Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Design and Synthesis of N^1,N^8-Diacetylspermidine Analogues Having A Linker with Desired Functional Groups

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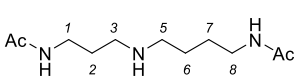
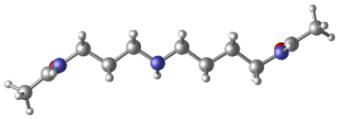
1. Computational chemistry

Conformational Search: All conformational searches were performed using the Monte Carlo Multiple Minimum (MCMM)[1] search protocol as implemented by MacroModel[2] (ver. 10.8) with the solvation model in H_2O and an OPLS-2005 force field. All conformations obtained from torsional sampling (maximum number of steps: 1000, 100 steps per rotable bond). Each geometry of the generated conformer was optimized employing a Polak-Ribière Conjugate gradient (PRCG) method with a convergence criteria of 0.05 and a maximum of 2500 iterations. The results of these conformational searches were manually analyzed and stable conformations were considered for further optimization at the density functional theory (DFT) level.

DFT calculation: All DFT calculations were performed using the Gaussian 09 program package[3], and the B3LYP hybrid functional. Optimized geometries for each conformer were subjected to single point calculations at a B3LYP/6-31G(d) level of theory. The DFT optimized structures were visualized by CYLview[4].

· DiAcSpd (Figure 2A)





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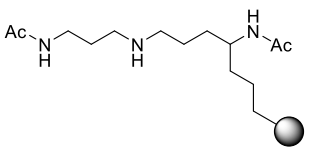
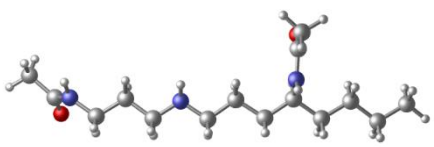
・ Substituted in C8 position (Figure 2B)





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12	6	0	0.667470	-0.977271	-0.612169
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14	6	0	3.045803	-0.899101	-0.728751
15	6	0	4.295221	-0.074438	-0.385794
16	6	0	5.593440	-0.783867	-0.809366
17	7	0	6.773879	0.019955	-0.501805
18	6	0	7.430093	0.026307	0.663940
19	6	0	8.643371	0.940036	0.761361
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46	1	0	9.393311	0.659740	0.021440
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50	1	0	-0.525545	-0.022708	0.918949
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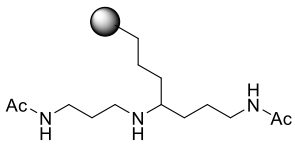
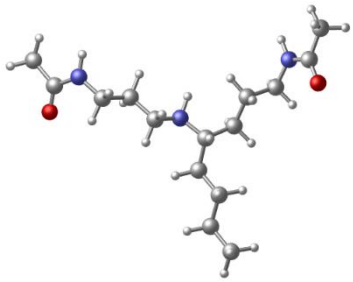
・ Substituted in C5 position (Figure 2B)





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14	6	0	-4.059165	-0.003243	-0.743409
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16	8	0	-1.263003	1.133376	0.040105
17	1	0	1.381682	2.558564	-0.693069
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21	1	0	2.067016	2.081433	1.779630
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24	1	0	1.990717	-0.705984	2.703729
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39	1	0	3.806949	0.345129	1.194437
40	6	0	3.618015	-0.924578	-0.551219

41	1	0	3.549999	-1.854455	0.012955
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45	1	0	5.735290	-0.649255	-0.208061
46	6	0	5.517442	-1.940346	-1.918803
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51	1	0	-3.736817	1.013475	-2.519383
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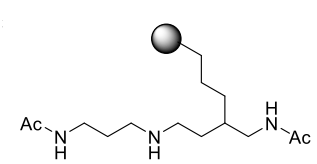
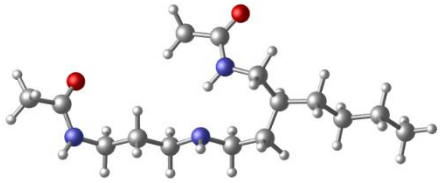
・ Substituted in C7 position (Figure 2C)





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8	7	0	0.581339	1.433866	0.213078
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18	6	0	-5.779565	-0.074167	0.001745
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24	1	0	6.804646	-0.482276	1.244736
25	1	0	6.931521	0.805209	0.071275
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28	1	0	4.328437	0.122951	1.649571
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41	1	0	-1.377026	-2.769690	-0.623240
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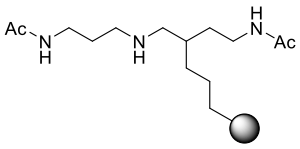
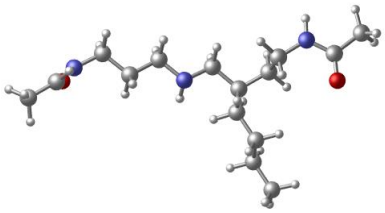
・ Substituted in C6 position (Figure 2C)





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3	6	0	-2.132913	2.711064	0.149370
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6	6	0	-2.740384	-0.380439	-0.520153
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12	6	0	-0.211030	-0.614398	-0.661785
13	7	0	1.028263	-0.272586	0.023706
14	6	0	2.128999	-1.056738	-0.519940
15	6	0	3.430620	-0.704618	0.214540
16	6	0	4.629602	-1.515932	-0.307516
17	7	0	5.857046	-1.192167	0.415365
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50	1	0	-1.328782	0.246393	0.969512
51	1	0	3.309622	-0.887937	1.283486

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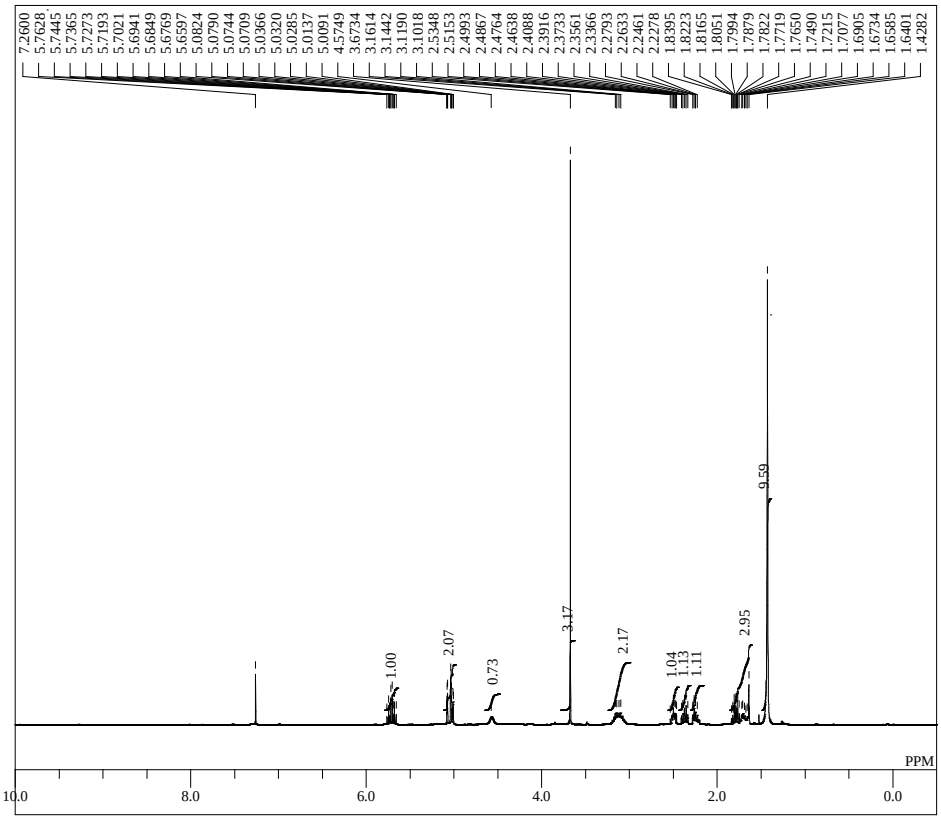
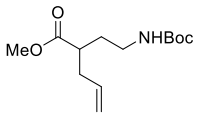
4. References

1. G. Chang, W. C. Guida, W. C. Still, An internal-coordinate Monte Carlo method for searching conformational space, *J. Am. Chem. Soc*., 1989, **111**, 4379‑4386.
2. MacroModel, Version 10.8, Schrödinger, LLC, New York, NY, 2014.
3. Gaussian 09, Revision A.02, M. J. Frisch, G. W. Trucks, H. B. Schlegel, G. E. Scuseria, M. A. Robb, J. R. Cheeseman, G. Scalmani, V. Barone, B. Mennucci, G. A. Petersson, H. Nakatsuji, M. Caricato, X. Li, H. P. Hratchian, A. F. Izmaylov, J. Bloino, G. Zheng, J. L. Sonnenberg, M. Hada, M. Ehara, K. Toyota, R. Fukuda, J. Hasegawa, M. Ishida, T. Nakajima, Y. Honda, O. Kitao, H. Nakai, T. Vreven, J. A. Montgomery, Jr., J. E. Peralta, F. Ogliaro, M. Bearpark, J. J. Heyd, E. Brothers, K. N. Kudin, V. N. Staroverov, R. Kobayashi, J. Normand, K. Raghavachari, A. Rendell, J. C. Burant, S. S. Iyengar, J. Tomasi, M. Cossi, N. Rega, J. M. Millam, M. Klene, J. E. Knox, J. B. Cross, V. Bakken, C. Adamo, J. Jaramillo, R. Gomperts, R. E. Stratmann, O. Yazyev, A. J. Austin, R. Cammi, C. Pomelli, J. W. Ochterski, R. L. Martin, K. Morokuma, V. G. Zakrzewski, G. A. Voth, P. Salvador, J. J. Dannenberg, S.Dapprich, A. D. Daniels, O. Farkas, J. B. Foresman, J. V. Ortiz, J. Cioslowski, D. J. Fox, Gaussian, Inc., Wallingford CT, 2009.
4. CYLview, 1.0b, Legault, C. Y., Université de Sherbrooke, 2009 (http://www.cylview.org).

^{1}H NMR spectrum of **10**

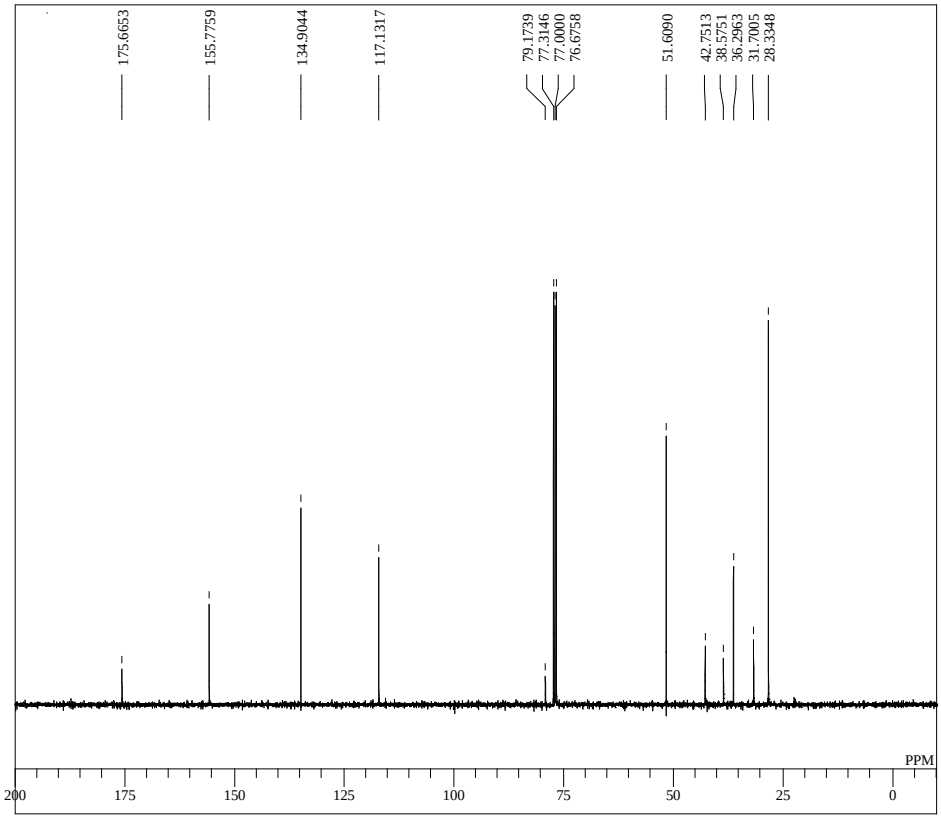


DFILE 1_DiAcSpd_step3_1H.als
COMNT single_pulse
DATIM 2018-06-14 19:36:50
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 13107
FREQU 6002.40 Hz
SCANS 8
ACQTM 2.1837 sec
PD 1.0000 sec
PW1 7.25 usec
IRNUC 1H
CTEMP 19.5 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 40

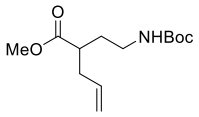


10

^{13}C NMR spectrum of **10**

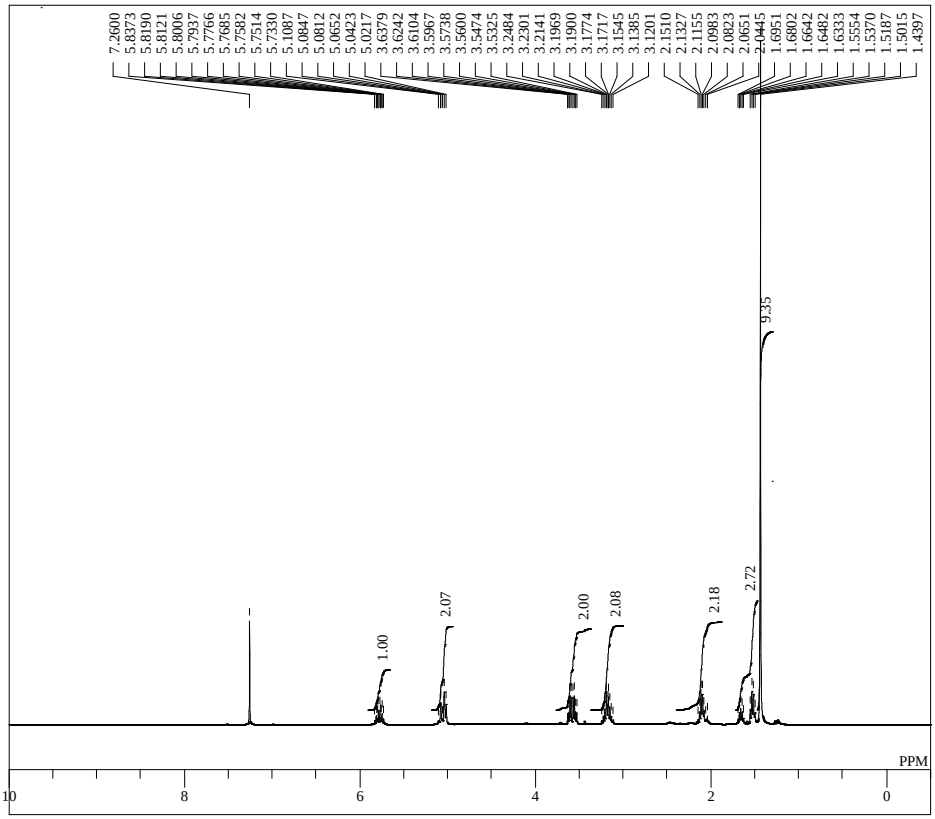


DFILE 1_DiAcSpd_step3_13C.als
COMNT single pulse decoupled gated NOE
DATIM 2018-06-15 10:07:27
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 26214
FREQU 25125.63 Hz
SCANS 451
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 3.17 usec
IRNUC 1H
CTEMP 19.9 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

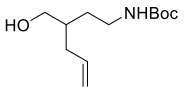


10

^{1}H NMR spectrum of **11**

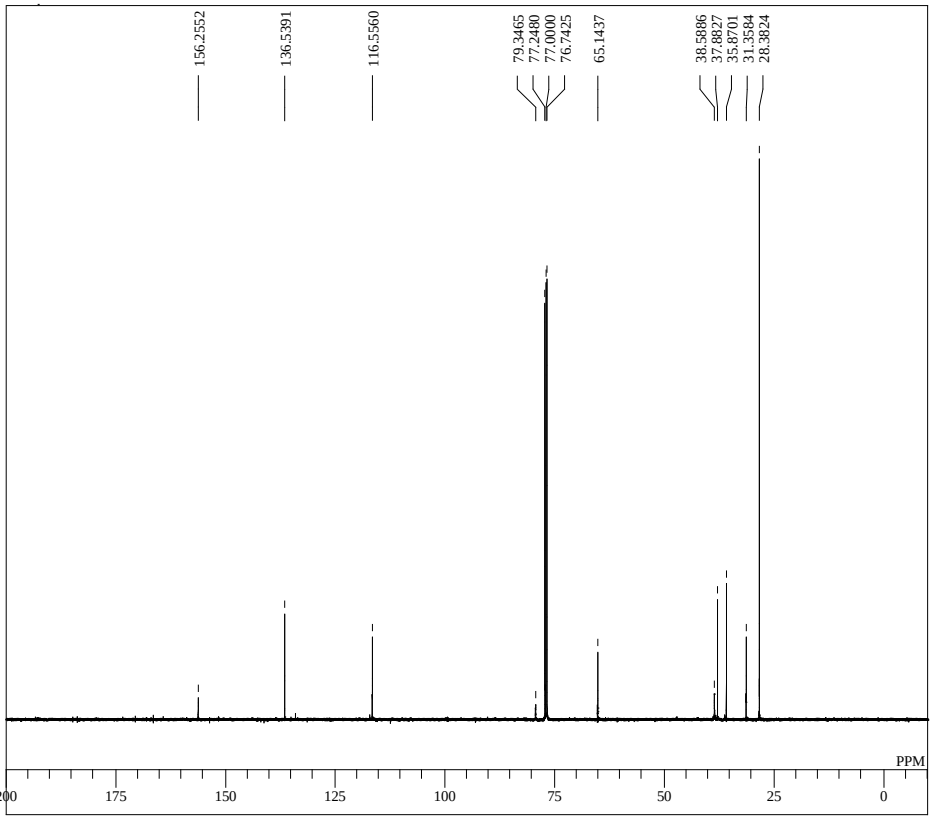


DFILE 2_DiAcSpd_step4_1H.als
COMNT single_pulse
DATIM 2018-04-10 18:12:02
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 13107
FREQU 6002.40 Hz
SCANS 16
ACQTM 2.1837 sec
PD 2.0000 sec
PW1 7.25 usec
IRNUC 1H
CTEMP 19.5 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 1.20 Hz
RGAIN 46

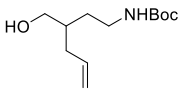


11

^{13}C NMR spectrum of **11**

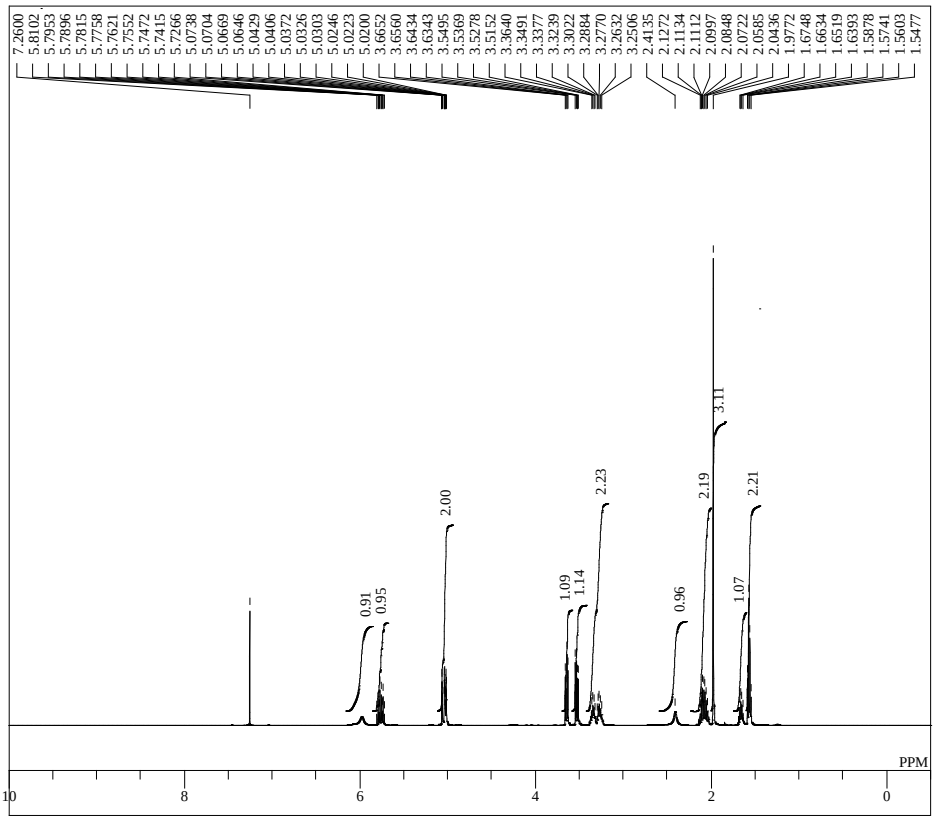


DFILE 2_DiAcSpd_step4_13C.als
COMNT single pulse decoupled gated NOE
DATIM 2018-04-10 20:09:35
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 600
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.93 usec
IRNUC 1H
CTEMP 20.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

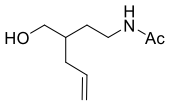


11

^{1}H NMR spectrum of **12**

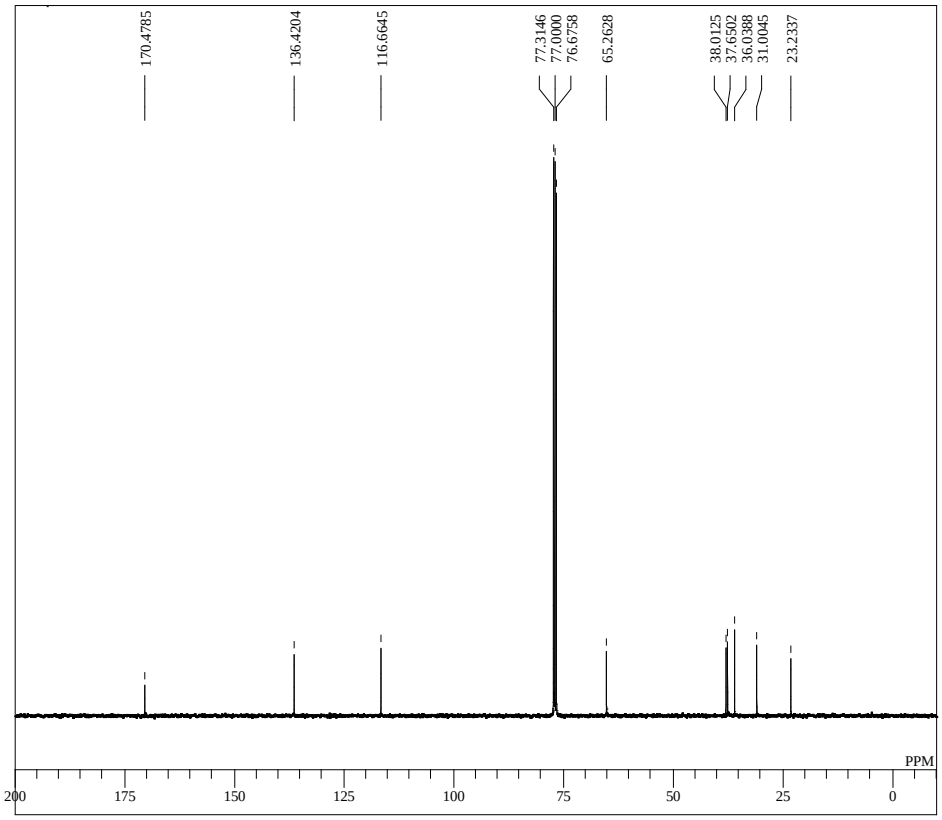


DFILE 3_DiAcSpd_step5_1H.als
COMNT single_pulse
DATIM 2018-04-13 15:34:33
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 16
ACQTM 1.7459 sec
PD 1.5000 sec
PW1 6.50 usec
IRNUC 1H
CTEMP 19.8 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 44

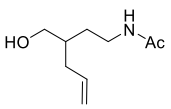


12

^{13}C NMR spectrum of **12**

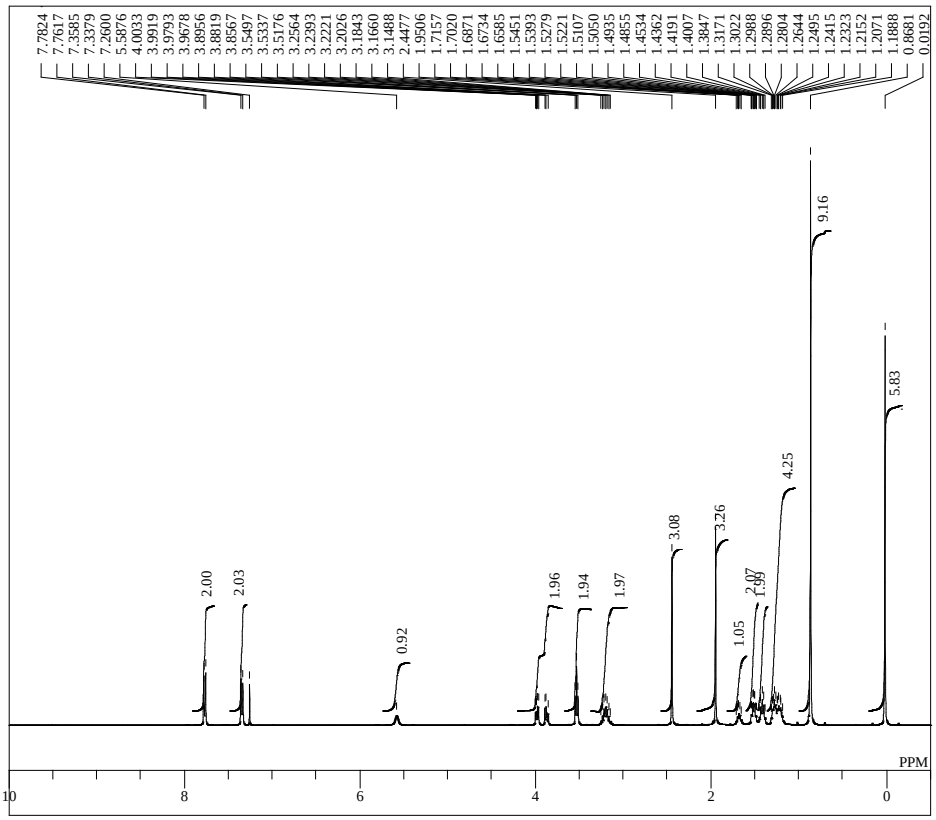


DFILE 3_DiAcSpd_step5_13C.als
COMNT single pulse decoupled gated NOE
DATIM 2018-04-13 16:51:25
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 26214
FREQU 25125.63 Hz
SCANS 400
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 3.17 usec
IRNUC 1H
CTEMP 19.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 60

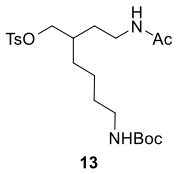


12

^{1}H NMR spectrum of **13**

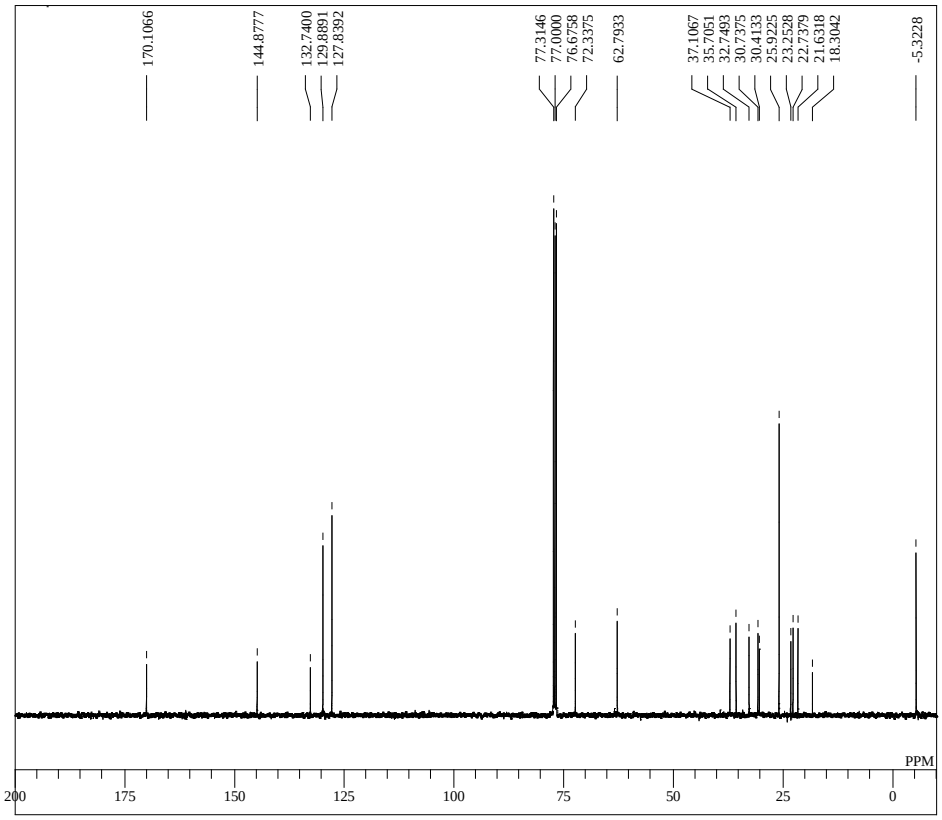


DFILE 4_DiAcSpd_step8_1H.als
COMNT single_pulse
DATIM 2018-04-12 18:07:24
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 13107
FREQU 6002.40 Hz
SCANS 16
ACQTM 2.1837 sec
PD 2.0000 sec
PW1 7.25 usec
IRNUC 1H
CTEMP 19.4 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 1.20 Hz
RGAIN 36

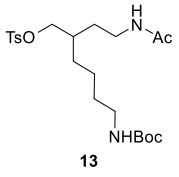


13

^{13}C NMR spectrum of **13**

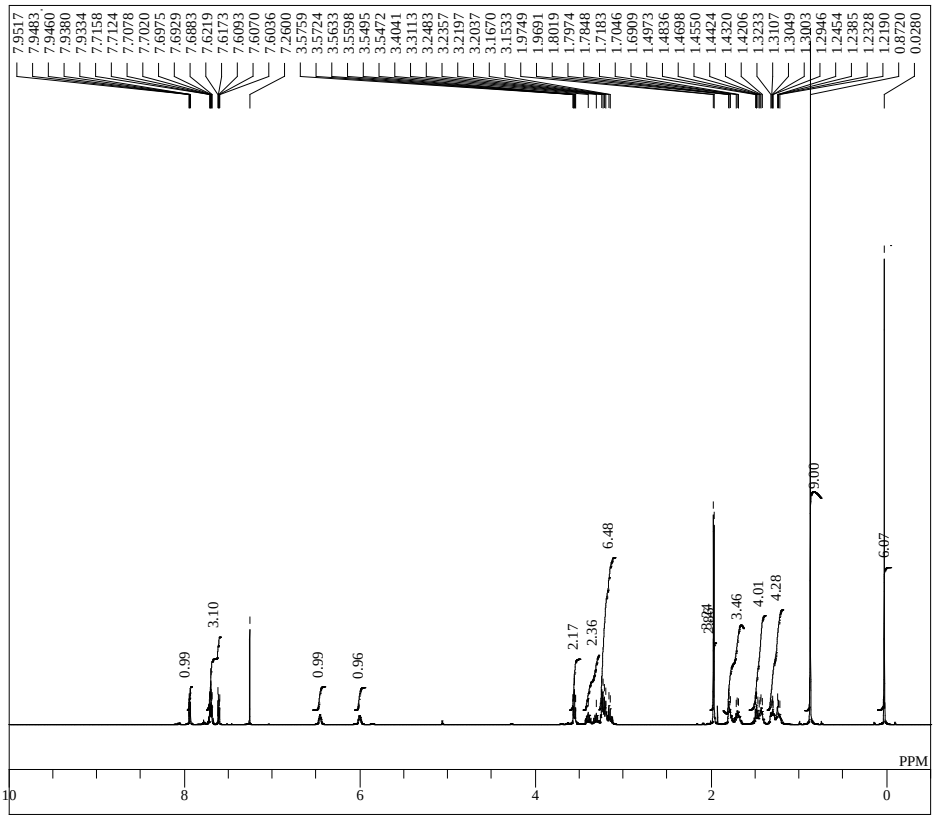


DFILE 4_DiAcSpd_step8_13C.als
COMNT single pulse decoupled gated NOE
DATIM 2018-04-12 18:10:58
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 26214
FREQU 25125.63 Hz
SCANS 150
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 3.17 usec
IRNUC 1H
CTEMP 19.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 60

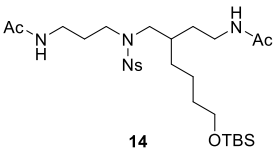


13

^{1}H NMR spectrum of **14**

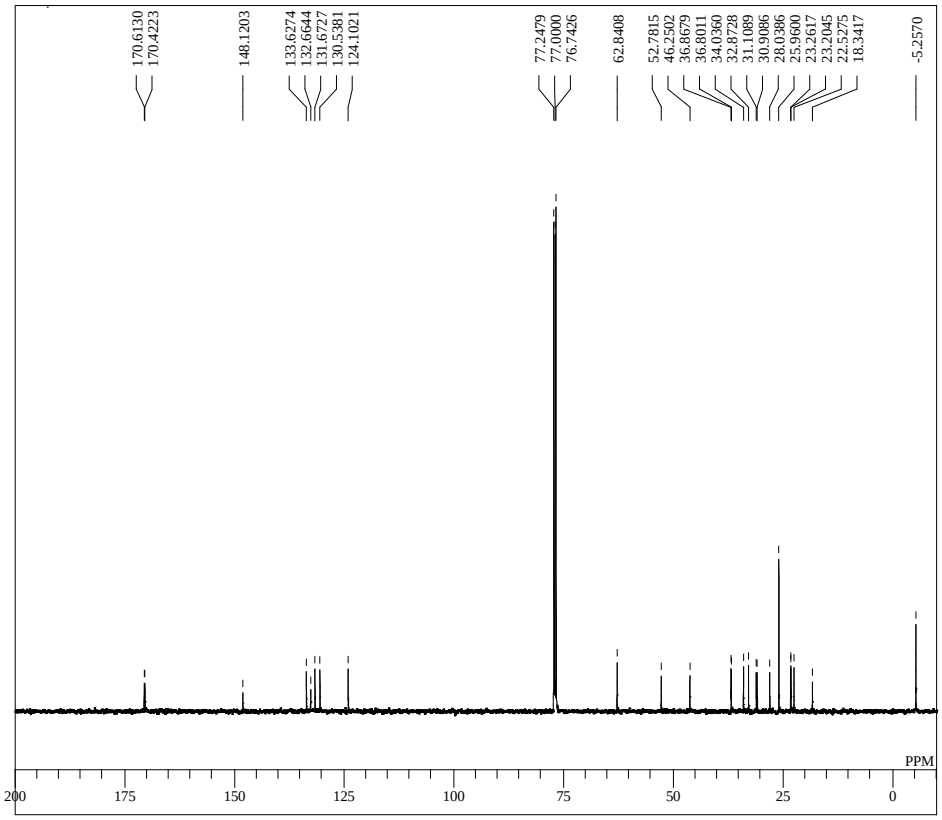


DFILE 5_DiAcSpd_step9_1H.als
COMNT single_pulse
DATIM 2018-04-10 13:29:49
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 32
ACQTM 1.7459 sec
PD 2.5000 sec
PW1 6.50 usec
IRNUC 1H
CTEMP 19.9 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 36

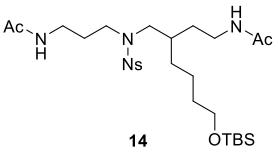


14

^{13}C NMR spectrum of **14**

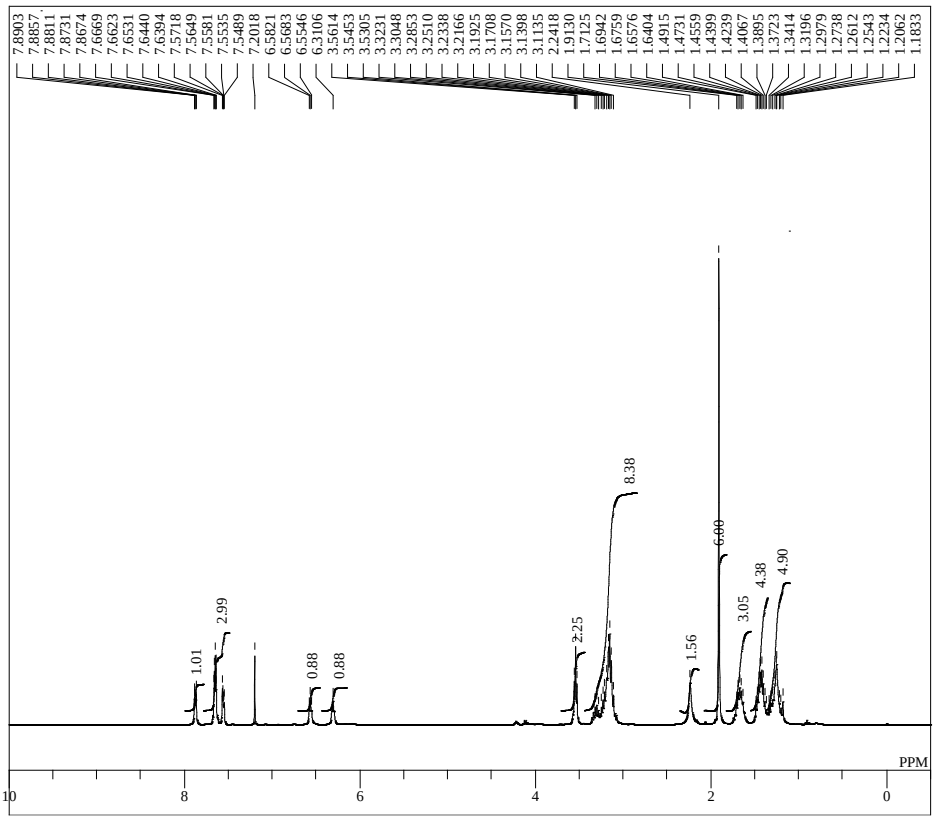


DFILE 5_DiAcSpd_step9_13C.als
COMNT single pulse decoupled gated NOE
DATIM 2018-04-08 03:01:23
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26224
FREQU 31446.54 Hz
SCANS 222
ACQTM 0.0000 sec
PD 2.0000 sec
PW1 3.93 usec
IRNUC 1H
CTEMP 20.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

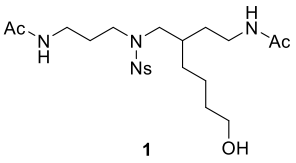


14

^{1}H NMR spectrum of **1**

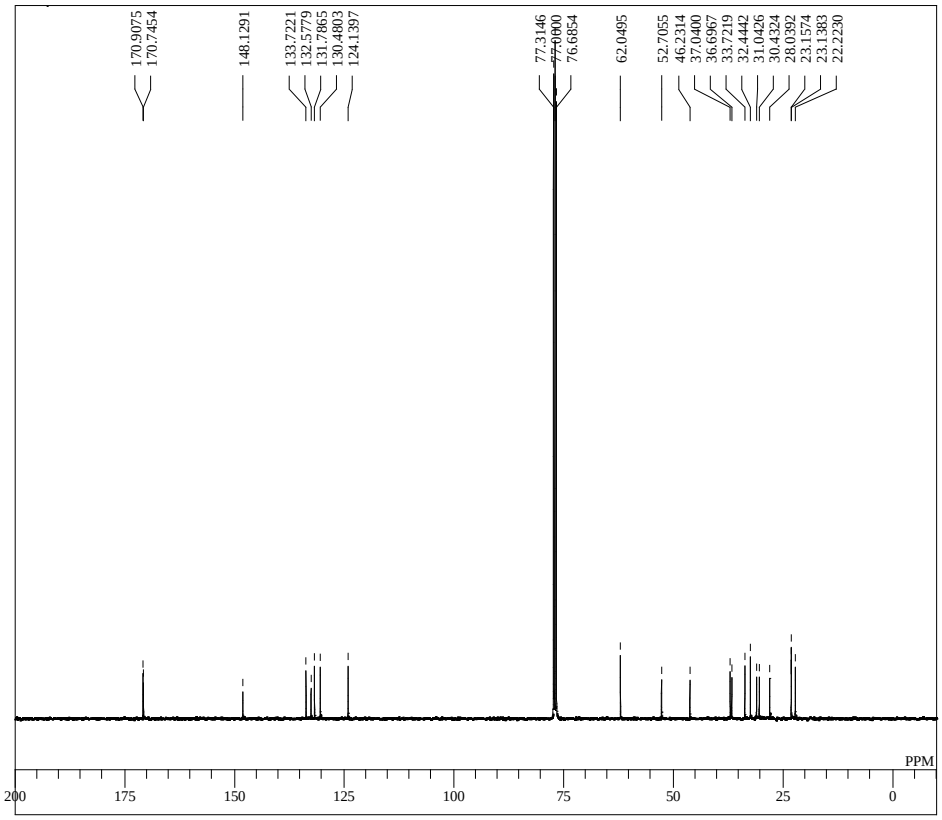


DFILE 6_DiAcSpd_step10_1H.als
COMNT single_pulse
DATIM 2018-04-09 21:08:06
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 13107
FREQU 6002.40 Hz
SCANS 14
ACQTM 2.1837 sec
PD 2.0000 sec
PW1 7.25 usec
IRNUC 1H
CTEMP 19.2 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 1.20 Hz
RGAIN 40

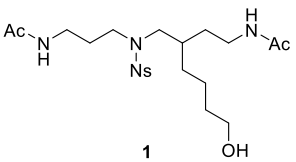


1

^{13}C NMR spectrum of **1**

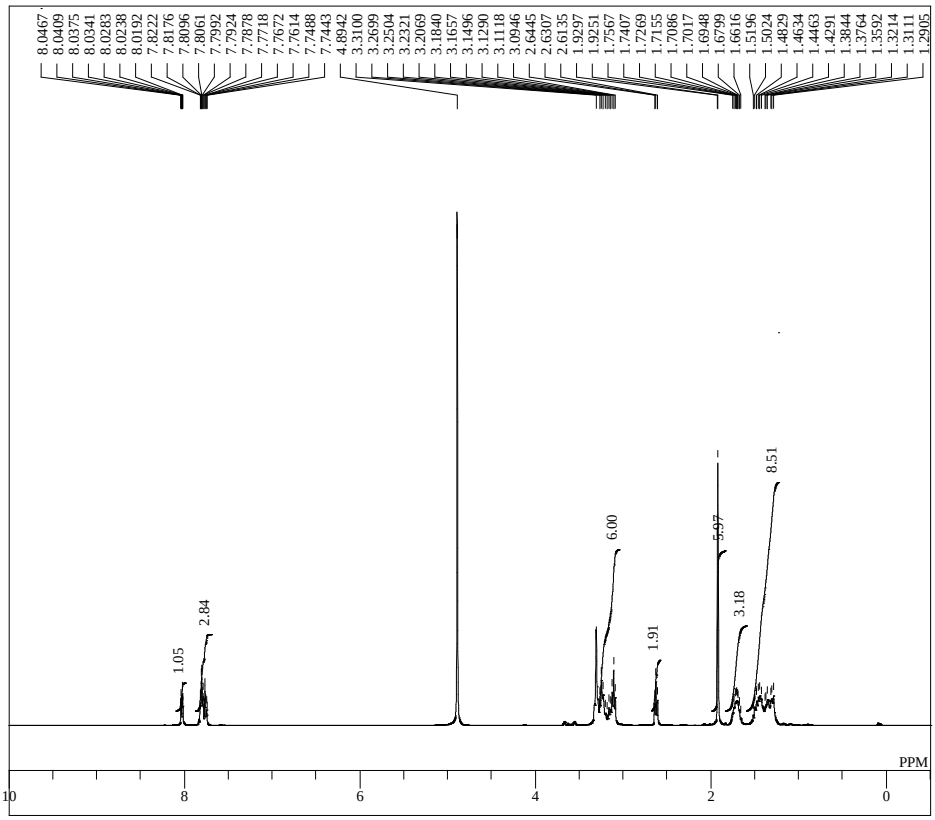


DFILE 6_DiAcSpd_step10_13C.als
COMNT single pulse decoupled gated NOE
DATIM 2018-04-09 21:15:16
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 26214
FREQU 25125.63 Hz
SCANS 901
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 3.17 usec
IRNUC 1H
CTEMP 19.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 60

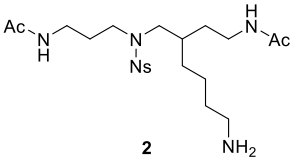


1

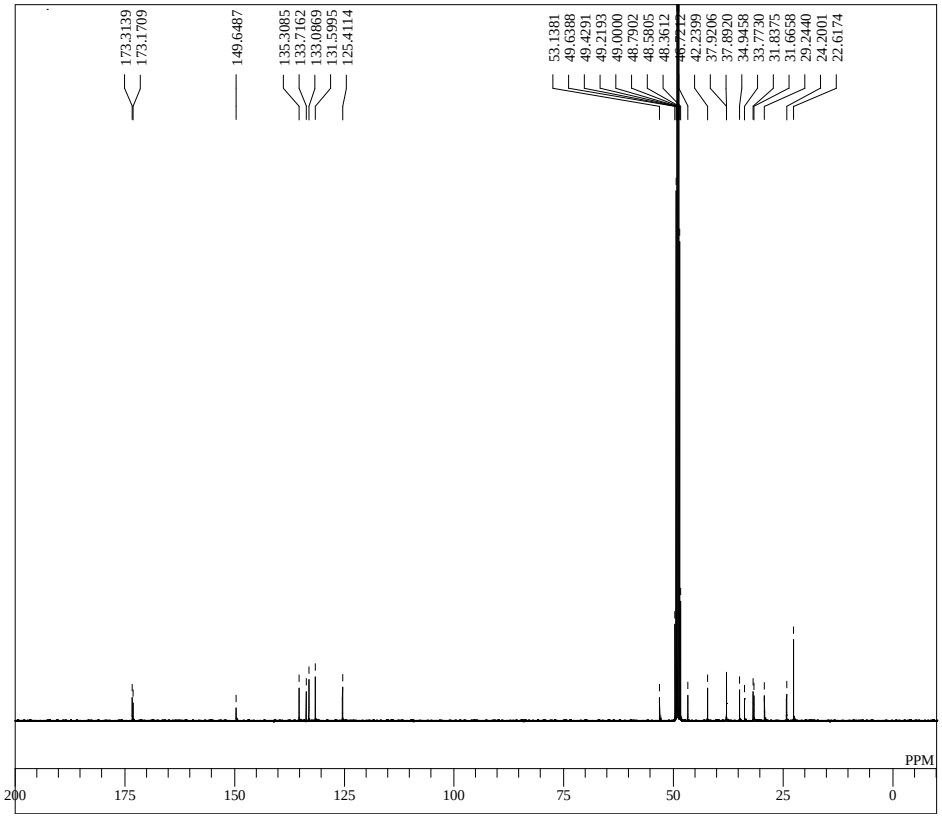
^{1}H NMR spectrum of **2**



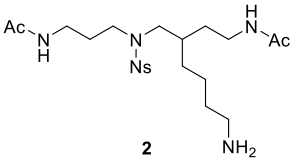
DFILE 7_DiAcSpd_step11_1H.als
COMNT single_pulse
DATIM 2018-06-04 01:07:26
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 13107
FREQU 6002.40 Hz
SCANS 16
ACQTM 2.1837 sec
PD 1.0000 sec
PW1 7.25 usec
IRNUC 1H
CTEMP 19.2 c
SLVNT CD3OD
EXREF 3.31 ppm
BF 0.12 Hz
RGAIN 38



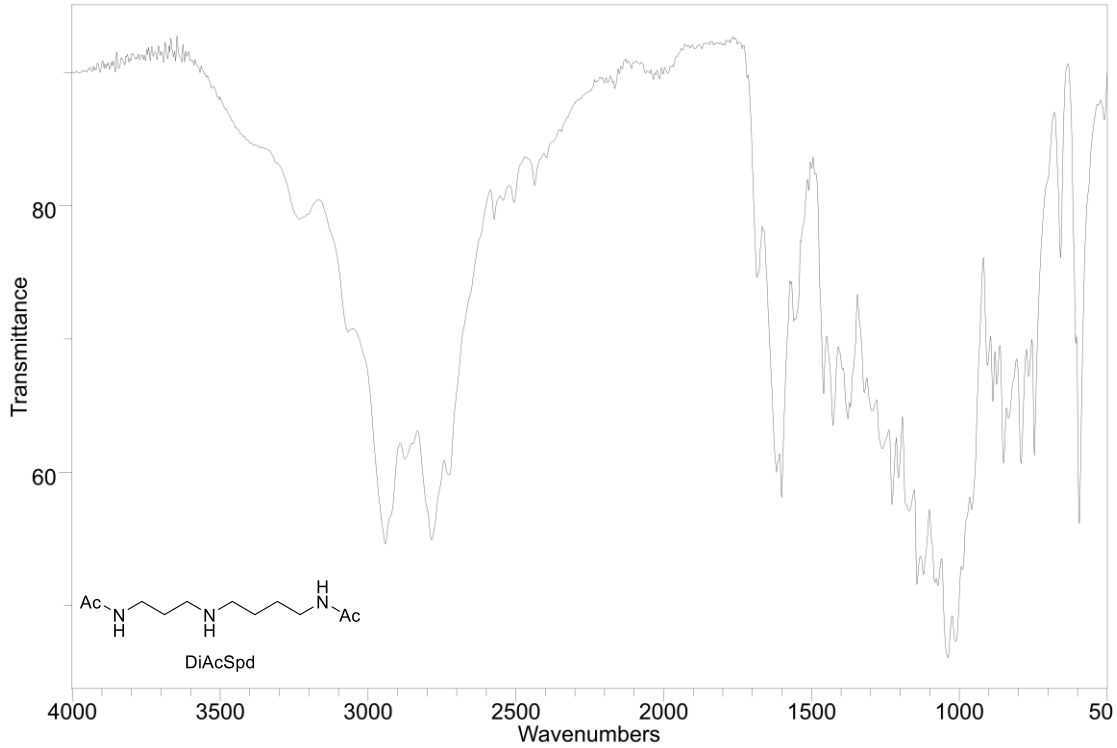
^{13}C NMR spectrum of **2**



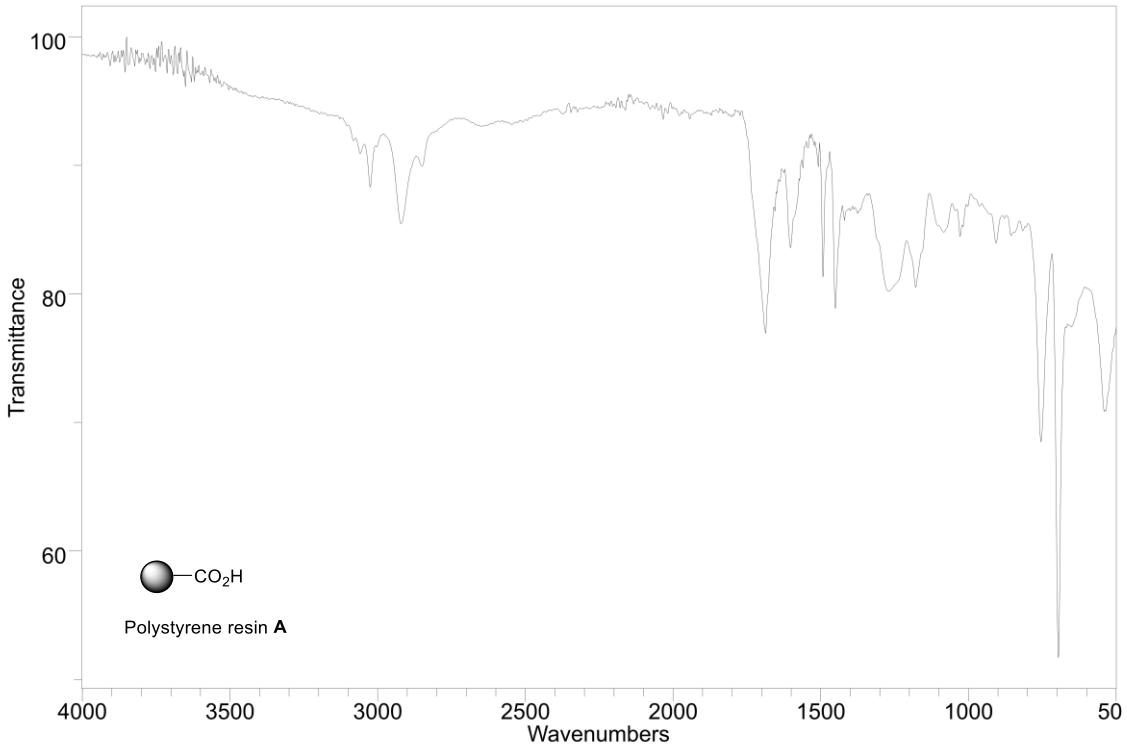
DFILE 7_DiAcSpd_step11_13C.als
COMNT single pulse decoupled gated NOE
DATIM 2018-06-04 01:13:22
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 26214
FREQU 25125.63 Hz
SCANS 1024
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 3.17 usec
IRNUC 1H
CTEMP 19.6 c
SLVNT CD3OD
EXREF 49.00 ppm
BF 0.12 Hz
RGAIN 60



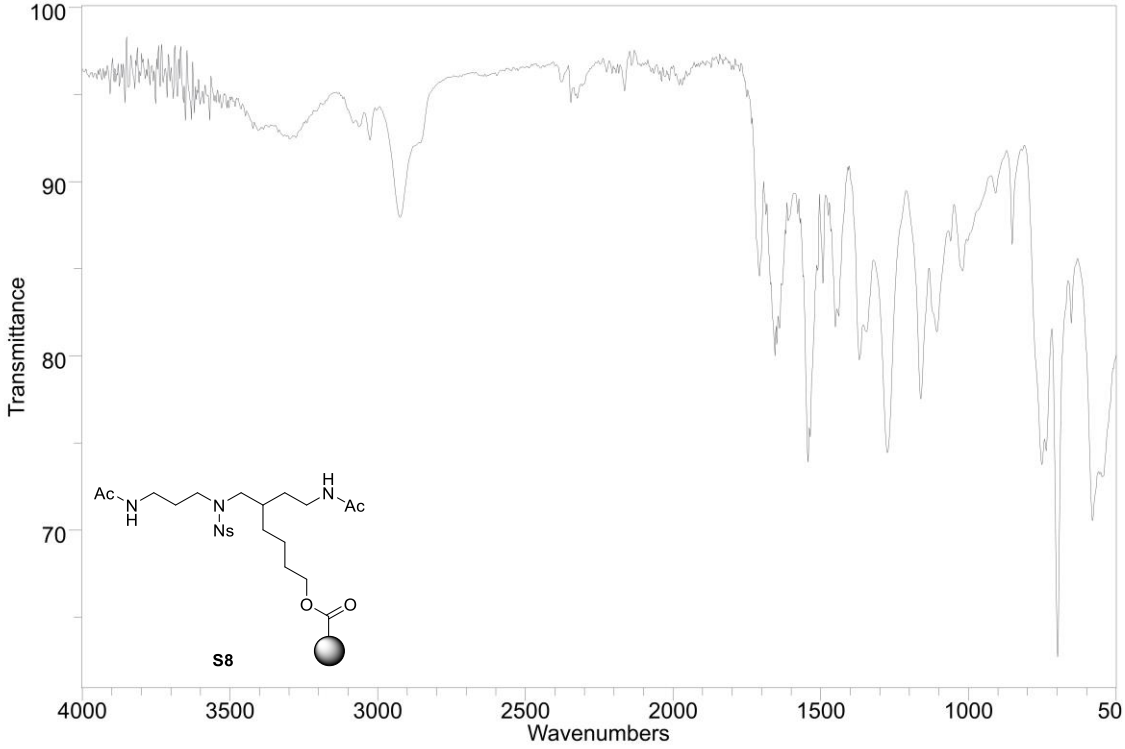
ATR-IR spectrum of DiAcSpd



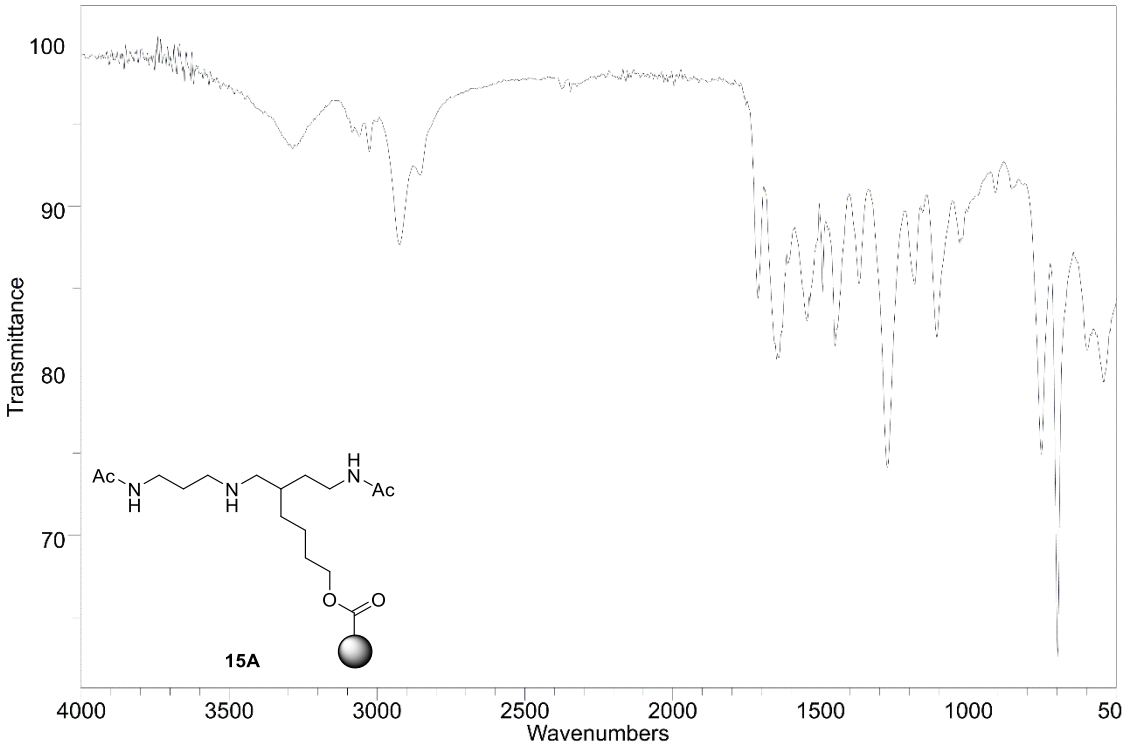
ATR-IR spectrum of resin A (polystyrene COOH resin)



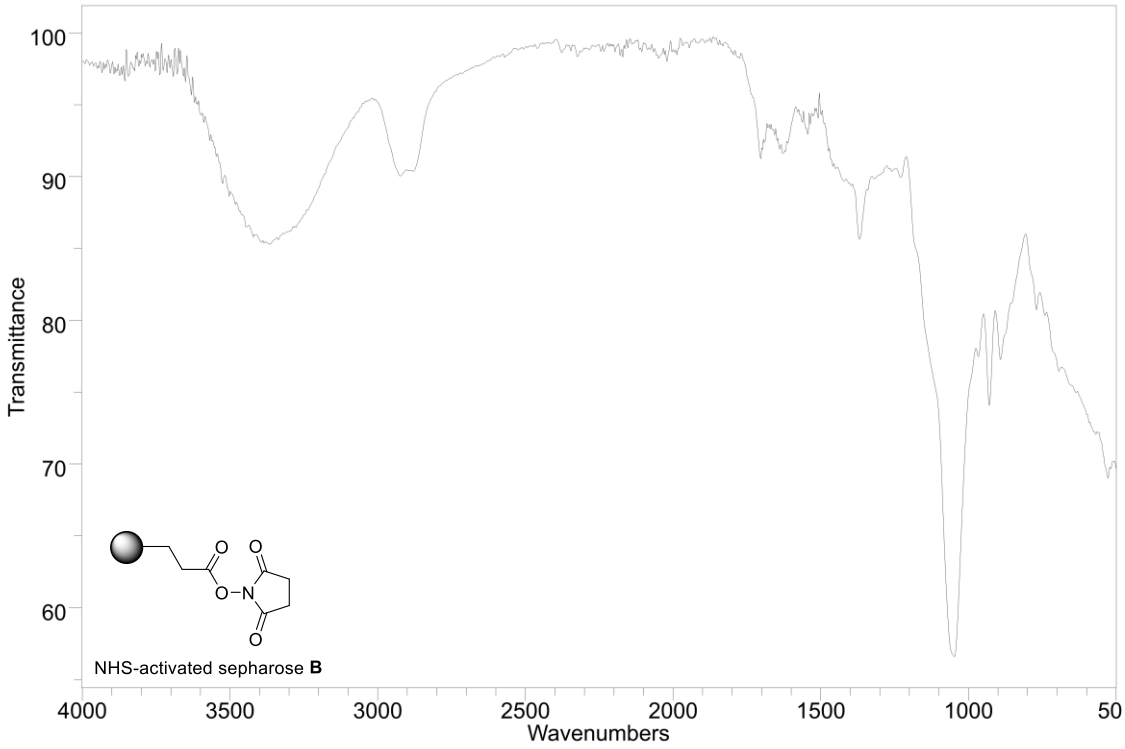
ATR-IR spectrum of **S8** (DiAcSpd-Ns immobilized on polystyrene COOH)



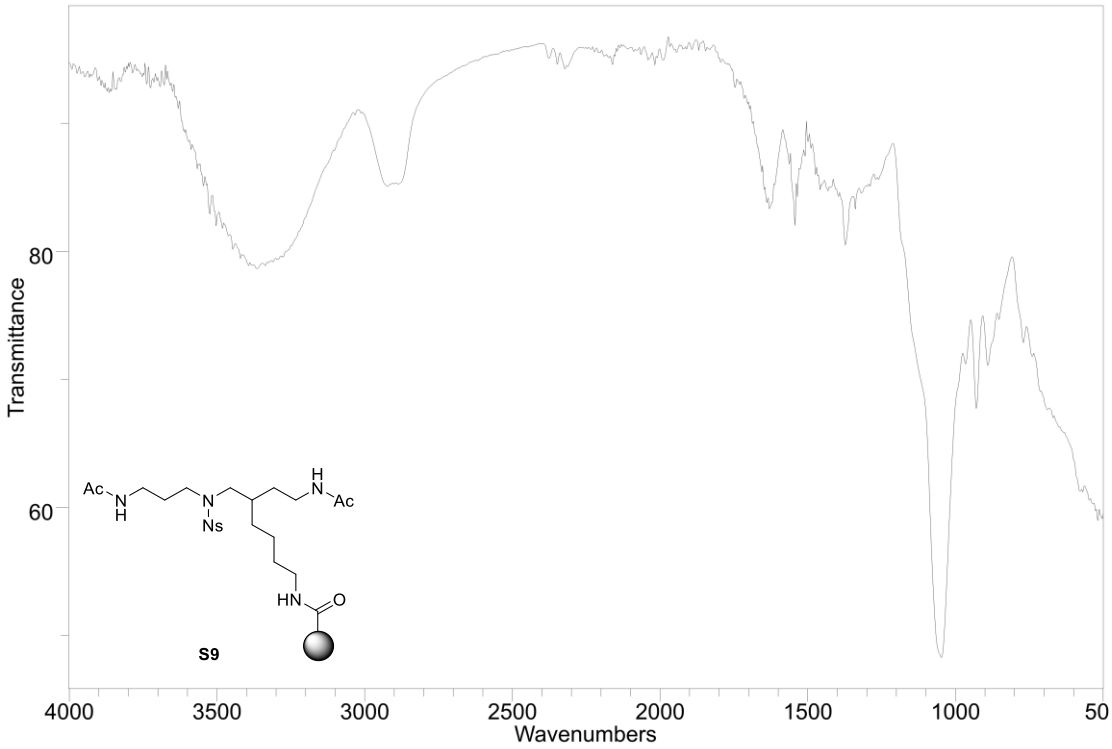
ATR-IR spectrum of **15A**



ATR-IR spectrum of resin B (NHS-activated sepharose)



ATR-IR spectrum of **S9** (DiAcSpd-Ns immobilized on NHS-activated sepharose)



ATR-IR spectrum of **15B**