

Hydrophobic deep eutectic solvents as water-immiscible extractants

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Table of contents	
Materials, preparation and liquid-liquid extraction method	ESI1
Analytical Methodologies	ESI2
IC results	ESI3
NMR spectra	ESI4
Densities and viscosities	ESI5
TGA thermograms	ESI6
DSC thermograms	ESI7
FTIR spectra	ESI8

ESI1: Materials, preparation and extraction method

Materials for the preparation

Tetrabutylammonium chloride (N₄₄₄₄-Cl) was purchased from Merck with a purity \geq 95 %, tetraheptylammonium chloride (N₇₇₇₇-Cl) was obtained from Acros Organics with a 95% purity and methyltrioctylammonium chloride (N₈₈₈₁-Cl), tetraoctylammonium chloride (N₈₈₈₈-Cl), methyltrioctylammonium bromide (N₈₈₈₁-Br) and tetraoctylammonium bromide (N₈₈₈₈-Br) were acquired from Sigma-Aldrich with purities of 97%, 97%, 97 % and 98%. Decanoic acid was purchased from Sigma-Aldrich with a purity higher than 98%.

Materials for the mixing with water and the extraction

Propionic acid (\geq 99.5%) and butyric acid (\geq 99%) were acquired from Sigma-Aldrich. Acetic acid (glacial, 100%) was obtained from Merck. These chemicals were used as provided. MilliQ water was used for the preparation of the solutions and for the mixing of the DESs with water, which was obtained from a Millipore Milli-Q[®] biocel, which used a Q-grade[®] column.

Preparation

The 6 deep eutectic solvents (DESs) were all prepared following the same experimental method. The hydrogen bond donor (HBD), decanoic acid, was first weighted in a flask. The hydrogen bond acceptors (HBAs), the quaternary ammonium salts, were first weighted in a weighing boat. The amount of HBA was mixed with the amount of HBD in the flask. The flask was then heated in an oil bath at a temperature of 35 °C until it was a clear liquid. The wall of the flask was rinsed with the DES inside the flask to ensure that all the solid was in the liquid phase. After the rinsing the flask was heated up again to 35 °C until again a clear liquid was obtained. Afterwards, the DES was maintained at room temperature (295 K) overnight.

Mixing with water

All the DESs were mixed with water to investigate the amount of water they would uptake. Centrifuge tubes (CELLSTAR[®]) of 50 mL were used, in which approximately 6 grams of water and 6 gram of DES was added. The mixture was vigorously stirred with a vortex mixer (VWR, analog Vortex Mixer) and afterwards placed in the centrifuge (Sigma 2-16 KL) for 30 min at 12.000 rpm. The phases were separated and further analyzed. From the top phase (DES-rich phase) the water content was determined using Karl Fischer titration and the bottom phase (water-rich phase) was analyzed with ion chromatography (IC).

Liquid-liquid extraction method

Liquid-liquid extractions were conducted using the prepared hydrophobic DESs as extractants at room temperature (295 K). Solutions of 1 wt% VFA in water were prepared, with a pH of 3.3. In centrifuge tubes, 2 mL aliquots of VFA solutions were mixed with 2 mL of hydrophobic DES. The mixtures were initially mixed for short time using a vortex and later for 2 hours, at room temperature, using an incubating shaker (IKA KS 4000 I). The samples were centrifuged (Sigma 2-16 KL) for 30 min at 3500 rpm. Thereafter, the DES-rich phase and water-rich phase were taken using a needled syringe. The concentration of the VFAs in the aqueous phase was determined by analysis of 1 mL samples using a

high performance liquid chromatography (HPLC). The same experimental methodology was used for the extraction experiments using trioctylamine.

ESI2: Analytical Methodologies

Karl Fischer

The water content in the prepared DESs was measured with the Karl Fischer titration method (795 KFT Titrino Metrohm Karl Fischer).

Anton-Paar

Density, ρ , and dynamic viscosity, η , measurements were conducted using an Anton Paar SVM 3000 Stabinger Viscometer. The measurements were performed in the temperature range of (288.15 - 323.15) K at atmospheric pressure. The temperature uncertainty in the temperature range of the equipment (288.15 to 378.15 K) is ± 0.02 K and the relative uncertainty of the dynamic viscosity is ± 0.35 %, while the absolute uncertainty in the density is $0.5 \text{ kg}\cdot\text{m}^{-3}$.

TGA

The decomposition temperatures of all the studied hydrophobic DESs were determined using a thermogravimetric analyzer (Perkin Elmer TGA 4000). The samples were heated in a ceramic crucible under a continuous nitrogen flow ($20 \text{ mL}\cdot\text{min}^{-1}$) and a gas pressure of 0.2 MPa. The thermal stability was determined by scanning in the temperature range of (303 – 673) K at heating rate of $5 \text{ K}\cdot\text{min}^{-1}$. The sample masses were typically between 15 and 30 mg and the weighing precision and sensitivity of the balance are $\pm 0.01\%$ and $1 \text{ }\mu\text{g}$, respectively.

DSC

The melting point and solid-solid transition temperatures of the prepared DESs were studied using differential scanning calorimetry (TA instruments, DSC Q100). The operating temperature range of the DSC is 123 to 873 K and the scanning rates range from 0.01 to $500 \text{ K}\cdot\text{min}^{-1}$. The measurements were conducted within a temperature range of 193 to 323 K at a heating rate of $5 \text{ K}\cdot\text{min}^{-1}$. The temperature measurement is accurate within ± 0.1 K. The calorimeter precision and sensitivity are $\pm 0.1\%$ and $0.2 \text{ }\mu\text{W}$, respectively.

NMR

Proton Nuclear Magnetic Resonance (^1H -NMR) spectra were recorded at $25 \text{ }^\circ\text{C}$ on a 400 MHz Bruker BZH 400/52. All samples were diluted with deuterated dimethyl sulfoxide (DMSO-d_6) without further pretreatment inside 8" Wilmad precision tubes with an outer diameter of 5 mm. A relaxation time of 3 s was applied and the spectra were recorded in 16 scans. The signals were automatically Fourier transformed with recording software VnmrJ. Further adjustments were performed with software package MestreNova, version 9.1.0-14011. Phase corrections and integrals were done manually, baselines were corrected with the Bernstein polynomial (6 parameters).

FTIR

All Fourier transformed infrared (FTIR) spectra were recorded on a PerkinElmer Spectrum Two spectrometer equipped with a Spectrum Two UATR (universal attenuated total reflectance) module. All samples were scanned over a wavenumber range of 450-4000 cm^{-1} . The spectra of the DESs were recorded in 16 scans, for the pure components 4 scans were sufficient. Prior to each measurement, the quality of the background signal was evaluated and a background spectrum was recorded using the same settings as for the sample measurement if necessary (residual peaks after cleaning > 0.2 % transmittance). For liquids, the crystal of the UATR module was completely covered with a small drop of the sample. Solids were pressed on the crystal until a maximum signal of at least 50 % transmittance was obtained. The spectra presented in ESI8 were submitted to an automatic baseline correction performed with PerkinElmer Spectrum software, Application version 10.03.08.0135, 2012.

HPLC

The aqueous phases after extraction were analyzed using an Agilent Technologies 1260 Infinity HPLC system, with a 300 mm x 7.8 mm Aminex HPX-87H column, using a UV detector operating at a wavelength of 210 nm. The injection volume was 10 μL , the temperature of the column was kept at 50 $^{\circ}\text{C}$ and 5mM aqueous sulphuric acid was used as eluent with a flow rate of 0.6 $\text{mL}\cdot\text{min}^{-1}$.

IC

The chloride and bromide concentrations were measured with a A Metrohm 761 Compact IC and 762 IC interface with a detection limit of 0.1 $\text{mg}\cdot\text{L}^{-1}$.

ESI3: IC results

Table S1: Results obtained from IC analysis

DESS	C_{anion} [mg·L ⁻¹]	C_{anion} [g·L ⁻¹]	C_{anion} [mol·L ⁻¹]	M_{salt} [g·mol ⁻¹]	C_{salt} [g·L ⁻¹]	C_{salt} [mg·g ⁻¹]	$m_{salt,leached}/$ $m_{salt,DES}$ [g·g ⁻¹]
DecA:N ₄₄₄₄ -Cl (2:1)	19800	19.80	0.558	277.92	155.21	155.21	0.3475
DecA:N ₈₈₈₁ -Cl (2:1)	1420	1.42	0.040	404.16	16.19	16.19	0.0300
DecA:N ₇₇₇₇ -Cl (2:1)	1040	1.04	0.029	446.24	13.09	13.09	0.0232
DecA:N ₈₈₈₈ -Cl (2:1)	807	0.80	0.023	502.34	11.43	11.43	0.0193
DecA:N ₈₈₈₁ -Br (2:1)	2330	2.33	0.029	448.61	13.08	13.08	0.0231
DecA:N ₈₈₈₈ -Br (2:1)	2080	2.08	0.026	446.79	14.23	32.08	0.0523

ESI4: NMR spectra

In this section, δ (for figures and tables respectively) are considered the chemical shift compared to internal reference tetramethylsilane (TMS). Peak shapes are reported as: *s*, singlet; *d*, doublet; *t*, triplet and *m*, multiplet. The structure formulas of each DES are drawn in the spectra. Each carbon atom is numbered and assigned to the appropriate peaks or peak groups, displayed in the first two columns of the tables and above the peaks in the spectra.

The degree of leaching of salt from the DES-rich phase to the water-rich phase after mixing can be derived from a decrease in the integral of the groups that can be assigned to the protons of the quaternary ammonium salt. This makes the $^1\text{H-NMR}$ spectra complementary to the previously presented ion chromatography (IC) results (ESI3). However, it should be noted that the accuracy of the integrals for specific peaks is not high enough to determine the leaching quantitatively. This is mainly caused by overlap of the peaks of decanoic acid (DecA) and the salt in the region of chemical shifts between 0 and 2 ppm and overlap of water and the quaternary ammonium salts in the 3-4 ppm region. Nevertheless, the spectra showed a clear qualitative similarity to the IC results, a significant loss of salt for the DecA: $\text{N}_{4444}\text{-Cl}$ 2:1 DES and much smaller losses for the salts with increased alkyl chain lengths. This is expressed by slight decreases of the integrals of the hydrocarbon chains in the salts, which are all normalized to the integral of the clear triplet of the CH_2 group next to the COOH of DecA that is set to 4 protons.

DecA:N₄₄₄₄-Cl (2:1)

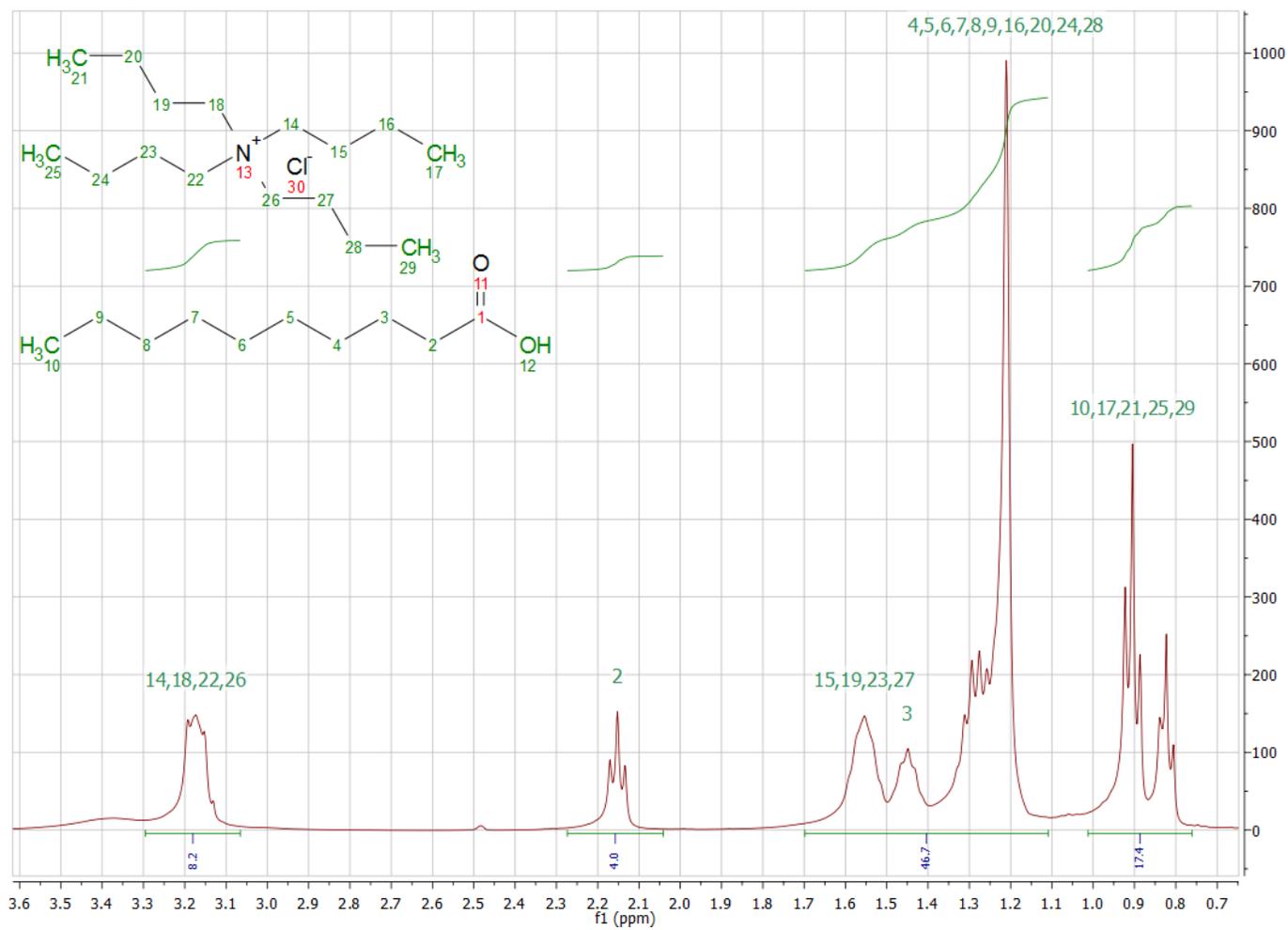


Figure S3: ¹H-NMR spectrum of DecA:N₄₄₄₄-Cl (2:1) before mixing with water.

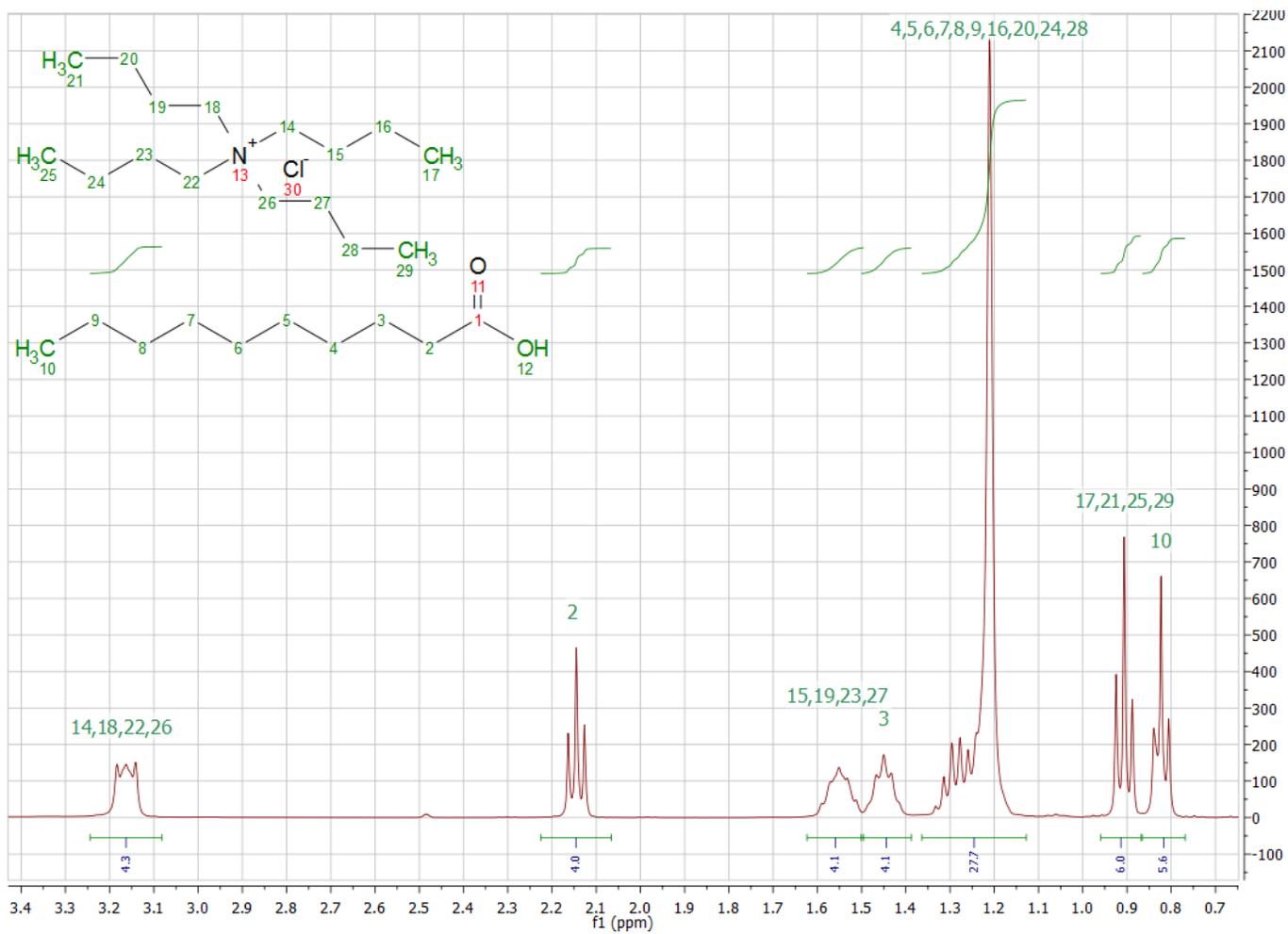


Figure S4: ¹H-NMR spectrum of DecA:N₄₄₄₄-Cl (2:1) after mixing with water.

Table S2: Peak assignments and integrals of the ¹H-NMR spectrum of DecA:N₄₄₄₄-Cl (2:1) before mixing with water

Groups		Before mixing with water			
DecA	N ₄₄₄₄ -Cl	Expected integral	δ (ppm)	Shape	Integral
2·C(10)H ₃	C(17,21,25,29)H ₃	18	0.76-1.01	m	17.4
2·C(3-9)H ₂	C(15,16,19,20,23,24,27,28)H ₂	44	1.11-1.70	m	46.7
2·C(2)H ₂	-	4	2.04-2.27	t	4.0
DMSO-d ₆	-	-	2.46-2.50	m	-
-	C(14,18,22,26)H ₂	8	3.11-3.21	m	8.2
H ₂ O, HDO	-	-	3.28-3.46	broad	-
C(28)OOH	-	2	11.75-12.7	broad	1.5

Table S3: Peak assignments and integrals of the ¹H-NMR spectrum of DecA:N₄₄₄₄-Cl (2:1) after mixing with water

Groups		After mixing with water		
DecA	N ₄₄₄₄ -Cl	δ (ppm)	Shape	Integral
2·C(10)H ₃	-	0.77-0.87	t	5.7
-	C(17,21,25,29)H ₃	0.87-0.96	t	6.1
2·C(4-9)H ₂	C(16,20,24,28)H ₂	1.13-1.36	m	28.1
2·C(3)H ₂	-	1.39-1.50		4.1
-	C(15,19,23,27)H ₂	1.50-1.62		4.2
2·C(2)H ₂	-	2.07-2.23	t	4.0
DMSO-d ₆	-	2.46-2.51	m	-
-	C(14,18,22,26)H ₂	3.08-3.24	m	4.3

DecA:N₈₈₈₁-Cl (2:1)

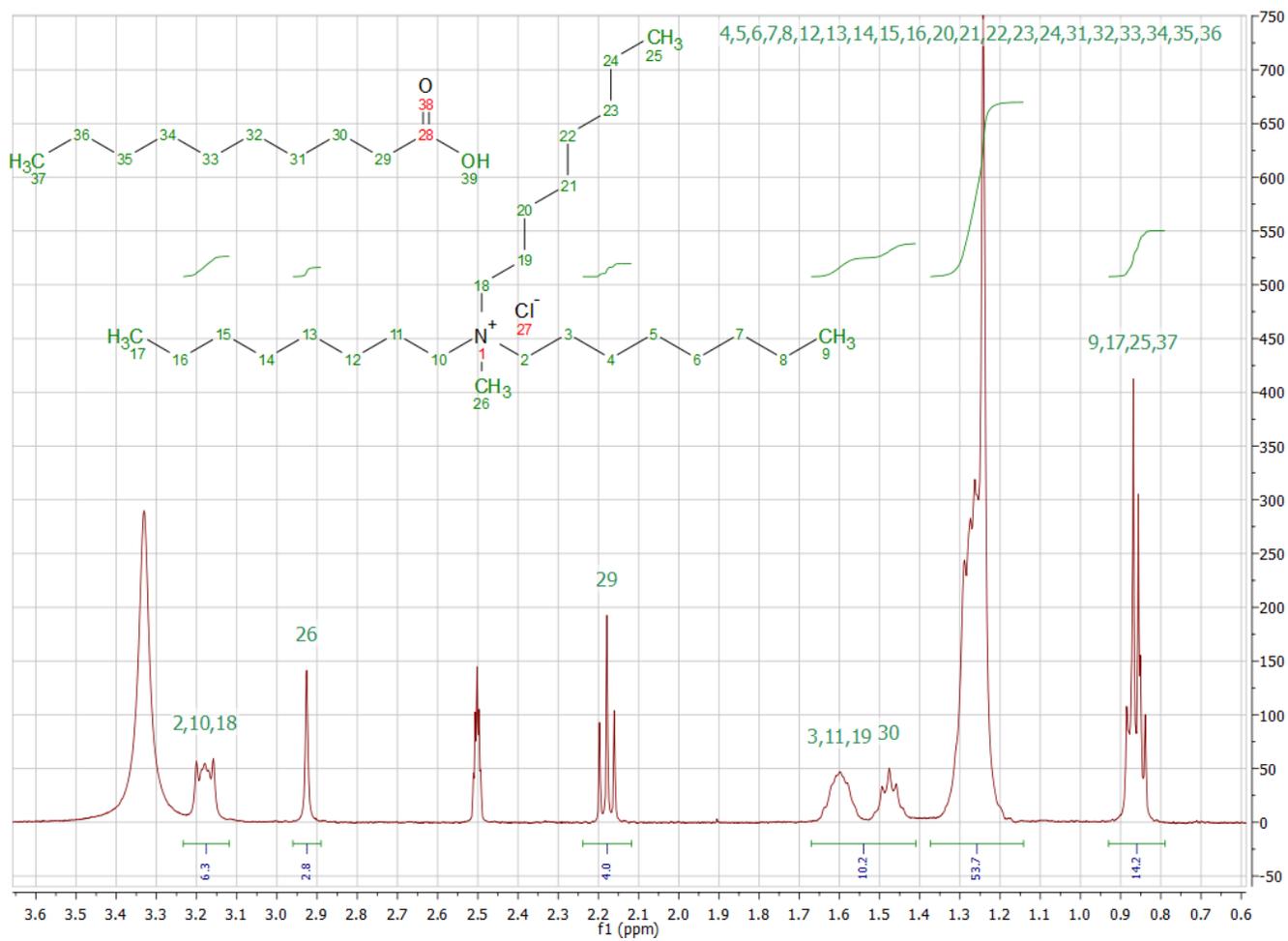


Figure S5: ¹H-NMR spectrum of DecA:N₈₈₈₁-Cl (2:1) before mixing with water.

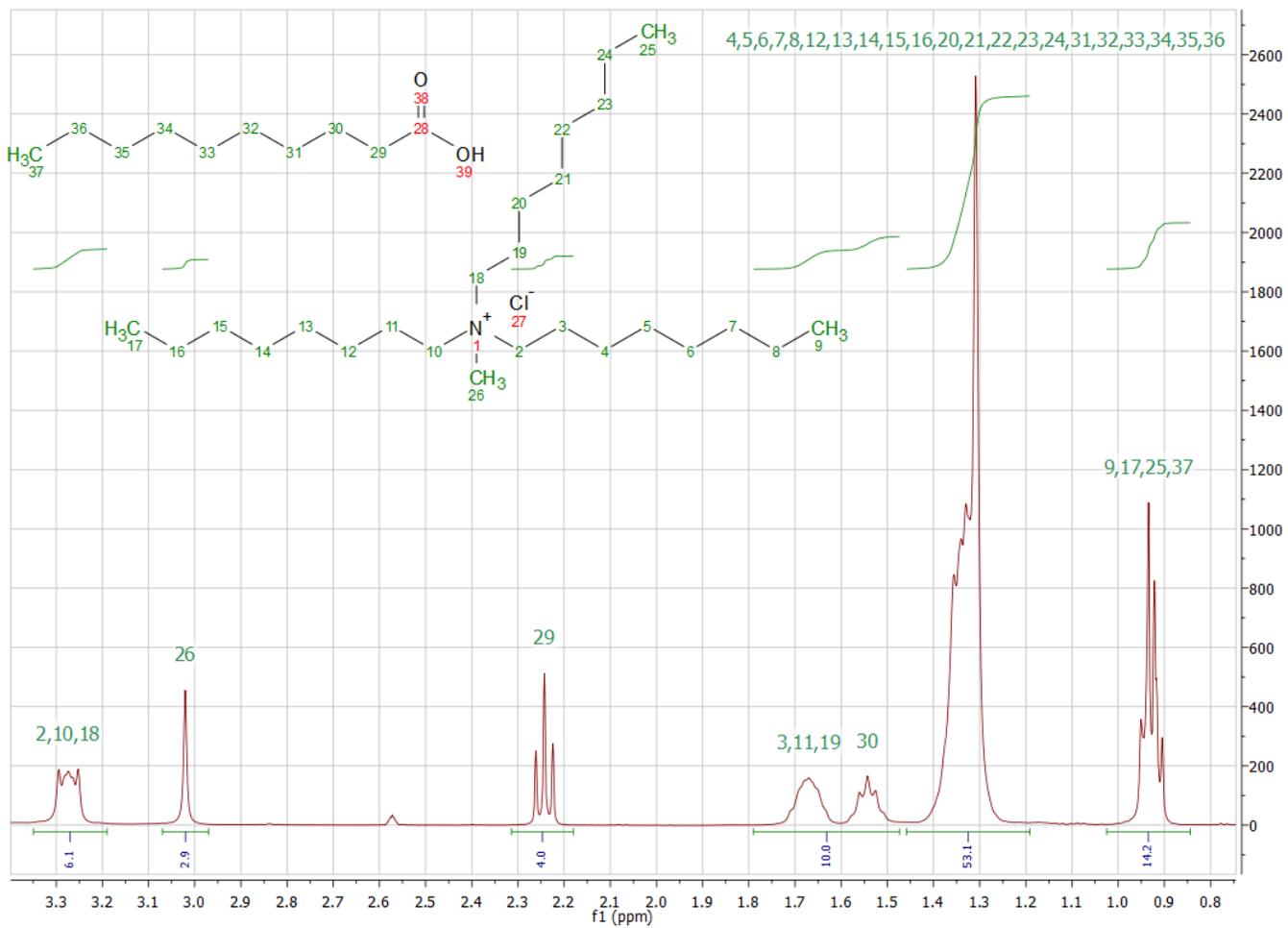


Figure S6: ¹H-NMR spectrum of DecA:N₈₈₈₁-Cl (2:1) after mixing with water.

Table S4: Peak assignments and integrals of the ¹H-NMR spectrum of DecA:N₈₈₈₁-Cl (2:1) before and after mixing with water

Groups		Before mixing with water			After mixing with water		
DecA	N ₈₈₈₁ -Cl	δ (ppm)	Shape	Integral	δ (ppm)	Shape	Integral
2·C(37)H ₃	C(9,17,25)H ₃	0.79-0.93	m	14.2	0.87-1.03	m	14.1
2·C(31-36)H ₂	C(4-8,12-16,20-24)H ₂	1.14-1.38	m	53.7	1.19-1.45	m	53.1
2·C(30)H ₂	C(3,11,19)H ₂	1.41-1.67	m	10.2	1.48-1.78	m	10.0
2·C(29)H ₂	-	2.12-2.24	t	4.0	2.19-2.31	t	4.0
DMSO-d ₆	-	2.48-2.53	m	-	2.55-2.59	m	-
-	C(26)H ₃	2.89-2.96	s	2.9	2.97-3.07	s	2.9
-	C(2,10,18)H ₂	3.13-3.23	m	6.3	3.19-3.35	m	6.1
H ₂ O, HDO	-	3.08-3.58	s	-	-	-	-
C(28)OOH	-	11.75-12.25	s,broad	weak	-	-	-

The difference in water content can be attributed to the usage of a different batch of DMSO-d₆ for sample preparation. The water content of this batch is ~3wt%. All other spectra were measured with a new, dry batch of DMSO-d₆.

DecA:N₇₇₇₇-Cl (2:1)

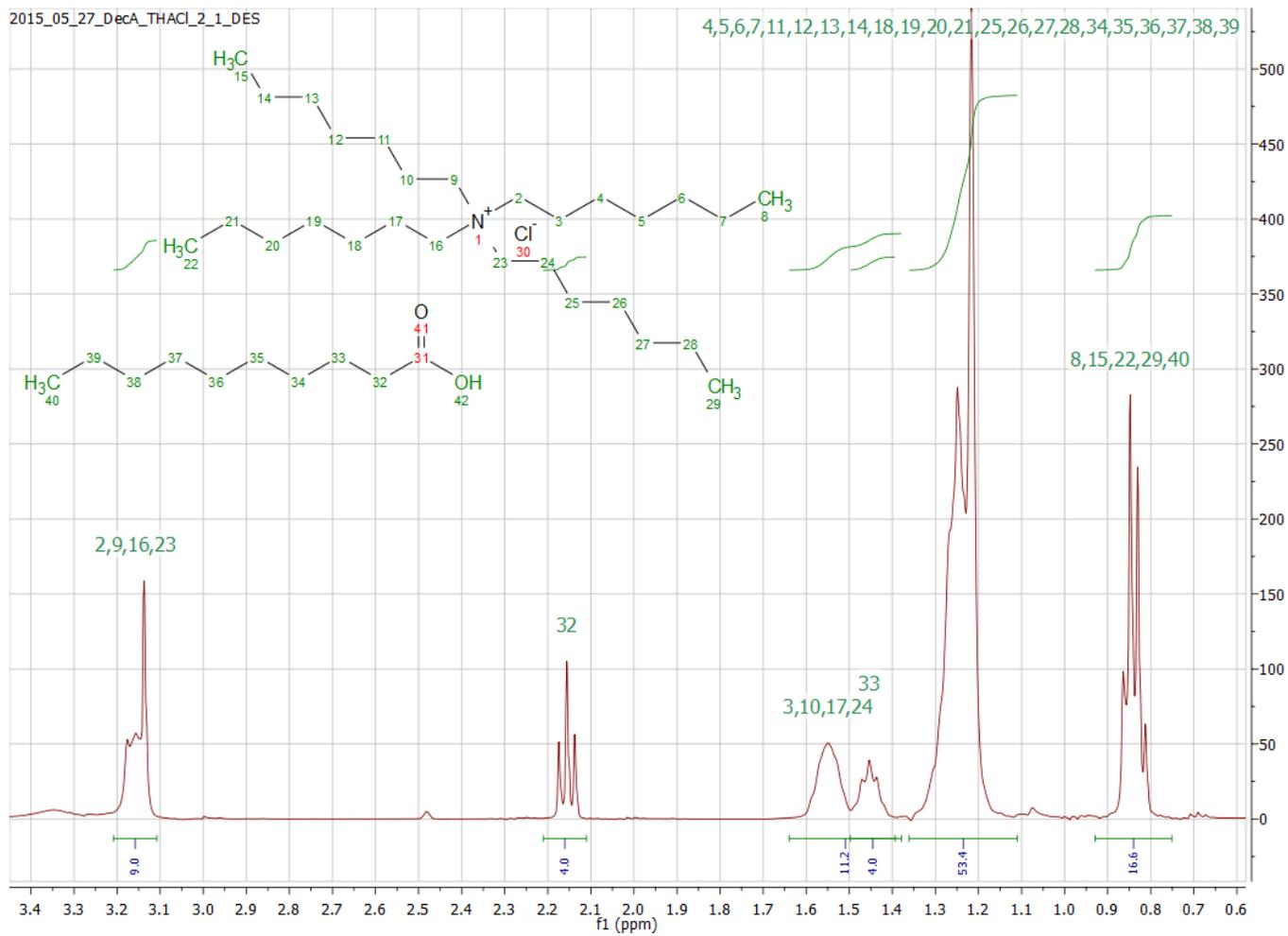


Figure S7: ¹H-NMR spectrum of DecA:N₇₇₇₇-Cl (2:1) before mixing with water.

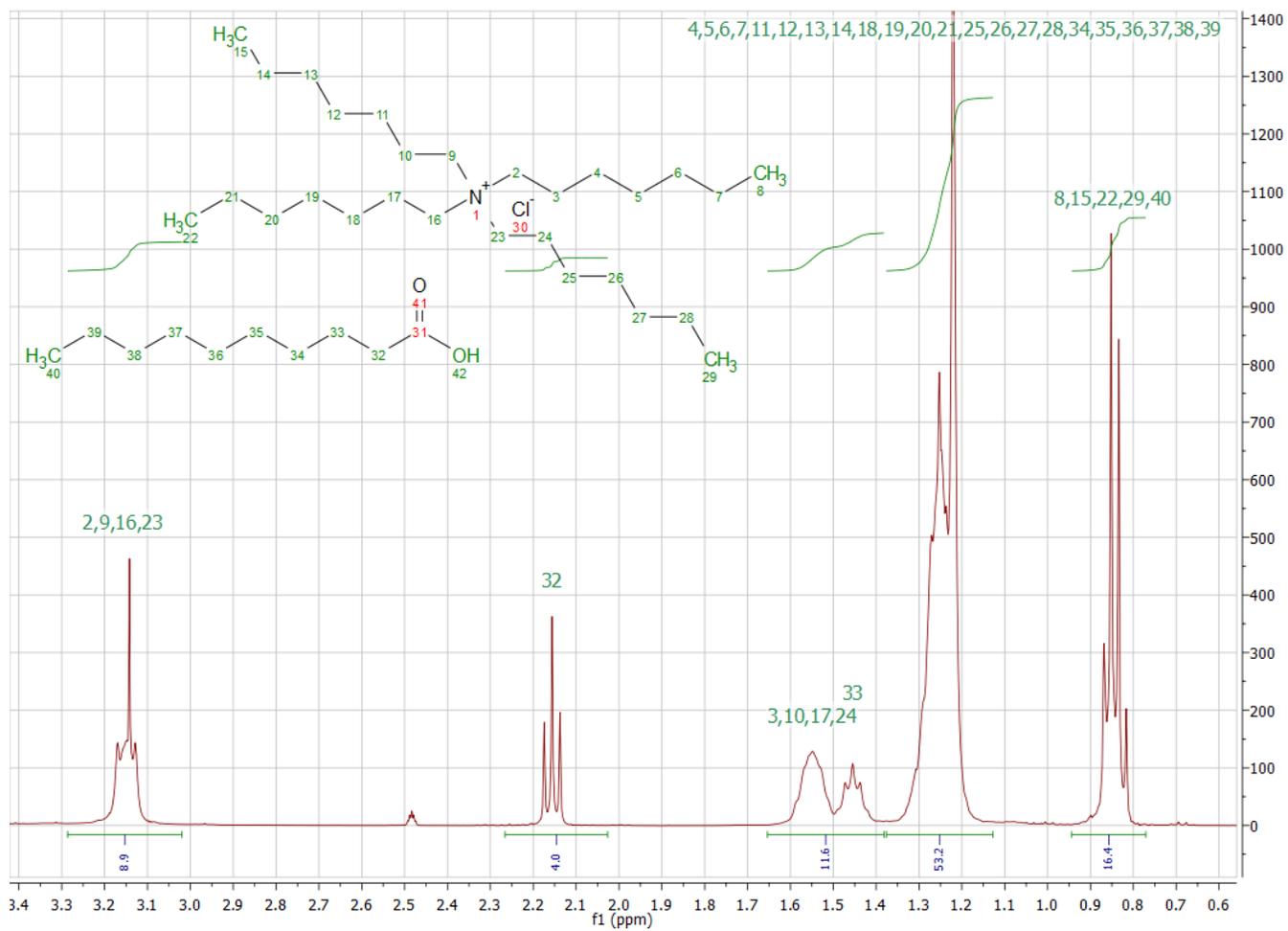


Figure S8: ¹H-NMR spectrum of Deca:N₇₇₇₇-Cl (2:1) after mixing with water.

Table S5: Peak assignments and integrals of the ¹H-NMR spectrum of DecA:N₇₇₇₇-Cl (2:1) before and after mixing with water

Groups		Before mixing with water			After mixing with water		
DecA	N ₇₇₇₇ -Cl	δ (ppm)	Shape	Integral	δ (ppm)	Shape	Integral
2·C(40)H ₃	C(8,15,22,29)H ₃	0.75-0.93	m	16.6	0.77-0.95	m	16.4
2·C(34-39)H ₂	C(4-7,11-14,18-21, 25-28)H ₂	1.11-1.36	m	53.4	1.13-1.37	m	53.2
2·C(33)H ₂	C(3,10,17,24)H ₂	1.38-1.64	m	11.2	1.38-1.65	m	11.6
2·C(32)H ₂	-	2.11-2.21	t	4.0	2.11-2.21	t	4.0
DMSO-d ₆	-	2.46-2.50	m	-	2.46-2.51	m	-
-	C(2,9,16,23)H ₂	3.11-3.21	m	9.1	3.02-3.28	m	8.9
H ₂ O, HDO	-	3.28-3.46	broad	-	-	-	-
C(28)OOH	-	11.75-12.7	broad	1.9	10.50-13.34	broad	0.4

DecA:N₈₈₈₈-Cl (2:1)

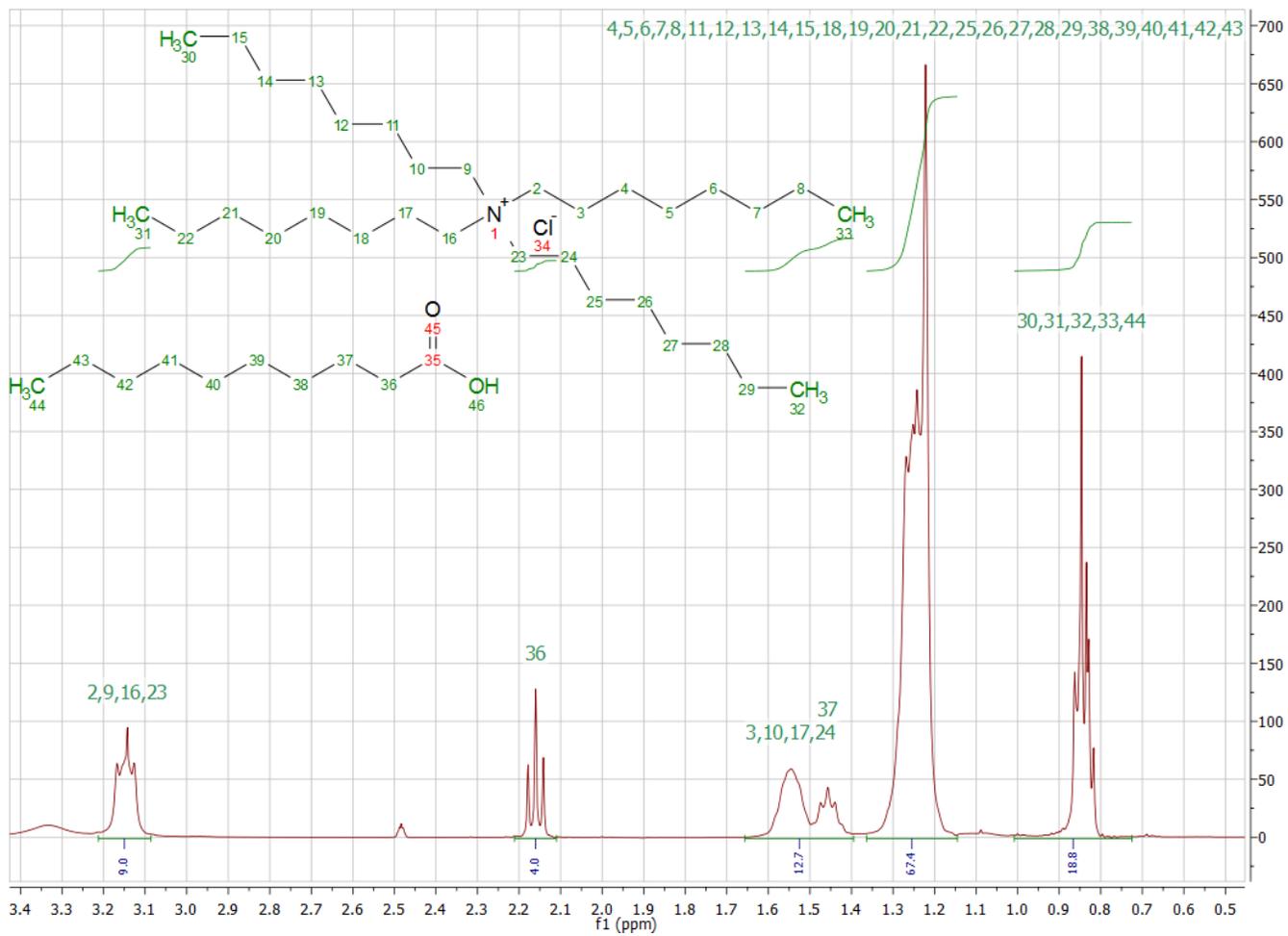


Figure S9: ¹H-NMR spectrum of DecA:N₈₈₈₈-Cl (2:1) before mixing with water.

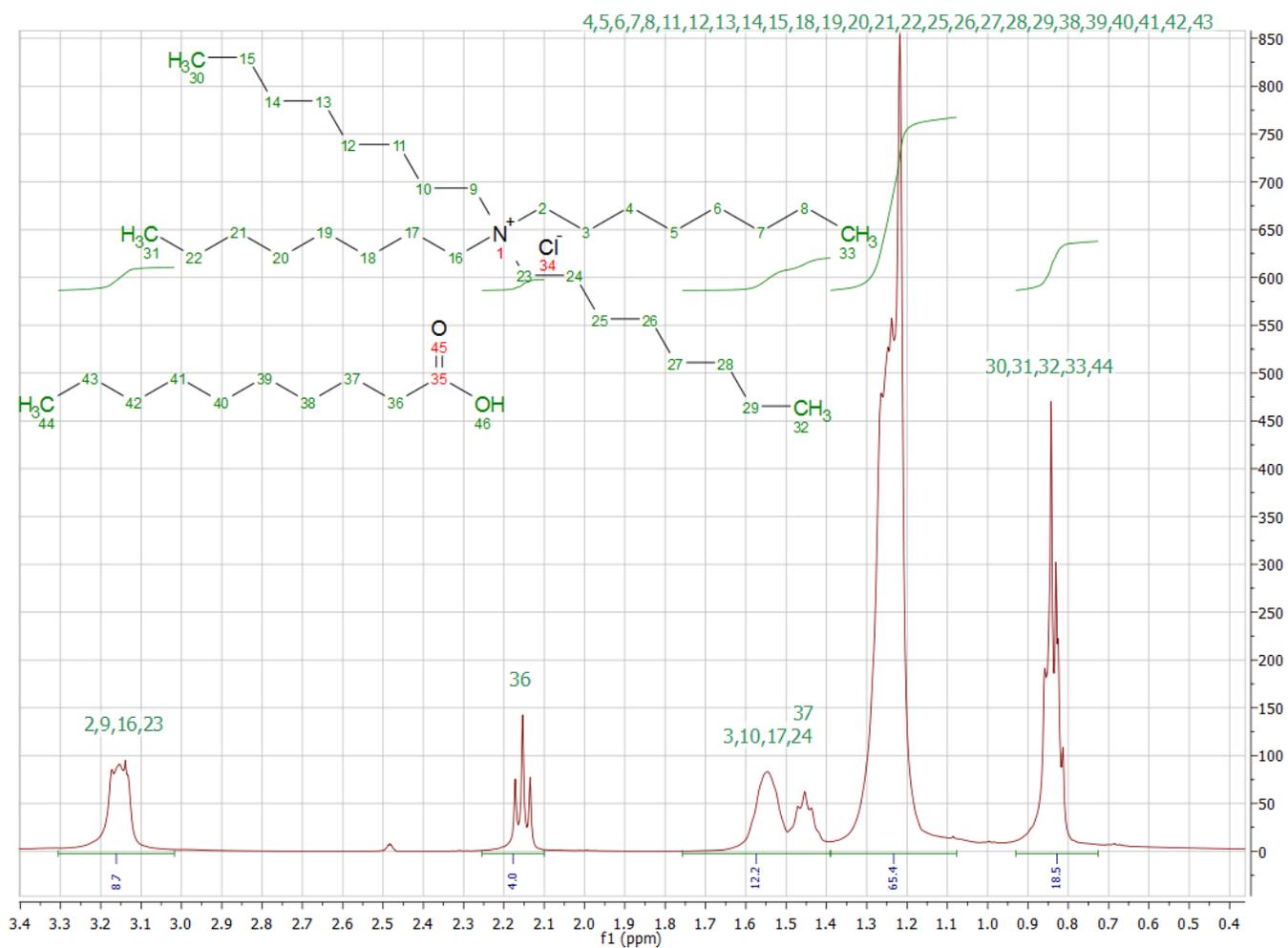


Figure S10: $^1\text{H-NMR}$ spectrum of Deca: $\text{N}_{888}\text{-Cl}$ (2:1) after mixing with water.

Table S6: Peak assignments and integrals of the ¹H-NMR spectrum of DecA:N₈₈₈-Cl (2:1) before and after mixing with water

Groups		Before mixing with water			After mixing with water		
DecA	N ₈₈₈ -Cl	δ (ppm)	Shape	Integral	δ (ppm)	Shape	Integral
2·C(44)H ₃	C(39,31,32,33)H ₃	0.78-0.92	m	18.8	0.78-0.92	m	18.5
2·C(38-43)H ₂	C(4-8,11-15,18-22, 25-29)H ₂	1.14-1.37	m	67.4	1.14-1.37	m	65.4
2·C(37)H ₂	C(3,10,17,24)H ₂	1.39-1.64	m	12.7	1.39-1.64	m	12.2
2·C(36)H ₂	-	2.11-2.21	t	4.0	2.11-2.21	t	4.0
DMSO-d ₆	-	2.46-2.50	m	-	2.46-2.50	m	-
-	C(2,9,16,23)H ₂	3.06-3.23	m	9.0	3.06-3.23	m	8.7
H ₂ O, HDO	-	3.25-3.88	s, broad	-	-	-	-
C(46)OOH	-	11.79-12.76	s	1.5	-	-	-

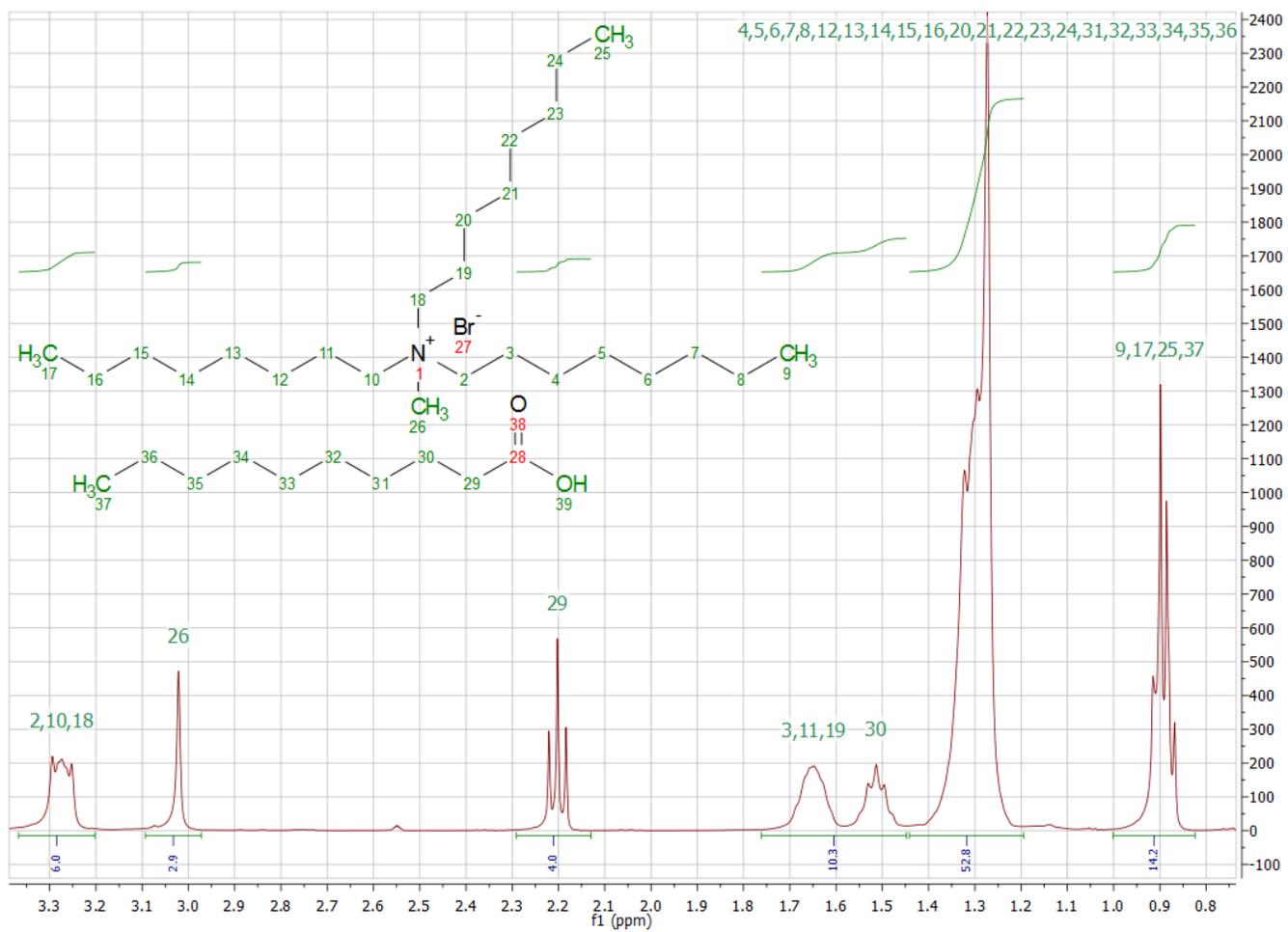


Figure S12: $^1\text{H-NMR}$ spectrum of DecA: $\text{N}_{881}\text{-Br}$ (2:1) after mixing with water.

Table S7: Peak assignments and integrals of the ¹H-NMR spectrum of DecA:N₈₈₈₁-Br (2:1) before and after mixing with water

Groups		Before mixing with water			After mixing with water		
DecA	N ₈₈₈₁ -Br	δ (ppm)	Shape	Integral	δ (ppm)	Shape	Integral
2·C(37)H ₃	C(9,17,25)H ₃	0.75-0.92	m	14.4	0.82-1.00	m	14.2
2·C(31-36)H ₂	C(4-8,12-16,20-24)H ₂	1.09-1.37	m	53.5	1.19-1.44	m	52.8
2·C(30)H ₂	C(3,11,19)H ₂	1.37-1.67	m	10.2	1.45-1.76	m	10.3
2·C(29)H ₂	-	2.08-2.21	t	4.0	2.13-2.29	t	4.0
DMSO-d ₆	-	-	m	-	2.53-2.57	m	-
-	C(26)H ₃	2.89-3.02	s	3.1	2.97-3.09	s	2.9
-	C(2,10,18)H ₂	3.12-3.28	m	5.9	3.20-3.37	m	6.0
H ₂ O, HDO	-	3.28-3.47	s	-	-	-	-
C(28)OOH	-	11.60-12.40	s,broad	1.7	-	-	-

DecA:N₈₈₈₈-Br (2:1)

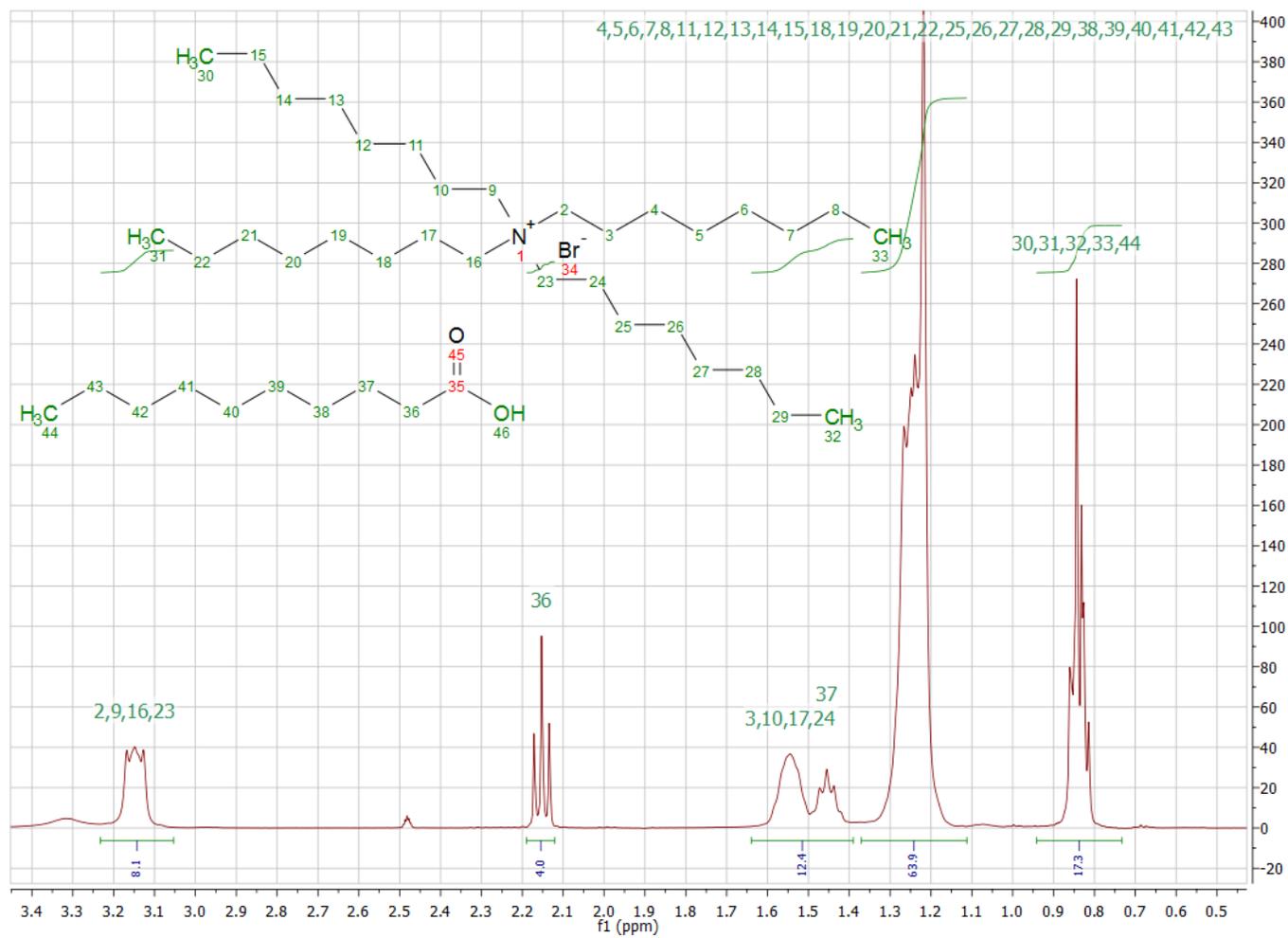


Figure S13: ¹H-NMR spectrum of DecA:N₈₈₈₈-Br (2:1) before mixing with water.

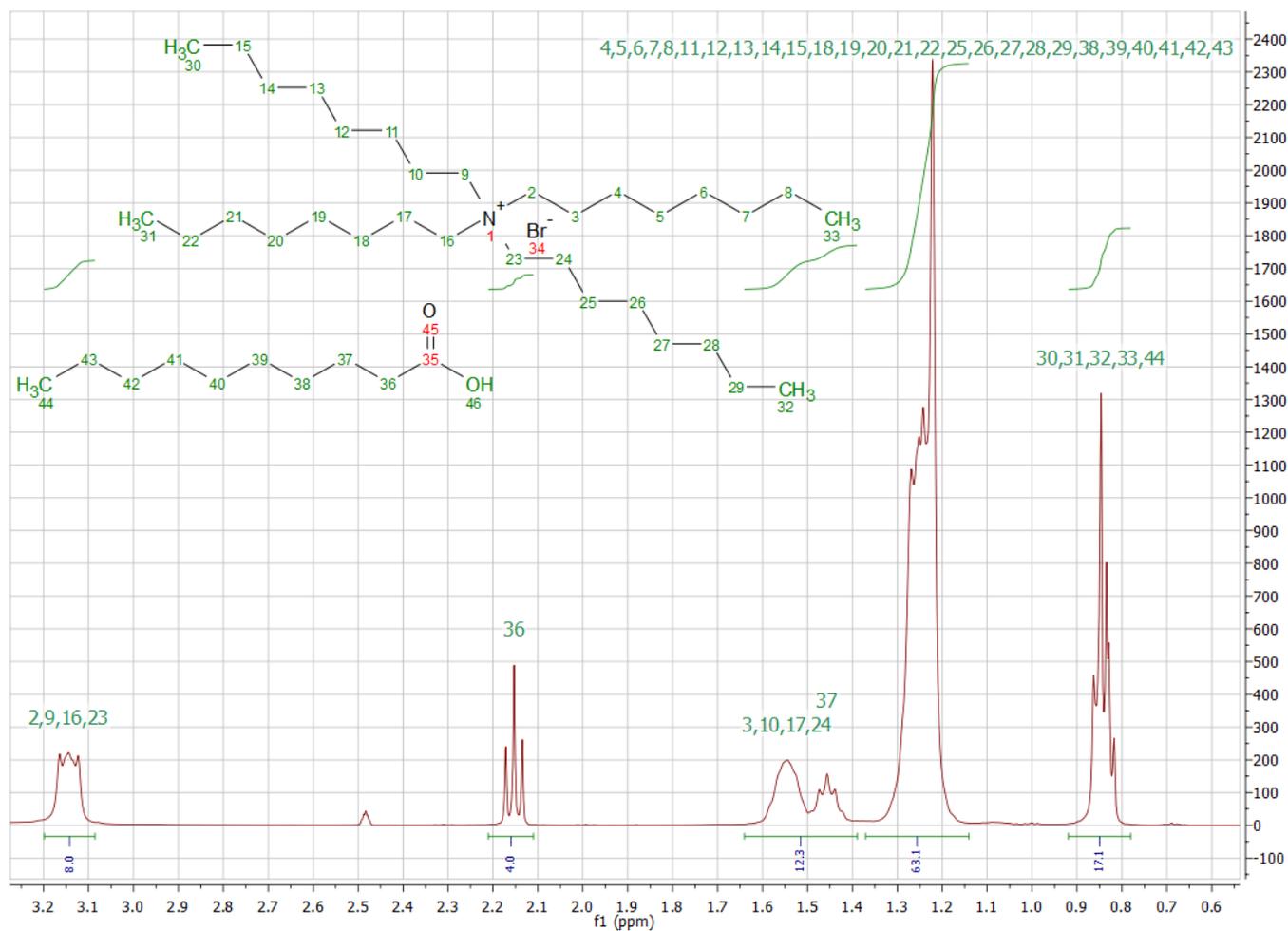


Figure S14: ¹H-NMR spectrum of DecA:N₈₈₈-Br (2:1) before mixing with water.

Table S8: Peak assignments and integrals of the ¹H-NMR spectrum of DecA:N₈₈₈-Br (2:1) before and after mixing with water

Groups		Before mixing with water			After mixing with water		
DecA	N ₈₈₈ -Br	δ (ppm)	Shape	Integral	δ (ppm)	Shape	Integral
2·C(44)H ₃	C(39,31,32,33)H ₃	0.78-0.92	m	17.3	0.78-0.92	m	17.1
2·C(38-43)H ₂	C(4-8,11-15,18-22, 25-29)H ₂	1.14-1.37	m	63.9	1.14-1.37	m	63.1
2·C(37)H ₂	C(3,10,17,24)H ₂	1.39-1.64	m	12.4	1.39-1.64	m	12.3
2·C(36)H ₂	-	2.11-2.21	t	4.0	2.11-2.21	t	4.0
DMSO-d ₆	-	2.46-2.50	m	-	2.46-2.50	m	-
-	C(2,9,16,23)H ₂	3.06-3.23	m	8.1	3.06-3.23	m	8.0
H ₂ O, HDO	-	3.25-3.88	s, broad	-	-	-	-
C(46)OOH	-	11.79-12.76	s	1.7	11.79-12.76	broad	0.9

ES15: Densities and viscosities

Table S9: Experimentally determined densities (ρ) of the studied DESs as function of temperature (T)

T/K	$\rho/\text{kg}\cdot\text{m}^{-3}$					
	DecA:N ₄₄₄₄ -Cl (2:1)	DecA:N ₈₈₈₁ -Cl (2:1)	DecA:N ₇₇₇₇ -Cl (2:1)	DecA:N ₈₈₈₈ -Cl (2:1)	DecA:N ₈₈₈₁ -Br (2:1)	DecA:N ₈₈₈₈ -Br (2:1)
288.15	923.2	902.7	897.2	895.3	948.9	936.4
293.15	919.9	899.6	893.9	892.1	945.6	933.1
298.15	916.8	896.4	890.7	888.9	942.2	929.8
303.15	913.6	893.2	887.5	885.7	938.8	926.5
308.15	910.4	890.0	884.3	882.5	935.6	923.2
313.15	907.3	886.9	881.1	879.4	932.3	920.0
318.15	904.2	883.8	877.9	876.3	929.0	916.8
323.15	901.0	880.7	874.7	873.2	925.8	913.6

Table S10: Experimentally determined values of the dynamic viscosity (η) of the studied DESs as function of temperature (T)

T/K	$\eta/\text{mPa}\cdot\text{s}$					
	DecA:N ₄₄₄₄ -Cl (2:1)	DecA:N ₈₈₈₁ -Cl (2:1)	DecA:N ₇₇₇₇ -Cl (2:1)	DecA:N ₈₈₈₈ -Cl (2:1)	DecA:N ₈₈₈₁ -Br (2:1)	DecA:N ₈₈₈₈ -Br (2:1)
288.15	526.39	1706.23	307.09	929.18	1186.97	1273.07
293.15	368.54	1138.73	227.96	654.18	814.53	889.57
298.15	265.26	783.41	172.87	472.58	576.53	636.36
303.15	195.05	552.32	133.19	348.61	416.93	464.04
308.15	146.16	398.23	104.19	261.97	307.25	344.61
313.15	111.52	293.12	82.73	200.26	230.39	260.41
318.15	86.53	219.87	66.61	155.52	175.72	199.89
323.15	68.27	190.56	54.32	122.61	136.15	155.69

ESI6: TGA thermograms

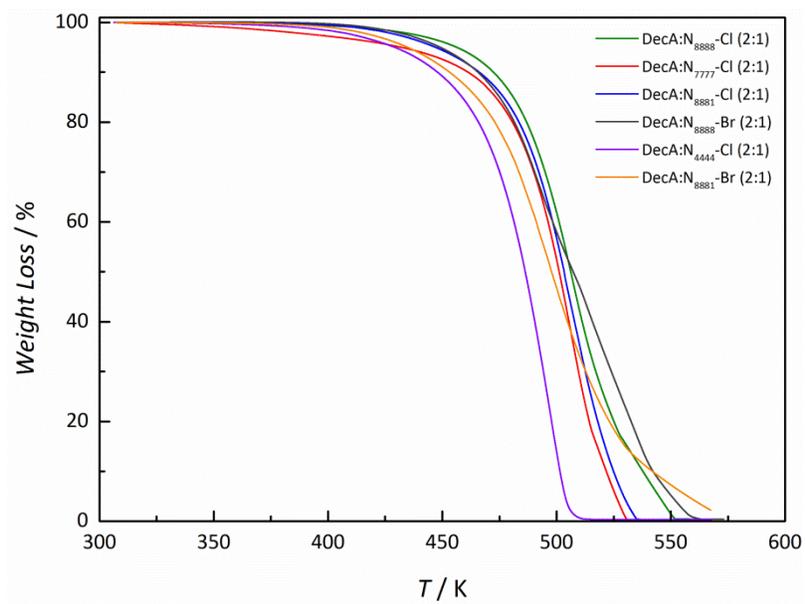


Figure S15: TGA curves for all the studied DESs.

ESI7: DSC thermograms

DecA:N₄₄₄₄-Cl (2:1)

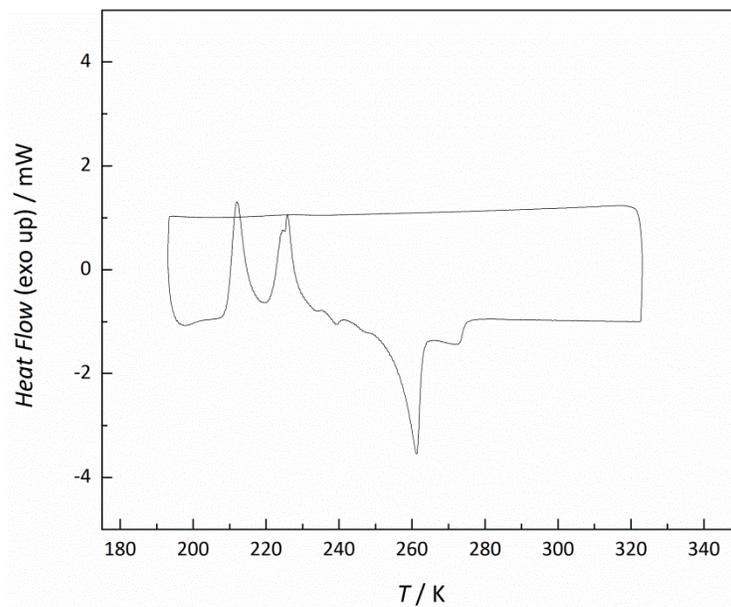


Figure S16: DSC thermogram for the DecA:N₄₄₄₄-Cl (2:1).

DecA:N₈₈₈₁-Cl (2:1)

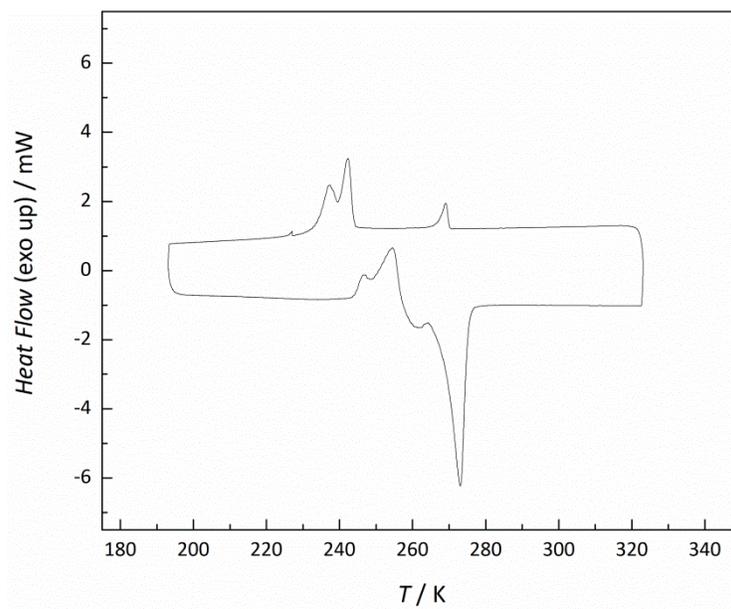


Figure S17: DSC thermogram for the DecA:N₈₈₈₁-Cl (2:1).

DecA:N₇₇₇-Cl (2:1)

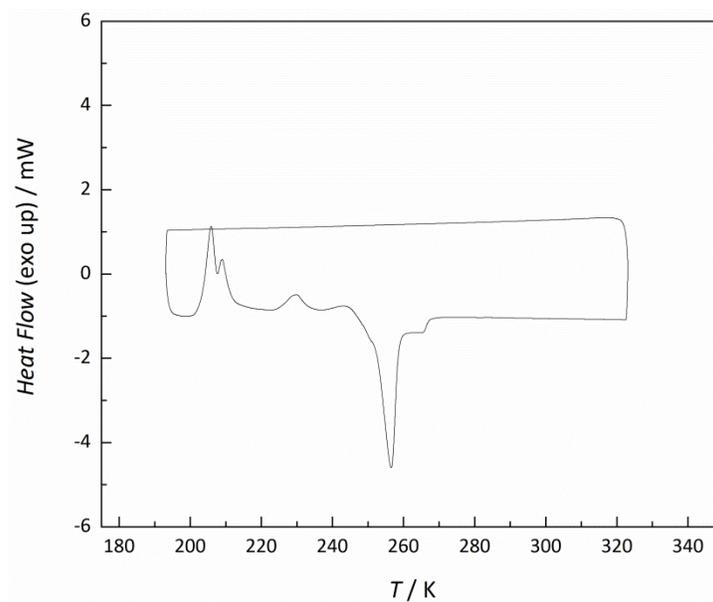


Figure S18: DSC thermogram for the DecA:N₇₇₇-Cl (2:1).

DecA:N₈₈₈-Cl (2:1)

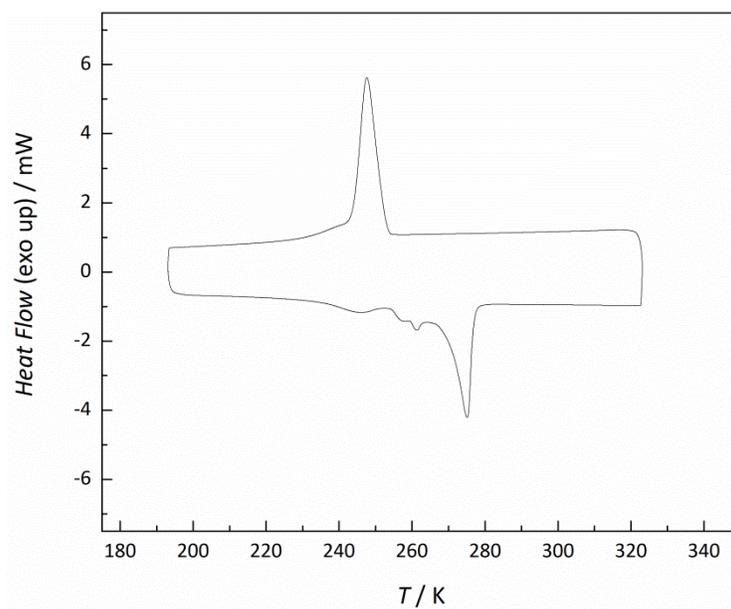


Figure S19: DSC thermogram for the DecA:N₈₈₈-Cl (2:1).

DecA:N₈₈₈₁-Br (2:1)

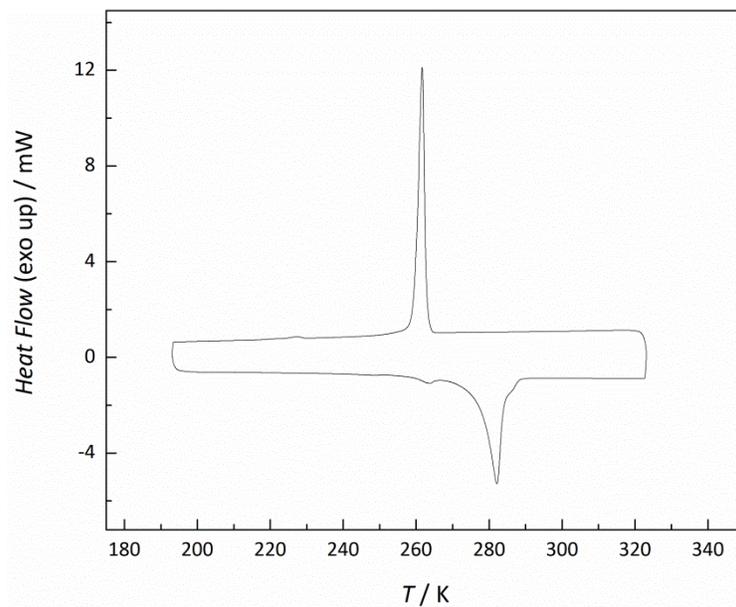


Figure S20: DSC thermogram for the DecA:N₈₈₈₁-Br (2:1).

DecA:N₈₈₈₈-Br (2:1)

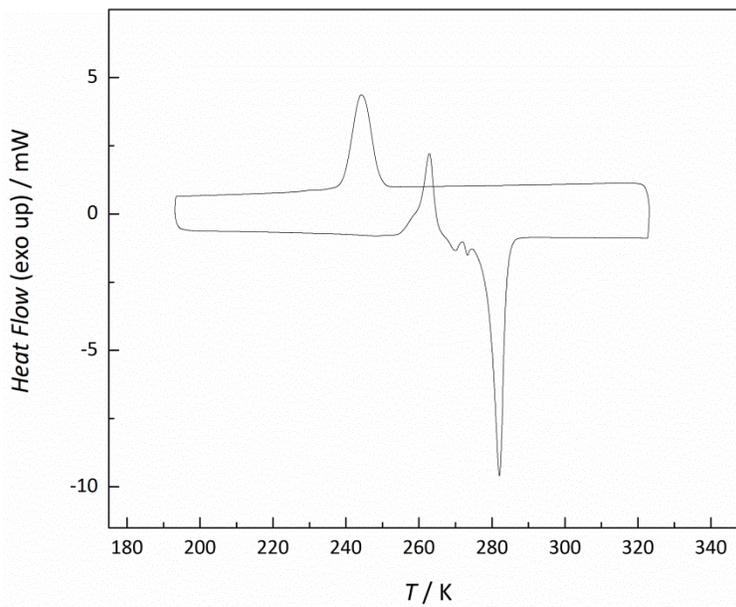


Figure S21: DSC thermogram for the DecA:N₈₈₈₈-Br (2:1).

ESI8: FTIR spectra

DecA:N₄₄₄₄-Cl (2:1)

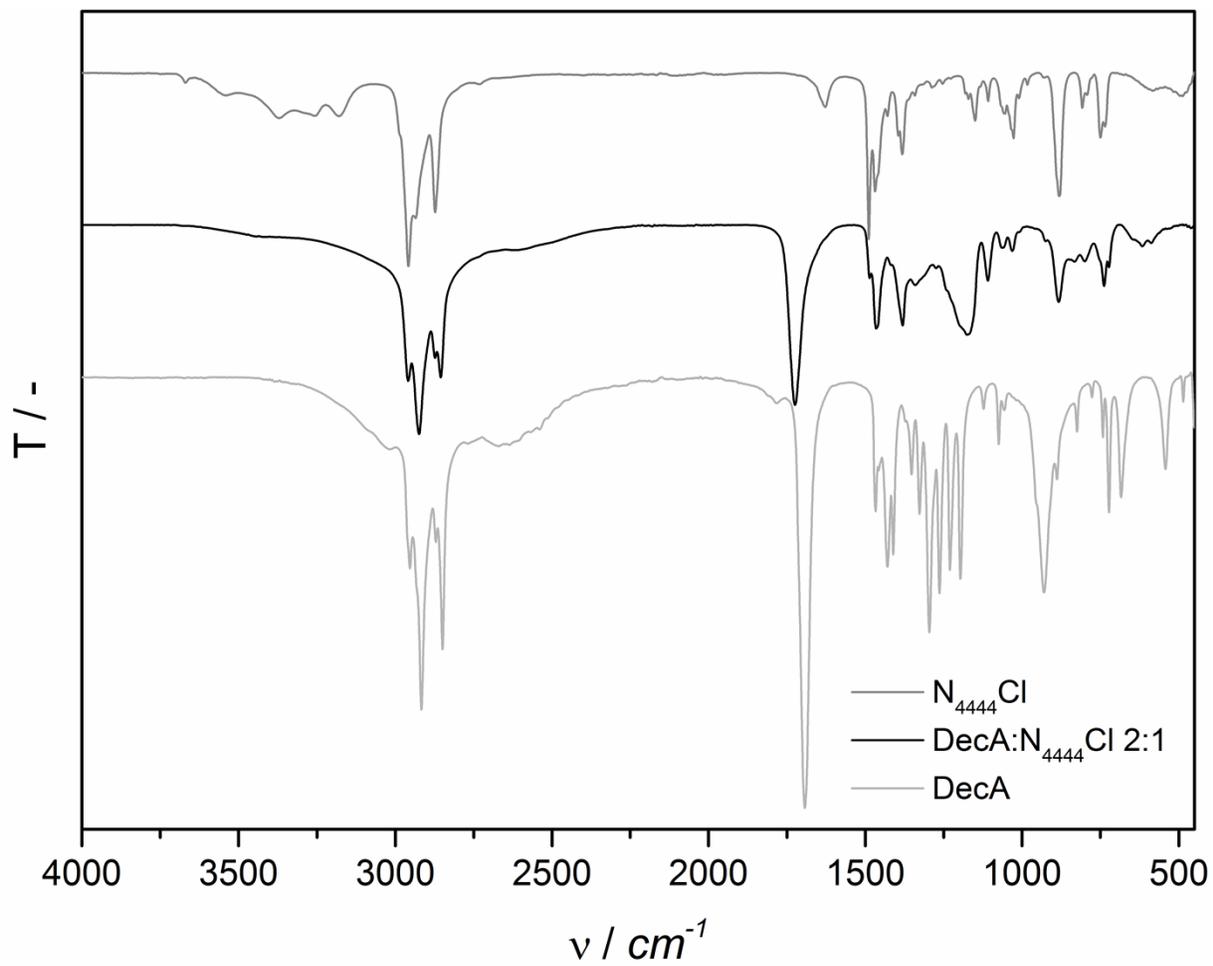


Figure S22: Stacked FTIR spectra of DecA:N₄₄₄₄-Cl (2:1) and its starting components.

DecA:N₈₈₈₁-Cl (2:1)

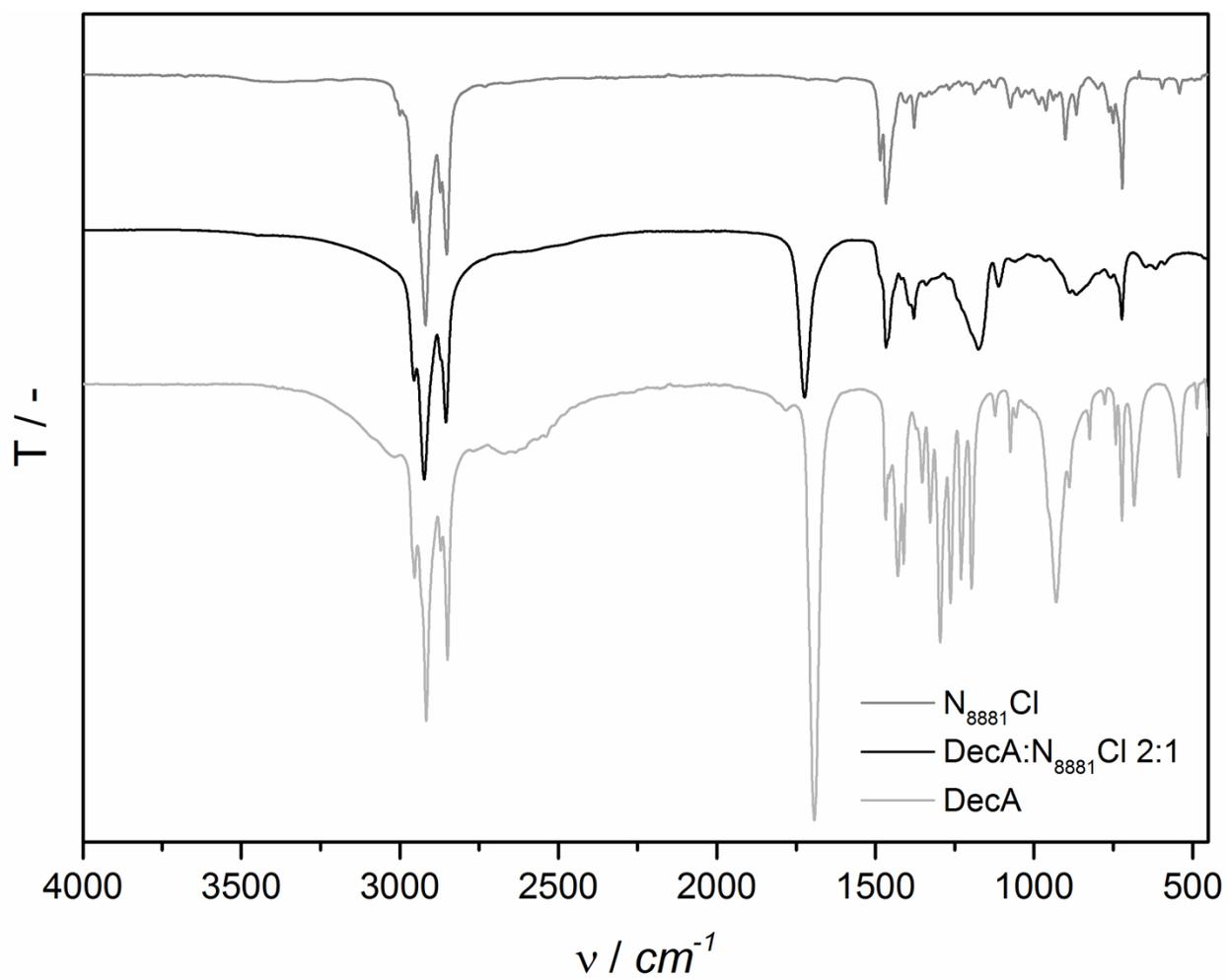


Figure S23: Stacked FTIR spectra of DecA:N₈₈₈₁-Cl (2:1) and its starting components.

DecA:N₇₇₇₇-Cl (2:1)

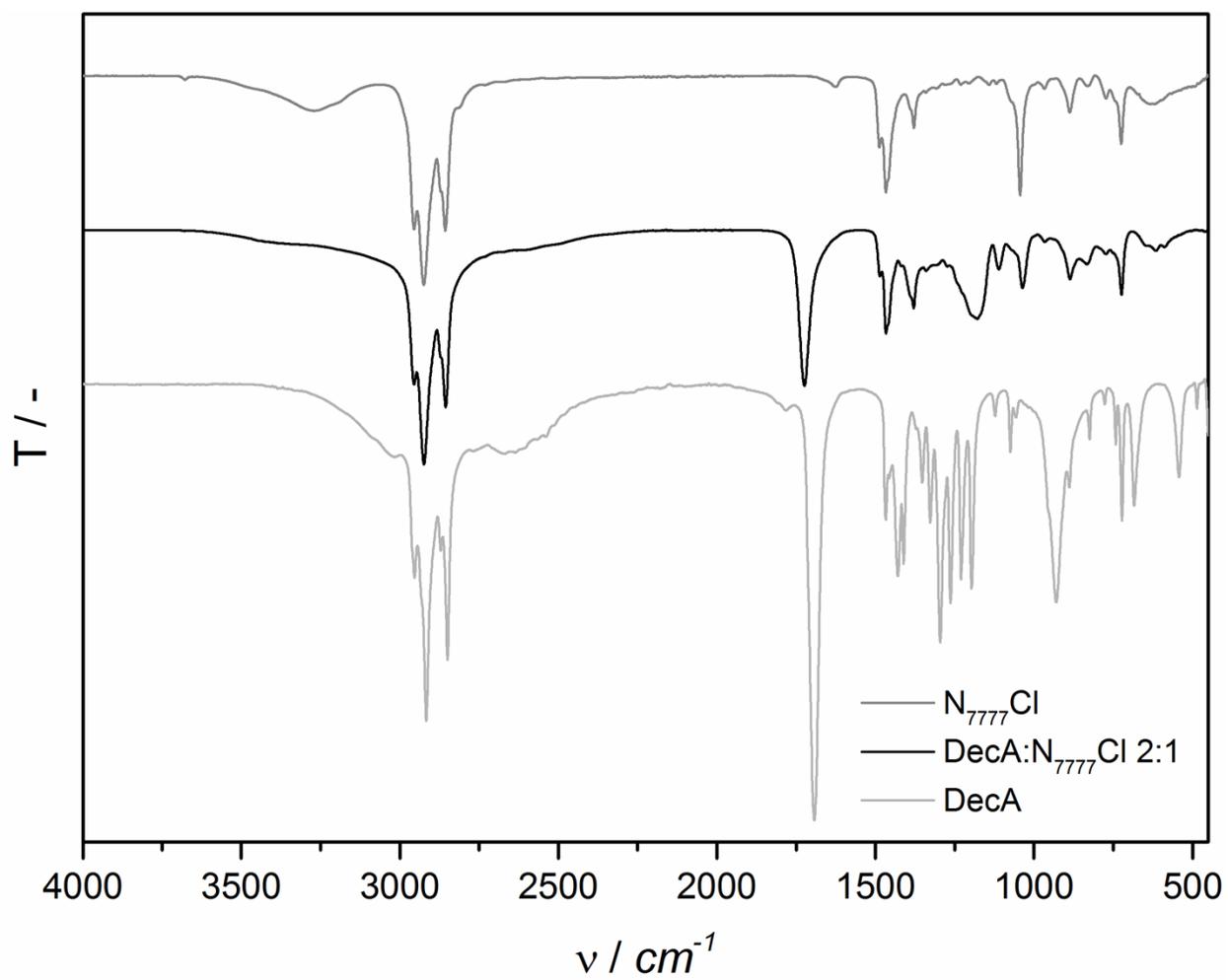


Figure S24: Stacked FTIR spectra of DecA:N₇₇₇₇-Cl (2:1) and its starting components.

DecA:N₈₈₈₈-Cl (2:1)

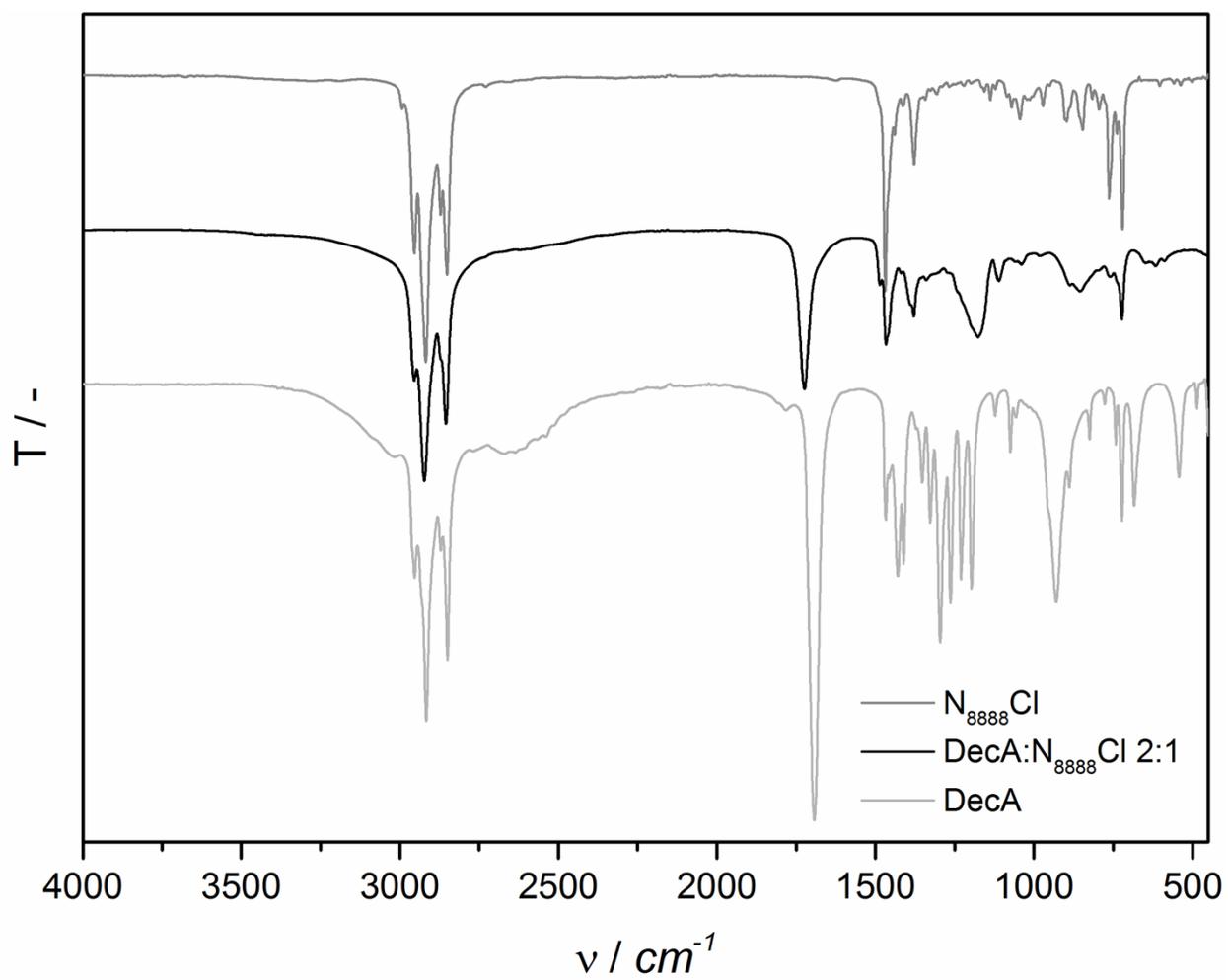


Figure S25: Stacked FTIR spectra of DecA:N₈₈₈₈-Cl (2:1) and its starting components.

DecA:N₈₈₈₁-Br (2:1)

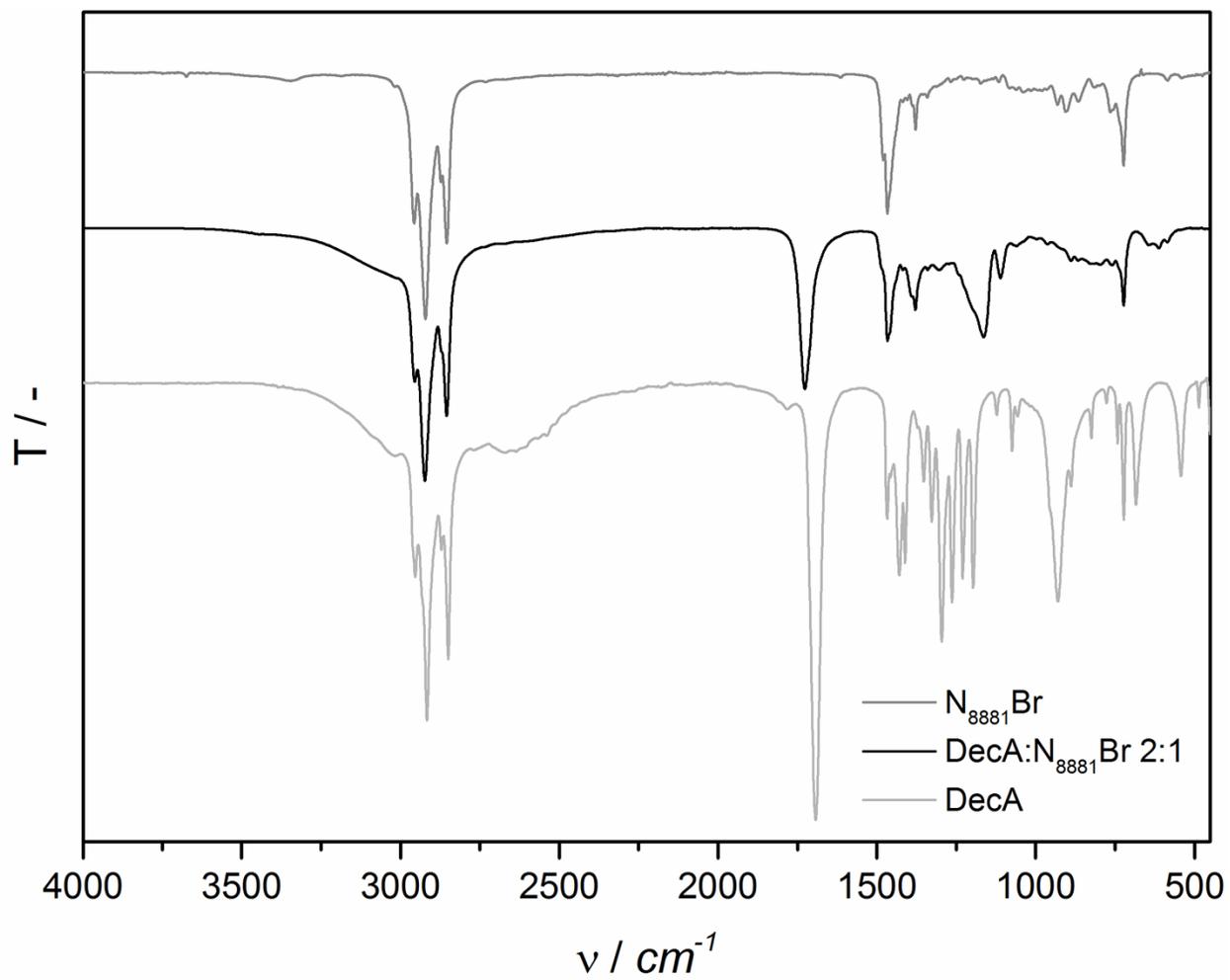


Figure S26: Stacked FTIR spectra of DecA:N₈₈₈₁-Br (2:1) and its starting components.

DecA:N₈₈₈₈-Br (2:1)

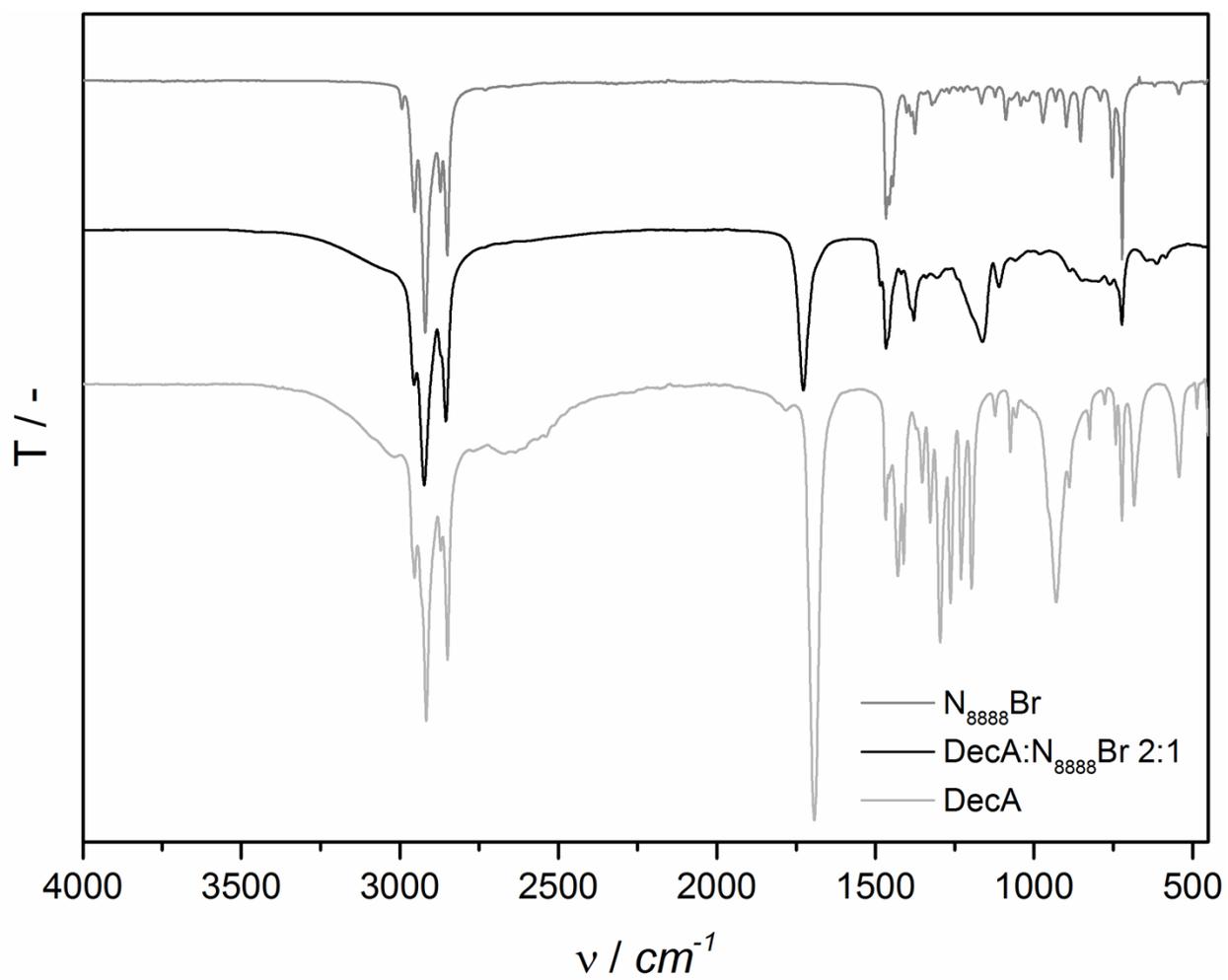


Figure S27: Stacked FTIR spectra of DecA:N₈₈₈₈-Br (2:1) and its starting components.