Appendix to Chapter 2, entitled:

Electronic Effects in Covalent Adaptable Furan-Maleimide Networks

Accompanying Chapter 2 of the PhD thesis entitled 'The Design and Tuning of Boronate-Tetraazaadamantane Covalent Adaptable Networks' written by Simon van Hurne.

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1. Contents

2. Materials

Commercially available chemicals were not further purified before use.

1,8-octanediamine, 4,4′-oxydianiline, 4,4′-diaminodiphenyl sulfide, epichlorohydrin, furfuryl alcohol, poly(propylene glycol) bis(2-aminopropyl ether) M_n≈2000, tetrabutylammonium bromide and triethylamine were obtained from TCI. 4,4′-Diaminobenzophenone, DMAP, hydrochloric acid and Jeffamine ED-600 were obtained from Merck. 4-Aminophenyl sulfone was supplied by Acros Organics. 1,1′-(Methylenedi-4,1-phenylene)bismaleimide was supplied by Alfa Aesar. Acetic acid, 2-furoyl chloride, furfuryl mercaptan and maleic anhydride were provided by Fischer Scientific. Pentaerythritol triacrylate was bought from abcr GmbH. Sodium carbonate was bought from VWR International. Ethyl acetate, tetrahydrofuran and methanol were bought from Biosolve. Sodium hydroxide tablets (Baker analysed) was purchased from Boom B.V. Boomlab.

All chemicals were used without purification.

3. Equipment

3.1. NMR

NMR characterisation was performed on a Bruker AV400 400 MHz (9.4 T) NMR.

3.2. Rheology

The materials were studied using a Anton Paar MCR 702e space rheometer with a piezo oven combined with a 10 mm plate-plate or extensional DMA linear drive setup.

3.3. IR

IR characterisation was done on a Bruker Tensor 27. The spectra were recorded with a resolution of 4 cm^{-1}, a sample scan time of 16 scans and a background scan time of 16 scans. The data was recorded for the range of 4000–400 cm^{-1}. All samples, except the deposited materials, were measured using ATR. The measurements involving the deposition of crosslinked networks were performed on KBr pellets in combination with FTIR.

3.4. Mass spectrometry

Mass spectrometry data were acquired using MS-ESI Thermo scientific Exactive in positive mode. The compounds were ionized without fragmentation at 0 kV spray voltage and with a capillary temperature of 150 °C.

3.5. TGA

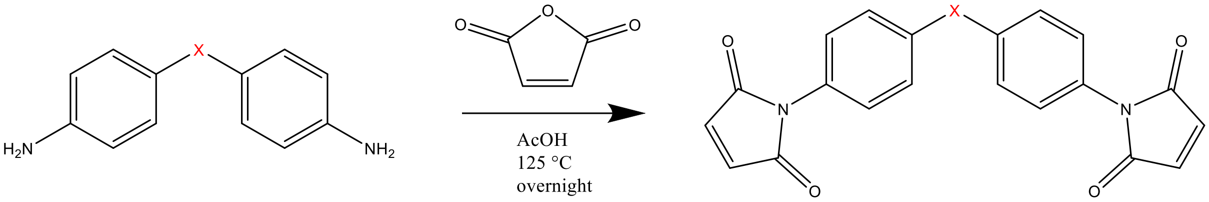
TGA experiments were performed on a Perkin Elmer STA 6000 under a nitrogen atmosphere. The initial temperature of 30 °C was held for 1 minute, whereafter the temperature was gradually increased to 900 °C with a heating rate of 10.00 °C/min. The final temperature was held for 10 min.

3.6. DSC

DSC was performed on a PerkinElmer DSC 8000 over the temperature range of –50 to 200 °C with a temperature increase of 5 °C/min.

4. Methods

4.1. General synthesis of bismaleimide linkers (**1-5**)



Supplementary Scheme S1 General synthesis of bismaleimide linkers.

Maleic acid (1.5 g, 15.1 mmol) and 3.8 mmol bismaleimide precursor (see Supplementary Table S1) were dissolved in 25 ml glacial acetic acid. This solution was then refluxed for 12 hours at 125°C while stirring. The solution was subsequently cooled to room temperature and neutralized by addition of a saturated sodium carbonate solution (≈150-200 ml). The solution was then extracted with 5 × 50 ml ethyl acetate. The organic fractions were collected and washed with 5 × 50 ml of 1 M HCl. Afterwards the solvent was removed by rotary evaporation and the solids were dissolved in 60 ml acetone followed by a precipitation by addition of 100 ml ice cold water. The flask was then put in an ice bath for 30 min. Afterwards the solution was filtered and the precipitate was washed with ice cold water (≈200 ml). The solids were collected and dried overnight.

bMCO: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 (d, J = 8.7 Hz, 4H), 7.57 (d, J = 8.7 Hz, 4H), 6.92 (s, 4H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 194.44, 168.96, 136.17, 135.13, 134.46, 130.89, 125.27, 77.33, 77.01, 76.70.

bMO: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 (d, J = 8.9 Hz, 4H), 7.13 (d, J = 8.9 Hz, 4H), 6.87 (s, 4H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.54, 156.30, 134.24, 127.75, 126.56, 119.54, 77.33, 77.02, 76.70.

bMS: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 (d, J = 8.6 Hz, 4H), 7.33 (d, J = 8.7 Hz, 4H), 6.87 (s, 4H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.25, 135.18, 134.31, 131.69, 130.37, 126.61, 77.33, 77.01, 76.69.

$bMSO_2$: ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (d, J = 8.8 Hz, 4H), 7.63 (d, J = 8.8 Hz, 4H), 6.90 (s, 4H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.57, 139.88, 135.97, 134.51, 128.76, 125.78, 77.32, 77.01, 76.69.

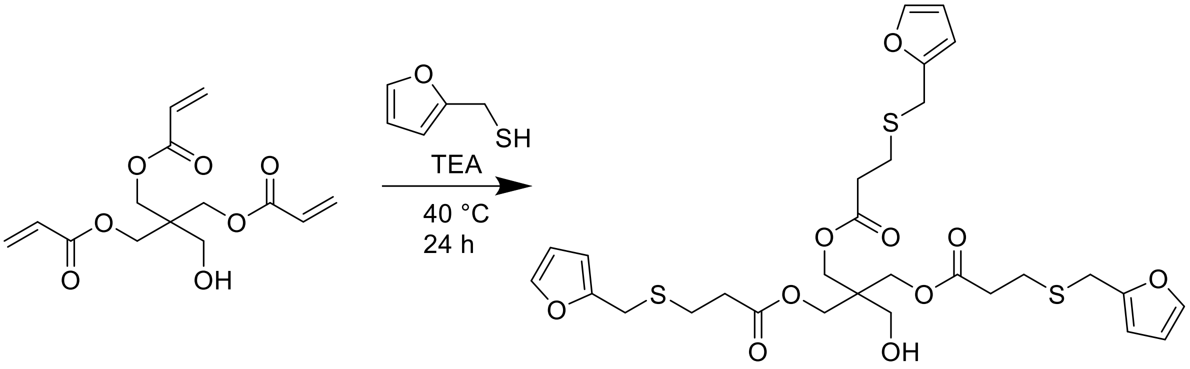
Supplementary Table S1 Amounts and yields of bismaleimide linkers.

Bismaleimide precursor	**Name Product**	**g/mol precursor**	**g/mol product**	**g product**	**Yield (%)**
4,4′-Diaminobenzophenone	bMCO	212.25	372.34	1.0	69
4,4′-Oxydianiline	bMO	200.24	360.33	1.1	71
4,4′-Diaminodiphenyl sulfide	bMS	216.30	376.39	0.9	65
4-Aminophenyl sulfone	$bMSO_2$	248.30	408.38	0.8	52

Supplementary Table S2 Dart-MS found m/z values for the synthesised bismaleimide linkers.

Name Product	**Adduct**	**$(m/z)_{exp}$**	**$(m/z)_{theo}$**
bMCO	$[M+H]^+$	373.0821	373.08
bMO	$[M+NH_4]^+$	378.1084	378.11
bMS	$[M+NH_4]^+$	394.0856	394.09
$bMSO_2$	$[M+NH_4]^+$	426.0755	426.08

4.2. Synthesis of pentaerythritol tris(3-(furfurylthiol)-propionate) [PTF] (**6**) Adapted from Adzima *et al.* (2008)[S1]

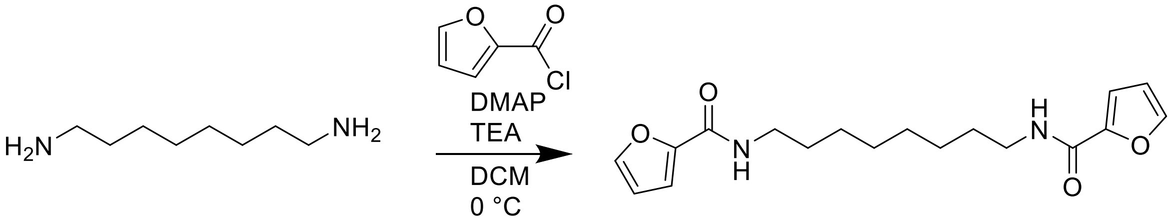


Supplementary Scheme S2 Synthesis of Pentaerythritol Tris(3-(furfurylthiol)-propionate).

4.9 ml (19 mmol) pentaerythritol triacrylate was mixed with 6.4 ml (63 mmol) furfuryl mercaptan and 0.44 ml (3.15 mmol) triethylamine. the reaction mixture was heated at 40 °C for 24 hours while stirring. After the reaction had completed, the remaining furfuryl mercaptan and triethylamine were removed under vacuum at 50 °C and finally under nitrogen flow in the fume hood. A viscous oil was obtained (10.9 g, 89.7%).

4.3. Synthesis of *N*,*N*'-(octane-1,8-diyl)bis(furan-2-carboxamide) (**7**) Adapted from Mayo and Adronov (2013)[S2]

All glassware used was dried in an oven at 120 °C before use.

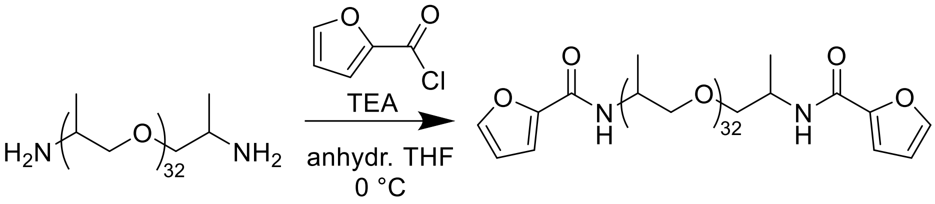


Supplementary Scheme S3 Synthesis of N,N'-(Octane-1,8-diyl)bis(furan-2-carboxamide).

10.0 g (69.3 mmol) 1,8-octanediamine, 19.6 ml (141 mmol) triethylamine and 0.17 g (1.35 mmol) DMAP were dissolved in 200 ml anhydrous DCM under argon. This solution was cooled on an ice bath. A stock solution of 2-furoyl chloride was prepared by dissolving 14.4 ml (146 mmol) 2-furyol chloride in 50 ml anhydrous DCM under argon. The 2-furyol chloride solution was then added to the cooled 1,8-octadiamine solution via dropwise addition. After complete addition the reaction mixture was allowed to stir at room temperature for 1 hour after which the solvent was evaporated via rotary evaporation. The obtained solid was washed with ~2 L of water via vacuum filtration. The solid was then dried overnight in a vacuum oven at 50 °C. A white powder was obtained (14.5 g , 65%).

4.4. Synthesis of PPG-bis-furan (**8**)

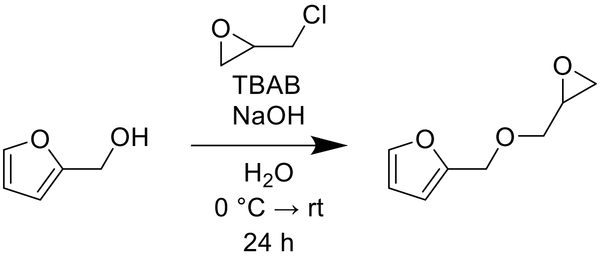
All glassware used was dried in an oven at 120 °C before use.



Supplementary Scheme S4 Synthesis of PPG-bis-furan.

20.0 ml (10.0 mmol) poly(propylene glycol) bis(2-aminopropyl ether) M_n 2000 Da and 4.18 ml (30 mmol) triethylamine were dissolved in 50 ml anhydrous THF. This solution was then purged for 30 min with N_2 in an ice bath. After purging a N_2 filled balloon was added. The reaction mixture was cooled with an ice bath to 0 °C 6.0 ml (60.7 mmol) 2-furfuroyl chloride was dissolved in 2 ml anhydrous THF and added dropwise to the reaction mixture under stirring. After complete addition the reaction was allowed to stir for 1 hour at room temperature. The solvent was evaporated and the solid was redissolved in 30 ml H_2O: MeOH 1:1 and divided over 3 dialysis tubes. This was then dialysed (1 kDA cut-off) against H_2O: MeOH 1:1 for 2 days (medium was refreshed 3 times per day). Next the solvent was evaporated and the product was dried in a 70 °C oven till the weight stabilised, resulting in a brown/yellowish oil was obtained (16.4 g, 77.7% yield).

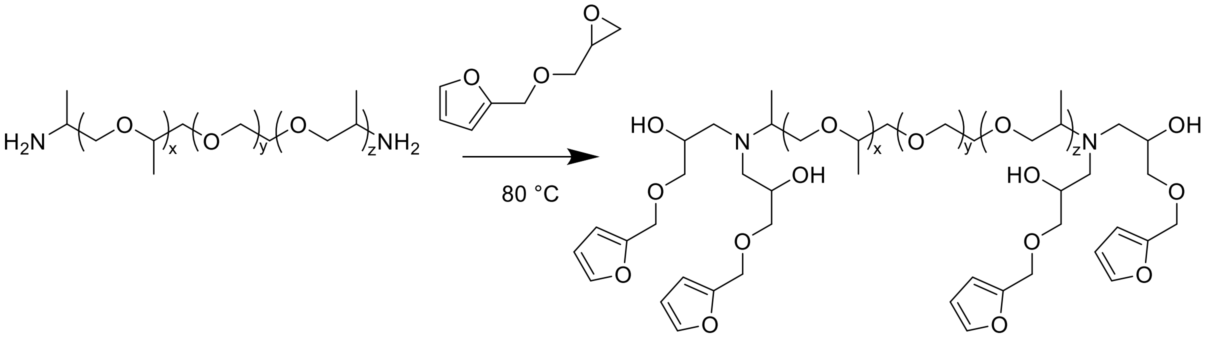
4.5. Synthesis of furfuryl glycidyl ether (adapted from Ye *et al.*)[S3]



Supplementary Scheme 5 Synthesis of furfuryl glycidyl ether.

15 g NaOH was dissolved in 22.5 ml DI water in a 250 ml round bottom flask. The flask was then cooled to 0 °C in an ice bath. 150 mg (0.465 mmol) TBAB was added to the reaction mixture. After the TBAB had fully dissolved, 90 g (0.98 mmol) epichlorohydrin was added. Then 15 g (0.15 mmol) furfuryl alcohol was added to the mixture in a dropwise manner. The reaction was stirred on ice for another four hours, after which the reaction was continued at room temperature for another 20 hours. After the reaction was completed, the mixture was extracted using 2× 100 ml diethyl ether and 1x 100 ml ethyl acetate. The organic layers were collected and washed with 3× 100 ml DI water and 2x 100 ml brine. After washing the organic layer was dried with $MgSO_4$. The solvent was removed and the remaining epichlorohydrin was evaporated using a rotavap at 50 °C and 33 mbar for 5 hours. The last remaining epichlorohydrin was removed using a silica plug run with 50/50 ethyl acetate/petroleum ether 40/60. This resulted in a yellow oil. The yield was 16.7 g (72.2%).

4.6. Synthesis of four-armed furan linker FA4 (**9**) (as reported by McReynolds *et al.*)[S4]



Supplementary Scheme 6 Synthesis of furan linker FA4.

10.0 g (16.7 mmol) of Jeffamine ED-600 and 10.28 g (66.7 mmol) of furfuryl glycidyl ether were mixed in a round bottom flask and stirred at 80 °C for 48 h. The reaction mixture went from a deep yellow to a dark brown oil. Conversion was quantitative as determined by NMR.

4.7. Preparation of **FA3-alkyl/PPG** networks

Materials were prepared by dissolving the bismaleimide, *N*,*N*'-(octane-1,8-diyl)bis(furan-2-carboxamide) or PPG-bis-furan and pentaerythritol tris(3-(furfurylthiol)-propionate) in DCM : methanol 95:5 in a 1:1 molar equivalence of the reactive groups. The solution was then mixed and evaporated. The resulting solid was powderised and pressed into shape with a circular 10 mm die and pressed for 20 seconds at 120 °C and 1 ton. Then sample was then heated at 70 °C for at least for a duration of 2 days.

4.8. Network deposition on KBr pellets (**FA3-alkyl**)

Stock solution of the different networks were prepared by dissolving low amounts of the (0.019 mmol) **bMX** linker, (0.0057 mmol) *N*,*N*'-(octane-1,8-diyl)bis(furan-2-carboxamide) and (0.0089 mmol) pentaerythritol tris(3-(furfurylthiol)-propionate) in 0.1 ml DCM. After mixing 3 drops were deposited on a KBr pellet (400 mg) and dried in the fume hood at room temperature. After evaporation the samples were measured to obtain the starting intensity. The samples were then stored for at least 3 days each at 70, 80, 90 and 100 °C in a dry oven. All samples were prepared in triplicate. The IR absorbance was measured for each temperature and compared to the starting intensity. The maleimide peak at 690 cm^{-1} was integrated using an end point weighted baseline fit and compared for all samples.

The samples with **bMSO$_2$** were dissolved in 0.4 ml DCM due to solubility issues.

The samples with **bMCO** were dissolved in 0.6 ml DCM + 0.1 ml THF + 0.1 ml ACN due to solubility issues. The solution was extensively sonicated and vortexed before use.

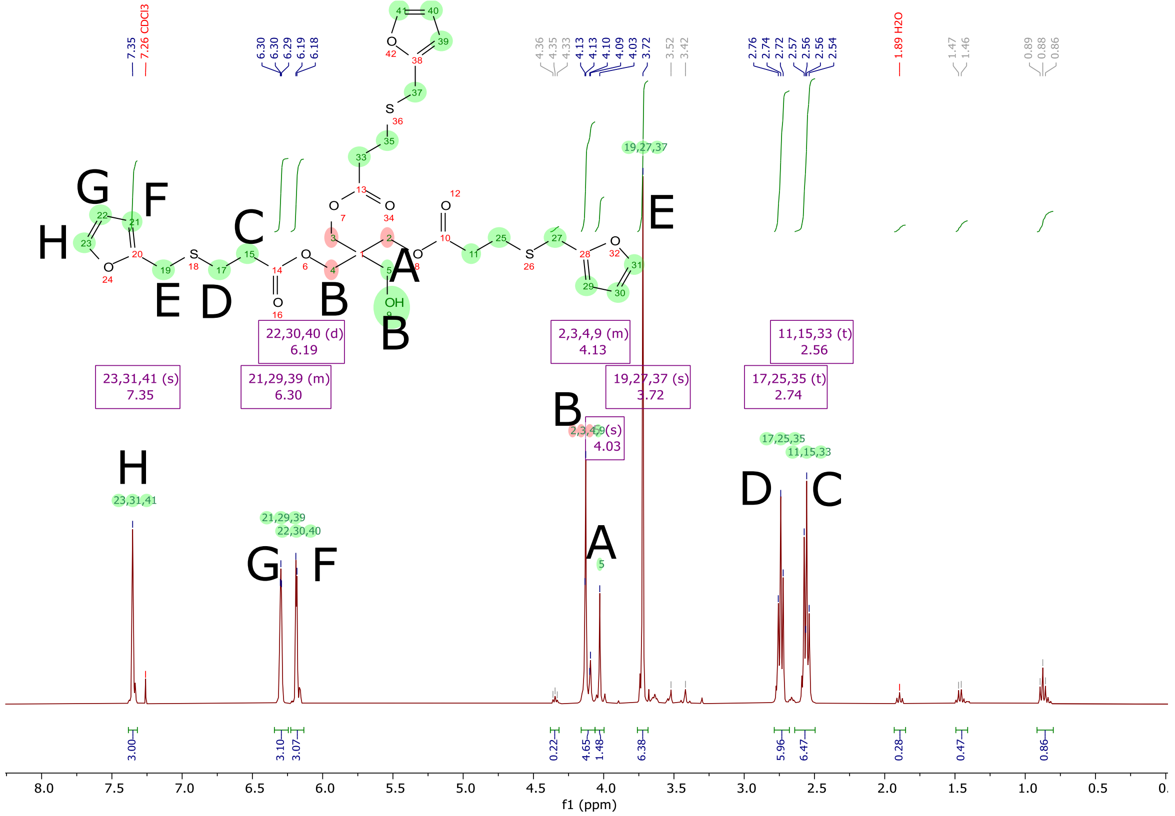
4.9. Preparation of **FA4** networks

352.8 mg (0.290 mmol) **FA4** and (0.348 mmol) **bMX** linker were separately dissolved in 1 ml DMF. The molar ratio used was 0.6 maleimide/furan functional group. Both stock solutions were mixed and vortexed for 10 seconds. This solution was then cast in a plastic sample tube with a diameter of 1.5 cm. after drying in the oven at 70 °C a film was obtained. This film was then trimmed to a circle with a diameter of 1 cm for rheology.

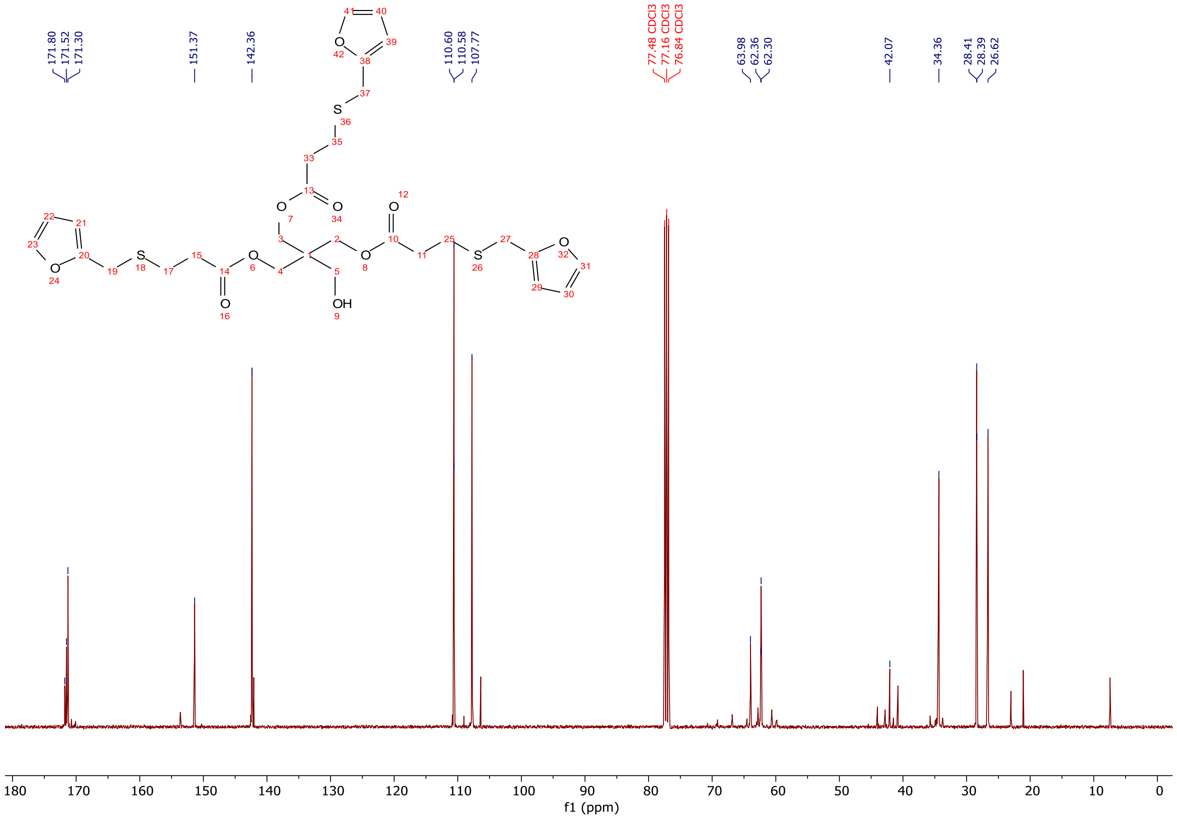
4.10. **FA4** network IR measurements

17.64 mg (0.0145 mmol) **FA4** and 0.0174 mmol **bMX** linker were dissolved in 1 ml DCM. The solution was vortexed for 10 seconds. 1-2 drops of stock solution were deposited on a Teflon sheet. After evaporation of the solvent under air flow, the sample was transferred to the crystal of an ATR-IR that was already equilibrated to the set temperature. The sample was then measured every minute (16 scans, 4 cm^{-1} resolution).

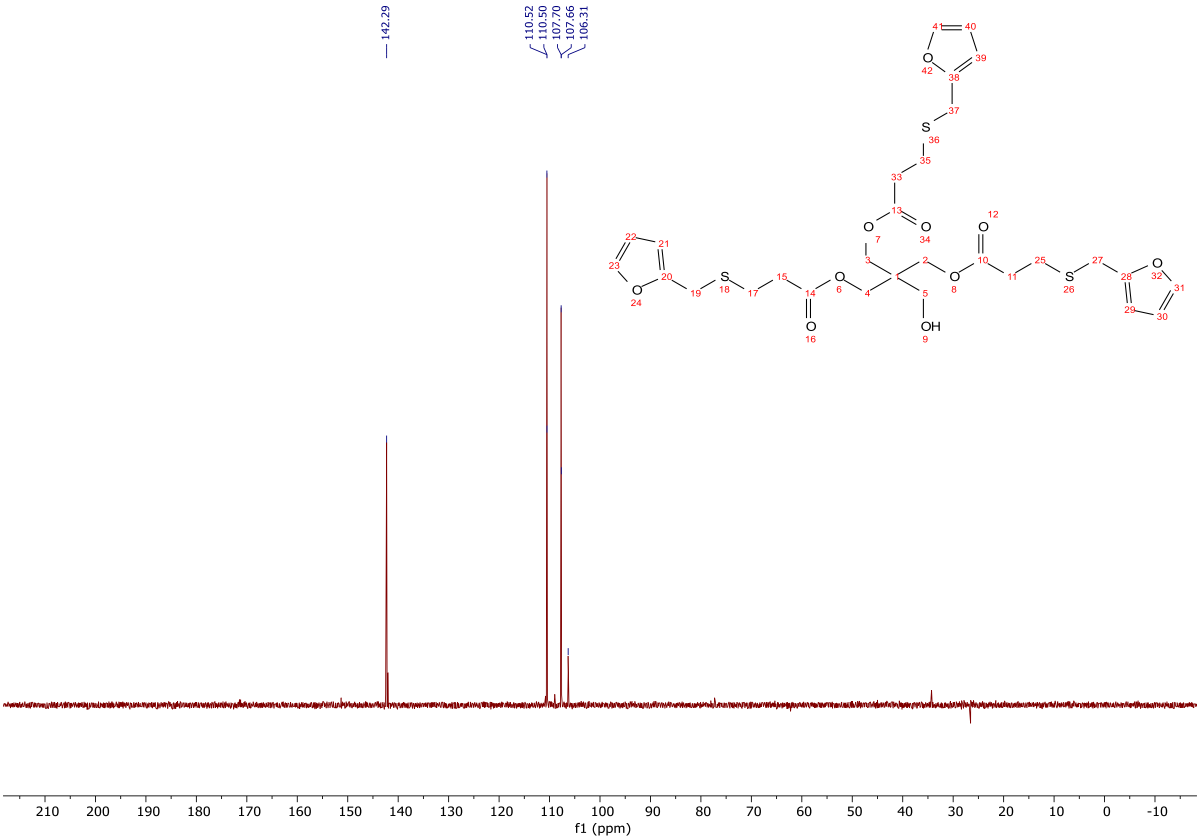
5. NMR spectra



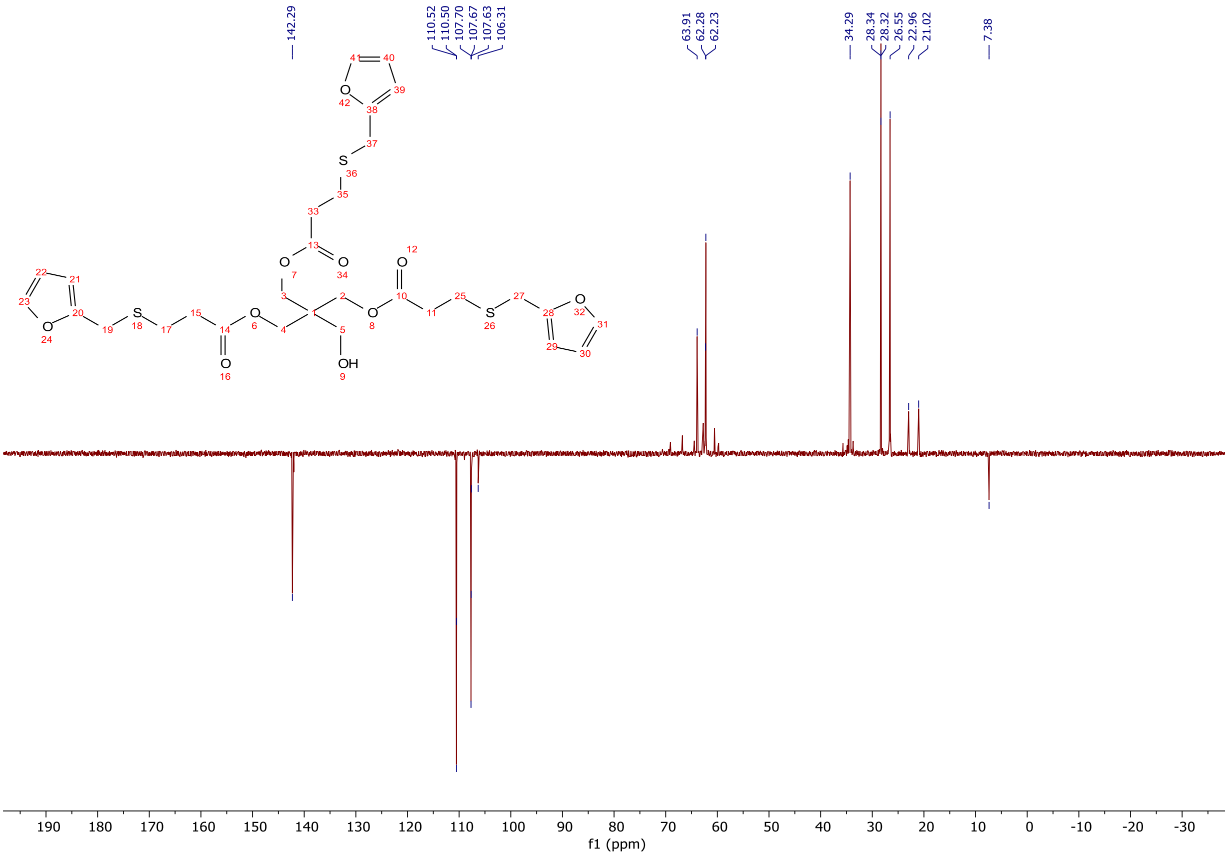
Supplementary Figure S1 ^{1}H-NMR spectrum of pentaerythritol tris(3-(furfurylthiol)-propionate) in $CDCl_3$.



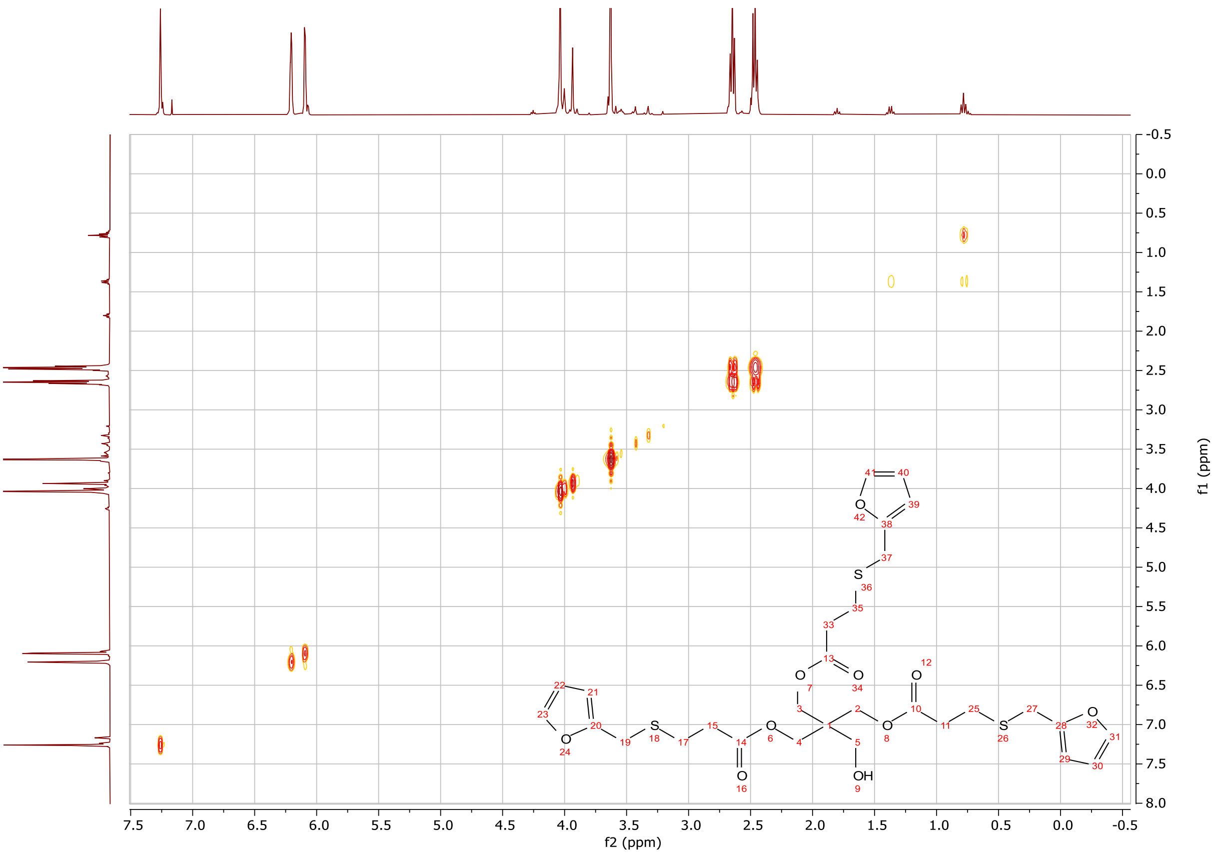
Supplementary Figure S2 ^{13}C-NMR spectrum of pentaerythritol tris(3-(furfurylthiol)-propionate) in $CDCl_3$.



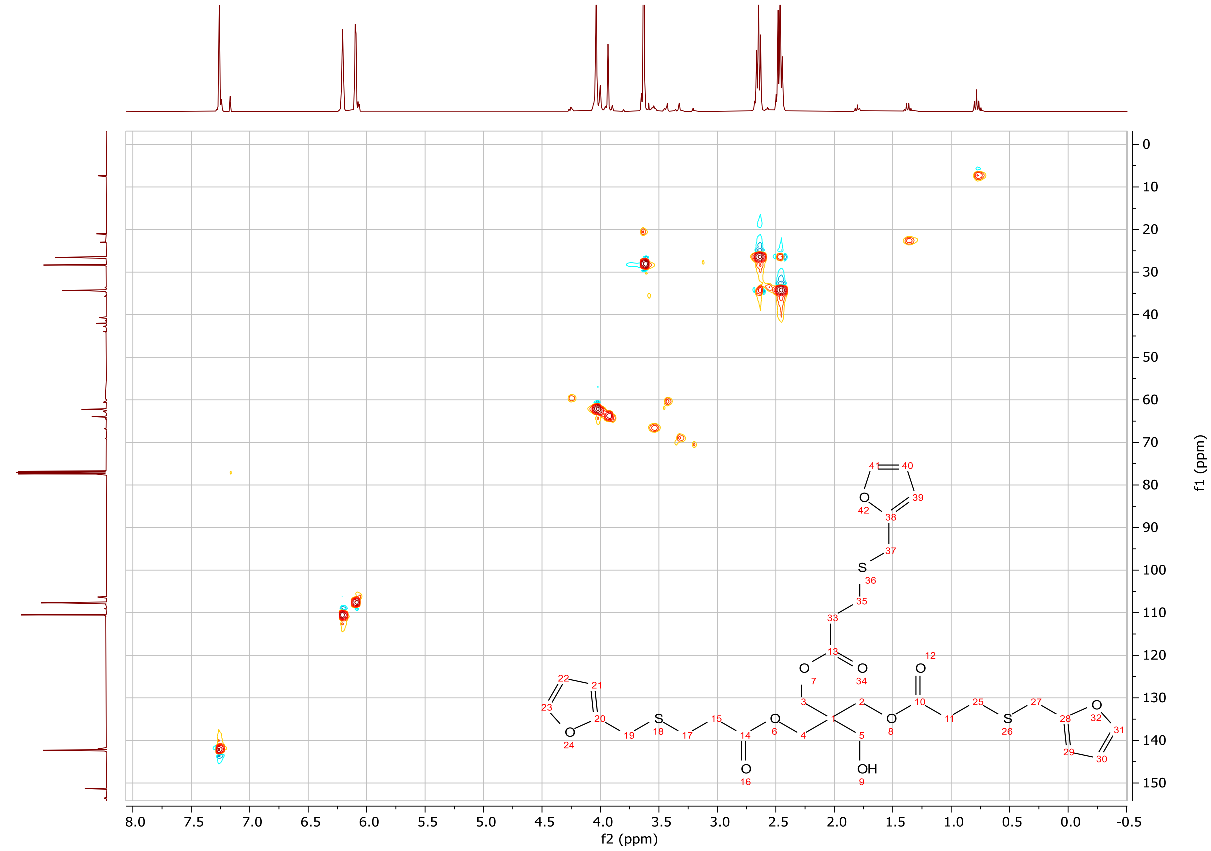
Supplementary Figure S3 DEPT90 spectrum of pentaerythritol tris(3-(furfurylthiol)-propionate) in CDCl$_3$.



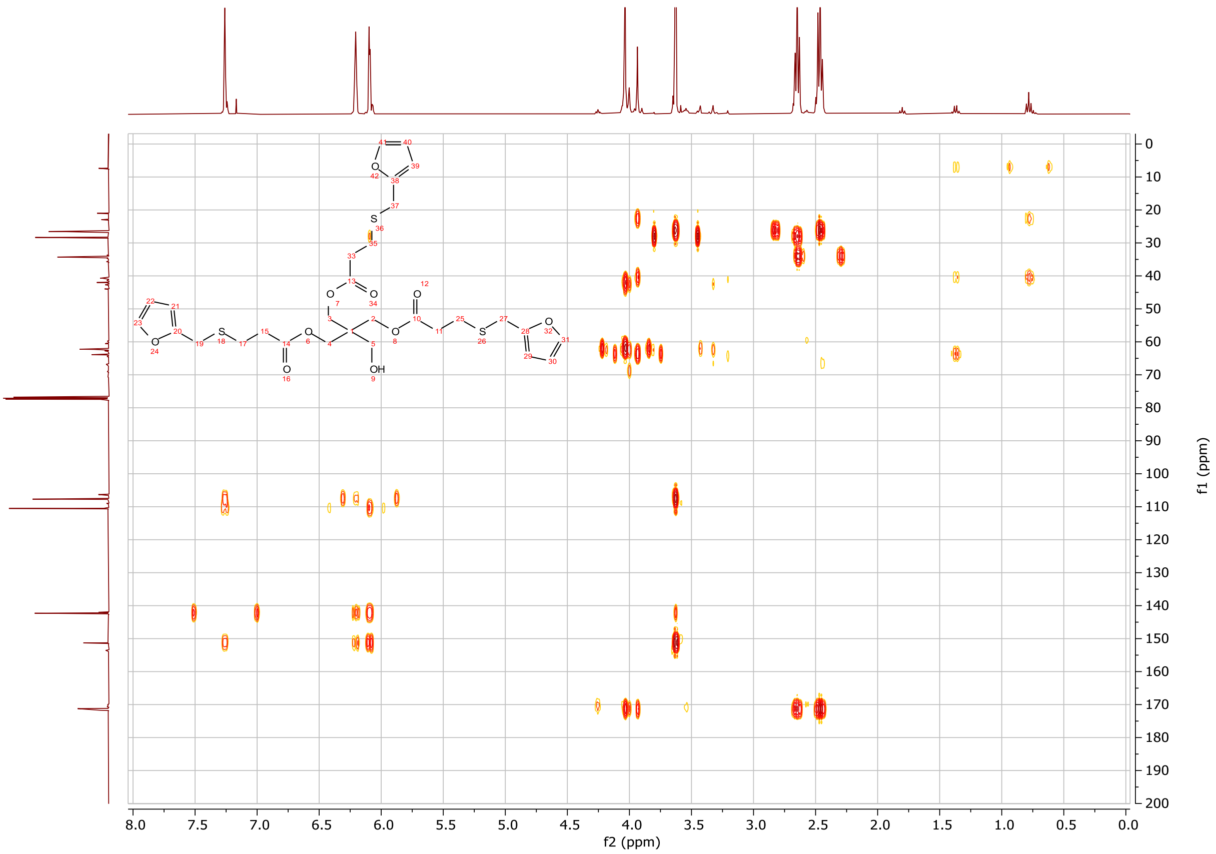
Supplementary Figure S4 DEPT135 spectrum of pentaerythritol tris(3-(furfurylthiol)-propionate) in CDCl$_3$.



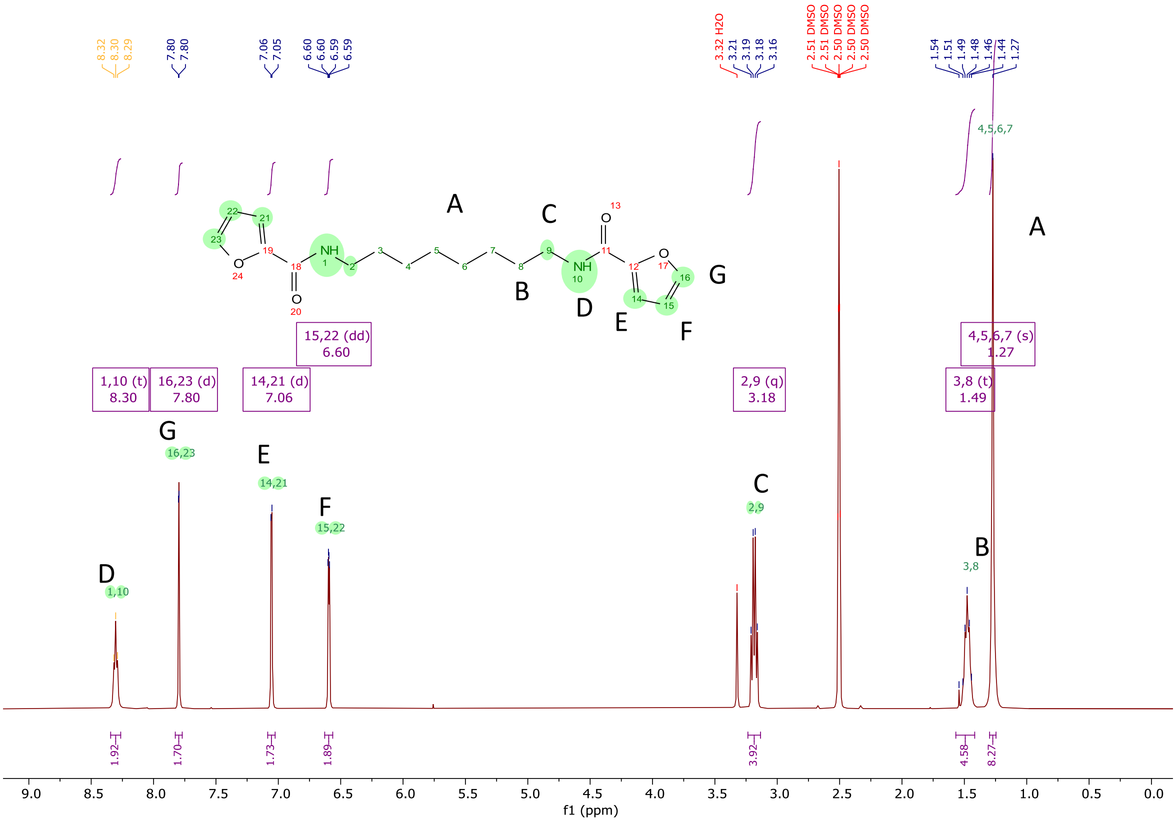
Supplementary Figure S5 COSY spectrum of pentaerythritol tris(3-(furfurylthiol)-propionate) in $CDCl_3$.



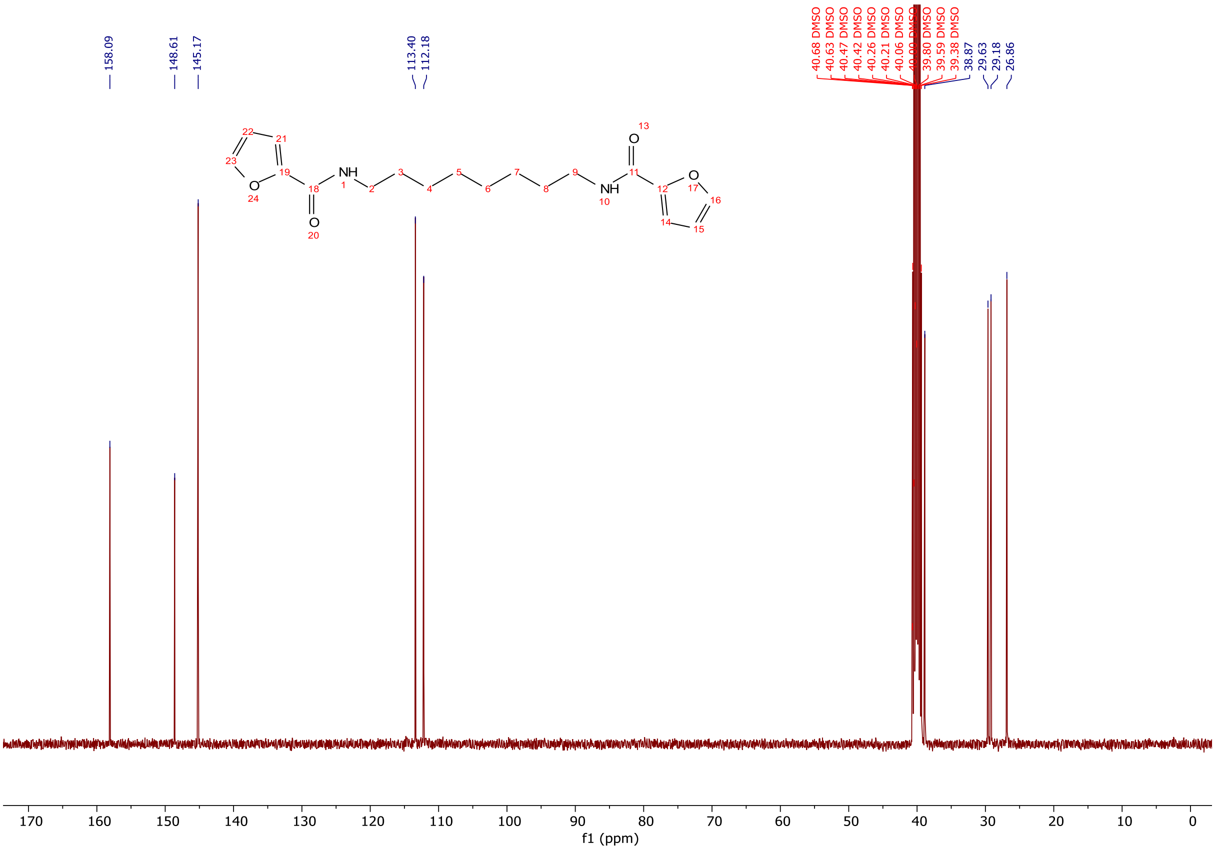
Supplementary Figure S6 HSQC spectrum of pentaerythritol tris(3-(furfurylthiol)-propionate) in $CDCl_3$.



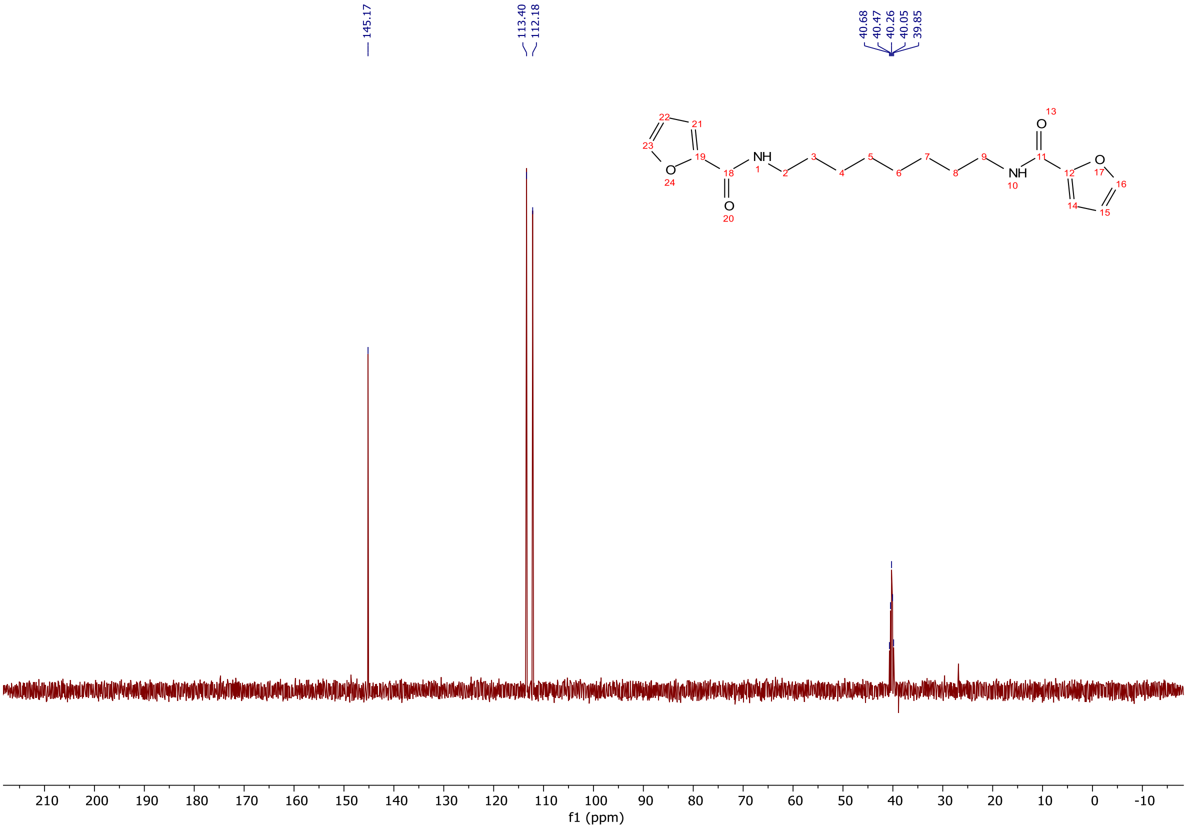
Supplementary Figure S7 HMBC spectrum of pentaerythritol tris(3-(furfurylthiol)-propionate) in $CDCl_3$.



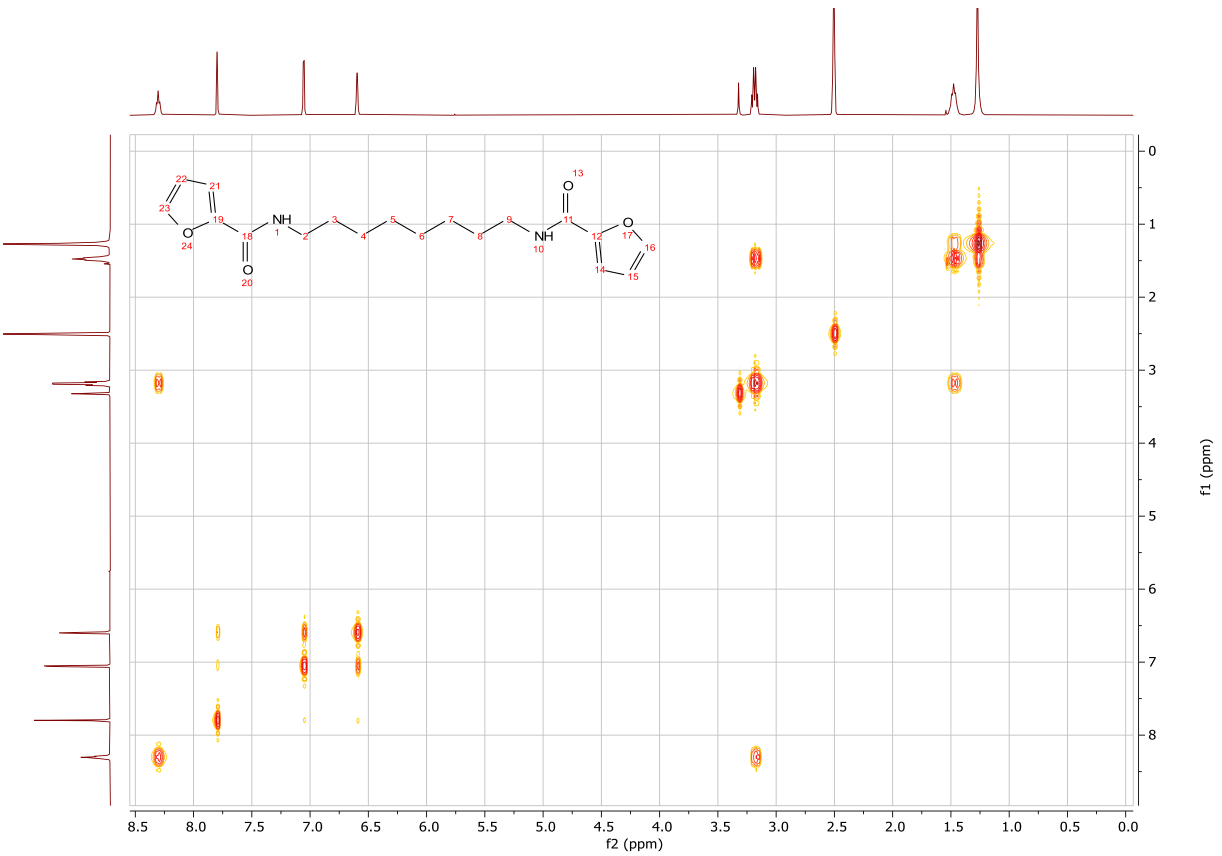
Supplementary Figure S8 ^{1}H-NMR spectrum of N,N'-(octane-1,8-diyl)bis(furan-2-carboxamide) in d_6-DMSO.



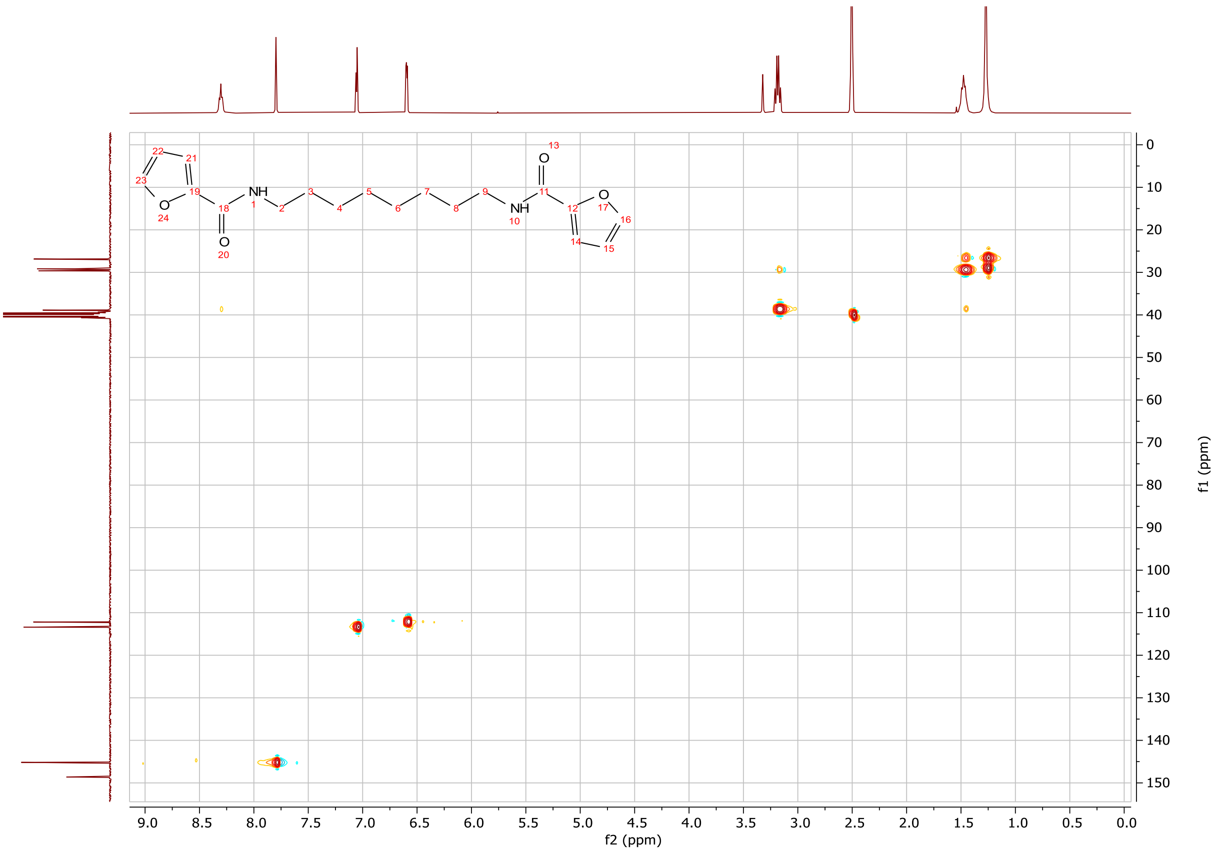
Supplementary Figure S9 ^{13}C-NMR spectrum of N,N'-(octane-1,8-diyl)bis(furan-2-carboxamide) in d$_6$-DMSO.



Supplementary Figure S10 DEPT90 of N,N'-(octane-1,8-diyl)bis(furan-2-carboxamide) in d$_6$-DMSO.

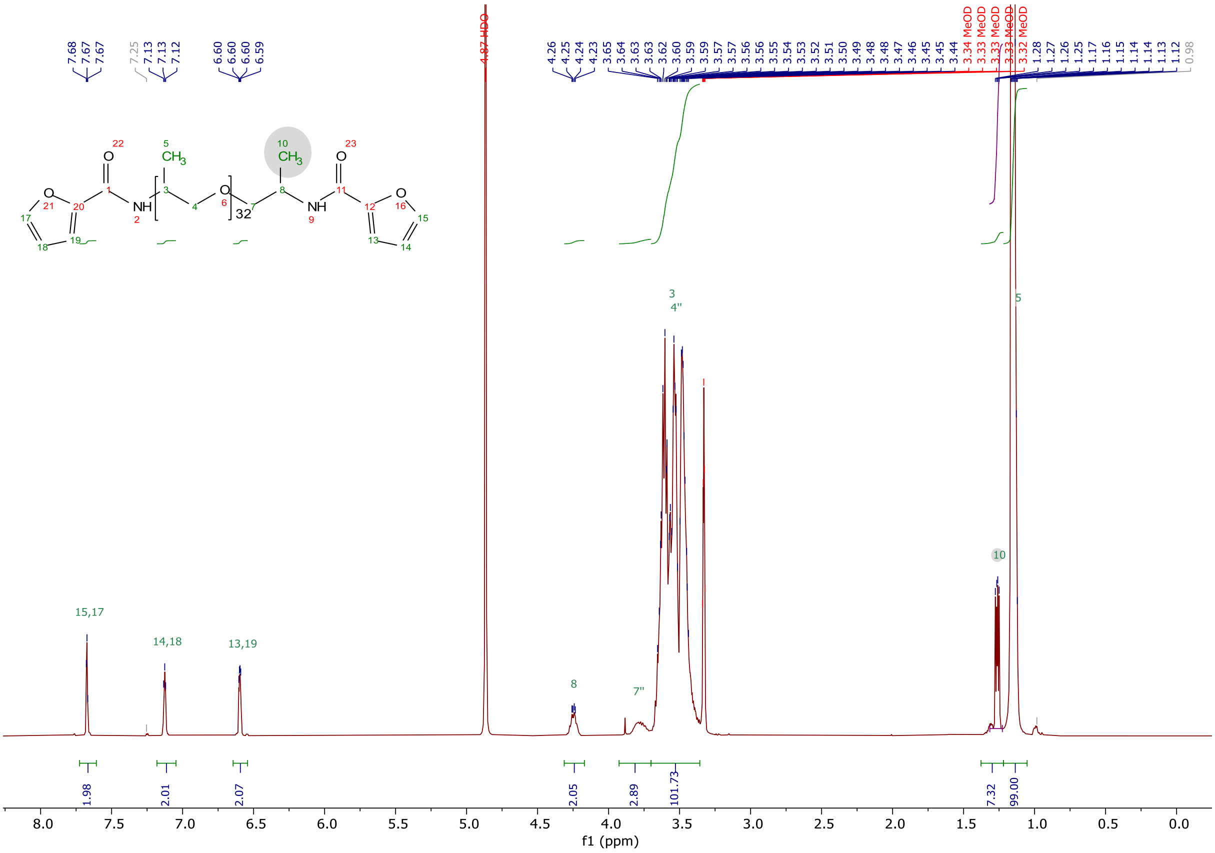


Supplementary Figure S11 COSY of of N,N'-(octane-1,8-diyl)bis(furan-2-carboxamide) in d_6-DMSO.

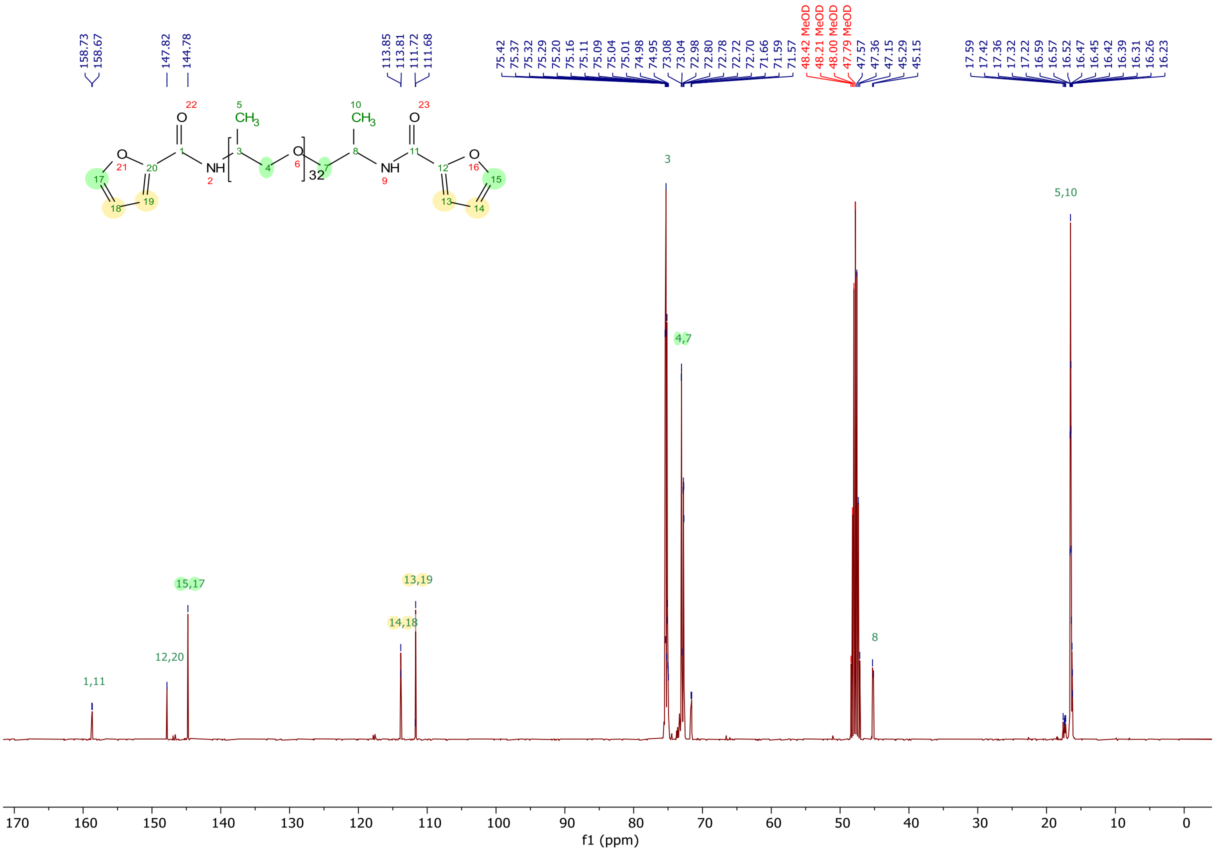


Supplementary Figure S12 HSQC of N,N'-(octane-1,8-diyl)bis(furan-2-carboxamide) in d_6-DMSO.

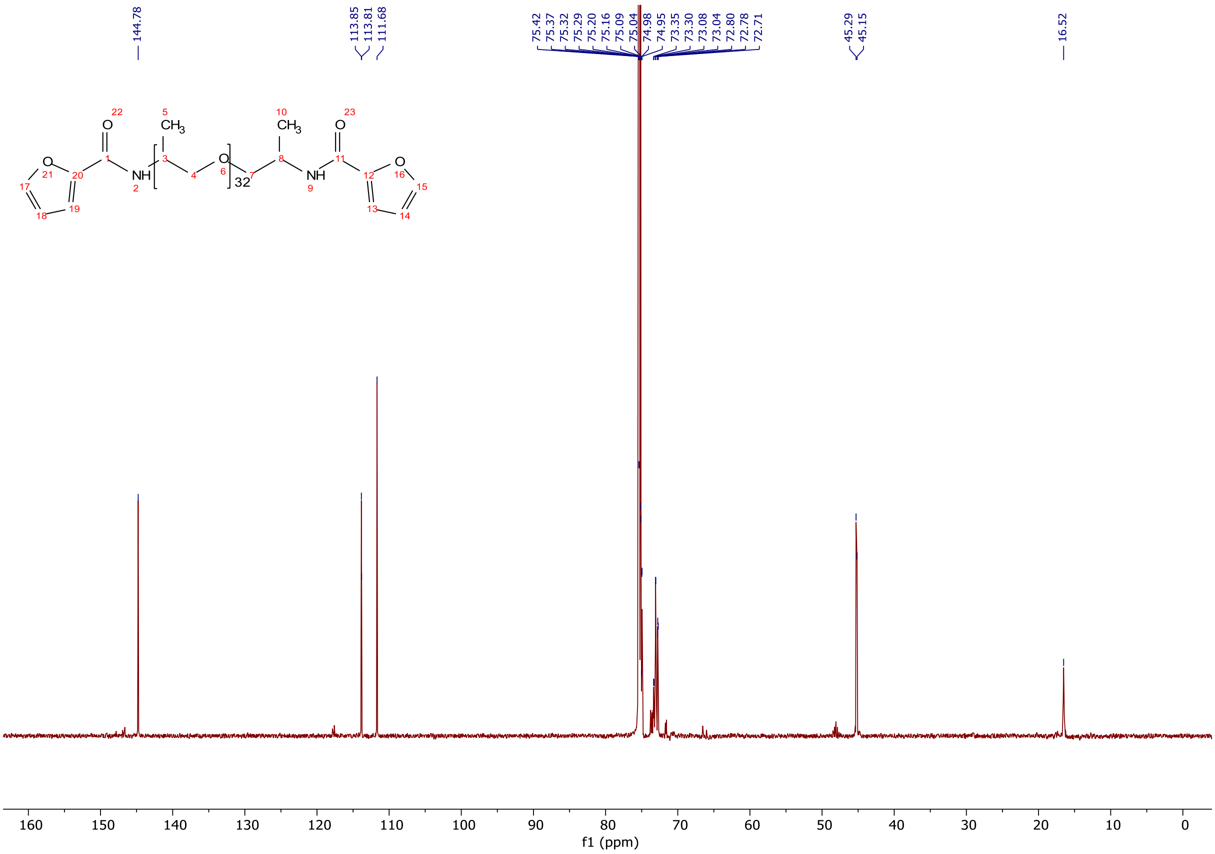
Supplementary Figure S13 HMBC of N,N'-(octane-1,8-diyl)bis(furan-2-carboxamide) in d_6-DMSO.



*Supplementary Figure S14 ^{1}H-NMR spectrum of **PPG-bis-furan** in d_4-MeOD.*



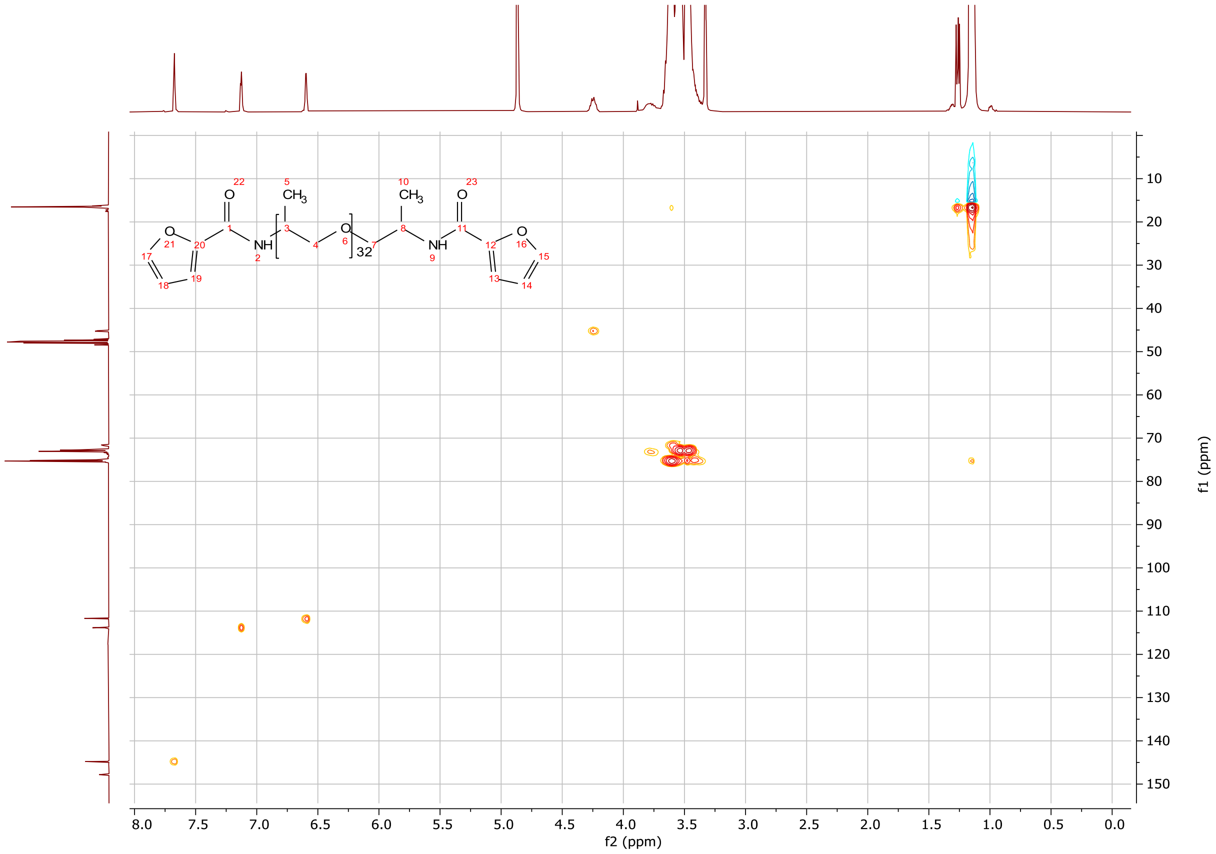
Supplementary Figure S15 ^{13}C-NMR spectrum of ***PPG-bis-furan*** *in d_4-MeOD.*



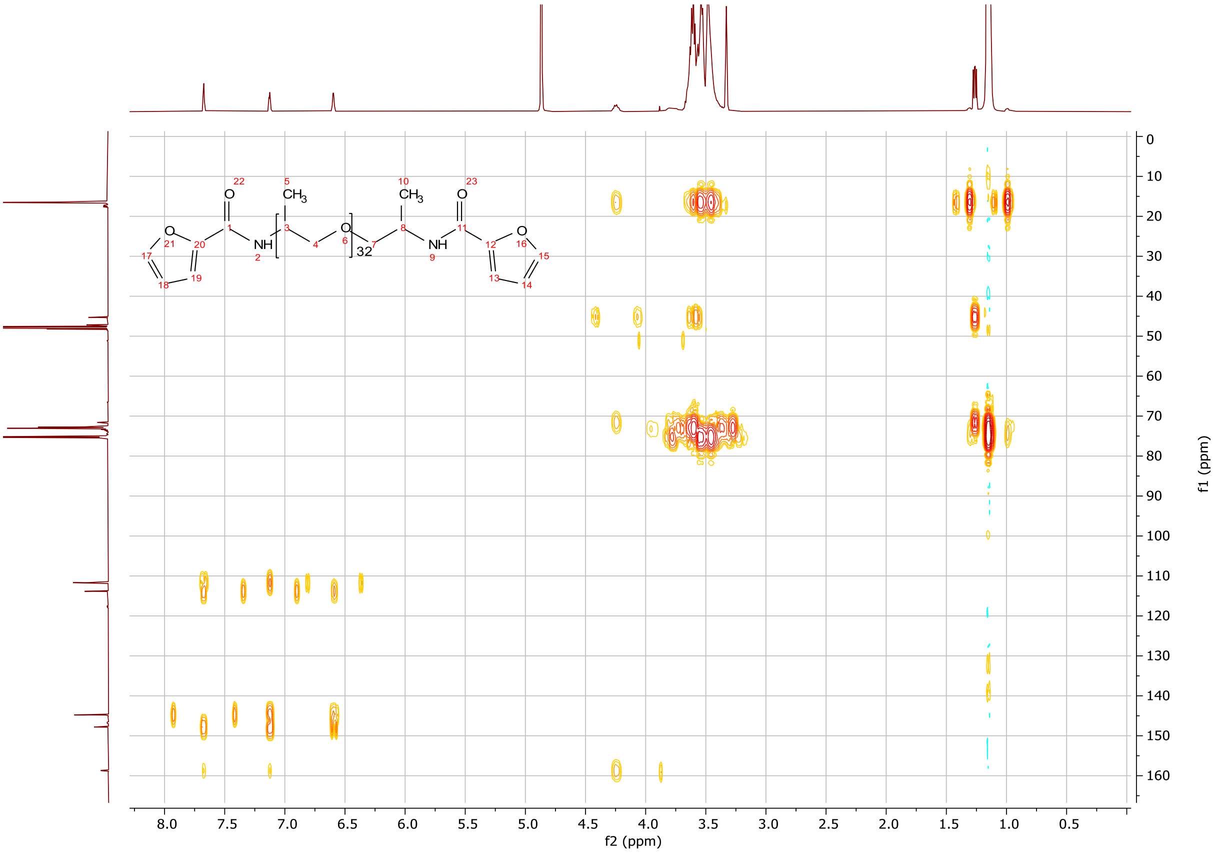
Supplementary Figure S16 DEPT90 spectrum of ***PPG-bis-furan*** *in d_4-MeOD.*

Supplementary Figure S17 DEPT135 spectrum of ***PPG-bis-furan*** *in* d_4*-MeOD.*

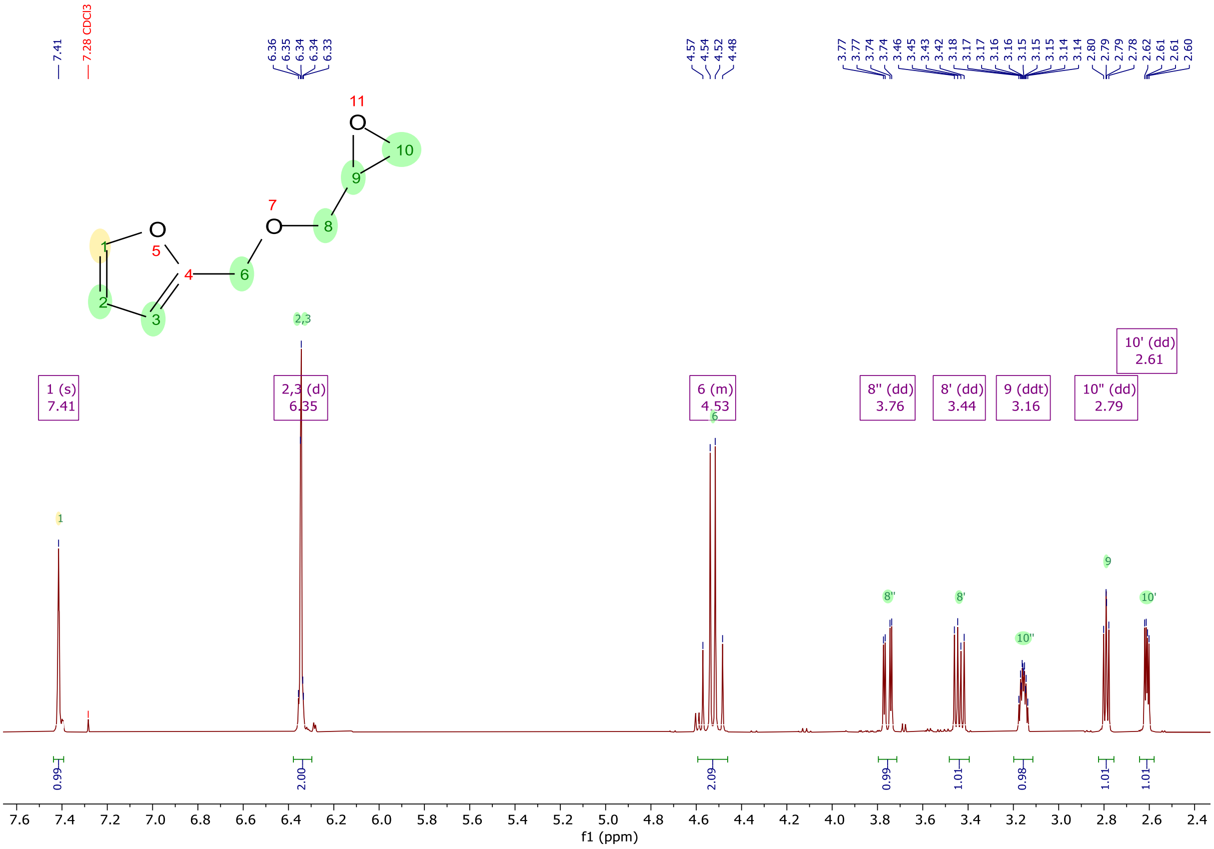
Supplementary Figure S18 COSY spectrum of ***PPG-bis-furan*** *in* d_4*-MeOD.*



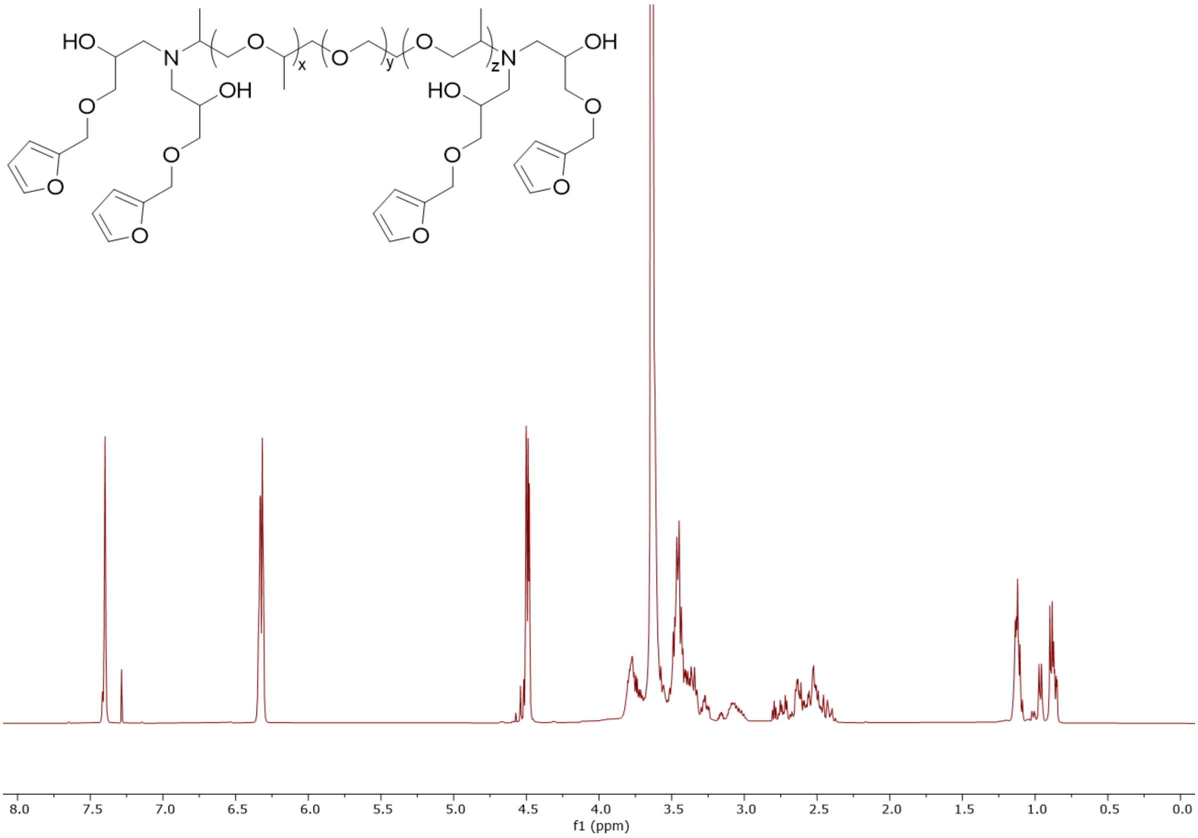
Supplementary Figure S19 HSQC spectrum of ***PPG-bis-furan*** *in* d_4*-MeOD.*



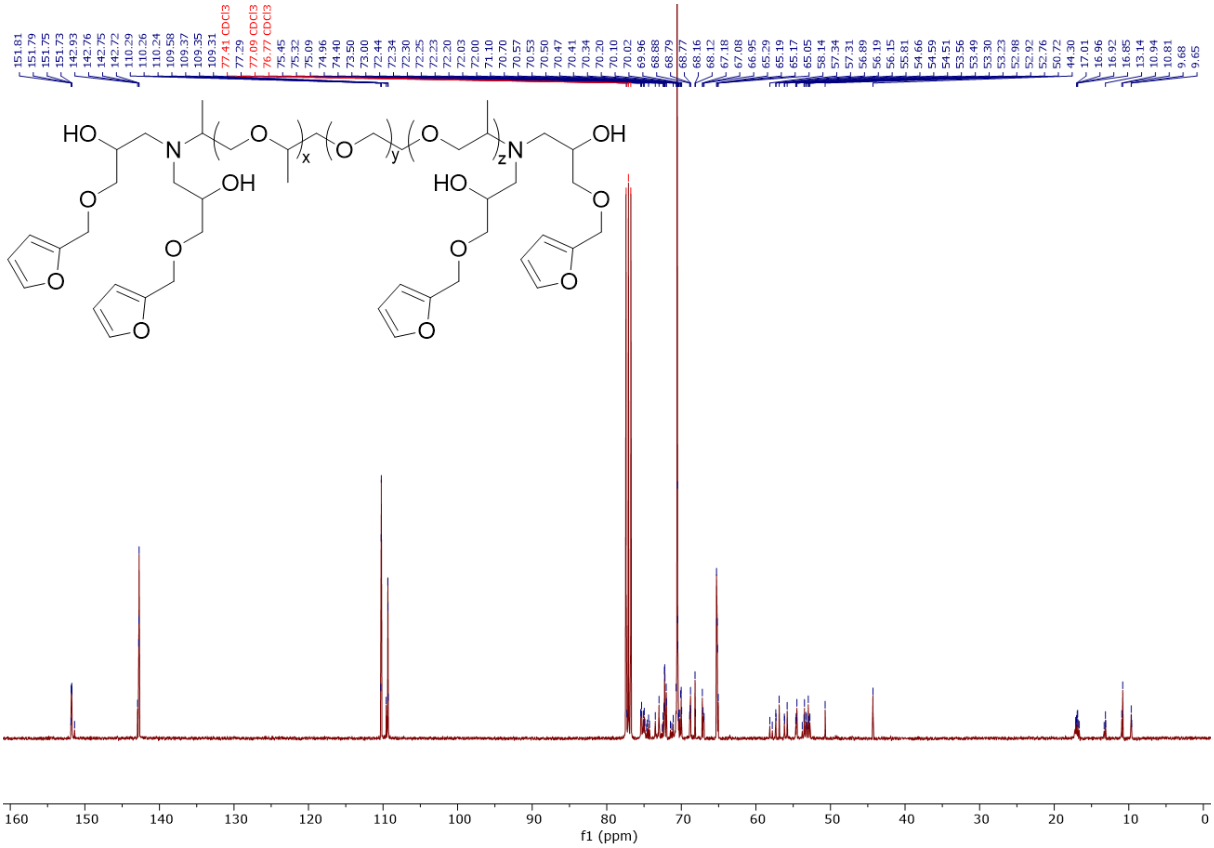
Supplementary Figure S20 HMBC spectrum of ***PPG-bis-furan*** *in* d_4*-MeOD.*



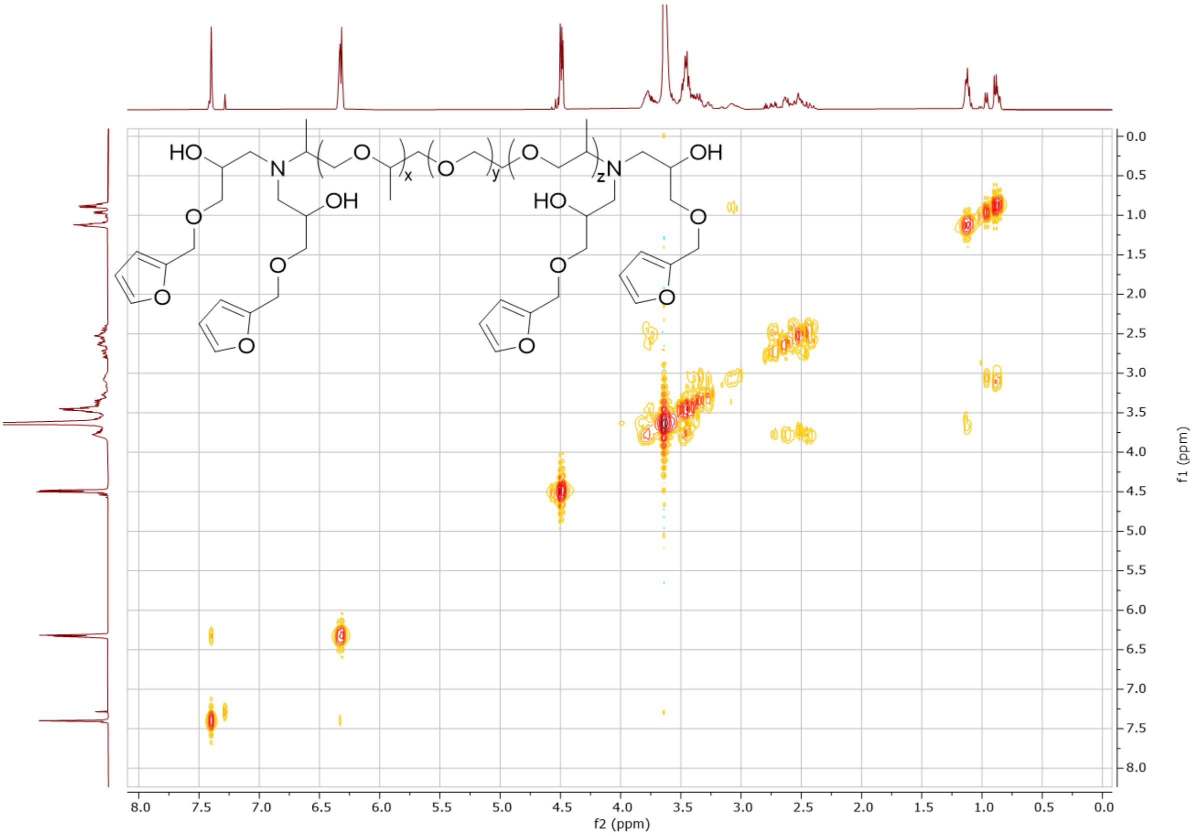
Supplementary Figure S21 ^{1}H-NMR spectrum of furfuryl glycidyl ether in CDCl$_3$.



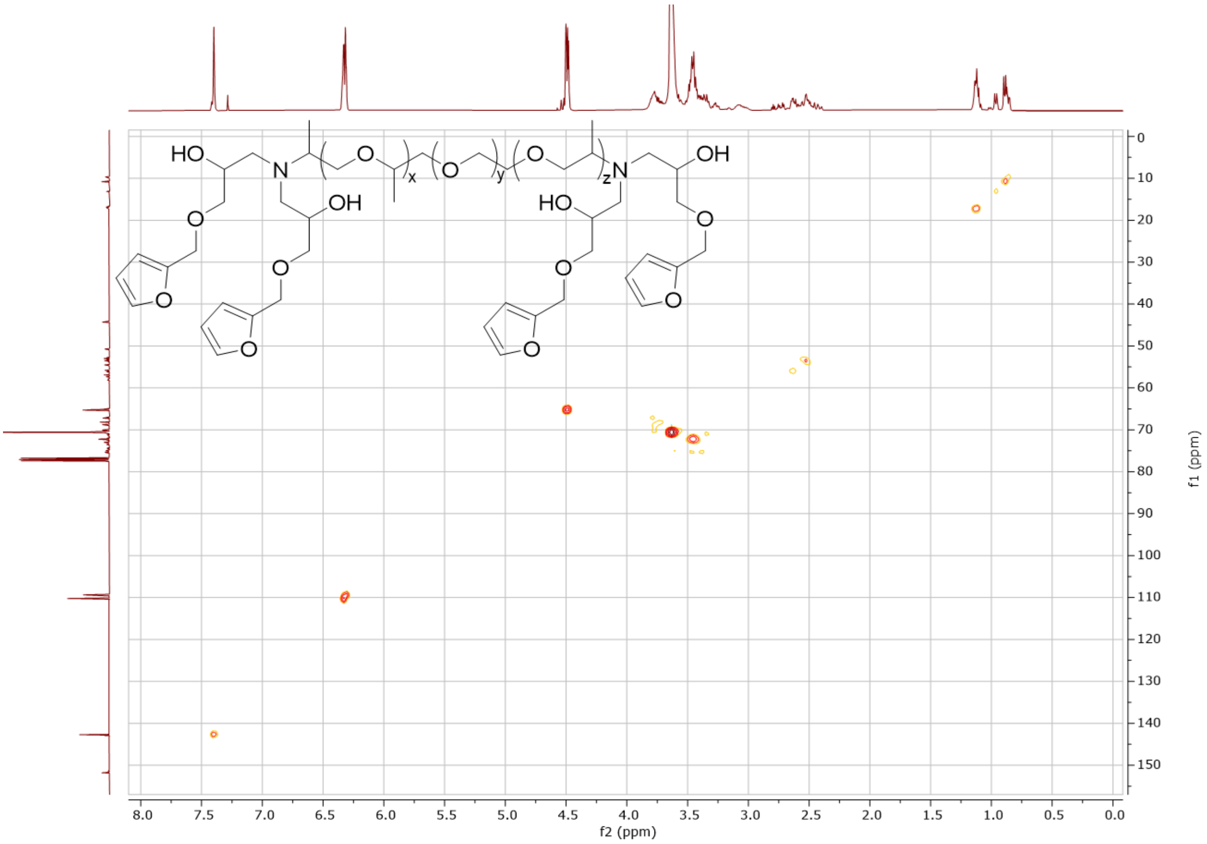
*Supplementary Figure S22 ^{1}H-NMR spectrum of the four-armed furan linker **FA4** in CDCl$_3$.*



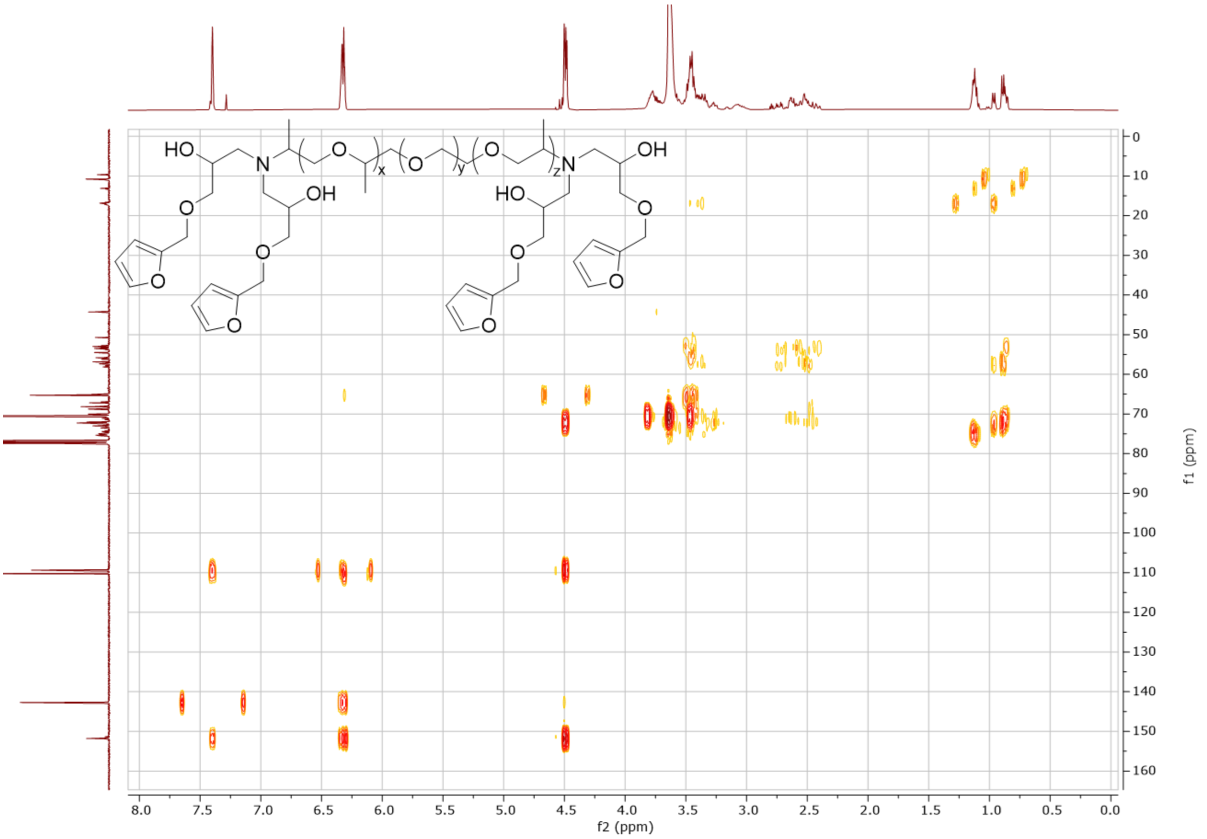
*Supplementary Figure S23 ^{13}C-NMR spectrum of the four-armed furan linker **FA4** in $CDCl_3$.*



*Supplementary Figure S24 COSY spectrum of the four-armed furan linker **FA4** in $CDCl_3$.*

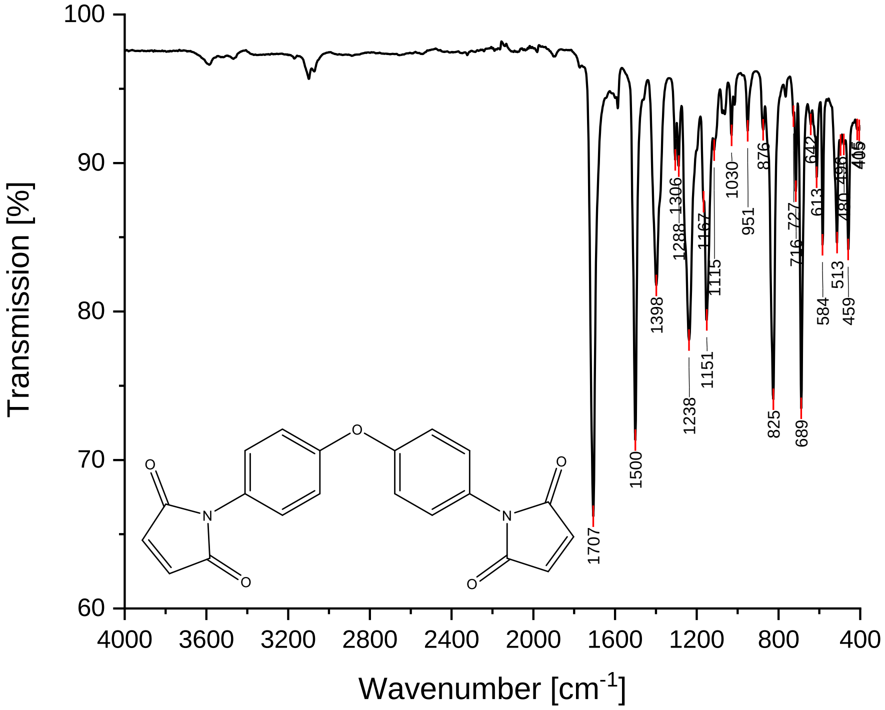


Supplementary Figure S25 HSQC spectrum of the four-armed furan linker ***FA4*** *in $CDCl_3$.*

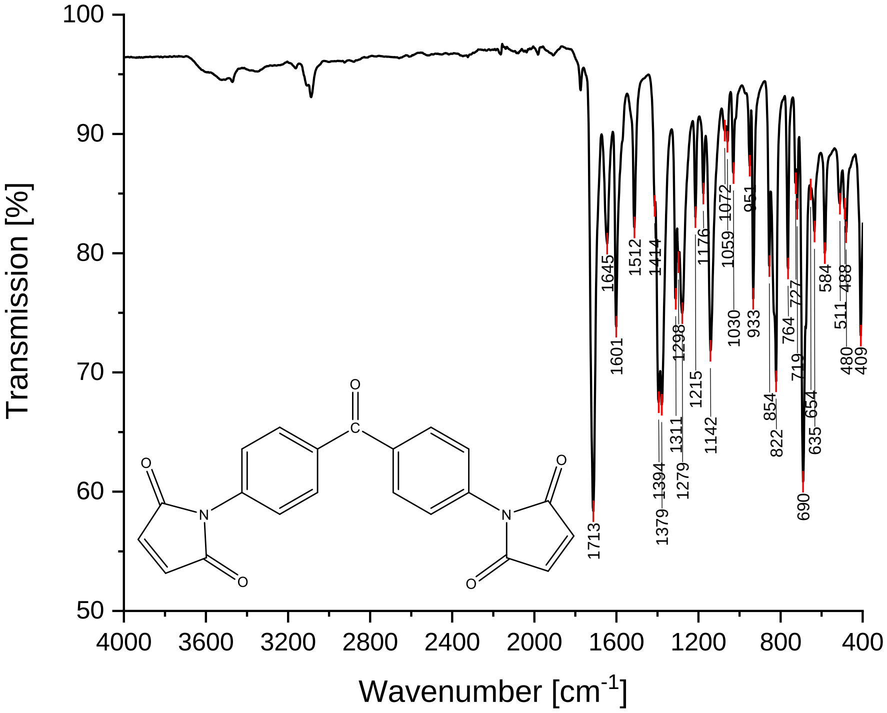


Supplementary Figure S26 HMBC spectrum of the four-armed furan linker ***FA4*** *in $CDCl_3$.*

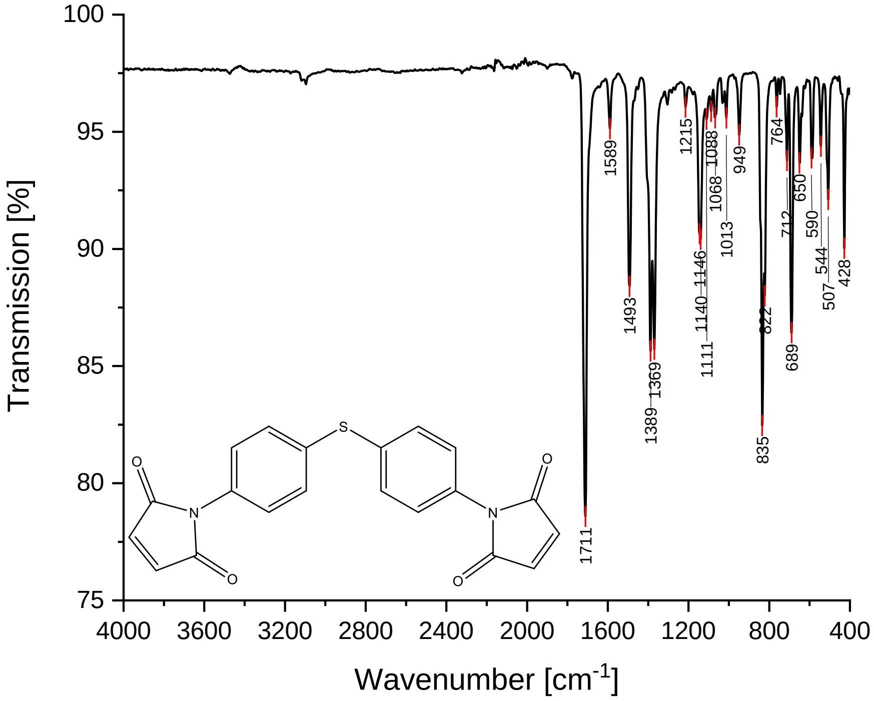
6. IR spectra



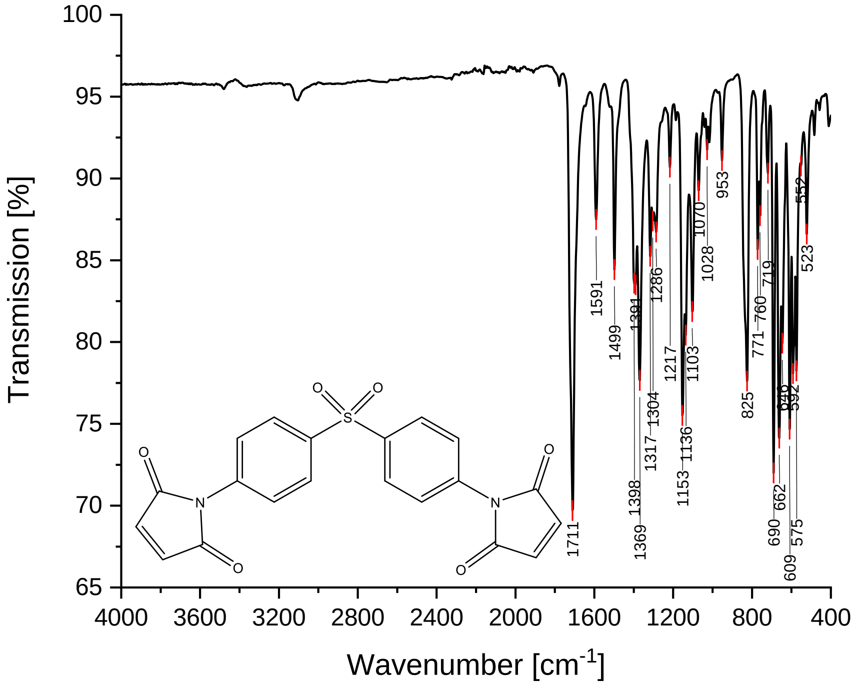
*Supplementary Figure S27 IR spectrum of bismaleimide linker **bMO**.*



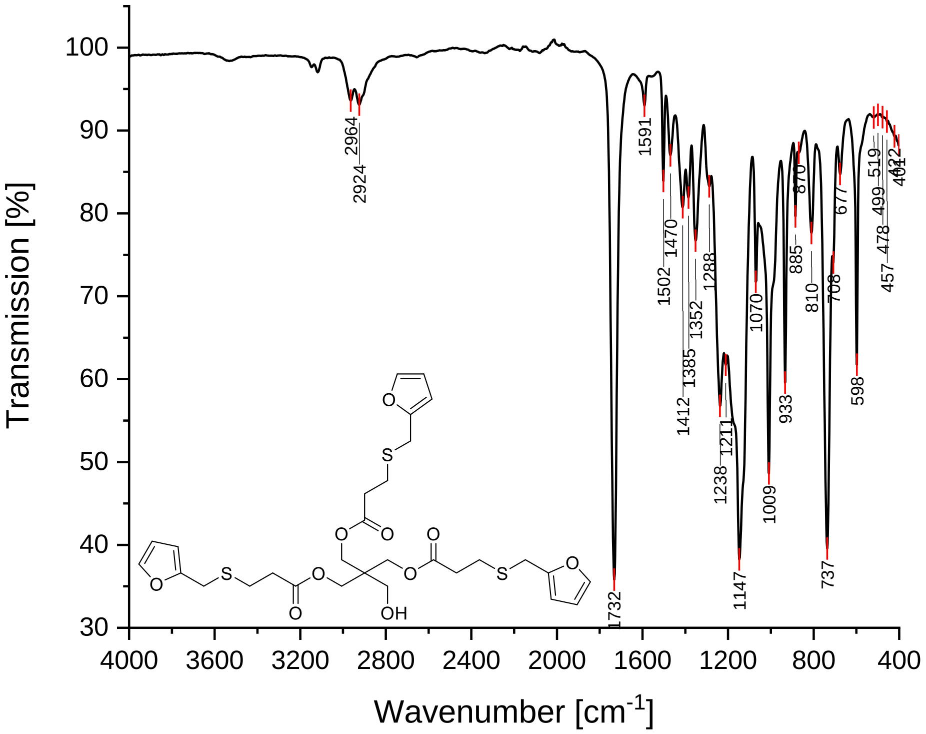
*Supplementary Figure S28 IR spectrum of bismaleimide linker **bMCO**.*



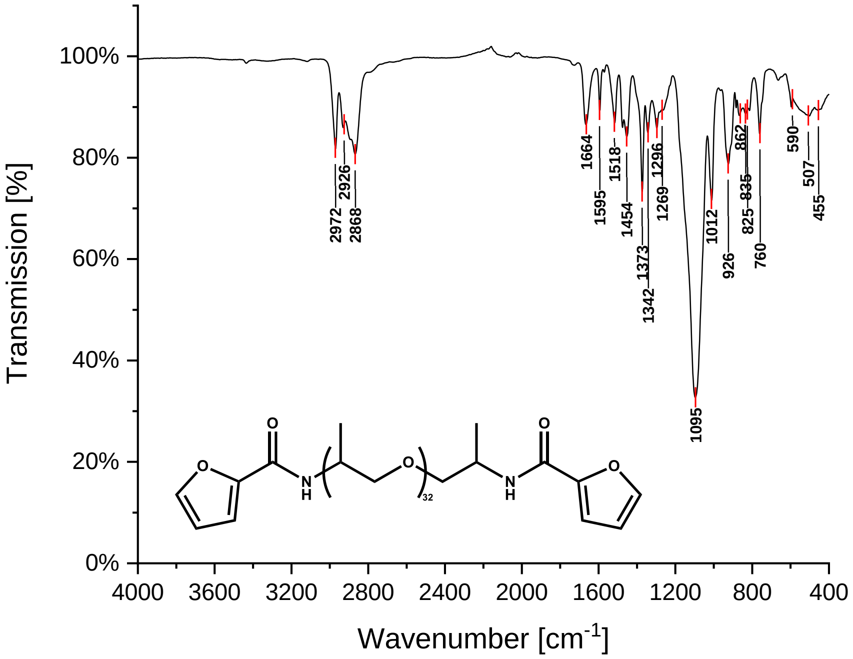
Supplementary Figure S29 IR spectrum of bismaleimide linker ***bMS****.*



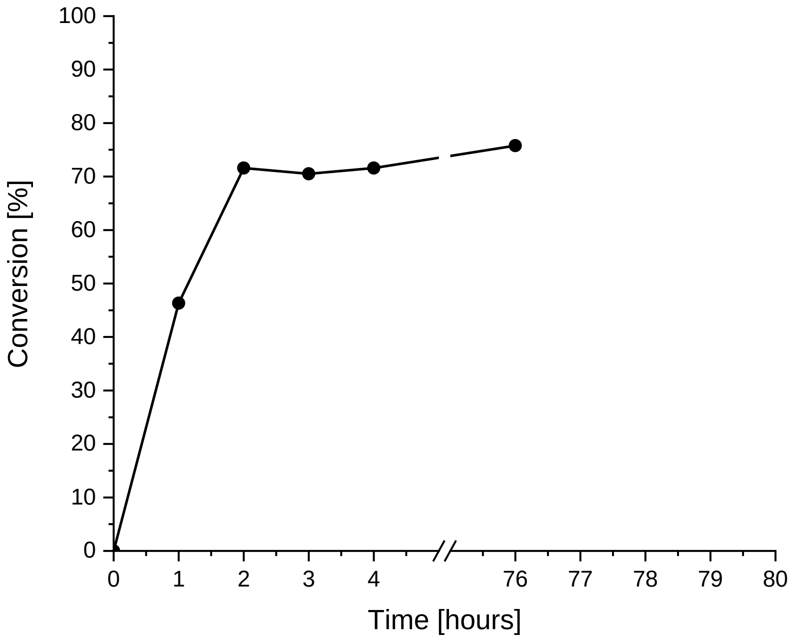
Supplementary Figure S30 IR spectrum of bismaleimide linker ***bMSO$_2$****.*



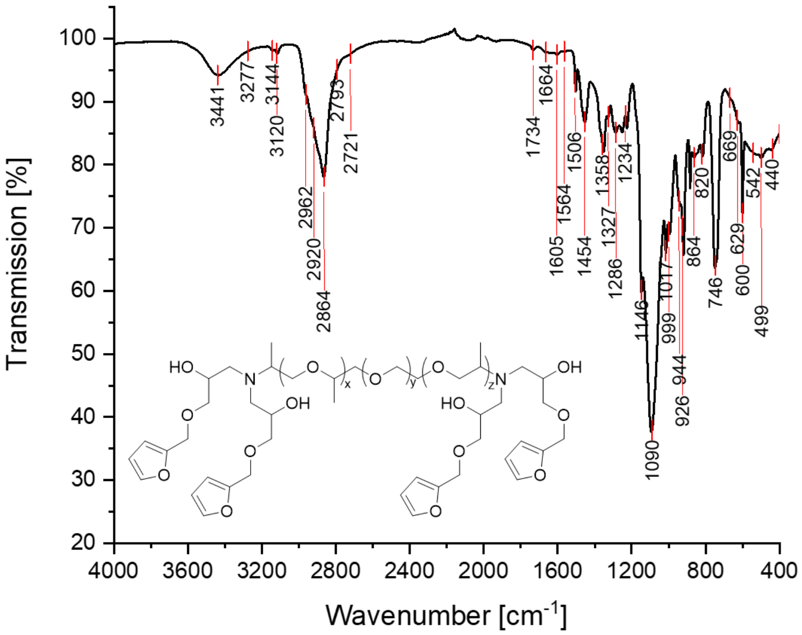
Supplementary Figure S31 IR spectrum of pentaerythritol tris(3-(furfurylthiol)-propionate).



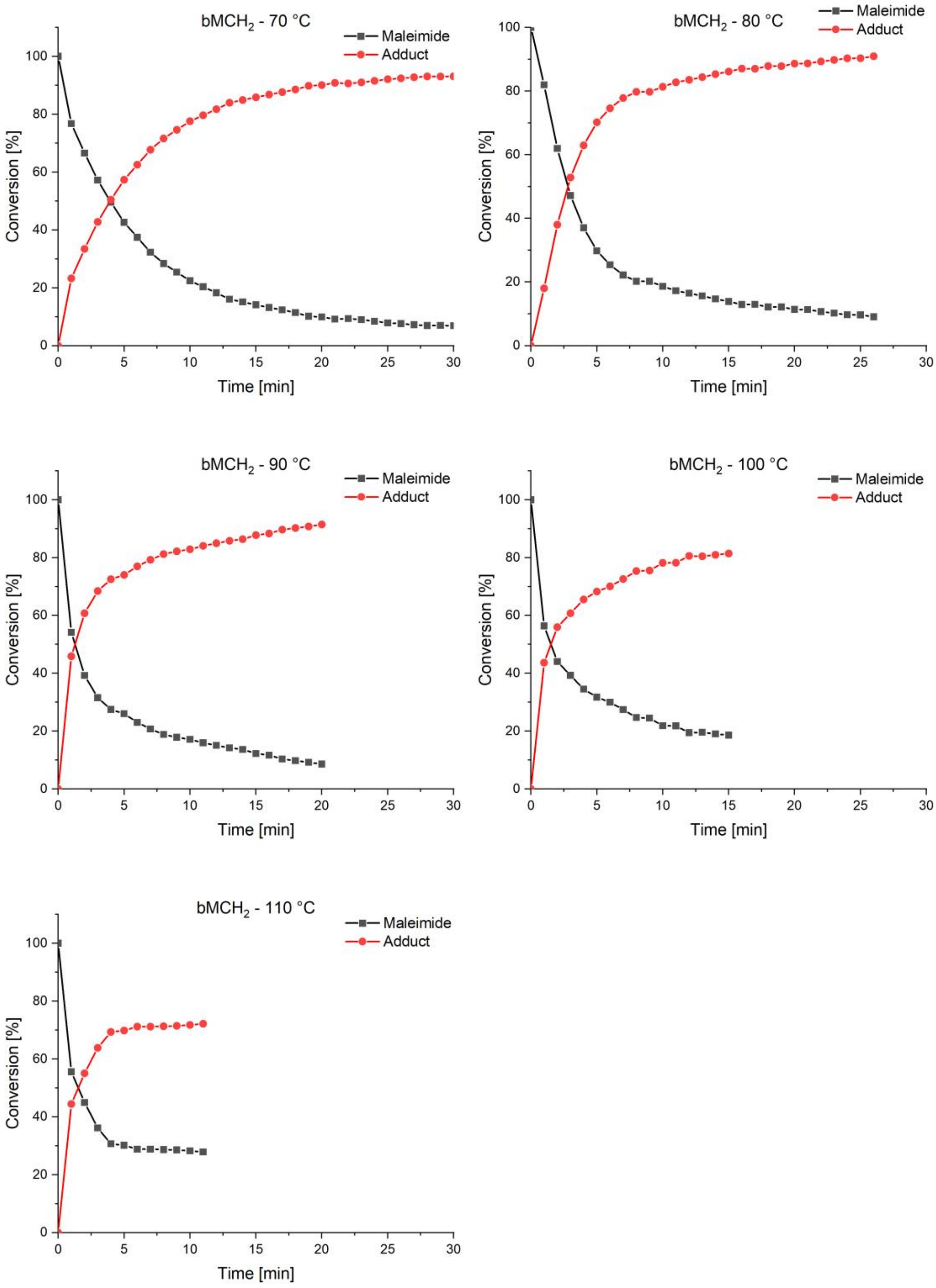
*Supplementary Figure S32 IR spectrum of **PPG-bis-furan**.*



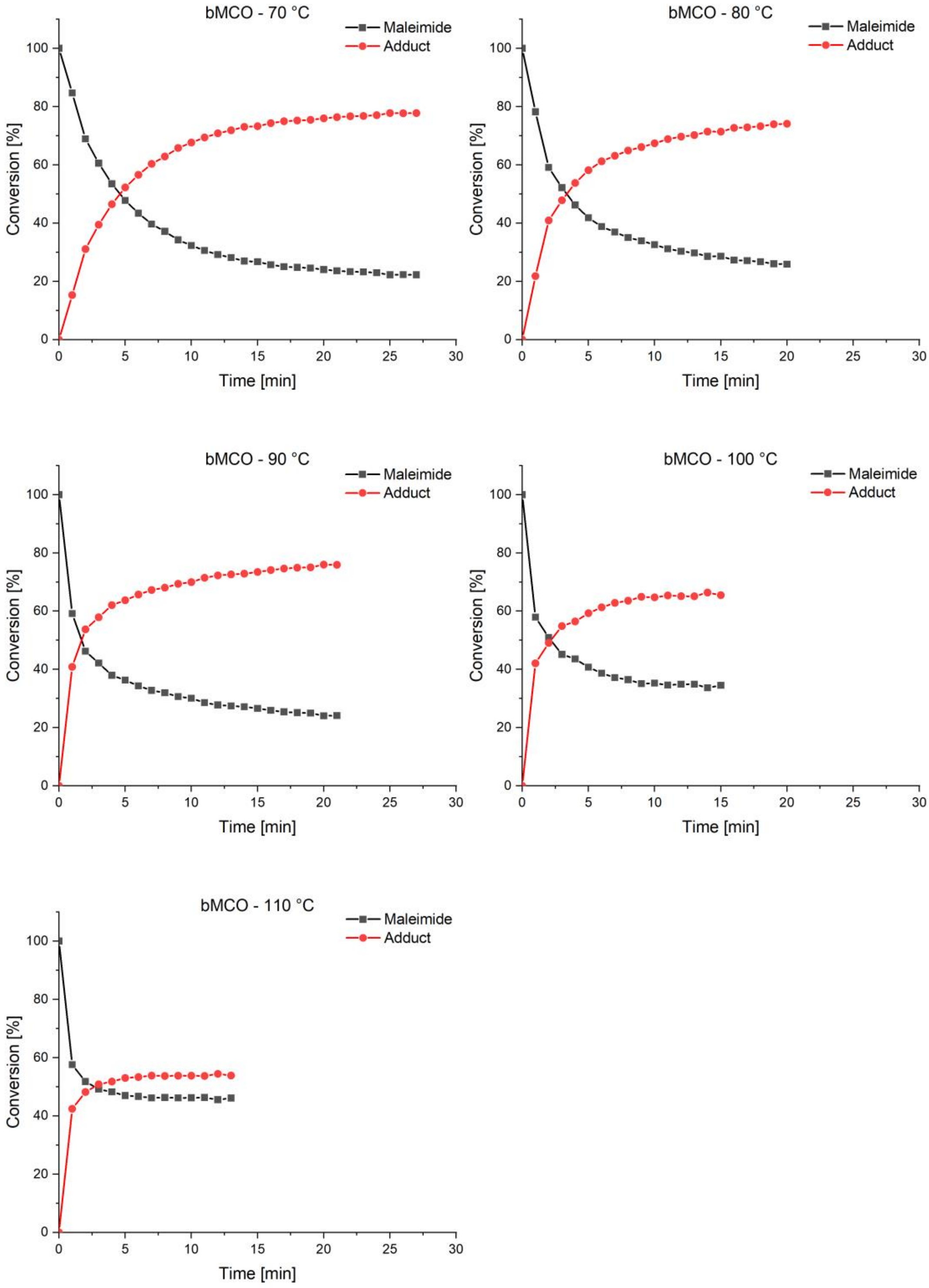
Supplementary Figure S33 Maleimide conversion as calculated from the 690 cm^{-1} peak area in a 10% crosslinked ***PPG-furan*** *network.*



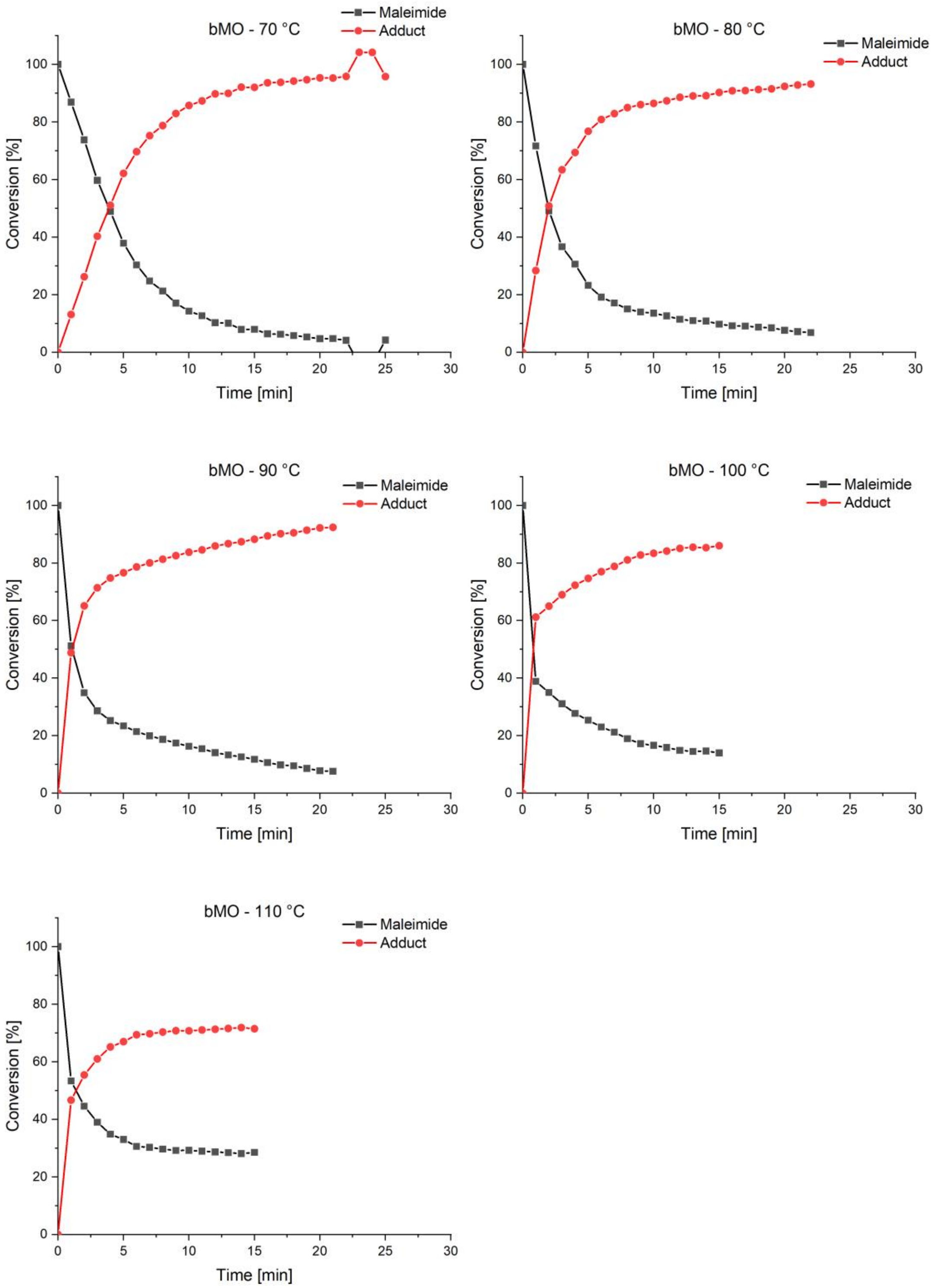
Supplementary Figure S34 IR spectrum of the four-armed furan linker ***FA4****.*



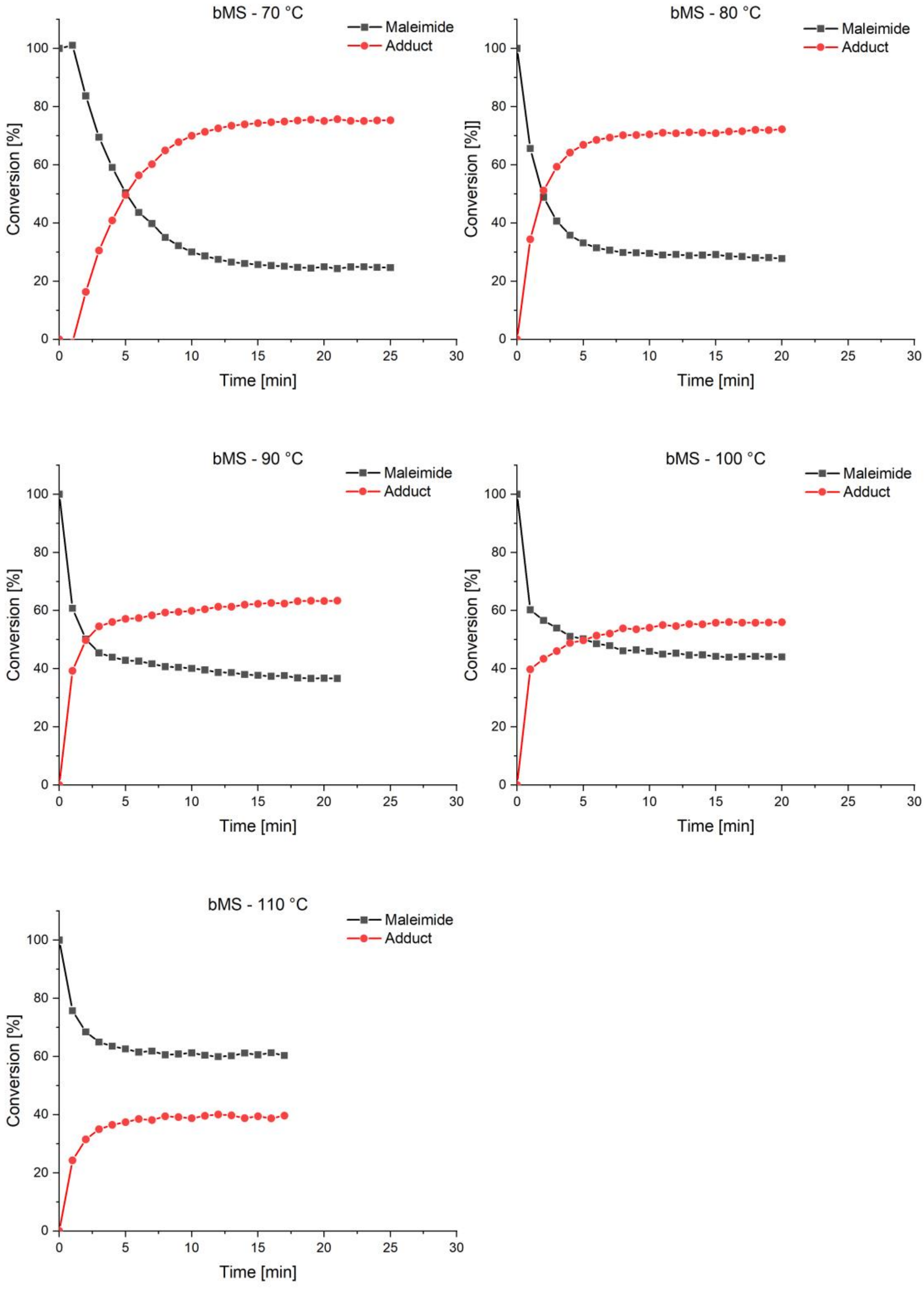
Supplementary Figure S35 IR conversion data at different temperatures of the ***FA4*** *network with the* ***bMCH$_2$*** *linker.*



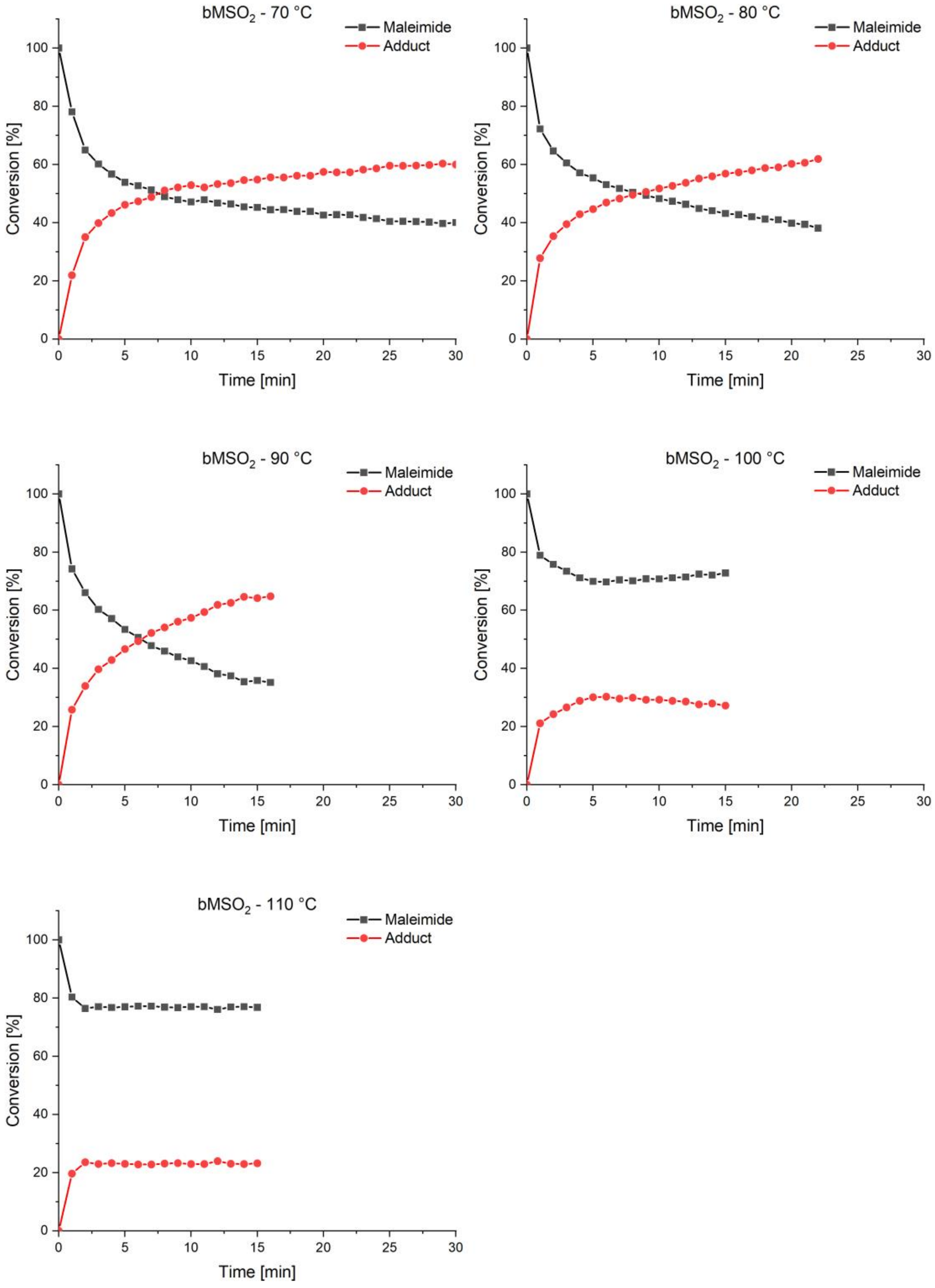
Supplementary Figure S36 IR conversion data at different temperatures of the ***FA4*** *network with the* ***bMCO*** *linker.*



*Supplementary Figure S37 IR conversion data at different temperatures of the **FA4** network with the **bMO** linker.*

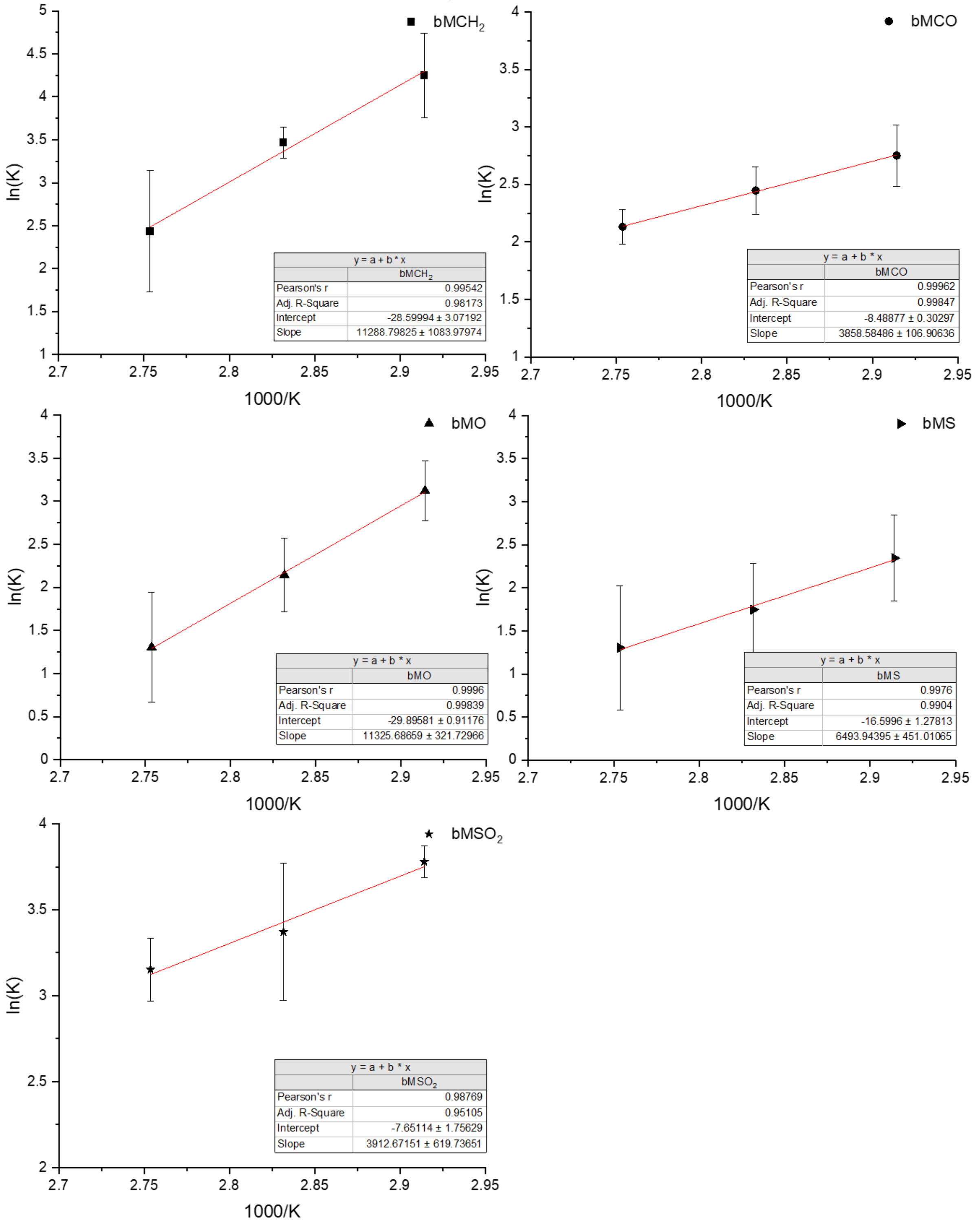


*Supplementary Figure S38 IR conversion data at different temperatures of the **FA4** network with the **bMS** linker.*



Supplementary Figure 39 IR conversion data at different temperatures of the ***FA4*** *network with the* ***bMSO$_2$*** *linker.*

7. Van ‘t Hoff plots of **FA3-alkyl** networks



y = a + b * x	
	bMCH$_2$
Pearson's r	0.99542
Adj. R-Square	0.98173
Intercept	-28.59994 ± 3.07192
Slope	11288.79825 ± 1083.97974

y = a + b * x	
	bMCO
Pearson's r	0.99962
Adj. R-Square	0.99847
Intercept	-8.48877 ± 0.30297
Slope	3858.58486 ± 106.90636

y = a + b * x	
	bMO
Pearson's r	0.9996
Adj. R-Square	0.99839
Intercept	-29.89581 ± 0.91176
Slope	11325.68659 ± 321.72966

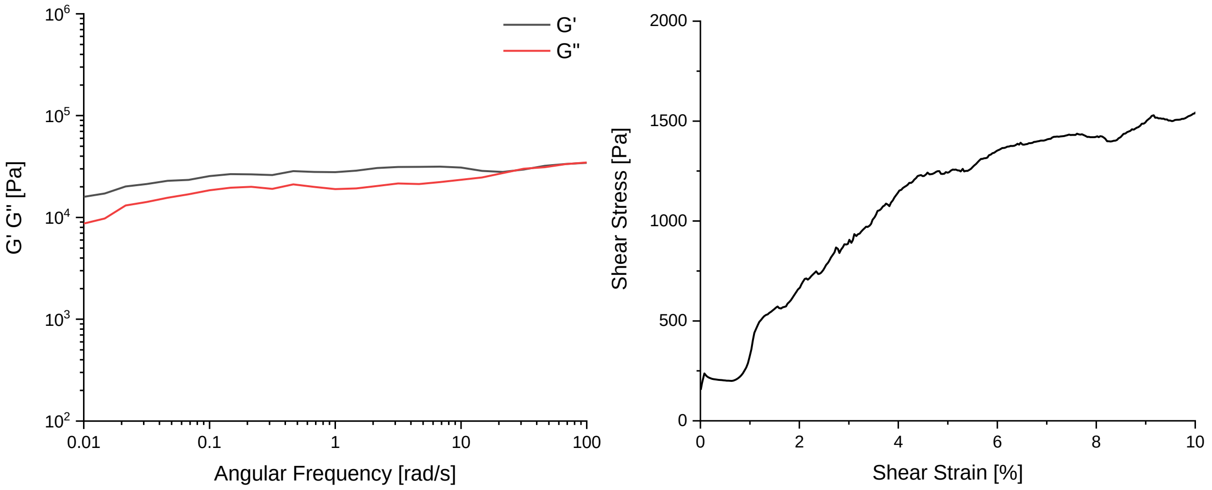
y = a + b * x	
	bMS
Pearson's r	0.9976
Adj. R-Square	0.9904
Intercept	-16.5996 ± 1.27813
Slope	6493.94395 ± 451.01065

y = a + b * x	
	bMSO$_2$
Pearson's r	0.98769
Adj. R-Square	0.95105
Intercept	-7.65114 ± 1.75629
Slope	3912.67151 ± 619.73651

*Supplementary Figure S40 Van ‘t Hoff plots of FA3-alkyl networks made using bMX linkers (n=3 for **bMCH$_2$, bMO, bMS** and **bMSO$_2$**; n=2 for **bMCO**).*

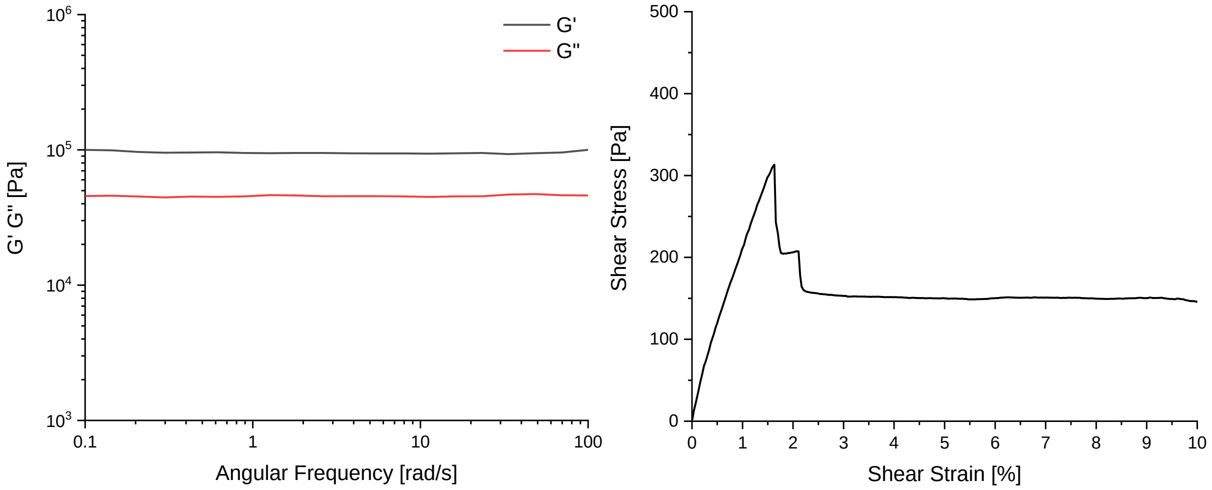
8. Rheology data

8.1. **FA3-alkyl** networks

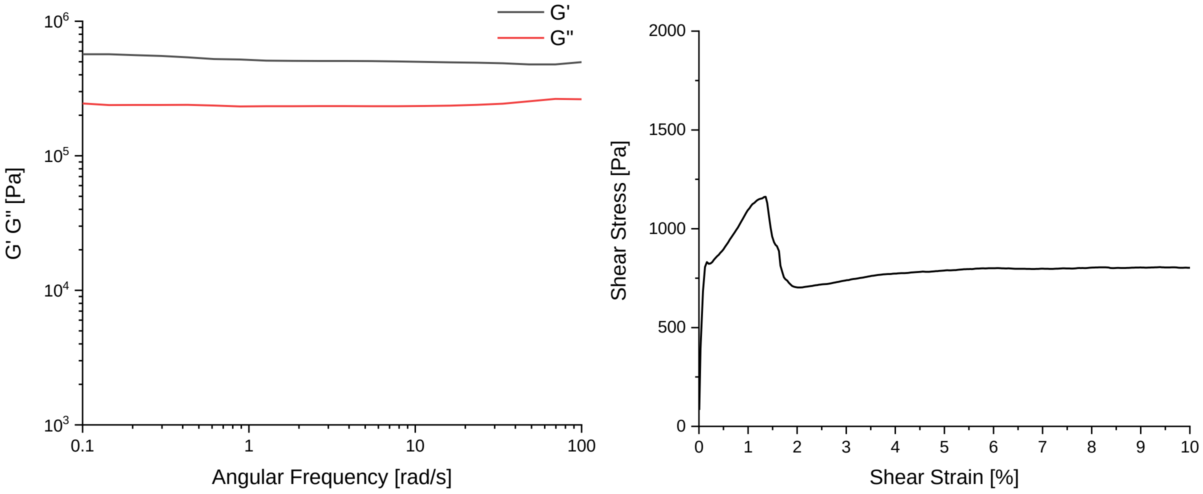


*Supplementary Figure S41 Frequency sweep of a **FA3-alkyl** network with a **bMS** linker at 100 °C and a strain of 0.025% (left) and a shear experiment of a **FA3-alkyl** network with a **bMS** linker at 100 °C (right).*

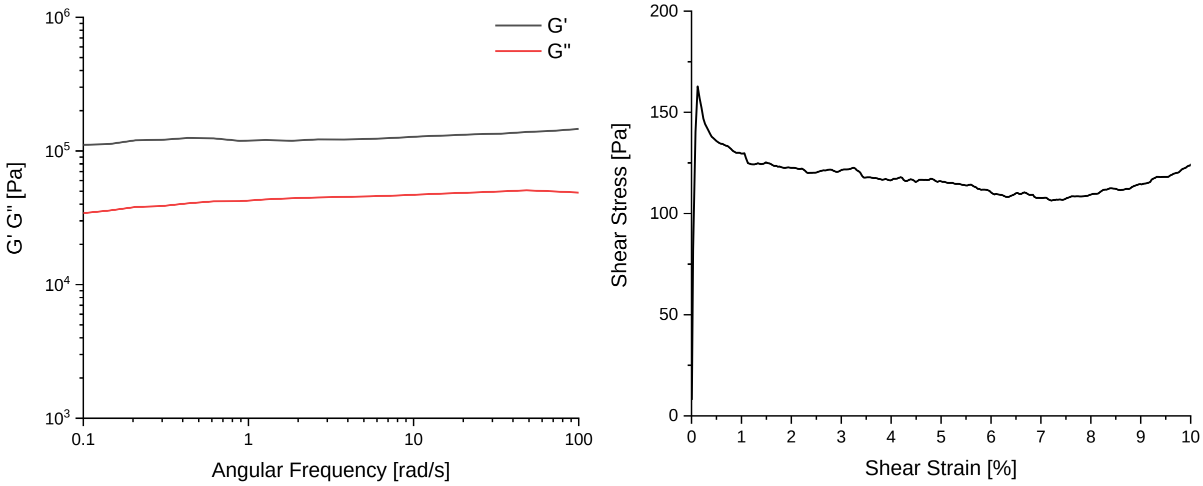
8.2. **FA3-PPG** networks



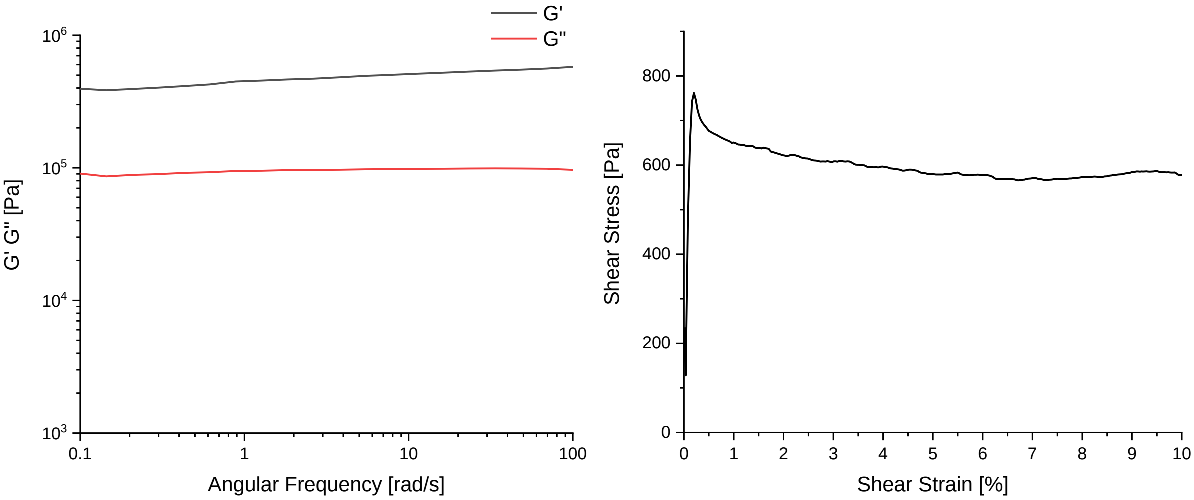
*Supplementary Figure S42 Frequency sweep of a **FA3-PPG** network with a **bMCH$_2$** linker at 70 °C and 0.5% strain (left). A shear experiment of a **FA3-PPG** network with a **bMCH$_2$** linker at 100 °C (right). This sample was crosslinked with 50% of the furans coming from the crosslinker and 50% from the linear furan linker.*



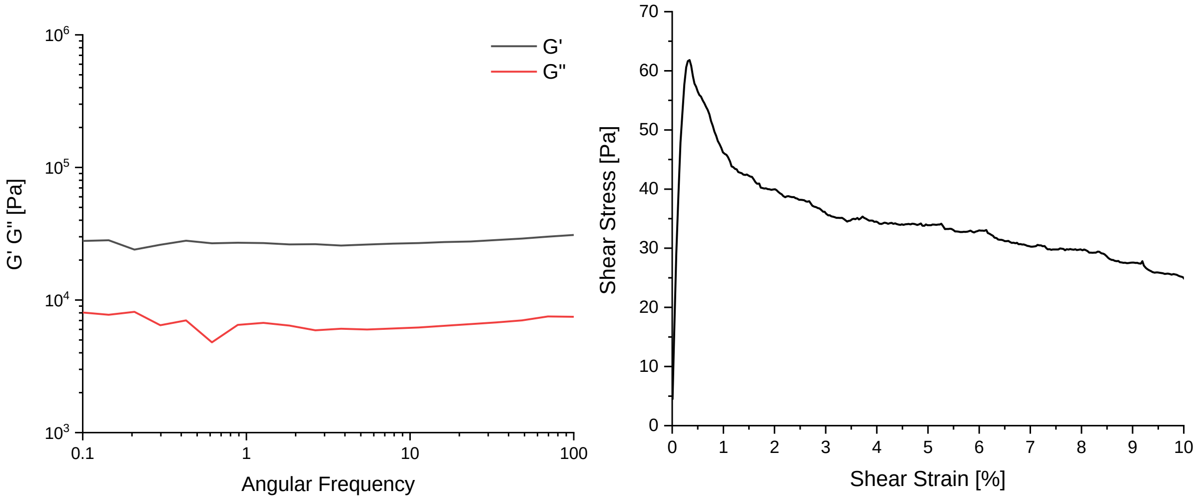
Supplementary Figure S43 Frequency sweep of a ***FA3-PPG*** *network with a* ***bMCH₂*** *linker at 70 °C and 0.5% strain (left). A shear experiment of a* ***FA3-PPG*** *network with a* ***bMCH₂*** *linker at 100 °C (right). This sample was crosslinked with 25% of the furans coming from the crosslinker and 75% from the linear furan linker.*



Supplementary Figure S44 Frequency sweep of a ***FA3-PPG*** *network with a* ***bMCH₂*** *linker at 70 °C and 0.5% strain (left). A shear experiment of a* ***FA3-PPG*** *network with a* ***bMCH₂*** *linker at 70 °C (right). This sample was crosslinked with 10% of the furans coming from the crosslinker and 90% from the linear furan linker.*

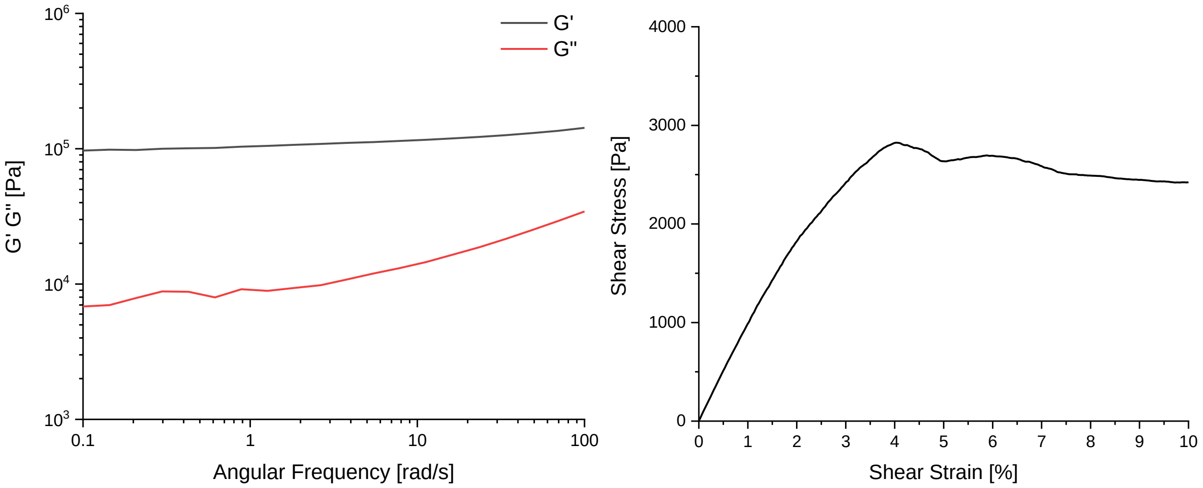


Supplementary Figure S45 Frequency sweep of a ***FA3-PPG*** *network with a* ***bMCH$_2$*** *linker at 70 °C and 0.5% strain (left). Shear experiment of a* ***FA3-PPG*** *network with a* ***bMCH$_2$*** *linker at 70 °C (right). This sample was crosslinked with 5% of the furans coming from the crosslinker and 99% from the linear furan linker.*

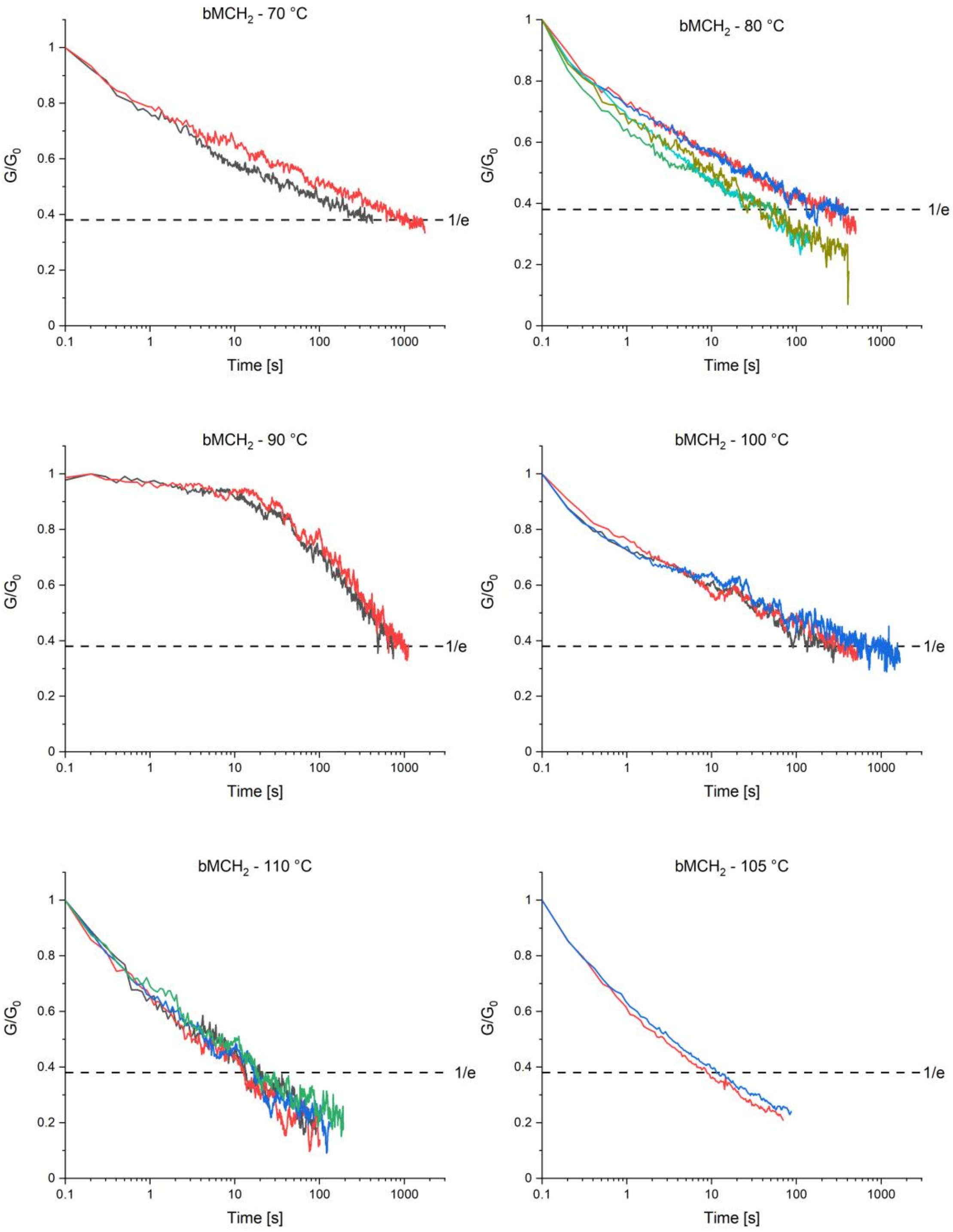


Supplementary Figure S46 Frequency sweep of a ***FA3-PPG*** *network with a* ***bMCH$_2$*** *linker at 70 °C and 0.5% strain (left). A shear experiment of a* ***FA3-PPG*** *network with a* ***bMCH$_2$*** *linker at 70 °C (right). This sample was crosslinked with 1% of the furans coming from the crosslinker and 99% from the linear furan linker.*

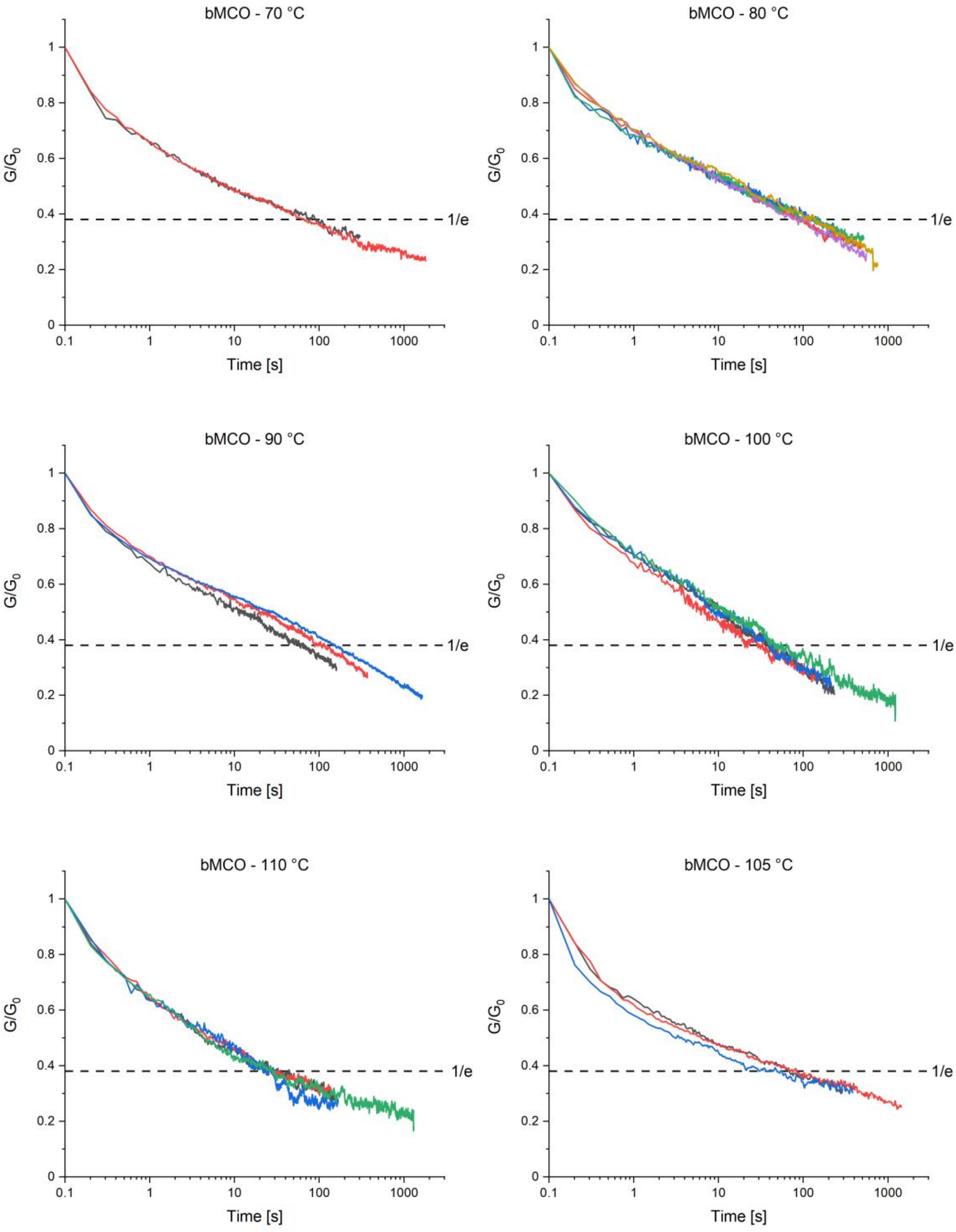
8.3. **FA4** networks



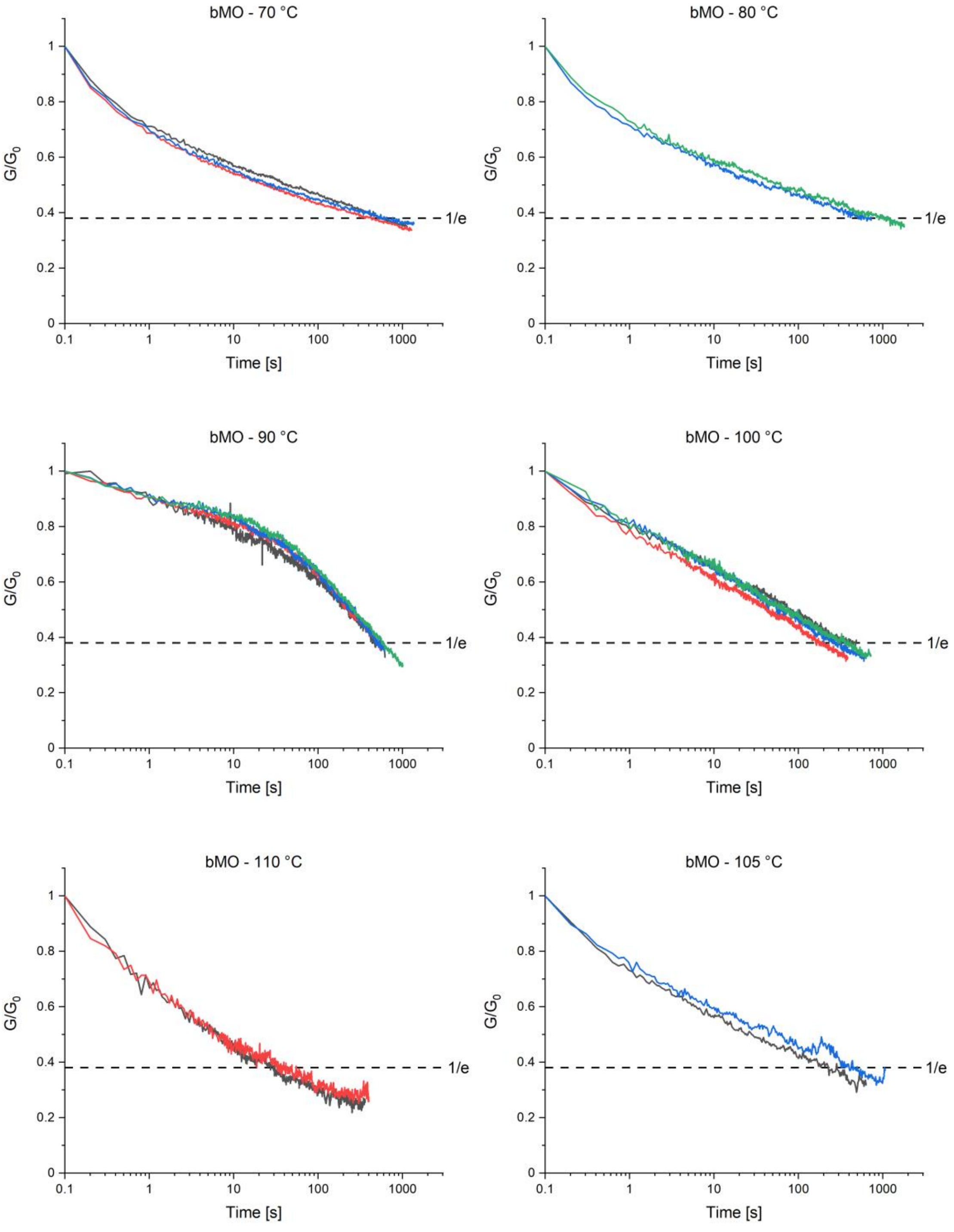
Supplementary Figure S47 Frequency sweep of a ***FA4*** *network with a* ***bMCH$_2$*** *linker at 70 °C and 0.1% strain (left). A shear experiment of a* ***FA4*** *network with a* ***bMCH$_2$*** *linker at 70 °C (right).*



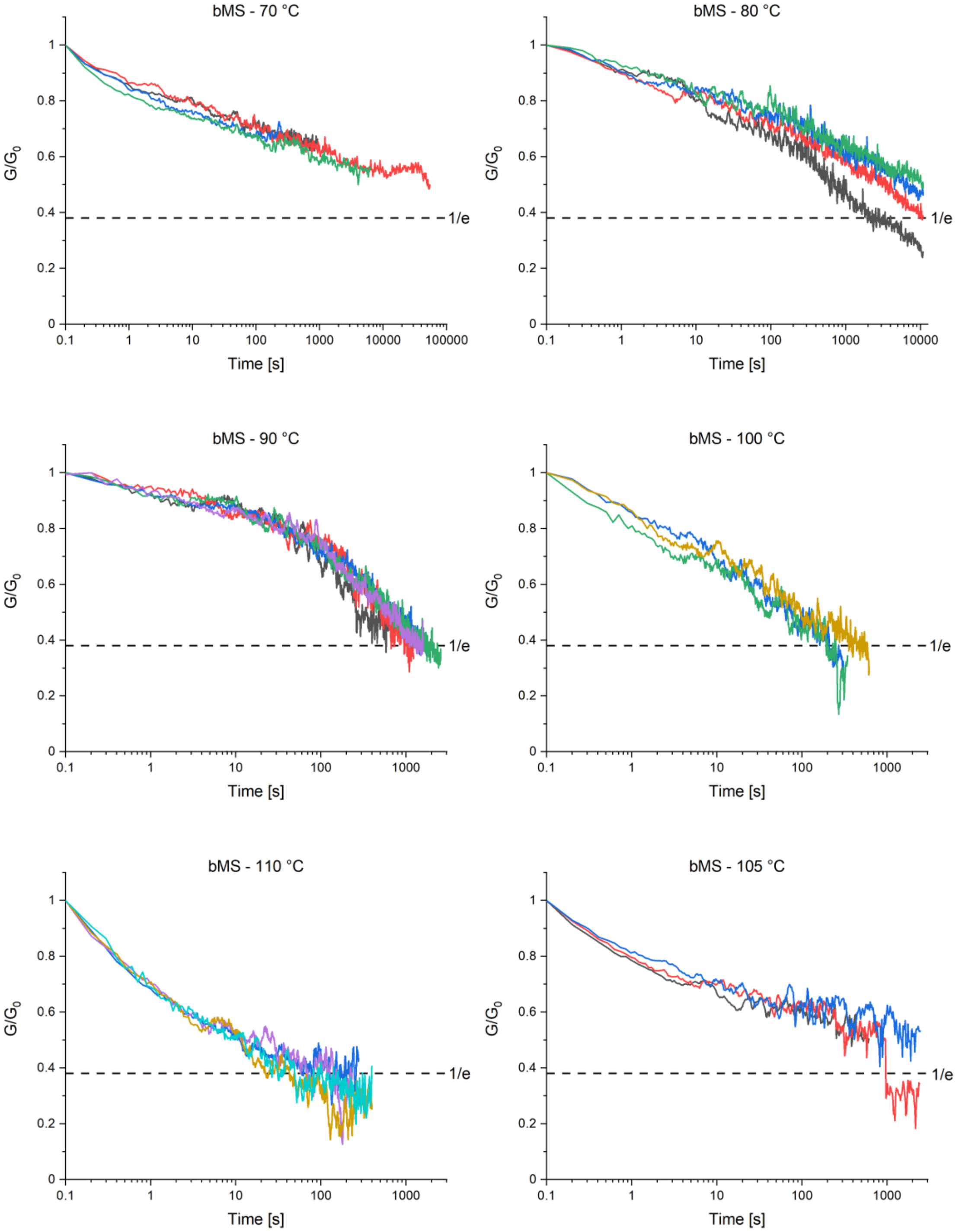
*Supplementary Figure S48 Relaxation data for a **FA4 bMCH₂** network at different temperatures with 0.1% strain. The multiple curves per subfigure indicate repeat experiments.*



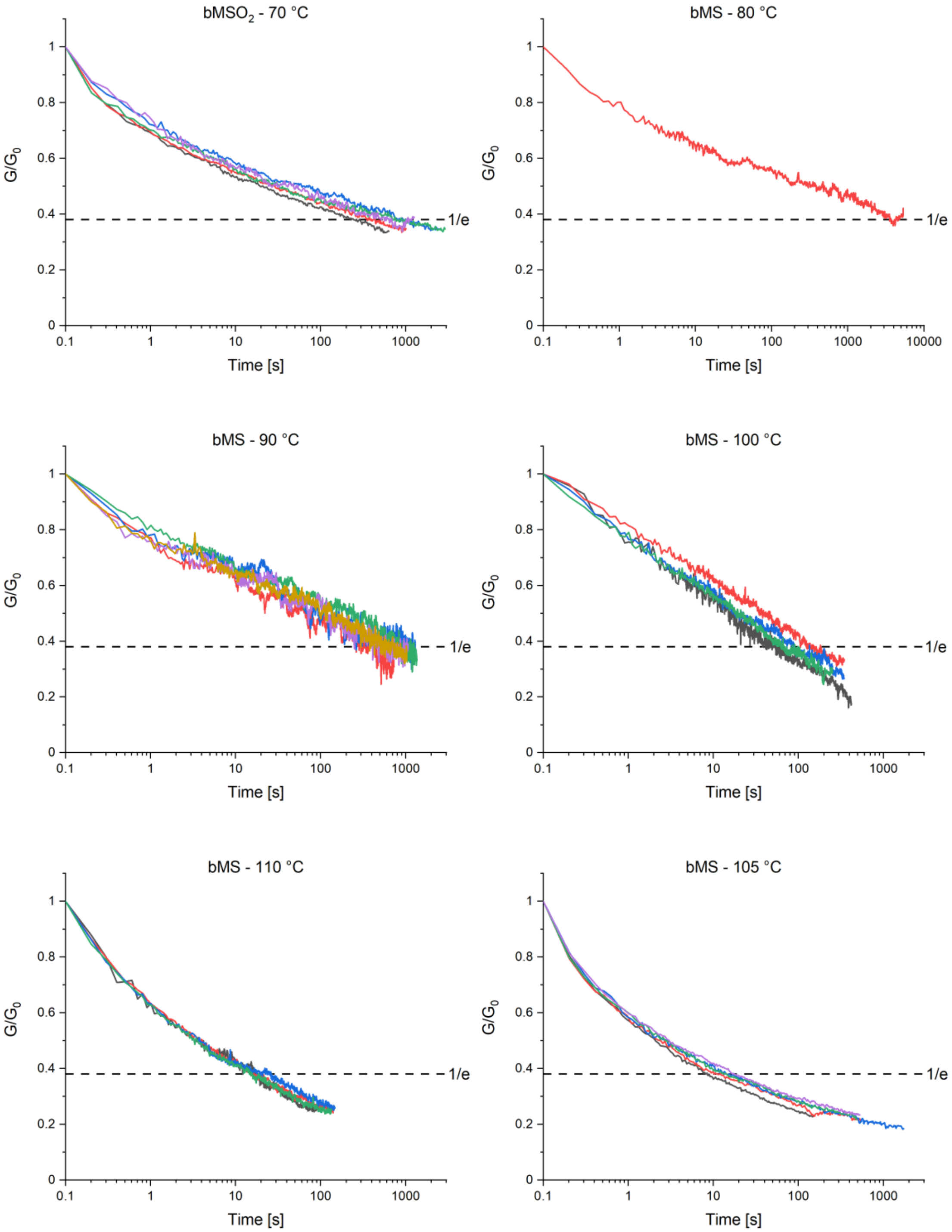
Supplementary Figure S49 Relaxation data for a ***FA4 bMCO*** *network at different temperatures with 0.1% strain. The multiple curves per subfigure indicate repeat experiments.*



Supplementary Figure S50 Relaxation data for a ***FA4 bMO*** *network at different temperatures with 0.1% strain. The multiple curves per subfigure indicate repeat experiments.*



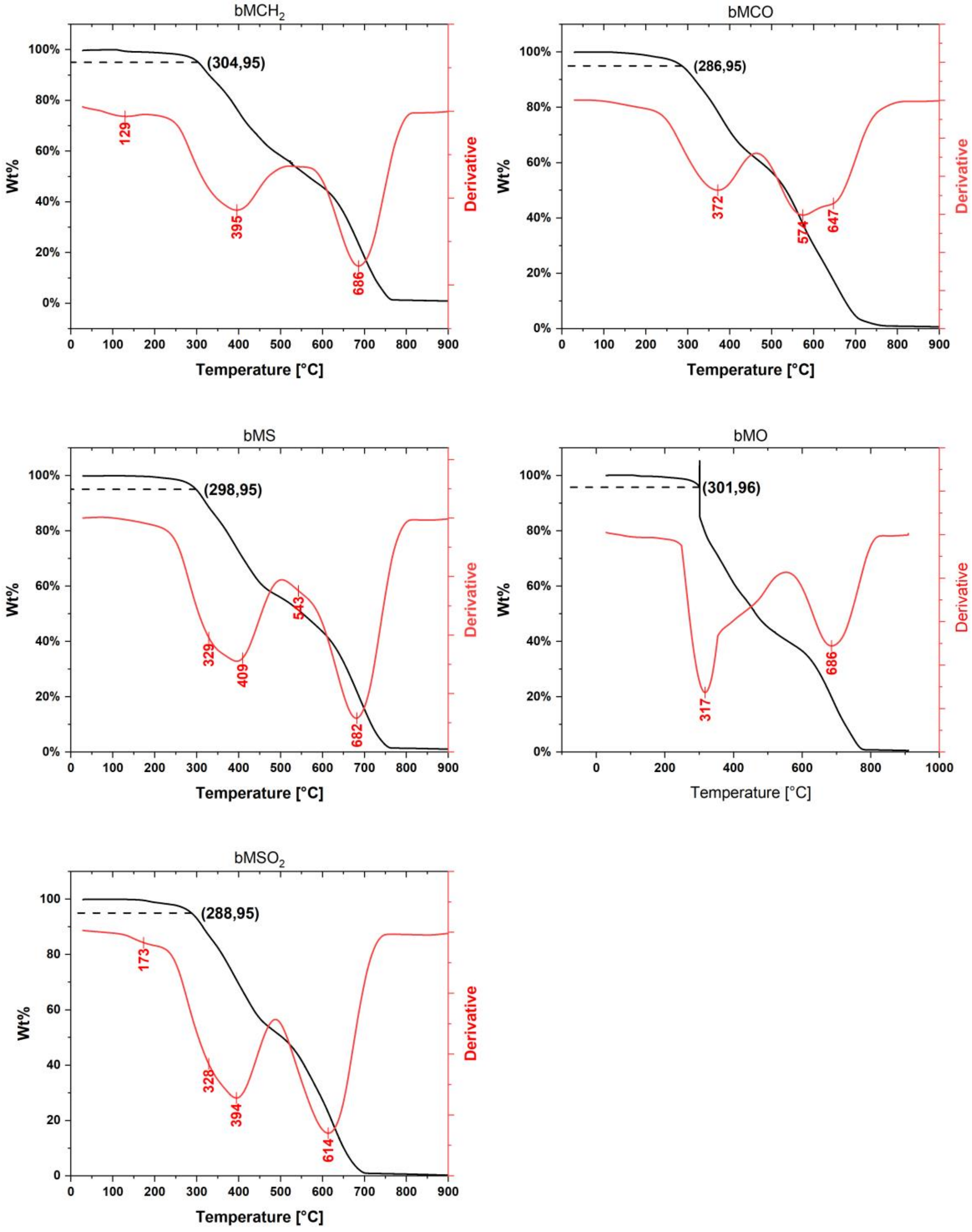
Supplementary Figure S51 Relaxation data for a ***FA4 bMS*** *network at different temperatures with 0.1% strain. The multiple curves per subfigure indicate repeat experiments.*



*Supplementary Figure S52 Relaxation data for a **FA4 bMSO$_2$** network at different temperatures with 0.1% strain. The multiple curves per subfigure indicate repeat experiments.*

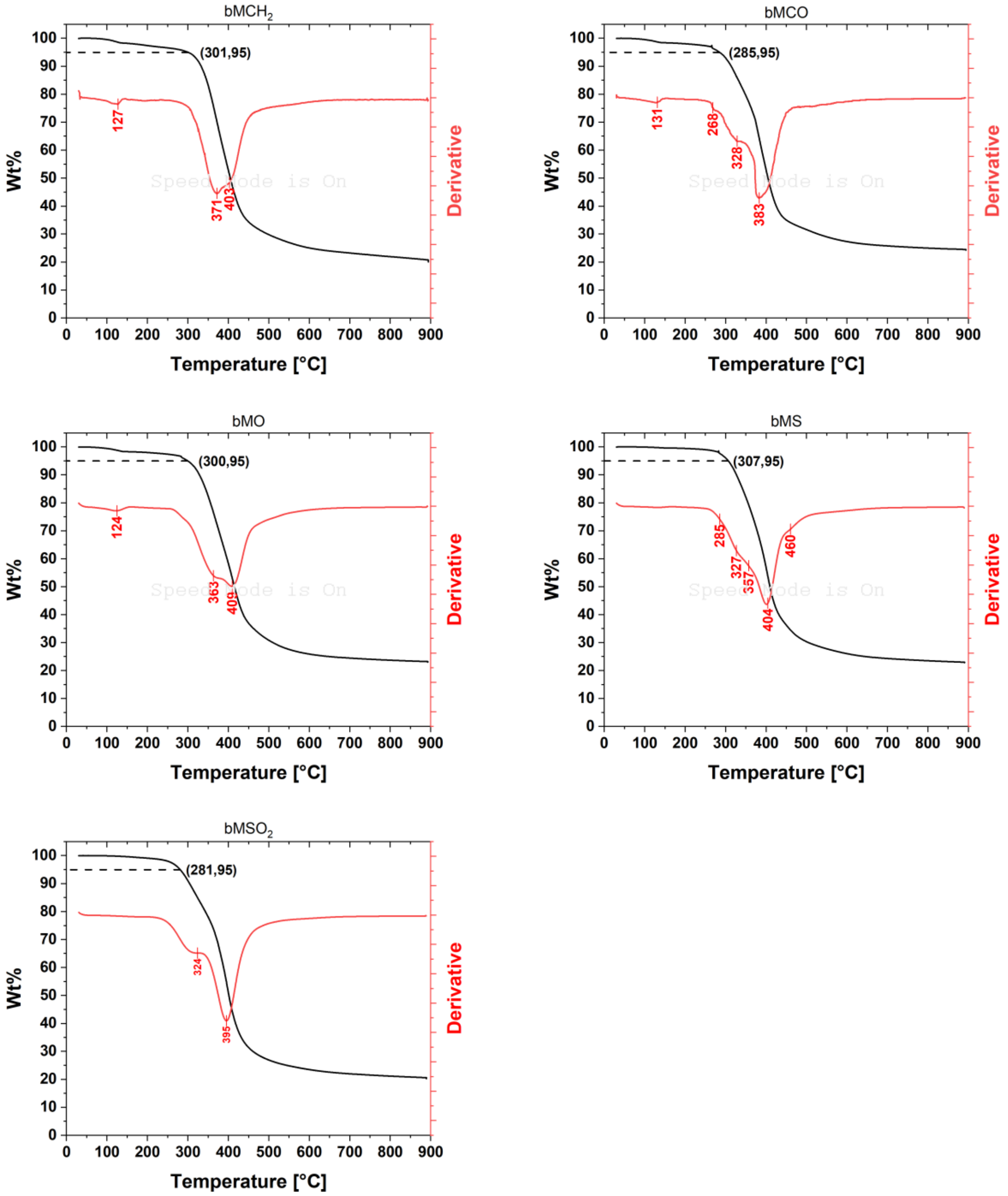
9. TGA data

9.1. **FA3-alkyl** network



*Supplementary Figure S53 TGA data under synthetic air atmosphere of **FA3-alkyl** networks.*

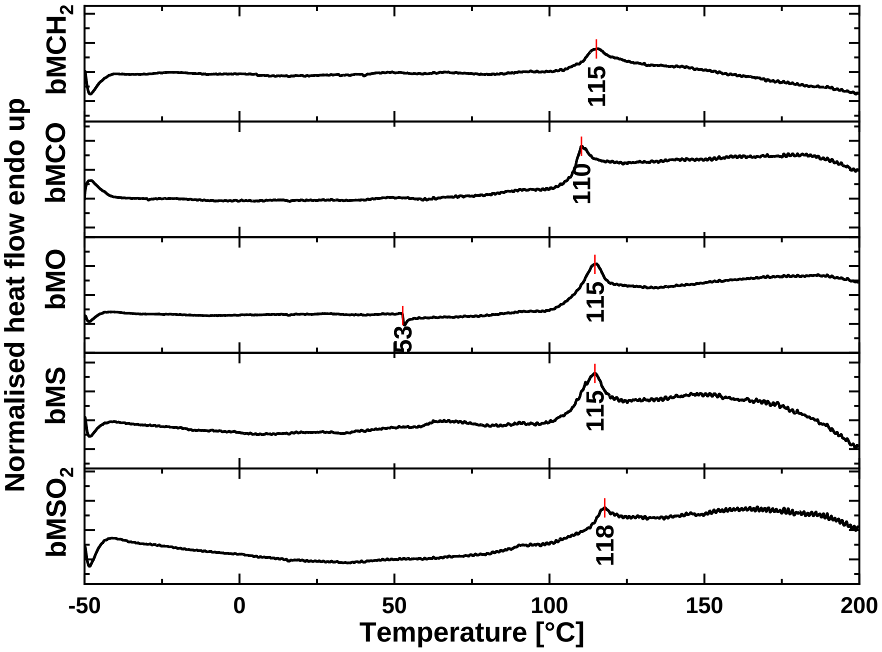
9.2. **FA4** network



*Supplementary Figure S54 TGA data under N_2 atmosphere of **FA4** networks.*

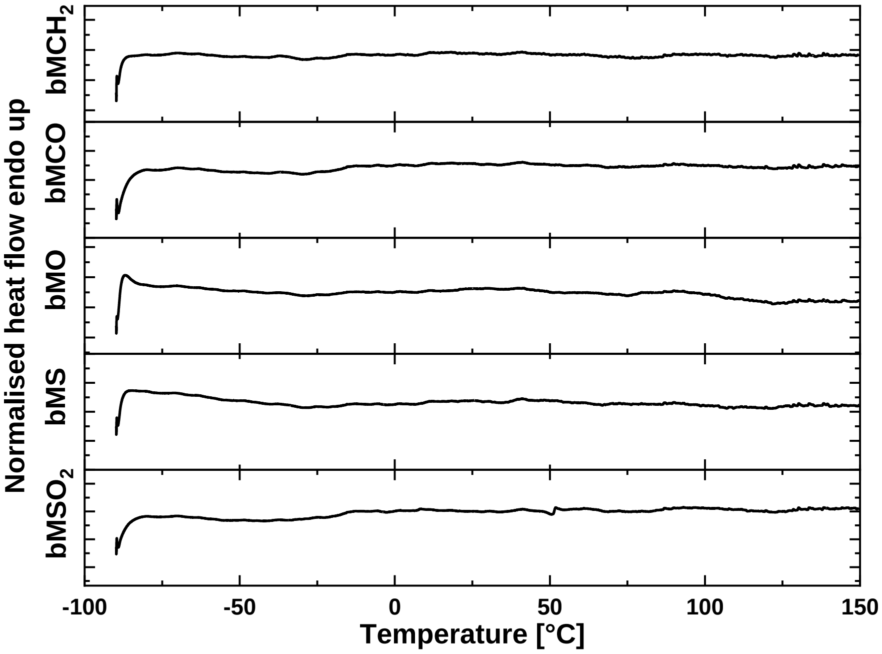
10. DSC data

10.1. **FA3-alkyl** networks



Supplementary Figure S55 Normalised second heat run of the ***FA3-alkyl*** *networks.*

10.2. **FA4** networks



Supplementary Figure S56 Normalised second heat run of the ***FA4*** *networks.*

11. References

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