Supporting Information

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1. Synthesis of Cross-linkers

1.1 General

All NMR spectra were recorded on a Bruker Avance 500 (^{1}H 500 MHz, ^{13}C 125 MHz) and on a Bruker Avance III HD (^{1}H 700 MHz, ^{13}C 175 MHz) in $CDCl_3$, MeOH-d_4 and CD_2Cl_2. The signals were referenced to the residual solvent signal and the peak assignment was supported by HSQC-, COSY-, and HMBC experiments. For easier comparison of NMR spectra, atom numbering may deviate from the IUPAC nomenclature. IR spectra were recorded on a Bruker FT-IR spectrometer Vektor 22 with MKII Golden Gate Single Reflection Diamant ATR system. Mass spectra (ESI) were measured on a Bruker Daltonics micro-TOF-Q spectrometer. Elemental analyses were recorded on a Carlo Erba Strumentazione Elemental Analyzer, Modell 1106. Melting points were obtained with a Olympus BX50 microscope with a Linkam TP93 temperature control. Column chromatography was performed using silica gel 60 by Fluka, grain size 40 - 63 µm. Merck Kieselgel 60 F254 plates (0.25 mm thickness on aluminium) were used for thin layer chromatography and the substances were visualized with permanganate reagent or phosphomolybdic acid solution. All chemicals were used as purchased unless otherwise stated. CH_2Cl_2 and NEt_3 were dried over CaH_2 and THF was dried over potassium by heating at reflux and subsequent distillation. Petroleum ether (PE), ethyl acetate and CH_2Cl_2 for column chromatography were distilled prior to use. Methyl iodide was freshly distilled.

All chemical syntheses reported here were carried out from the previously published diamines **C_n NH_2** (n = 4, 6, 8, 10).[1]

1.2 General Procedures

Synthesis of Neutral Acrylamide or Methacrylamide Cross-linkers (C_n AAm or C_n MeAAm, GP 1)
Following a procedure from Tominey,[2] a solution of the diamine **C_n NH_2**[1] (3.30 mmol, 1.00 equiv.) in abs. dichloromethane (120 mL) was treated alternately with abs. triethylamine (13.2 mmol, 4.00 equiv.) and acryloyl chloride or methacryloyl chloride (6.93 mmol, 2.10 equiv.) at 0 °C under N_2 atmosphere. The reaction mixture was left to stir at room temperature for 15 h. Afterwards, the solvent was evaporated under reduced pressure at 30 °C. The residue was taken up in THF and the insoluble $HNEt_3Cl$ was filtered off. The filtrate was concentrated and the crude product was purified via column chromatography on silica gel.

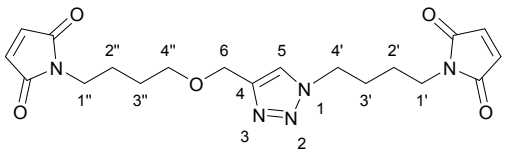
Synthesis of Neutral Vinylsulfonamide Cross-linkers (C_n VSAm, GP 2)
Following a procedure from Martinez,[3] a solution of the diamine **C_n NH_2**[1] (2.41 mmol, 1.00 equiv.) in abs. dichloromethane (100 mL) was treated alternately with abs. triethylamine (14.5 mmol, 6.00 equiv.) and 2-chloroethanesulfonyl chloride (5.06 mmol, 2.10 equiv.) at 0 °C under N_2 atmosphere. The reaction mixture was stirred at room temperature for 20 h. After evaporation of the solvent under reduced pressure at 30 °C, the residue was taken up in THF and the insoluble $HNEt_3Cl$ was filtered off. The filtrate was concentrated and the crude product was purified via column chromatography on silica gel.

Synthesis of Triazolium Iodide Cross-linkers (GP 3)
The respectrive triazole (0.20 mmol, 1.00 equiv.) was dissolved in acetonitrile or DMF (3 mL) and treated with iodomethane (4.00 mmol, 20.0 equiv.).[1] The reaction mixture was stirred at 30 °C until full

conversion was determined via TLC and ^{1}H-NMR. The solvent was evaporated and the triazolium crosslinkers used as obtained. If necessary, the products were purified via column chromatography on silica gel previously treated with diluted hydroiodic acid.

1.3 Maleimide Cross-linker C_4 Mal

1-[4-(4-{[4-(2,5-Dioxo-2,5-dihydro-1*H*-pyrrol-1-yl)butoxy]methyl}-1*H*-1,2,3-triazol-1-yl)-butyl]-1*H*-pyrrole-2,5-dione (C_4 Mal). The C_4 maleimide crosslinker was obtained following the previously published procedure.[1] Diamine **C_4 NH_2** (0.78 g, 3.22 mmol) was dissolved in EtOH (135 mL) and NEt_3 (1.07 mL, 0.78 g, 7.73 mmol) and maleic anhydride (0.76 g, 7.73 mmol) were added. The reaction mixture was refluxed for 6 h. After evaporation of the solvent, the residue was taken up in acetic anhydride (30 mL) and sodium acetate (0.88 g, 6.44 mmol) and stirred at 70 °C for 4 h and at room temperature for further 11 h. Then, H_2O (50 mL) and CH_2Cl_2 (50 mL) were added. The organic layer was washed with H_2O (2 x 50 mL), dried over $MgSO_4$ and concentrated. The crude product was obtained after column chromatography (SiO_2, PE/EtOAc 1:2, 1:4, 0:1), redissolved in MeOH and the insoluble solid was filtered off. The filtrate was evaporated to give the pure maleimide **C_4 Mal** as a colourless solid (0.34 g, 0.84 mmol, 26%). R_f = 0.51 (EtOAc, $KMnO_4$). ^{1}H-NMR (300 MHz, MeOH-d_4): δ = 1.49 – 1.69 (m, 6H, 2‘-H, 2’’-H, 3’’-H), 1.83 – 1.95 (m, 2H, 3‘-H), 3.45 – 3.57 (m, 6H, 1‘-H, 1‘‘-H, 4’’-H), 4.44 (t, J = 7.1 Hz, 2H, 4‘-H), 4.56 (s, 2H, 6-H), 6.79, 6.80 (2 s, 4H, CH_{Mal}), 7.96 (s, 1H, 5-H) ppm; ^{13}C-NMR (75 MHz, MeOH-d_4): δ = 26.3, 26.4, 27.8, 28.4 (C-2‘, C-2‘‘, C-3‘, C-3‘‘), 37.7, 38.3 (C-1’, C-1’’), 50.6 (C-4’), 64.7 (C-6), 70.7 (C-4’’), 125.0 (C-5), 135.3, 135.4 (*C*H$_{Mal}$), 146.2 (C-4), 172.5, 172.6 (C=O) ppm. The spectral data are in accordance with previously published values.[1]

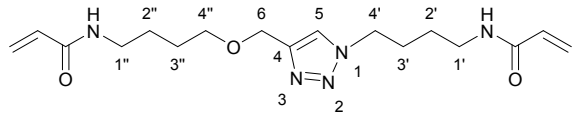


$C_{19}H_{23}N_5O_5$
401.42 g/mol

C_4 Mal

1.4 Neutral Acrylamide Cross-linkers C_nAAm

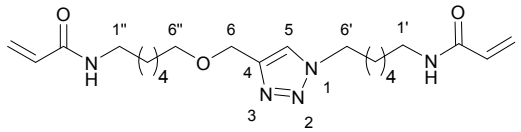
***N*-{4-[4-({[4-(Acryloylamino)butyl]oxy}methyl)-1*H*-1,2,3-triazol-1-yl]butyl}acrylamide (C_4 AAm).** According to GP 1, from diamine **C_4 NH_2** (1.15 g, 4.77 mmol), abs. NEt_3 (2.64 mL, 1.93 g, 19.1 mmol) and acryloyl chloride (0.81 mL, 0.91 g, 10.0 mmol) in abs. CH_2Cl_2 (160 mL), chromatography with CH_2Cl_2/MeOH 40:1, 20:1 to 10:1; yield: 0.84 g, 2.40 mmol, 50%, colourless solid, R_f = 0.43 (CH_2Cl_2/MeOH 10:1, $KMnO_4$). Mp. 105 °C. ^{1}H-NMR (500 MHz, $CDCl_3$): δ = 1.48 – 1.56 (m, 2H, 2‘-H), 1.56 – 1.65 (m, 4H, 2’’-H, 3’’-H), 1.89 – 1.97 (m, 2H, 3‘-H), 3.26 – 3.35 (m, 4H, 1‘-H, 1‘‘-H), 3.52 (t, J = 5.6 Hz, 2H, 4’’-H), 4.37 (t, J = 6.9 Hz, 2H, 4‘-H), 4.58 (s, 2H, 6-H), 5.56 – 5.61 (m, 2H, CH=CH_2), 6.06 – 6.15 (m, 2H, C*H*=CH$_2$), 6.19 – 6.27 (m, 2H, CH=CH_2), 6.38 (br s, 1H, N*H*), 6.56 (br s, 1H, N*H*), 7.57 (s, 1H, 5-H) ppm; ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 26.4, 26.6, 27.0, 27.7 (C-2‘, C-2‘‘, C-3‘, C-3‘‘), 38.7, 39.3 (C-1’, C-1’’), 49.9 (C-4’), 64.3 (C-6), 70.2 (C-4’’), 122.7 (C-5), 126.2, 126.4 (CH=*C*H$_2$), 131.0, 131.2 (*C*H=CH$_2$), 145.3 (C-4), 165.9, 166.0 (C=O) ppm. The spectral data are in accordance with previously published values.[4]



$C_{17}H_{27}N_5O_3$
349.44 g/mol

C_4 AAm

***N*-{6-[4-({[6-(Acryloylamino)hexyl]oxy}methyl)-1*H*-1,2,3-triazol-1-yl]hexyl}acrylamide (C_6 AAm).**

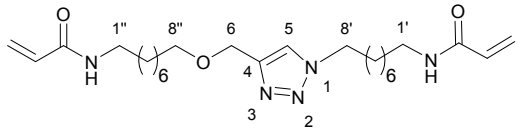


C_6 AAm

$C_{21}H_{35}N_5O_3$
405.54 g/mol

According to GP 1, from diamine **C_6 NH_2** (0.99 g, 3.32 mmol), abs. NEt_3 (1.84 mL, 1.34 g, 13.3 mmol) and acryloyl chloride (0.57 mL, 0.63 g, 6.97 mmol) in abs. CH_2Cl_2 (150 mL), chromatography with CH_2Cl_2/MeOH 40:1, 20:1 to 10:1; yield: 0.45 g, 1.10 mmol, 33%, colourless solid, R_f = 0.53 (CH_2Cl_2/MeOH 10:1, $KMnO_4$). Mp. 112 °C. ^{1}H-NMR (500 MHz, $CDCl_3$): δ = 1.25 – 1.39 (m, 8H, CH_2), 1.46 – 1.54 (m, 4H, 2'-H, 2''-H), 1.54 – 1.60 (m, 2H, 5''-H), 1.83 – 1.93 (m, 2H, 5'-H), 3.24 – 3.33 (m, 4H, 1'-H, 1''-H), 3.49 (t, *J* = 6.5 Hz, 2H, 6''-H), 4.33 (t, *J* = 7.0 Hz, 2H, 6'-H), 4.58 (s, 2H, 6-H), 5.55 –5.63 (m, 2H, CH=C*H*$_2$), 6.07 – 6.17 (m, 3H, C*H*=CH$_2$, N*H*), 6.18 – 6.28 (m, 3H, CH=C*H*$_2$, N*H*), 7.52 (s, 1H, 5-H) ppm. ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 25.9, 26.1, 26.2, 26.7, 29.4, 29.5 (2C), 30.2 (CH_2), 39.3, 39.6 (C-1', C-1''), 50.2 (C-6'), 64.3 (C-6), 70.6 (C-6''), 122.5 (C-5), 126.15, 126.24 (CH=*C*H$_2$), 131.1, 131.2 (*C*H=CH$_2$), 145.5 (C-4), 165.8, 165.9 (C=O) ppm. FT-IR (ATR, $CDCl_3$): $\tilde{\nu}$ = 3282 (b), 3077 (w), 2933 (s), 2860 (m), 1657 (vs), 1624 (s), 1549 (vs), 1462 (m), 1439 (m), 1408 (m), 1375 (w), 1316 (w), 1244 (m), 1097 (m), 1056 (m), 986 (m), 958 (w), 806 (w), 723 (w) cm^{-1}. MS (ESI): *m*/*z* = 428.26 [M + Na]$^+$, 400.26. HRMS (ESI): calcd. for [$C_{21}H_{35}N_5O_3Na$]$^+$ 428.2632, found 428.2630 [M + Na]$^+$. Elemental analysis: calcd. (%) for $C_{21}H_{35}N_5O_3$: C 62.20, H 8.70, N 17.27; found: C 62.07, H 8.40, N 17.08.

***N*-{8-[4-({[8-(Acryloylamino)octyl]oxy}methyl)-1*H*-1,2,3-triazol-1-yl]octyl}acrylamide (C_8 AAm).**

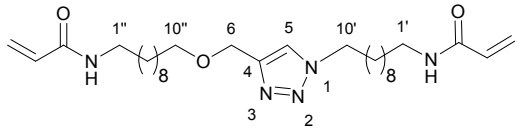


C_8 AAm

$C_{25}H_{43}N_5O_3$
461.65 g/mol

According to GP 1, from diamine **C_8 NH_2** (0.69 g, 1.94 mmol), abs. NEt_3 (1.08 mL, 0.79 g, 7.76 mmol) and acryloyl chloride (0.33 mL, 0.37 g, 4.07 mmol) in abs. CH_2Cl_2 (60 mL), chromatography with CH_2Cl_2/MeOH 40:1 to 10:1; yield: 0.28 g, 0.60 mmol, 31%, colourless solid, R_f = 0.55 (CH_2Cl_2/MeOH 10:1, $KMnO_4$). Mp. 98 °C. ^{1}H-NMR (500 MHz, $CDCl_3$): δ = 1.25 – 1.35 (m, 16H, CH_2), 1.47 – 1.54 (m, 4H, CH_2), 1.56 – 1.61 (m, 2H, CH_2), 1.84 – 1.91 (m, 2H, CH_2), 3.27 – 3.32 (m, 4H, 1'-H, 1''-H), 3.50 (t, *J* = 6.7 Hz, 2H, 8''-H), 4.33 (t, *J* = 7.4 Hz, 2H, 8'-H), 4.60 (s, 2H, 6-H), 5.59 – 5.63 (m, 2H, CH=C*H*$_2$), 5.93 (br s, 2H, N*H*), 6.07 – 6.14 (m, 2H, C*H*=CH$_2$), 6.23 – 6.29 (m, 2H, CH=C*H*$_2$), 7.52 (s, 1H, 5-H) ppm. ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 26.1, 26.4, 26.8, 26.9, 28.8, 29.0, 29.2, 29.3, 29.56, 29.58, 29.7, 30.3 (CH_2), 39.6, 39.7 (C-1', C-1''), 50.4 (C-8'), 64.4 (C-6), 70.8 (C-8''), 122.4 (C-5), 126.19, 126.23 (2 x CH=*C*H$_2$), 131.1, 131.2 (2 x *C*H=CH$_2$), 145.5 (C-4), 165.72, 165.73 (2 x C=O) ppm. FT-IR (ATR, $CDCl_3$): $\tilde{\nu}$ = 3298 (br), 3077 (w), 2925 (s), 2852 (m), 1653 (s), 1623 (s), 1623 (s), 1541 (s), 1470 (m), 1408 (m), 1379 (w), 1334 (w), 1312 (w), 1254 (w), 1237 (m), 1153 (w), 1114 (m), 1055 (m), 989 (m), 953 (m), 919 (w), 853 (w), 806 (w), 723 (w), 680 (w) cm^{-1}. MS (ESI): *m*/*z* = 484.33 [M + Na]$^+$, 456.32. HRMS (ESI): calcd. for [$C_{25}H_{43}N_5O_3Na$]$^+$ 484.3258, found 484.3254 [M + Na]$^+$. Elemental analysis: calcd. (%) for $C_{23}H_{43}N_5O_3$: C 65.04, H 9.39, N 15.17; found: C 64.85, H 9.22, N 14.99.

***N*-{10-[4-({[10-(Acryloylamino)decyl]oxy}methyl)-1*H*-1,2,3-triazol-1-yl]decyl}acrylamide (C_{10} AAm).** According to GP 1, from diamine C_{10} NH_2 (0.67 g, 1.62 mmol), abs. NEt_3 (0.90 mL, 0.66 g, 6.49 mmol) and acryloyl chloride (0.28 mL, 0.31 g, 3.41 mmol) in abs. CH_2Cl_2 (60 mL), chromatography with CH_2Cl_2/MeOH 80:1 to 10:1; yield: 0.23 g, 0.49 mmol, 30%, colourless solid, R_f = 0.57 (CH_2Cl_2/MeOH 10:1, $KMnO_4$). Mp. 108 °C. ^{1}H-NMR (500 MHz, $CDCl_3$): δ = 1.23 – 1.33 (m, 24H, CH_2), 1.48 – 1.55 (m, 4H, CH_2), 1.56 - 1.61 (m, 2H, CH_2), 1.85 – 1.92 (m, 2H, CH_2), 3.28 – 3.34 (m, 4H, 1'-H, 1''-H), 3.50 (t, J = 6.7 Hz, 2H, 10''-H), 4.33 (t, J = 7.4 Hz, 2H, 10'-H), 4.60 (s, 2H, 6-H), 5.58 – 5.63 (m, 2H, CH=CH_2), 5.82 (br s, 1H, N*H*), 5.87 (br s, 1H, N*H*), 6.06 – 6.14 (m, 2H, C*H*=CH_2), 6.22 – 6.29 (m, 2H, CH=CH_2), 7.51 (s, 1H, 5-H) ppm. ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 26.2, 26.5, 26.9, 27.0, 28.9, 29.2, 29.26, 29.32, 29.33, 29.45, 29.48, 29.52, 29.61, 29.64, 29.7, 30.3 (CH_2), 39.70, 39.74 (C-1', C-1''), 50.4 (C-10'), 64.5 (C-6), 70.9 (C-10''), 122.3 (C-5), 126.2 (2 x CH=*C*H_2), 131.1 (2 x *C*H=CH_2), 145.6 (C-4), 165.7 (2 x C=O) ppm. FT-IR (ATR, $CDCl_3$): $\tilde{\nu}$ = 3298 (br), 3077 (w), 2919 (s), 2851 (s), 1654 (s), 1624 (s), 1542 (s), 1471 (m), 1408 (m), 1379 (w), 1313 (w), 1244 (m), 1231 (w), 1155 (w), 990 (w), 954 (w), 922 (w), 806 (w), 721 (w), 681 (w) cm^{-1}. MS (ESI): *m*/*z* = 540.39 [M + Na]$^+$. HRMS (ESI): calcd. for [$C_{29}H_{51}N_5O_3$Na]$^+$ 540.3884, found 540.3873 [M + Na]$^+$. Elemental analysis: calcd. (%) for $C_{29}H_{51}N_5O_3$: C 67.27, H 9.93, N 13.53; found: C 66.79, H 9.32, N 13.35.

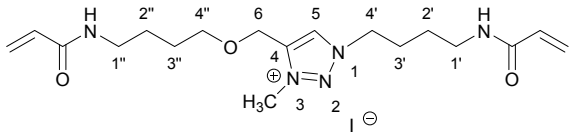


$C_{29}H_{51}N_5O_3$
517.76 g/mol

C_{10} AAm

1.5 Triazolium-Acrylamide Cross-linkers Me-C_n^+ I^- AAm

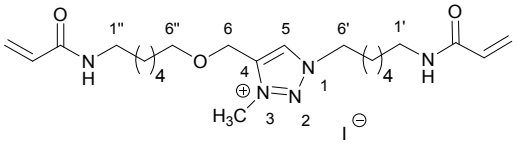
3-[4-(Acryloylamino)butyl]-5-({[4-(acryloylamino)butyl]oxy}methyl)-1-methyl-1*H*-1,2,3-triazol-3-ium iodide (Me-C_4^+ I^- AAm). Following GP 3, from triazole C_4 **AAm** (70 mg, 0.20 mmol) and CH_3I (0.25 mL, 0.57 g, 4.00 mmol) in CH_3CN (3 mL), 4 d at 30 °C, yield: 92 mg, 0.19 mmol, 94%, yellow oil, R_f = 0.16 (CH_2Cl_2/MeOH 10:1, $KMnO_4$). ^{1}H-NMR (700 MHz, CD_2Cl_2): δ = 1.60 – 1.72 (m, 6H, 2'-H, 2''-H, 3''-H), 2.09 – 2.15 (m, 2H, 3'-H), 3.26 – 3.30 (m, 2H, 1'-H or 1''-H), 3.32 – 3.37 (m, 2H, 1''-H or 1'-H), 3.65 (t, J = 6.0 Hz, 2H, 4''-H), 4.31 (s, 3H, NCH_3), 4.74 (t, J = 7.3 Hz, 2H, 4'-H), 4.84 (s, 2H, 6-H), 5.54 – 5.58 (m, 2H, CH=CH_2), 6.15 – 6.22 (m, 2H, CH=CH_2), 6.29 – 6.41 (m, 2H, C*H*=CH_2), 6.84 (br s, 1H, N*H*), 7.35 (br s, 1H, N*H*), 9.27 (s, 1H, 5-H) ppm. ^{13}C-NMR (175 MHz, CD_2Cl_2): δ = 26.0, 26.3, 27.0, 27.3 (C-2', C-2'', C-3', C-3''), 38.2, 39.2 (C-1', C-1''), 39.5 (N*C*H_3), 54.3 (C-4'), 61.2 (C-6), 71.9 (C-4''), 125.60, 125.64 (CH=*C*H_2), 130.6 (C-5), 132.0 (2 x *C*H=CH_2), 141.5 (C-4), 165.9, 166.2 (C=O) ppm. The spectral data are in accordance with previously published values.[4]



$C_{18}H_{30}IN_5O_3$
491.37 g/mol

Me-C_4^+ I^- AAm

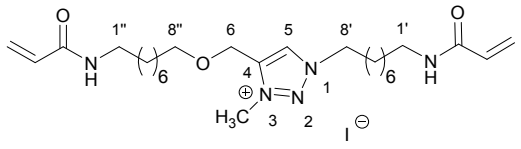
1-[6-(Acryloylamino)hexyl]-4-({[6-(acryloylamino)hexyl]oxy}methyl)-3-methyl-1*H*-1,2,3-triazol-3-ium iodide (Me-C_6^+ I^- AAm). Following GP 3, from triazole **C_6 AAm** (70.0 mg, 0.17 mmol) and CH_3I (0.49 g, 0.21 mL, 3.45 mmol) in CH_3CN (5 mL), 10 d at 30 °C, yield: 92 mg, 0.168 mmol, 97%, yellow solid, R_f = 0.23 (CH_2Cl_2/MeOH 10:1, $KMnO_4$), Mp. 102 °C. ^{1}H-NMR (500 MHz, CD_3OD): δ = 1.34 – 1.48 (m, 8H, CH_2), 1.51 – 1.61 (m, 4H, CH_2), 1.61 - 1.70 (m, 2H, 5''-H), 1.99 – 2.08 (m, 2H, 5'-H), 3.22 – 3.28 (m, 4H, 1'-H, 1''-H), 3.62 (t, *J* = 6.3 Hz, 2H, 6''-H), 4.31 (s, 3H, NCH_3), 4.63 (t, *J* = 7.1 Hz, 2H, 6'-H), 4.80 (s, 2H, 6-H), 5.62 – 5.67 (m, 2H, CH=CH_2), 6.17 – 6.28 (m, 4H, CH=CH_2, CH=CH_2), 8.76 (s, 1H, 5-H) ppm. ^{13}C-NMR (125 MHz, CD_3OD): δ = 26.68, 26.73, 27.1, 27.7, 30.0, 30.2, 30.3, 30.4 (8 x CH_2), 38.9 (NCH$_3$), 40.0, 40.2 (C-1', C-1''), 55.0 (C-6'), 61.5 (C-6), 72.7 (C-6''), 126.47, 126.52 (CH=CH$_2$), 130.4 (C-5), 132.11, 132.14 (CH=CH$_2$), 142.6 (C-4), 168.08, 168.11 (C=O) ppm. FT-IR (ATR, CD_2Cl_2): $\tilde{\nu}$ = 3449 (br), 3277 (br), 3066 (m), 2931 (s), 2859 (m), 1659 (vs), 1624 (s), 1542 (s), 1457 (m), 1407 (m), 1358 (w), 1315 (m), 1242 (m), 1098 (m), 988 (m), 961 (m), 806 (m), 656 (w) cm^{-1}. MS (ESI): *m*/*z* = 420.29 $[M]^+$. HRMS (ESI): calcd. for $[C_{22}H_{38}N_5O_3]^+$ 420.2969, found 420.2946 $[M]^+$. Elemental analysis: calcd. (%) for $C_{22}H_{38}IN_5O_3$: C 48.26, H 7.00, N 12.79; found: C 48.12, H 6.72, N 12.62.



Me-C_6^+ I^- AAm

$C_{22}H_{38}IN_5O_3$
547.48 g/mol

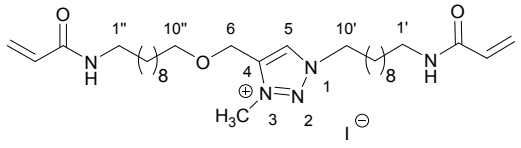
1-[8-(Acryloylamino)octyl]-4-({[8-(acryloylamino)decyl]oxy}methyl)-3-methyl-1*H*-1,2,3-triazol-3-ium iodide (Me-C_8^+ I^- AAm). Following GP 3, from triazole **C_8 AAm** (70.0 mg, 0.15 mmol) and CH_3I (0.19 mL, 0.43 g, 3.04 mmol) in CH_3CN (1 mL), 8 d at 30 °C, yield: 87 mg, 0.14 mmol, 93%, yellow solid, R_f = 0.25 (CH_2Cl_2/MeOH 10:1, $KMnO_4$), Mp. 99 °C. ^{1}H-NMR (500 MHz, CD_2Cl_2): δ = 1.29 – 1.41 (m, 16H, CH_2), 1.49 – 1.56 (m, 4H, CH_2), 1.57 - 1.64 (m, 2H, CH_2), 2.00 – 2.07 (m, 2H, CH_2), 3.22 – 3.27 (m, 4H, 1'-H, 1''-H), 3.61 (t, *J* = 6.7 Hz, 2H, 8''-H), 4.33 (s, 3H, NCH_3), 4.69 (t, *J* = 7.4 Hz, 2H, 8'-H), 4.86 (s, 2H, 6-H), 5.54 – 5.58 (m, 2H, CH=CH_2), 6.14 – 6.34 (m, 4H, CH=CH_2, CH=CH_2), 6.49 (br s, 1H, NH), 6.70 (br s, 1H, NH), 9.29 (s, 1H, 5-H) ppm. ^{13}C-NMR (125 MHz, CD_2Cl_2): δ = 26.2, 26.3, 27.0, 27.2, 28.9, 29.0, 29.48, 29.50, 29.7, 29.75, 29.77, 29.9 (CH_2), 39.65 (NCH_3), 39.69, 39.9 (C-1', C-1''), 54.9 (C-8'), 61.1 (C-6), 72.6 (C-8''), 125.6, 125.7 (2 x CH=CH$_2$), 130.8 (C-5), 132.1, 132.3 (2 x CH=CH$_2$), 141.3 (C-4), 165.9, 166.0 (2 x C=O) ppm. FT-IR (ATR, CD_2Cl_2): $\tilde{\nu}$ = 3455 (br), 3261 (br), 3065 (br), 2927 (s), 2855 (m), 1657 (s), 1623 (s), 1538 (s), 1460 (m), 1405 (m), 1357 (w), 1315 (m), 1239 (m), 1172 (w), 1100 (m), 988 (m), 955 (m), 806 (m), 708 (m), 654 (w), 483 (w) cm^{-1}. MS (ESI): *m*/*z* = 476.36 $[M]^+$. HRMS (ESI): calcd. for $[C_{26}H_{46}N_5O_3]^+$ 476.3595, found 476.3586 $[M]^+$. Elemental analysis: calcd. (%) for $C_{26}H_{46}IN_5O_3$: C 51.74, H 7.68, N 11.60; found: C 51.76, H 7.48, N 11.17.



Me-C_8^+ I^- AAm

$C_{26}H_{46}IN_5O_3$
603.59 g/mol

1-[10-(Acryloylamino)decyl]-4-({[10-(acryloylamino)decyl]oxy}methyl)-3-methyl-1*H*-1,2,3-triazol-3-ium iodide (Me-C_{10}^+ I^- AAm). Following GP 3, from triazole **C_{10} AAm** (70.0 mg, 0.14 mmol) and CH_3I (0.17 mL, 0.38 g, 2.70 mmol) in DMF (1 mL), 4 d at 30 °C, chromatography on HI-silica with CH_2Cl_2/MeOH 10:1; yield: 17 mg, 25.8 µmol, 18%, yellow solid, R_f = 0.21 (CH_2Cl_2/MeOH 10:1, $KMnO_4$),



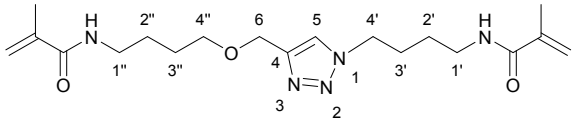
Me-C_{10}^+ I^- AAm

$C_{30}H_{54}IN_5O_3$
659.70 g/mol

Mp. 92 °C. ^{1}H-NMR (500 MHz, $CDCl_3$): δ = 1.24 – 1.37 (m, 24H, CH_2), 1.50 – 1.57 (m, 4H, CH_2), 1.58 - 1.63 (m, 2H, CH_2), 2.01 – 2.08 (m, 2H, CH_2), 3.26 – 3.34 (m, 4H, 1‘-H, 1‘‘-H), 3.61 (t, *J* = 6.7 Hz, 2H, 10’’-H), 4.36 (s, 3H, NCH_3), 4.70 (t, *J* = 7.4 Hz, 2H, 10’-H), 4.90 (s, 2H, 6-H), 5.57 – 5.63 (m, 2H, CH=CH_2), 6.04 – 6.28 (m, 6H, N*H*, C*H*=CH_2, CH=CH_2), 9.25 (s, 1H, H-5) ppm. ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 25.96, 26.04, 26.8, 26.9, 28.7, 28.98, 29.03, 29.1, 29.20, 29.23, 29.3, 29.37, 29.44, 29.5, 29.6 (CH_2), 39.4 (NCH_3), 39.6, 39.7 (C-1’, C-1’’), 54.6 (C-10’), 60.9 (C-6), 72.4 (C-10’’), 126.1, 126.2 (CH=*C*H_2), 130.5 (C-5), 131.2, 131.3 (*C*H=CH_2), 140.8 (C-4), 165.8, 165.9 (C=O) ppm. FT-IR (ATR, $CDCl_3$): $\tilde{\nu}$ = 3441 (br), 3266 (br), 3065 (w), 2925 (s), 2853 (s), 2763 (w), 1656 (s), 1622 (s), 1540 (s), 1462 (m), 1406 (m), 1371 (w), 1315 (w), 1242 (m), 1169 (w), 1100 (m), 1021 (w), 987 (w), 957 (w), 917 (m), 806 (m), 726 (s), 643 (m), 483 (w) cm^{-1}. MS (ESI): *m*/*z* = 532.42 $[M]^+$, 252.21, 156.18. HRMS (ESI): calcd. for $[C_{30}H_{54}N_5O_3]^+$ 532.4221, found 532.4223 $[M]^+$.

1.6 Neutral Methacrylamide Cross-linkers C_n MeAAm

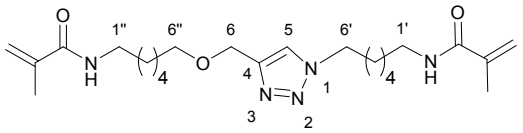
***N*-{4-[4-({[4-(Methacryloylamino)butyl]oxy}methyl)-1*H*-1,2,3-triazol-4-yl]butyl}-2-methylacrylamide (C_4 MeAAm).** According to GP 1, from diamine **C_4 NH_2** (0.78 g, 3.22 mmol), abs. NEt_3 (1.80 mL, 1.30 g, 12.9 mmol) and methacryloyl chloride (0.65 mL, 0.71 g, 6.76 mmol) in abs. CH_2Cl_2 (100 mL), chromatography with CH_2Cl_2/MeOH 40:1, 20:1 to 10:1; yield: 560 mg, 1.48 mmol, 46%, colourless solid, R_f = 0.47 (CH_2Cl_2/MeOH 10:1, $KMnO_4$). Mp. 86 °C. ^{1}H-NMR (500 MHz, $CDCl_3$): δ = 1.52 – 1.59 (m, 2H, 2‘-H), 1.59 – 1.67 (m, 4H, 2’’-H, 3’’-H), 1.91 – 1.98 (m, 2H, 3‘-H), 1.92, 1.94 (2 s, 6H, 2 x CH_3), 3.27 – 3.35 (m, 4H, 1‘-H, 1‘‘-H), 3.54 (t, *J* = 5.6 Hz, 2H, 4’’-H), 4.38 (t, *J* = 6.9 Hz, 2H, 4‘-H), 4.60 (s, 2H, 6-H), 5.28, 5.31 (2 s, 2H, C(CH_3)=CH_2), 5.63, 5.67 (2 s, 2H, C(CH_3)=CH_2), 6.08 (br s, 1H, N*H*), 6.17 (br s, 1H, N*H*), 7.56 (s, 1H, 5-H) ppm. ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 18.8 (2 x CH_3), 26.5, 26.7, 27.0, 27.7 (C-2‘, C-2‘‘, C-3‘, C-3‘‘), 38.8, 39.4 (C-1’, C-1’’), 49.9 (C-4’), 64.4 (C-6), 70.2 (C-4’’), 119.3, 119.7 (C(CH_3)=*C*H_2), 122.6 (C-5), 140.0, 140.3 (*C*(CH_3)=CH_2), 145.4 (C-4), 168.6, 168.8 (C=O) ppm. FT-IR (ATR, $CDCl_3$): $\tilde{\nu}$ = 3323 (br), 3135 (w), 3081 (w), 2932 (m), 2866 (m), 1655 (vs), 1614 (vs), 1533 (vs), 1452 (m), 1376 (m), 1321 (m), 1219 (s), 1138 (m), 1099 (m), 1053 (m), 928 (m), 808 (w), 651 (w) cm^{-1}. MS (ESI): *m*/*z* = 400.23 $[M + Na]^+$, 372.22. HRMS (ESI): calcd. for $[C_{19}H_{31}N_5O_3Na]^+$ 400.2319, found 400.2305 $[M + Na]^+$. Elemental analysis: calcd. (%) for $C_{19}H_{31}N_5O_3$: C 60.45, H 8.28, N 18.55; found: C 60.37, H 8.01, N 18.45.



C_4 MeAAm

$C_{19}H_{31}N_5O_3$
377.49 g/mol

***N*-{6-[4-({[6-(Methacryloylamino)hexyl]oxy}methyl)-1*H*-1,2,3-triazol-4-yl]hexyl}-2-methylacrylamide (C_6 MeAAm).** According to GP 1, from diamine **C_6 NH_2** (0.99 g, 3.32 mmol), abs. NEt_3 (1.84 mL, 1.34 g, 13.3 mmol) and methacryloyl chloride (0.67 mL, 0.73 g, 6.97 mmol) in abs. CH_2Cl_2 (120 mL), chromatography with CH_2Cl_2/MeOH 40:1 to 20:1; yield: 440 mg, 1.02 mmol, 31%, colourless solid,

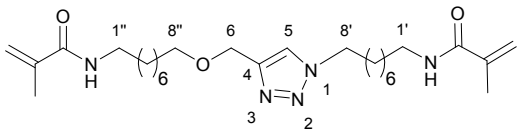


$C_{23}H_{39}N_5O_3$
433.60 g/mol

C_6 MeAAm

R_f = 0.51 (CH_2Cl_2/MeOH 10:1, $KMnO_4$). Mp. 53 °C. ^{1}H-NMR (700 MHz, $CDCl_3$): δ = 1.29 – 1.38 (m, 8H, CH_2), 1.47 – 1.53 (m, 4H, 2'-H, 2''-H), 1.54 – 1.59 (m, 2H, 5''-H), 1.85 – 1.91 (m, 2H, 5'-H), 1.93 (s, 6H, 2 x CH_3), 3.23 – 3.28 (m, 4H, 1'-H, 1''-H), 3.49 (t, *J* = 6.5 Hz, 2H, 6''-H), 4.32 (t, *J* = 7.2 Hz, 2H, 6'-H), 4.58 (s, 2H, 6-H), 5.27, 5.28 (2 s, 2H, $C(CH_3)=CH_2$), 5.64, 5.65 (2 s, 2H, $C(CH_3)=CH_2$), 5.95 (br s, 1H, N*H*), 6.03 (br s, 1H, N*H*), 7.51 (s, 1H, 5-H) ppm. ^{13}C-NMR (175 MHz, $CDCl_3$): δ = 18.7 (2 C, CH_3), 25.8, 26.0, 26.1, 26.7, 29.3, 29.45, 29.47, 30.1 (8 x CH_2), 39.3, 39.6 (C-1', C-1''), 50.1 (C-6'), 64.3 (C-6), 70.6 (C-6''), 119.1, 119.2 ($C(CH_3)=CH_2$), 122.2 (C-5), 140.16, 140.24 ($C(CH_3)=CH_2$), 145.4 (C-4), 168.47, 168.51 (C=O) ppm. FT-IR (ATR, $CDCl_3$): $\tilde{\nu}$ = 3327 (br), 3132 (w), 3084 (w), 2931 (s), 2859 (m), 1655 (vs), 1613 (vs), 1532 (vs), 1454 (m), 1437 (m), 1375 (m), 1323 (m), 1218 (s), 1096 (s), 1053 (m), 927 (m), 808 (w), 731 (w), 651 (w) cm^{-1}. MS (ESI): *m*/*z* = 456.29 $[M + Na]^+$, 428.29. HRMS (ESI): calcd. for $[C_{23}H_{39}N_5O_3Na]^+$ 456.2945, found 456.2942 $[M + Na]^+$. Elemental analysis: calcd. (%) for $C_{23}H_{39}N_5O_3$: C 63.71, H 9.07, N 16.15; found: C 63.60, H 8.88, N 16.13.

***N*-{8-[4-({[8-(Methacryloylamino)octyl]oxy}methyl)-1*H*-1,2,3-triazol-4-yl]octyl}-2-methylacrylamide (C_8 MeAAm).** According to GP 1, from diamine **C_8 NH_2** (0.37 g, 1.04 mmol), abs. NEt_3 (0.58 mL, 0.42 g, 4.16 mmol) and methacryloyl chloride (0.21 mL, 0.23 g, 2.18 mmol) in abs. CH_2Cl_2 (50 mL), chromatography with CH_2Cl_2/MeOH 40:1, 20:1 to 10:1; yield: 0.33 g, 0.67 mmol, 65%, colourless solid,

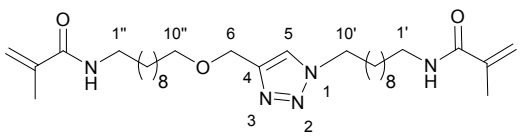


$C_{27}H_{47}N_5O_3$
489.69 g/mol

C_8 MeAAm

R_f = 0.57 (CH_2Cl_2/MeOH 10:1, $KMnO_4$). Mp. 66 °C. ^{1}H-NMR (500 MHz, $CDCl_3$): δ = 1.25 – 1.35 (m, 16H, CH_2), 1.47 – 1.54 (m, 4H, CH_2), 1.55 – 1.60 (m, 2H, CH_2), 1.86 – 1.90 (m, 2H, CH_2), 1.95 (s, 6H, 2 x CH_3), 3.27 (m, 4H, 1'-H, 1''-H), 3.50 (t, *J* = 6.7 Hz, 2H, 8''-H), 4.32 (t, *J* = 7.4 Hz, 2H, 8'-H), 4.60 (s, 2H, 6-H), 5.29 (s, 2H, $C(CH_3)=CH_2$), 5.65 (s, 2H, $C(CH_3)=CH_2$), 5.88 (br s, 2H, N*H*), 7.51 (s, 1H, 5-H) ppm. ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 18.8 (2 x CH_3), 26.1, 26.5, 26.9, 27.0, 28.9, 29.1, 29.3, 29.4, 29.6, 29.65, 29.69, 30.3 (CH_2), 39.7, 39.8 (C-1', C-1''), 50.4 (C-8'), 64.5 (C-6), 70.9 (C-8''), 119.2, 119.3 ($C(CH_3)=CH_2$), 122.3 (C-5), 140.36, 140.38 ($C(CH_3)=CH_2$), 145.6 (C-4), 168.6 (2 x C=O) ppm. FT-IR (ATR, $CDCl_3$): $\tilde{\nu}$ = 3330 (br), 2927 (s), 2855 (m), 1656 (s), 1615 (s), 1533 (s), 1455 (m), 1375 (m), 1318 (w), 1218 (m), 1136 (w), 1099 (m), 1053 (w), 927 (w), 808 (w), 724 (w), 652 (w) cm^{-1}. MS (ESI): *m*/*z* = 512.36 $[M + Na]^+$, 484.35, 182.15. HRMS (ESI): calcd. for $[C_{27}H_{47}N_5O_3Na]^+$ 512.3571, found 512.3568 $[M + Na]^+$. Elemental analysis: calcd. (%) for $C_{27}H_{47}N_5O_3$: C 66.23, H 9.67, N 14.30; found: C 65.93, H 9.39, N 14.07.

***N*-{10-[4-({[10-(Methacryloylamino)decyl]oxy}methyl)-1*H*-1,2,3-triazol-4-yl]decyl}-2-methylacrylamide (C_{10} MeAAm).** According to GP 1,



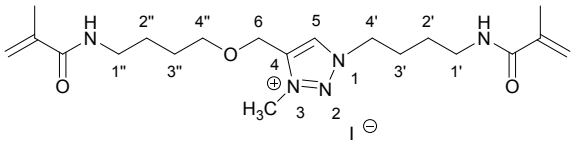
$C_{31}H_{55}N_5O_3$
545.80 g/mol

C_{10} MeAAm

from diamine **C_{10} NH_2** (0.60 g, 1.47 mmol), abs. NEt_3 (0.60 mL, 0.82 g, 5.88 mmol) and methacryloyl chloride (0.30 mL, 0.32 g, 3.09 mmol) in abs. CH_2Cl_2 (75 mL), chromatography with CH_2Cl_2/MeOH 80:1 to 20:1; yield: 0.58 g, 1.06 mmol, 72%, colourless solid, R_f = 0.69 (CH_2Cl_2/MeOH 10:1, $KMnO_4$). Mp. 77 °C. ^{1}H-NMR (500 MHz, $CDCl_3$): δ = 1.22 – 1.32 (m, 24H, CH_2), 1.47 – 1.54 (m, 4H, CH_2), 1.54 – 1.59 (m, 2H, CH_2), 1.85 – 1.89 (m, 2H, CH_2), 1.94 (s, 6H, 2 x CH_3), 3.24 – 3.29 (m, 4H, 1'-H, 1''-H), 3.49 (t, J = 6.7 Hz, 2H, 10''-H), 4.31 (t, J = 7.4 Hz, 2H, 10'-H), 4.59 (s, 2H, 6-H), 5.28 (s, 2H, C(CH_3)=CH_2), 5.65 (s, 2H, C(CH_3)=CH_2), 5.91 (br s, 2H, NH), 7.51 (s, 1H, 5-H) ppm. ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 18.8 (2 x CH_3), 26.2, 26.5, 26.95, 27.00, 29.0, 29.2, 29.30, 29.33, 29.4, 29.47, 29.50, 29.53, 29.62, 29.64, 29.7, 30.3 (CH_2), 39.77, 39.80 (C-1', C-1''), 50.4 (C-10'), 64.4 (C-6), 70.9 (C-10''), 119.2 (2 x C(CH_3)=CH_2), 122.2 (C-5), 140.4 (2 x C(CH_3)=CH_2), 145.5 (C-4), 168.5 (2 x C=O) ppm. FT-IR (ATR, $CDCl_3$): $\tilde{\nu}$ = 3325 (br), 3142 (br), 3096 (br), 2917 (s), 2849 (s), 1652 (s), 1612 (s), 1525 (s), 1470 (s), 1377 (w), 1342 (w), 1320 (w), 1215 (m), 1149 (w), 1119 (s), 1053 (m), 978 (w), 918 (m), 872 (w), 803 (w), 720 (m), 649 (m), 593 (w) cm^{-1}. MS (ESI): m/z = 584.39, 568.42 $[M + Na]^+$, 540.41, 292.69, 210.18. HRMS (ESI): calcd. for $[C_{31}H_{55}N_5O_3Na]^+$ 568.4197, found 568.4199 $[M + Na]^+$. Elemental analysis: calcd. (%) for $C_{31}H_{55}N_5O_3$: C 68.22, H 10.16, N 12.83; found: C 67.71, H 9.83, N 12.65.

1.7 Triazolium-Methacrylamide Cross-linkers Me-C_n^+ I^- MeAAm

3-[4-(Methacryloylamino)butyl]-5-({[4-(methacryloylamino)butyl]oxy}methyl)-1-methyl-1*H*-1,2,3-triazol-3-ium iodide (Me-C_4^+ I^- MeAAm). Following GP 3, from triazole **C_4 MeAAm** (0.10 g, 0.27 mmol) and CH_3I (0.76 g, 0.33 mL, 5.35 mmol) in CH_3CN (3 mL), 7 d at 30 °C, chromatography on HI-silica with CH_2Cl_2/MeOH 15:1 to 5:1; yield: 99 mg, 0.19 mmol, 71%, yellow oil,

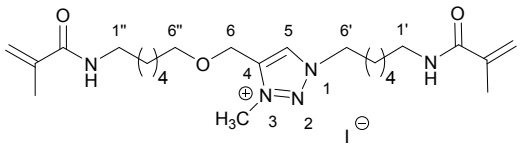


$C_{20}H_{34}IN_5O_3$
519.43 g/mol

Me-C_4^+ I^- MeAAm

R_f = 0.21 (CH_2Cl_2/MeOH 10:1, $KMnO_4$). ^{1}H-NMR (700 MHz, CD_2Cl_2): δ = 1.60 – 1.67 (m, 4H, 2''-H, 3''-H), 1.67 – 1.72 (m, 2H, 2'-H), 1.93, 1.94 (2 s, 6H, 2 x CH_3), 2.06 – 2.11 (m, 2H, 3'-H), 3.25 – 3.28 (m, 2H, 1''-H), 3.32 – 3.35 (m, 2H, 1'-H), 3.66 (t, J = 5.9 Hz, 2H, 4''-H), 4.32 (s, 3H, NCH_3), 4.77 (t, J = 7.3 Hz, 2H, 4'-H), 4.86 (s, 2H, 6-H), 5.28 – 5.31 (m, 2H, C(CH_3)=CH_2), 5.70, 5.79 (2 s, 2H, C(CH_3)=CH_2), 6.49 (br s, 1H, NH), 6.98 (br s, 1H, NH), 9.29 (s, 1H, 5-H) ppm. ^{13}C-NMR (175 MHz, CD_2Cl_2): δ = 19.1, 19.3 (2 x CH_3), 26.2, 26.5, 26.9, 27.1 (C-2', C-2'', C-3', C-3''), 38.3 (C-1'), 39.4 (C-1''), 39.6 (NCH_3), 54.2 (C-4'), 61.2 (C-6), 71.9 (C-4''), 119.5, 120.0 (C(CH_3)=CH_2), 130.7 (C-5), 140.3, 140.6, 141.3 (C-4, C(CH_3)=CH_2), 168.7, 168.9 (C=O) ppm. FT-IR (ATR, CD_2Cl_2): $\tilde{\nu}$ = 3306 (br), 3079 (w), 2927 (m), 2868 (w), 1723 (m), 1655 (vs), 1611 (vs), 1527 (vs), 1452 (s), 1375 (m), 1314 (m), 1218 (s), 1101 (s), 954 (m), 929 (m), 807 (w), 707 (s), 644 (m) cm^{-1}. MS (ESI): m/z = 392.26 $[M]^+$, 140.11. HRMS (ESI): calcd. for $[C_{20}H_{34}N_5O_3]^+$ 392.2656, found 392.2650 $[M]^+$. Elemental analysis: calcd. (%) for $C_{20}H_{34}IN_5O_3$: C 46.25, H 6.60, N 13.48; found: C 45.56, H 6.58, N 12.59.

1-[6-(Methacryloylamino)hexyl]-4-({[6-(methacryloylamino)hexyl]oxy}methyl)-3-methyl-1*H*-1,2,3-triazol-3-ium iodide (Me-C_6^+ I^- MeAAm). Following GP 3, from triazole **C_6 MeAAm** (0.10 g, 0.24 mmol) and CH_3I (0.29 mL, 0.67 g, 4.70 mmol) in CH_3CN (3 mL), 3 d at 30 °C,

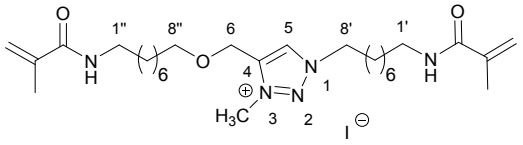


$C_{24}H_{42}IN_5O_3$
575.54 g/mol

Me-C_6^+ I^- MeAAm

chromatography on HI-silica with CH_2Cl_2/MeOH 10:1 to 5:1; yield: 0.11 g, 0.19 mmol, 79%, yellow oil, R_f = 0.32 (CH_2Cl_2/MeOH 10:1, $KMnO_4$). ^{1}H-NMR (700 MHz, $CDCl_3$): δ = 1.30 – 1.45 (m, 8H, CH_2), 1.51 – 1.62 (m, 6H, CH_2), 1.94 (s, 6H, 2 x CH_3), 2.02 – 2.08 (m, 2H, 5'-H), 3.23 – 3.28 (m, 4H, 1'-H, 1''-H), 3.59 (t, J = 6.4 Hz, 2H, 6''-H), 4.36 (s, 3H, NCH_3), 4.70 (t, J = 7.2 Hz, 2H, 6'-H), 4.88 (s, 2H, 6-H), 5.28 (s, 2H, C(CH_3)=CH_2), 5.69, 5.73 (2 s, 2H, C(CH_3)=CH_2), 6.21 (br s, 1H, NH), 6.47 (br s, 1H, NH), 9.36 (s, 1H, 5-H) ppm. ^{13}C-NMR (175 MHz, $CDCl_3$): δ = 18.9, 19.0 (2 x CH_3), 25.4, 25.5, 25.8, 26.4, 28.9, 29.1, 29.2, 29.3 (8 x CH_2), 39.0 (C-1' or C-1''), 39.37 (NCH$_3$), 39.39 (C-1'' or C-1'), 54.3 (C-6'), 60.8 (C-6), 72.0 (C-6''), 119.4, 119.7 (C(CH_3)=CH$_2$), 130.5 (C-5), 139.9, 140.1, 140.8 (C-4, C(CH_3)=CH_2), 168.6, 168.7 (C=O) ppm. FT-IR (ATR, $CDCl_3$): $\tilde{\nu}$ = 3308 (br), 3078 (w), 2932 (s), 2859 (m), 1655 (vs), 1612 (vs), 1527 (vs), 1454 (s), 1375 (m), 1316 (m), 1216 (m), 1098 (m), 1009 (w), 921 (m), 809 (w), 728 (m), 643 (m) cm^{-1}. MS (ESI): m/z = 448.33 [M]$^+$. HRMS (ESI): cald. for [$C_{24}H_{42}N_5O_3$]$^+$ 448.3282, found 448.3279 [M]$^+$. Elemental analysis: calcd. (%) for $C_{24}H_{42}IN_5O_3$: C 50.09, H 7.36, N 12.17; found: C 48.77, H 7.01, N 11.62.

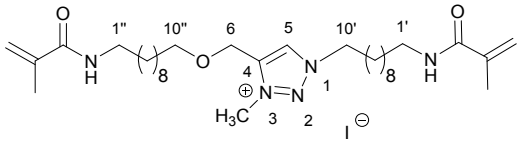
1-[8-(Methacryloylamino)octyl]-4-({[8-(methacryloylamino)octyl]oxy}methyl)-3-methyl-1*H*-1,2,3-triazol-3-ium iodide (Me-C_8^+ I^- MeAAm). Following GP 3, from triazole **C_8 MeAAm** (90 mg, 0.18 mmol) and CH_3I (0.23 mL, 0.52 g, 3.68 mmol) in CH_3CN (1 mL), 4 d at 30 °C, yield: 0.11 g, 0.17 mmol, 99%, yellow wax, R_f = 0.42 (CH_2Cl_2/MeOH 10:1, $KMnO_4$). ^{1}H-NMR (500 MHz, $CDCl_3$): δ = 1.28 – 1.40 (m, 16H, CH_2), 1.50 – 1.56 (m, 4H, CH_2), 1.58 – 1.62 (m, 2H, CH_2), 1.95 (s, 6H, 2 x CH_3), 2.01 – 2.08 (m, 2H, CH_2), 3.25 – 3.31 (m, 4H, 1'-H, 1''-H), 3.59 (t, J = 6.7 Hz, 2H, 8''-H), 4.36 (s, 3H, NCH_3), 4.70 (t, J = 7.4 Hz, 2H, 8'-H), 4.89 (s, 2H, 6-H), 5.29 (s, 2H, C(CH_3)=CH_2), 5.66 – 5.72 (m, 2H, C(CH_3)=CH_2), 5.99 (br s, 1H, NH), 6.12 (br s, 1H, NH), 9.44 (s, 1H, 5-H) ppm. ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 18.9, 19.0 (2 x CH$_3$), 25.91, 25.94, 26.6, 26.8, 28.5, 28.7, 29.1, 29.2, 29.40, 29.42, 29.5, 29.6 (CH_2), 39.4 (NCH$_3$), 39.6, 39.7 (C-1', C-1''), 54.4 (C-8'), 60.8 (C-6), 72.3 (C-8''), 119.4, 119.5 (C(CH_3)=CH$_2$), 130.8 (C-5), 140.2, 140.3 (C(CH_3)=CH_2), 140.7 (C-4), 168.58, 168.61 (C=O) ppm. FT-IR (ATR, $CDCl_3$): $\tilde{\nu}$ = 3307 (br), 3078 (w), 2927 (s), 2855 (m), 1656 (s), 1614 (s), 1530 (s), 1455 (m), 1374 (w), 1318 (m), 1217 (m), 1102 (m), 929 (w), 808 (w), 724 (w), 645 (w) cm^{-1}. MS (ESI): m/z = 504.39 [M]$^+$, 224.17. HRMS (ESI): calcd. for [$C_{28}H_{50}N_5O_3$]$^+$ 504.3908, found 504.3911 [M]$^+$. Elemental analysis: calcd. (%) for $C_{28}H_{50}IN_5O_3$: C 53.24, H 7.98, N 11.09; found: C 52.82, H 8.23, N 10.77.



Me-C_8^+ I^- MeAAm

$C_{28}H_{50}IN_5O_3$
631.63 g/mol

1-[10-(Methacryloylamino)decyl]-4-({[10-(methacryloylamino)decyl]oxy}methyl)-3-methyl-1*H*-1,2,3-triazol-3-ium iodide (Me-C_{10}^+ I^- MeAAm). Following GP 3, from triazole **C_{10} MeAAm** (0.12 g, 0.22 mmol) and CH_3I (0.27 mL, 0.62 g, 4.38 mmol) in CH_3CN (1.5 mL), 4 d at 30 °C, yield: 0.15 g, 0.22 mmol, 99%, yellow solid, R_f = 0.47 (CH_2Cl_2/MeOH 10:1, $KMnO_4$), Mp. 60 °C. ^{1}H-NMR



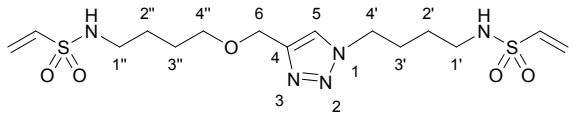
Me-C_{10}^+ I^- MeAAm

$C_{32}H_{58}IN_5O_3$
687.74 g/mol

(500 MHz, $CDCl_3$): δ = 1.22 – 1.42 (m, 24H, CH_2), 1.50 – 1.57 (m, 4H, CH_2), 1.57 – 1.61 (m, 2H, CH_2), 1.96 (s, 6H, 2 x C*H*$_3$), 2.02 – 2.07 (m, 2H, CH_2), 3.26 – 3.33 (m, 4H, 1'-H, 1''-H), 3.60 (t, *J* = 6.7 Hz, 2H, 10''-H), 4.36 (s, 3H, NC*H*$_3$), 4.70 (t, *J* = 7.4 Hz, 2H, 10'-H), 4.89 (s, 2H, 6-H), 5.30 (s, 2H, C(CH_3)=C*H*$_2$), 5.66 – 5.69 (m, 2H, C(CH_3)=C*H*$_2$), 5.87 (br s, 1H, N*H*), 5.93 (br s, 1H, N*H*), 9.44 (s, 1H, 5-H) ppm. ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 18.90, 18.93 (CH_3), 26.1, 26.2, 26.9, 27.0, 28.8, 29.2, 29.26, 29.29, 29.34, 29.46, 29.48, 29.52, 29.58, 29.62, 29.7 (CH_2), 39.3 (N*C*H$_3$), 39.77, 39.81 (C-1', C-1''), 54.6 (C-10'), 60.9 (C-6), 72.4 (C-10''), 119.3, 119.4 (C(CH_3)=*C*H$_2$), 130.9 (C-5), 140.4 (2 x *C*(CH_3)=CH_2), 140.7 (C-4), 168.6 (2 x C=O) ppm. FT-IR (ATR, $CDCl_3$): $\tilde{\nu}$ = 3309 (br), 3068 (w), 2926 (s), 2854 (m), 1656 (s), 1615 (s), 1530 (s), 1456 (m), 1374 (w), 1318 (w), 1217 (w), 1103 (m), 928 (w), 809 (w), 722 (w), 645 (w) cm^{-1}. MS (ESI): *m*/*z* = 560.45 $[M]^+$, 518.40, 295.21, 252.21, 224.20. HRMS (ESI): calcd. for $[C_{32}H_{58}N_5O_3]^+$ 560.4534, found 560.4520 $[M]^+$. Elemental analysis: calcd. (%) for $C_{32}H_{58}I_1N_5O_3$: C 55.89, H 8.50, N 10.18; found: C 55.53, H 8.36, N 9.76.

1.8 Neutral Vinylsulfonamide Cross-linkers C_n VSAm

***N*-{4-[4-({4-[(Vinylsulfonyl)amino]butoxy}methyl)-1*H*-1,2,3-triazol-1-yl]butyl}ethylenesulfonamide (C_4 VSAm).** Following GP 2, from diamine **C_4 NH_2** (0.67 g, 2.41 mmol), abs. NEt_3 (2.00 mL, 1.46 g, 14.5 mmol) and 2-chloroethanesulfonyl chloride (0.53 mL, 0.83 g, 5.06 mmol) in abs. CH_2Cl_2 (100 mL), chromatography

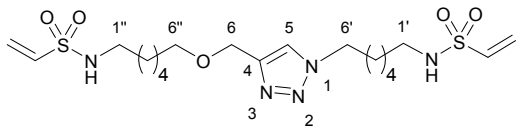


C_4 VSAm

$C_{15}H_{27}N_5O_5S_2$
421.53 g/mol

with CH_2Cl_2/MeOH 40:1 to 5:1; yield: 0.18 g, 0.42 mmol, 18%, colourless oil, R_f = 0.07 (PE/EtOAc 1:4, $KMnO_4$). ^{1}H-NMR (500 MHz, CD_2Cl_2): δ = 1.50 – 1.59 (m, 2H, 2'-H), 1.59 – 1.69 (m, 4H, 2''-H, 3''-H), 1.93 – 2.01 (m, 2H, 3'-H), 2.95 – 3.05 (m, 4H, 1'-H, 1''-H), 3.53 (t, *J* = 5.7 Hz, 2H, 4''-H), 4.37 (t, *J* = 6.9 Hz, 2H, 4'-H), 4.57 (s, 2H, 6-H), 4.92 (br s, 1H, N*H*), 5.11 (br s, 1H, N*H*), 5.90 – 5.95 (m, 2H, CH=C*H*$_2$), 6.12 – 6.20 (m, 2H, CH=C*H*$_2$), 6.44 – 6.54 (m, 2H, C*H*=CH_2), 7.62 (s, 1H, 5-H) ppm. ^{13}C-NMR (125 MHz, CD_2Cl_2): δ = 27.1, 27.2, 27.4, 27.6 (C-2', C-3', C-2'', C-3''), 42.7, 43.3 (C-1', C-1''), 50.0 (C-4'), 64.5 (C-6), 70.5 (C-4''), 123.2 (C-5), 126.7, 126.9 (CH=*C*H$_2$), 136.2, 136.3 (*C*H=CH_2), 145.3 (C-4) ppm. FT-IR (ATR, CD_2Cl_2): $\tilde{\nu}$ = 3281 (br), 3141 (w), 2943 (w), 2867 (w), 1434 (m), 1385 (m), 1321 (vs), 1256 (w), 1222 (w), 1142 (vs), 1081 (s), 956 (s), 779 (w), 735 (s), 709 (s), 657 (s), 547 (s), 498 (m) cm^{-1}. MS (ESI): *m*/*z* = 444.13 $[M + Na]^+$. HRMS (ESI): calcd. for $[C_{15}H_{27}N_5O_5S_2Na]^+$ 444.1346, found 444.1336 $[M + Na]^+$. Elemental analysis: calcd. (%) for $C_{15}H_{27}N_5O_5S_2$: C 42.74, H 6.46, N 16.61; found: C 42.86, H 6.38, N 16.18.

***N*-(6-{4-[({6-[(Vinylsulfonyl)amino]hexyl}oxy)methyl]-1*H*-1,2,3-triazol-1-yl}hexyl)ethylenesulfonamide (C_6 VSAm).** Following GP 2, from diamine **C_6 NH_2** (0.99 g, 3.28 mmol), abs. NEt_3 (2.70 mL, 1.99 g, 19.7 mmol) and 2-chloroethanesulfonyl chloride (0.72 mL, 1.12 g, 6.89 mmol) in abs. CH_2Cl_2 (120 mL), chromatography with CH_2Cl_2/MeOH 20:1 to 10:1 or EtOAc/MeOH 40:1, 20:1 to 10:1;

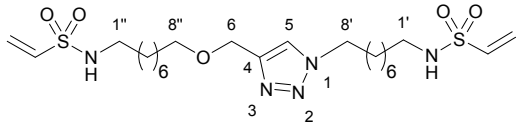


C_6 VSAm

$C_{19}H_{35}N_5O_5S_2$
477.64 g/mol

yield: 0.55 g, 1.15 mmol, 35%, colourless oil, R_f = 0.17 (PE/EtOAc 1:4, $KMnO_4$). ^{1}H-NMR (500 MHz, CD_2Cl_2): δ = 1.27 – 1.41 (m, 8H, 3'-H, 3''-H, 4'-H, 4''-H), 1.49 – 1.60 (m, 6H, 2'-H, 2''-H, 5''-H), 1.85 – 1.93 (m, 2H, 5'-H), 2.93 – 3.01 (m, 4H, 1'-H, 1''-H), 3.48 (t, *J* = 6.6 Hz, 2H, 6''-H), 4.33 (t, *J* = 7.1 Hz, 2H, 6'-H), 4.55 (s, 2H, 6-H), 4.63 (br s, 2H, N*H*), 5.93 (2 d, *J* = 10.1 Hz, 2H, CH=CH_2), 6.18 (d, *J* = 16.6 Hz, 2H, CH=CH_2), 6.51 (2 dd, *J* = 10.1, 16.6 Hz, 2H, C*H*=CH_2), 7.54 (s, 1H, 5-H) ppm. ^{13}C-NMR (125 MHz, CD_2Cl_2): δ = 26.0, 26.16, 26.22, 26.6 (C-3', C-3'', C-4', C-4''), 29.8, 30.0, 30.1, 30.4 (C-2', C-2'', C-5', C-5''), 43.2, 43.4 (C-1', C-1''), 50.4 (C-6'), 64.5 (C-6), 70.7 (C-6''), 122.8 (C-5), 126.7, 126.8 (CH=*C*H_2), 136.3, 136.4 (*C*H=CH_2), 145.6 (C-4) ppm. FT-IR (ATR, CD_2Cl_2): $\tilde{\nu}$ = 3283 (br), 3139 (w), 2935 (m), 2860 (m), 1433 (m), 1384 (m), 1322 (vs), 1256 (w), 1221 (w), 1144 (vs), 1088 (s), 1058 (m), 969 (m), 734 (s), 660 (m), 548 (m), 501 (w) cm^{-1}. MS (ESI): *m*/*z* = 500.20 $[M + Na]^+$, 478.21 $[M + H]^+$, 408.20, 243.12, 120.01. HRMS (ESI): calcd. for $[C_{19}H_{35}N_5O_5S_2Na]^+$ 500.1972, found 500.1959 $[M + Na]^+$. Elemental analysis: calcd. (%) for $C_{19}H_{35}N_5O_5S_2$: C 47.78, H 7.39, N 14.66; found: C 47.79, H 7.14, N 14.39.

***N*-(8-{4-[({8-[(Vinylsulfonyl)amino]octyl}oxy)methyl]-1*H*-1,2,3-triazol-1-yl}octyl)ethylenesulfonamide (C_8 VSAm).** Following GP 2, from diamine **C_8 NH_2** (1.09 g, 3.00 mmol), abs. NEt_3 (2.50 mL, 1.82 g, 18.0 mmol) and 2-chloroethanesulfonyl chloride (0.66 mL, 1.03 g, 6.30 mmol) in abs. CH_2Cl_2 (120 mL), chromatography with PE/EtOAc 1:2 to 1:4; yield: 0.80 g, 1.50 mmol, 50%, colourless

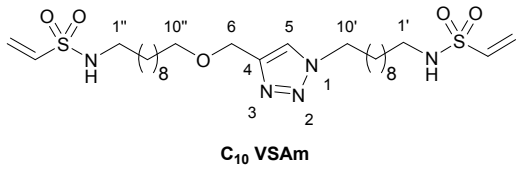


C_8 VSAm

$C_{23}H_{43}N_5O_5S_2$
533.75 g/mol

solid, R_f = 0.35 (PE/EtOAc 1:4, $KMnO_4$). Mp. 39 °C. ^{1}H-NMR (500 MHz, CD_2Cl_2): δ = 1.25 – 1.36 (m, 16H, CH_2), 1.48 – 1.59 (m, 6H, 2'-H, 2''-H, 7''-H), 1.84 – 1.91 (m, 2H, 7'-H), 2.94 – 3.00 (m, 4H, 1'-H, 1''-H), 3.47 (t, *J* = 6.5 Hz, 2H, 8''-H), 4.32 (t, *J* = 7.1 Hz, 2H, 8'-H), 4.55 (s, 2H, 6-H), 4.65 (br s, 2H, N*H*), 5.93 (d, *J* = 10.1 Hz, 2H, CH=CH_2), 6.18 (d, *J* = 16.6 Hz, 2H, CH=CH_2), 6.51 (2 dd, *J* = 10.1, 16.6 Hz, 2H, C*H*=CH_2), 7.53 (s, 1H, 5-H) ppm. ^{13}C-NMR (125 MHz, CD_2Cl_2): δ = 26.3, 26.6, 26.7, 26.8, 29.1, 29.2, 29.3, 29.5 (CH_2), 29.9, 30.1, 30.2, 30.5 (C-2', C-2'', C-7', C-7''), 43.0, 43.5 (C-1', C-1''), 50.6 (C-8'), 64.5 (C-6), 70.9 (C-8''), 122.7 (C-5), 126.65, 126.68 (CH=*C*H_2), 136.38, 136.39 (*C*H=CH_2), 145.6 (C-4) ppm. FT-IR (ATR, CD_2Cl_2): $\tilde{\nu}$ = 3282 (br), 3139 (w), 2929 (m), 2856 (m), 1461 (w), 1434 (w), 1384 (w), 1324 (vs), 1255 (w), 1222 (w), 1145 (vs), 1087 (s), 1056 (m), 955 (s), 732 (s), 709 (vs), 658 (s), 548 (s), 496 (m) cm^{-1}. MS (ESI): *m*/*z* = 556.26 $[M + Na]^+$, 464.26. HRMS (ESI): calcd. for $[C_{23}H_{43}N_5O_5S_2Na]^+$ 556.2598, found 556.2589 $[M + Na]^+$. Elemental analysis: calcd. (%) for $C_{23}H_{43}N_5O_5S_2$: C 51.76, H 8.12, N 13.12; found: C 51.86, H 8.09, N 13.37.

***N*-(10-{4-[({10-[(Vinylsulfonyl)amino]decyl}oxy)methyl]-1*H*-1,2,3-triazol-1-yl}decyl)ethylene-sulfonamide (C_{10} VSAm).** Following GP 2, from diamine **C_{10} NH_2** (0.36 g, 0.78 mmol), abs. NEt_3 (0.65 mL, 0.47 g, 4.68 mmol) and 2-chloroethanesulfonyl chloride (0.17 mL, 0.27 g, 1.64 mmol) in abs. CH_2Cl_2 (40 mL), chromatography with PE/EtOAc 1:1 to 1:2; yield: 0.21 g, 0.36 mmol, 46%, colourless solid, R_f = 0.52 (PE/EtOAc 1:4, $KMnO_4$). Mp. 58 °C. ^{1}H-NMR (500 MHz, CD_2Cl_2): δ = 1.23 – 1.35 (m, 24H, CH_2), 1.48 – 1.60 (m, 6H, 2'-H, 2''-H, 9''-H), 1.84 – 1.92 (m, 2H, 9'-H), 2.97 (q, *J* = 6.8 Hz, 4H, 1'-H, 1''-H), 3.47 (t, *J* = 6.7 Hz, 2H, 10''-H), 4.31 (t, *J* = 7.2 Hz, 2H, 10'-H), 4.55 (s, 2H, 6-H), 4.62 (br s, 1H, N*H*), 4.66 (br s, 1H, N*H*), 5.92 (d, *J* = 10.0 Hz, 2H, CH=CH_2), 6.18 (d, *J* = 16.6 Hz, 2H, CH=CH_2), 6.51 (2 dd, *J* = 10.0, 16.6 Hz, 2H, C*H*=CH_2), 7.53 (s, 1H, 5-H) ppm. ^{13}C-NMR (125 MHz, CD_2Cl_2): δ = 26.4, 26.7, 26.8, 26.9, 29.2, 29.3, 29.4, 29.5, 29.6, 29.7, 29.8, 30.0, 30.20, 30.22, 30.6 (CH_2), 43.45, 43.47 (C-1', C-1''), 50.6 (C-10'), 64.5 (C-6), 71.0 (C-10''), 122.7 (C-5), 126.62, 126.64 (CH=*C*H_2), 136.39 (*C*H=CH_2), 145.6 (C-4) ppm. FT-IR (ATR, CD_2Cl_2): $\tilde{\nu}$ = 3282 (br), 3140 (w), 2924 (s), 2853 (s), 1462 (m), 1435 (m), 1383 (w), 1324 (vs), 1255 (w), 1221 (w), 1144 (vs), 1089 (s), 967 (s), 733 (s), 659 (s), 548 (m), 496 (m) cm^{-1}. MS (ESI): *m*/*z* = 612.32 $[M + Na]^+$. HRMS (ESI): calcd. for $[C_{27}H_{51}N_5O_5S_2Na]^+$ 612.3224, found 612.3230 $[M + Na]^+$. Elemental analysis: calcd. (%) for $C_{27}H_{51}N_5O_5S_2$: C 54.98, H 8.72, N 11.87; found: C 55.09, H 8.48, N 11.94.

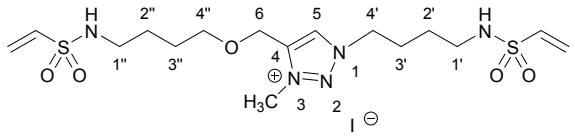


C_{10} VSAm

$C_{27}H_{51}N_5O_5S_2$
589.86 g/mol

1.9 Triazolium-Vinylsulfonamide Cross-linkers Me-C_n^+ I^- VSAm

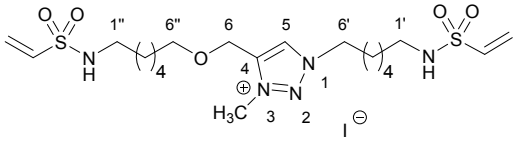
3-Methyl-4-({4-[(vinylsulfonyl)amino]butoxy}methyl)-1-{4-[(vinylsulfonyl)amino]butyl}-1H-1,2,3-triazol-3-ium iodide (Me-C_4^+ I^- VSAm). Following GP 3, from triazole **C_4 VSAm** (83 mg, 0.20 mmol) and CH_3I (0.25 mL, 0.56 g, 3.94 mmol) in CH_3CN (5 mL), 6 d at 30 °C, yield: 108 mg, 0.19 mmol, 97%, yellow oil, R_f = 0.16 (CH_2Cl_2/MeOH 10:1, $KMnO_4$). ^{1}H-NMR (500 MHz, CD_3OD): δ = 1.59 – 1.67 (m, 4H, 2'-H, 2''-H), 1.67 – 1.75 (m, 2H, 3''-H), 2.09 – 2.17 (m, 2H, 3'-H), 2.96 (t, *J* = 6.7 Hz, 2H, 1'-H or 1''-H), 3.00 (t, *J* = 6.7 Hz, 2H, 1''-H or 1'-H), 3.65 (t, *J* = 6.0 Hz, 2H, 4''-H), 4.32 (s, 3H, NCH_3), 4.67 (t, *J* = 7.2 Hz, 2H, 4'-H), 4.67 (s, 2H, 6-H), 5.94 – 6.01 (m, 2H, CH=CH_2), 6.08 – 6.17 (m, 2H, CH=CH_2), 6.59 – 6.69 (m, 2H, C*H*=CH_2), 8.75 (s, 1H, 5-H) ppm. ^{13}C-NMR (125 MHz, CD_3OD): δ = 27.3, 27.49, 27.54, 27.6 (C-2', C-3', C-2'', C-3''), 39.0 (NCH_3), 42.8, 43.5 (C-1', C-1''), 54.6 (C-4'), 61.5 (C-6), 72.1 (C-4''), 126.4, 126.7 (CH=*C*H_2), 130.6 (C-5), 137.5, 137.7 (*C*H=CH_2), 142.5 (C-4) ppm. FT-IR (ATR, CD_2Cl_2): $\tilde{\nu}$ = 3108 (br), 2942 (w), 2870 (w), 1422 (m), 1385 (w), 1321 (vs), 1145 (vs), 1076 (s), 970 (m), 862 (w), 802 (w), 741 (m), 658 (m), 547 (m), 495 (m) cm^{-1}. MS (ESI): *m*/*z* = 436.17 $[M]^+$, 381.30, 353.26. HRMS (ESI): calcd. for $[C_{16}H_{30}N_5O_5S_2]^+$ 436.1683, found 436.1697 $[M]^+$. Elemental analysis: calcd. (%) for $C_{16}H_{30}IN_5O_5S_2$: C 34.11, H 5.37, N 12.43; found: C 34.51, H 5.28, N 12.16.



Me-C_4^+ I^- VSAm

$C_{16}H_{30}IN_5O_5S_2$
563.47 g/mol

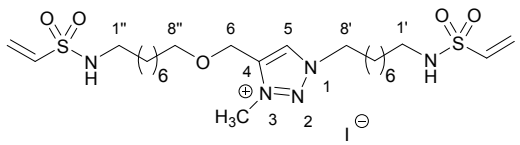
3-Methyl-1-{6-[(vinylsulfonyl)amino]hexyl}-4-[({6-[(vinylsulfonyl)amino]hexyl}oxy)methyl]-1*H*-1,2,3-triazol-3-ium-iodid (Me-C_6^+ I^- VSAm). Following GP 3, from triazole **C_6 VSAm** (0.13 g, 0.28 mmol) and CH_3I (0.34 mL, 0.79 g, 5.53 mmol) in CH_3CN (7 mL), 7 d at 30 °C, yield: 0.12 g, 0.20 mmol, 73%, yellow oil, R_f = 0.39 (CH_2Cl_2/MeOH 10:1, $KMnO_4$). ^{1}H-NMR (500 MHz, CD_2Cl_2): δ = 1.34 – 1.48 (m, 8H, 3‘-H, 3‘‘-H, 4‘-H, 4‘‘-H), 1.53 – 1.67 (m, 6H, 2‘-H, 2‘‘-H, 5‘‘-H), 2.02 – 2.12 (m, 2H, 5‘-H), 2.90 – 2.98 (m, 4H, 1‘-H, 1‘‘-H), 3.62 (t, *J* = 6.3 Hz, 2H, 6’’-H), 4.33 (s, 3H, NCH_3), 4.70 (t, *J* = 7.1 Hz, 2H, 6‘-H), 4.87 (s, 2H, 6-H), 5.41 (t, *J* = 6.1 Hz, 1H, N*H*), 5.64 (t, *J* = 6.1 Hz, 1H, N*H*), 5.92 (2 d, *J* = 9.9 Hz, 2H, CH=CH_2), 6.13 (2 d, *J* = 16.6 Hz, 2H, CH=CH_2), 6.59 (2 dd, *J* = 9.9, 16.6 Hz, 2H, C*H*=CH_2), 9.11 (s, 1H, 5-H) ppm. ^{13}C-NMR (125 MHz, CD_2Cl_2): δ = 25.5, 25.7, 25.8, 26.4 (C-3‘, C-3‘‘, C-4‘‘, C-4‘‘), 29.22, 29.26, 29.31, 29.8 (C-2‘, C-2‘‘, C-5‘‘, C-5‘‘), 39.5 (NCH_3), 42.9, 43.2 (C-1’, C-1’’), 54.5 (C-6’), 61.0 (C-6), 72.1 (C-6’’), 126.37, 126.43 (CH=*C*H_2), 130.6 (C-5), 136.59, 136.63 (*C*H=CH_2), 141.2 (C-4) ppm. FT-IR (ATR, CD_2Cl_2): $\tilde{\nu}$ = 3262 (br), 3122 (br), 2935 (m), 2861 (m), 1458 (w), 1423 (m), 1384 (w), 1321 (vs), 1255 (w), 1146 (vs), 1088 (m), 970 (m), 852 (w), 807 (w), 737 (m), 660 (m), 549 (m), 498 (w) cm^{-1}. MS (ESI): *m*/*z* = 548.22, 492.24 $[M]^+$, 400.24, 303.15 $[C_{12}H_{23}N_4O_3S_2]^+$, 120.01. HRMS (ESI): calcd. for $[C_{20}H_{38}N_5O_5S_2]^+$ 492.2309, found 492.2307 $[M]^+$. Elemental analysis: calcd. (%) for $C_{20}H_{38}IN_5O_5S_2$: C 38.77, H 6.18, N 11.30; found: C 38.37, H 5.91, N 10.84.



Me-C_6^+ I^- VSAm

$C_{20}H_{38}IN_5O_5S_2$
619.58 g/mol

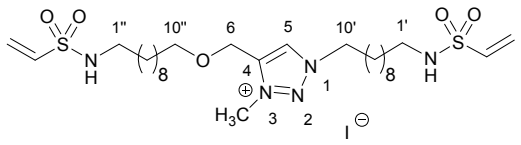
3-Methyl-1-{8-[(vinylsulfonyl)amino]octyl}-4-[({8-[(vinylsulfonyl)amino]octyl}oxy)methyl]-1*H*-1,2,3-triazol-3-ium-iodid (Me-C_8^+ I^- VSAm). Following GP 3, from triazole **C_8 VSAm** (0.16 g, 0.30 mmol) and CH_3I (0.38 mL, 0.86 g, 6.03 mmol) in CH_3CN (7.5 mL), 7 d at 30 °C, yield: 0.15 g, 0.22 mmol, 73%, yellow oil, R_f = 0.47 (CH_2Cl_2/MeOH 10:1, $KMnO_4$). ^{1}H-NMR (500 MHz, CD_2Cl_2): δ = 1.27 – 1.44 (m, 16H, CH_2), 1.51 – 1.64 (m, 6H, 2‘-H, 2‘‘-H, 7‘‘-H), 2.00 – 2.09 (m, 2H, 7‘-H), 2.90 – 2.98 (m, 4H, 1‘-H, 1‘‘-H), 3.61 (t, *J* = 6.4 Hz, 2H, 8’’-H), 4.32 (s, 3H, NCH_3), 4.69 (t, *J* = 7.2 Hz, 2H, 8‘-H), 4.87 (s, 2H, 6-H), 5.20 (t, *J* = 6.0 Hz, 1H, N*H*), 5.39 (t, *J* = 6.0 Hz, 1H, N*H*), 5.91 (2 d, *J* = 10.0 Hz, 2H, CH=CH_2), 6.14 (2 d, *J* = 16.6 Hz, 2H, CH=CH_2), 6.57 (2 dd, *J* = 10.0, 16.6 Hz, 2H, C*H*=CH_2), 9.19 (s, 1H, 5-H) ppm. ^{13}C-NMR (125 MHz, CD_2Cl_2): δ = 25.9, 26.0, 26.4, 26.6, 28.5, 28.6, 29.1, 29.2, 29.5, 29.8, 30.0 (CH_2), 39.4 (NCH_3), 43.2, 43.4 (C-1’, C-1’’), 54.7 (C-8’), 60.9 (C-6), 72.3 (C-8’’), 126.3, 126.4 (CH=*C*H_2), 130.7 (C-5), 136.56, 136.63 (*C*H=CH_2), 141.0 (C-4) ppm. FT-IR (ATR, CD_2Cl_2): $\tilde{\nu}$ = 3255 (w), 3107 (br), 2928 (m), 2856 (w), 1460 (w), 1423 (m), 1384 (w), 1322 (vs), 1255 (w), 1146 (vs), 1080 (m), 968 (m), 848 (w), 805 (w), 741 (m), 659 (m), 548 (m), 493 (w) cm^{-1}. MS (ESI): *m*/*z* = 548.29 $[M]^+$, 331.16 $[C_{14}H_{27}N_4O_3S]^+$, 226.93, 158.94, 119.98, 96.02. HRMS (ESI): calcd. for $[C_{24}H_{46}N_5O_5S_2]^+$ 548.2935, found 548.2926 $[M]^+$. Elemental analysis: calcd. (%) for $C_{24}H_{46}IN_5O_5S_2$: C 42.66, H 6.86, N 10.37; found: C 41.98, H 6.59, N 10.13.



Me-C_8^+ I^- VSAm

$C_{24}H_{46}IN_5O_5S_2$
675.69 g/mol

3-Methyl-1-{10-[(vinylsulfonyl)amino]decyl}-4-[({10-[(vinylsulfonyl)amino]decyl}oxy)methyl]-1*H*-1,2,3-triazol-3-ium-iodid (Me-C_{10}^+ I^- VSAm). Following GP 3, from triazole **C_{10} VSAm** (51 mg, 86 µmol) and CH_3I (0.11 mL, 0.25 g, 1.73 mmol) in CH_3CN (6 mL), 8 d at 30 °C, yield: 43 mg, 59 µmol, 68%, yellow oil, R_f = 0.55 (CH_2Cl_2/MeOH 10:1, $KMnO_4$). ^{1}H-NMR (700 MHz, CD_2Cl_2): δ = 1.25 – 1.43 (m, 24H, CH_2), 1.51 – 1.57 (m, 4H, 2‘-H, 2‘‘-H), 1.58 – 1.64 (m, 2H, 9‘‘-H), 2.01 – 2.07 (m, 2H, 9‘-H), 2.92 – 2.98 (m, 4H, 1‘-H, 1‘‘-H), 3.61 (t, *J* = 6.7 Hz, 2H, 10’’-H), 4.32 (s, 3H, NCH_3), 4.69 (t, *J* = 7.5 Hz, 2H, 10‘-H), 4.87 (s, 2H, 6-H), 4.90 (t, *J* = 5.7 Hz, 1H, N*H*), 5.03 (t, *J* = 5.7 Hz, 1H, N*H*), 5.92 (2 d, *J* = 10.0 Hz, 2H, CH=CH_2), 6.16 (2 d, *J* = 16.6 Hz, 2H, CH=CH_2), 6.56 (2 dd, *J* = 10.0, 16.6 Hz, 2H, C*H*=CH_2), 9.20 (s, 1H, 5-H) ppm. ^{13}C-NMR (175 MHz, CD_2Cl_2): δ = 26.2, 26.3, 26.7, 26.8, 28.9, 29.08, 29.16, 29.28, 29.29, 29.48, 29.56, 29.58, 29.7, 30.0, 30.2 (CH_2), 39.4 (NCH_3), 43.39, 43.44 (C-1’, C-1’’), 54.8 (C-10’), 60.9 (C-6), 72.4 (C-10’’), 126.45, 126.53 (CH=*C*H_2), 130.7 (C-5), 136.5, 136.6 (*C*H=CH_2), 141.0 (C-4) ppm. FT-IR (ATR, CD_2Cl_2): $\tilde{\nu}$ = 3134 (br), 2928 (vs), 2854 (s), 1462 (w), 1425 (m), 1383 (w), 1325 (vs), 1255 (w), 1149 (vs), 1094 (m), 970 (m), 740 (m), 661 (m), 557 (w) cm^{-1}. MS (ESI): *m*/*z* = 604.35 $[M]^+$, 512.35 $[C_{26}H_{50}N_5O_3S]^+$, 359.20 $[C_{16}H_{31}N_4O_3S]^+$. HRMS (ESI): calcd. for $[C_{28}H_{54}N_5O_5S_2]^+$ 604.3561, found 604.3551 $[M]^+$. Elemental analysis: calcd. (%) for $C_{28}H_{54}IN_5O_5S_2$: C 45.96, H 7.44, N 9.57; found: C 46.23, H 7.60, N 9.45.

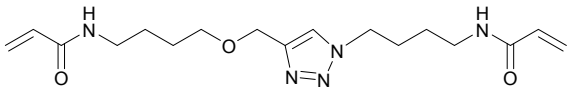


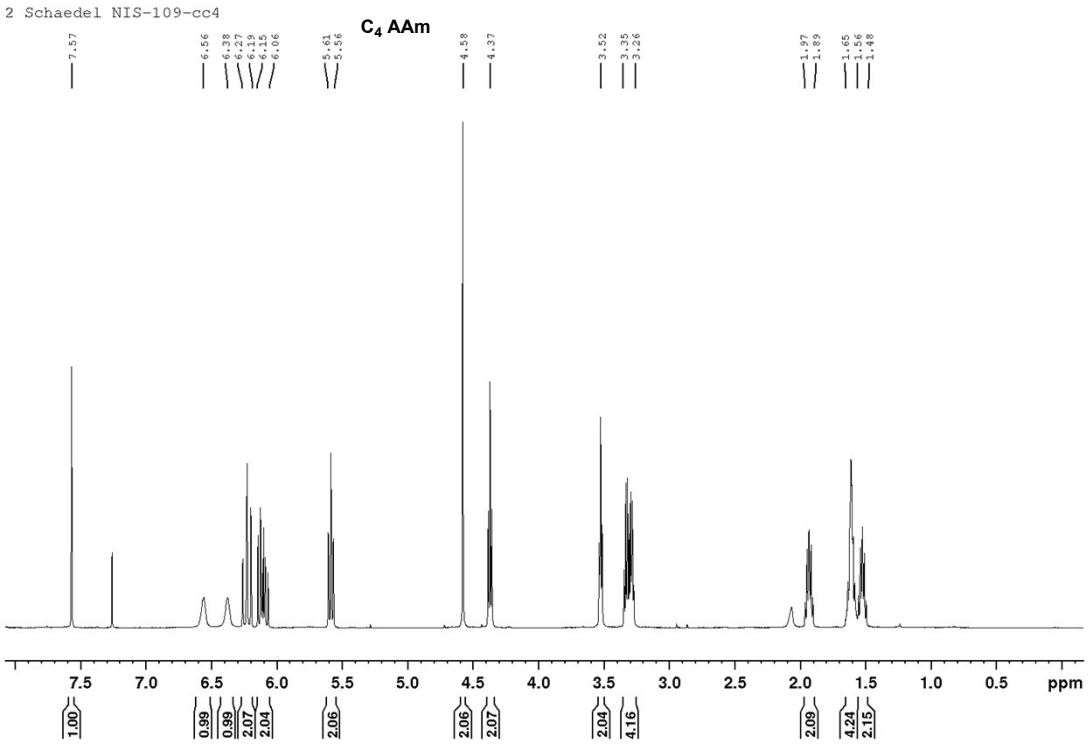
Me-C_{10}^+ I^- VSAm

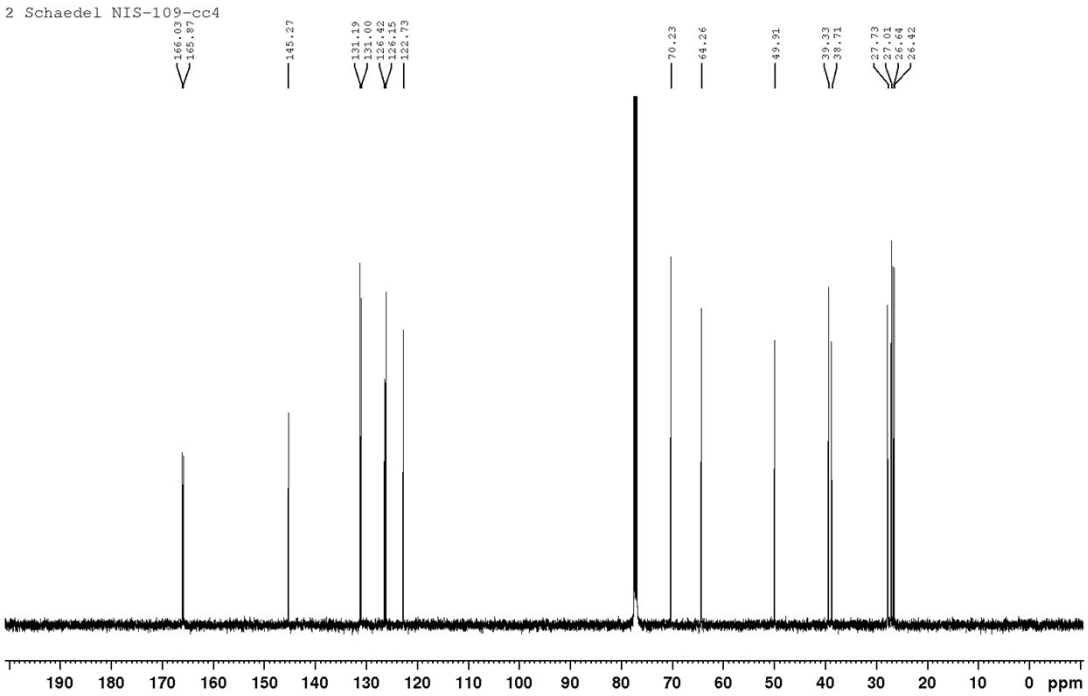
$C_{28}H_{54}IN_5O_5S_2$
731.26 g/mol

2. ^{1}H- and ^{13}C-NMR Spectra of Cross-linkers

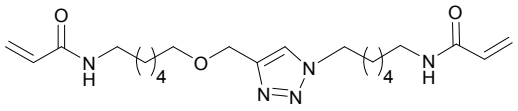
C_4 AAm



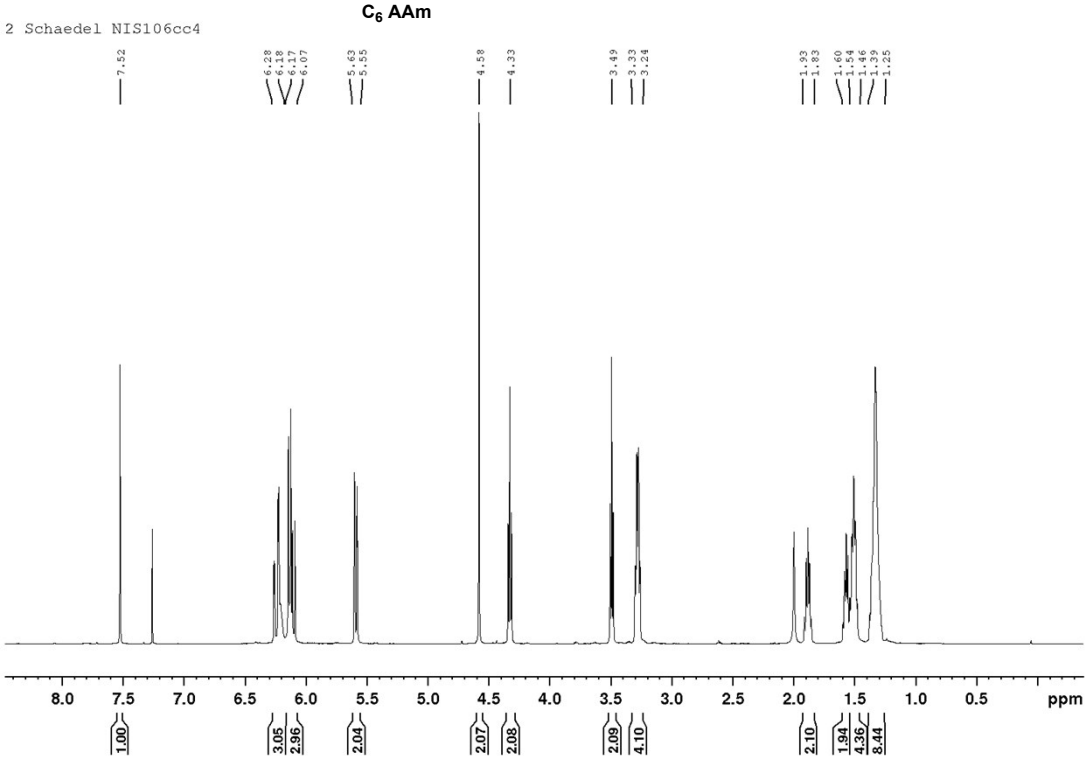


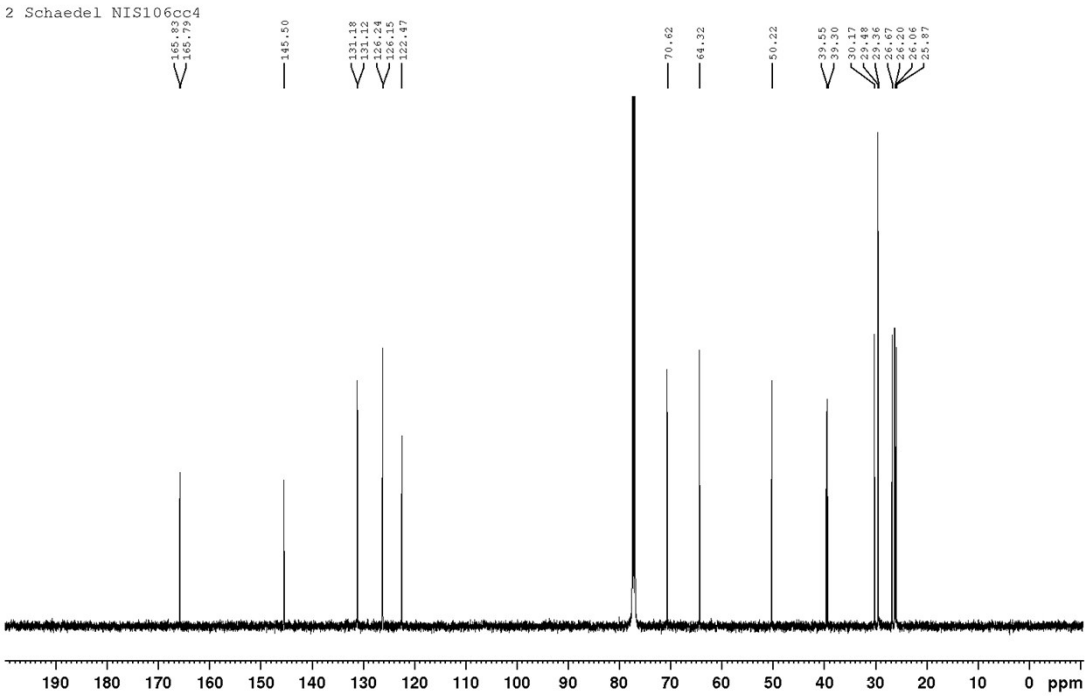


C_6 AAm

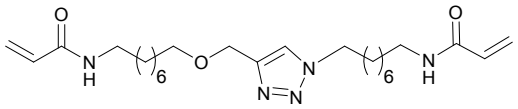

O
N
H
4
O
N
N=N
4
N
H
O

C_6 AAm

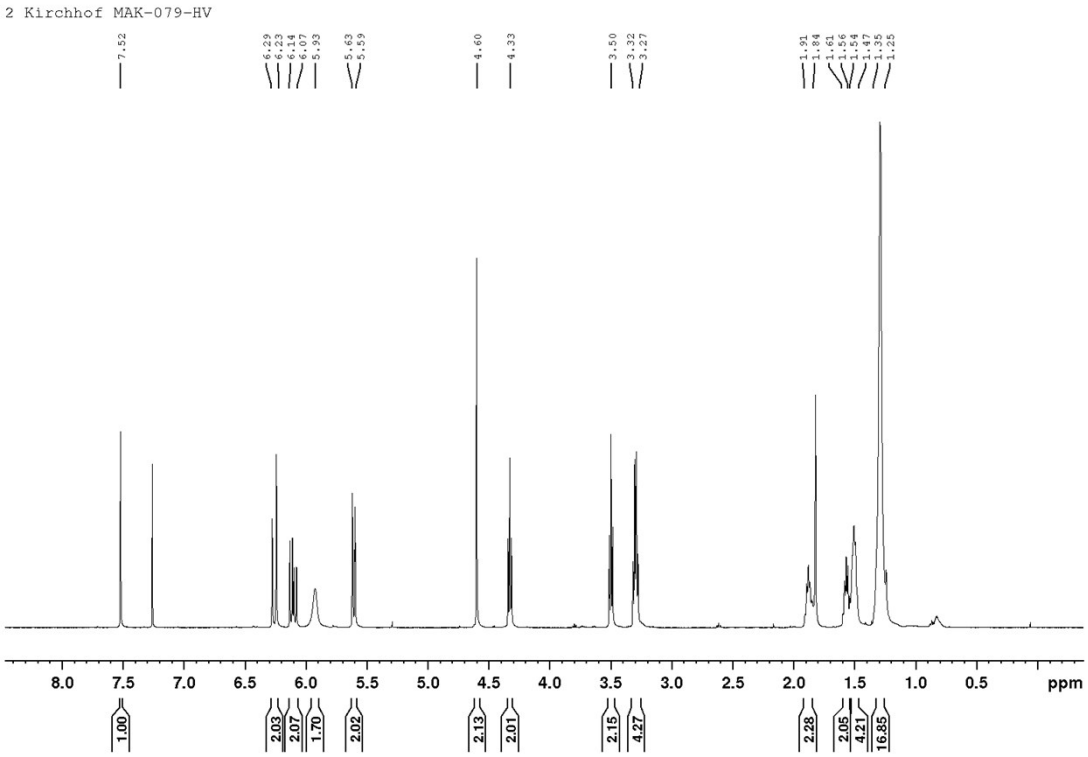

2 Schaedel NIS106cc4
7.52
6.28
6.18
6.17
6.07
5.63
5.55
4.58
4.33
3.49
3.33
3.24
1.93
1.83
1.60
1.54
1.46
1.38
1.25
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.00
3.05
2.96
2.04
2.07
2.08
2.09
4.10
2.10
1.94
4.36
8.44

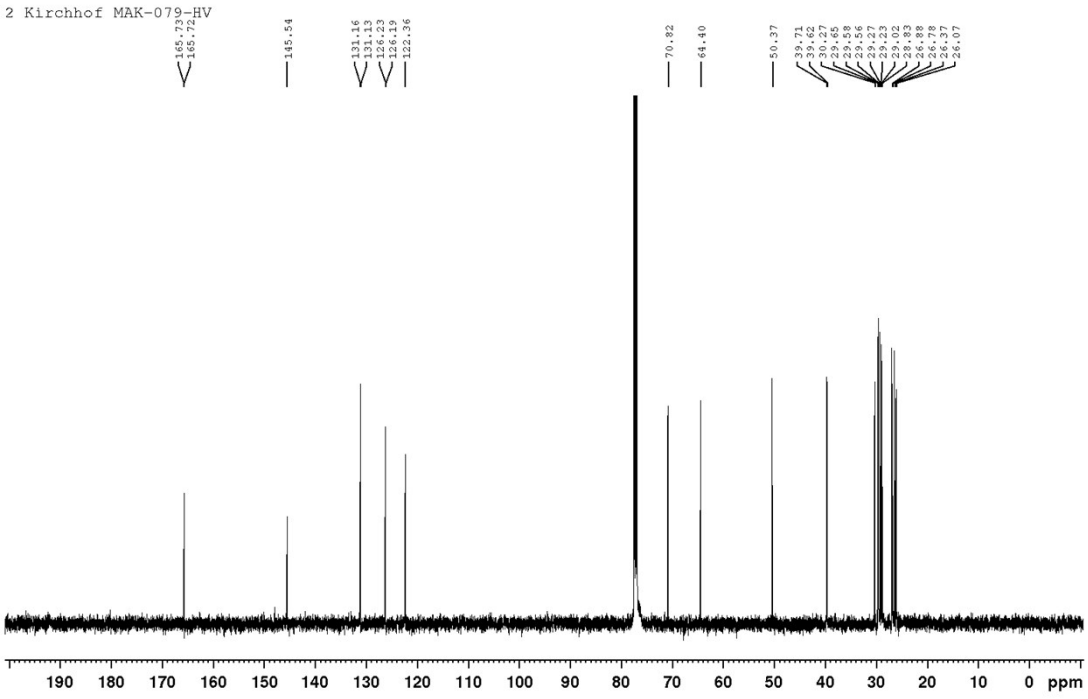

2 Schaedel NIS106cc4
165.83
165.79
145.50
131.18
131.12
126.24
126.15
122.47
70.62
64.32
50.22
39.55
39.30
30.17
29.48
29.36
26.67
26.20
26.06
25.87
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

C_8 AAm

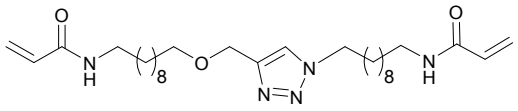


C_8 AAm

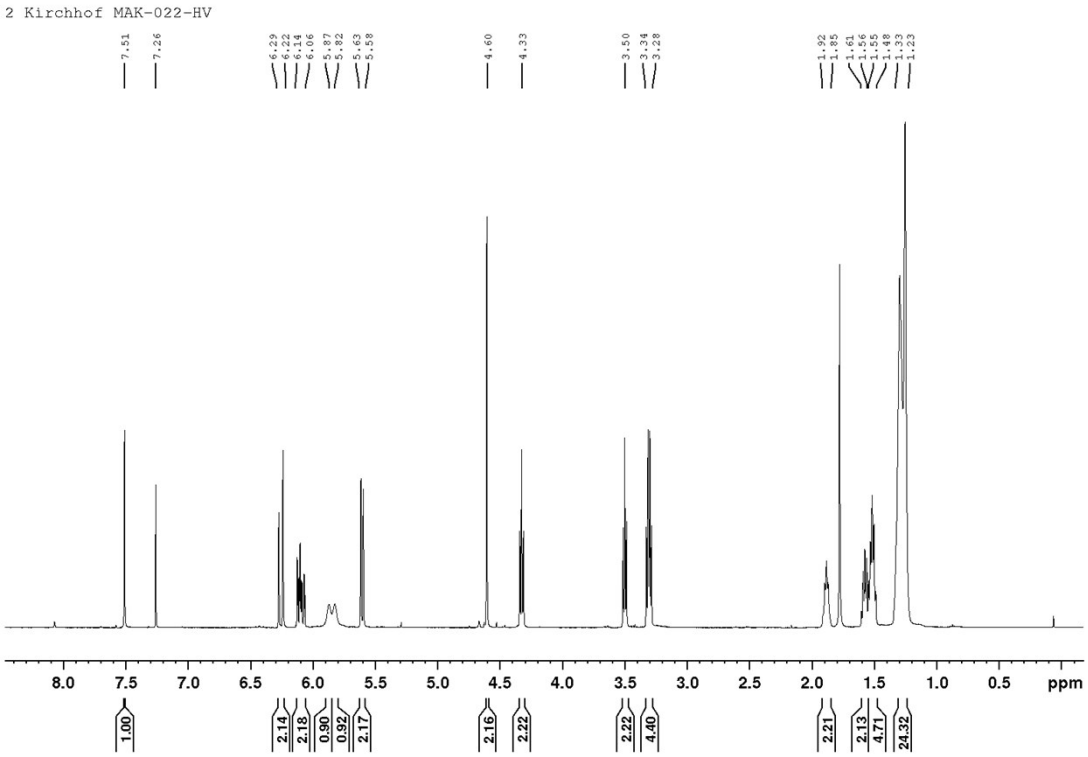

2 Kirchhof MAK-079-HV
7.52
6.29
6.23
6.14
6.07
5.93
5.63
5.59
4.60
4.33
3.50
3.32
3.27
1.91
1.84
1.61
1.56
1.54
1.47
1.35
1.25
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.00
2.03
2.07
1.70
2.02
2.13
2.01
2.15
4.27
2.28
2.05
4.21
16.85

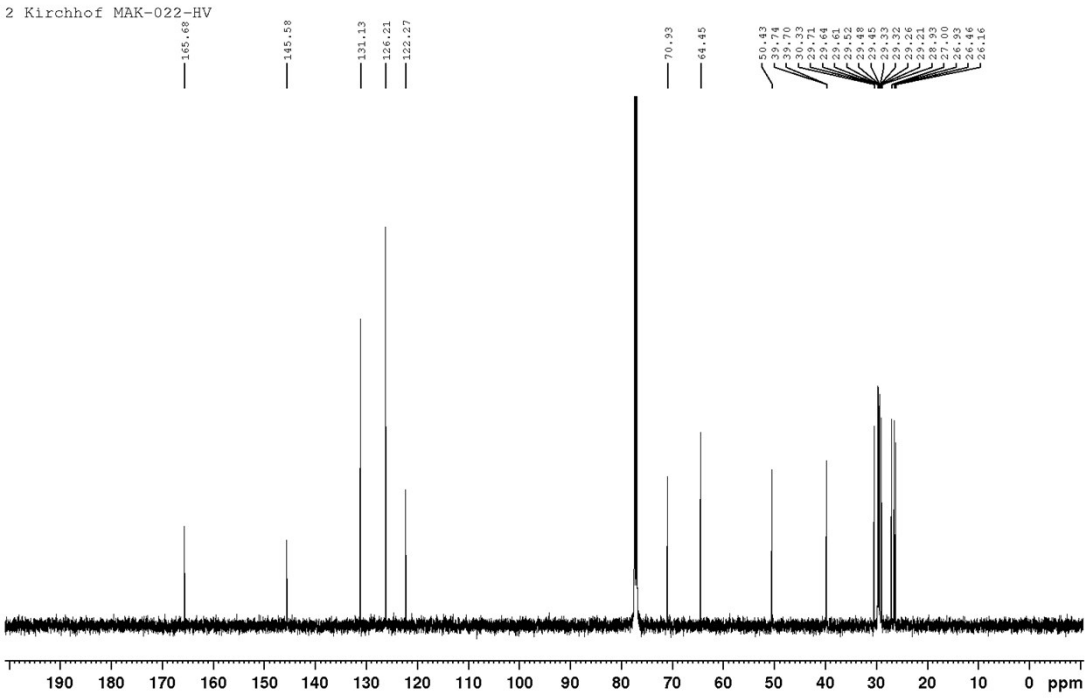

2 Kirchhof MAK-079-HV
165.73
165.72
145.54
131.16
131.13
126.23
126.19
122.36
70.82
64.40
50.37
39.71
39.62
30.27
29.65
29.58
29.56
29.27
29.23
29.02
28.83
26.88
26.78
26.37
26.07
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

C_{10} AAm

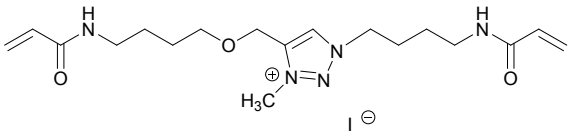


C_{10} AAm

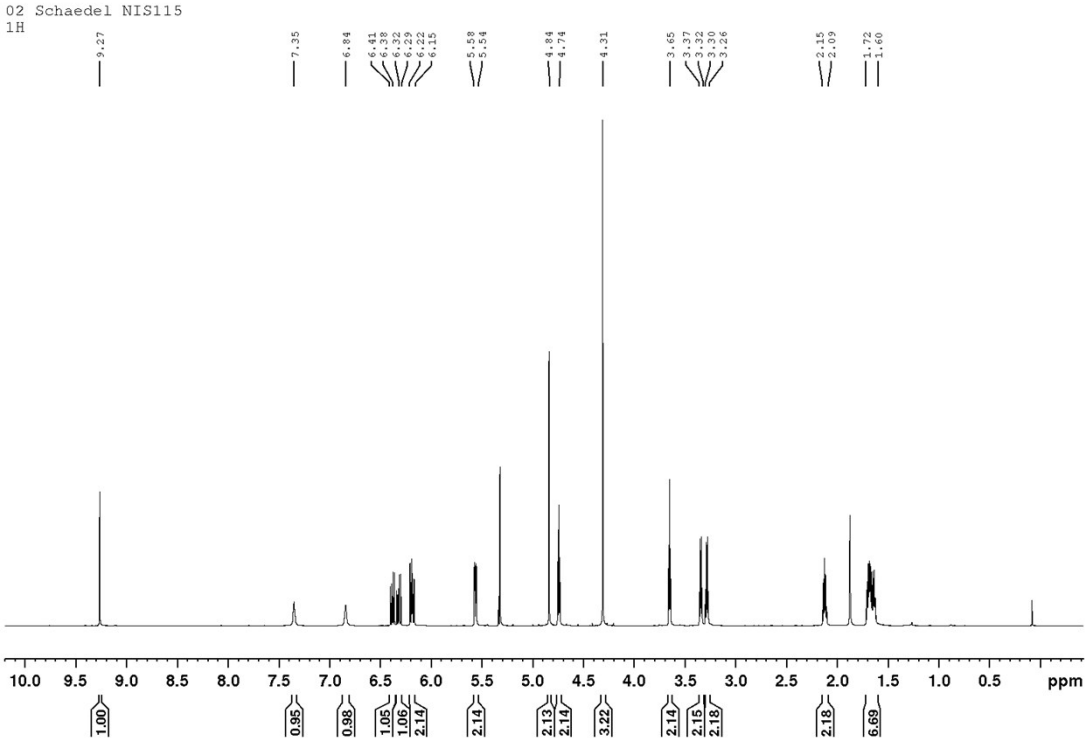


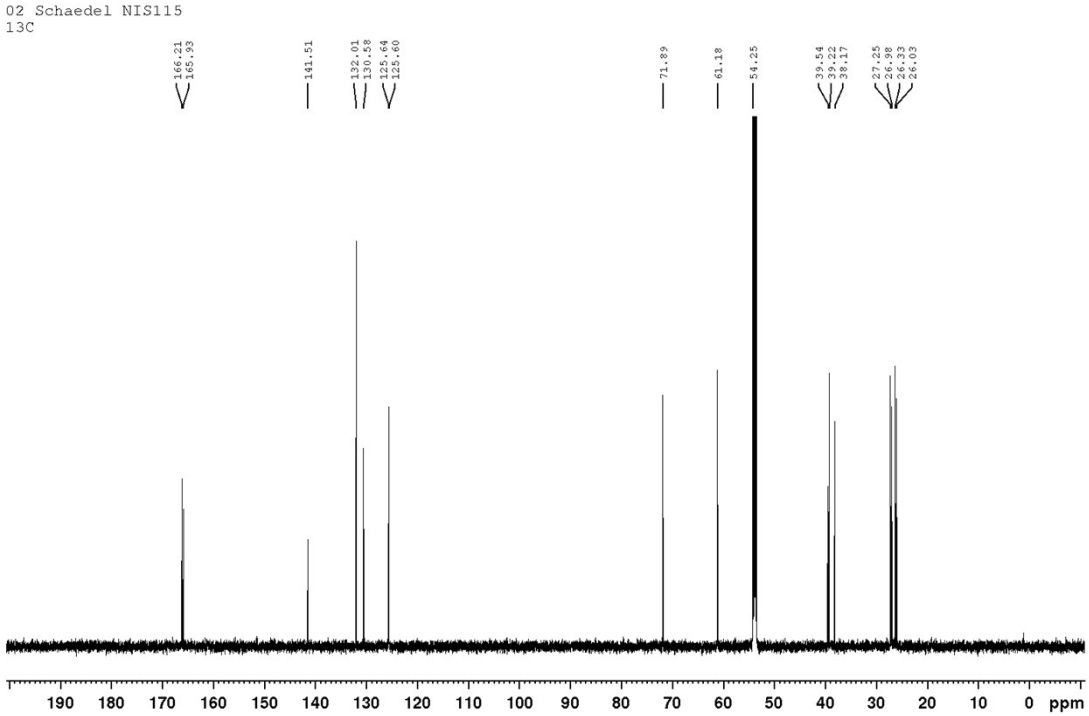


Me-C_4^+ I^- Aam

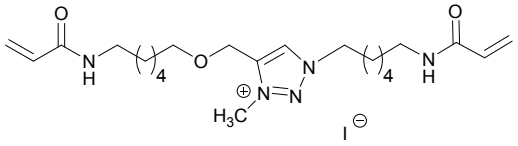


Me-C_4^+ I^- AAm

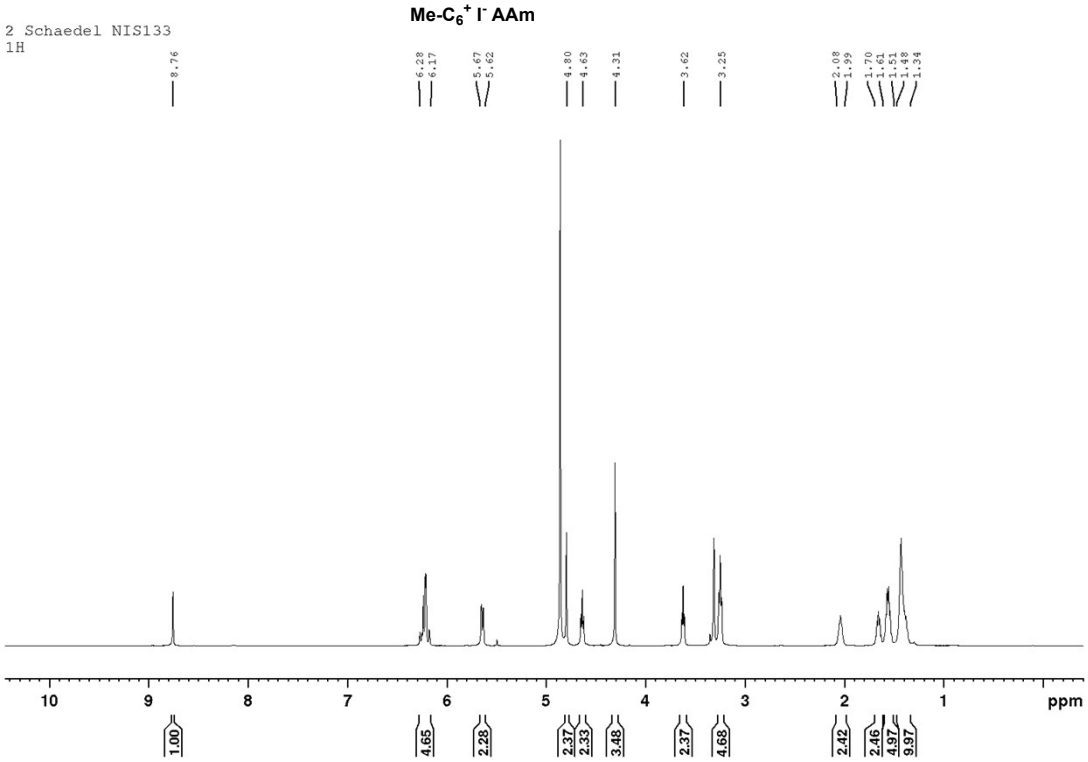


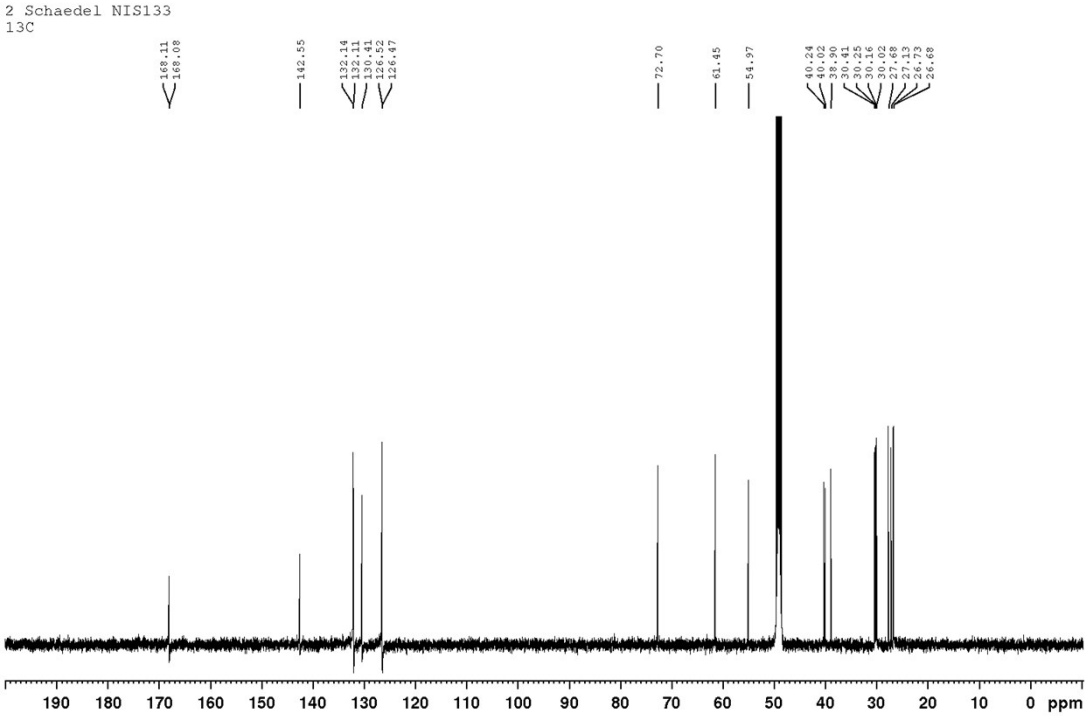


Me-C_6^+ I^- Aam

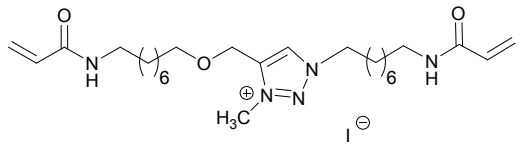


Me-C_6^+ I^- AAm

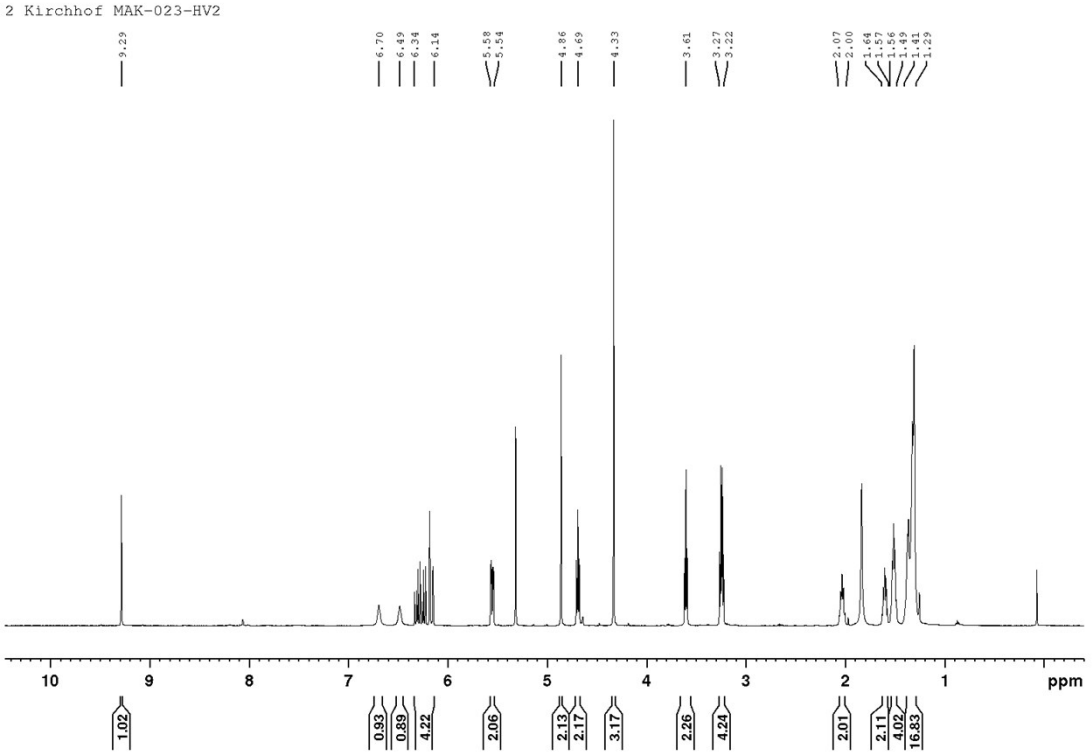


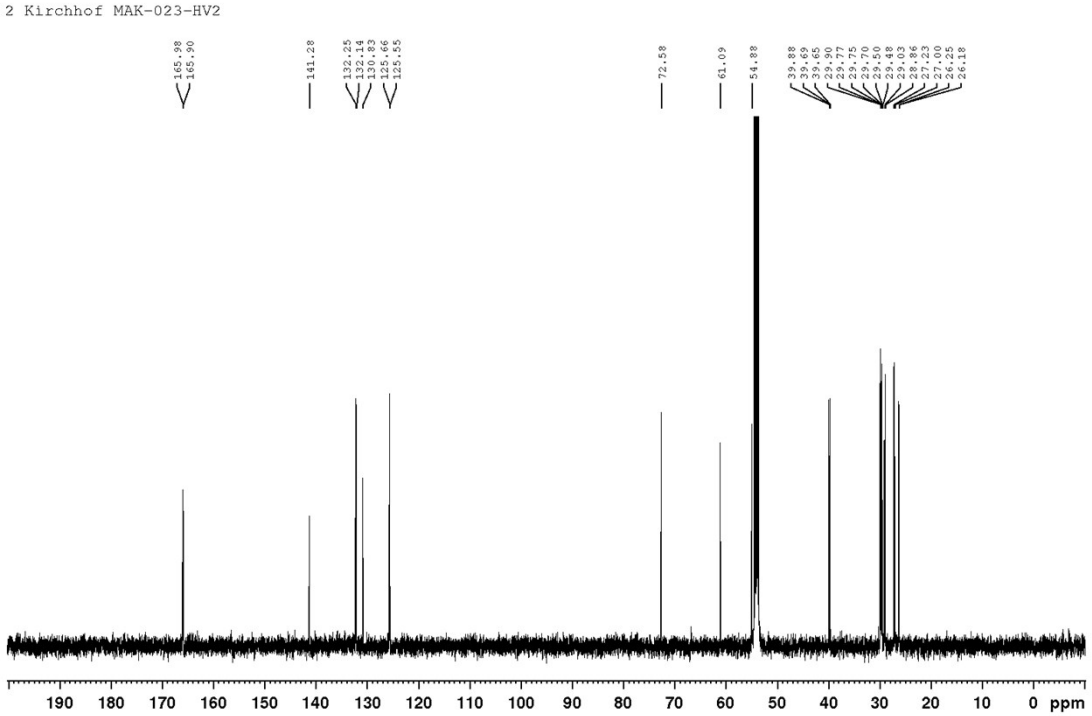


Me-C_8^+ I^- Aam

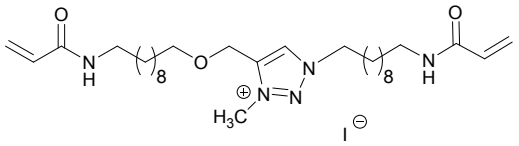


Me-C_8^+ I^- AAm

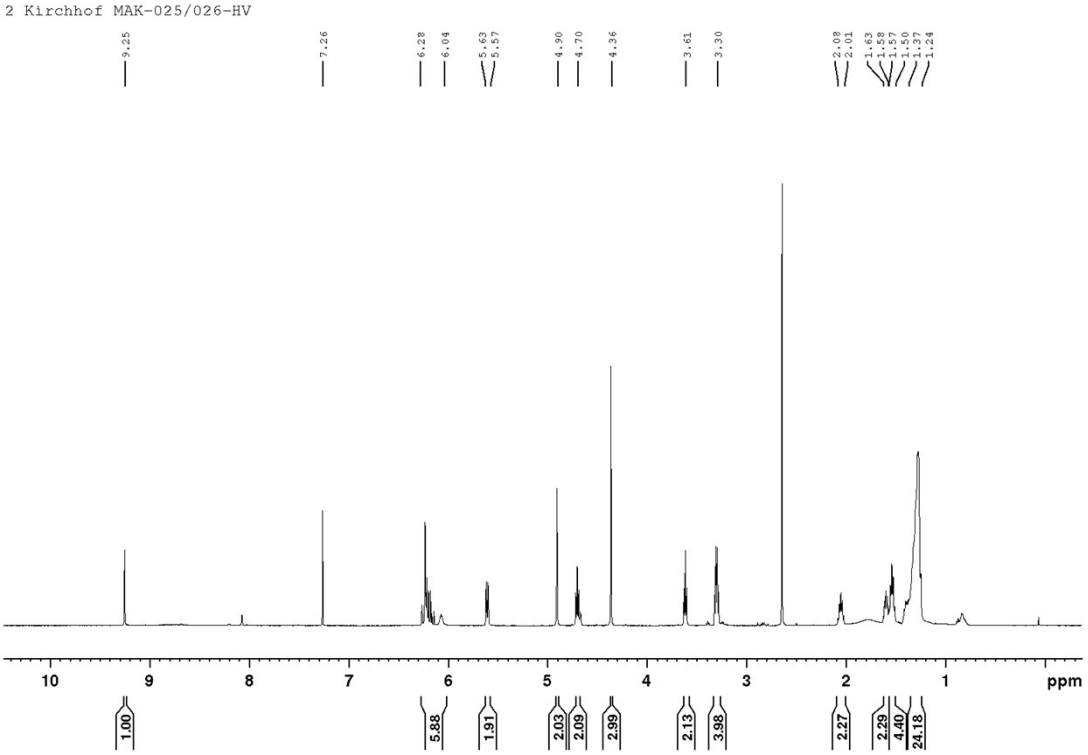


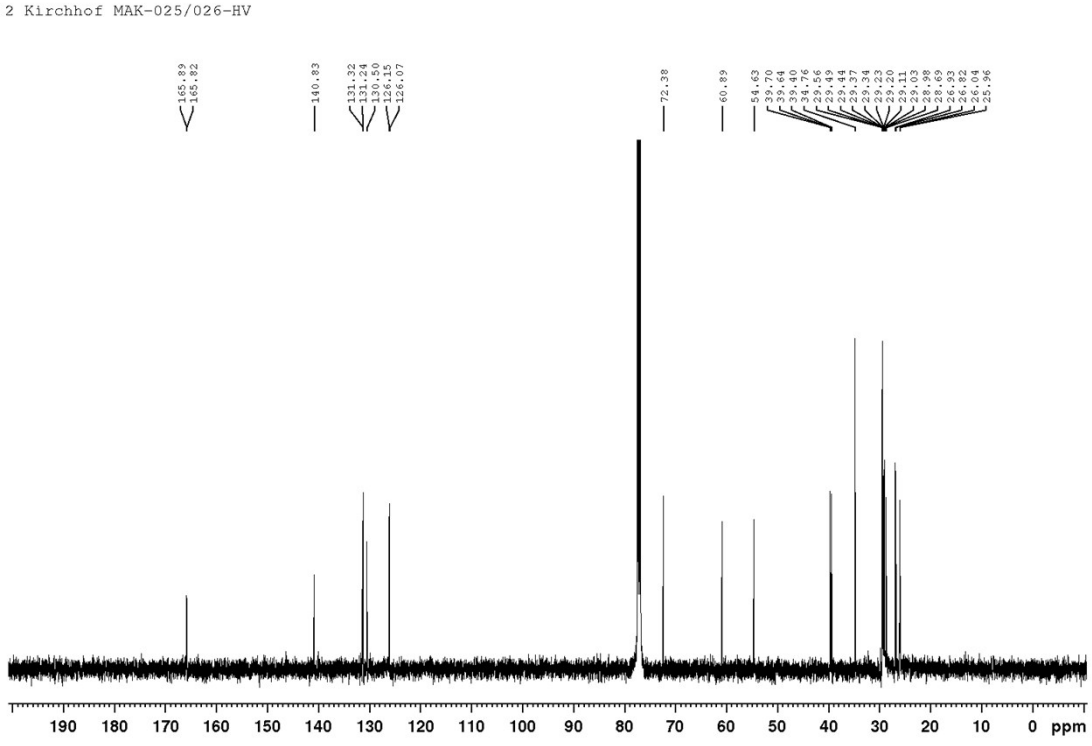


Me-C_{10}^{+} I^{-} Aam

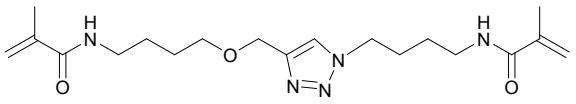


Me-C_{10}^{+} I^{-} AAm

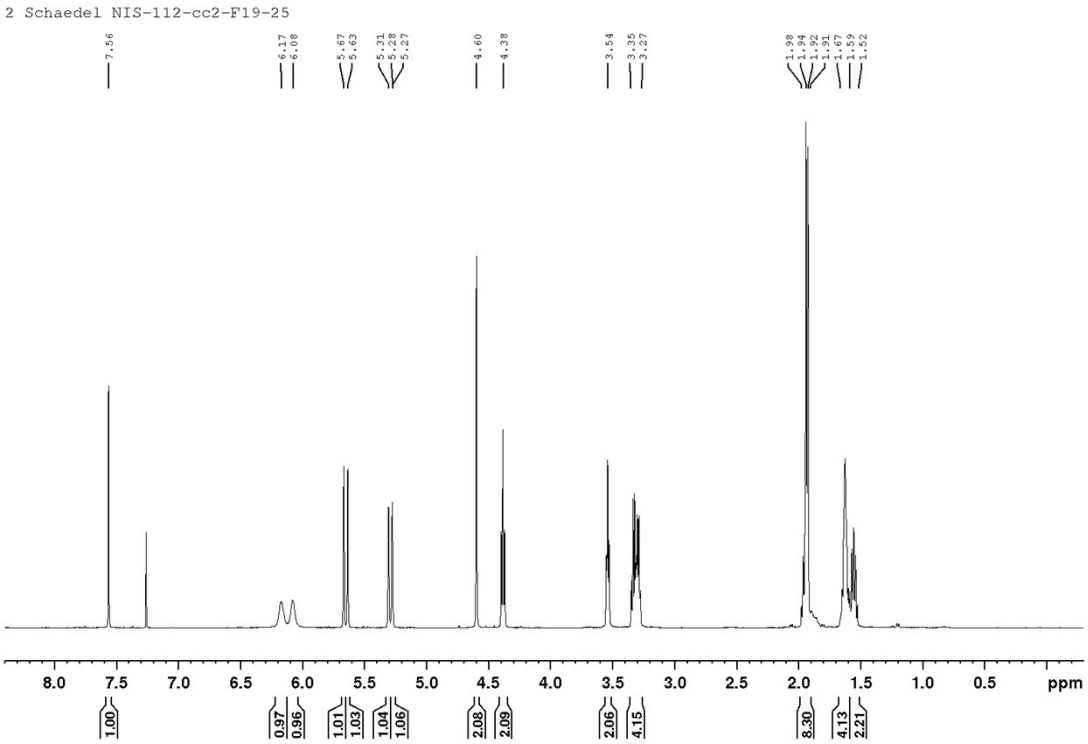


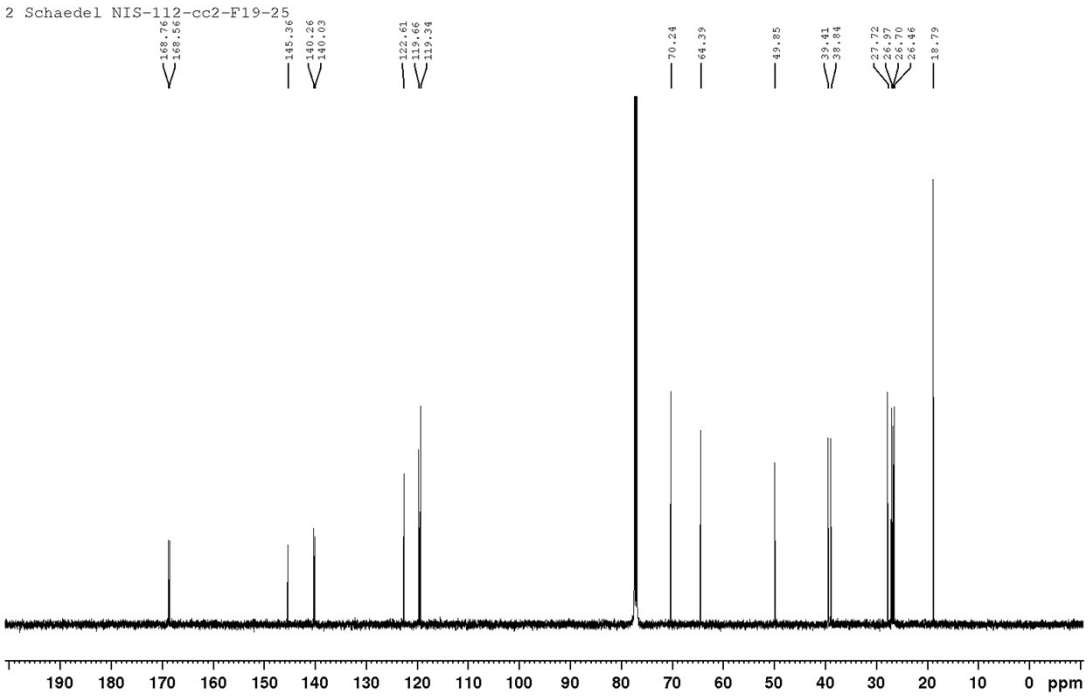


C_4 MeAAm

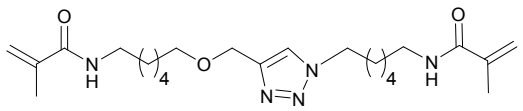


C_4 MeAAm

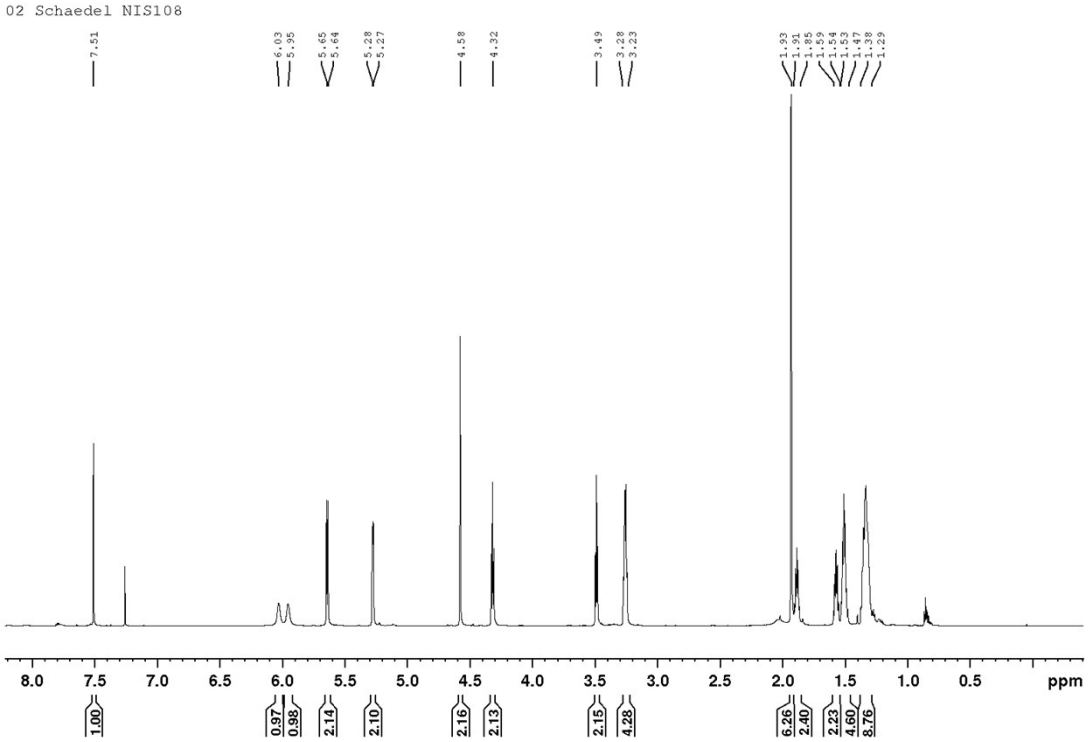


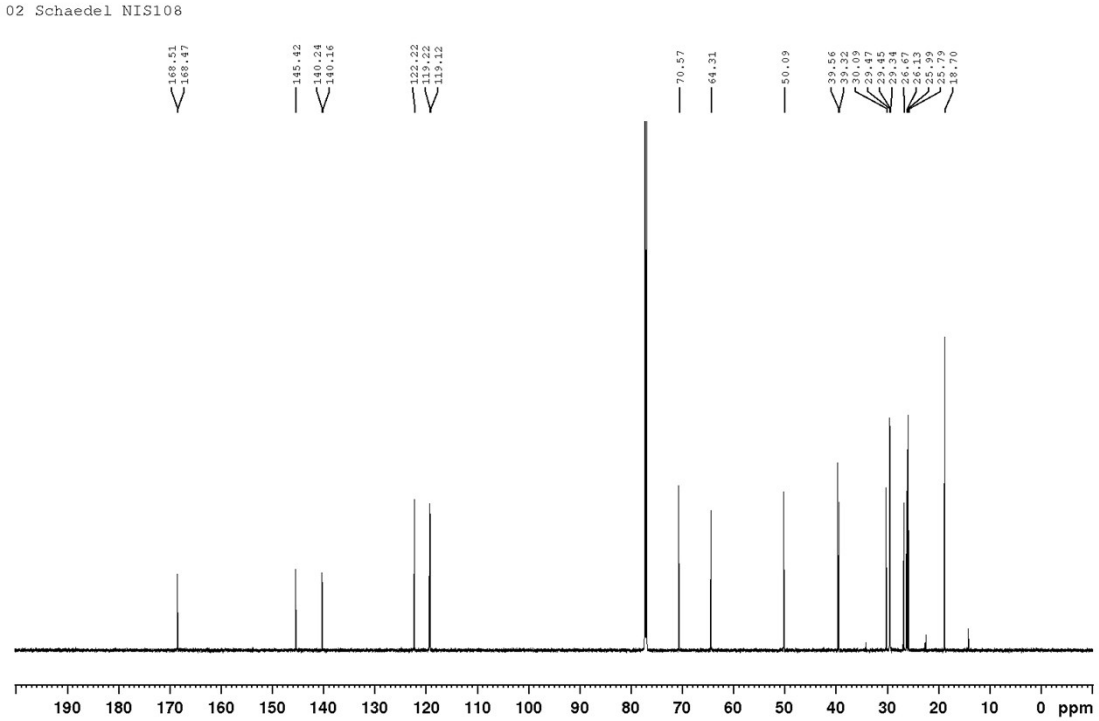


C_6 MeAAm

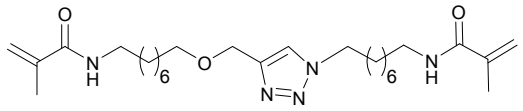


C_6 MeAAm

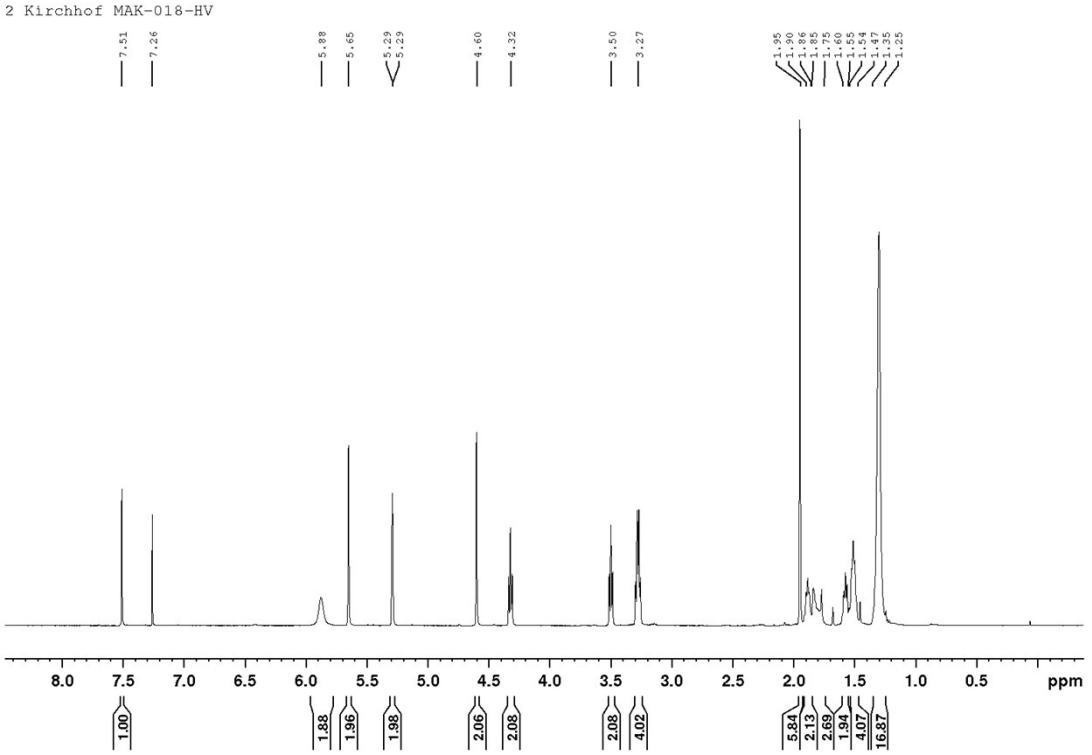


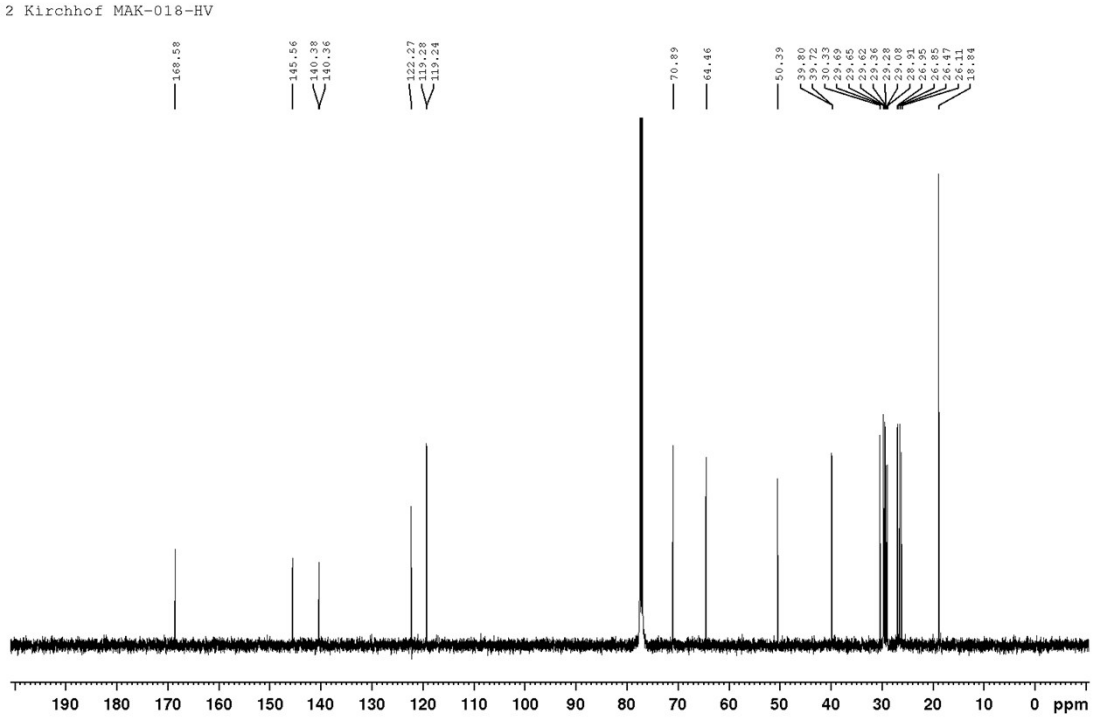


C_8 MeAAm

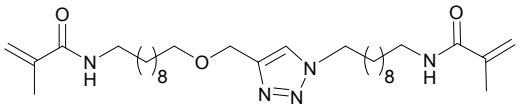


C_8 MeAAm

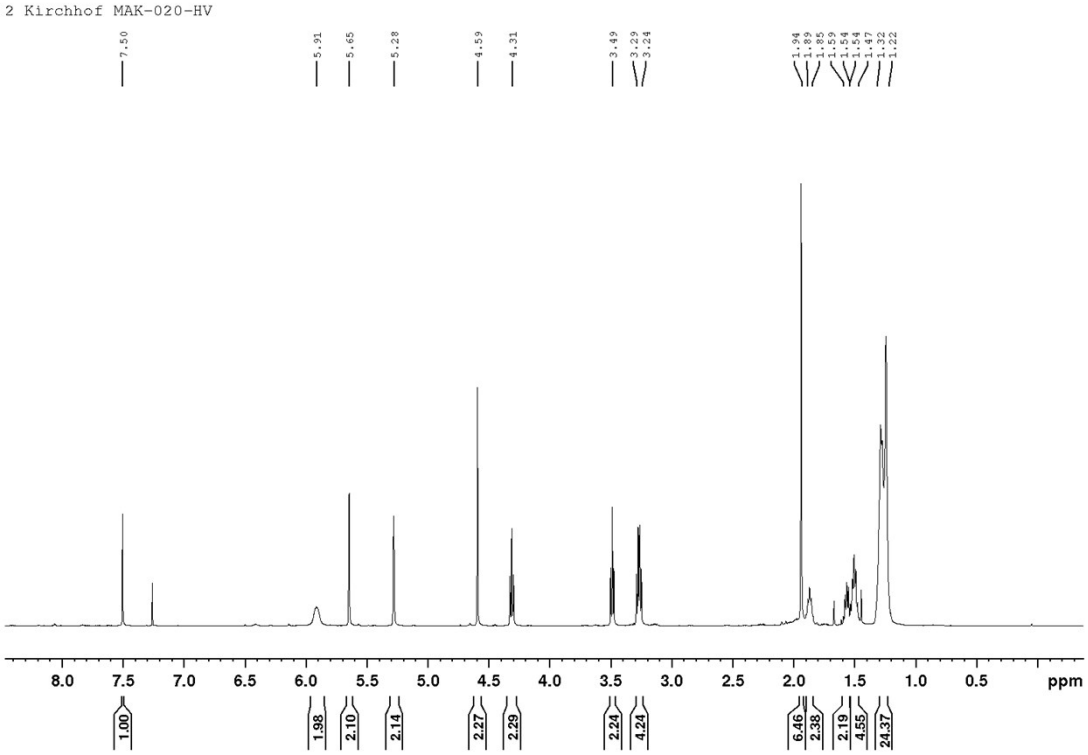


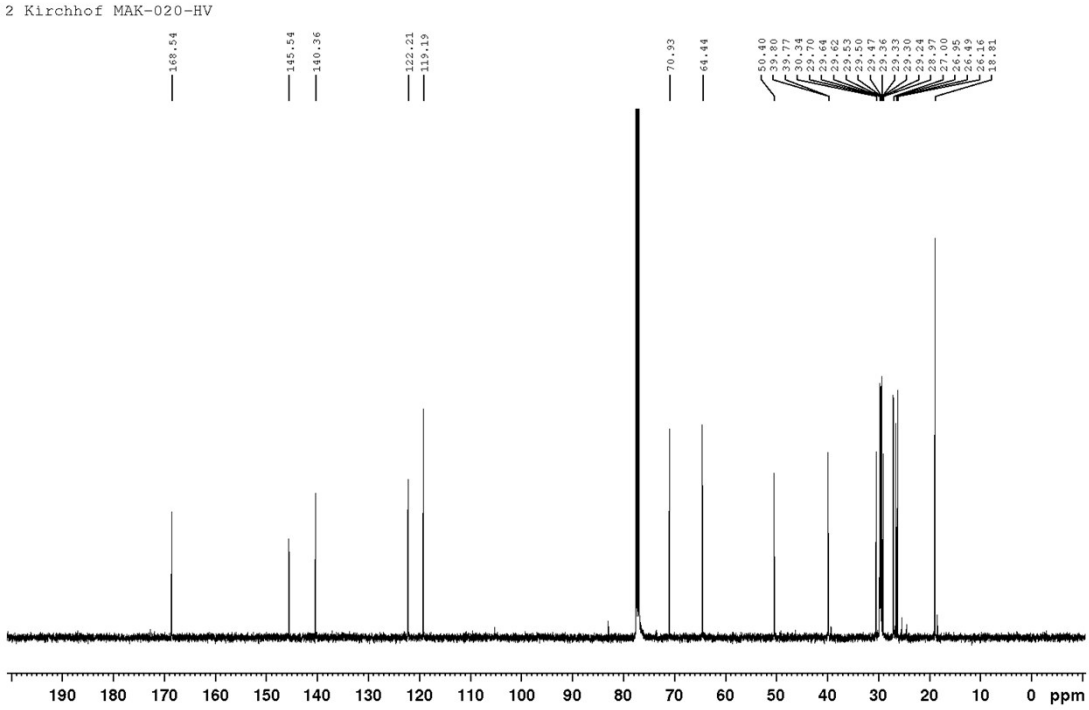


C_{10} MeAAm

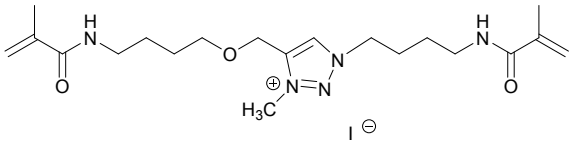


C_{10} MeAAm

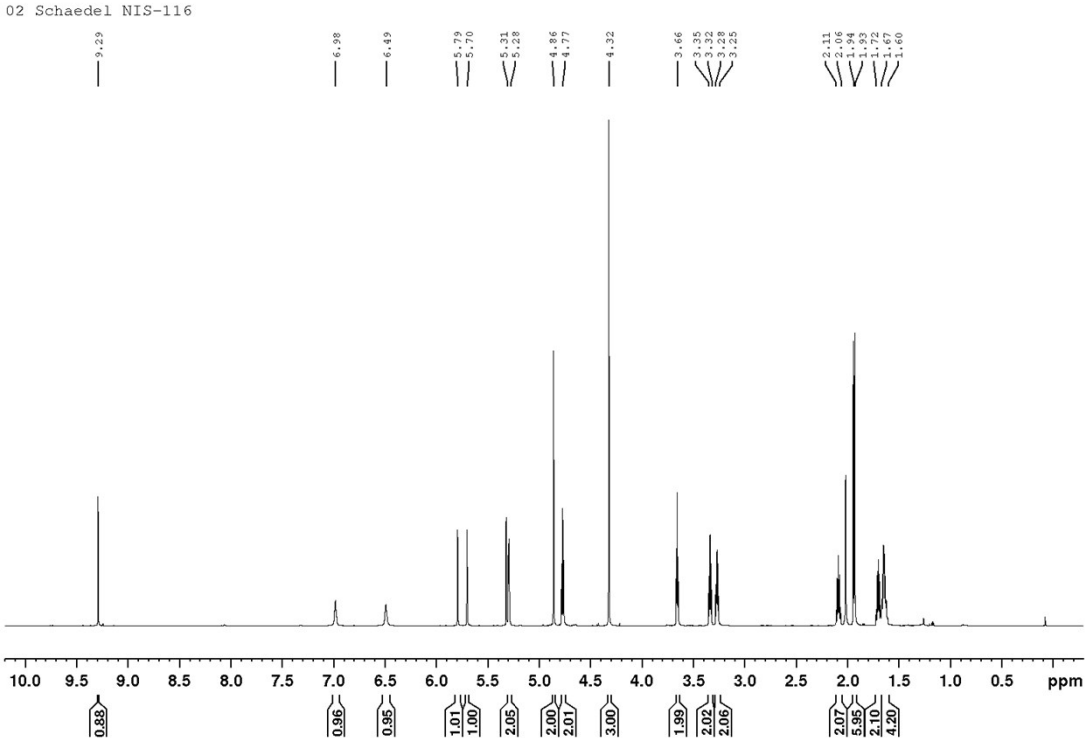


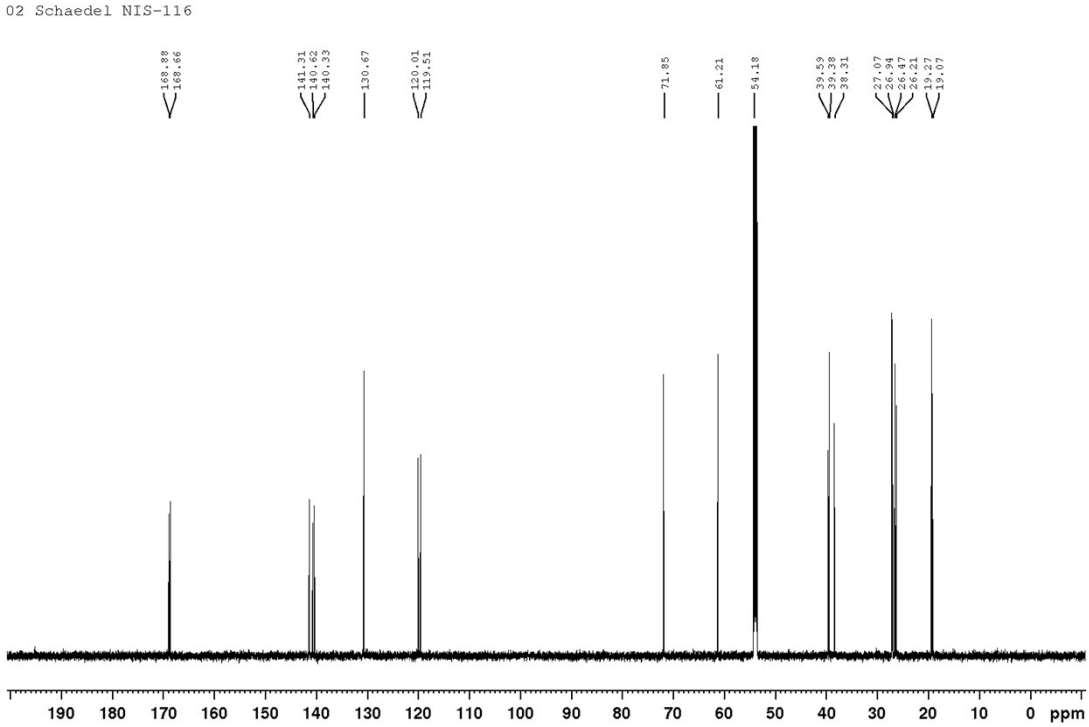


Me-C_4^+ I^- MeAAm

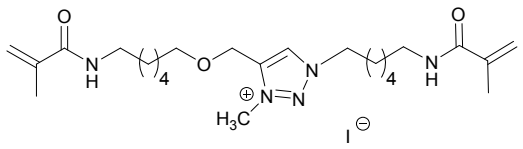


Me-C_4^+ I^- MeAAm

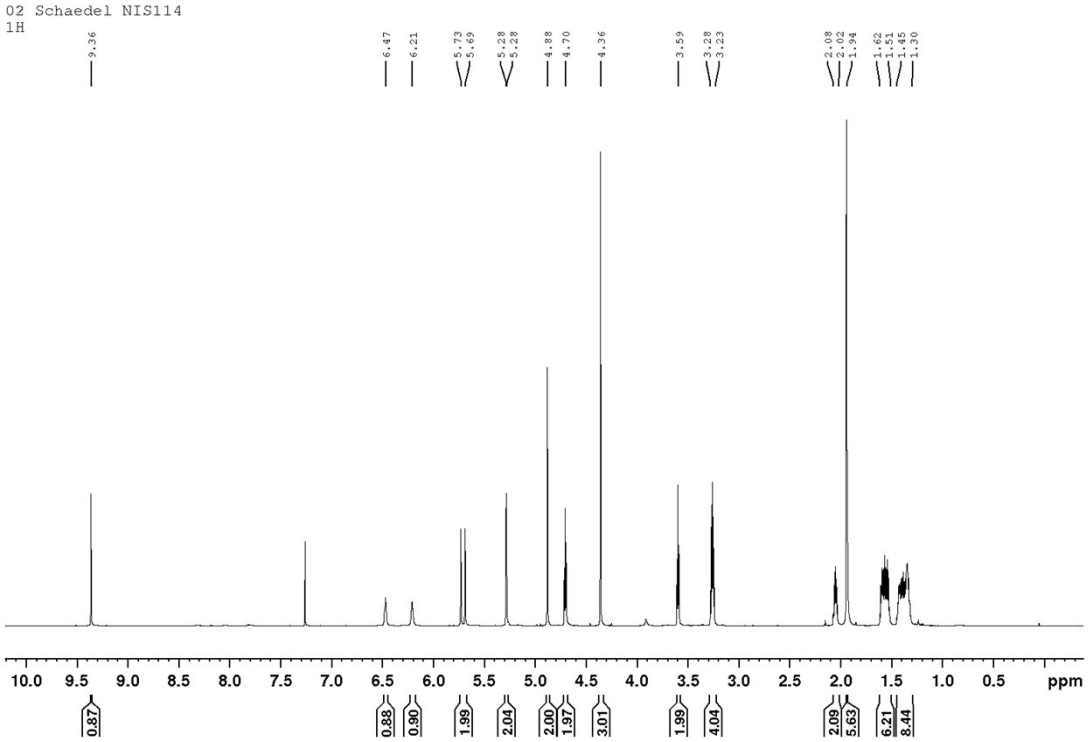


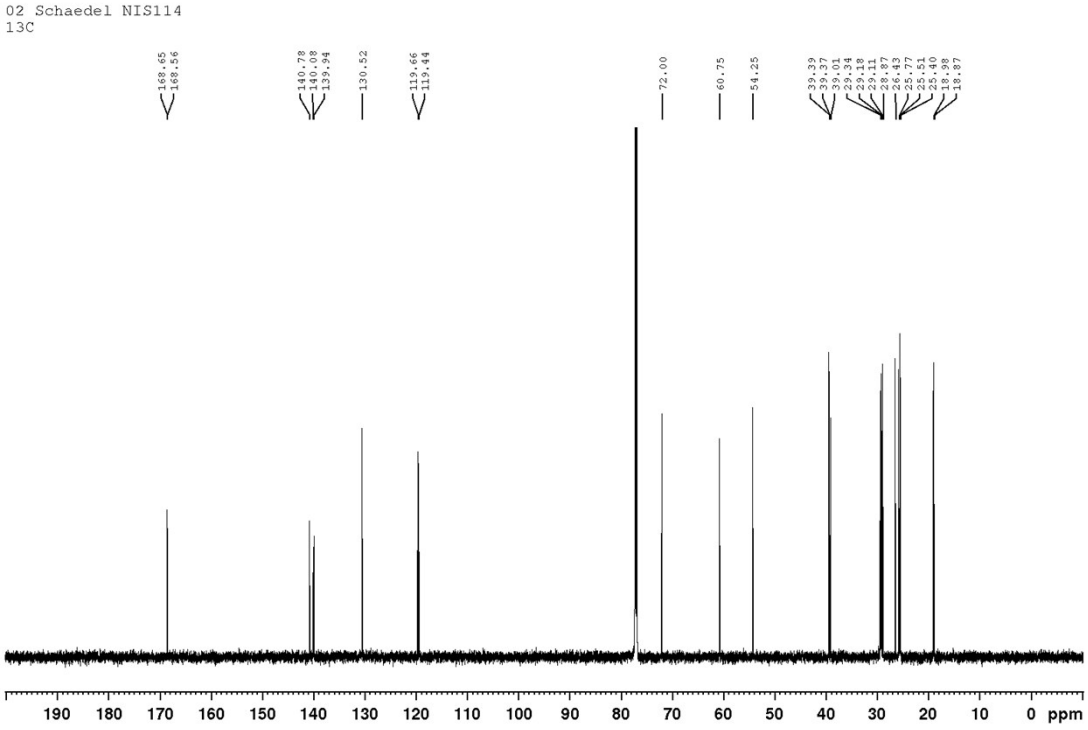


Me-C_6^+ I^- MeAAm

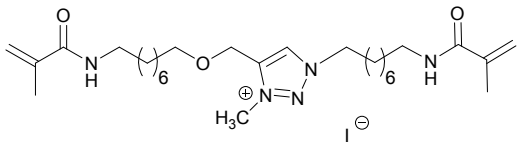


Me-C_6^+ I^- MeAAm

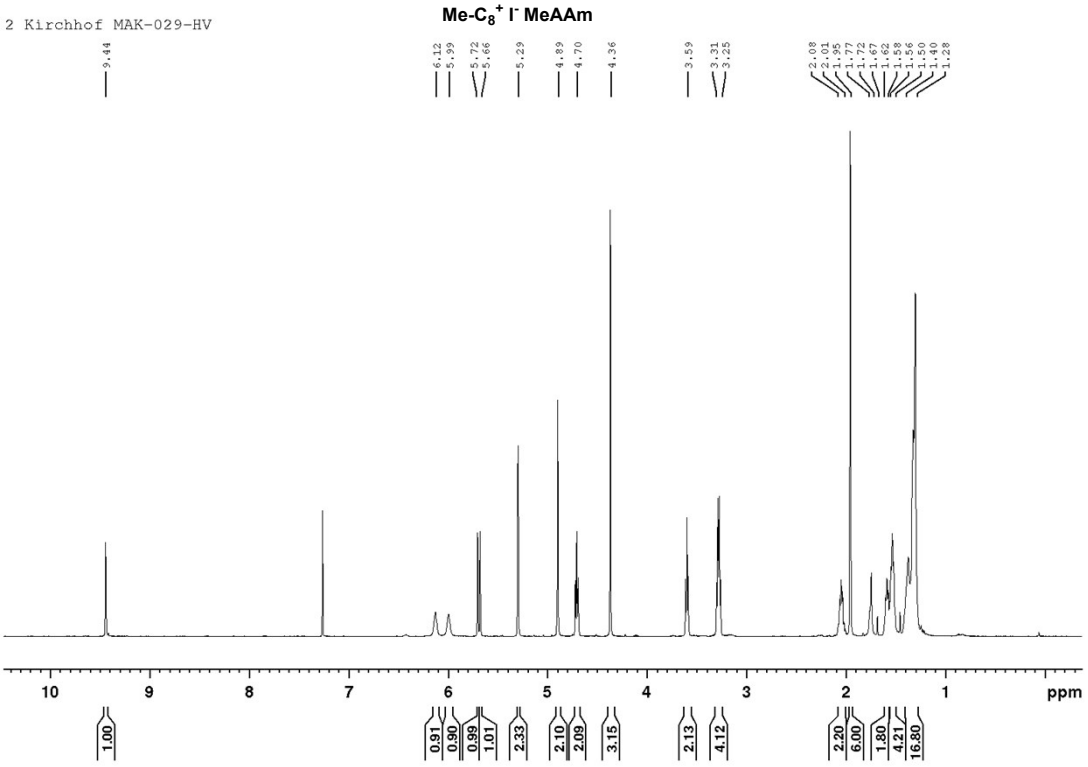


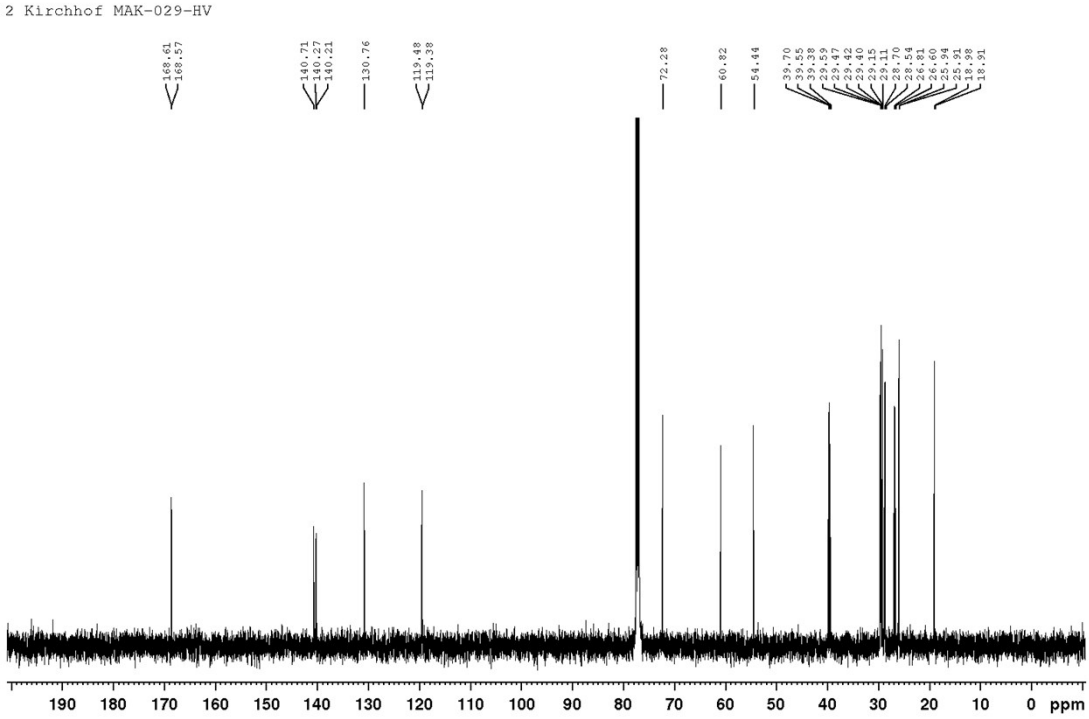


Me-C_8^+ I^- MeAAm

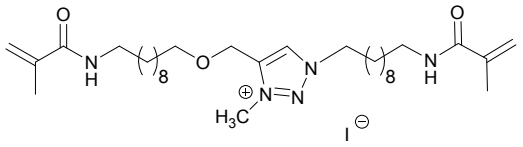


Me-C_8^+ I^- MeAAm

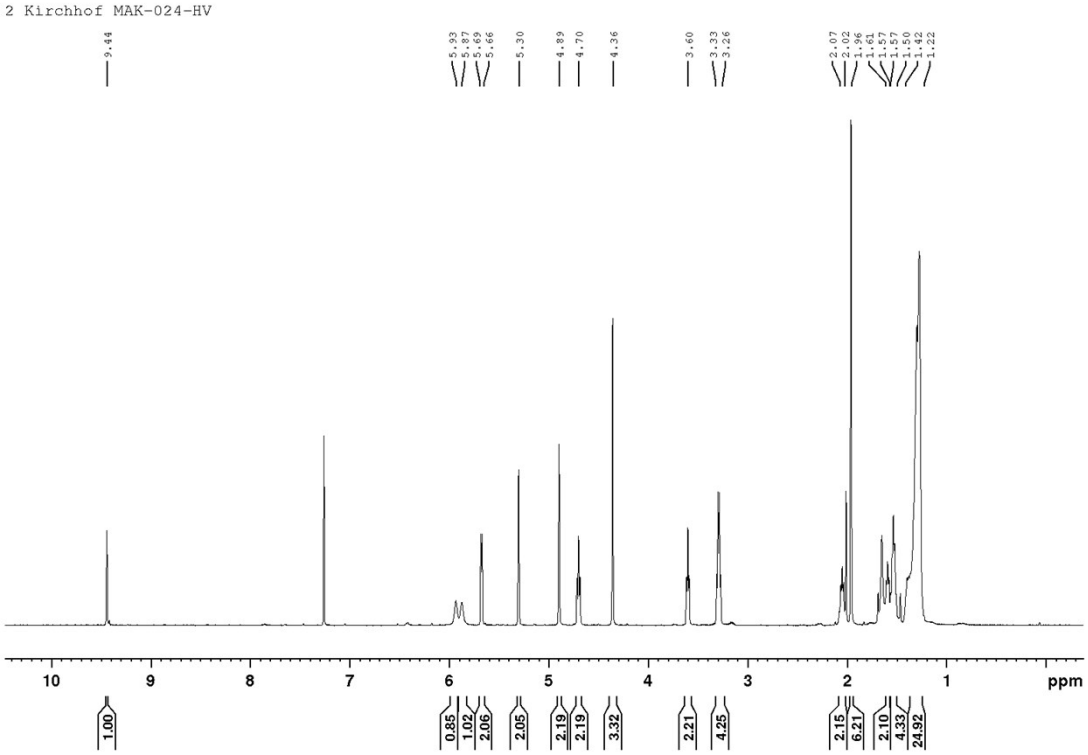


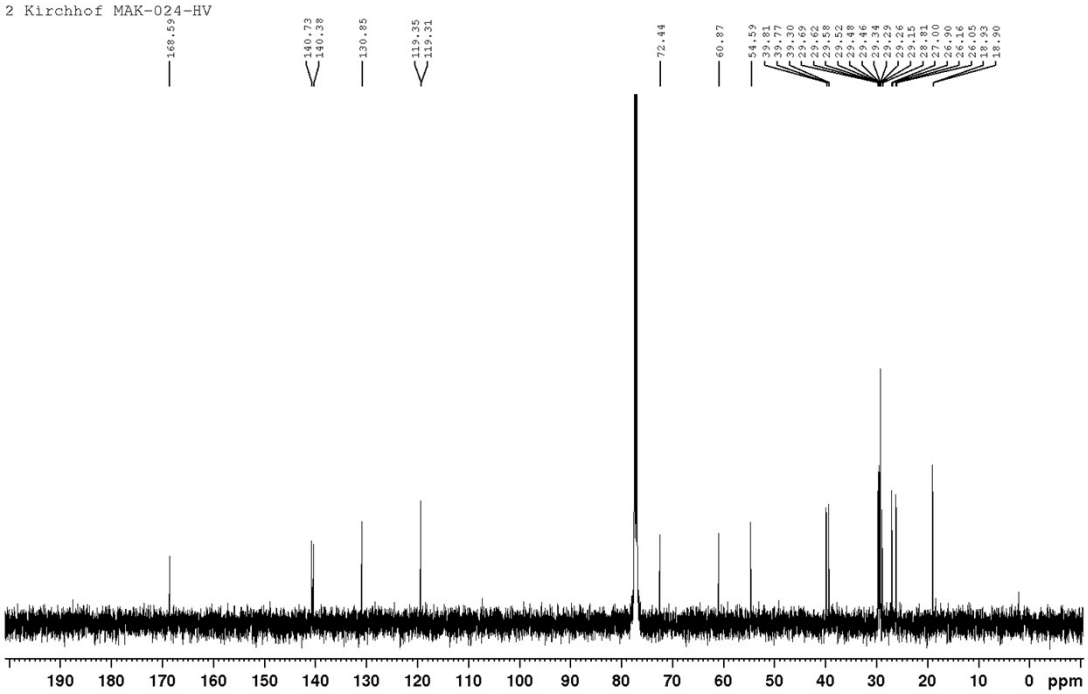


Me-C_{10}^{+} I^{-} MeAAm

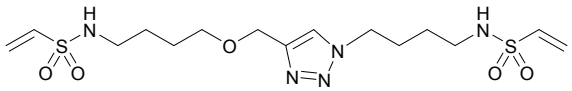


Me-C_{10}^{+} I^{-} MeAAm

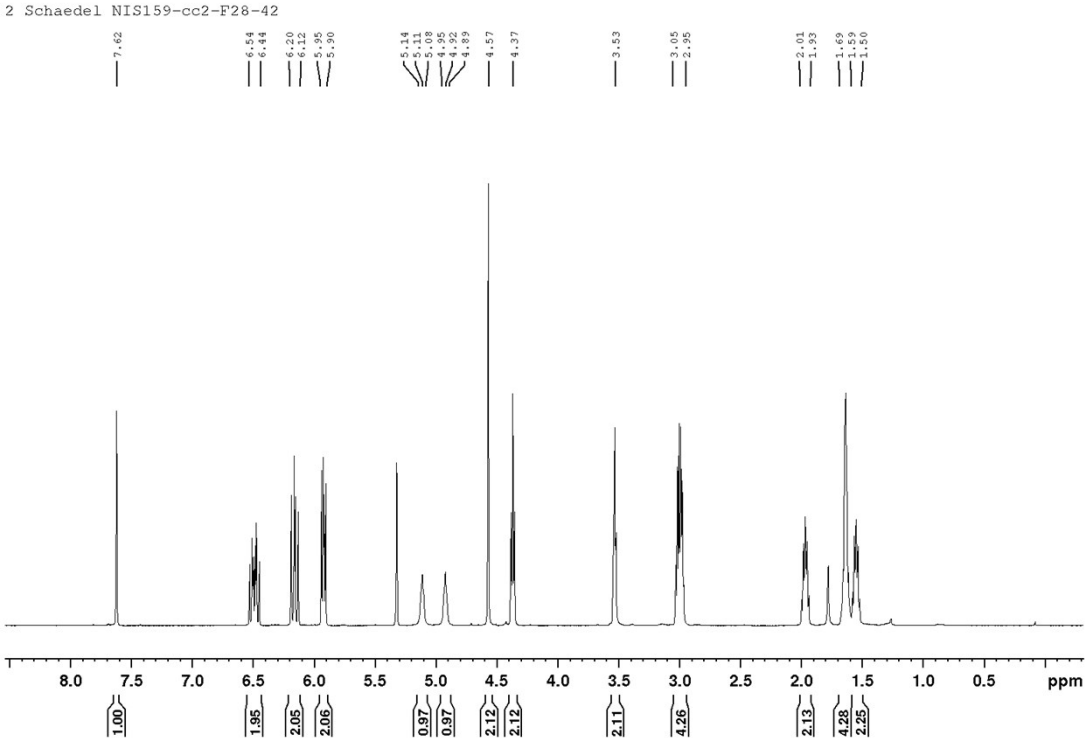


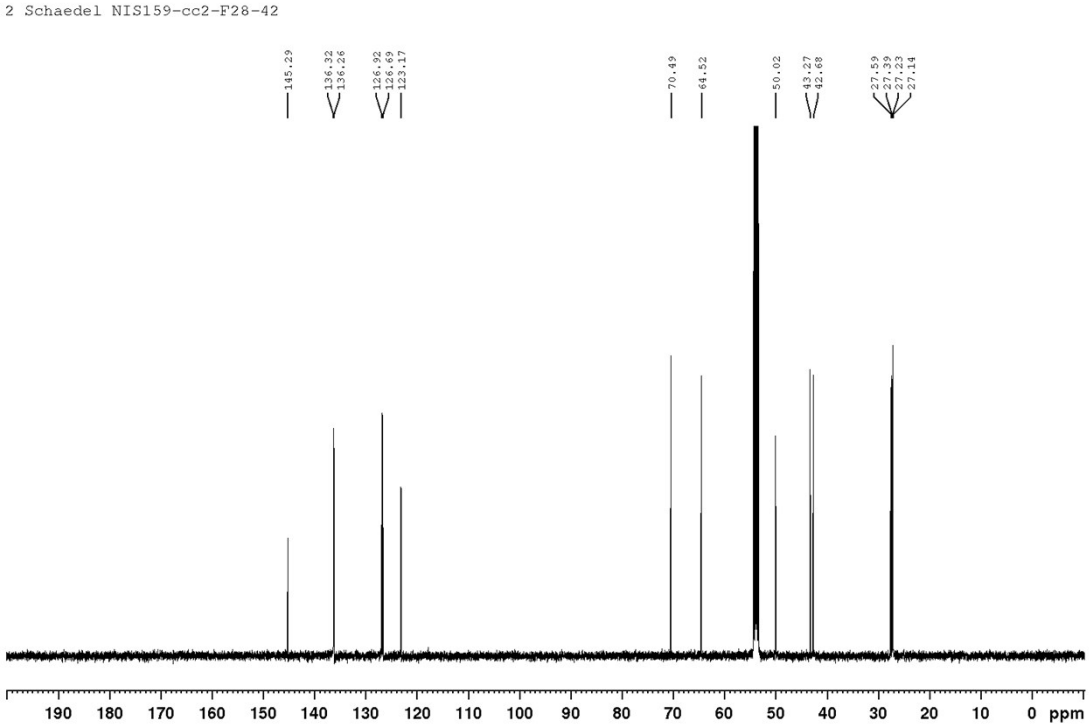


C_4 VSAm

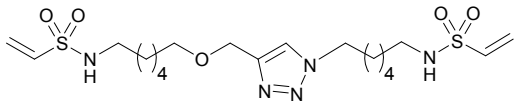


C_4 VSAm

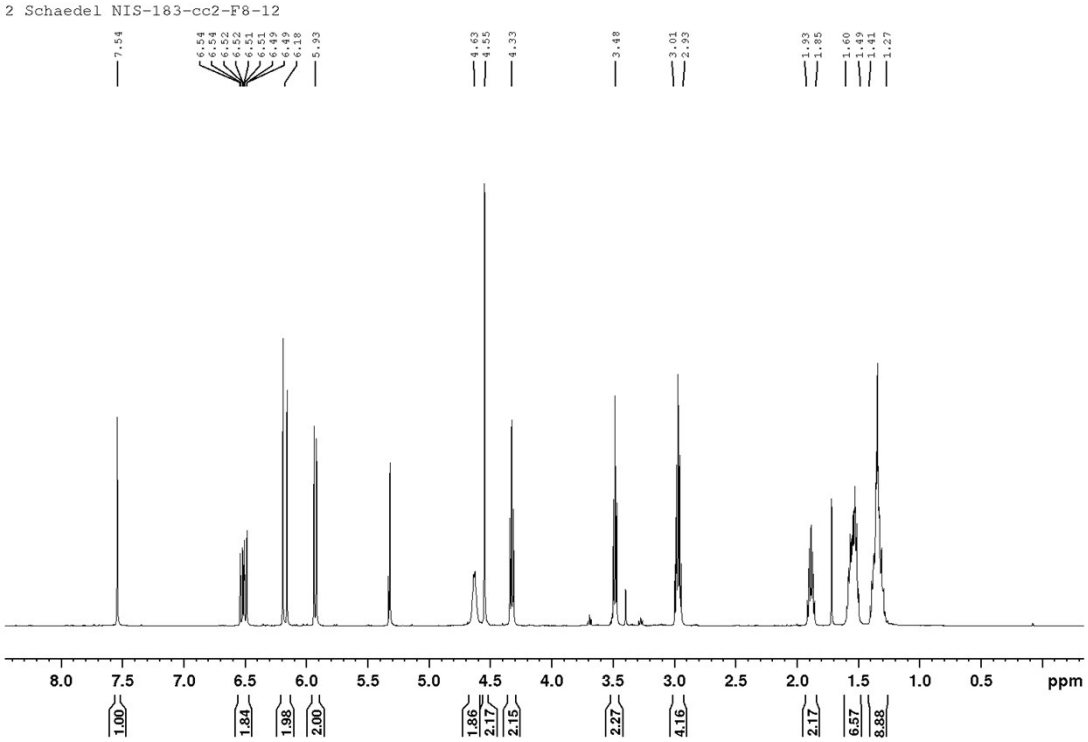


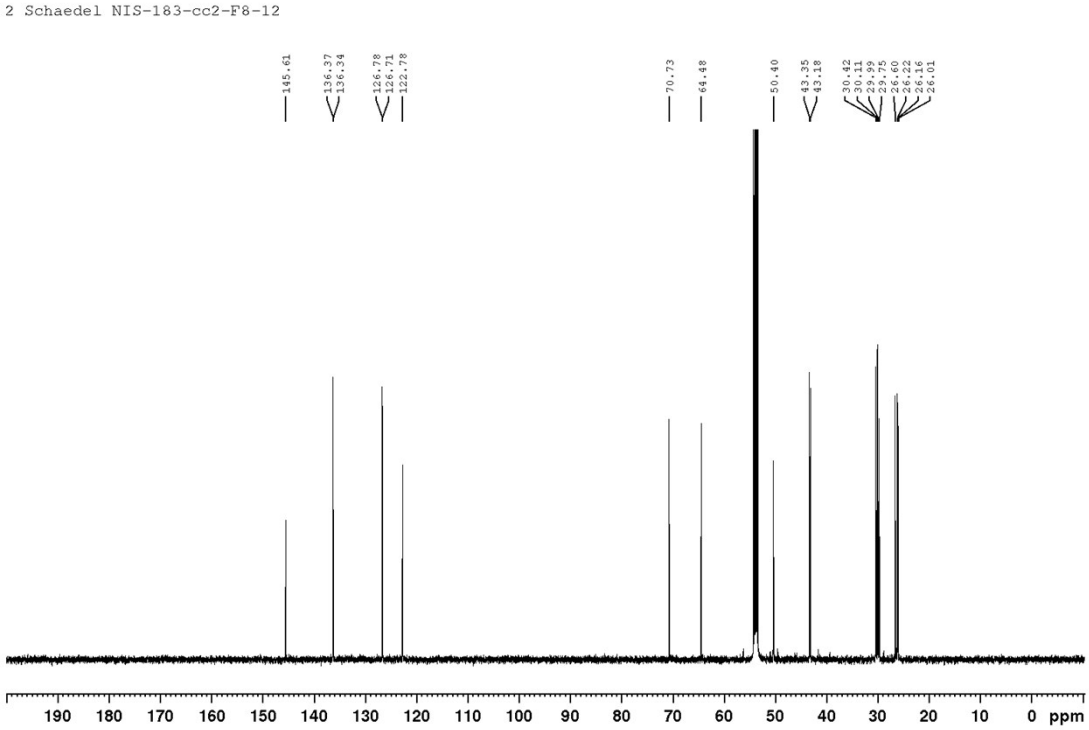


C_6 VSAm

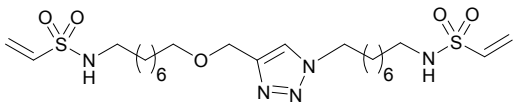


C_6 VSAm

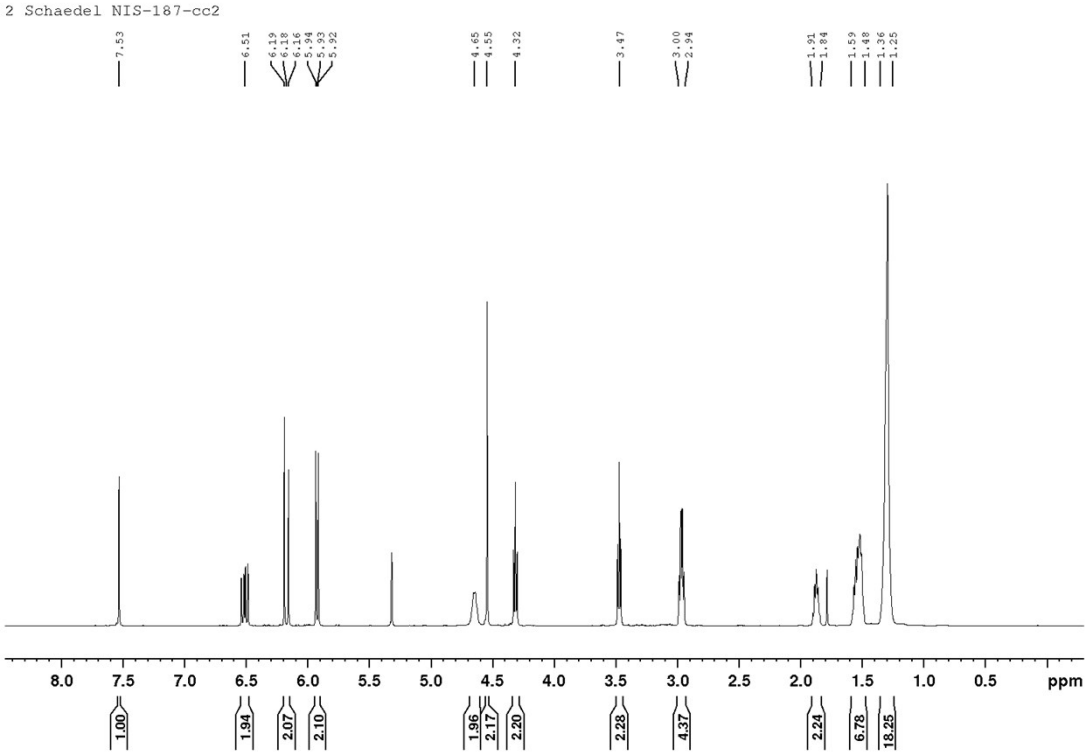


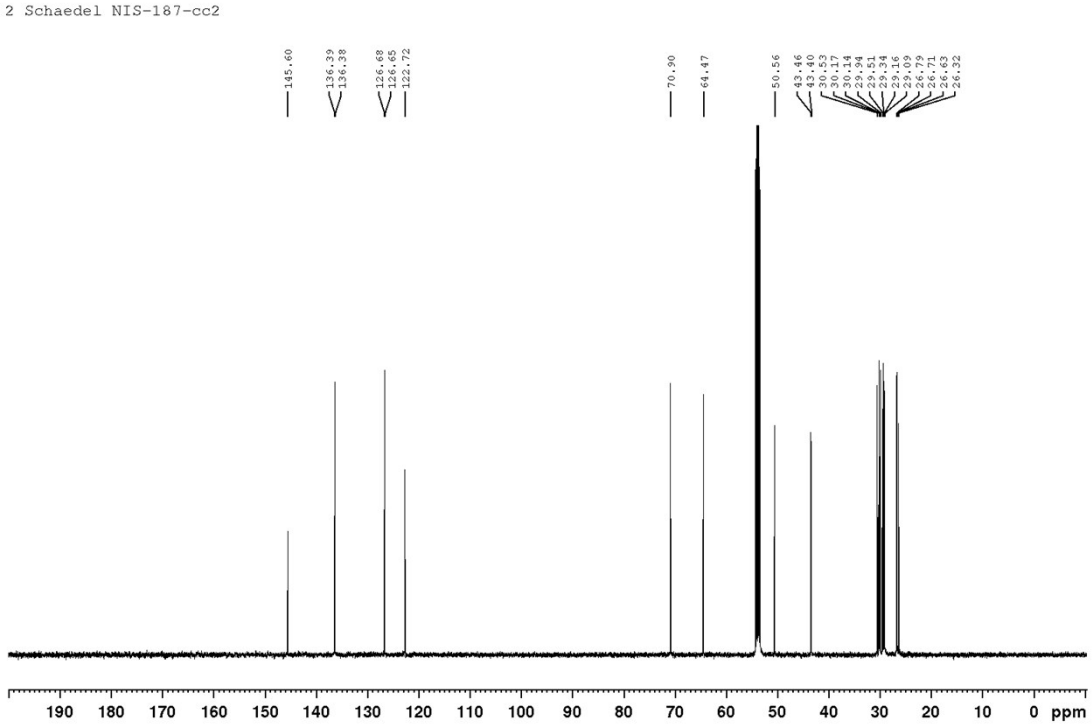


C_8 VSAm

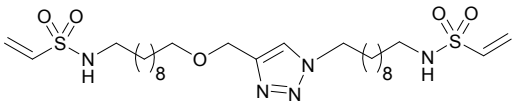


C_8 VSAm

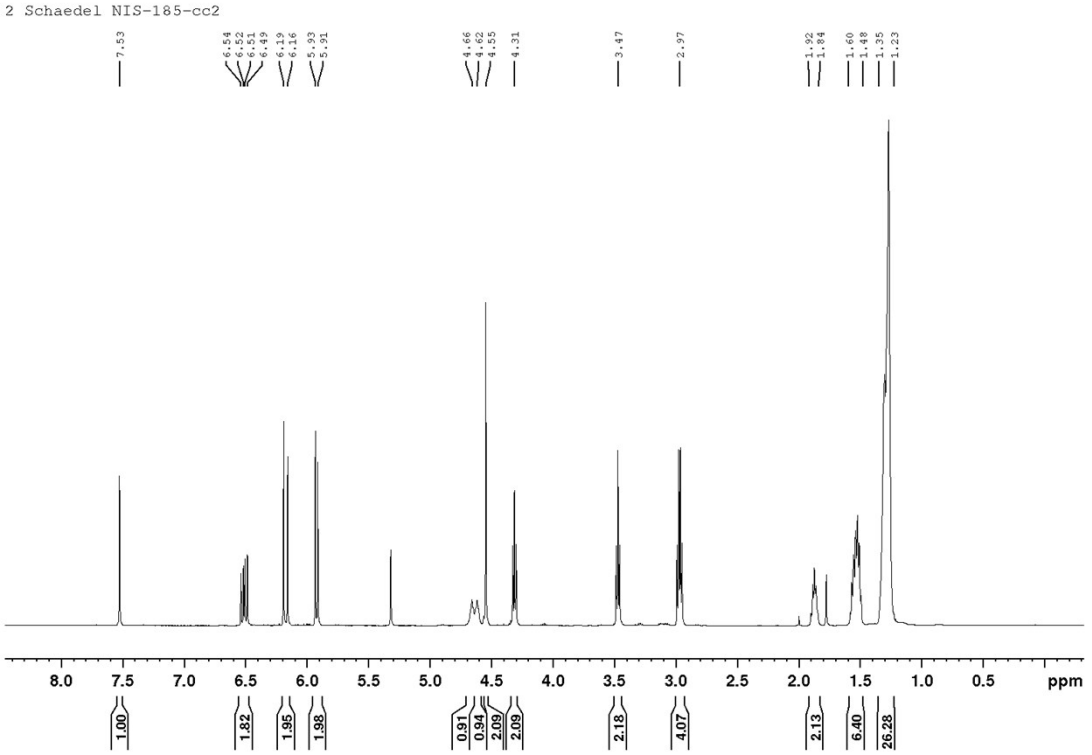


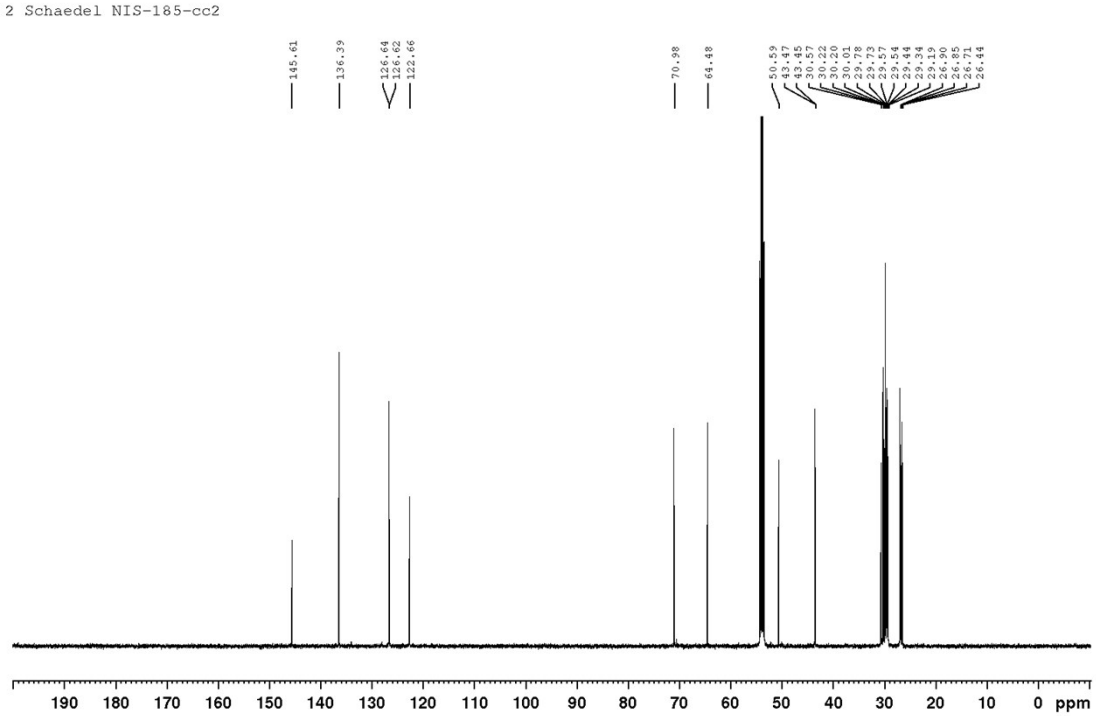


C_{10} VSAm

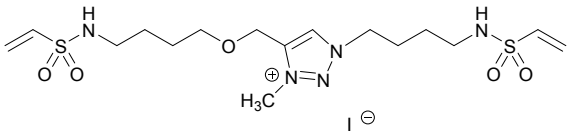


C_{10} VSAm

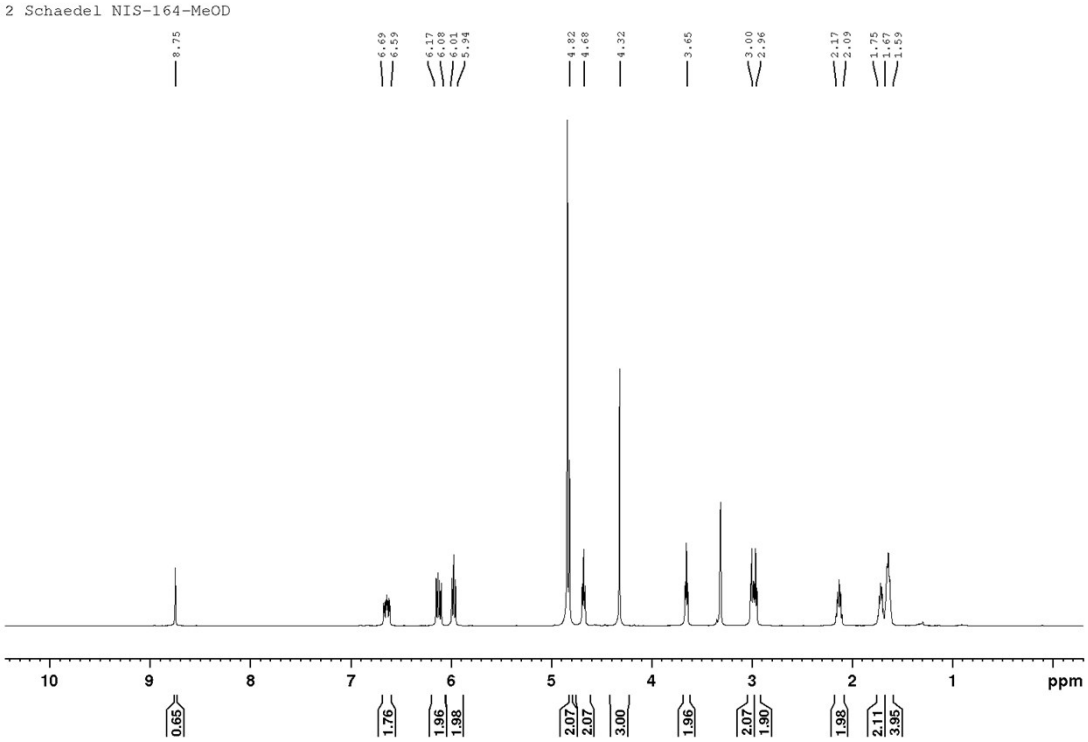


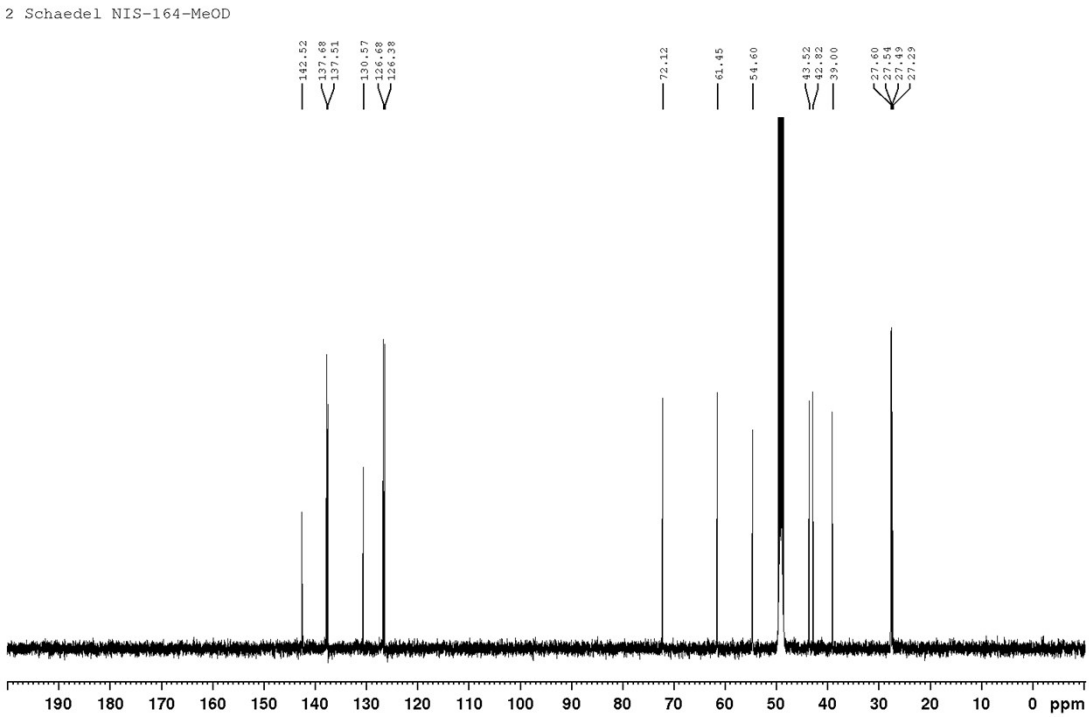


Me-C_4^+ I^- VSAm

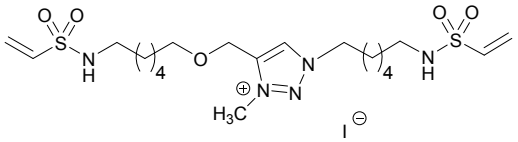


Me-C_4^+ I^- VSAm

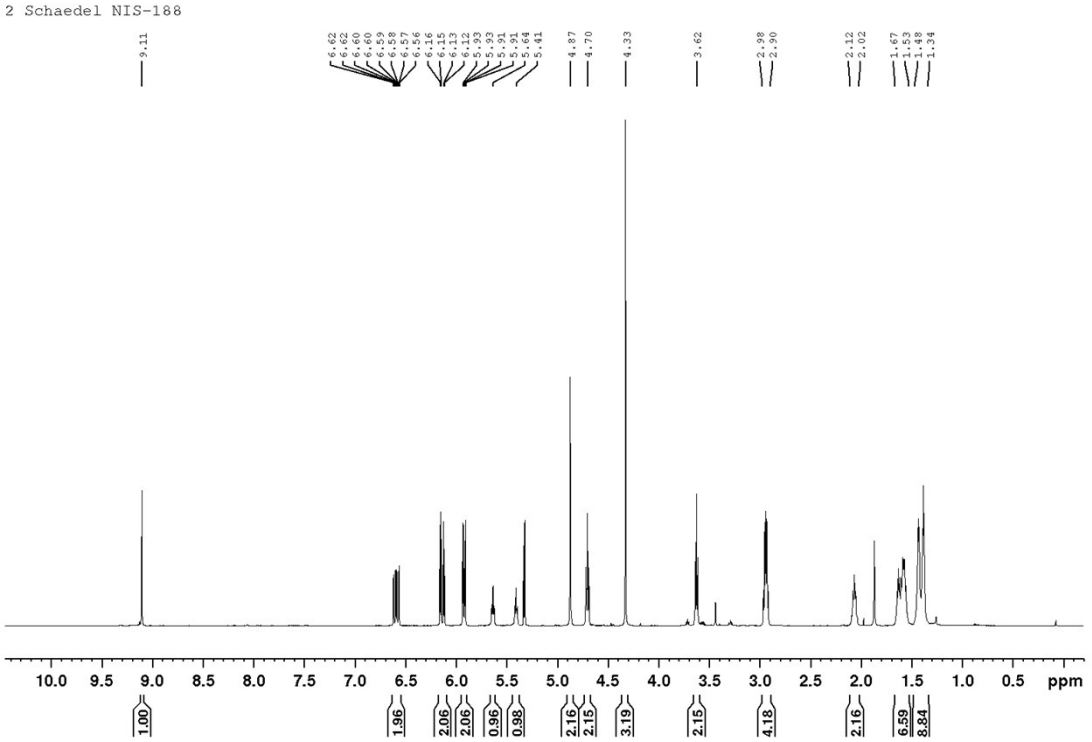


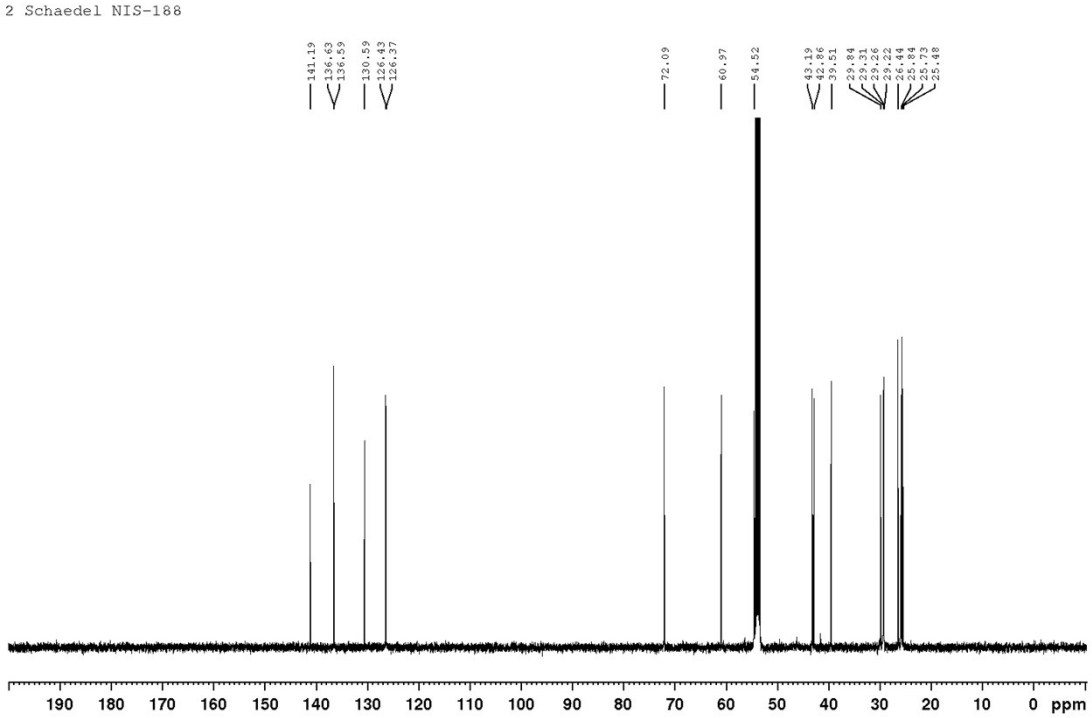


Me-C_6^+ I^- VSAm

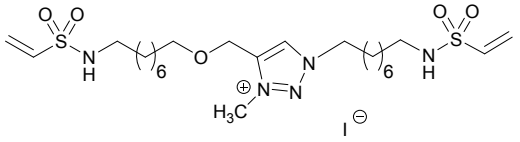


Me-C_6^+ I^- VSAm

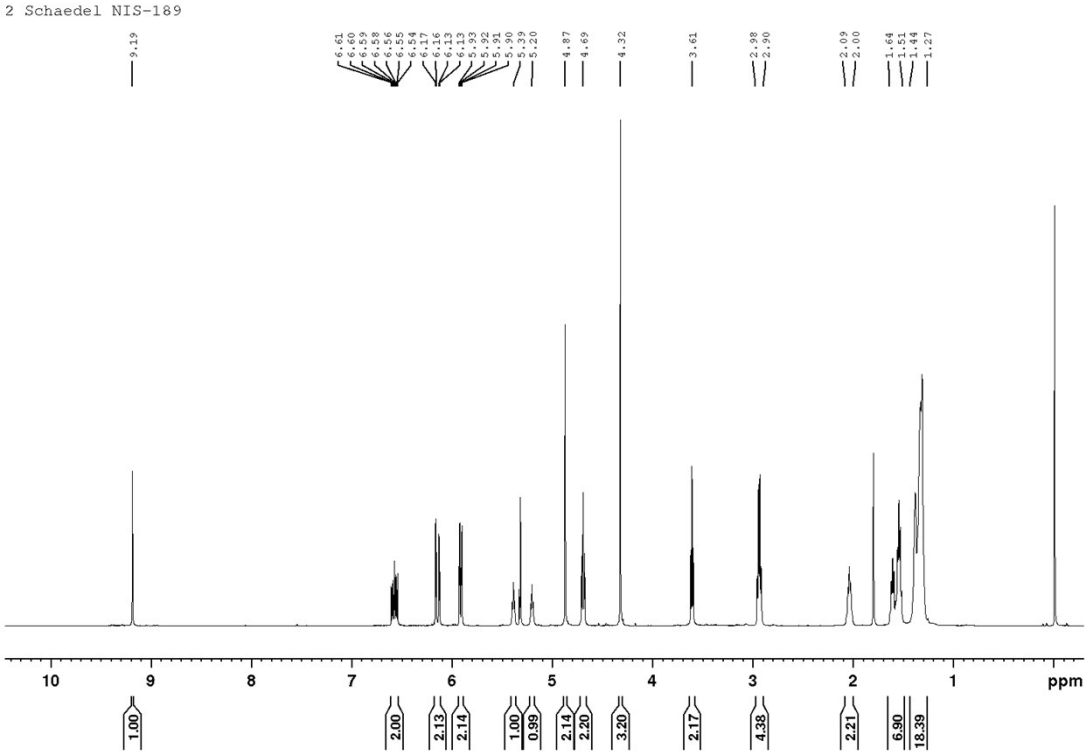


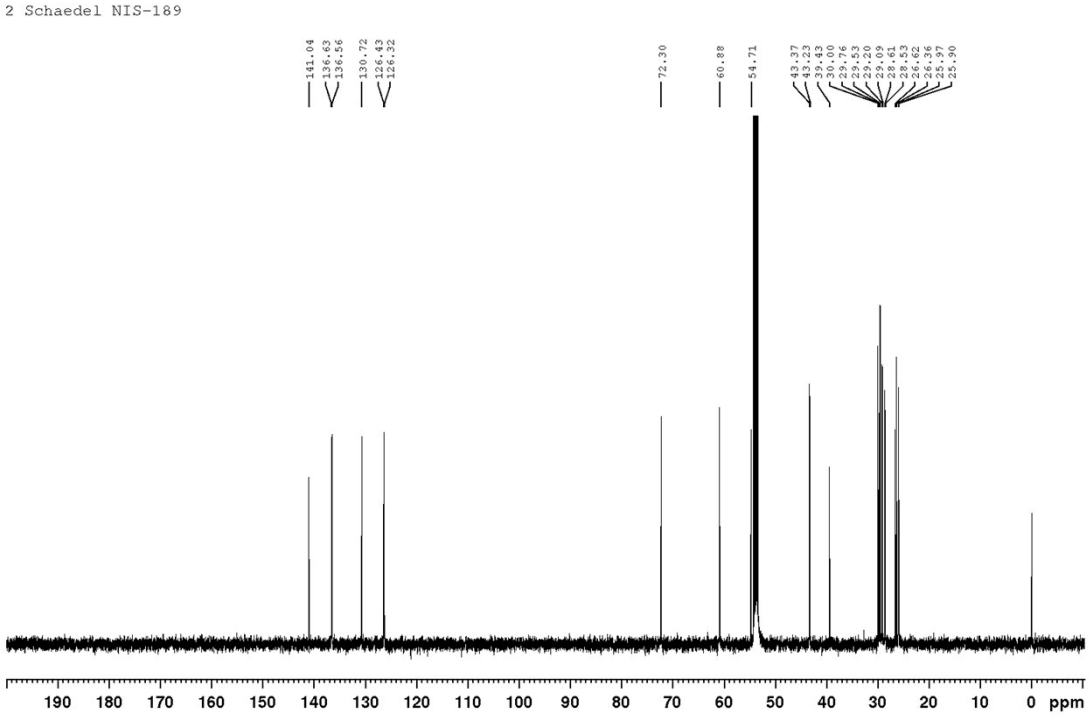


Me-C_8^+ I^- VSAm

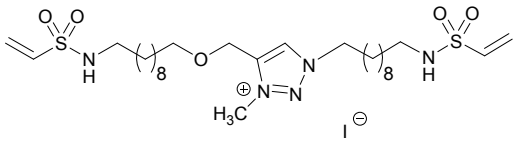


Me-C_8^+ I^- VSAm

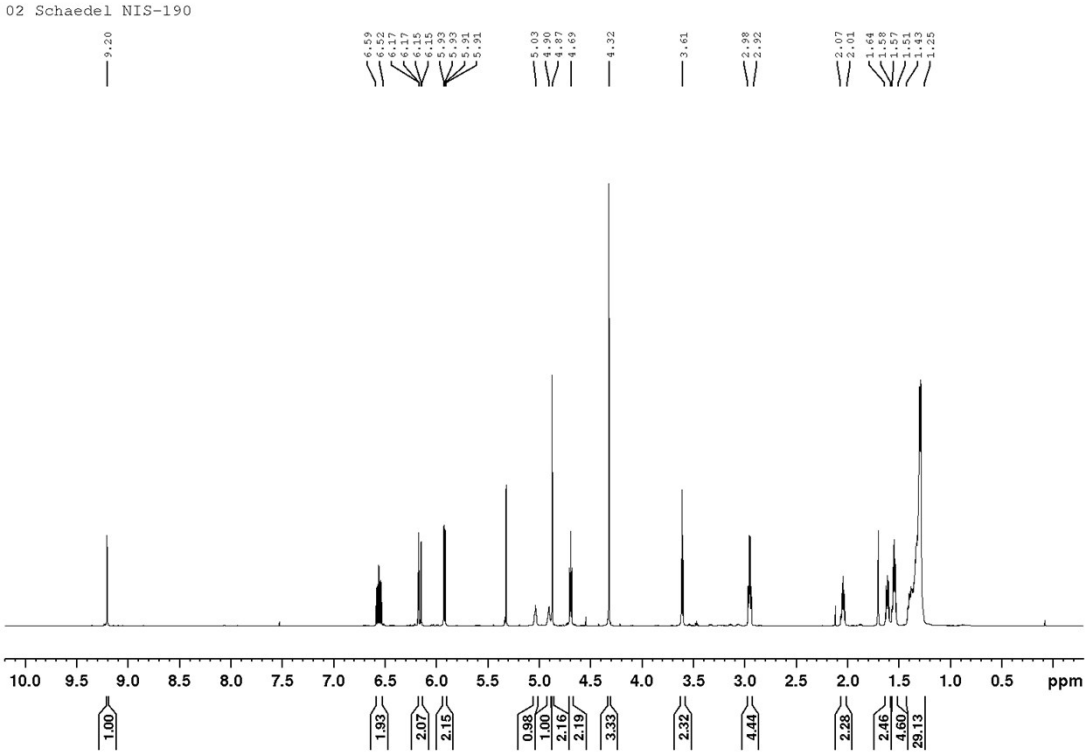


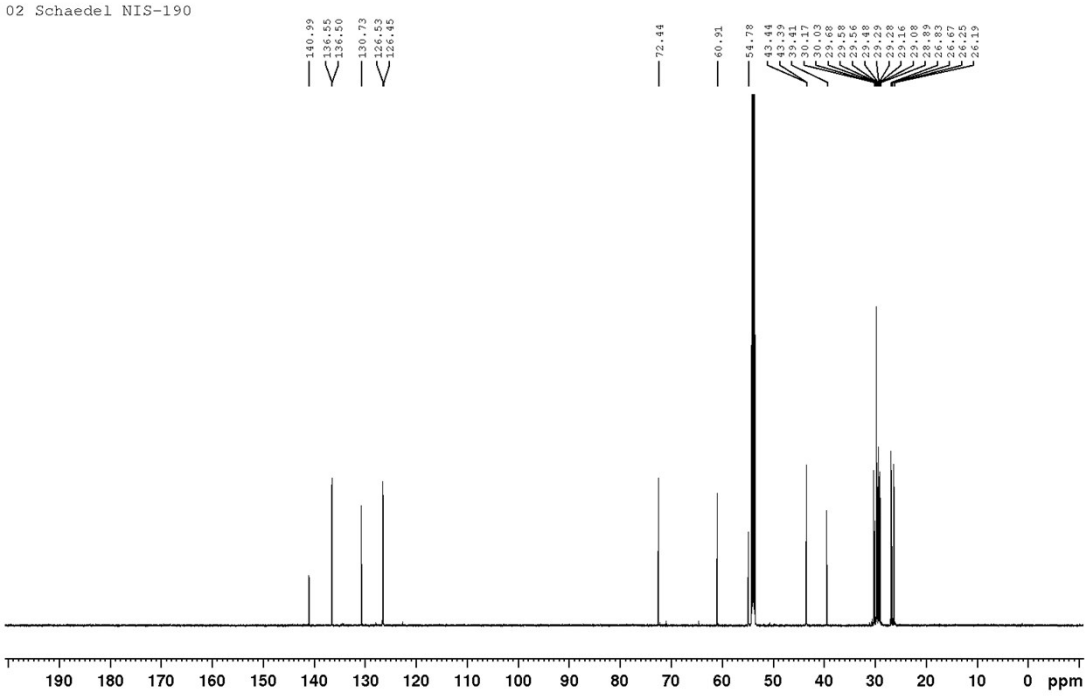


Me-C_{10}^{+} I^{-} VSAm



Me-C_{10}^{+} I^{-} VSAm





3. Hydrogel formulation of charged cross-linker

With **Me-$C_8$$^+I^-$VSAm** hydrogels formulation were prepared, but no gelation occurred. Instead, the solution turned yellow and by adding amylose solution turned violet as an identification of iodine (Fig. 1).

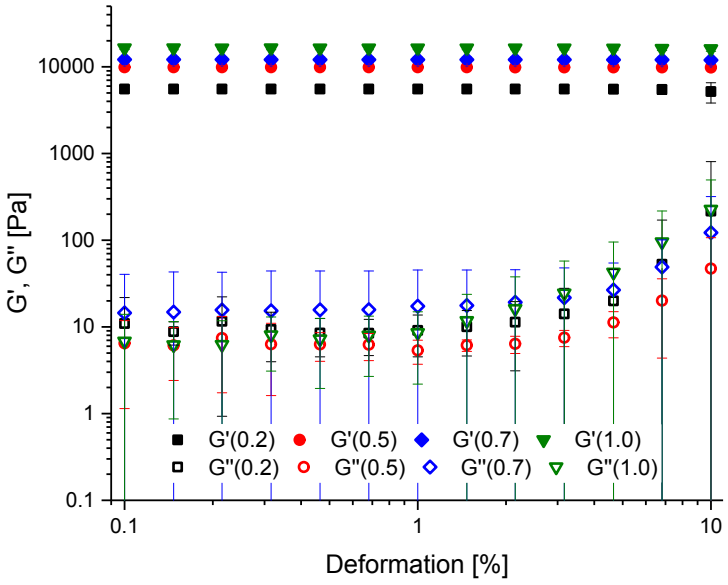
Figure 1

4. Amplitude and frequency sweeps of swollen hydrogels

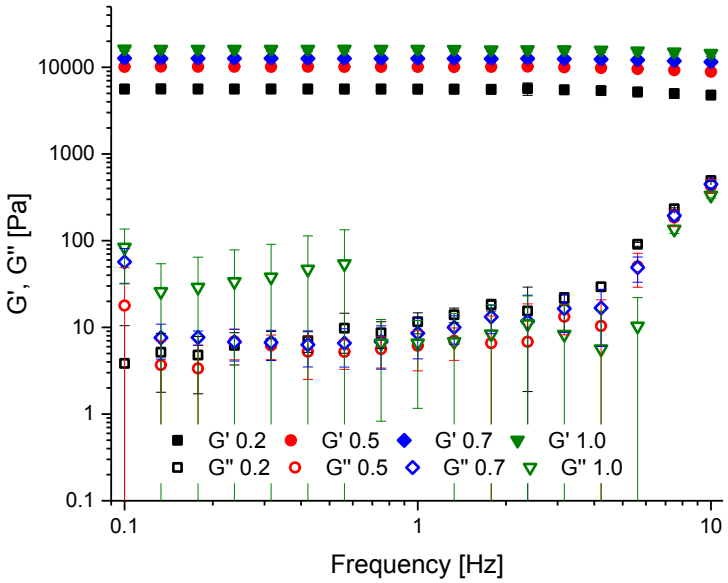
4.1 Acrylamide gels

MBA

Amplitude sweeps

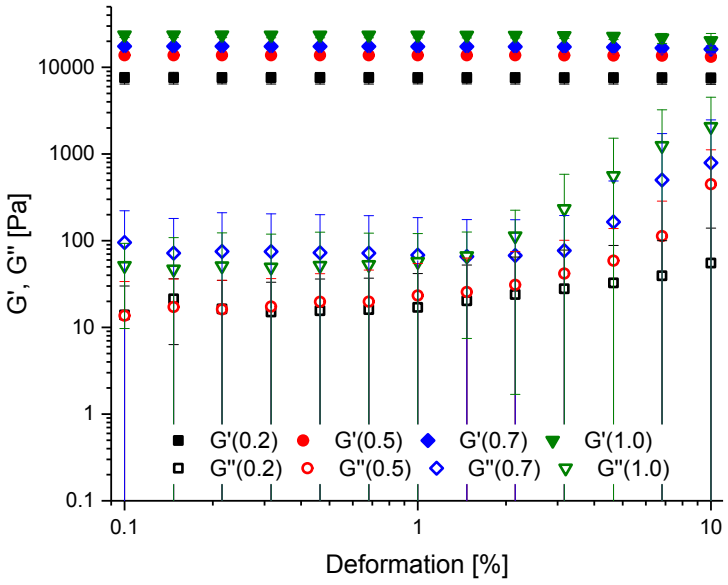


Frequency sweeps

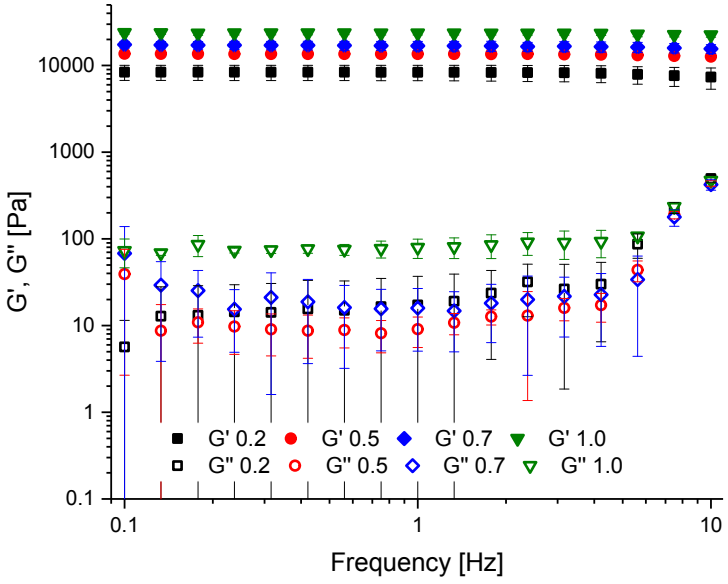

G', G'' [Pa]
10000
1000
100
10
1
0.1
0.1
1
10
Frequency [Hz]
G' 0.2
G' 0.5
G' 0.7
G' 1.0
G'' 0.2
G'' 0.5
G'' 0.7
G'' 1.0

C_4 AAm

Amplitude sweeps

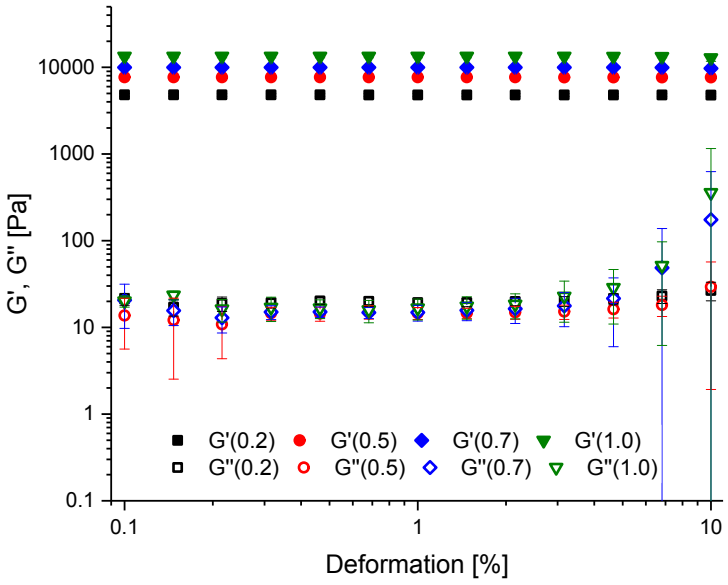


Frequency sweeps

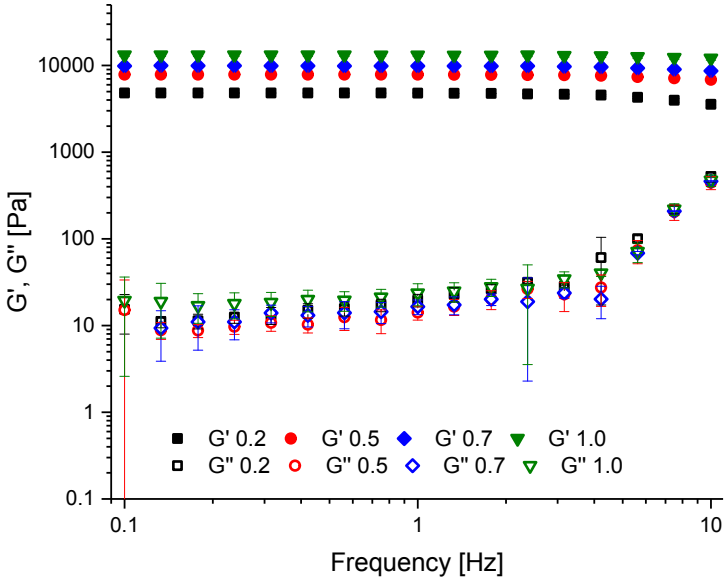


C_4 MeAAm

Amplitude sweeps

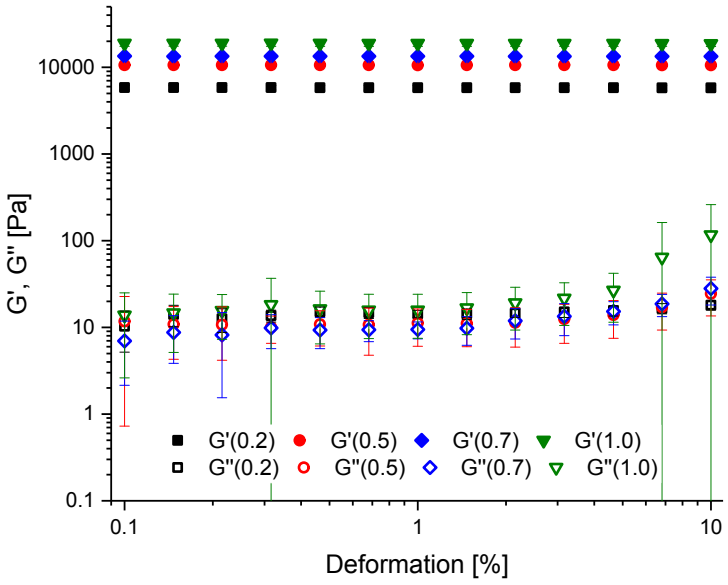


Frequency sweeps

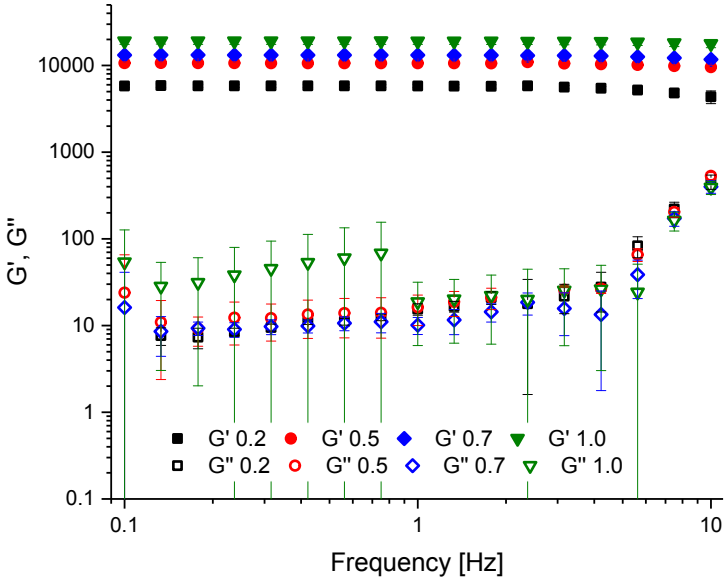


C_4 MaI

Amplitude sweeps

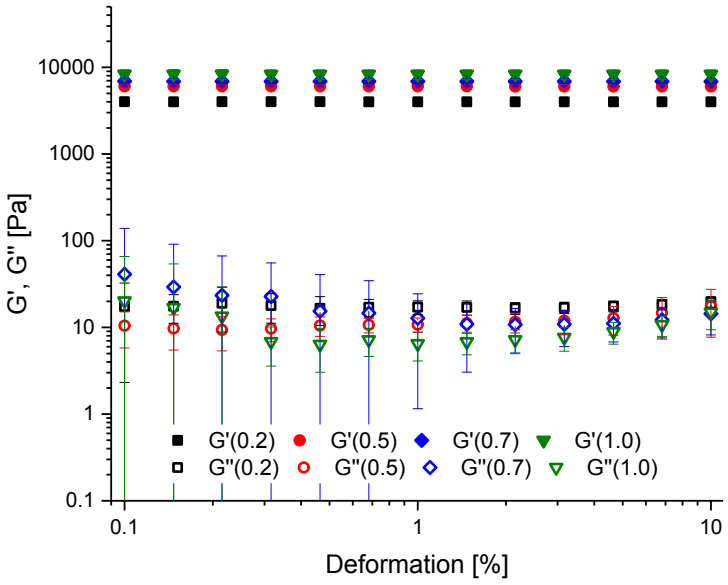


Frequency sweeps

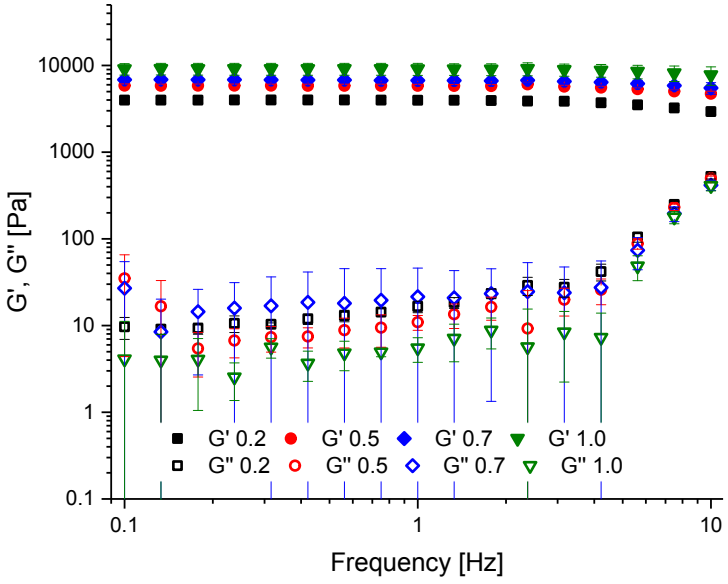


C_4 VSAm

Amplitude sweeps



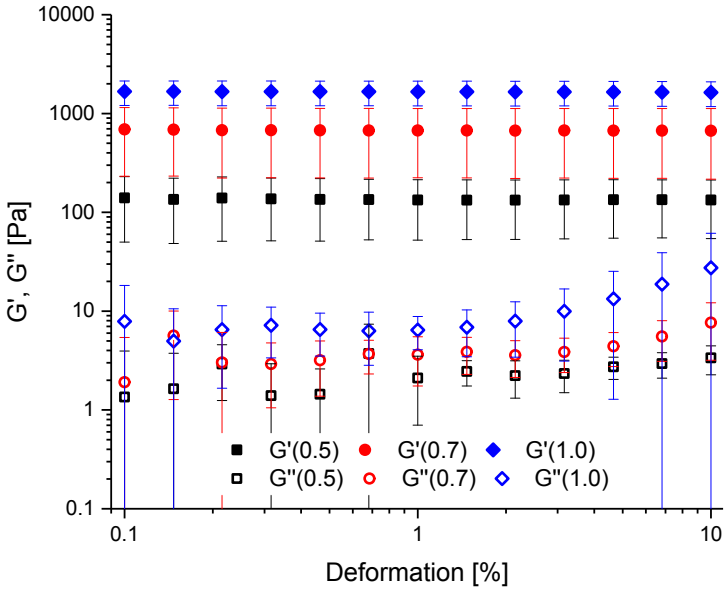
Frequency sweeps



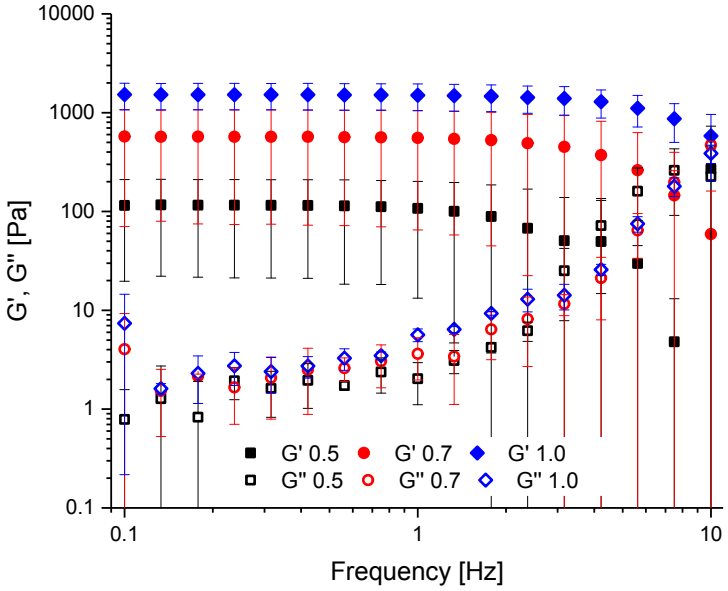
4.2 PDMAAm hydrogels

MBA

Amplitude sweeps

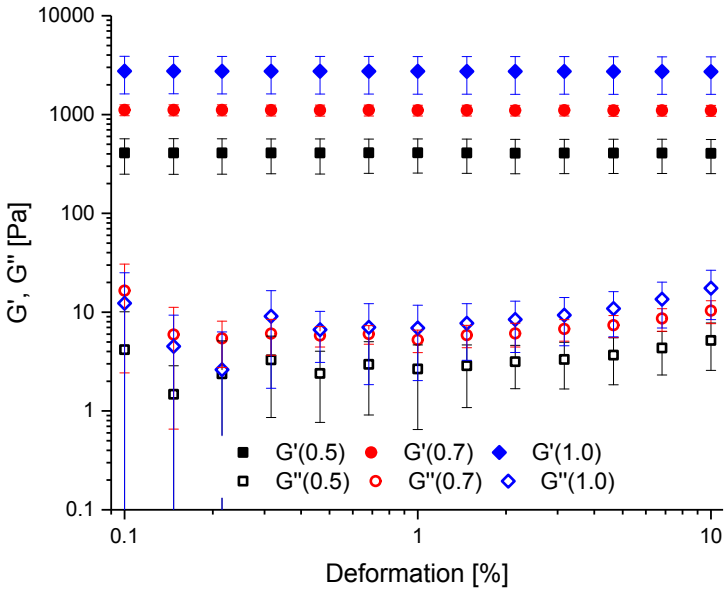


Frequency sweeps

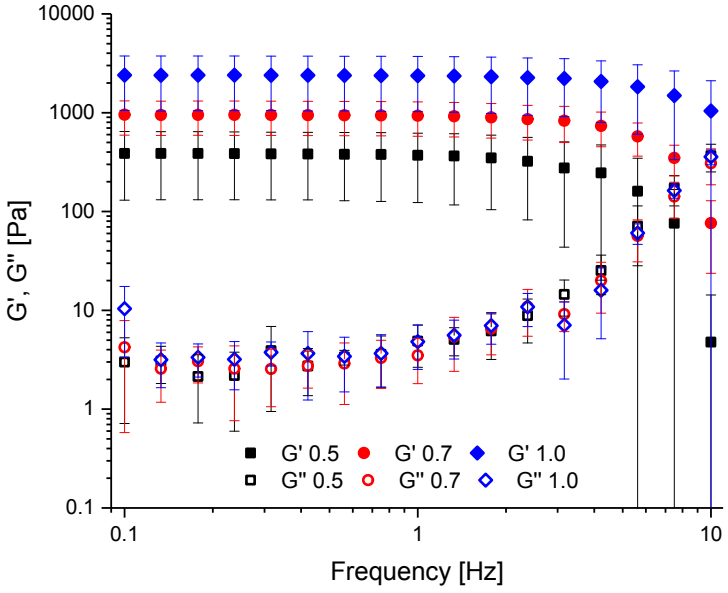


C_4 AAm

Amplitude sweeps

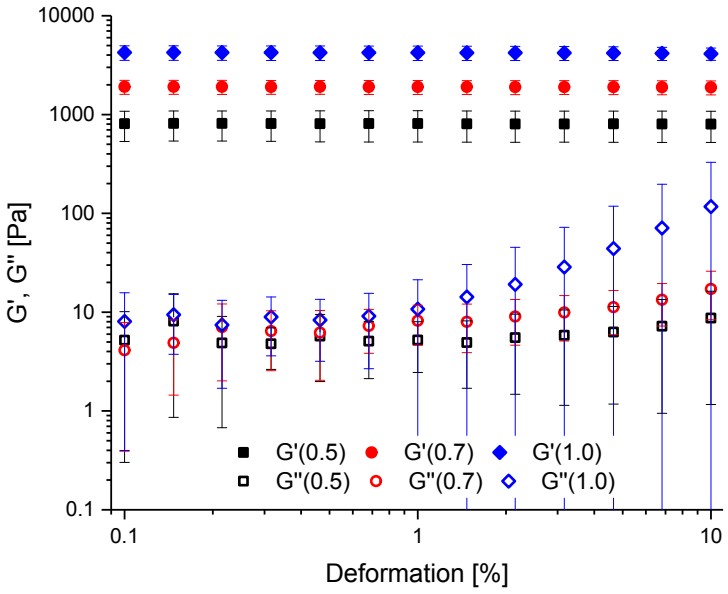


Frequency sweeps

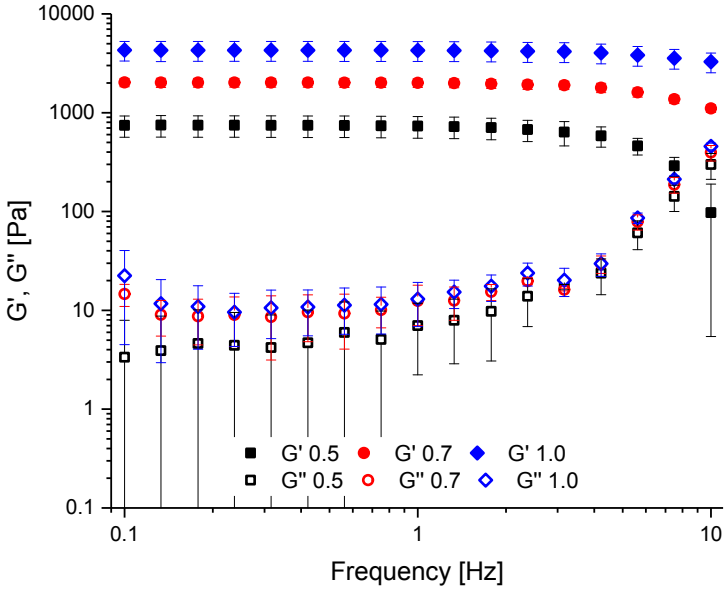


C_6 AAm

Amplitude sweeps

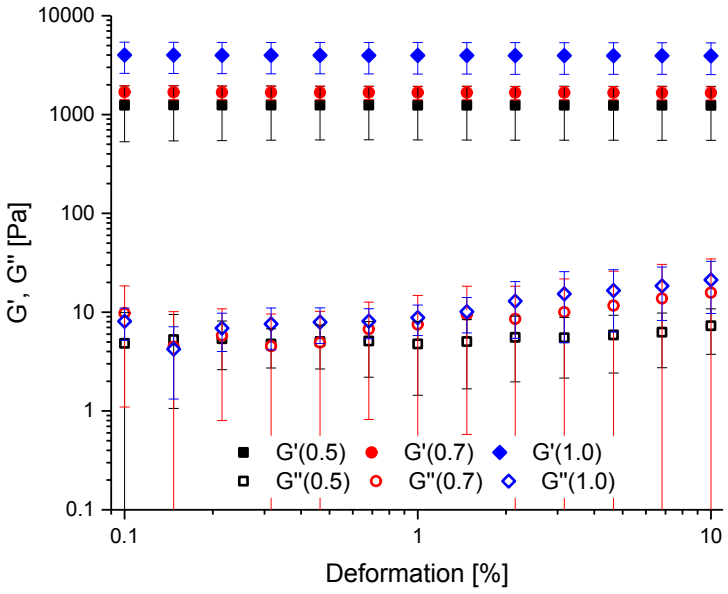


Frequency sweeps

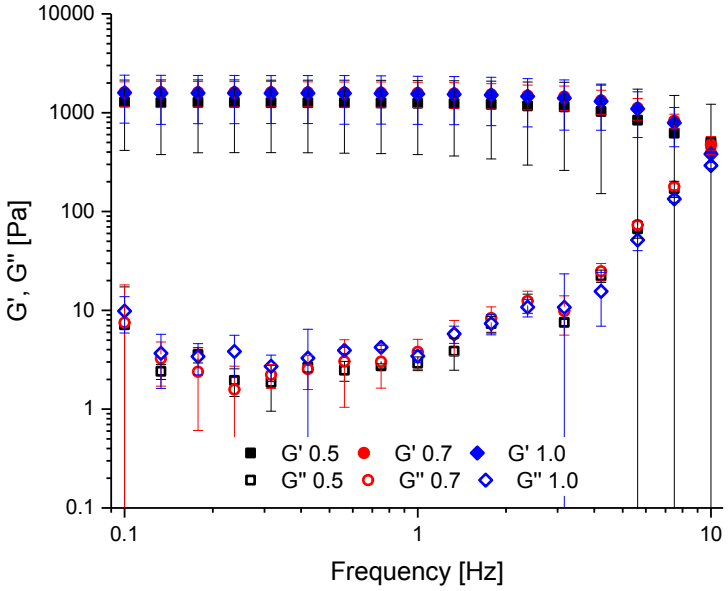


C_8 AAm

Amplitude sweeps

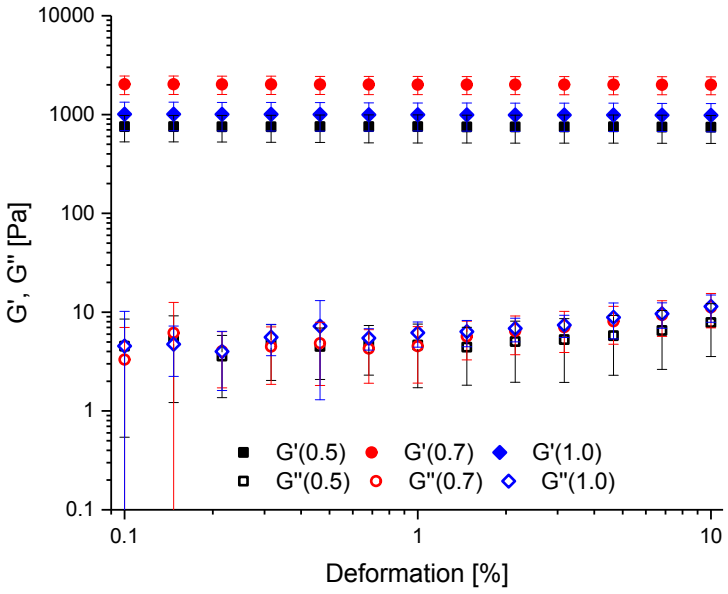


Frequency sweeps

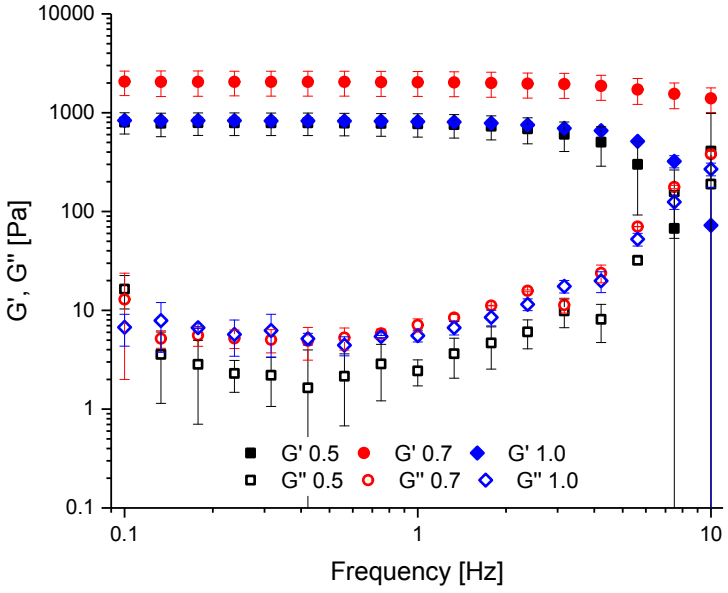


C_{10} AAm

Amplitude sweeps

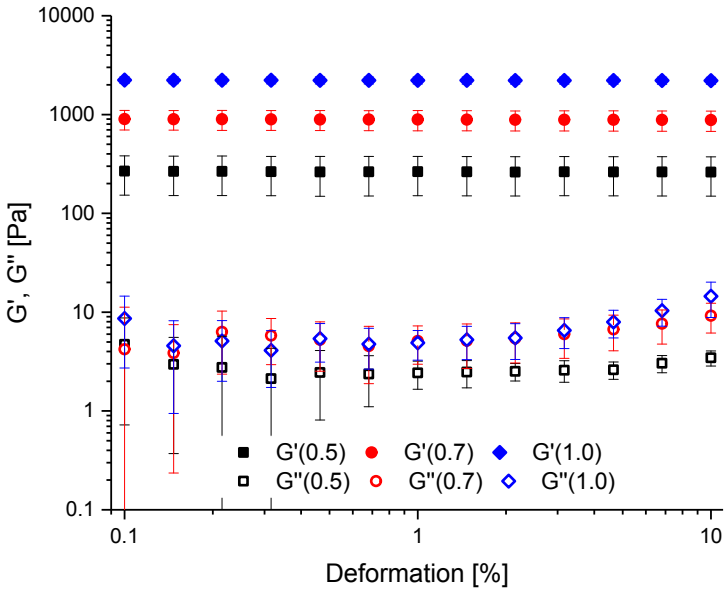


Frequency sweeps

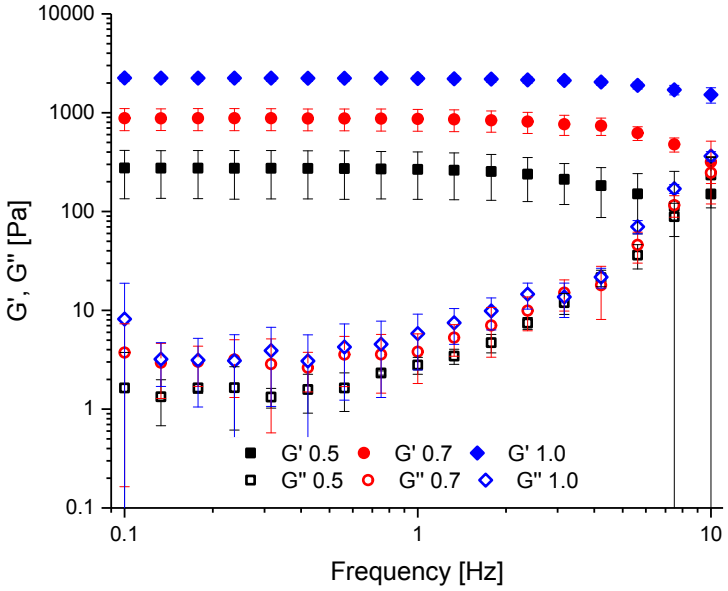


C_4 MeAAm

Amplitude sweeps

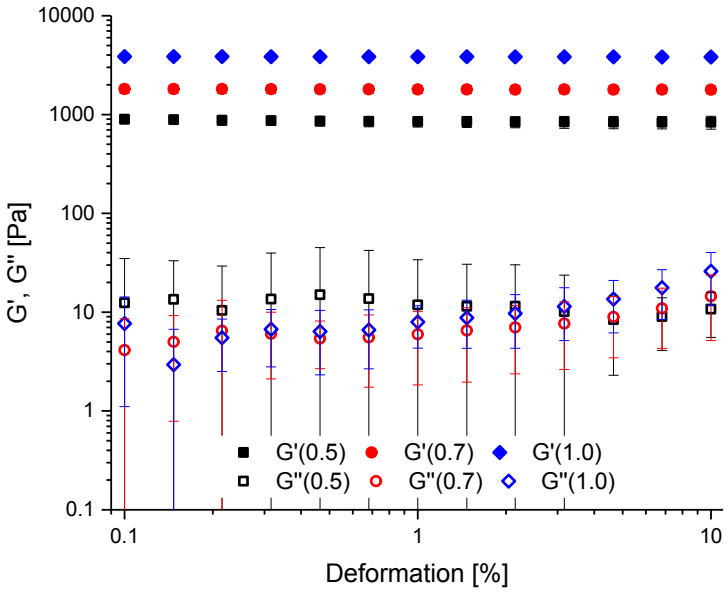


Frequency sweeps

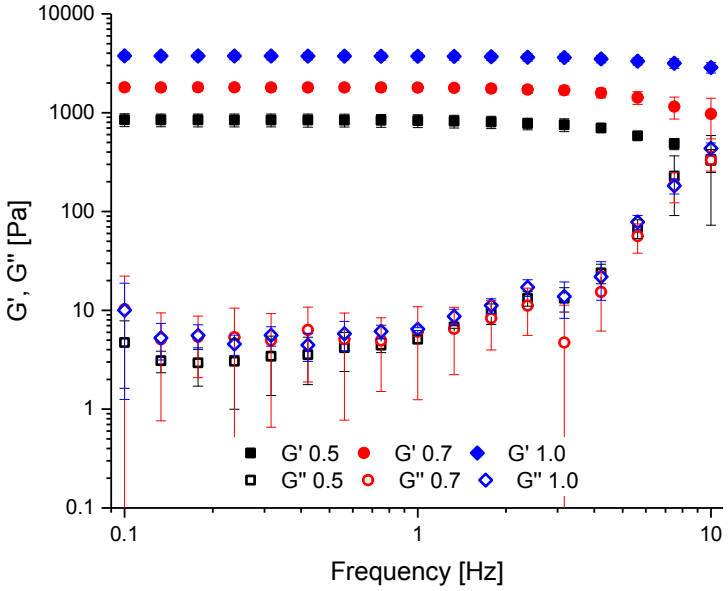


C_6 MeAAm

Amplitude sweeps

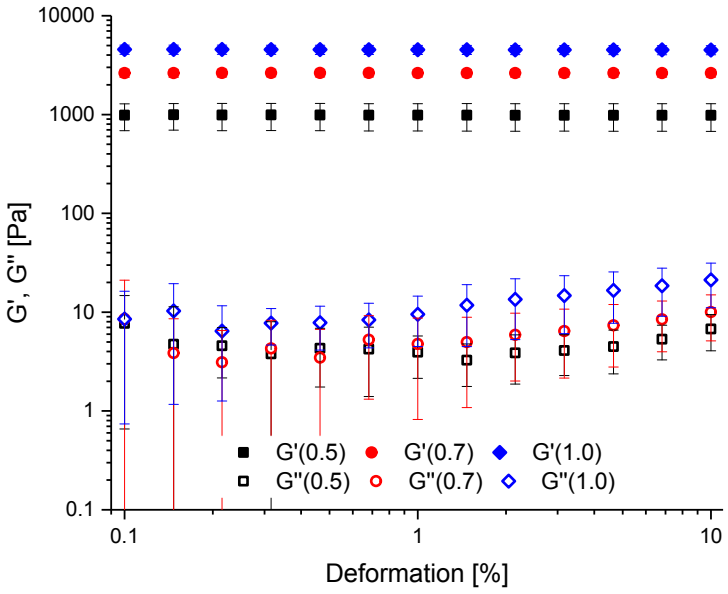


Frequency sweeps

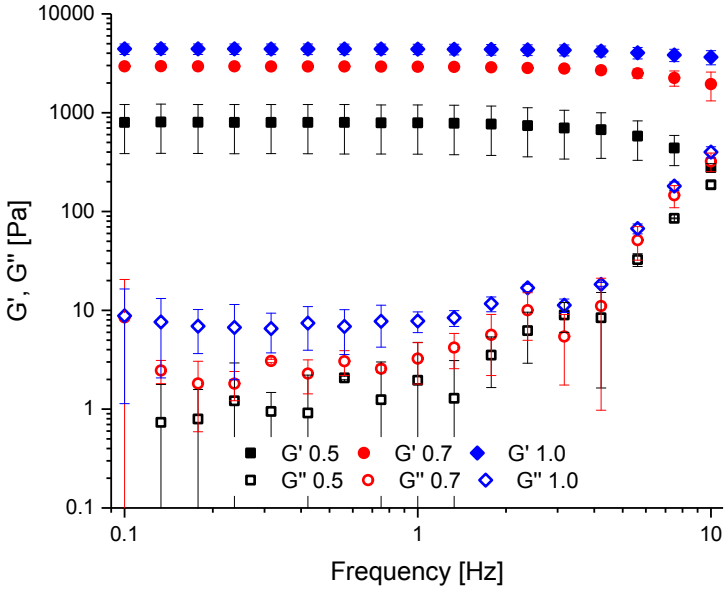


C_8 MeAAm

Amplitude sweeps

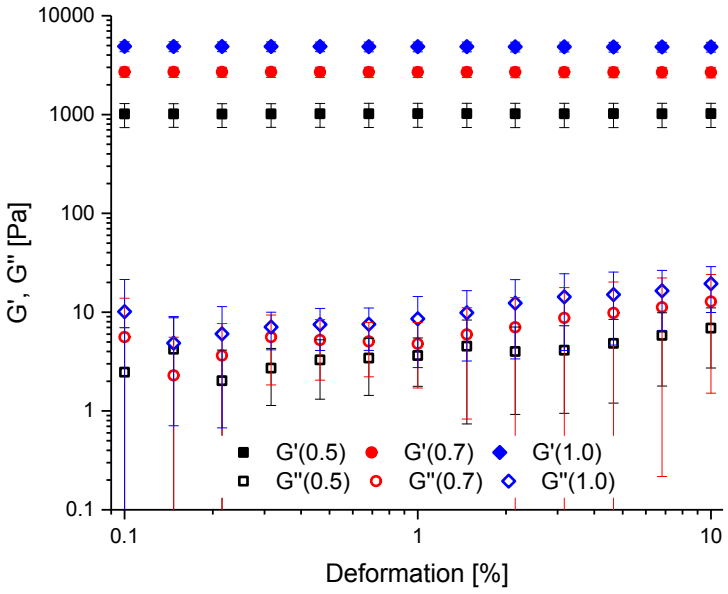


Frequency sweeps

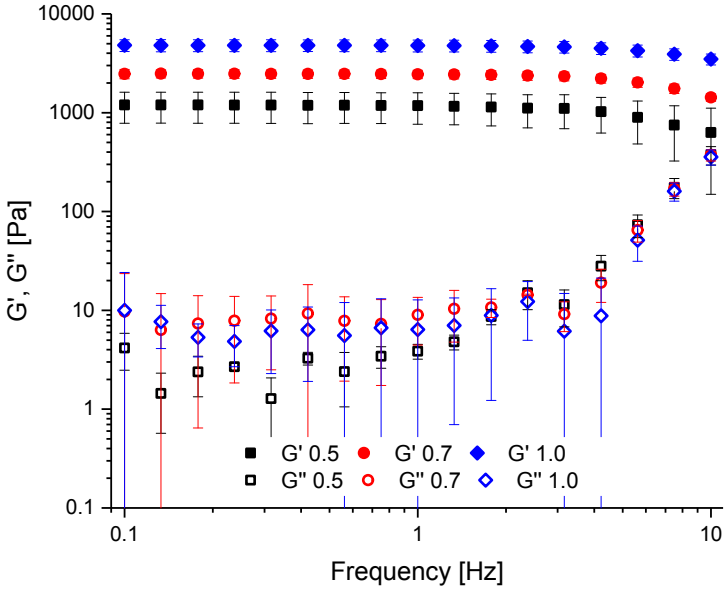


C_{10} MeAAm

Amplitude sweeps



Frequency sweeps



5. Mesh size calculation

$$\xi = \frac{1}{\sqrt[3]{\phi}} \sqrt{\frac{2\,M_c}{M_0}}\, l \sqrt{C}$$

With $l = 0.154\ nm$ as length of the C-C-bond; $C = 6.32$ as characteristic ratio for PAAm and $C = 6.7$ for PDMAAm

PAAm hydrogels

			by swelling		by rheology	
cross-linker	X [%]	ϕ	M_c [g mol^{-1}]	ξ [nm]	M_c [g mol^{-1}]	ξ [nm]
MBA	0.2	0.07536	6 478	12.4	29 776	26.5
	0.5	0.09689	3 405	8.3	18 171	19.1
	0.7	0.10628	2 678	7.1	15 307	17.0
	1.0	0.11866	2 006	5.9	11 885	14.4
C_4 AAm	0.2	0.09024	4 090	9.3	23 191	22.0
	0.5	0.11141	2 367	6.6	13 656	15.8
	0.7	0.12464	1 762	5.5	11 239	13.8
	1.0	0.1392	1 314	4.5	8 656	11.7
C_4 MeAAm	0.2	0.07603	6 335	12.2	34 469	28.5
	0.5	0.09221	3 869	8.9	22 994	21.8
	0.7	0.10167	3 005	7.6	18 288	18.8
	1.0	0.11411	2 223	6.3	14 188	15.9
C_4 MaI	0.2	0.08273	5 110	10.7	29 257	25.5
	0.5	0.10627	2 678	7.1	17 472	18.1
	0.7	0.11266	2 299	6.4	14 081	16.0
	1.0	0.13079	1 551	5.0	10 526	13.1
C_4 VSAm	0.2	0.07334	6 939	12.9	40 883	31.4
	0.5	0.08479	4 799	10.2	28 476	24.9
	0.7	0.09177	3 917	9.0	25 630	23.0
	1.0	0.09619	3 469	8.3	21 590	20.8

PDMAAm hydrogels

			by swelling		by rheology	
cross-linker	X [%]	ϕ	M_c [g mol^{-1}]	ξ [nm]	M_c [g mol^{-1}]	ξ [nm]
MBA	0.5	0.01511	93 951	70.2	657 035	185.6
	0.7	0.02865	32 574	33.4	160 753	74.2
	1.0	0.04066	18 238	22.2	74 355	44.9
C_4 MeAAm	0.5	0.0237	44 587	41.6	384 998	122.3
	0.7	0.03489	23 506	26.6	129 834	62.4
	1.0	0.04801	13 838	18.3	58 014	37.5
C_6 MeAAm	0.5	0.03203	27 083	29.3	132 160	64.8
	0.7	0.04318	16 506	20.7	69 073	42.4
	1.0	0.05935	9 711	14.3	35 882	27.5
C_8 MeAAm	0.5	0.03542	22 926	26.1	117 907	59.2
	0.7	0.05181	12 188	16.8	50 153	34.0
	1.0	0.06214	8 990	13.6	30 918	25.1
C_{10} MeAAm	0.5	0.0375	20 857	24.4	116 038	57.6
	0.7	0.04756	14 056	18.5	47 674	34.1
	1.0	0.06212	8 995	13.6	28 811	24.3
C_4 AAm	0.5	0.02358	44 963	41.9	247 108	98.2
	0.7	0.03249	26 451	28.9	102 243	56.7
	1.0	0.04468	15 595	19.9	45 867	34.2
C_6 AAm	0.5	0.031	28 589	30.5	137 292	66.8
	0.7	0.04272	16 802	21.0	64 972	41.3
	1.0	0.06161	9 120	13.7	33 084	26.1
C_8 AAm	0.5	0.02825	33 341	33.9	86 738	54.8
	0.7	0.04183	17 400	21.5	73 575	44.2
	1.0	0.05576	10 780	15.4	34 105	27.4
C_{10} AAm	0.5	0.03334	25 344	28.0	151 179	68.4
	0.7	0.04731	14 180	18.6	63 725	39.5
	1.0	0.03432	24 156	27.1	110 100	57.8

6. Biological studies of cross-linkers

Cross-linker stock solutions were prepared in DMSO and diluted for the shown studies.

Cytotoxicity of cross-linkers: mouse fibroblast cells L929 were incubated for 3 days with the test compounds and cell viability was evaluated using the alamarBlue® or the WST assay.[5,6]

Antimicrobial activity: growth of the Gram negative bacteria *Escherichia coli* K12 and its deletion mutant *Escherichia coli Δ*TolC in liquid culture was monitored via the turbidity of the bacterial suspension (optical density at 600 nm) after 24 h incubation of the bacteria with the cross-linkers.

First all compounds were tested at the single concentration of 100 µM to identify compounds with biological activity (resulting residual cell viability / microbial growth < 50 %). The influence of the concentration on cytoxicity and / or antimicrobial activity was subsequently investigated using only the active compounds allowing the determination of the IC_{50} (concentration leading to 50% residual growth or viability) by fitting the dose-response curve by non-linear regression.

Results of the biological studies of the cross-linkers:

Compound ID	IC_{50} Growth inhibition E. coli TolC [µM]	IC_{50} Growth inhibition S. aureus [µM]	Cell viability IC50 [µM]
C4 Mal	n.a.	n.a.	2.2
C6 Mal	n.a.	n.a.	1.8
C8 Mal	n.a.	n.a.	6.0 +/- 0.6
C10 Mal	n.a.	n.a.	> 50
Me-C4$^+$I$^-$ Mal	n.a.	n.a.	29,5 +/- 0,7
Me-C6$^+$I$^-$ Mal	n.a.	n.a.	> 50
Me-C8$^+$I$^-$ Mal	n.a.	n.a.	34.6 +/- 0.6
Me-C10$^+$I$^-$ Mal	9.1 +/- 1,1	36.1 +/- 0.6	18.25 +/- 0.96
C4 AAm	n.a.	n.a.	n.a.
C6 Aam	n.a.	n.a.	n.a.
C8 Aam	n.a.	n.a.	n.a.
C10 Aam	n.a.	n.a.	n.a.
Me-C4$^+$I$^-$ Aam	n.a.	n.a.	n.a.
Me-C6$^+$I$^-$ Aam	n.a.	n.a.	n.a.
Me-C8$^+$I$^-$ Aam	n.a.	n.a.	n.a.
Me-C10$^+$I$^-$ Aam	13.7 +/- 1.8	52 +/- 5	15.1+/-0.3
C4 MeAAm	n.a.	n.a.	n.a.
C6 MeAAm	n.a.	n.a.	n.a.
C8 MeAAm	n.a.	n.a.	n.a.
C10 MeAAm	n.a.	n.a.	n.a.
Me-C4$^+$I$^-$ MeAm	n.a.	n.a.	n.a.
Me-C6 $^+$I$^-$ MeAm	n.a.	n.a.	n.a.
Me-C8$^+$I$^-$ MeAm	n.a.	n.a.	35.4 +/- 0.3
Me-C10$^+$I$^-$ MeAm	7.2 +/- 0.5	21.5 +/-0.6	20.4 +/- 0.5
C4 VSAm	n.a.	n.a.	n.a.
C6 VSAm	n.a.	n.a.	16.6 +/- 0.7
C8 VSAm	n.a.	n.a.	11.0 +/- 2.6
C10 VSAm	n.a.	n.a.	n.a.
Me-C4$^+$I$^-$ VSAm	n.a.	n.a.	n.a.
Me-C6$^+$I$^-$ VSAm	n.a.	n.a.	n.a.
Me-C8$^+$I$^-$ VSAm	n.a.	n.a.	n.a.
Me-C10$^+$I$^-$ VSAm	4.7 +/- 0,5	8.3 +/- 0.7	16.4 +/- 2.6

n.a.: not active

7. References

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[4] P. S. Hegger, J. Kupka, B. B. Minsky, N. Schädel, N. Petri, S. Laschat, H. Boehm, C*hemistrySelect* **2017**, *2*, 7701–7705.

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[6] P. Ngamwongsatit, P.P. Banada, W. Panbangred, A.K. Bhunia *J Microbiol Methods* **2008**, *73*, 211-215.