

Supplementary Materials

Synthesis of Novel Carborane-containing Derivatives of RGD Peptide

Alexander V. Vakhrushev ¹, Dmitry A. Gruzdev ^{1,*}, Alexander M. Demin ¹, Galina L. Levit ¹, and Victor P. Krasnov ^{1,*}

¹ Postovsky Institute of Organic Synthesis, Russian Academy of Sciences (Ural Branch), Ekaterinburg 620108, Russia

Table of Contents	Page
NMR Spectra	S2
Figure S1. ¹ H NMR spectrum of compound 4	S2
Figure S2. ¹³ C NMR spectrum of compound 4	S2
Figure S3. ¹ H NMR spectrum of compound 5	S3
Figure S4. ¹³ C NMR spectrum of compound 5	S3
Figure S5. ¹ H NMR spectrum of compound 6	S4
Figure S6. ¹³ C NMR spectrum of compound 6	S4
Figure S7. ¹ H NMR spectrum of compound 7	S5
Figure S8. ¹³ C NMR spectrum of compound 7	S5
Figure S9. ¹ H NMR spectrum of compound 2c	S6
Figure S10. ¹³ C NMR spectrum of compound 2c	S6
Figure S11. ¹ H NMR spectrum of compound 1a	S7
Figure S12. ¹¹ B NMR spectrum of compound 1a	S7
Figure S13. ¹³ C NMR spectrum of compound 1a	S8
Figure S14. ¹ H NMR spectrum of compound 1b	S8
Figure S15. ¹¹ B NMR spectrum of compound 1b	S9
Figure S16. ¹³ C NMR spectrum of compound 1b	S9
Figure S17. ¹ H NMR spectrum of compound 1c	S10
Figure S18. ¹¹ B NMR spectrum of compound 1c	S10
Figure S19. ¹³ C NMR spectrum of compound 1c	S11
HPLC Data	S12
Figure S20. HPLC of compound 4	S12
Figure S21. HPLC of compound 6	S12
Figure S22. HPLC of compound 7	S13
Figure S23. HPLC of compound 2c	S13
Figure S24. HPLC of compound 1a	S14
Figure S25. HPLC of compound 1b	S14
Figure S26. HPLC of compound 1c	S15

NMR Spectra

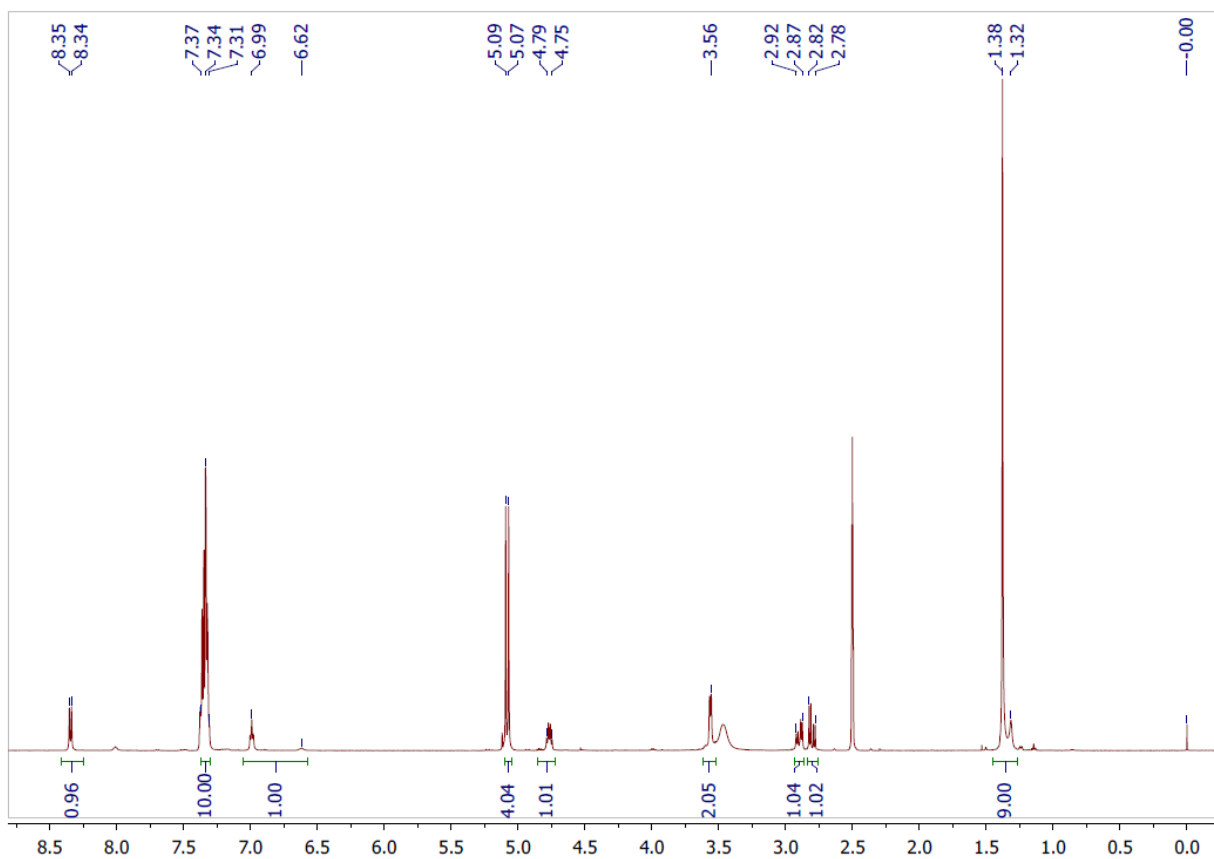


Figure S1. ¹H NMR spectrum of compound 4

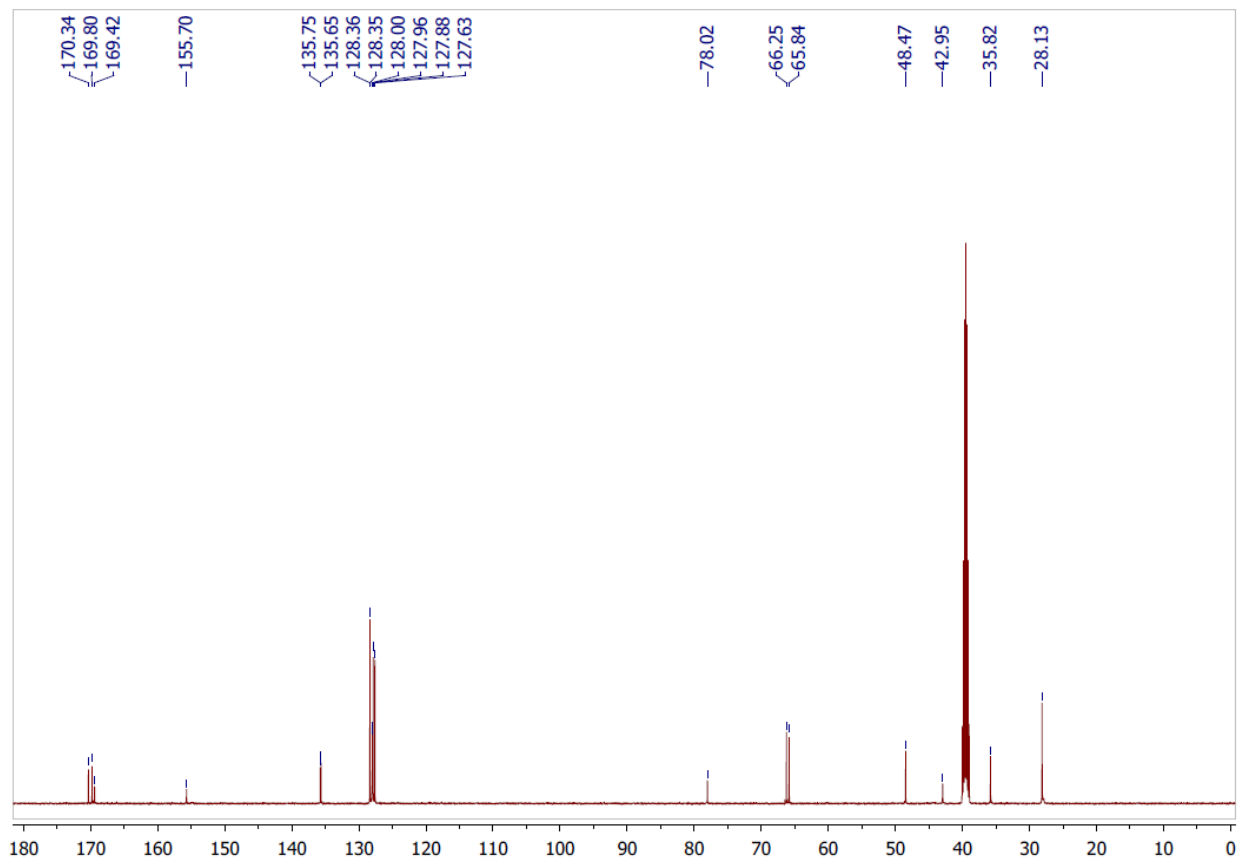


Figure S2. ¹³C NMR spectrum of compound 4

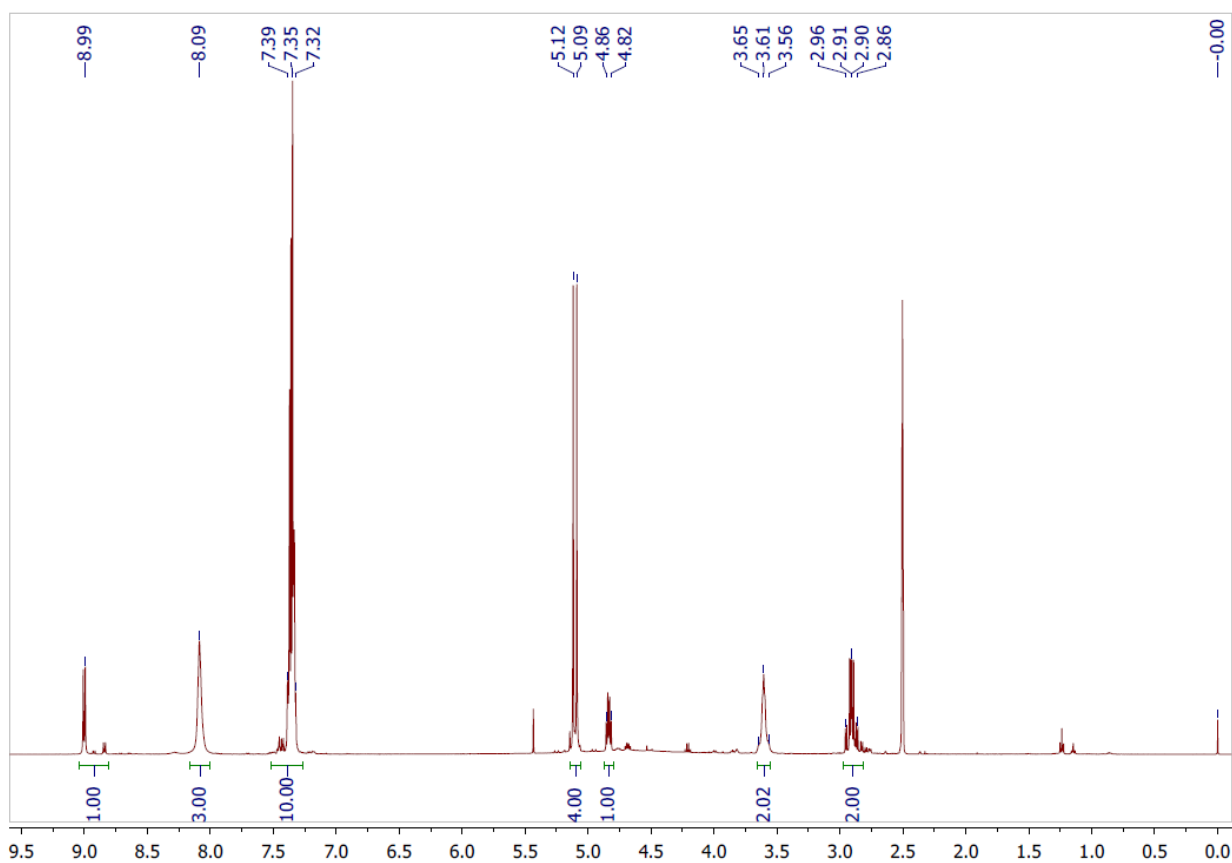


Figure S3. ¹H NMR spectrum of compound 5

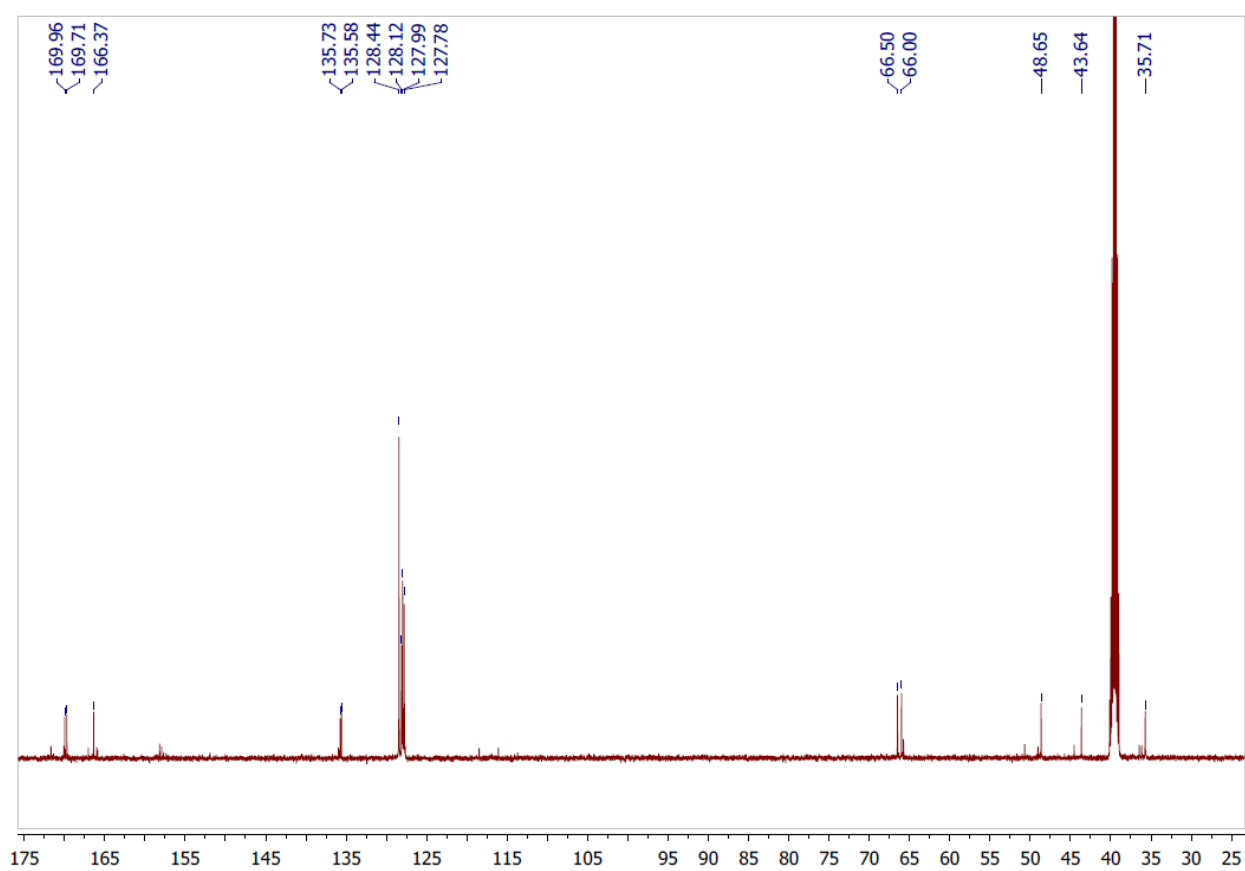


Figure S4. ¹³C NMR spectrum of compound 5

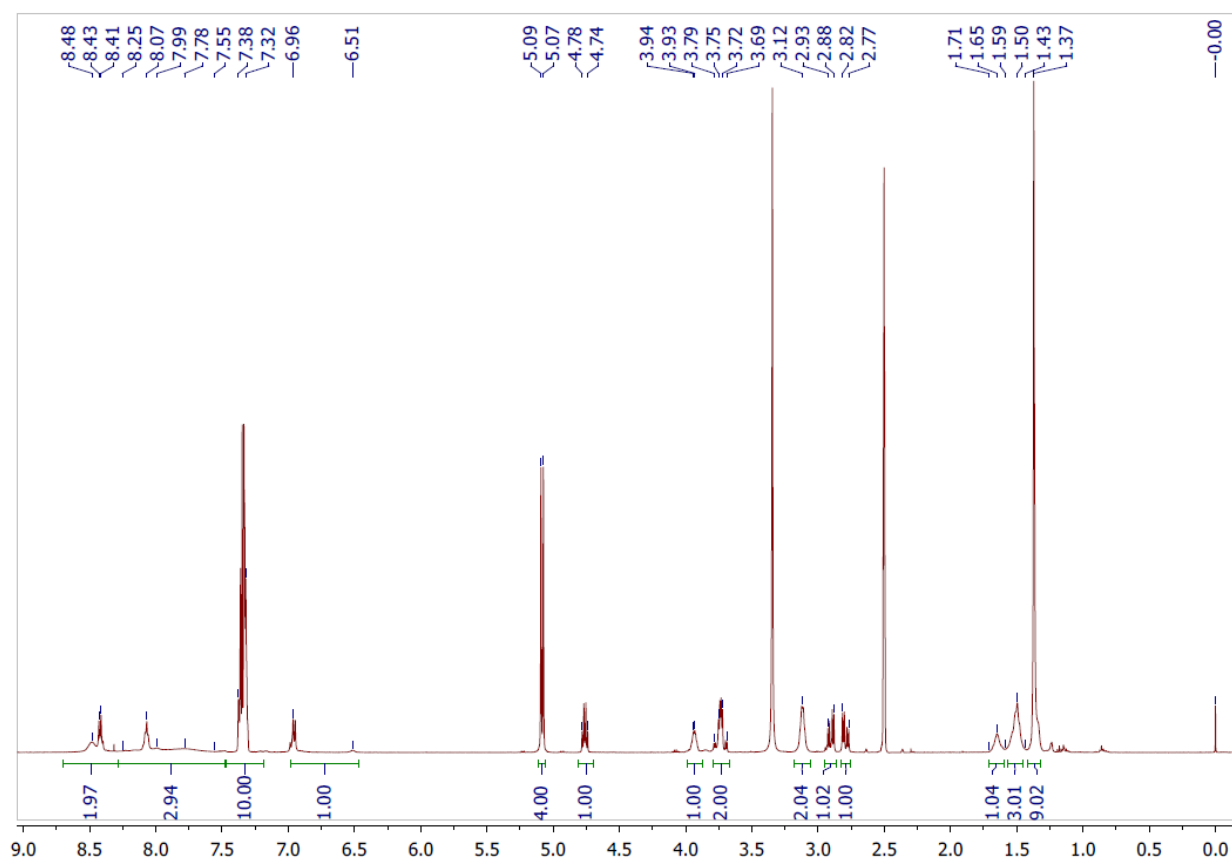


Figure S5. ^1H NMR spectrum of compound 6

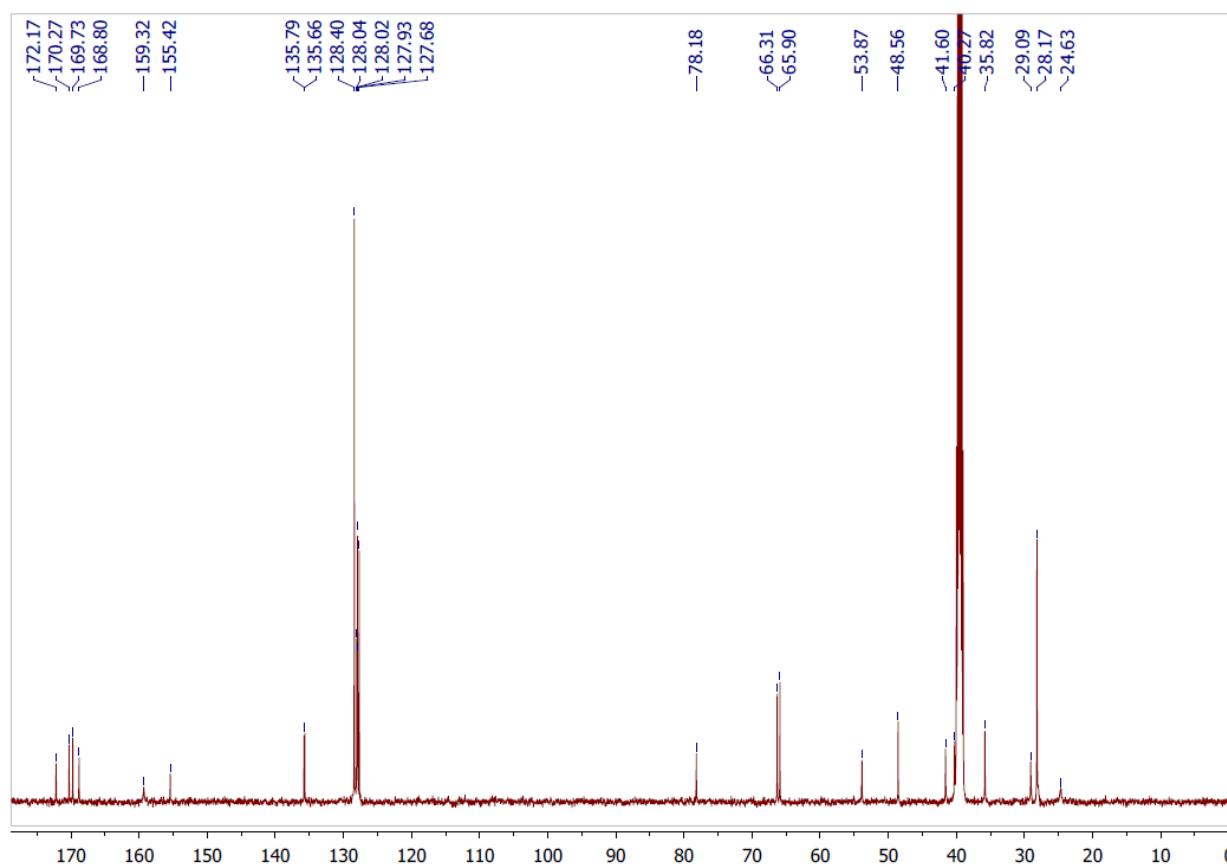


Figure S6. ^{13}C NMR spectrum of compound 6

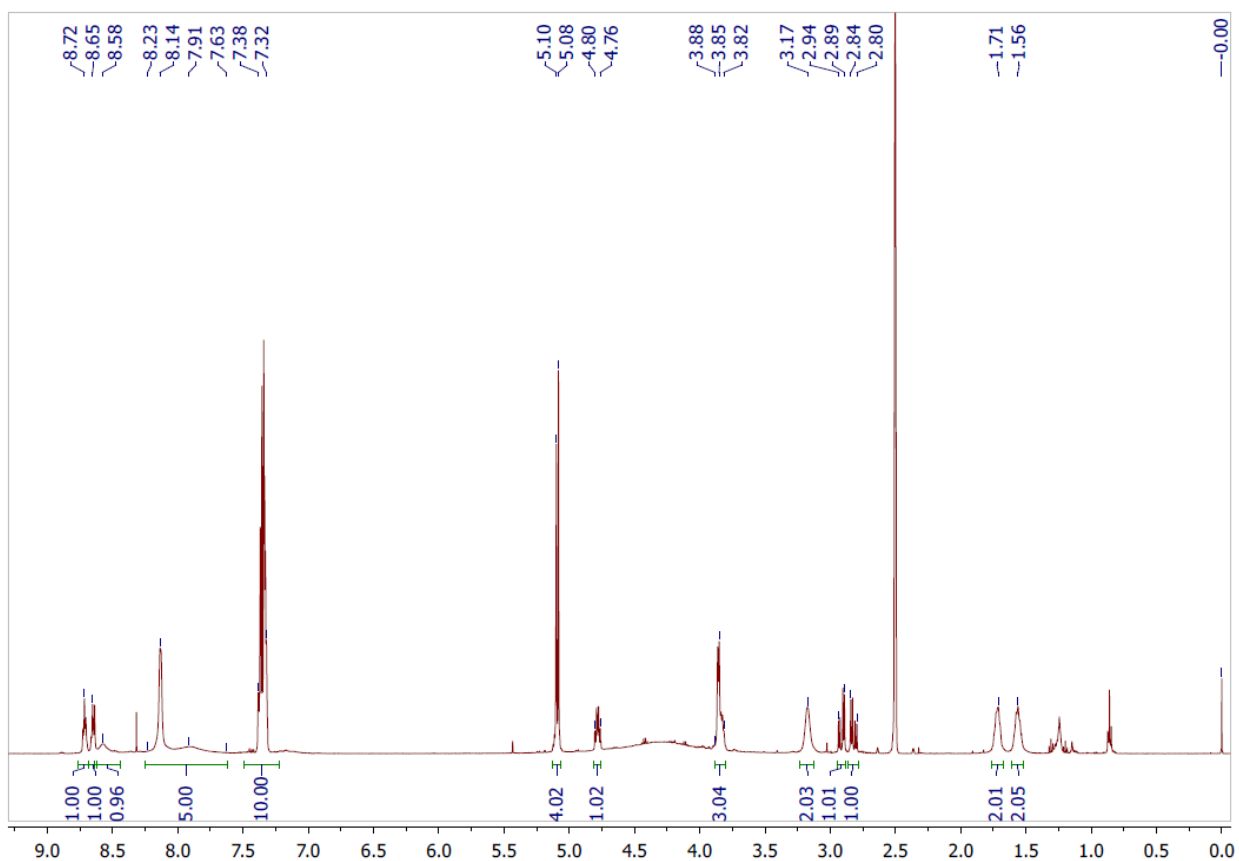


Figure S7. ^1H NMR spectrum of compound 7

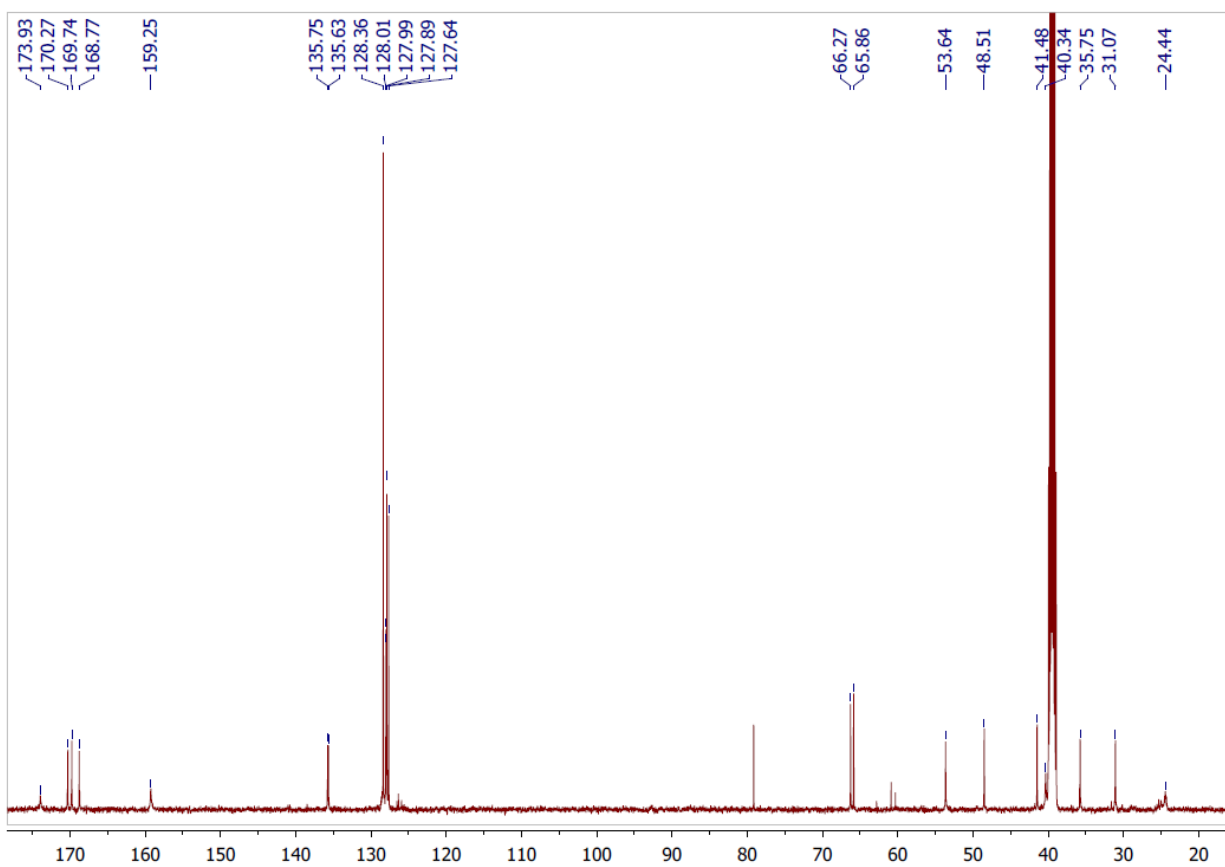


Figure S8. ^{13}C NMR spectrum of compound 7

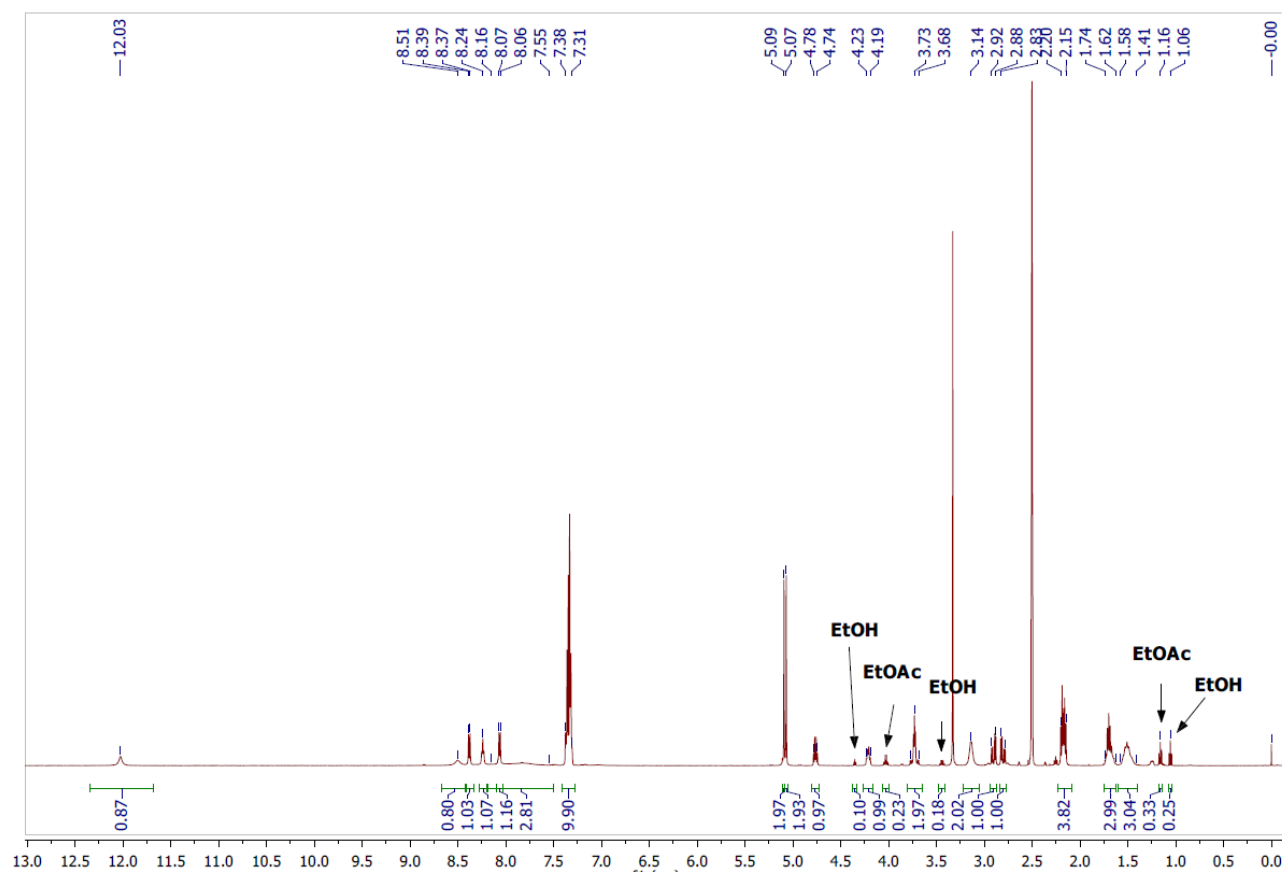


Figure S9. ^1H NMR spectrum of compound **2c**

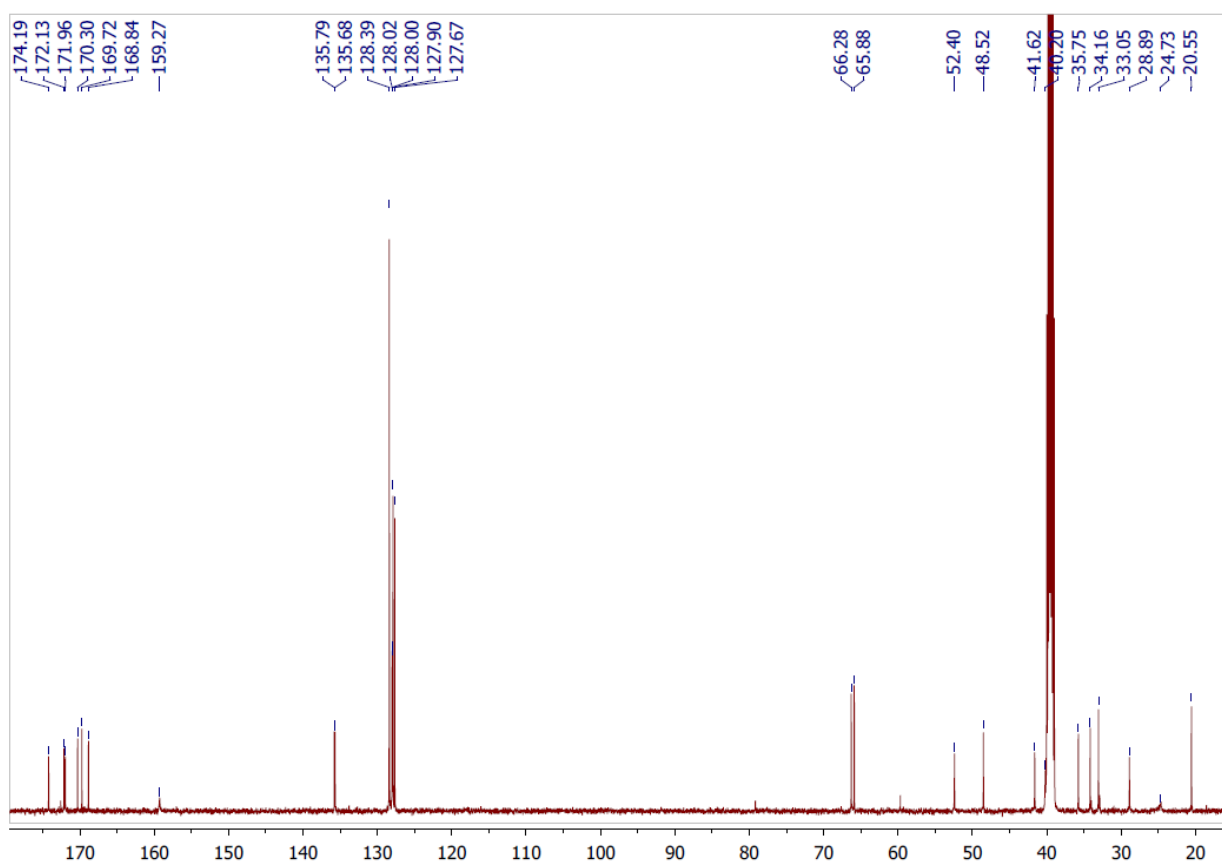


Figure S10. ^{13}C NMR spectrum of compound **2c**

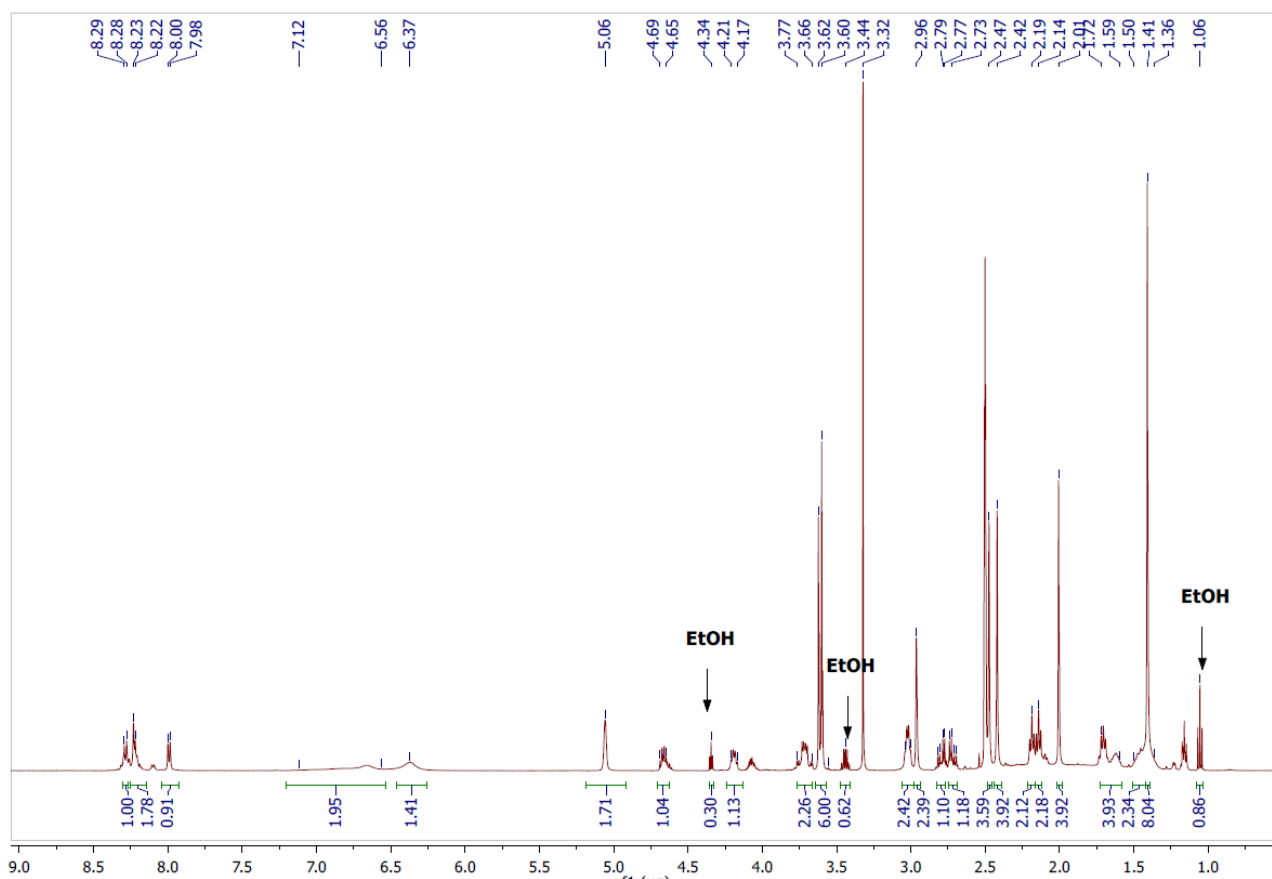


Figure S11. ¹H NMR spectrum of compound **1a**

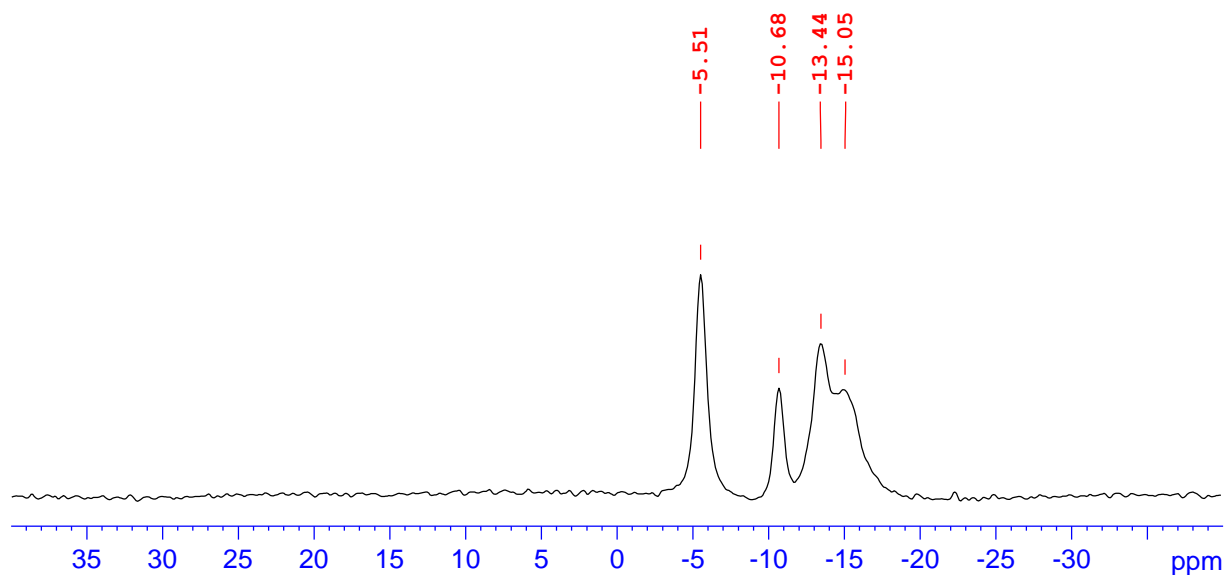


Figure S12. ¹¹B NMR spectrum of compound **1a**

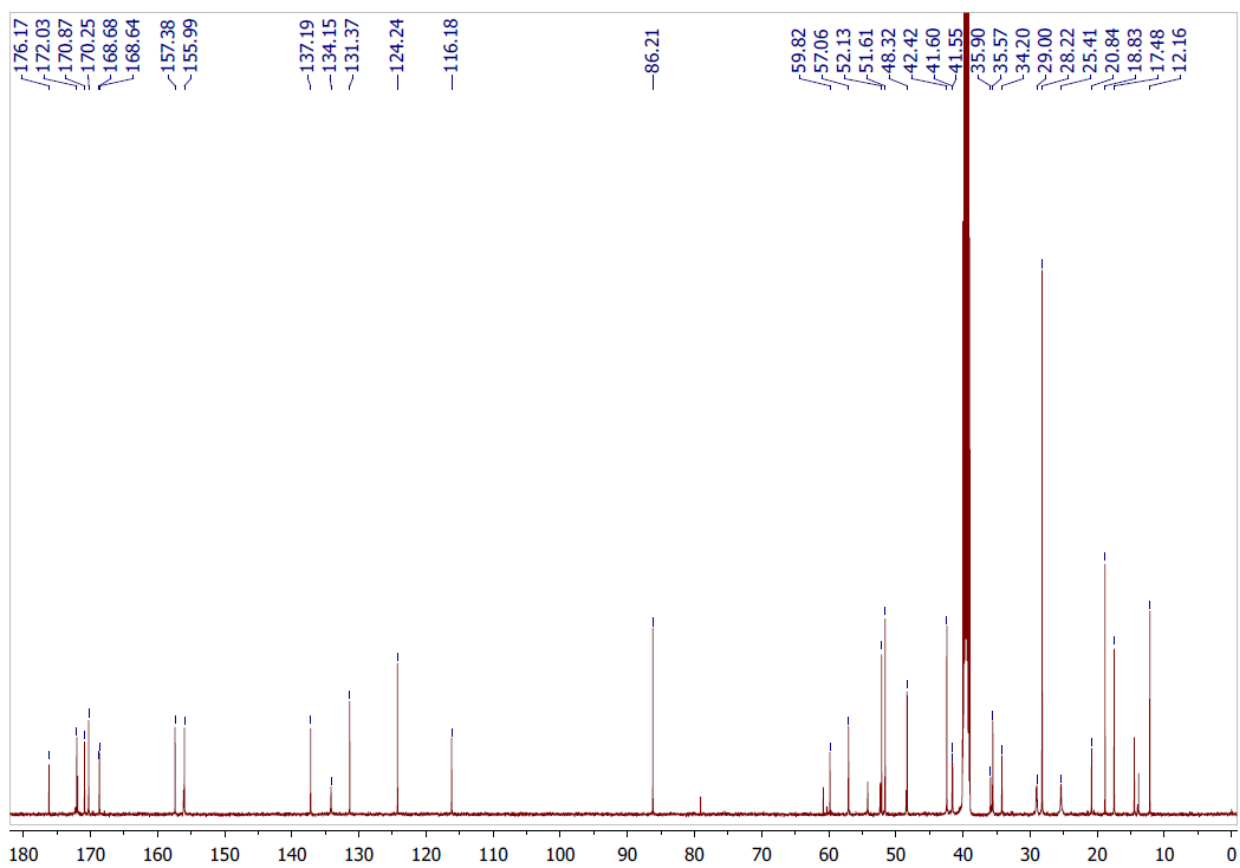


Figure S13. ¹³C NMR spectrum of compound **1a**

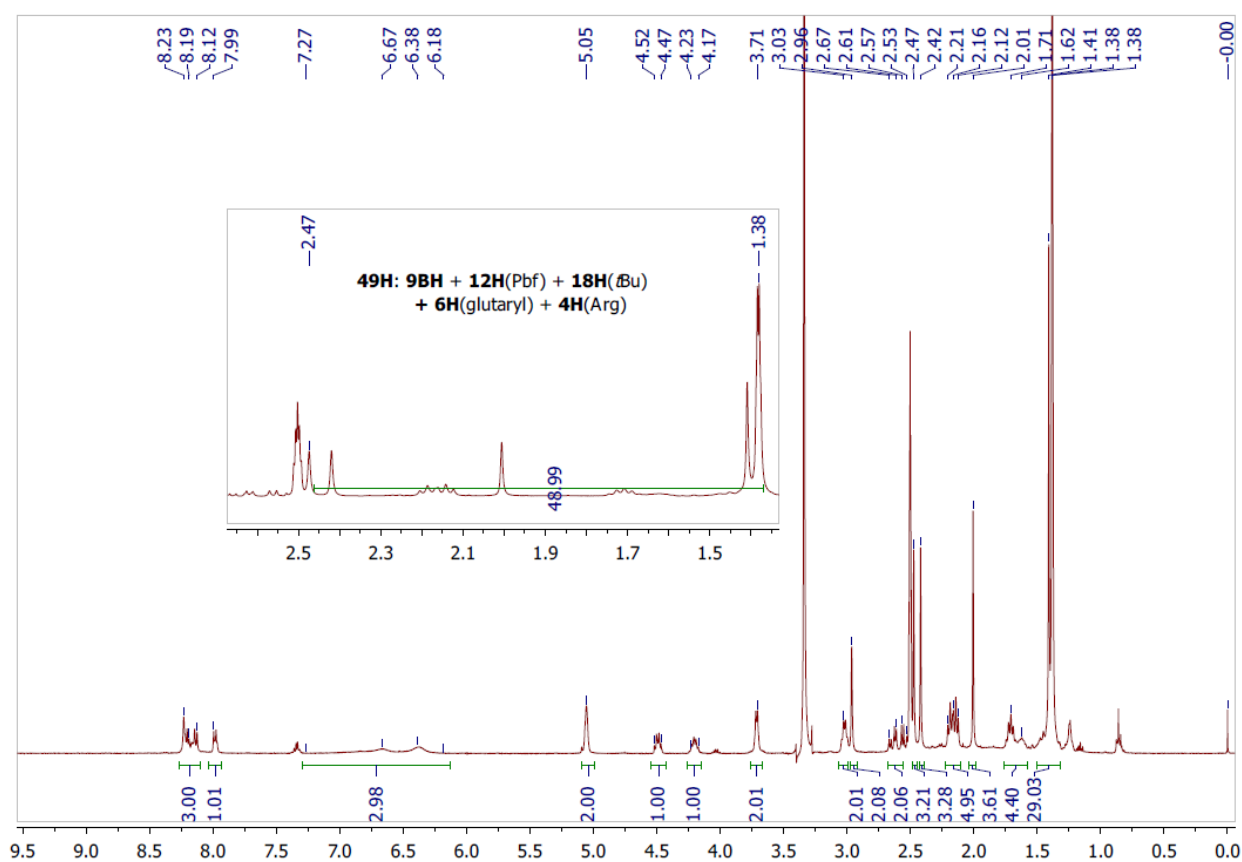


Figure S14. ¹H NMR spectrum of compound **1b**

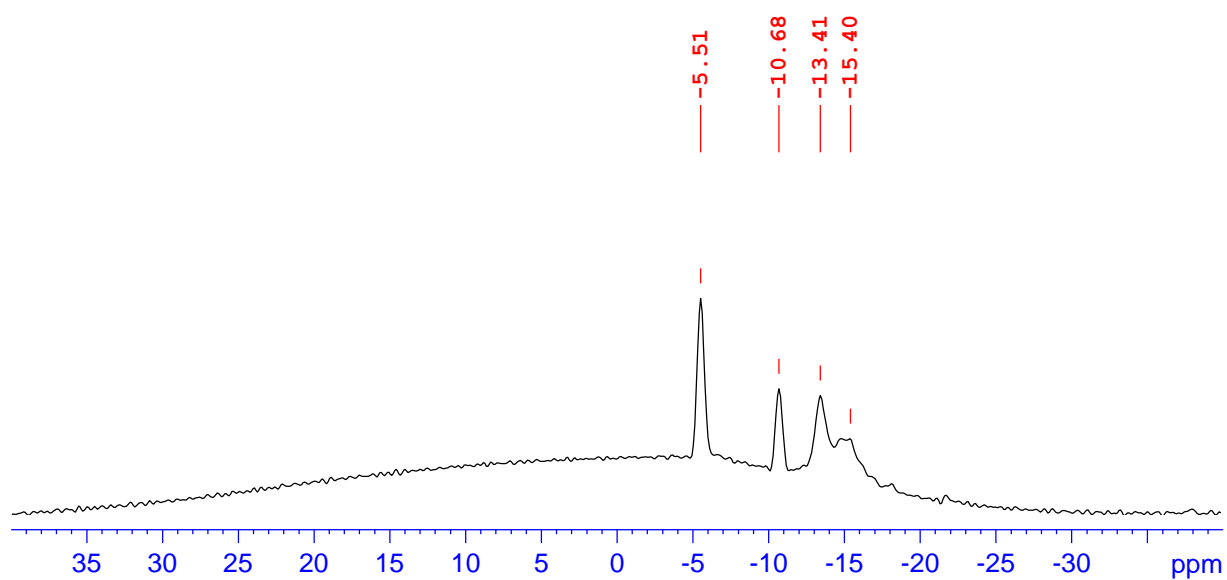


Figure S15. ^{11}B NMR spectrum of compound **1b**

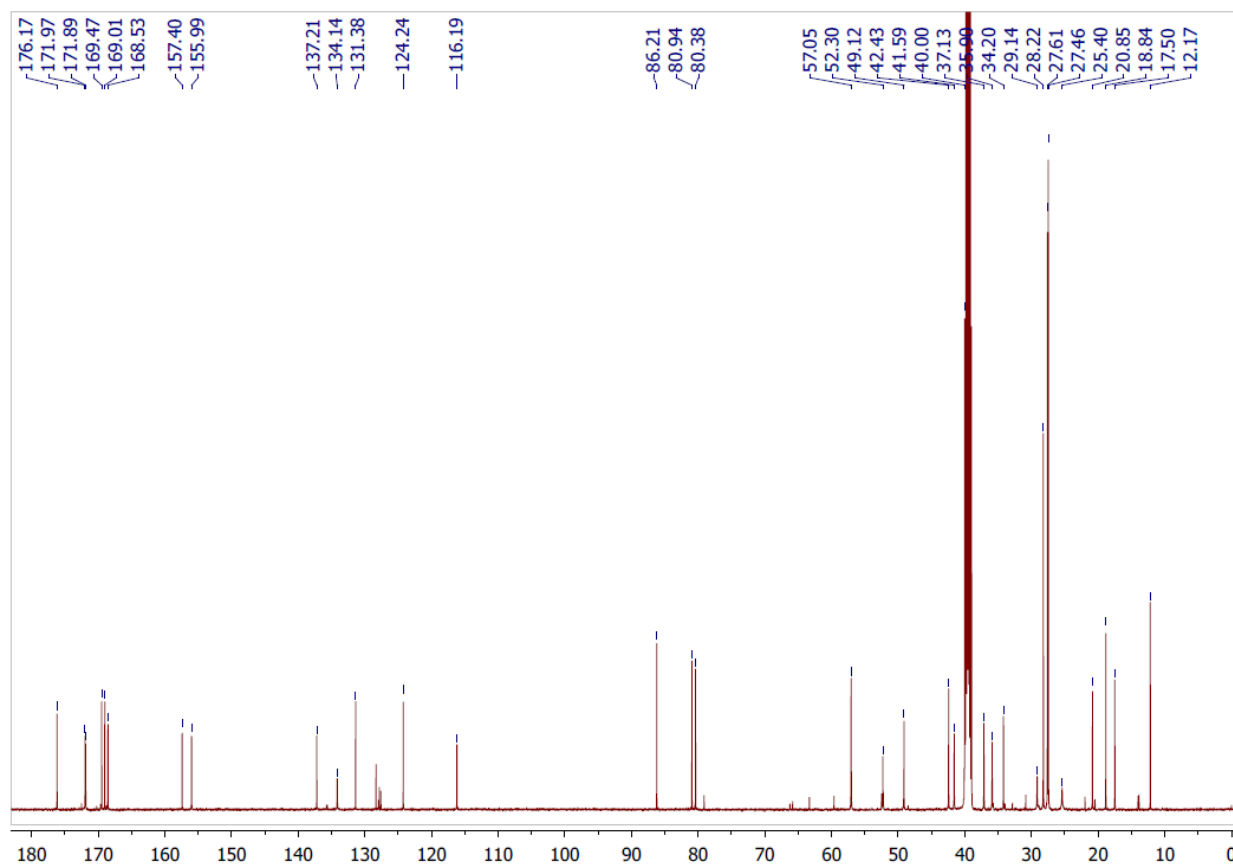


Figure S16. ^{13}C NMR spectrum of compound **1b**

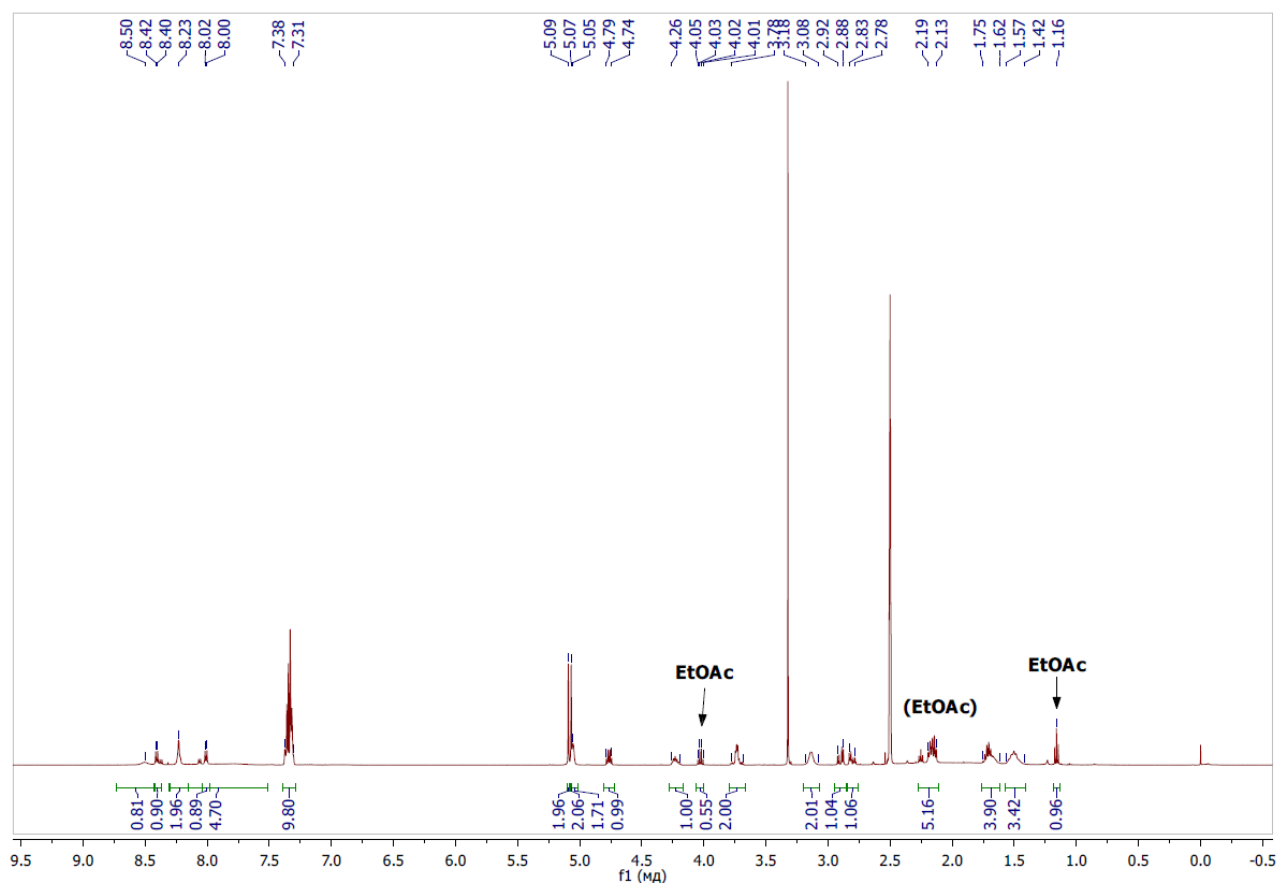


Figure S17. ^1H NMR spectrum of compound **1c**

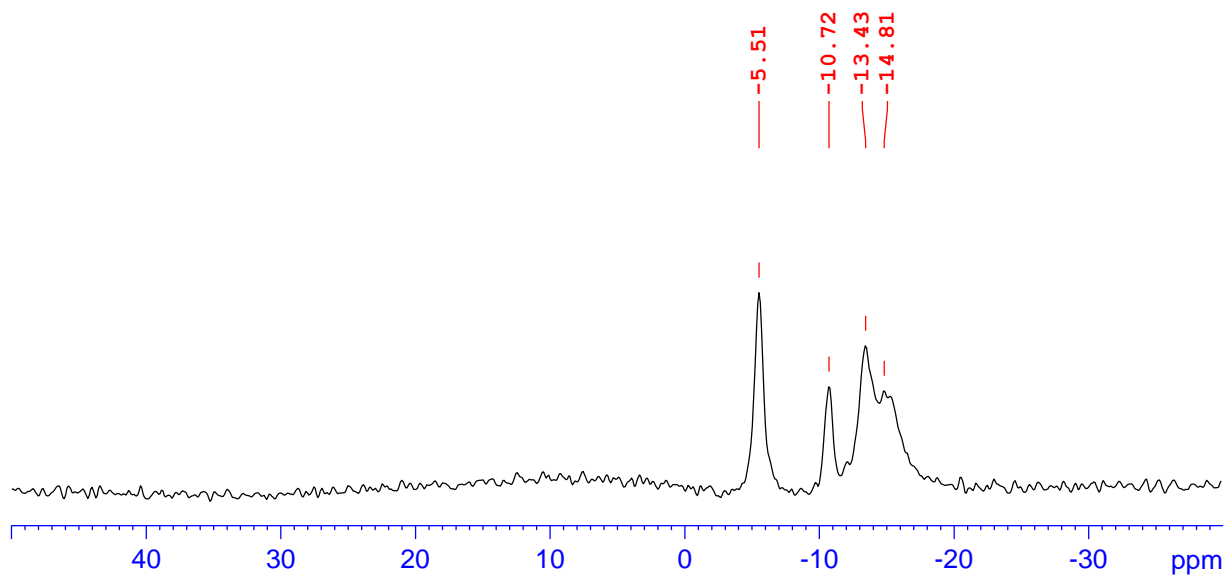


Figure S18. ^{11}B NMR spectrum of compound **1c**

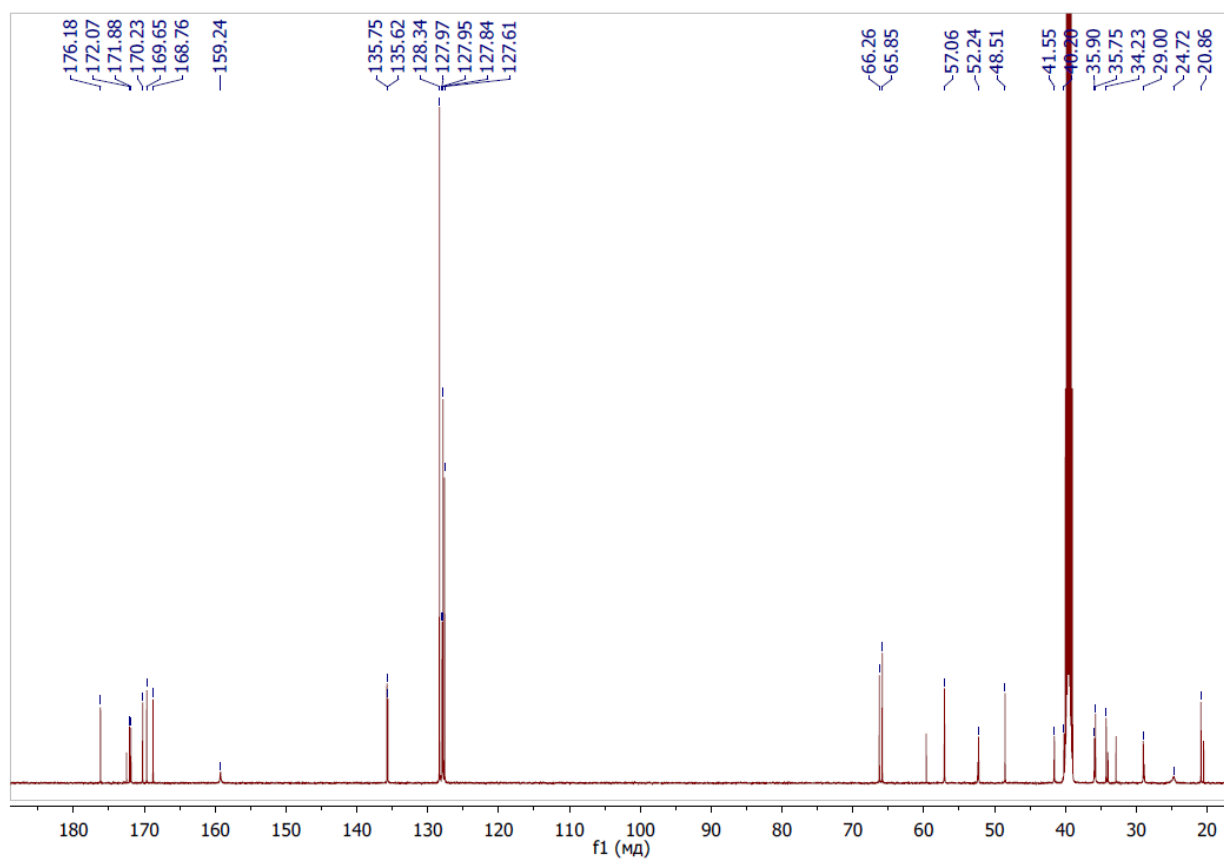
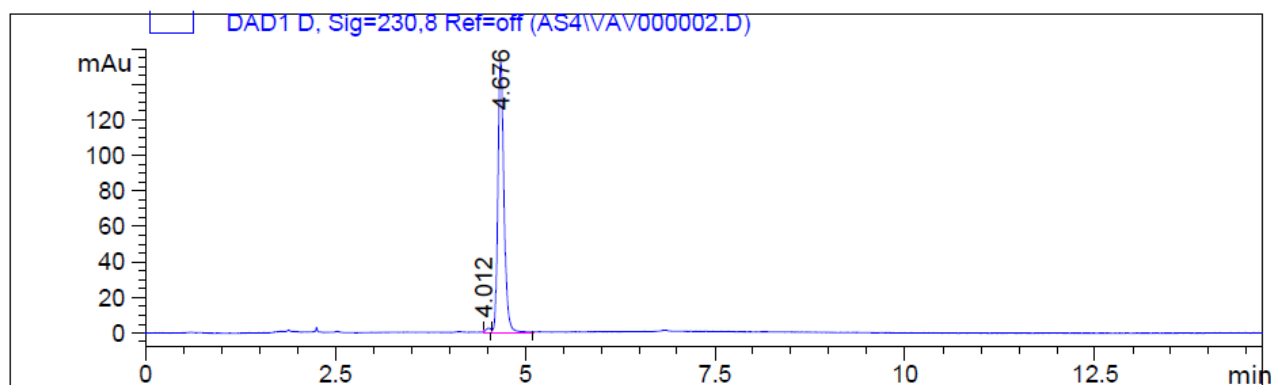


Figure S19. ^{13}C NMR spectrum of compound **1c**

HPLC Data

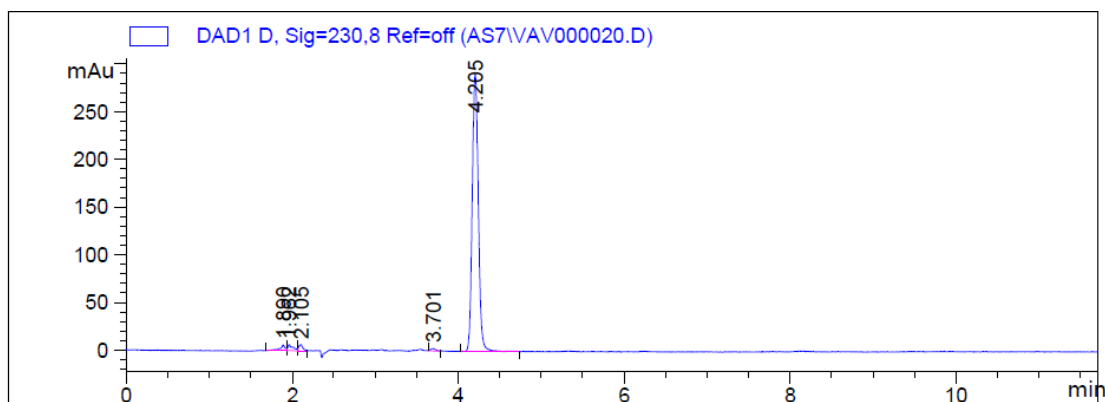


Signal 1: DAD1 D, Sig=230,8 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.676	VB	0.0870	846.00684	151.79439	98.9607
2	4.012	VB	0.0183	8.88488	1.55413	1.0393

Totals : 854.89172 153.34852

Figure S20. HPLC of compound **4** (Kromasil 100-5 C18, MeCN–H₂O 1 : 1, 0.8 mL/min, 35 °C, 230 nm)

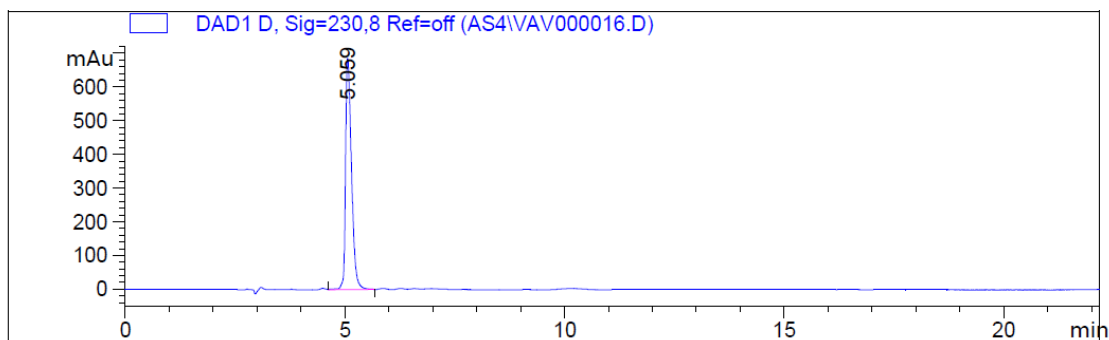


Signal 1: DAD1 D, Sig=230,8 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.890	MF	0.0719	4.41712	2.14745	0.2807
2	1.962	FM	0.0724	4.57223	2.31812	0.2906
3	2.105	FM	0.0542	3.44363	2.35420	0.2189
4	3.701	MM	0.0744	1.64553	1.21082	0.1046
5	4.205	BB	0.0797	1488.84937	291.11545	99.1052

Totals : 1502.92788 299.14604

Figure S21. HPLC of compound **6** (Kromasil 100-5 C18, MeCN–H₂O–AcOH 80: 20 : 0.0025, 0.8 mL/min, 35 °C, 230 nm)

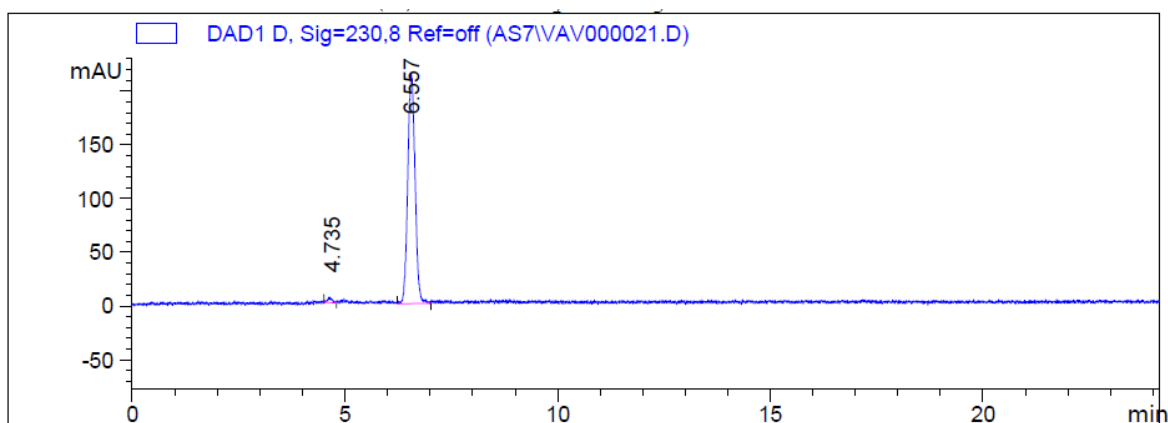


Signal 3: DAD1 D, Sig=230,8 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.059	VV	0.1459	6572.58545	685.54456	100.0000

Totals : 6572.58545 685.54456

Figure S22. HPLC of compound **7** (Kromasil 100-5 C18, MeCN–H₂O–CF₃CO₂H 70: 30 : 0.01, 0.8 mL/min, 35 °C, 230 nm)

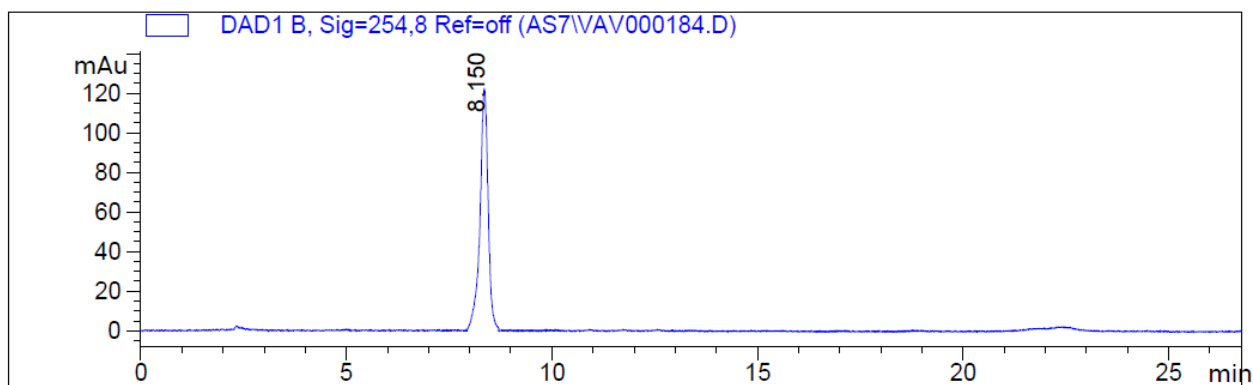


Signal 1: DAD1 D, Sig=230,8 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.735	BB	0.0710	7.65632	8.95126	0.2990
2	6.557	BB	0.1873	2552.85986	213.81601	99.7010

Totals : 2560.51618 222.76727

Figure S23. HPLC of compound **2c** (Kromasil 100-5 C18, MeCN–H₂O–AcOH 60 : 40 : 0.005, 0.8 mL/min, 35 °C, 230 nm)

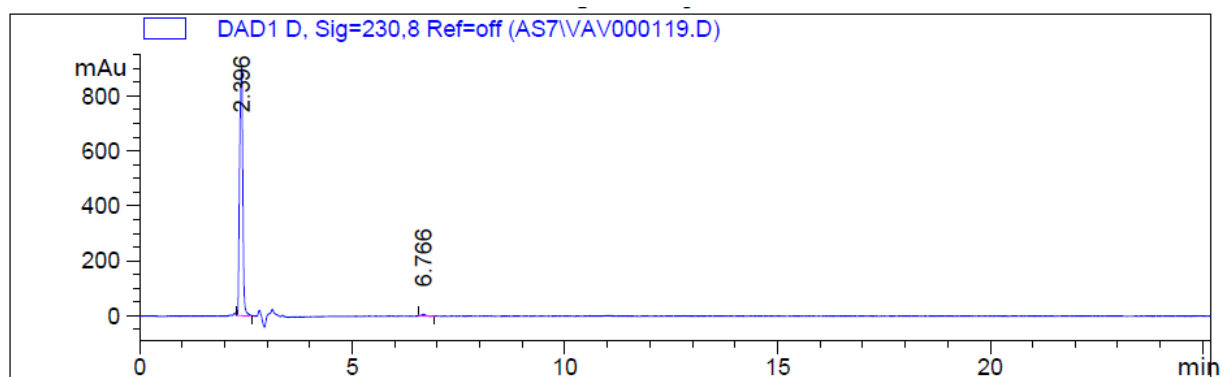


Signal 1: DAD1 B, Sig=254,8 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.150	VB	0.2490	2318.23071	131.92964	100.0000

Totals : 2318.23071 131.92964

Figure S24. HPLC of compound **1a** (Kromasil 100-5 C18, MeCN–H₂O 1 : 1, 0.8 mL/min, 35 °C, 254 nm)

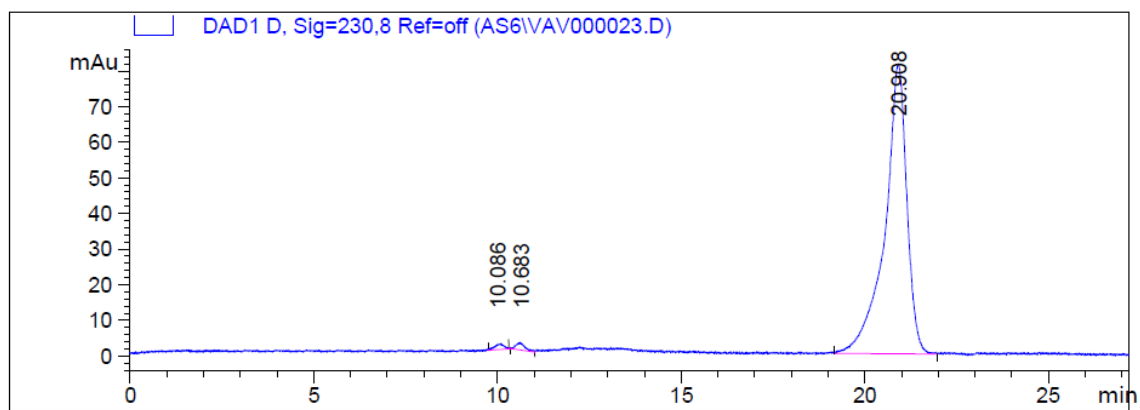


Signal 1: DAD1 D, Sig=230,8 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	2.396	BB	0.0862	4706.64990	910.28558	99.7007
2	6.766	BB	0.0644	14.12896	2.73546	0.2993

Totals : 4720.77886 913.02104

Figure S25. HPLC of compound **1b** (Kromasil 100-5 C18, MeCN–H₂O–AcOH 40 : 60 : 0.0025, 0.8 mL/min, 35 °C, 230 nm)



Signal 2: DAD1 D, Sig=230,8 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.086	BV	0.1274	16.88312	4.01685	0.4941
1	10.683	BV	0.1486	23.63285	4.56981	0.6917
1	20.908	BB	0.5838	3376.25488	81.08988	98.8142

Totals : 3416.77085 89.67654

Figure S26. HPLC of compound **1c** (Kromasil 100-5 C18, MeCN–0.06 M phosphate buffer solution (pH 7.0) 8 : 2, 0.8 mL/min, 35 °C, 230 nm)