Electronic Supplementary Information

Synthesis of tailored oxymethylene ether (OME) fuels via transacetalization reactions

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Content:

A. Reaction monitoring:

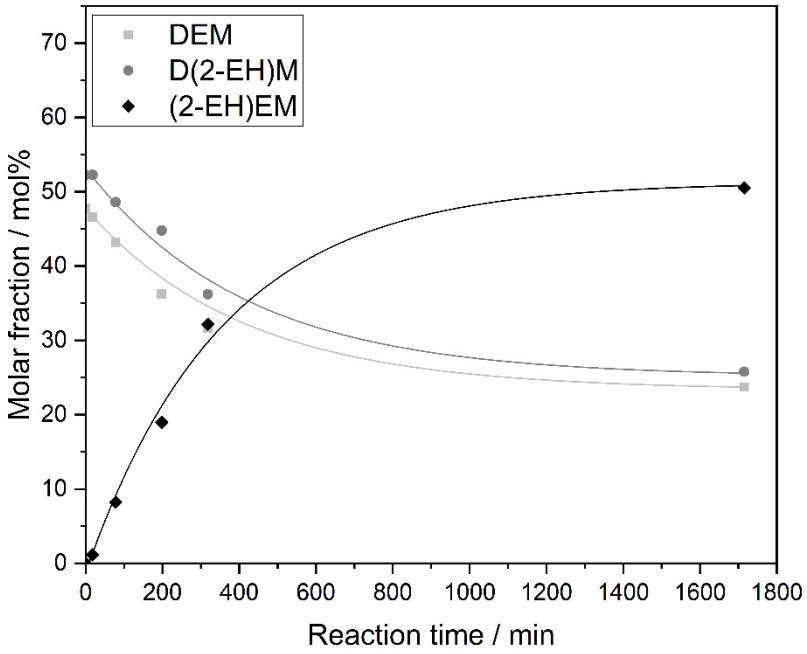


Figure A.1: Transacetalization reaction of D(2-EH)M with DEM catalyzed by zeolite BEA-25 (reaction conditions: 60 °C, 400 rpm, 0.78 wt% catalyst).

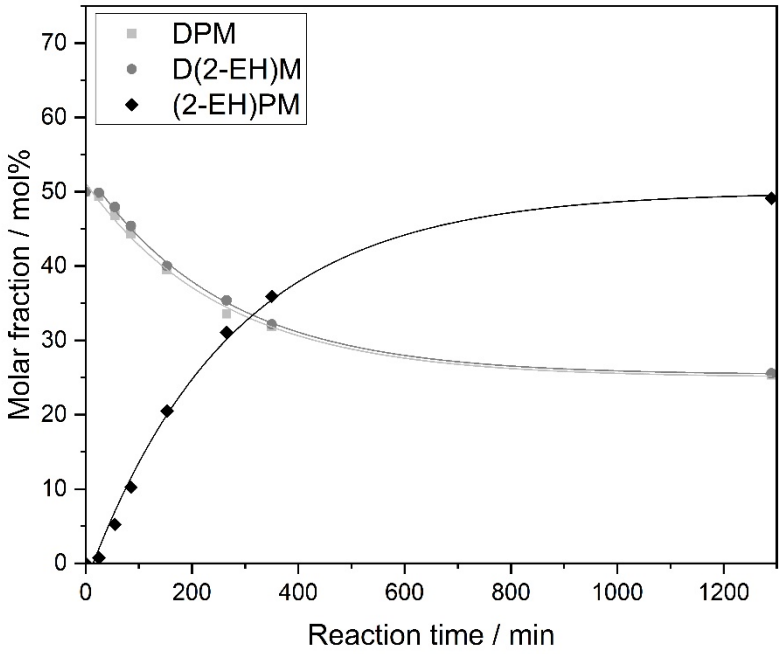


Figure A.2: Transacetalization reaction of D(2-EH)M with DPM catalysed by zeolite BEA-25 (reaction conditions: 80 °C, 400 rpm, 0.78 wt% catalyst).

B. NMR spectra

B.1 NMR spectra for DEM:

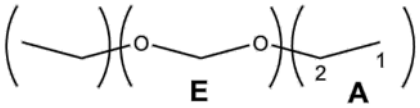


Figure B.1.1: Nomenclature for NMR analysis of DEM.

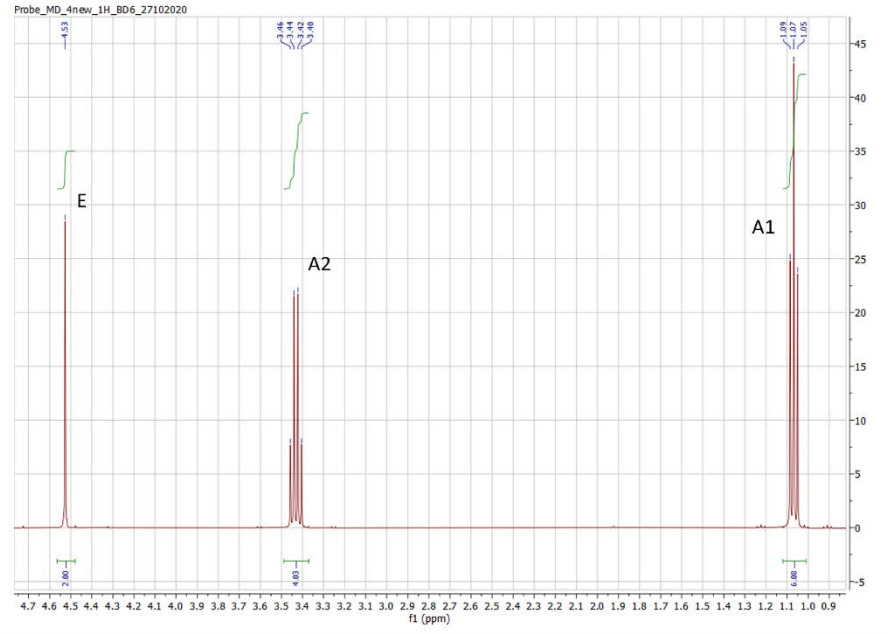


Figure B.1.2: ^{1}H-NMR spectrum for DEM (δ in ppm, J in Hz, in C_6D_6): 4.55 (s, 2H, **E**); 3.44 (q, 4H, **A2**, ^{3}JH-H 8 Hz); 0.87 (t, 6H, **A1**, ^{3}JH-H ~8 Hz).

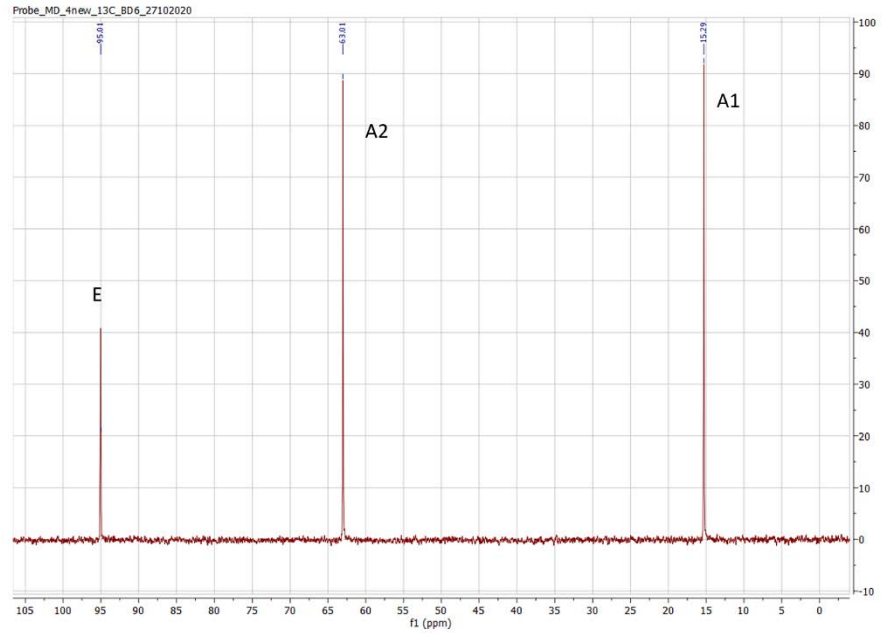


Figure B.1.3: ^{13}C-NMR spectrum for DEM (δ in ppm, J in Hz, in C_6D_6): 95.05 ppm (**E**, ^{1}JC-H= 161.0); 69.03 (**A2**, ^{1}JC-H= 141.0); 15.34 (**A1**, ^{1}JC-H= 126).

B.2 NMR spectra for DPM:

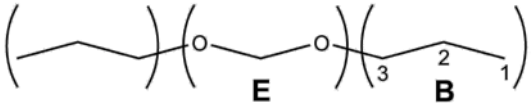


Figure B.2.1: Nomenclature for NMR analysis of DPM.

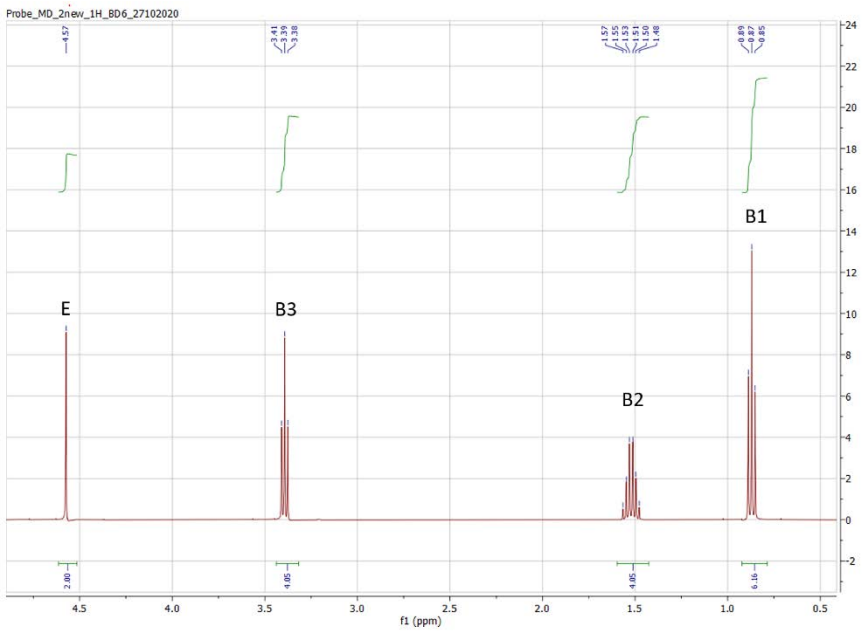


Figure B.2.2: ^{1}H-NMR spectrum for DPM (δ in ppm, J in Hz, in C_6D_6): 4.57 (s, 2H, **E**); 3.39 (d, 4H, **B3**, ^{3}JH-H= 4); 1.52 (m overlap, 4H, **B2**); 0.87 (pseudo t, overlap, 6H, **B1**, ^{3}JH-H = 8).

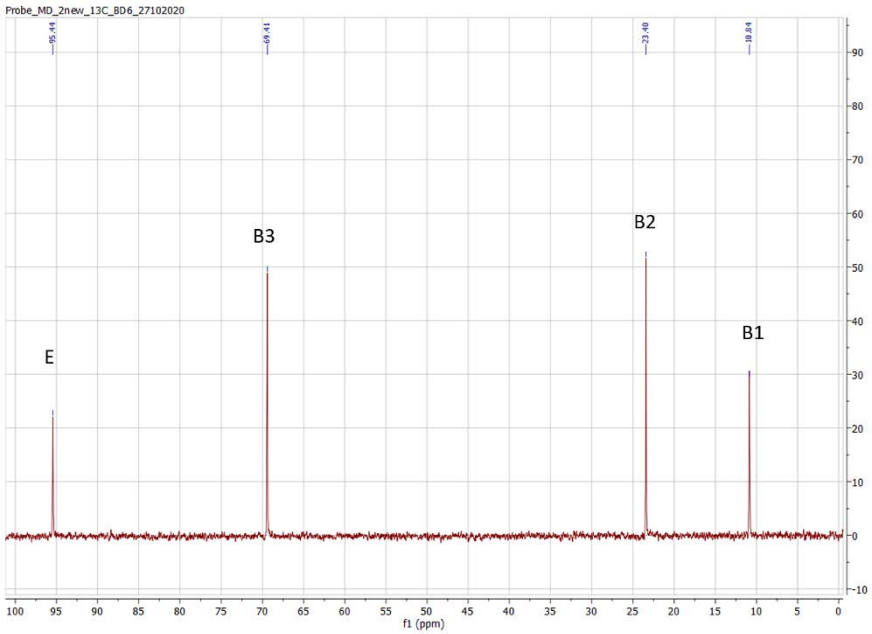


Figure B.2.3: ^{13}C-NMR spectrum for DPM (δ in ppm, J in Hz, in C_6D_6): 95.49 ppm (**E**, ^{1}JC-H=161.0 Hz); 69.46 (**B3**, ^{1}JC-H= 135.0 Hz); 23.43 (**B2**, ^{1}JC-H= 127.7 Hz); 10.87 (**B1**, ^{1}JC-H= 125.4 Hz).

B.3 NMR spectra for DBM:

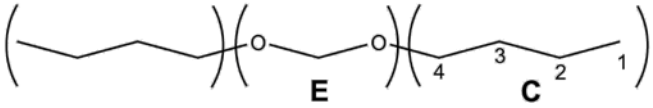


Figure B.3.1: Nomenclature for NMR analysis of DBM.

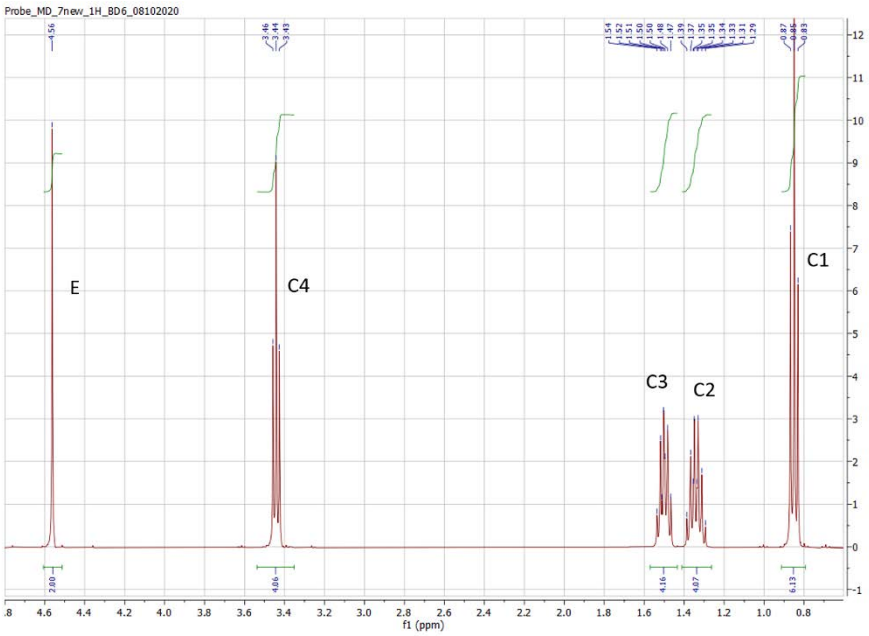


Figure B.3.2: ^{1}H-NMR spectrum for DBM (δ in ppm, J in Hz, in C_6D_6): 4.57 (s, 2H, **E**); 3.45 (t, 4H, **C4**, ^{3}JH-H= 8 Hz); 1.51 (quint. , 4H, **C3**); 1.32 (m , 4H, **C2**); 0.85 (t, 6H, **C1**, ^{3}JH-H= 8 Hz).

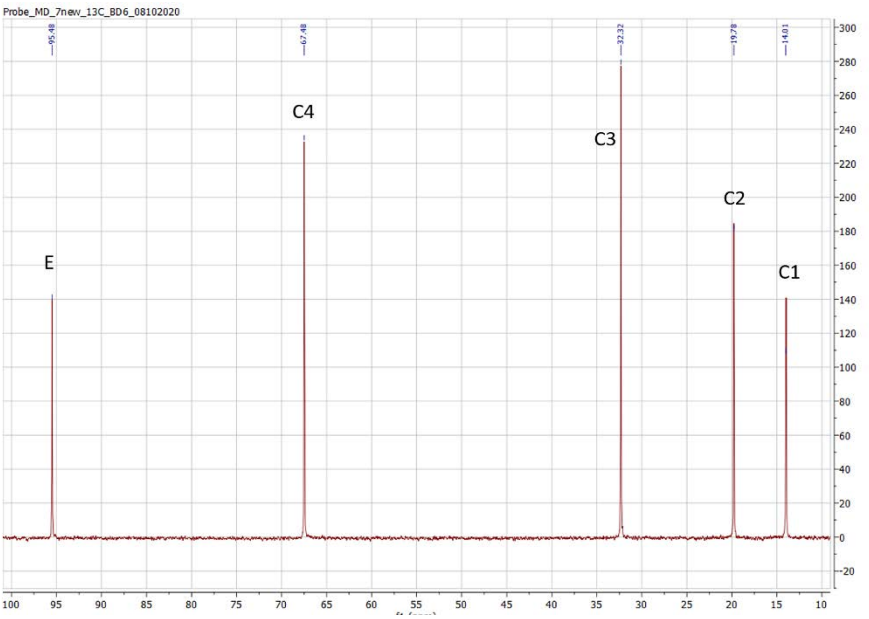


Figure B.3.3: ^{13}C-NMR spectrum for DBM (δ in ppm, J in Hz, in C_6D_6): 95.50 ppm (**E** ^{1}JC-H= 161.3); 67.51 (**C4** ^{1}JC-H= 141.2); 32.34 (**C3** ^{1}JC-H= 124.2); 19.80 (**C2** ^{1}JC-H= 124.2); 14.00 (**C1** ^{1}JC-H= 124.2).

B.4 NMR spectra for D(2-EH)M:

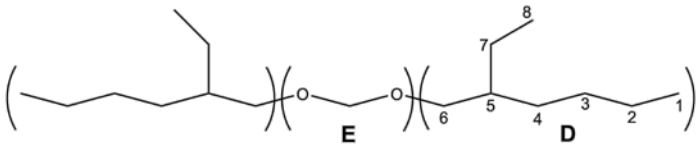


Figure B.4.1: Nomenclature for NMR analysis of D(2-EH)M.

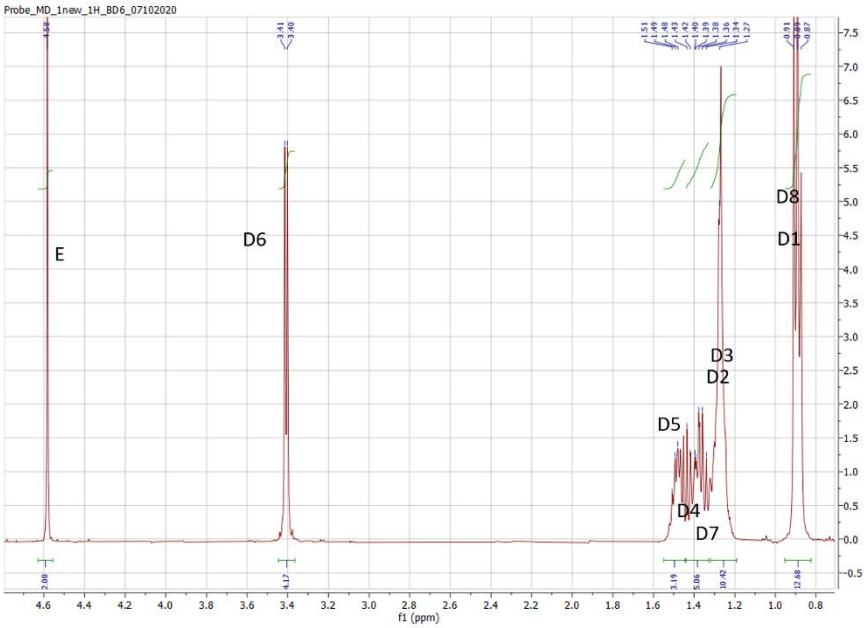


Figure B.4.2: ^{1}H-NMR spectrum for D(2-EH)M (δ in ppm, J in Hz, in C_6D_6): 4.50 (s, 2H, **E**) ; 3.40 (d, 2H, **D6**, ^{3}JH-H 4 Hz) ; 1.50-1.34 (m overlap, 8H, **D7**, **D5**) ; 1.27 (m overlap, 12H, **D2**, **D3**, **D4**) ; 0.89 (pseudo t overlap, 12H, **D1** & **D8** , ^{3}JH-H= 8 Hz).

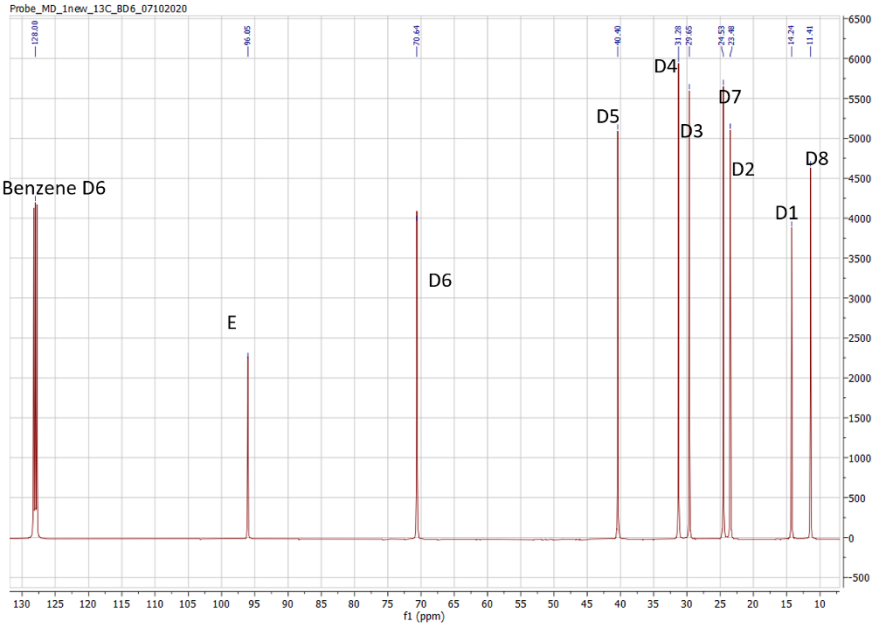


Figure B.4.3: ^{13}C-NMR spectrum for D(2-EH)M (δ in ppm, J in Hz, in C_6D_6): 96.05 ppm (**E** ^{1}JC-H= 160.9); 70.64 (**D6** ^{1}JC-H= 139.8); 40.40 (**D5** ^{1}JC-H= 126.6); 31.28 (**D4** ^{1}JC-H= 127.8); 29.65 (**D3** ^{1}JC-H= 126.9); 24.53 (**D7**, ^{1}JC-H= 126.4); 23.48 (**D2**, ^{1}JC-H= 124.5); 14.24 (**D1**, ^{1}JC-H= 124.3); 11.41 (**D8**, ^{1}JC-H= 124.6).

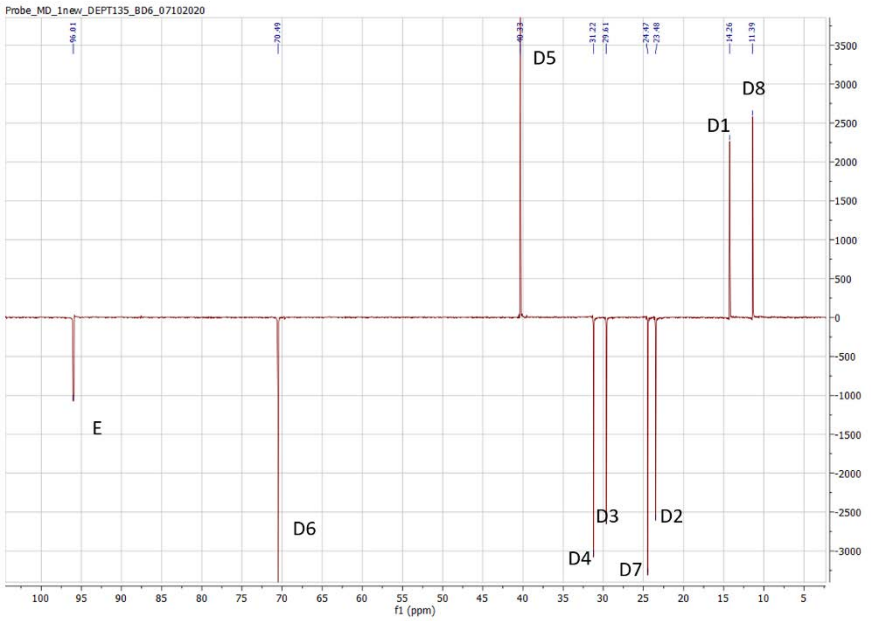


Figure B.4.4: ^{13}C ; DEPT135 spectrum for D(2-EH)M: δ (ppm, in C_6D_6).

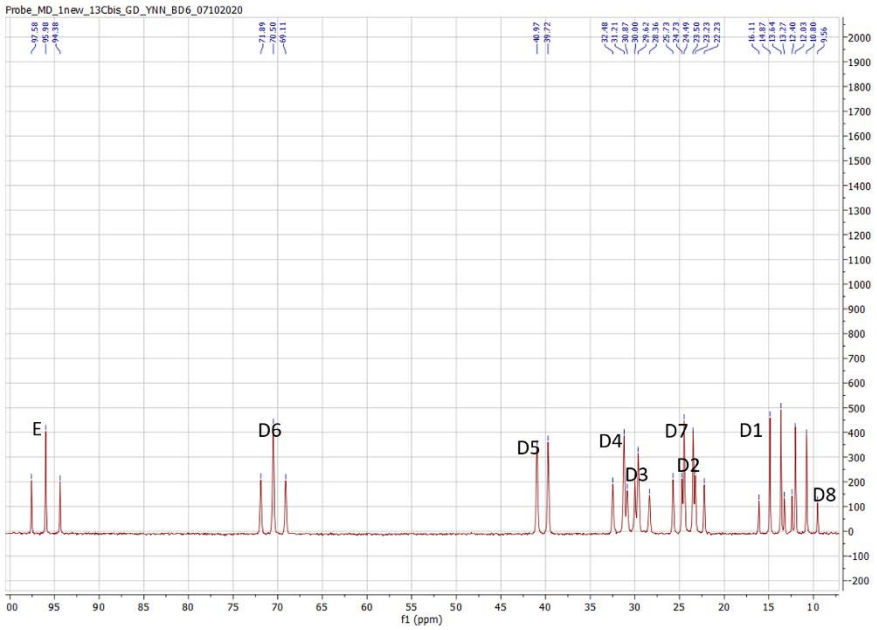


Figure B.4.5: ^{13}C spectrum recorded with ^{1}H coupling via Gated Decoupling measurement for D(2-EH)M: δ (ppm, in C_6D_6).

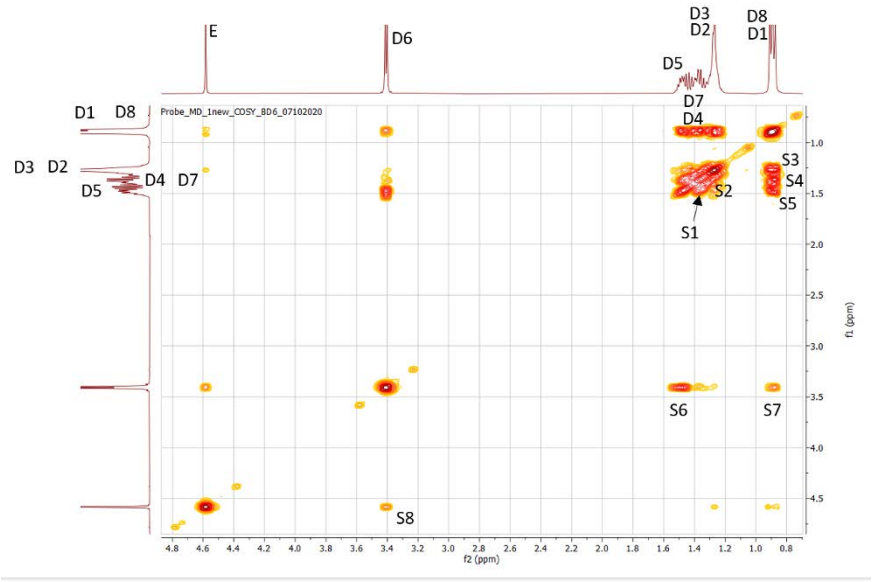


Figure B.4.6: $^{1}H,^{1}H$ COSY 2D spectrum for D(2-EH)M (δ in ppm, in C_6D_6).

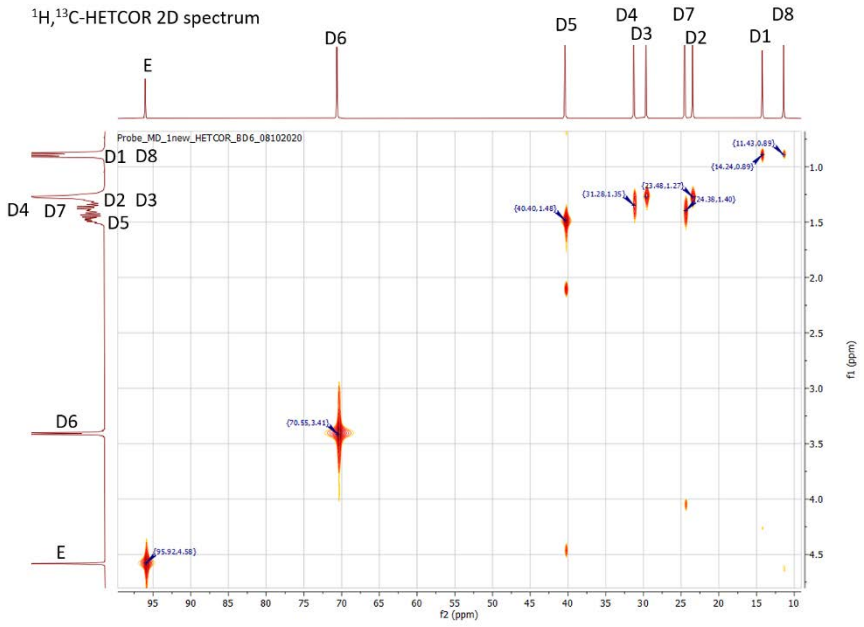


Figure B.4.7: $^{1}H,^{13}C$-HETCOR 2D spectrum for D(2-EH)M (δ in ppm, in C_6D_6).

B.5 NMR spectra for (2-EH)EM:

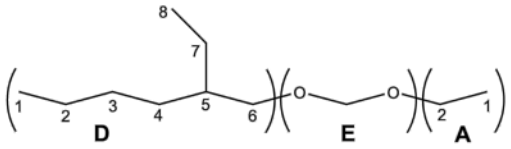


Figure B.5.1: Nomenclature for NMR analysis of (2-EH)EM.

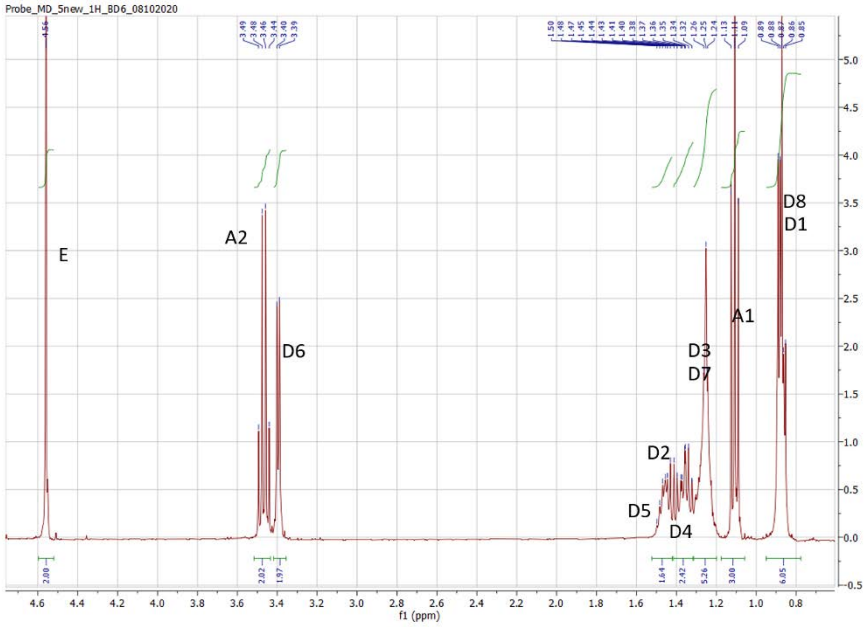


Figure B.5.2: ^{1}H-NMR spectrum for (2-EH)EM (δ in ppm, J in Hz, in C_6D_6, ref. @7.16ppm, J in Hz): 4.56 (s, 2H, **E**), 3.47 (q, 2H, **A2**, ^{3}JH,H 8.0), 3.40 (d, 2H, **D6**, ^{3}JH,H 8.0), 1.50-1.32 (m, 4H, **D5**, **D2**, **D4**), 1.25 (s, broad, 6H, **D7**, **D3**), 1.11 (t, 2H, **A1**, ^{3}JH,H 8.0); 0.87 (m as 2x t overlap, 9H, **D1**, **D8**).

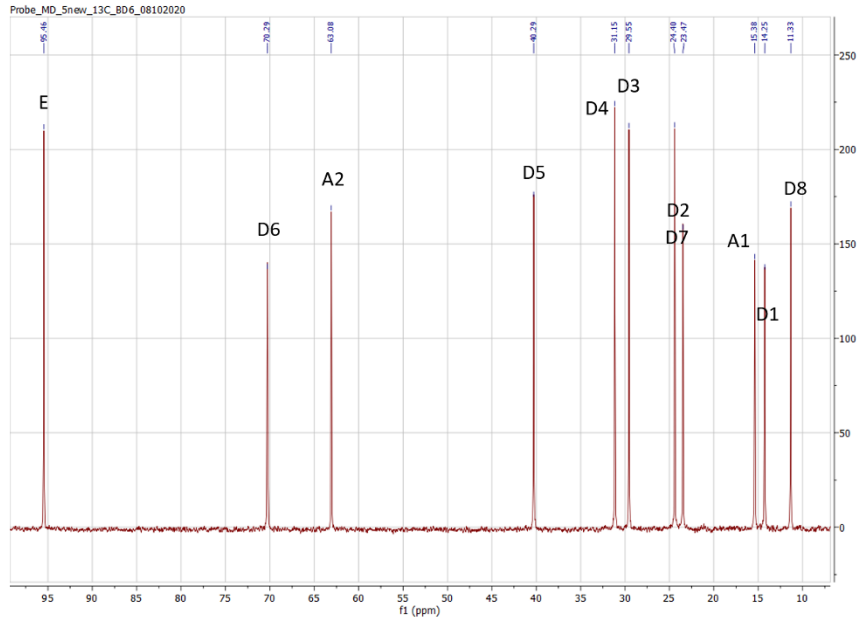


Figure B.5.3: ^{13}C-NMR spectrum for (2-EH)EM (δ in ppm, J in Hz, in C_6D_6): 95.46 ppm (**E**, ^{1}JC-H= 160.9), 70.29 (**D6**, ^{1}JC-H= 139.6), 63.08 (**A2**, ^{1}JC-H= 139.3), 40.29 (**D5**, ^{1}JC-H= 124.1), 31.15 (**D4**, ^{1}JC-H= 124.2), 29.55 (**D3**, ^{1}JC-H= 126.6), 24.40 (**D2**, ^{1}JC-H= 124.2), 23.48 (**D7**, ^{1}JC-H= 124.2), 15.38 (**A1**, ^{1}JC-H= 125.8), 14.25 (**D1**,^{1}JC-H= 124.3), 11.33 (**D8**, ^{1}JC-H= 124.5).

B.6 NMR spectra for (2-EH)PM:

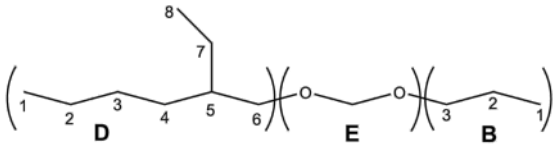


Figure B.6.1: Nomenclature for NMR analysis of (2-EH)PM.

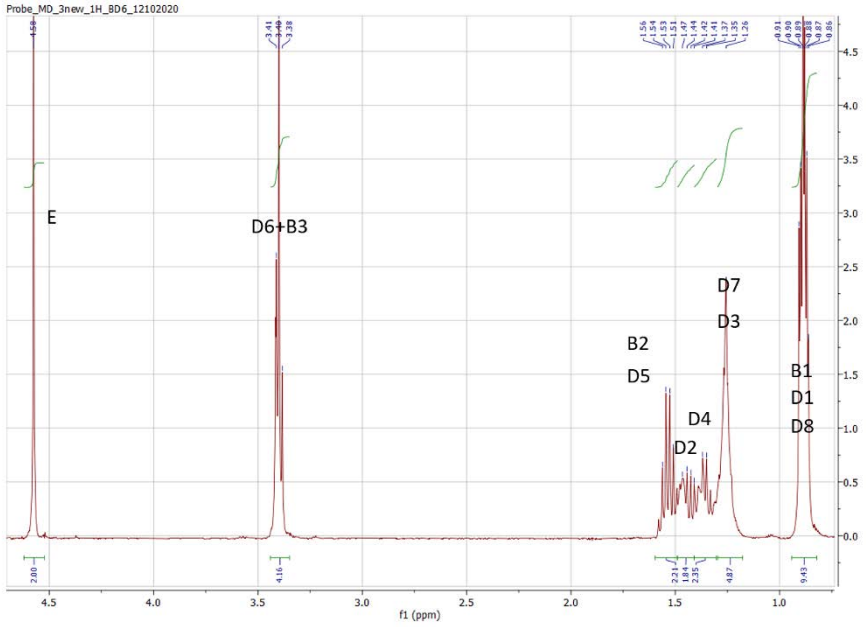


Figure B.6.2: ^{1}H-NMR spectrum for (2-EH)PM (δ in ppm, J in Hz, in C_6D_6): 4.58 (s, 2H, **E**), 3.40 (t overlap, 4H, **D6**, **B3** ^{3}JH,H 8.0 Hz), 1.56-1.33 (m overlap, 6H, **B2**, **D2**, **D4**, **D5**); 1.26 (m overlap, 5H, **D3**, **D7**); 0.88 (m, 9H, **B1**, **D1**, **D8**).

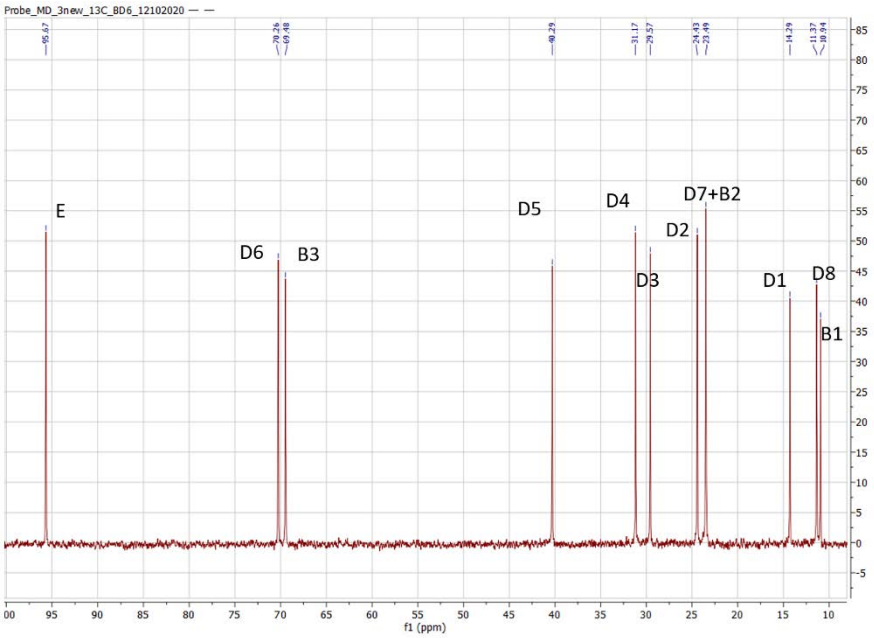


Figure B.6.3: ^{13}C-NMR spectrum for (2-EH)PM (δ in ppm, J in Hz, in C_6D_6): 95.67 ppm (**E,** ^{1}JC-H= 161.4); 70.26 (**D6,** ^{1}JC-H= 138.8); 69.48 (**B3,** ^{1}JC-H= 140.8); 40.29 (**D5,** ^{1}JC-H= 125.4); 31.17 (**D4,** ^{1}JC-H= 128.1); 29.57 (**D3,** ^{1}JC-H= 126.1); 24.43 (**D2,** ^{1}JC-H= 125.4); 23.49 (**D7**+**B2,** ^{1}JC-H= ~123.4); 14.29 (**D1,** ^{1}JC-H= 124.8); 11.37 (**D8,** ^{1}JC-H= 124.8); 10.94 (**B1,** ^{1}JC-H= 124.8).

B.7 NMR spectra for (2-EH)BM:

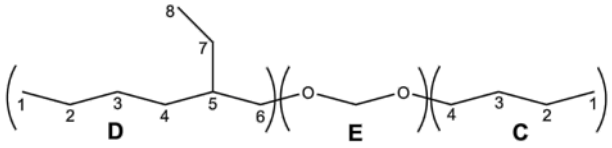


Figure B.7.1: Nomenclature for NMR analysis of (2-EH)BM.

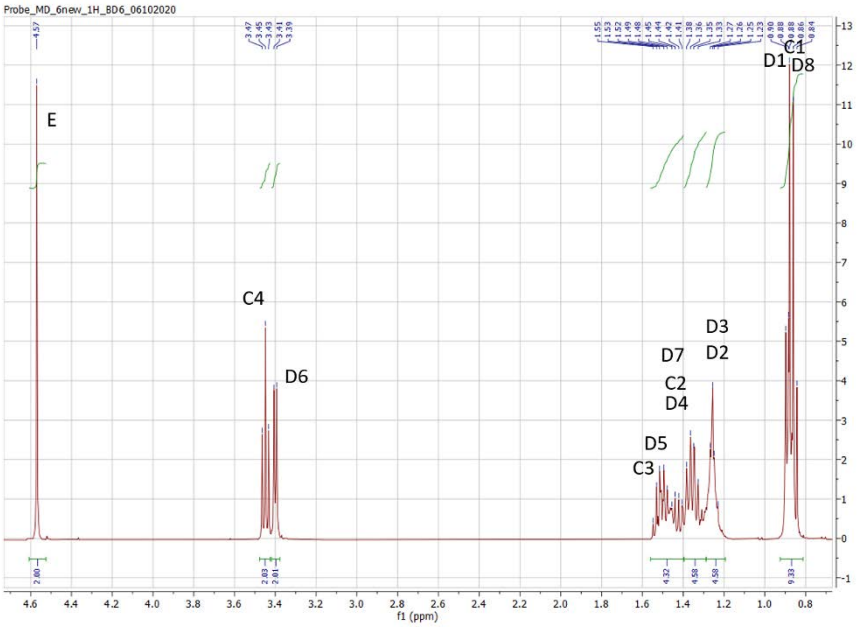


Figure B.7.2: ^{1}H-NMR spectrum for (2-EH)BM (δ in ppm, J in Hz, in C_6D_6): 4.57 (s, 2H, **E**), 3.45 (t, 2H, **C4**, ^{3}JH,H 8.0), 3.40 (d, 2H, **D6**, ^{3}JH,H 8.0), 1.55-1.33 (m, 8H, **C3**, **D5**, **D4**, **D7**), 1.26 (s, broad, 4H, **D3**, **D2**), 0.81 (m as 2x t overlap, 9H, **D1**, **C1**, **D8**).

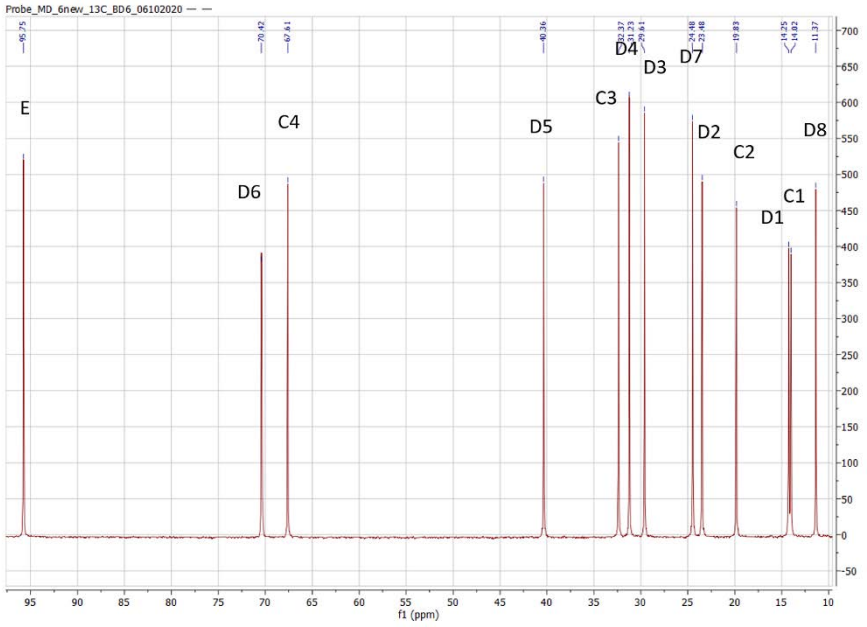


Figure B.7.3: ^{13}C-NMR spectrum for (2-EH)BM (δ in ppm, J in Hz, in C_6D_6): 95.75 ppm (**E,** ^{1}JC-H= 161.2), 70.41 (**D6,** ^{1}JC-H= 139.8), 67.61 (**C4,** ^{1}JC-H= 140.4), 40.36 (**D5,** ^{1}JC-H= 126.3), 32.37 (**C3,** ^{1}JC-H= ~122), 31.23 (**D4,** ^{1}JC-H= 126.1), 29.61 (**D3,** ^{1}JC-H= 122.1), 24.48 (**D7,** ^{1}JC-H= 124.1), 23.48 (**D2**, 124.1), 19.83 (**C2,** ^{1}JC-H= 122.1), 14.25 (**D1,** 124.1), 14.02 (**C1,** ^{1}JC-H= 124.1), 11.37 (**D8,** ^{1}JC-H= 124.5).

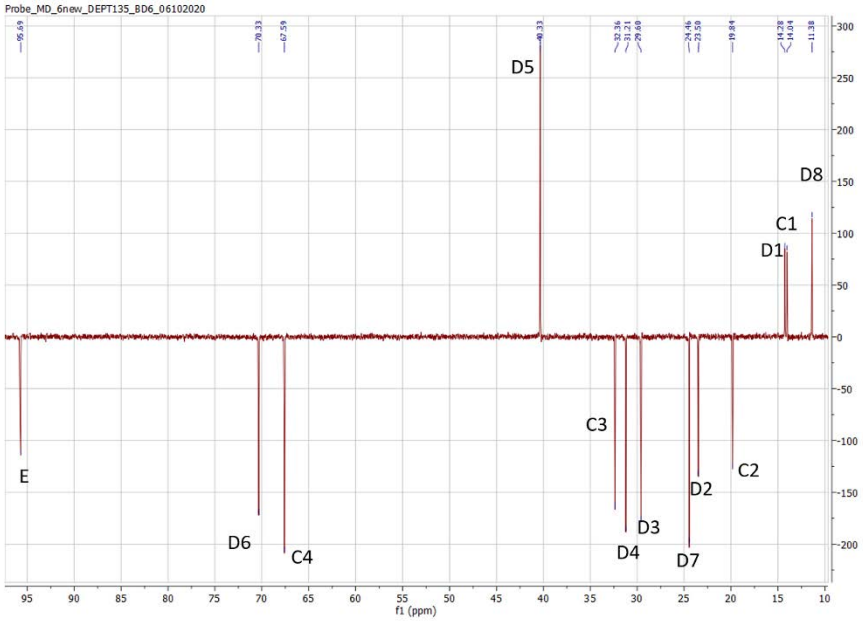


Figure B.7.4: ^{13}C ; DEPT135 spectrum for (2-EH)BM: δ (ppm, in C_6D_6).

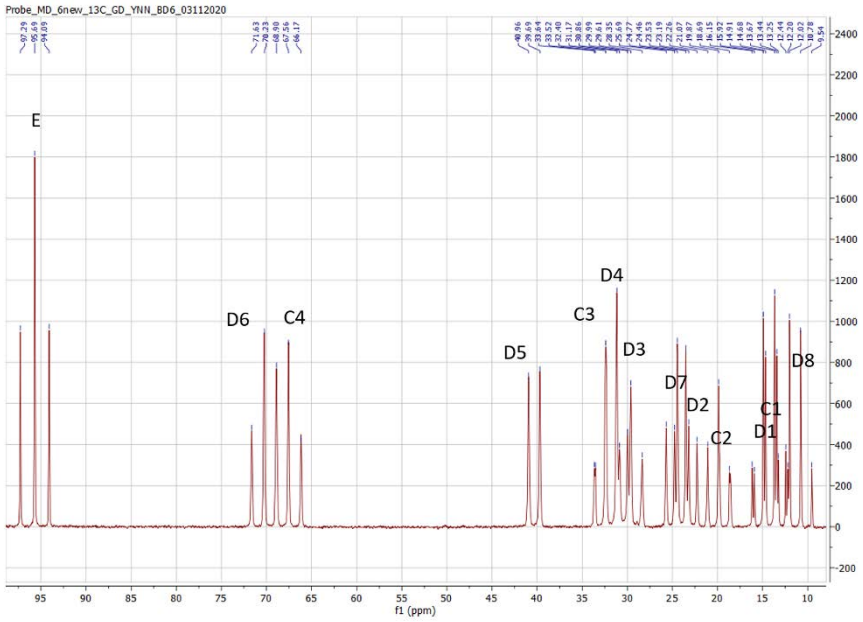


Figure B.7.5: ^{13}C spectrum recorded with ^{1}H coupling via Gated Decoupling measurement for (2-EH)BM (δ in ppm, in C_6D_6).

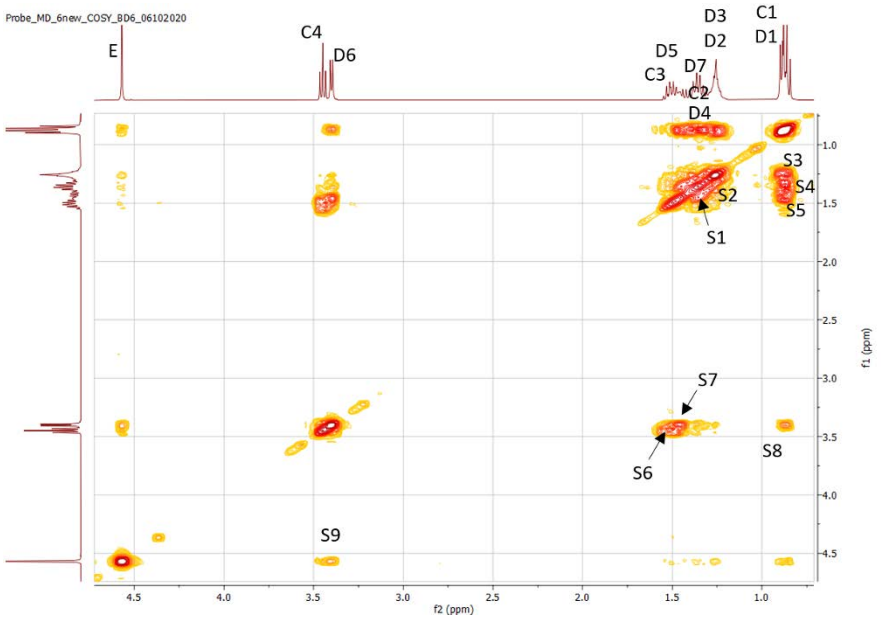


Figure B.7.6: $^{1}H,^{1}H$ COSY 2D spectrum for (2-EH)BM (δ in ppm, in C_6D_6).

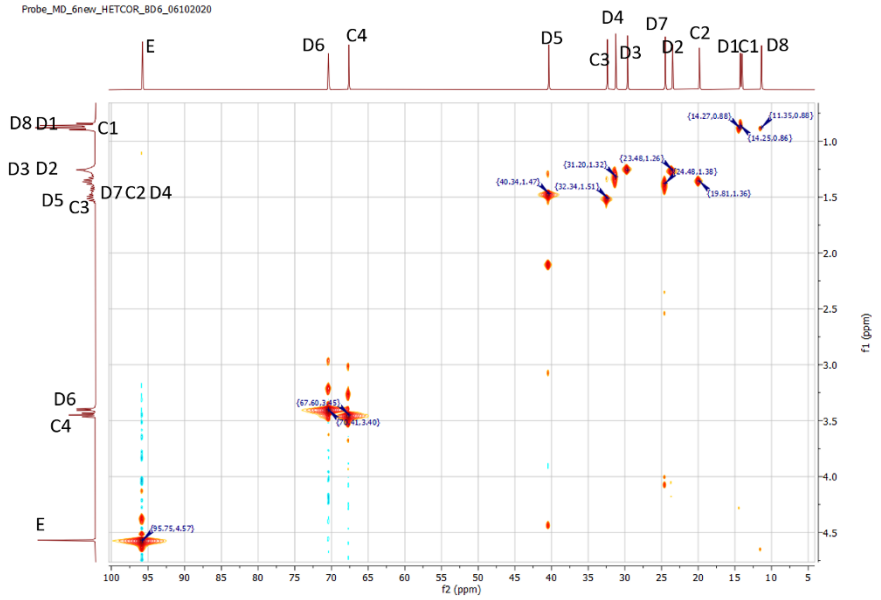


Figure B.7.7: $^{1}H,^{13}C$-HETCOR 2D spectrum for (2-EH)BM (δ in ppm, in C_6D_6).

C. FTIR spectra:

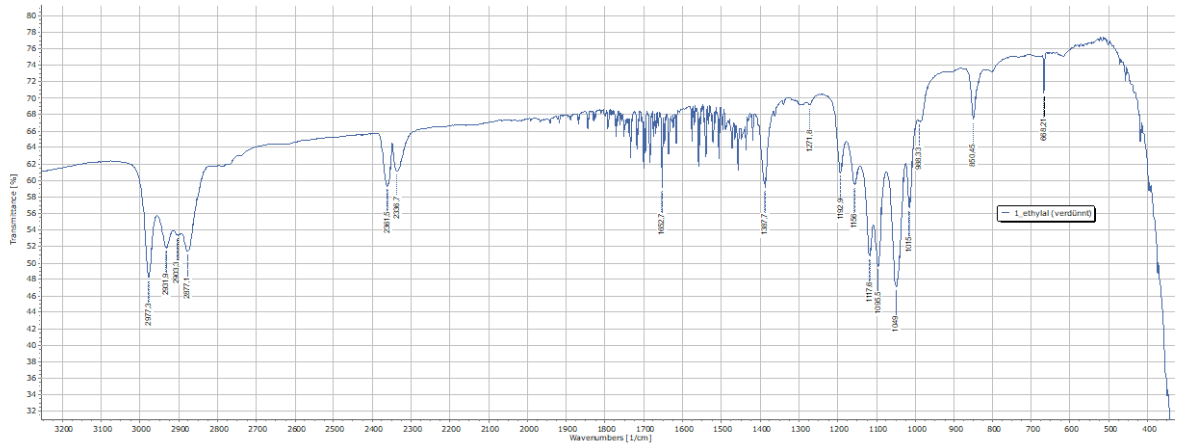


Figure C.1: FTIR spectrum of DEM.

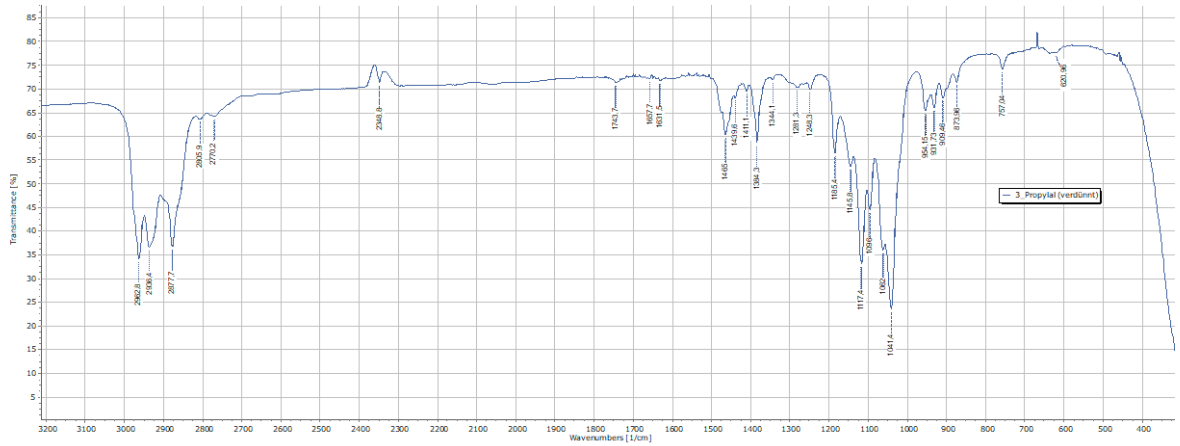


Figure C.2: FTIR spectrum of DPM.

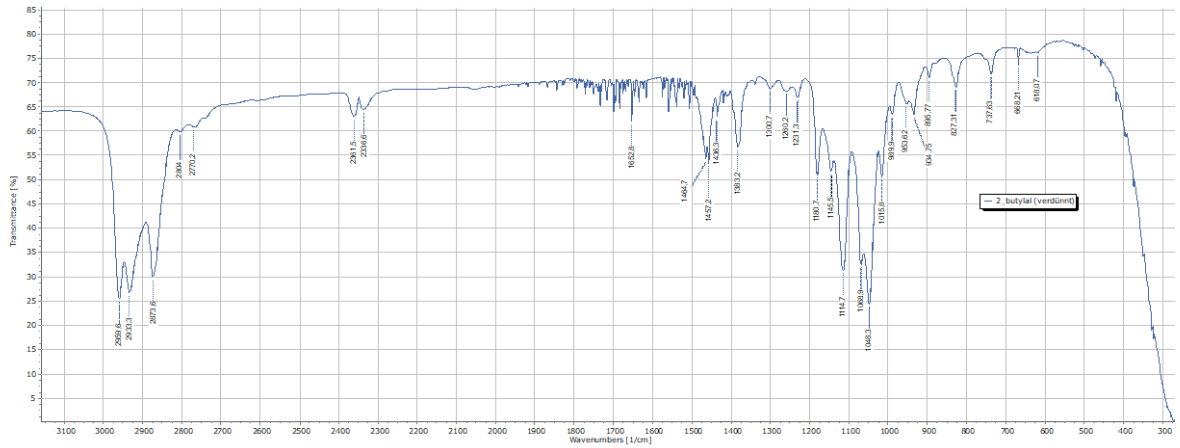


Figure C.3: FTIR spectrum of DBM.

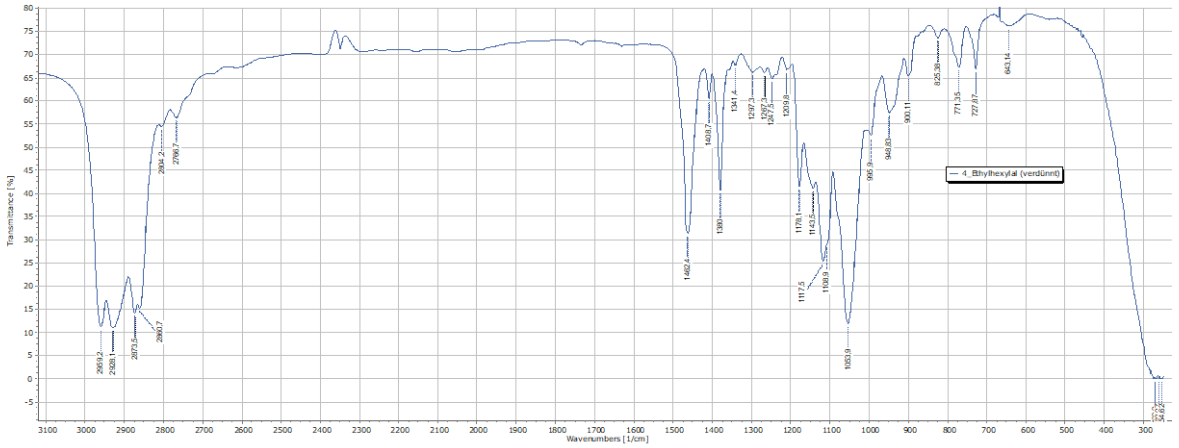


Figure C.4: FTIR spectrum of D(2-EH)M.

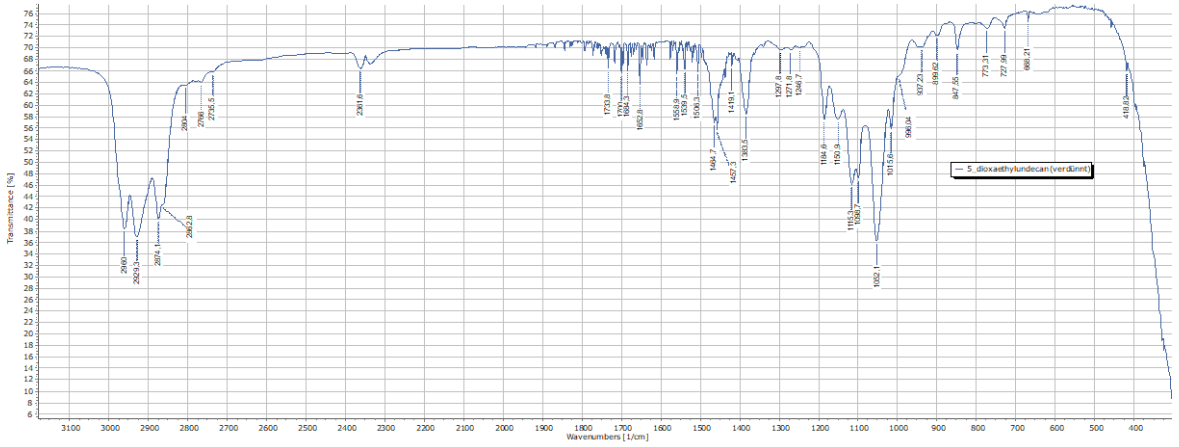


Figure C.5: FTIR spectrum of (2-EH)EM.

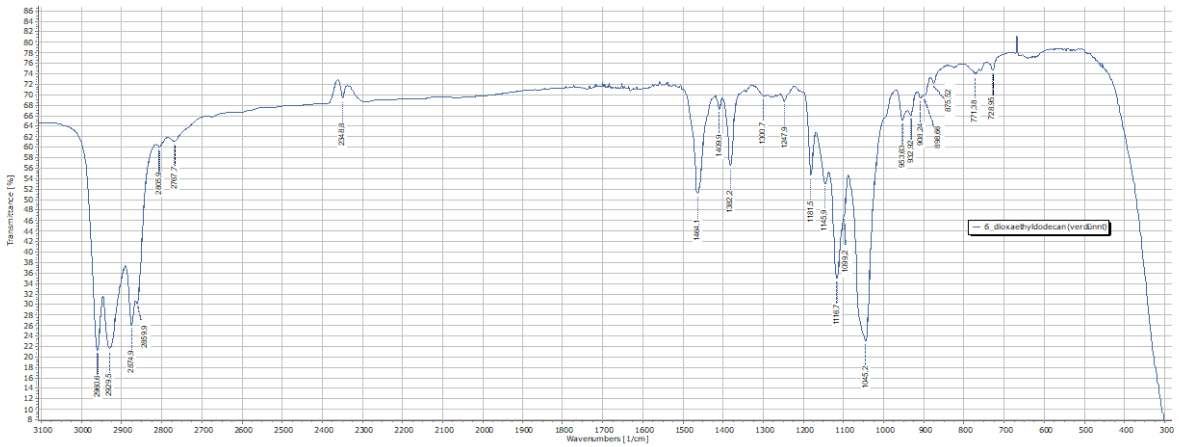


Figure C.6: FTIR spectrum of (2-EH)PM.

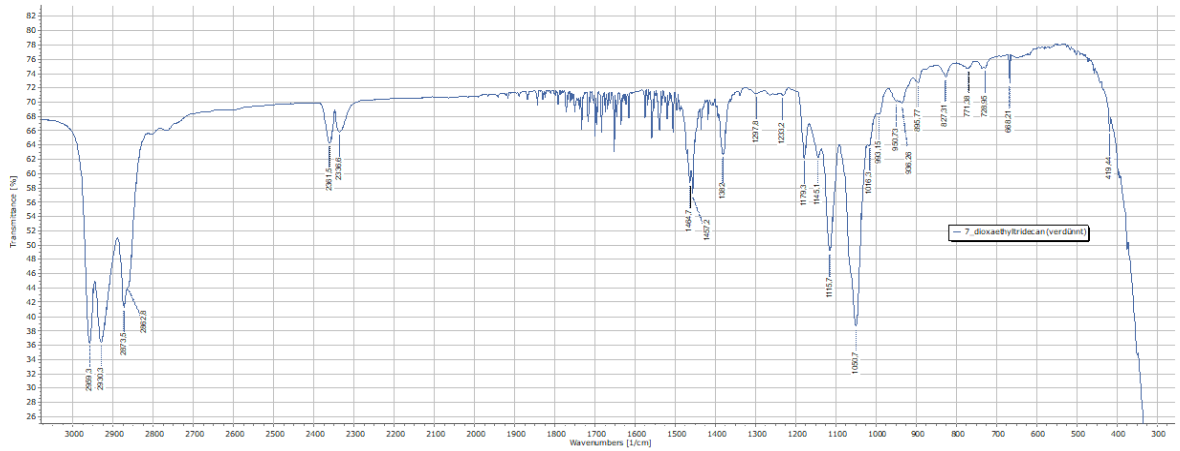


Figure C.7: FTIR spectrum of (2-EH)BM.

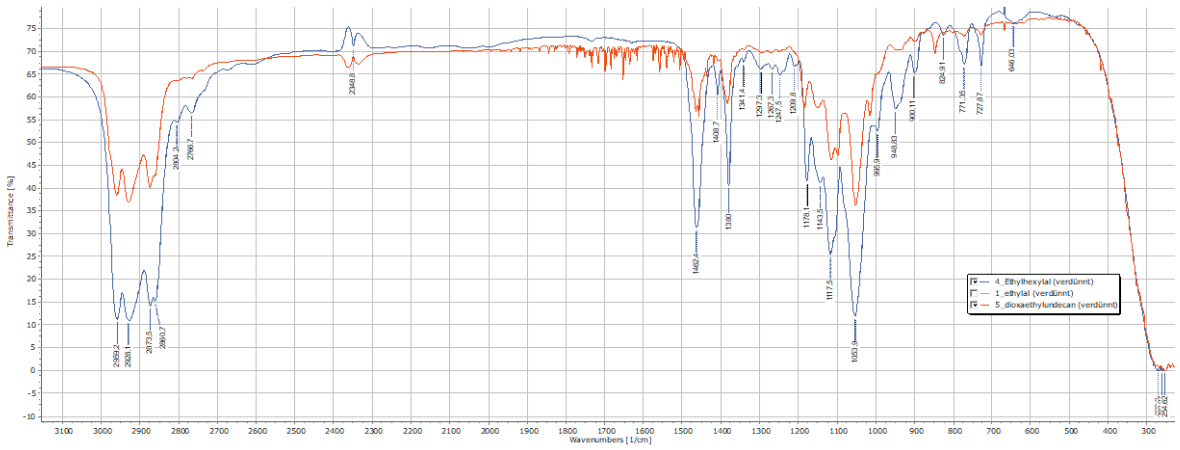


Figure C.8: Comparison of the FTIR spectra of D(2-EH)M and (2-EH)EM.

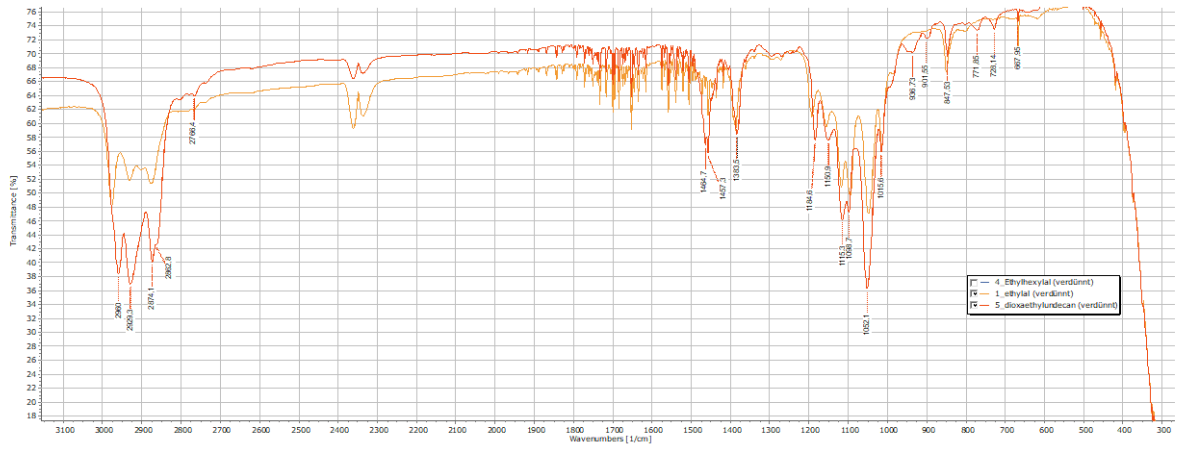


Figure C.9: Comparison of the FTIR spectra of DEM and (2-EH)EM.

D. Mass spectra:

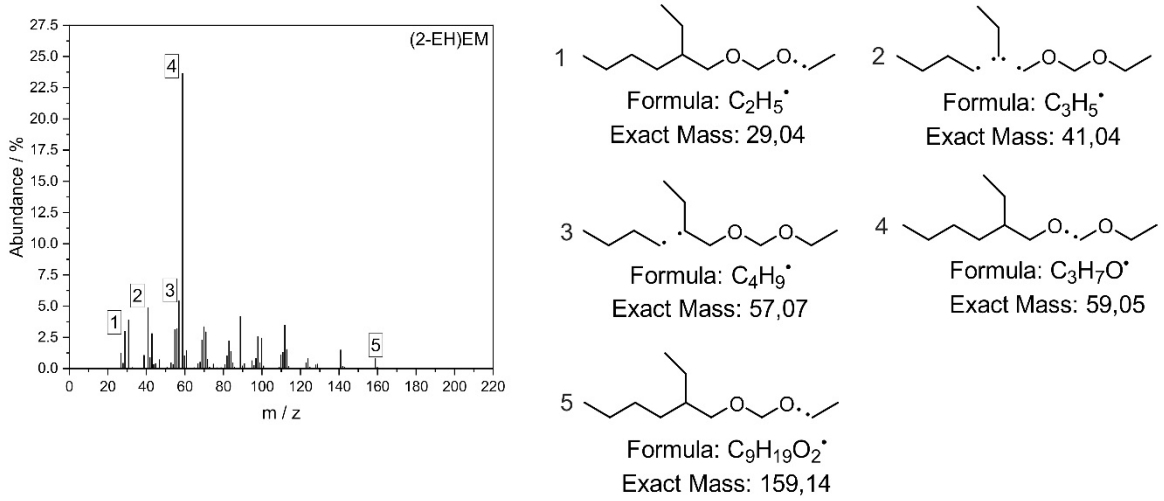


Figure D.1: Mass spectrum of (2-EH)EM and relevant fragments.

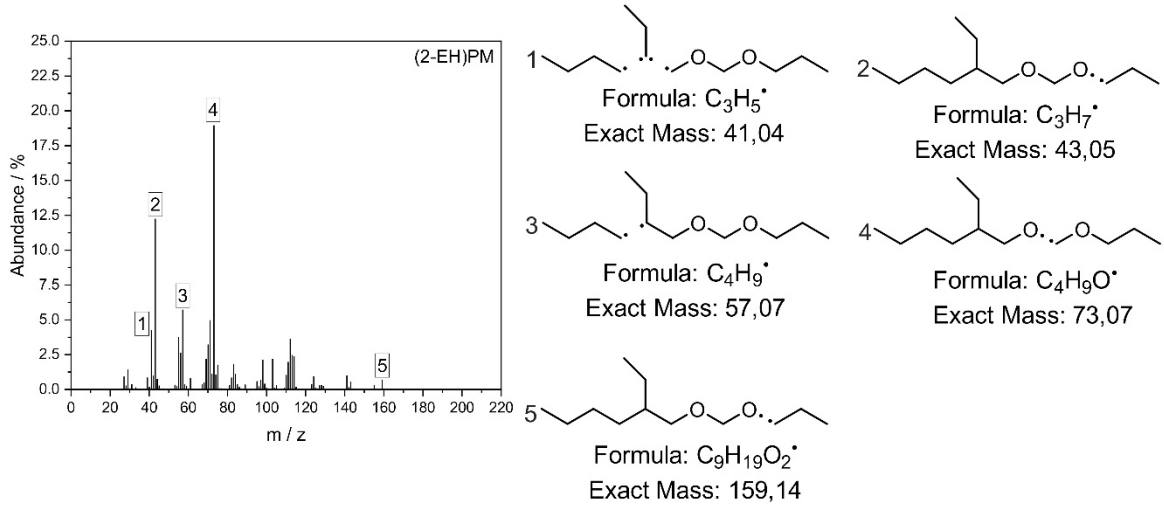


Figure D.2: Mass spectrum of (2-EH)PM and relevant fragments.

E. Tables:

Table E.1: Molecular structure, CAS number, chemical formula, molar mass and oxygen content of OMDMEs and n-alkanes.

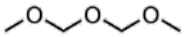
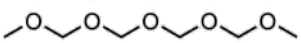
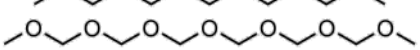
Compound	Molecular structure	CAS No.	Formula	Molar mass	Oxygen content
				g/mol	wt%
DMM ($OMDME_1$)		109-87-5	$C_3H_8O_2$	76.1	42.05
$OMDME_2$		628-90-0	$C_4H_{10}O_3$	106.1	45.24
$OMDME_3$		13353-03-2	$C_5H_{12}O_4$	136.2	46.99
$OMDME_4$		13352-75-5	$C_6H_{14}O_5$	166.2	48.13
$OMDME_5$		13352-76-6	$C_7H_{16}O_6$	196.2	48.93
$OMDME_6$		13352-77-7	$C_8H_{18}O_7$	226.2	49.51
n-heptane		142-82-5	C_7H_{16}	100.21	0.00
n-nonane		111-84-2	C_9H_{20}	128.26	0.00
n-undecane		1120-21-4	$C_{11}H_{24}$	156.31	0.00
n-tridecane		629-50-5	$C_{13}H_{28}$	184.37	0.00
n-tetradecane		629-59-4	$C_{14}H_{30}$	198.39	0.00
n-pentadecane		629-62-9	$C_{15}H_{32}$	212.42	0.00
n-nonadecane		629-92-5	$C_{19}H_{40}$	268.53	0.00

Table E.2: Physico-chemical properties of OMDMEs and n-alkanes.

Compound	Density at 20 °C	Molar volume	Melting point	Boiling point	Refractive index
	kg/m³	cm³/mol	°C	°C	-
DMM ($OMDME_1$)	859.3 [1]	88.6	-105.0 [1]	42.0 [1]	1.351 [1]
$OMDME_2$	971.0 [2 a]	109.3	-69.7 [3]	105.0 [3]	1.384 [4]
$OMDME_3$	1030.5 [4]	132.1	-42.5 [3]	155.9 [3]	1.396 [4]
$OMDME_4$	1073.7 [4]	154.8	-9.8 [3]	201.8 [3]	1.406 [4]
$OMDME_5$	1105.7 [4]	177.5	18.3 [3]	242.3 [3]	1.413 [4]
$OMDME_6$	1140.0 [5]	198.5	38.0 [5]	273.0 [5]	-
n-heptane	683.7 [6]	146.6	-90.6 [6]	98.4 [6]	1.386 [1 a]
n-nonane	719.2 [1]	178.3	-53.5 [6]	150.8 [6]	1.406 [1]
n-undecane	740.2 [1]	211.2	-25.6 [6]	195.9 [6]	1.416 [1]
n-tridecane	756.4 [1]	243.7	-5.3 [6]	235.4 [6]	1.426 [1]
n-tetradecane	759.6 [1]	261.2	6.0 [6]	253.5 [6]	1.429 [1]
n-pentadecane	768.5 [1]	276.4	10.0 [6]	270.6 [6]	1.432 [1]
n-nonadecane	785.5 [1]	341.9	32.0 [6]	330.1 [6]	1.441 [1]

[a] at 25 °C

Table E.3: Fuel properties of OMDMEs and n-alkanes.

Compound	Cetane number	Autoignition point	Flash point	Kinematic viscosity at 20 °C	HFRR	CFPP	Surface tension
	-	°C	°C	mm²/s	µm	°C	mN/m
DMM ($OMDME_1$)	28.0 [7 a]	237 [1]	-32 [1]	0.36 [4 b]	759 [8]	-	20.4 [9]
$OMDME_2$	68.0 [7 a]	230 [2]	16 [2]	0.56 [2 c]	545 [2 b]	-	27.0 [2 b]
$OMDME_3$	72.0 [7 a]	235 [4]	54 [4]	1.08 [4 b]	534 [4]	-	28.8 [4]
$OMDME_4$	84.0 [7 a]	235 [4]	88 [4]	1.72 [4 b]	465 [4]	-	30.7 [4]
$OMDME_5$	93.0 [7 a]	240 [4]	115 [4]	2.63 [4 b]	437 [4]	-	32.6 [4]
n-heptane	56.0 [10]	204 [10]	-7.4 [11]	0.61 [6]	-	-	20.3 [6]
n-nonane	72.0 [10]	205 [10]	28.8 [11]	0.97 [6]	-	-	22.9 [6]
n-undecane	81.0 [10]	240 [10]	61.1 [11]	1.61 [6]	-	-	24.7 [6]
n-tridecane	90.0 [10]	202 [10]	90.6 [11]	2.47 [6]	-	-	26.0 [6]
n-tetradecane	95.0 [10]	220 [10]	104.5 [11]	3.00 [6]	-	-	26.6 [6]
n-pentadecane	96.0 [10]	202 [10]	117.9 [11]	3.58 [6]	-	-	27.2 [6]
n-nonadecane	-	-	167.4 [11]	-	-	-	-

[a] determined according to IP Standard 617
[b] at 25 °C
[c] at 40 °C

Table E.4: Thermodynamic properties of OMDMEs and n-alkanes.

Compound	ΔH_f^0	ΔH_c^0	LHV	HHV
	kJ/mol	kJ/mol	MJ/kg	MJ/kg
DMM ($OMDME_1$)	-443.33	1880.52	22.40 [5]	24.71
$OMDME_2$	-597.06	2406.13	20.60 [5]	22.67
$OMDME_3$	-777.59	2904.94	19.40 [5]	21.33
$OMDME_4$	-946.33	3415.54	18.70 [5]	20.55
$OMDME_5$	-1137.87	3903.34	18.10 [5]	19.89
$OMDME_6$	-1320.19	4400.36	17.70 [5]	19.45
n-heptane	-223.98 [12]	4816.92 [12]	44.56	48.07
n-nonane	-274.97 [12]	6124.53 [12]	44.32	47.75
n-undecane	-325.96 [12]	7432.14 [12]	44.17	47.55
n-tridecane	-376.96 [12]	8739.74 [12]	44.06	47.40
n-tetradecane	-402.45 [12]	9393.55 [12]	44.02	47.35
n-pentadecane	-427.95 [12]	10047.35 [12]	43.99	47.30
n-nonadecane	-529.93 [12]	12662.57 [12]	43.88	47.16

Table E.5: Joback groups for the description of the investigated OMDAEs (all employed groups exhibit a non-ring structure).

Compound	Joback group			
	$-CH_3$	$-CH_2-$	>CH-	-O-
DEM	2	3	0	2
DPM	2	5	0	2
DBM	2	7	0	2
D(2-EH)M	4	11	2	2
(2-EH)EM	3	7	1	2
(2-EH)PM	3	8	1	2
(2-EH)BM	3	9	1	2
DMM	2	1	0	2
DPeM	2	9	0	2
DiBM	6	1	2	2

Table E.6: Joback parameters for the estimation of boiling points (T_b), melting points (T_m), dynamic viscosities (η_a and η_b) and enthalpies of fusion ΔH_f^0 of the investigated OMDAEs[13] (all employed groups exhibit a non-ring structure).

Estimation parameters	Joback group			
	$-CH_3$	$-CH_2-$	>CH-	-O-
T_b	23.58	22.88	21.74	22.42
T_m	-5.1	11.27	12.64	22.23
η_a	548.29	94.16	-322.15	122.09
η_b	-1.719	-0.199	1.187	-0.386
ΔH_f^0	-76.45	-20.64	29.89	-132.2

Table E.7: Data set used for fitting the regression function coefficients (part 1).

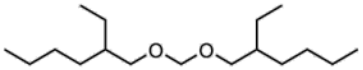
Component		Density at 20 °C	Molar volume	Boiling point	Refractive index
		kg/m³	cm³/mol	°C	-
DMM		-	88.6	42.0 [1]	1.351 [1]
DEM		829.7	125.5	87.1	1.373
DPM		834.6	158.4	135.2	1.393
DBM		835.4	191.8	178.8	1.406
DPeM		841.1 [14]	-	221.6 [15]	1.417 [14]
D(2-EH)M		848.2	321.2	285.1	1.435

Table E.8: Data set used for fitting the regression function coefficients (part 2).

Component	Molar mass	Flash point	Kinematic viscosity at 20 °C	Surface tension	ΔH_f^0	ΔH_c^0	LHV	HHV
	g/mol	°C	mm²/s	mN/m	kJ/mol	kJ/mol	MJ/kg	MJ/kg
DMM	76.1	-32.0 [1]	0.36 [4]	20.4 [9]	-443.33	1880.52	22.40 [5]	24.71
DEM	104.15	-5.0	0.52	21	-566.67	3115.86	27.38	29.92
DPM	132.20	29.5	0.85	23.1	-543.37	4497.84	31.36	34.02
DBM	160.26	60.5	1.22	24.3	-549.60	5850.29	33.76	36.51
DPeM	188.31	-	-	-	-	-	-	-
D(2-EH)M	272.47	133.5	4.56	27.1	-750.26	11084.35	37.78	40.68

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