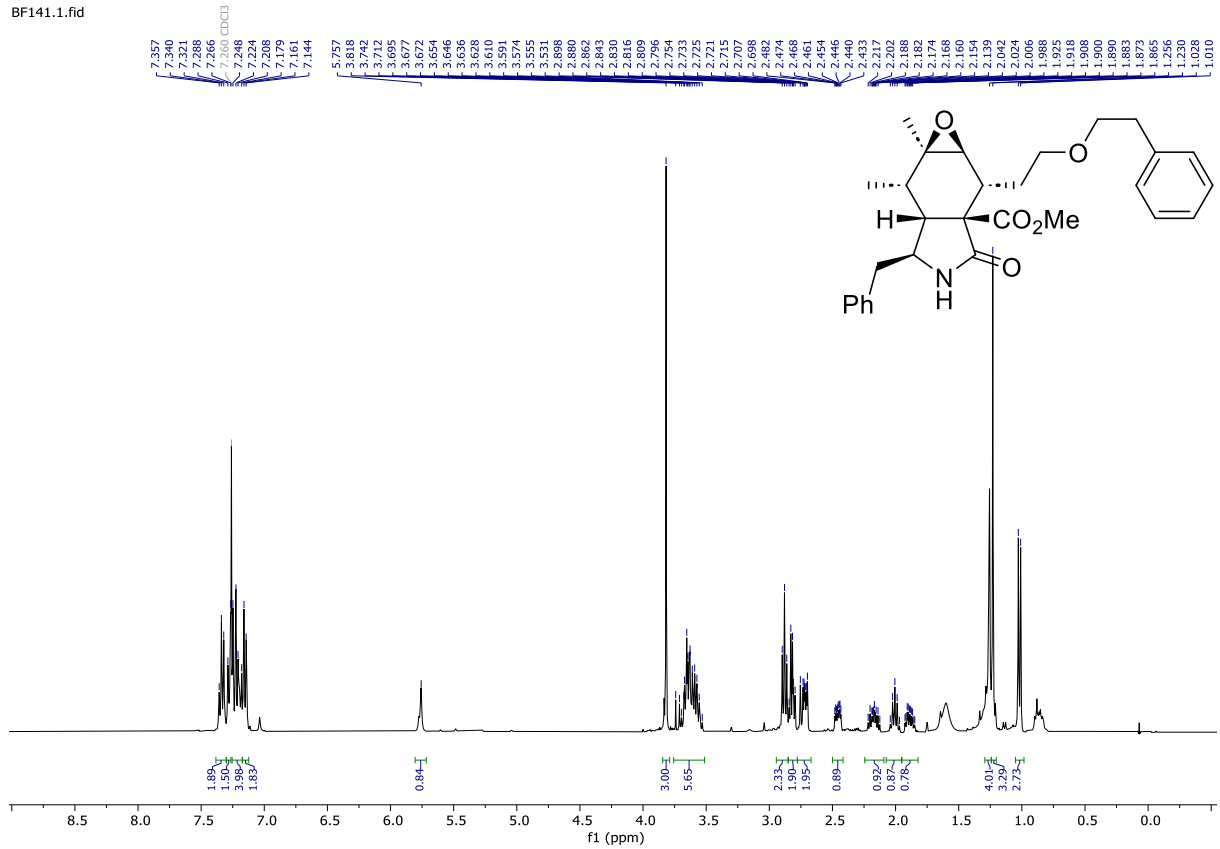
BF288-2F

Perlikova APT Experiment

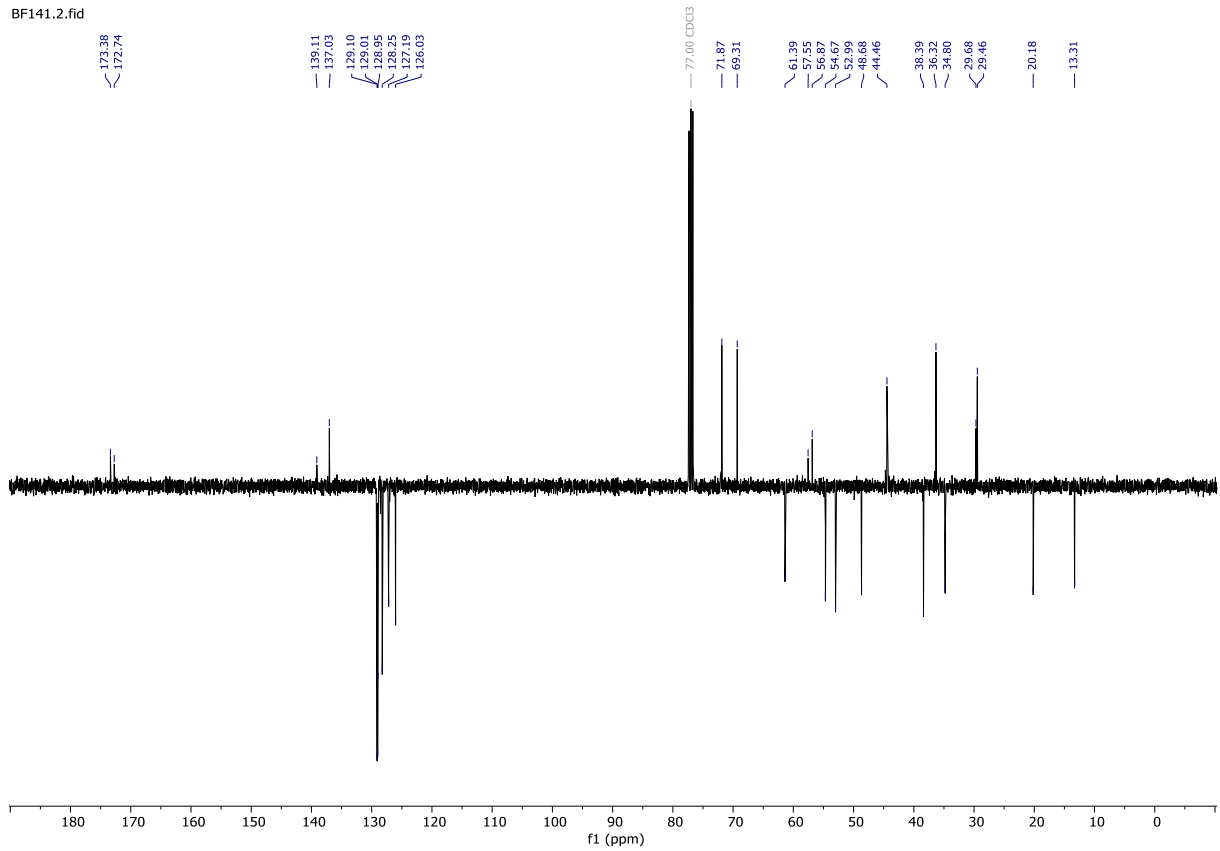


22a

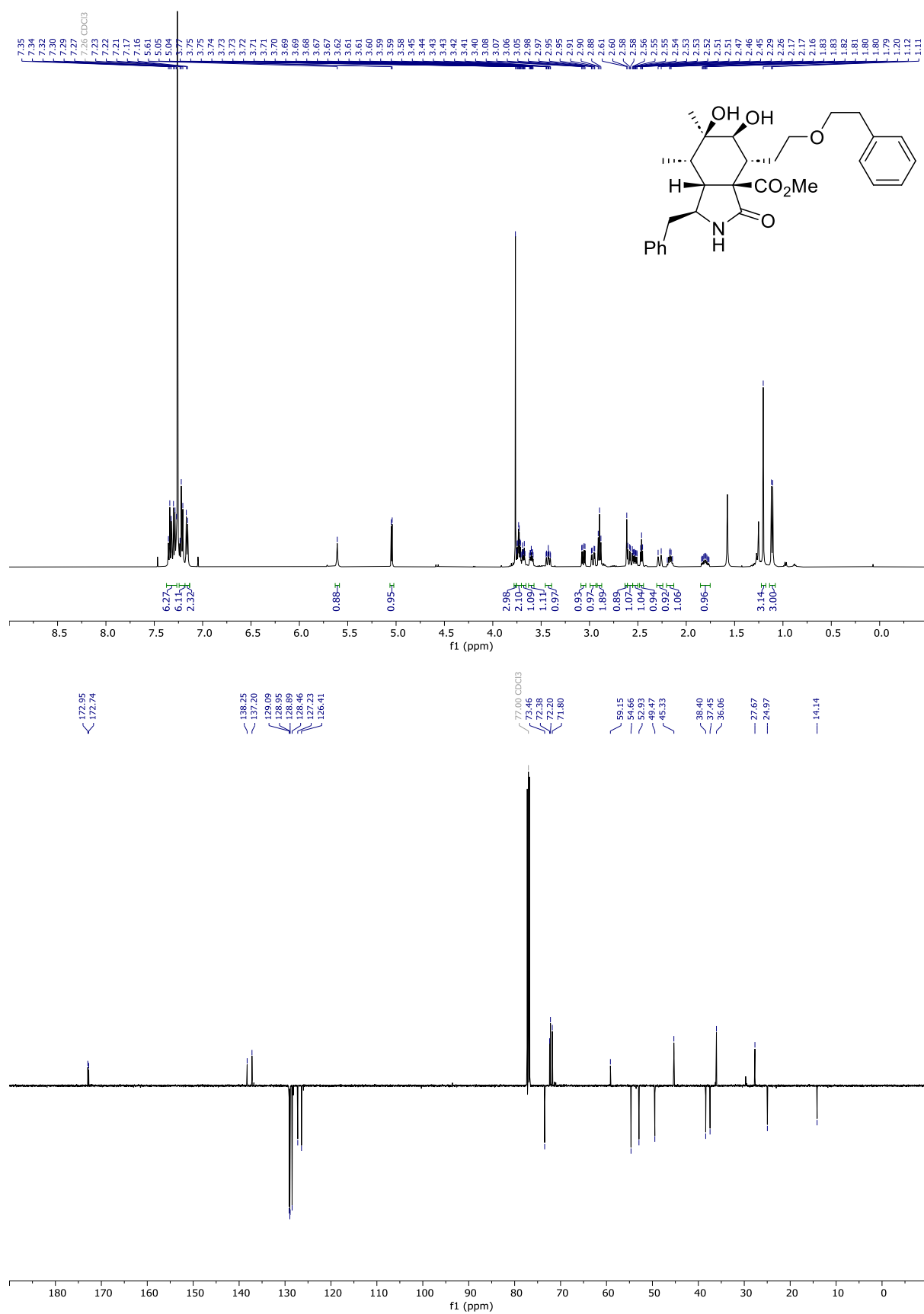
BF141.1.fid



BF141.2.fid



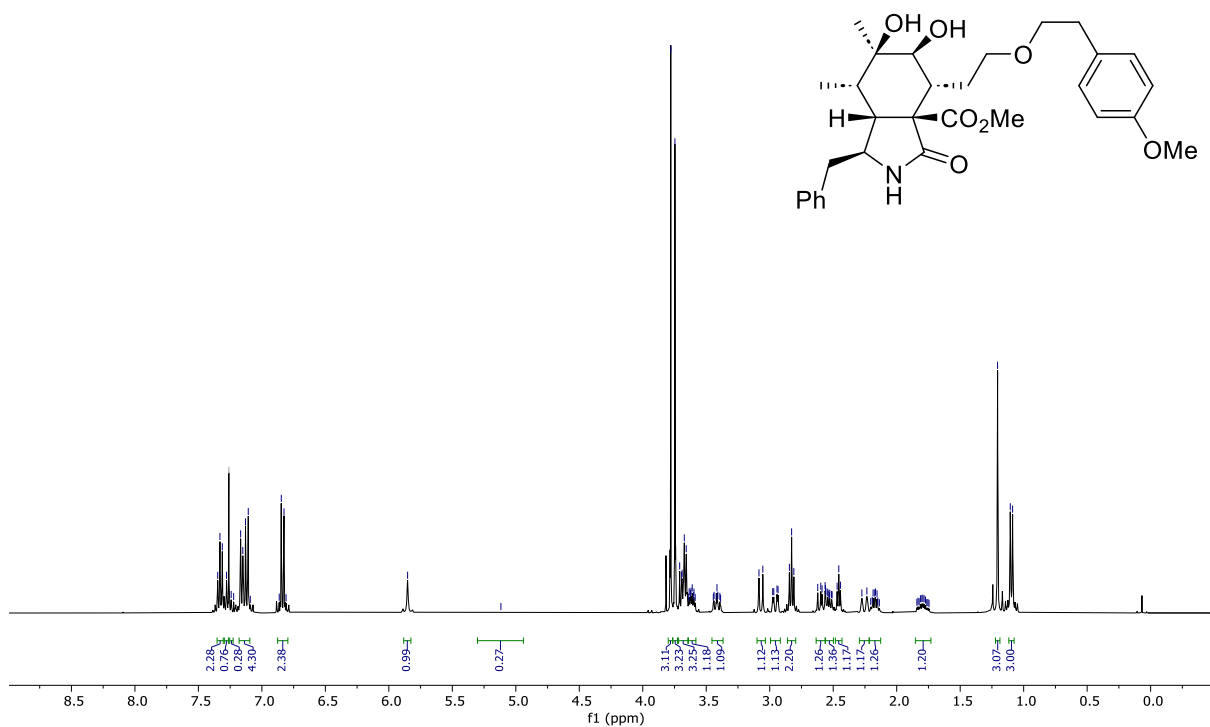
23a



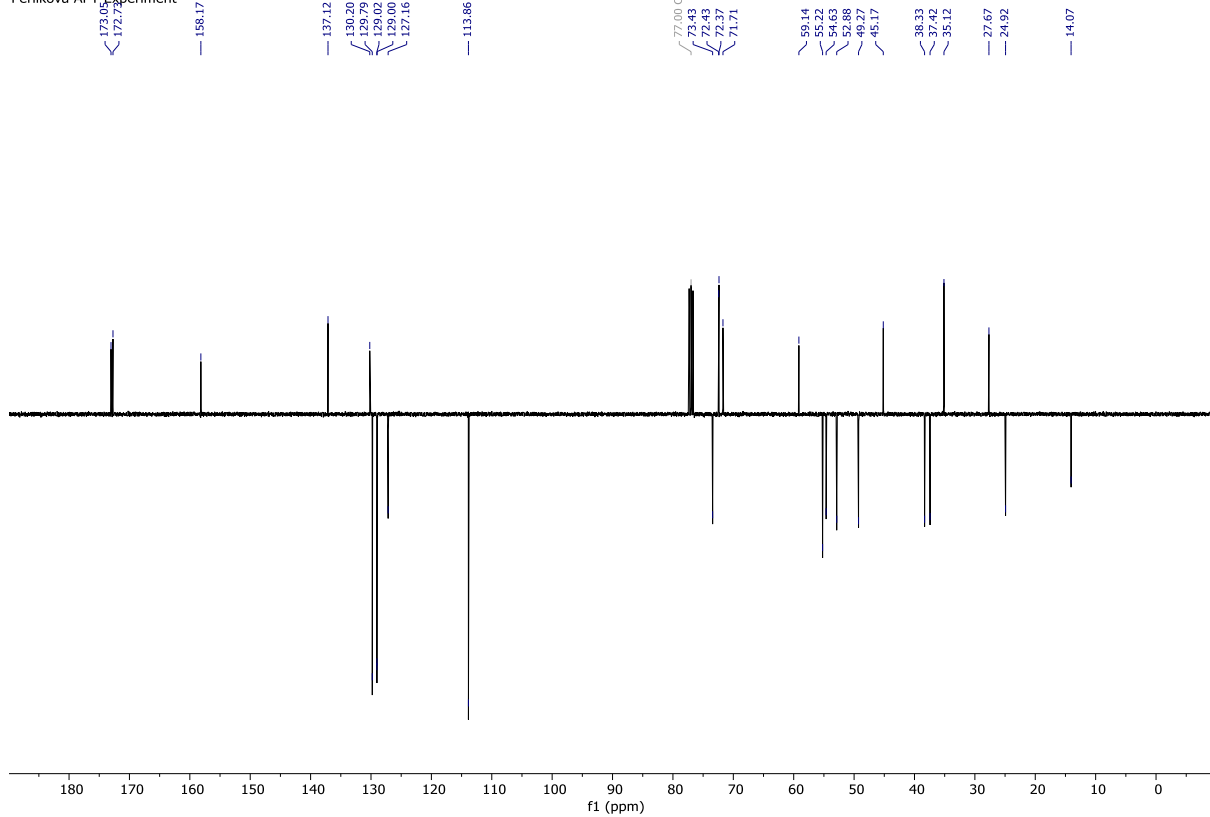
23b

BF291

7.35
7.33
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7.26 CDCl₃
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1.75
1.11
1.09

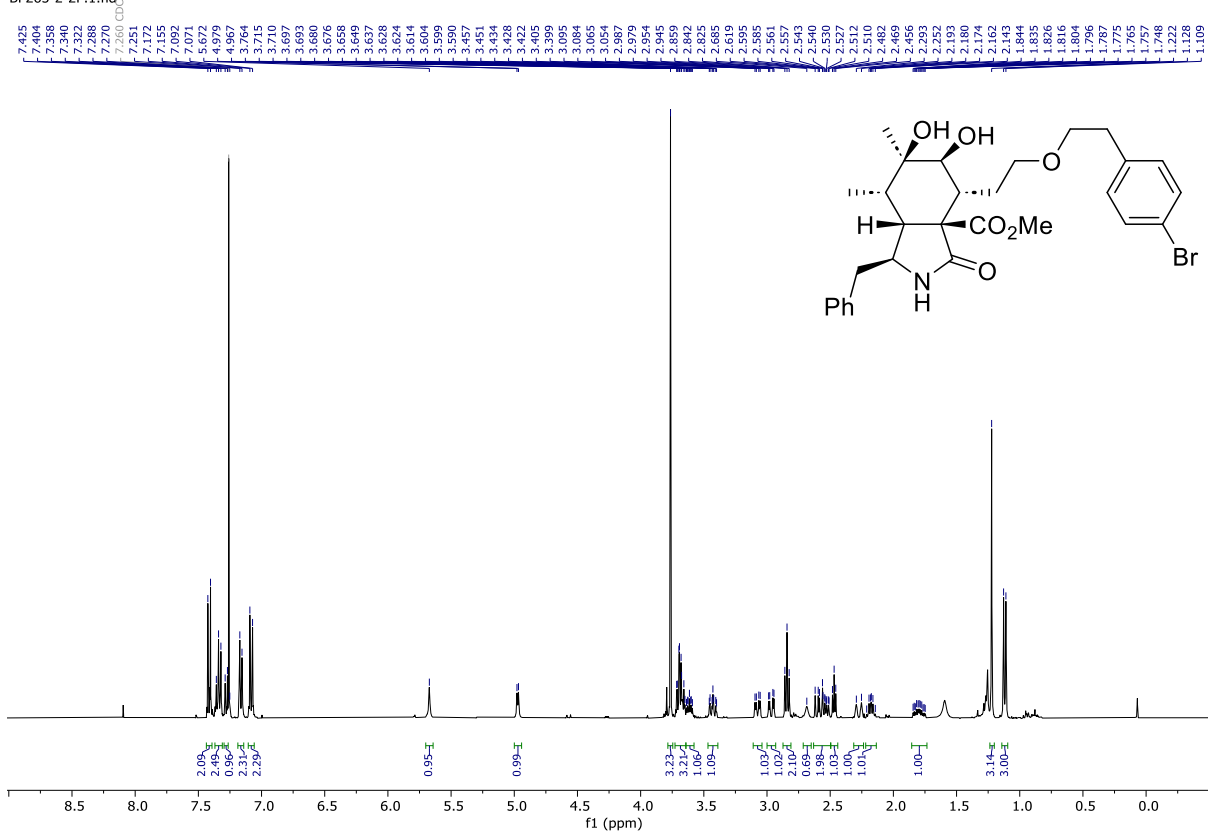


BF291
Perlikova APT Experiment

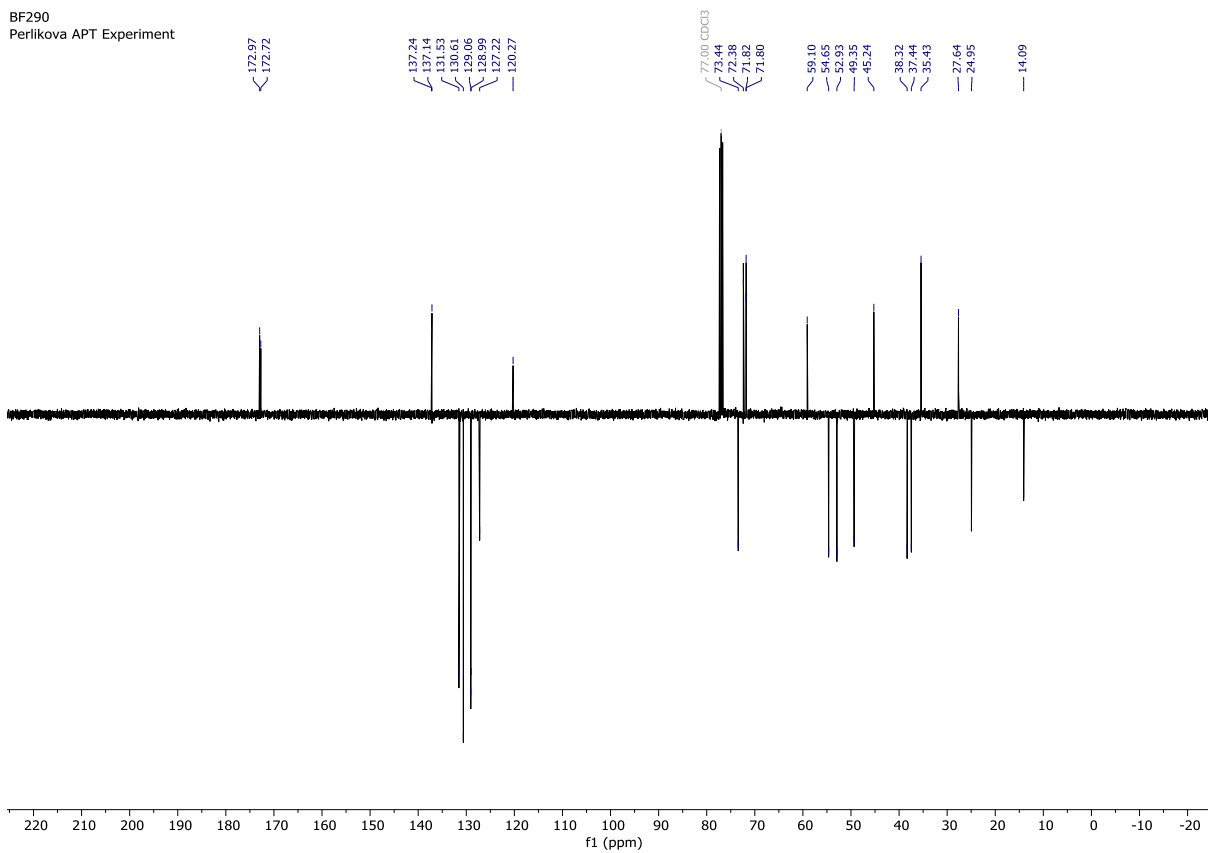


23h

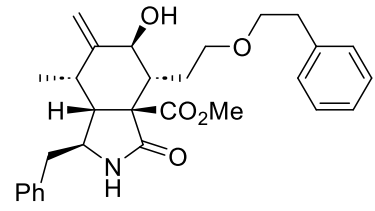
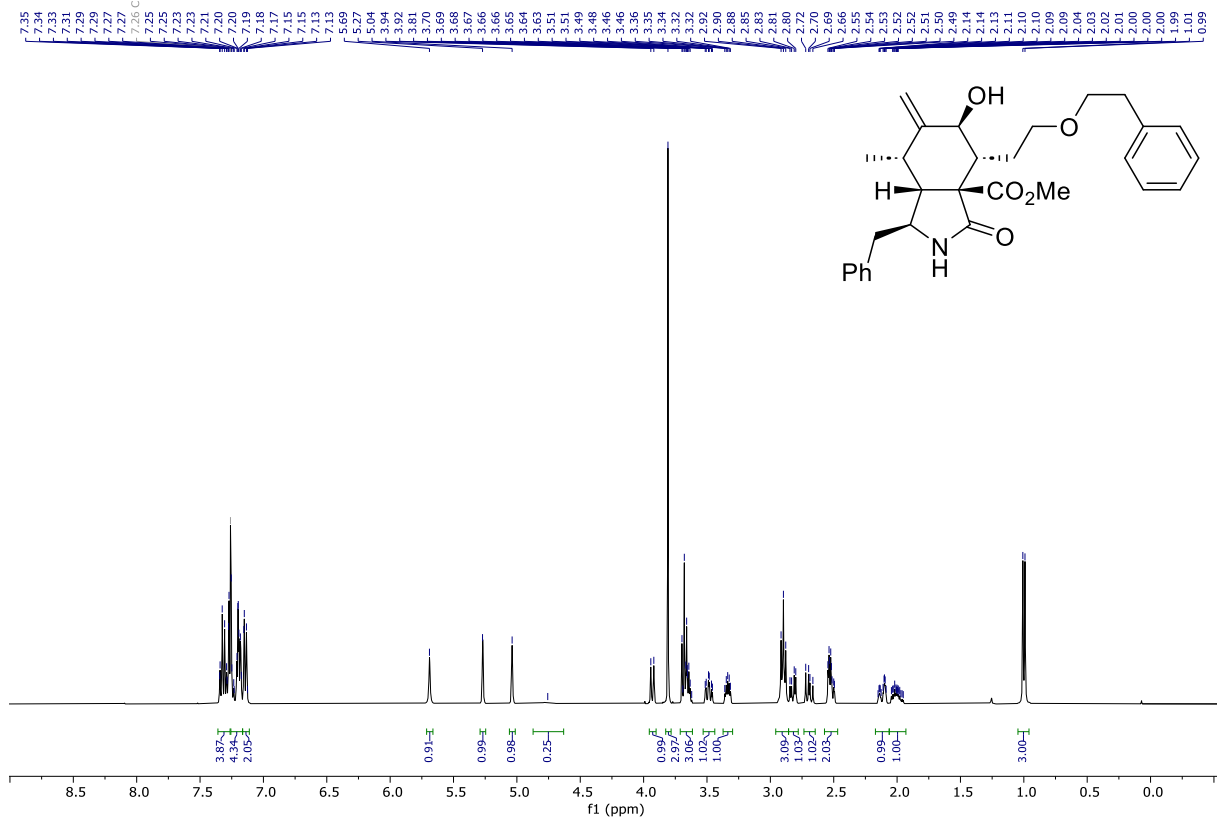
BF265-2-2F.1.fid



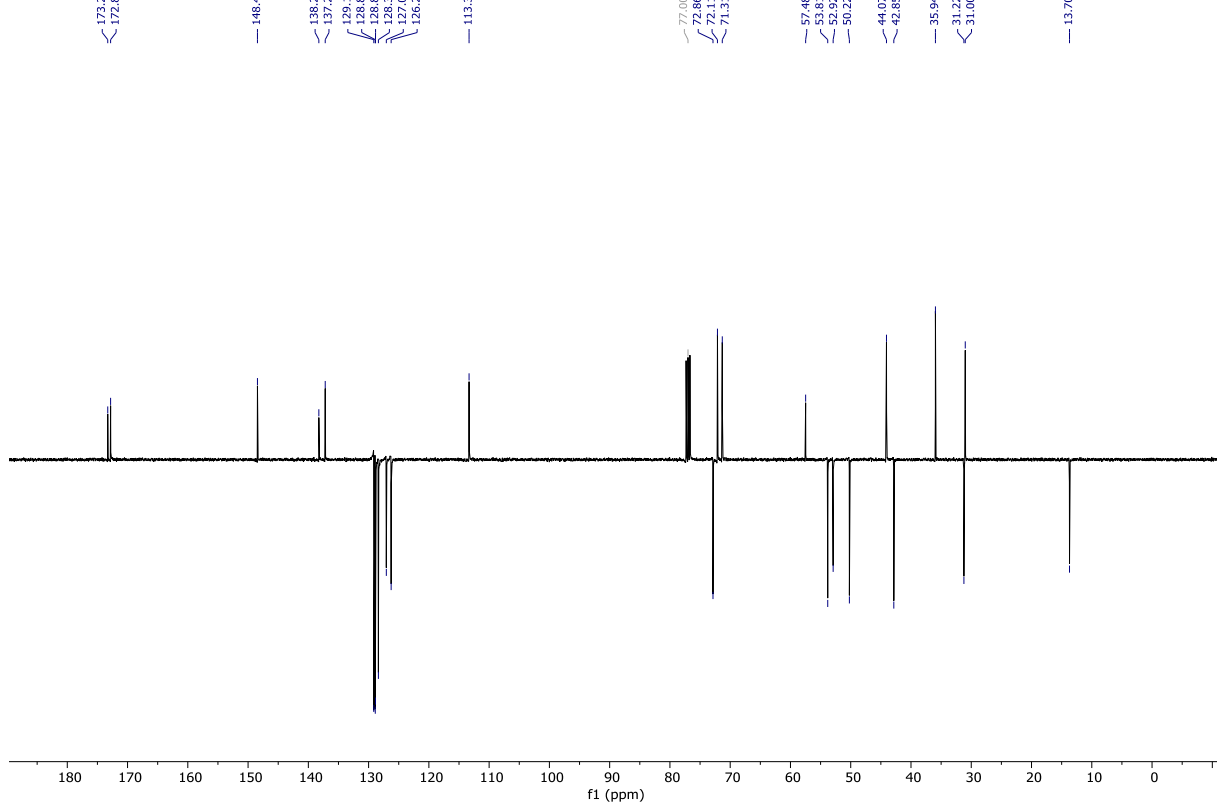
BF290
Perlikova APT Experiment



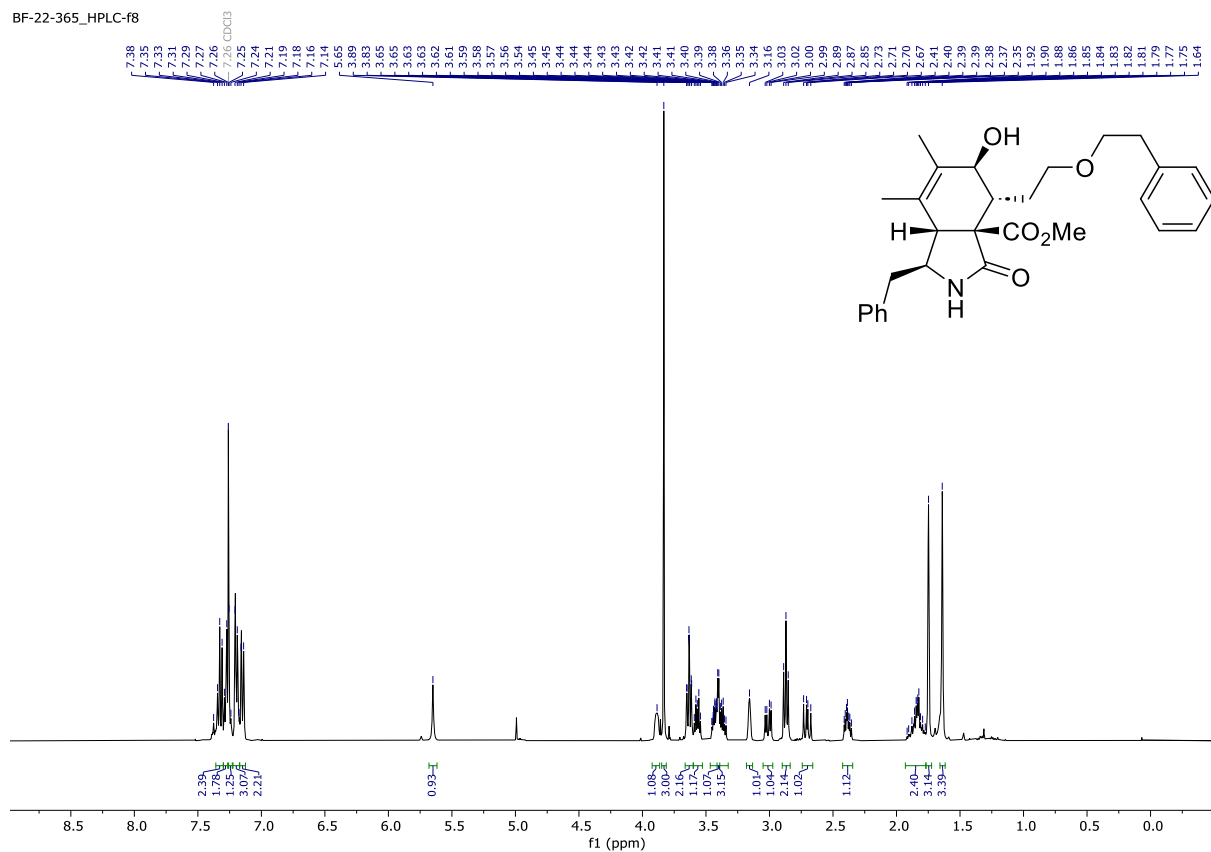
BF-299-HPLC-910



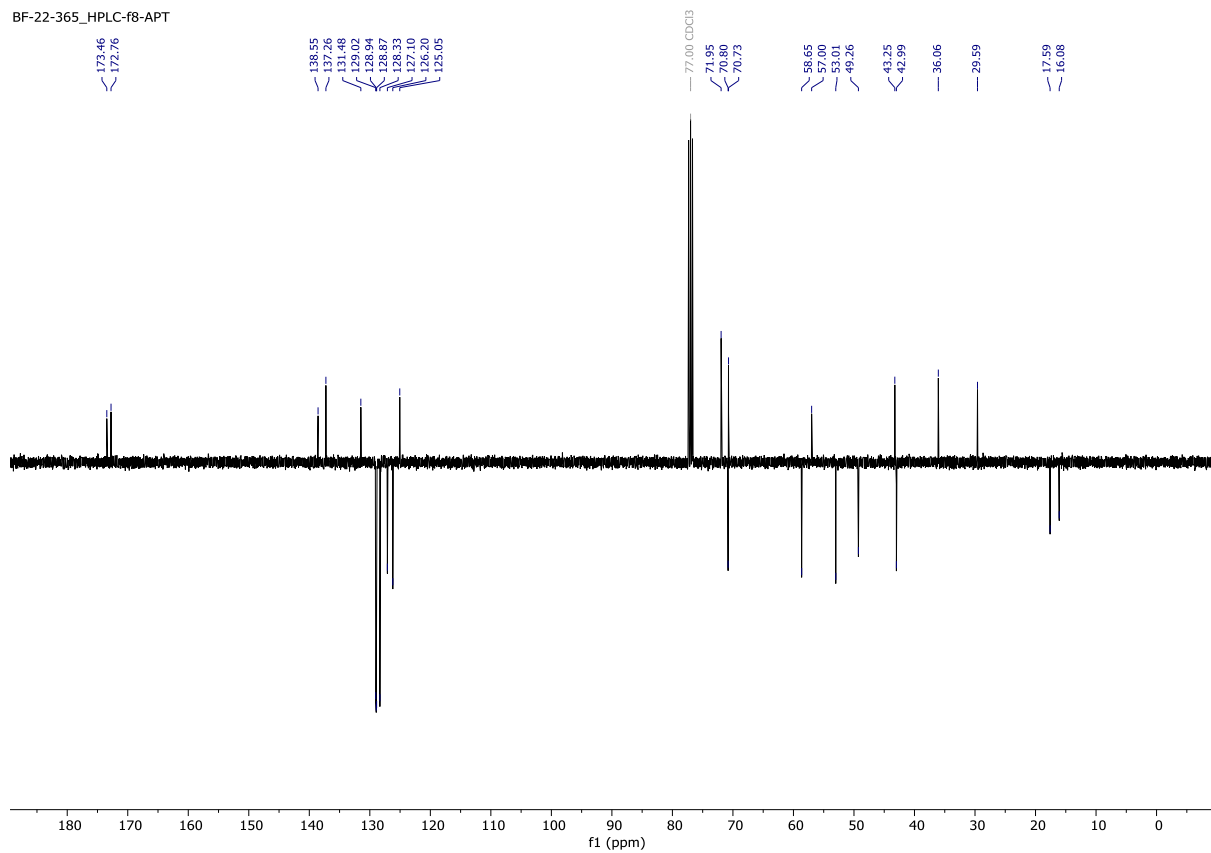
BF-299-HPLC-910
Perlikova APT Experiment

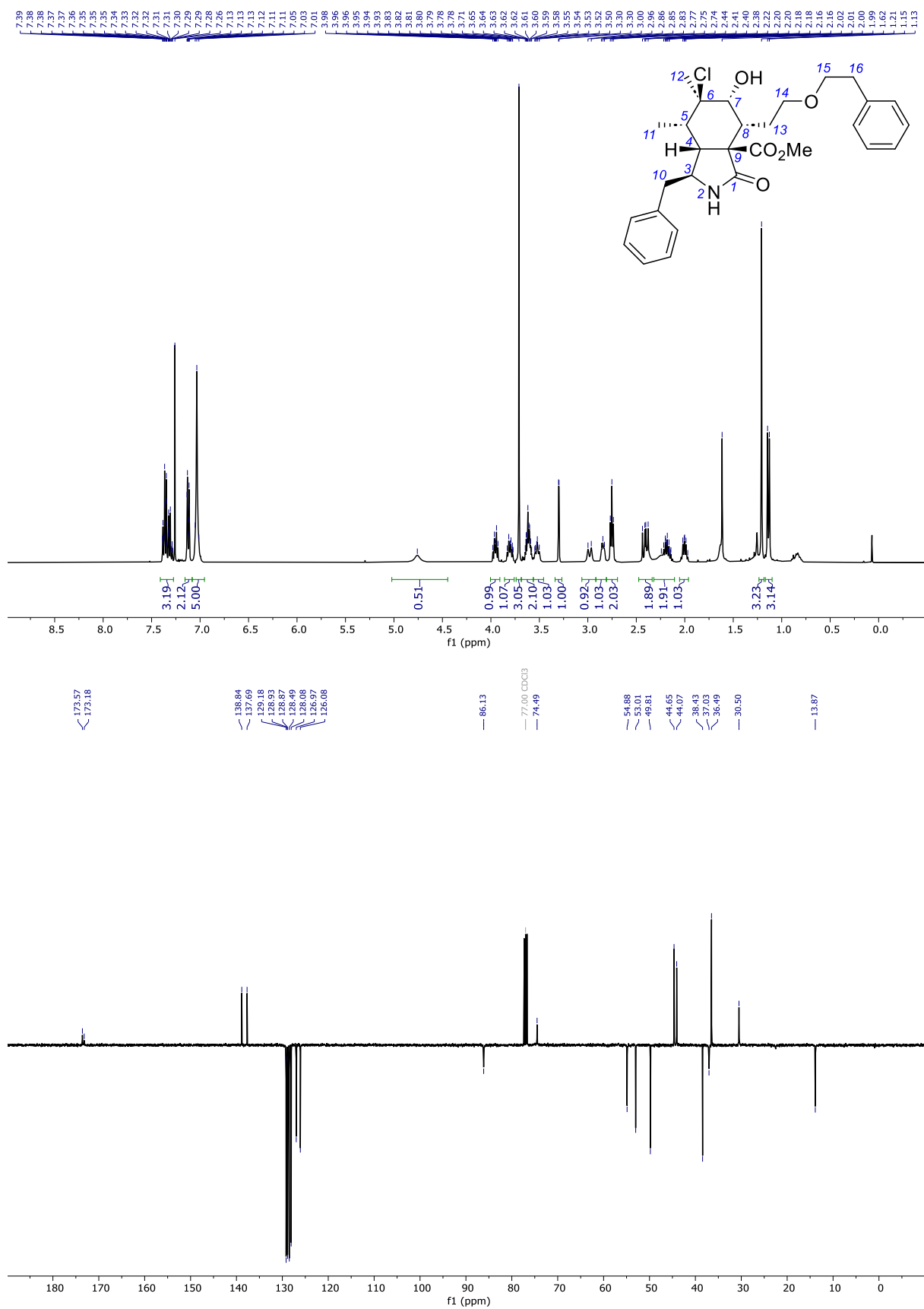


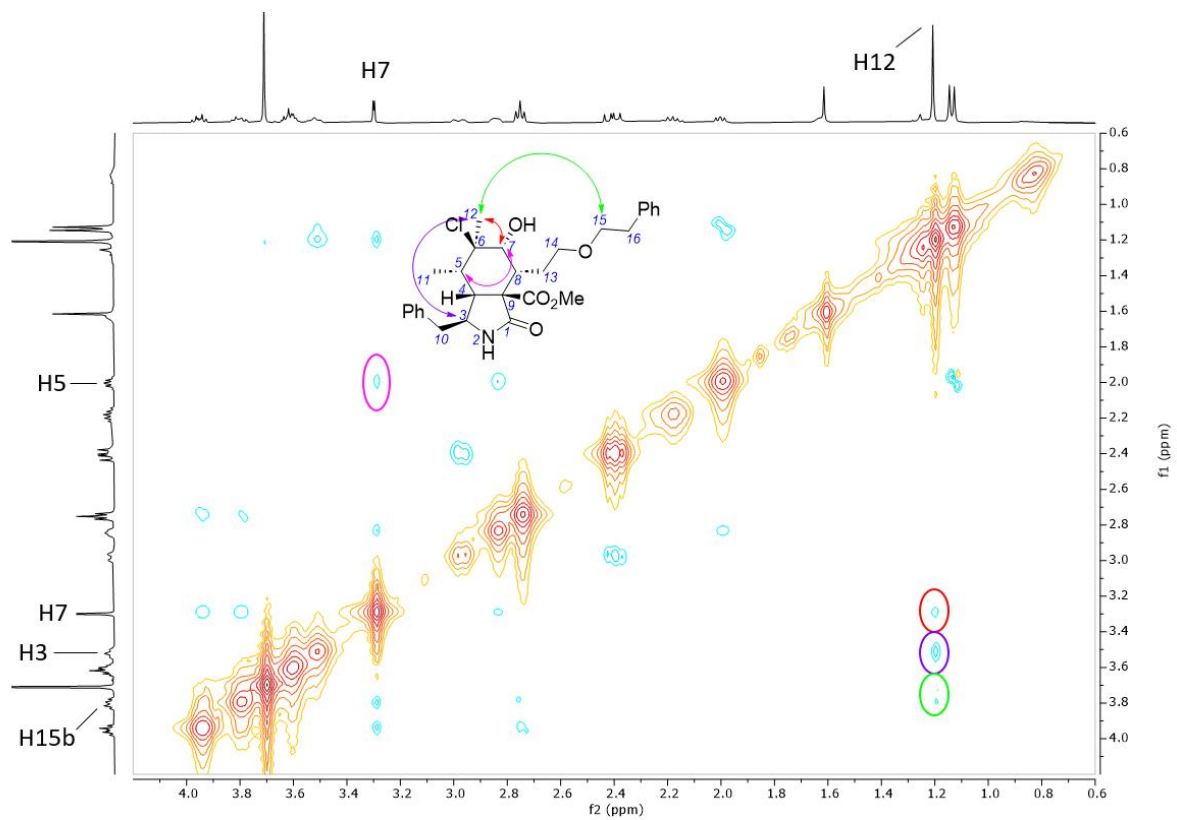
BF-22-365_HPLC-f8



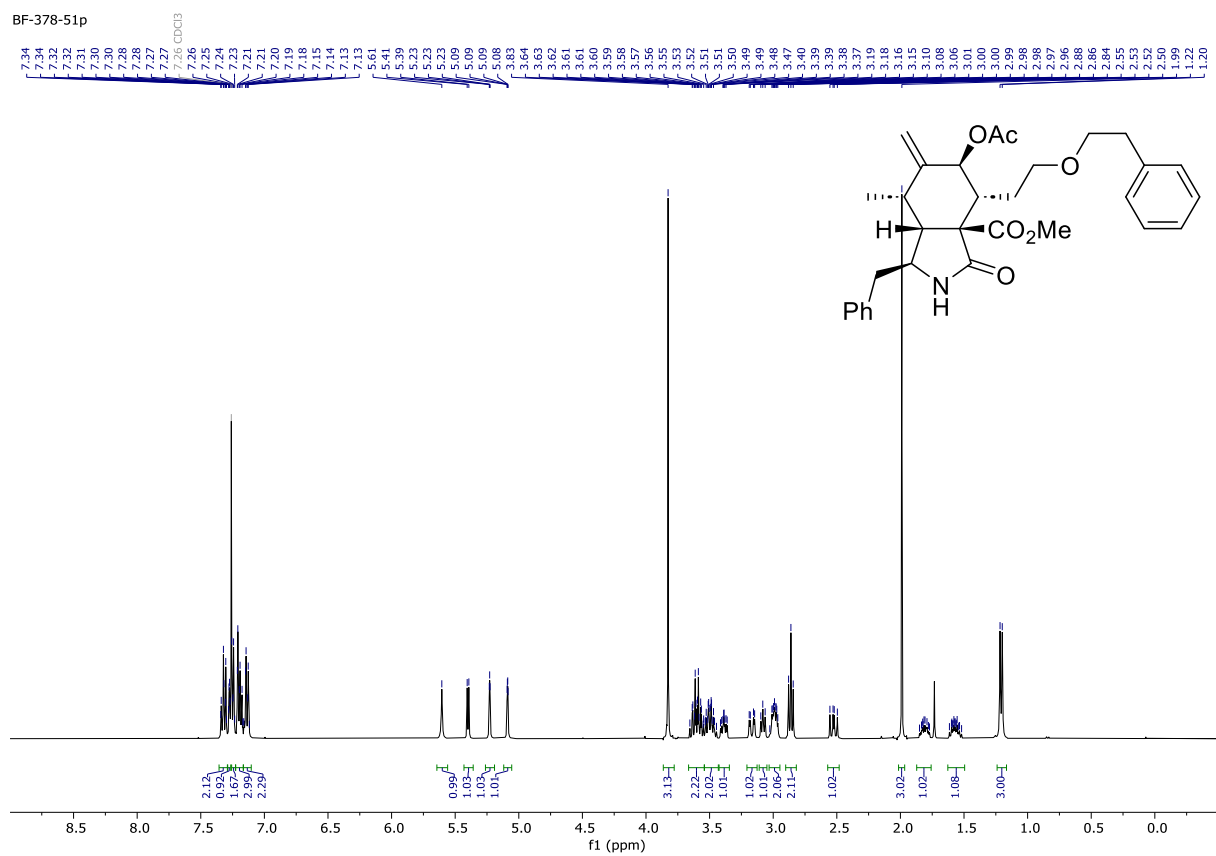
BF-22-365_HPLC-f8-APT





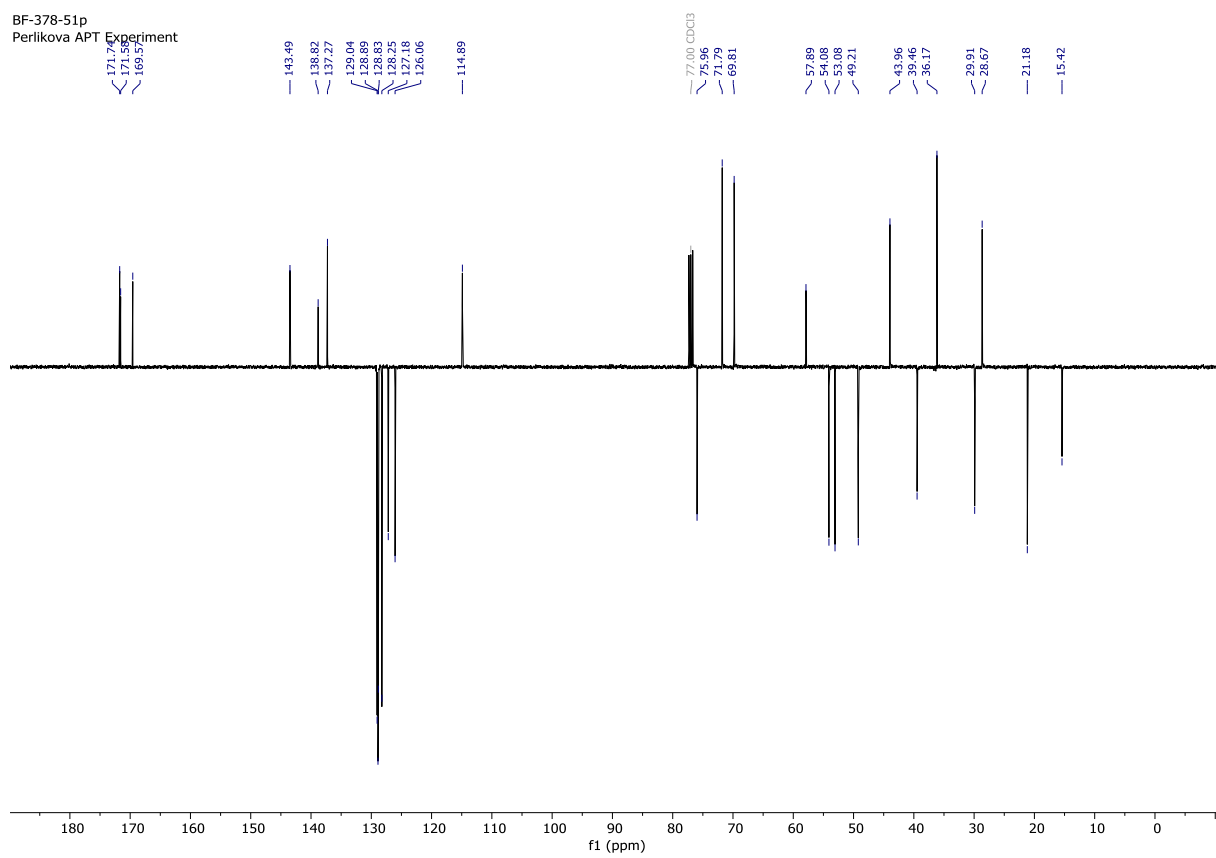


BF-378-51p

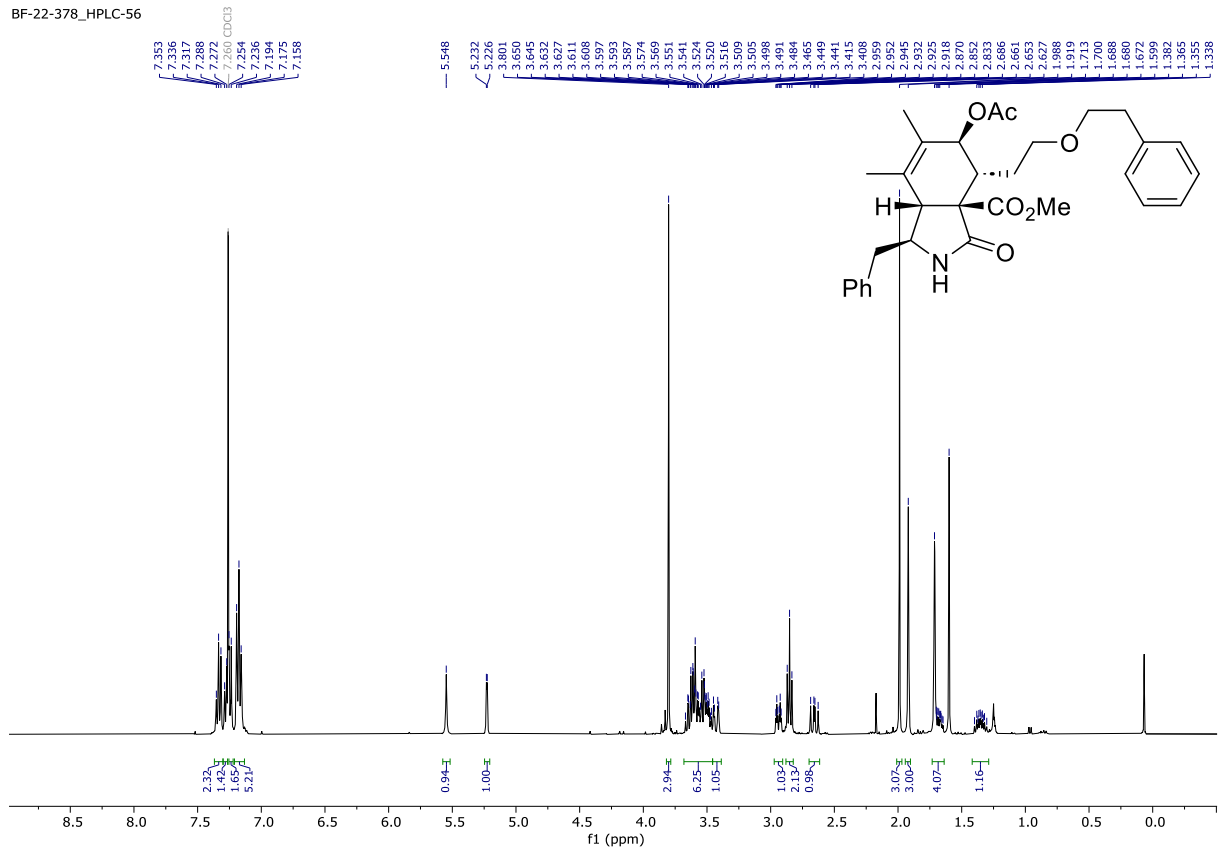


BF-378-51p

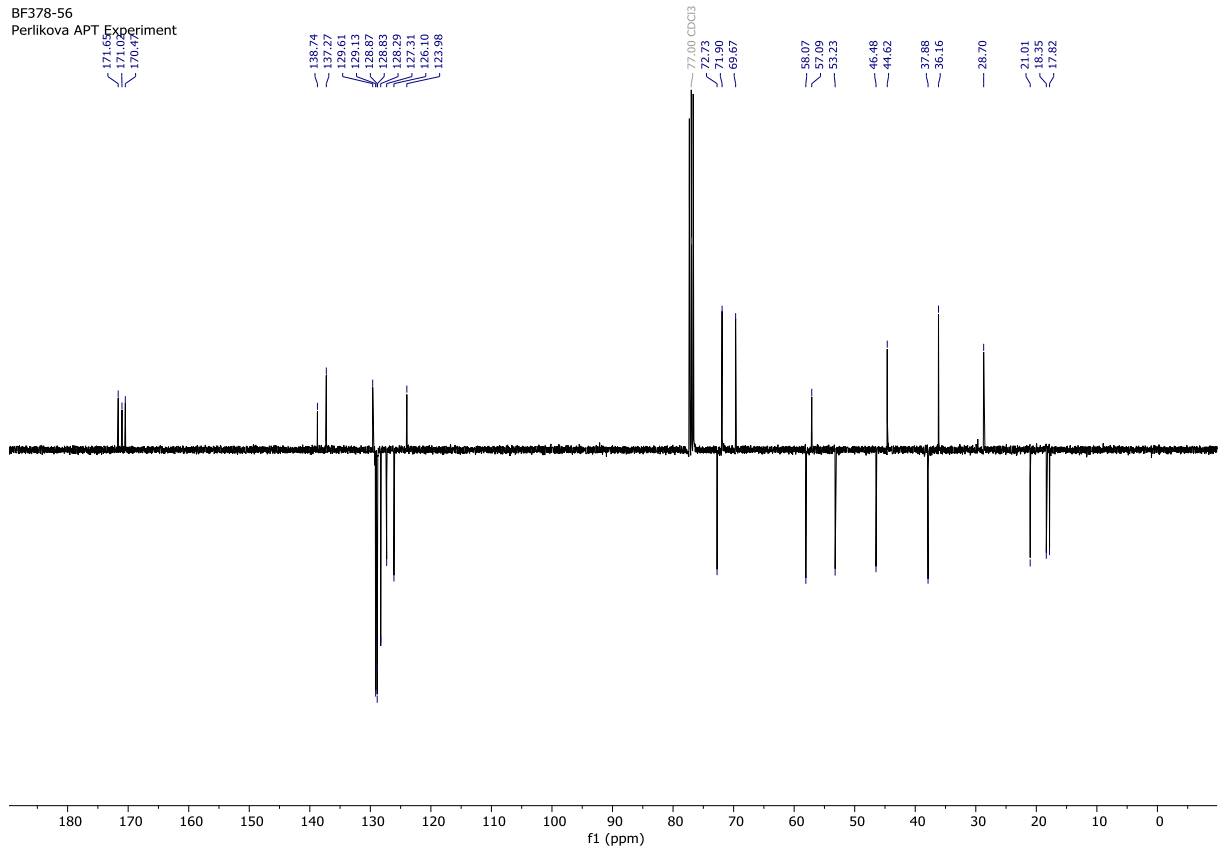
Perlikova APT Experiment



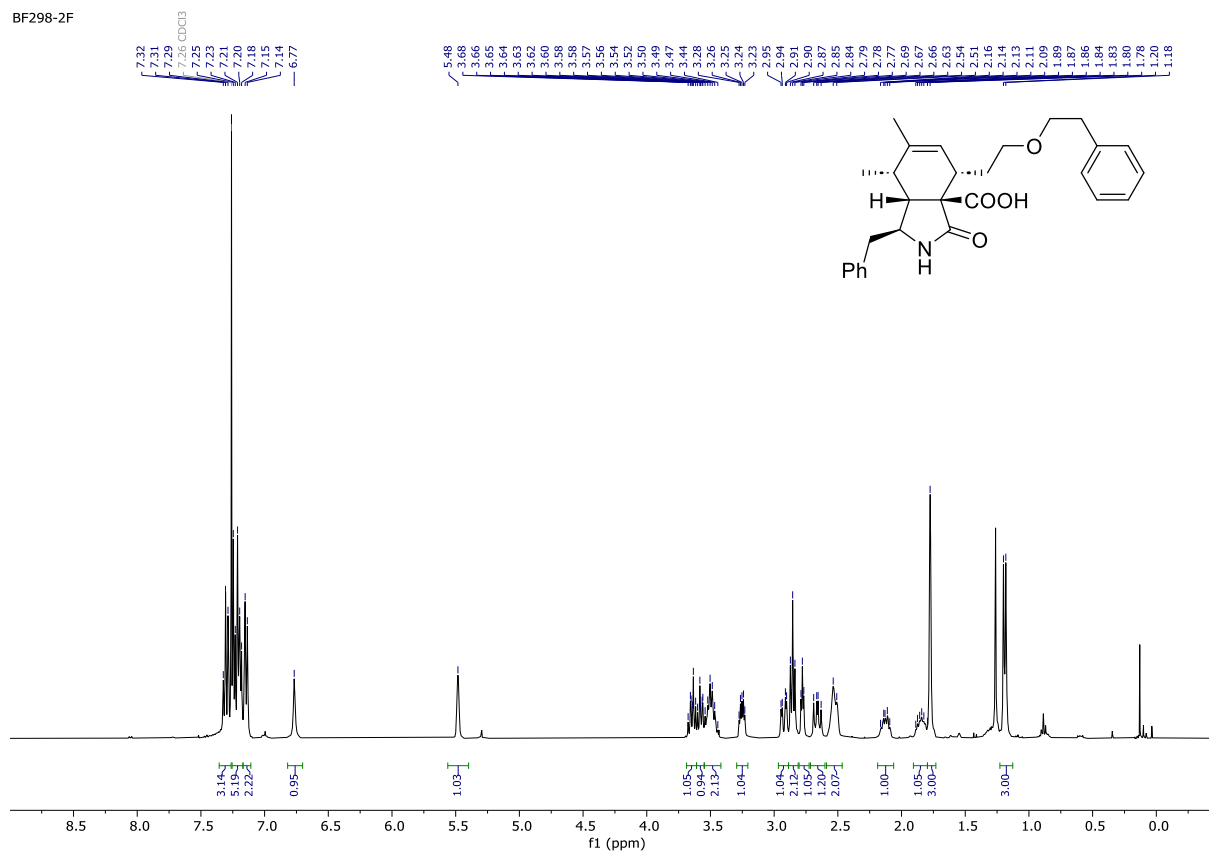
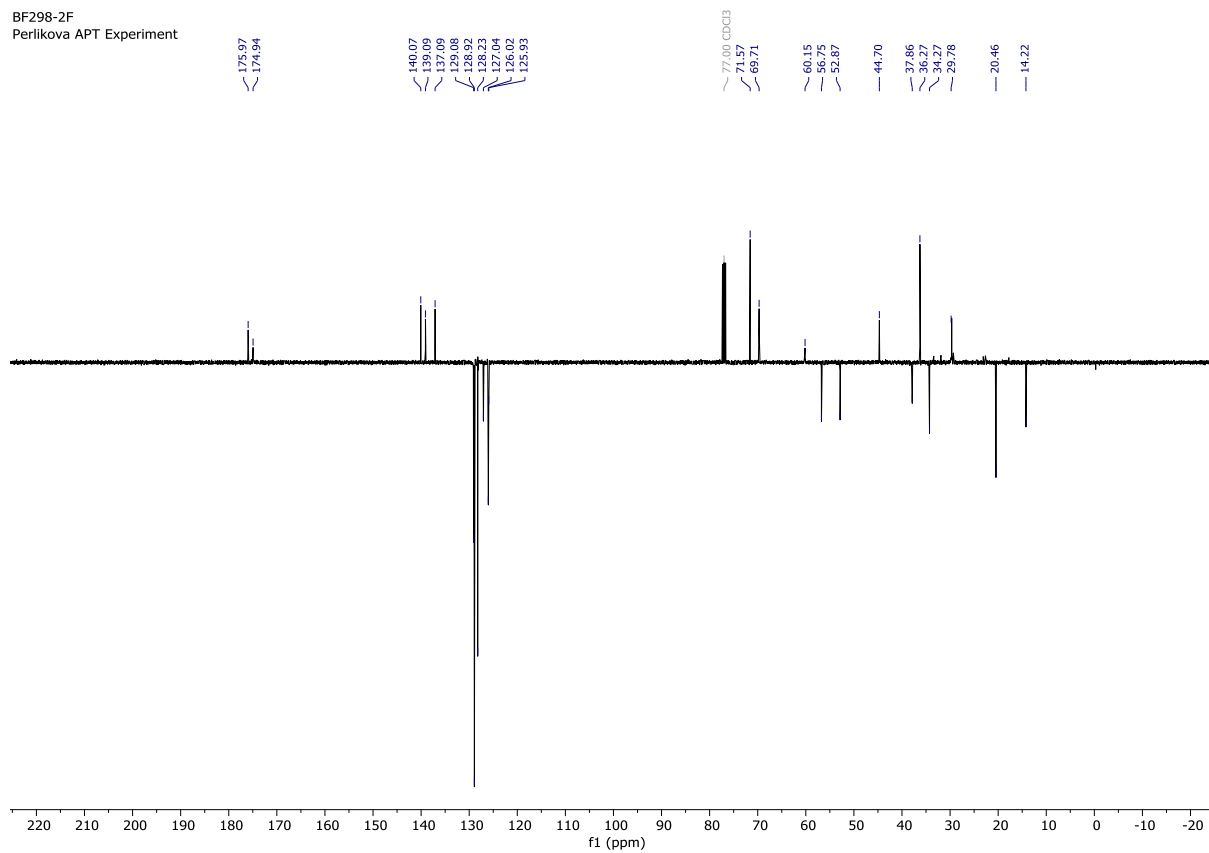
BF-22-378_HPLC-56



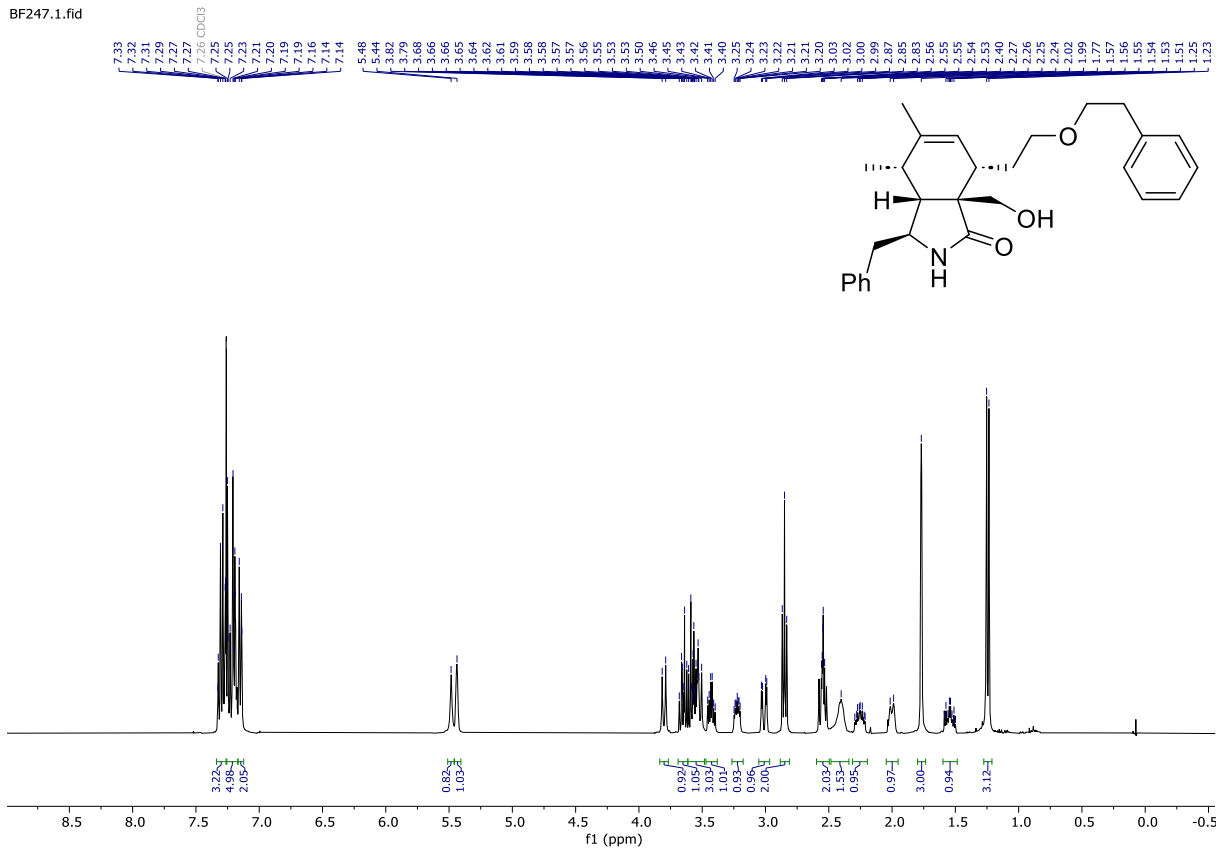
BF378-56
Perlikova APT Experiment



BF298-2F

BF298-2F
Perlikova APT Experiment

BF247.1.fid



BF247.2.fid

