ELECTRONIC SUPPORTING INFORMATION

"Sweet" ionic liquids comprising the acesulfame anion – synthesis, physicochemical properties and antifeedant activity towards stored product insects

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Acquisition Time (sec)	7.0000	*Date*	Jun 7 2019	*Date Stamp*	Jun 7 2019	*File Name*	D:\MOJE!!!\POBRANE\NMR\TR-WS-5.FID\FID		
Frequency (MHz)	399.91	*Nucleus*	1H	*Number of Transients*	64	*Original Points Count*	36611	*Points Count*	65536
Pulse Sequence	s2pul	*Receiver Gain*	18.00	*Solvent*	DMSO-d6	*Spectrum Offset (Hz)*	2273.0588	*Spectrum Type*	STANDARD
Sweep Width (Hz)	5230.13	*Temperature (degree C)*	AMBIENT TEMPERATURE						

^{1}H NMR (399.91 MHz, DMSO-d_6) δ [ppm] = 1.22 (t, J= 7.20 Hz, 3H, $CH_2C\underline{H}_3$), 1.90 (s, 3H, $OCC\underline{H}_3CHC$), 3.06 (s, 6H, $^+NC\underline{H}_3$), 3.40-3.54 (m, 8H, $^+NC\underline{H}_2CH_3$, $^+NC\underline{H}_2CH_2$, $OC\underline{H}_2C\underline{H}_2OH$), 3.81 (br. s, 2H, $^+NCH_2C\underline{H}_2O$), 4.71 (t, J= 5.20 Hz, 1H, $CH_2O\underline{H}$), 5.28 (s, 1H, $C{=}C\underline{H}C$).

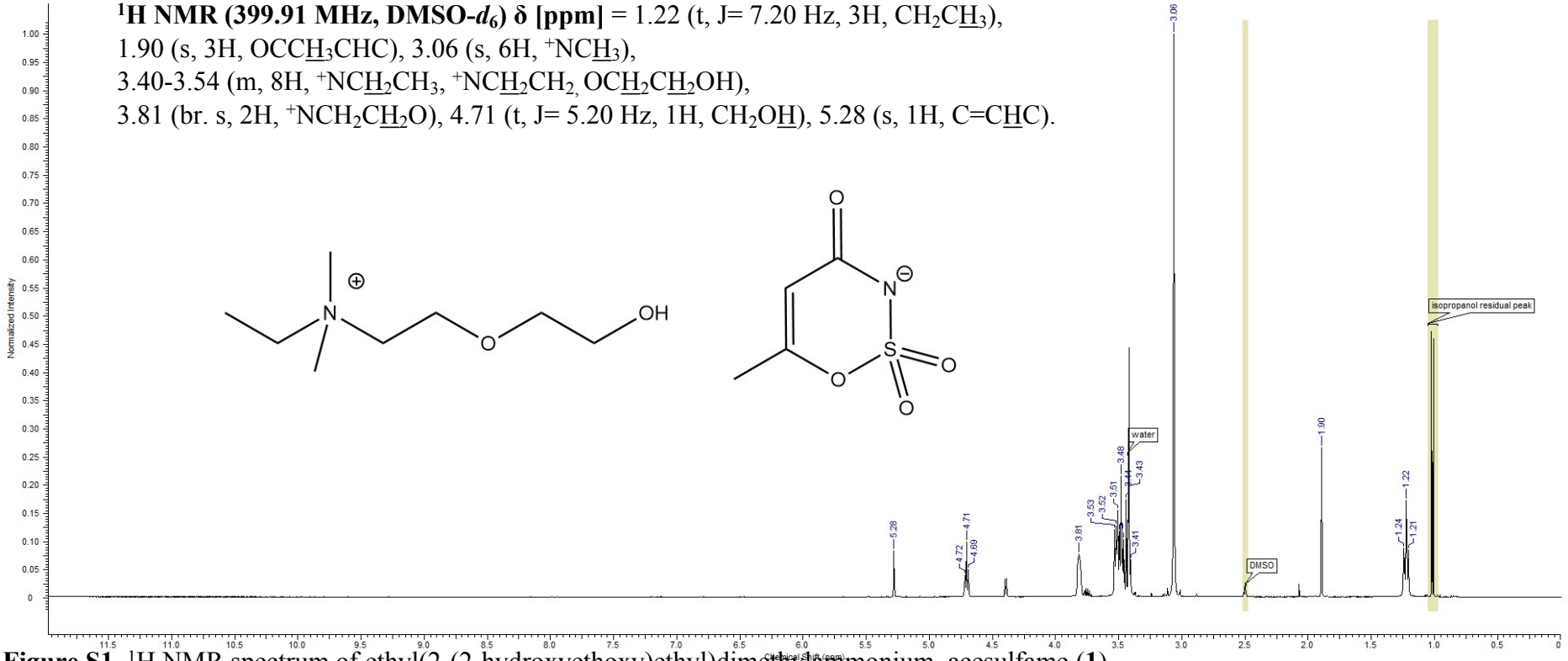


Figure S1. ^{1}H NMR spectrum of ethyl(2-(2-hydroxyethoxy)ethyl)dimethylammonium acesulfame (**1**).

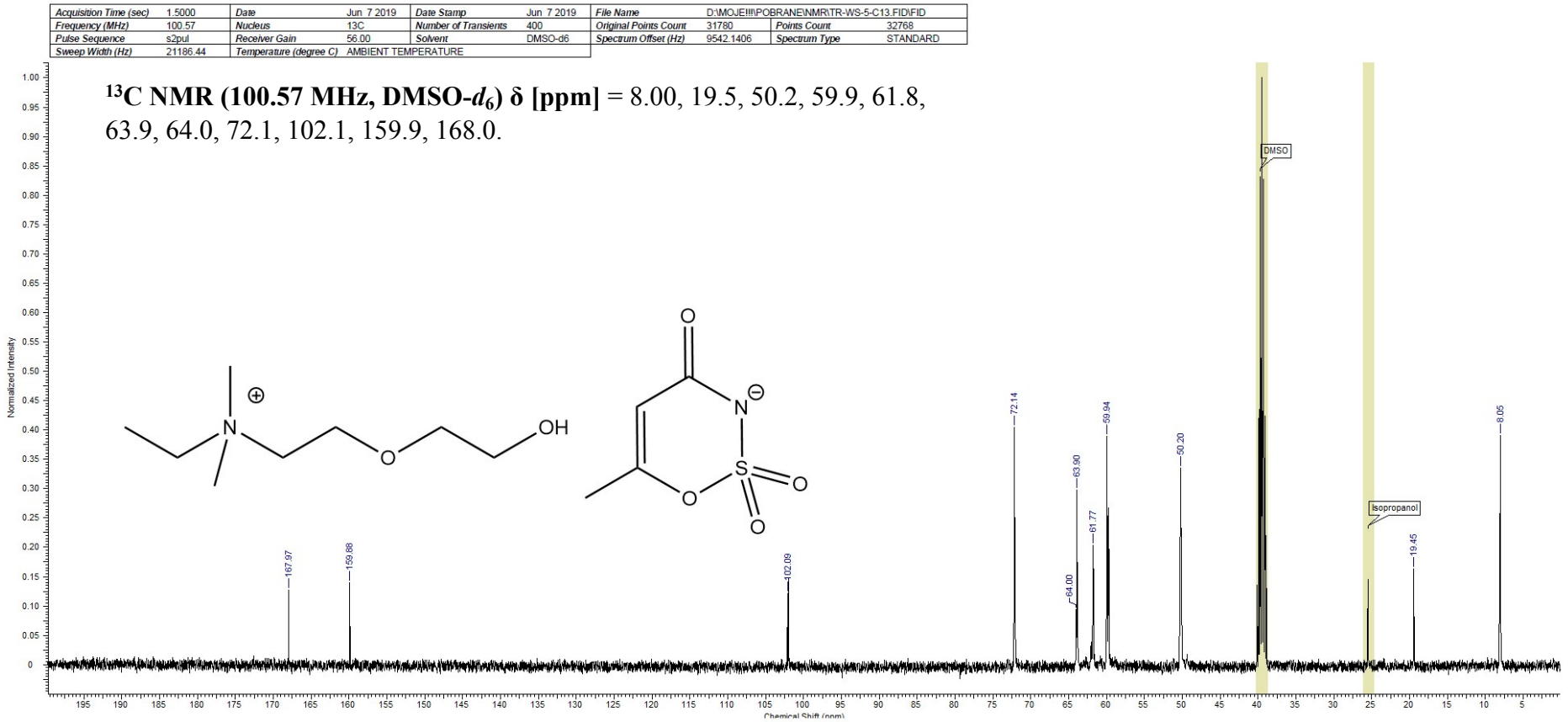


Figure S2. ^{13}C NMR spectrum of ethyl(2-(2-hydroxyethoxy)ethyl)dimethylammonium acesulfame (**1**).

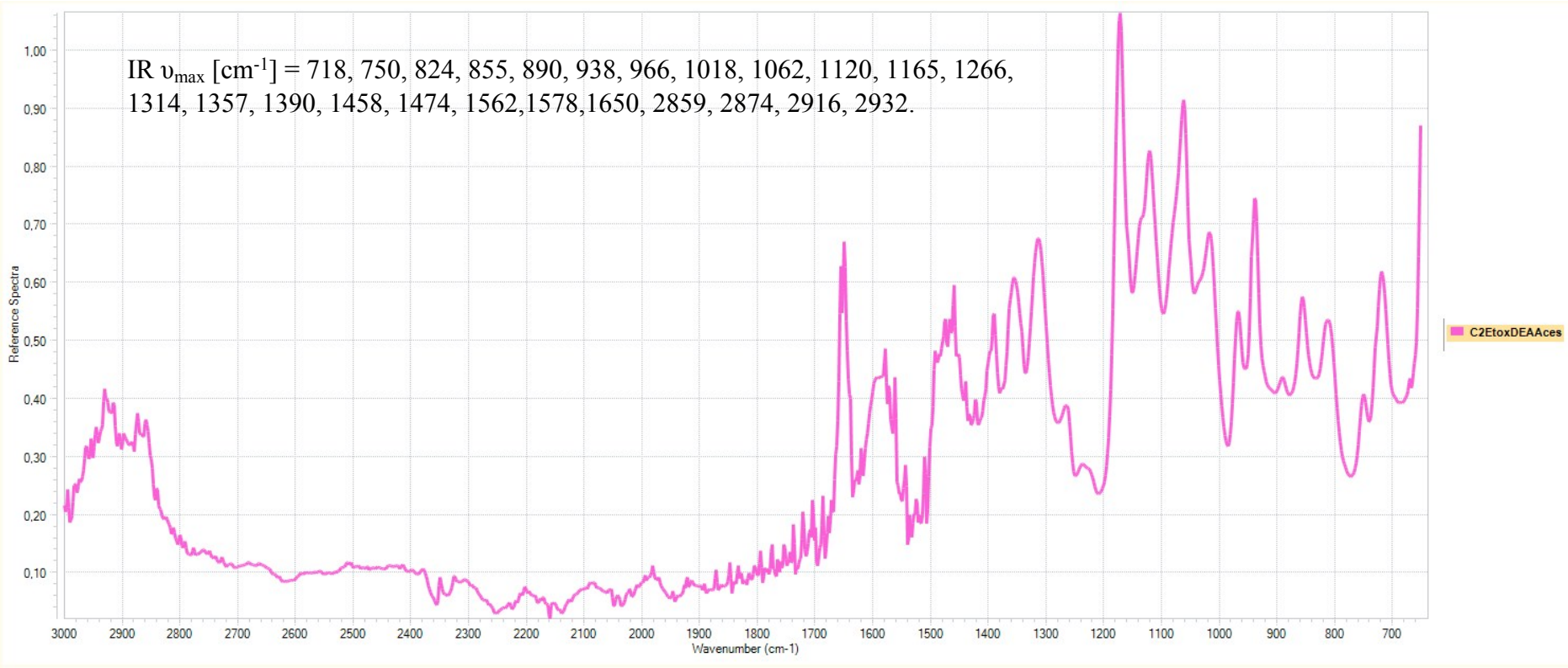
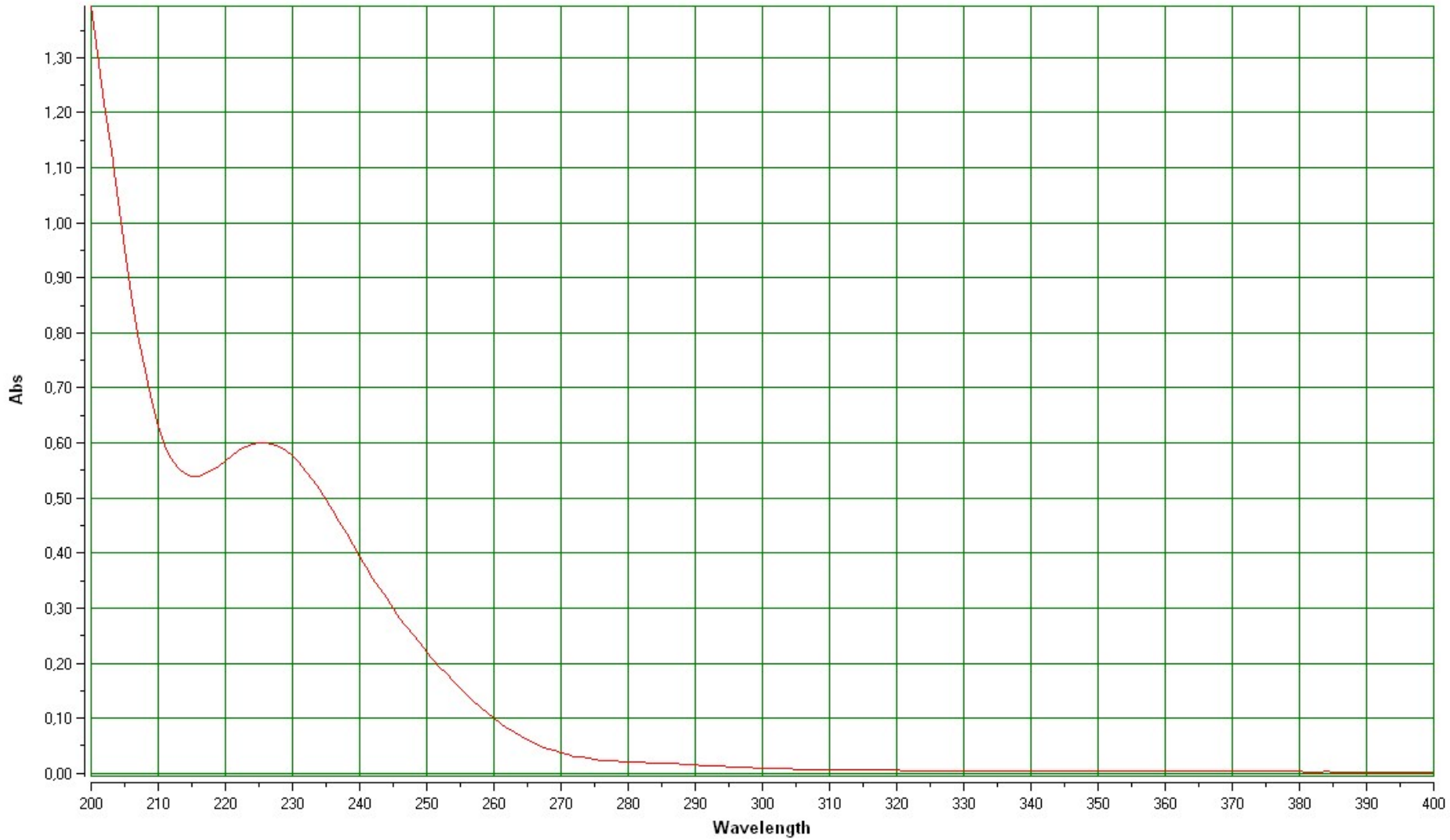


Figure S3. FT-IR spectrum of ethyl(2-(2-hydroxyethoxy)ethyl)dimethylammonium acesulfame (**1**).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Wavelength

Figure S4. UV spectrum of ethyl(2-(2-hydroxyethoxy)ethyl)dimethylammonium acesulfame (c $\approx 1{,}52 \times 10^{-4} \frac{mol}{dm^3}$) (**1**).

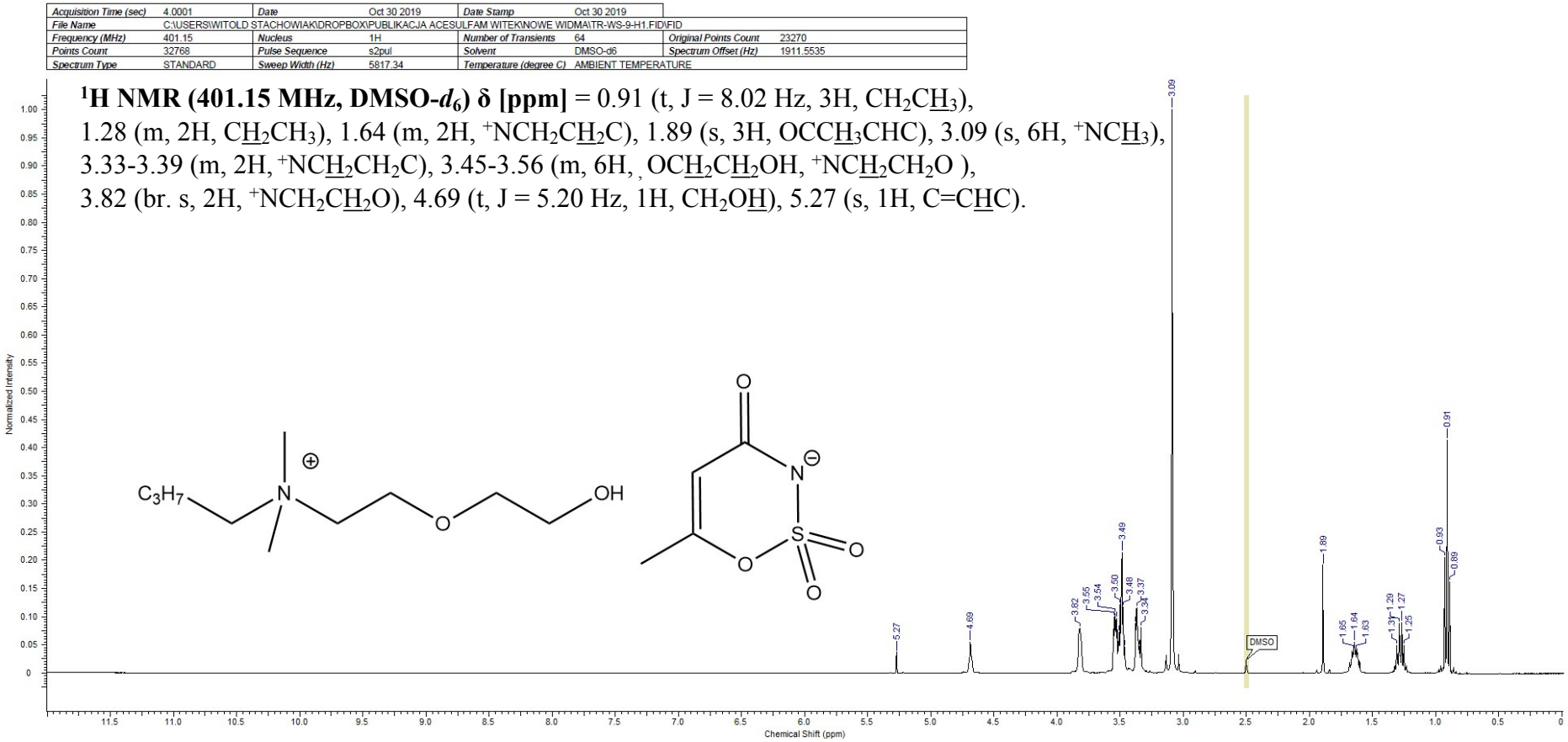


Figure S5. ^{1}H NMR spectrum of butyl(2-(2-hydroxyethoxy)ethyl)dimethylammonium acesulfame (**2**).

Acquisition Time (sec)	0.9500	*Date*	Oct 30 2019	*Date Stamp*	Oct 30 2019		
File Name	C:\USERS\WITOLD STACHOWIAK\DROPBOX\PUBLIKACJA ACESULFAM WITEK\NOWE WIDMA\TR-WS-9-C13.FID\FID						
Frequency (MHz)	100.88	*Nucleus*	13C	*Number of Transients*	2872	*Original Points Count*	24804
Points Count	32768	*Pulse Sequence*	s2pul	*Solvent*	DMSO-d6	*Spectrum Offset (Hz)*	11095.3574
Spectrum Type	STANDARD	*Sweep Width (Hz)*	26109.66	*Temperature (degree C)*	AMBIENT TEMPERATURE		

^{13}C NMR (100.88, DMSO-d_6) δ [ppm] = 13.4, 19.1, 19.4, 23.8, 50.7, 59.9, 62.2, 63.9, 72.1, 102.0, 159.6, 167.8.

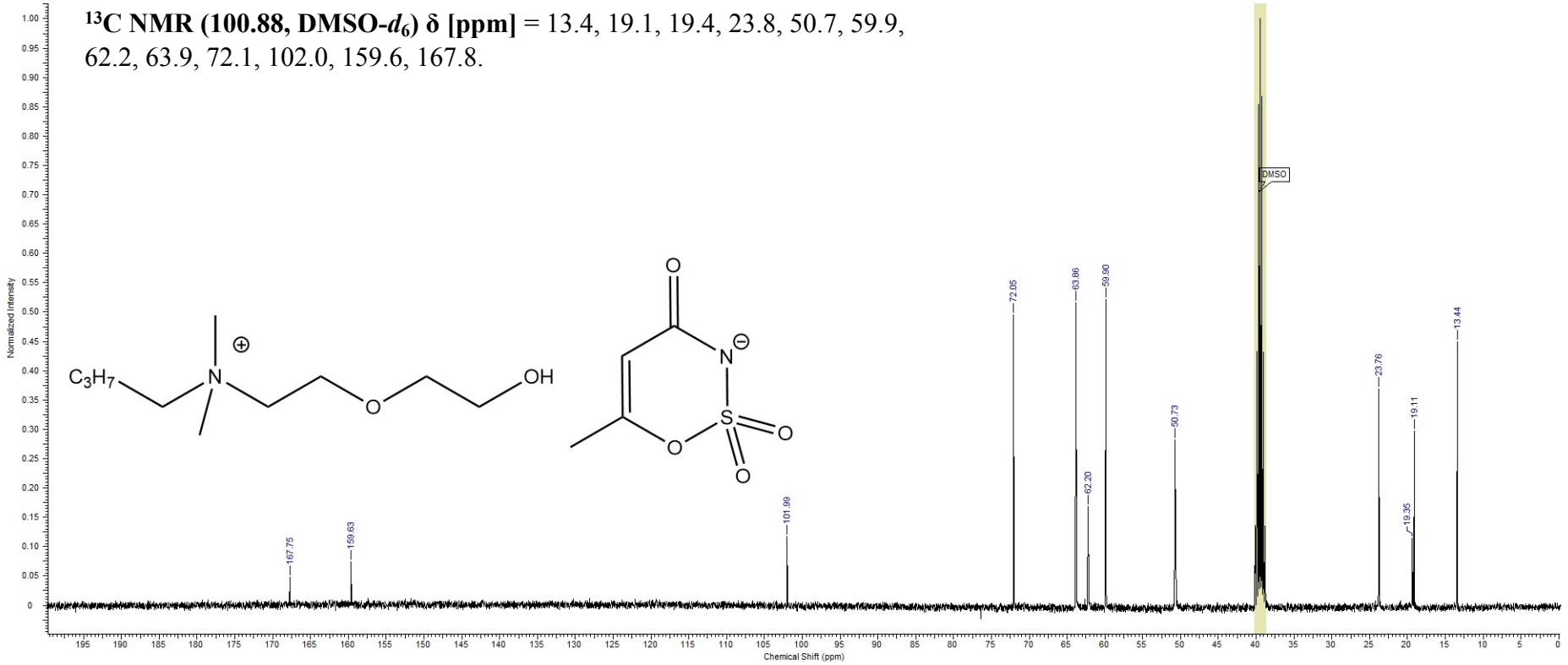


Figure S6. ^{13}C NMR spectrum of butyl(2-(2-hydroxyethoxy)ethyl)dimethylammonium acesulfame (**2**).

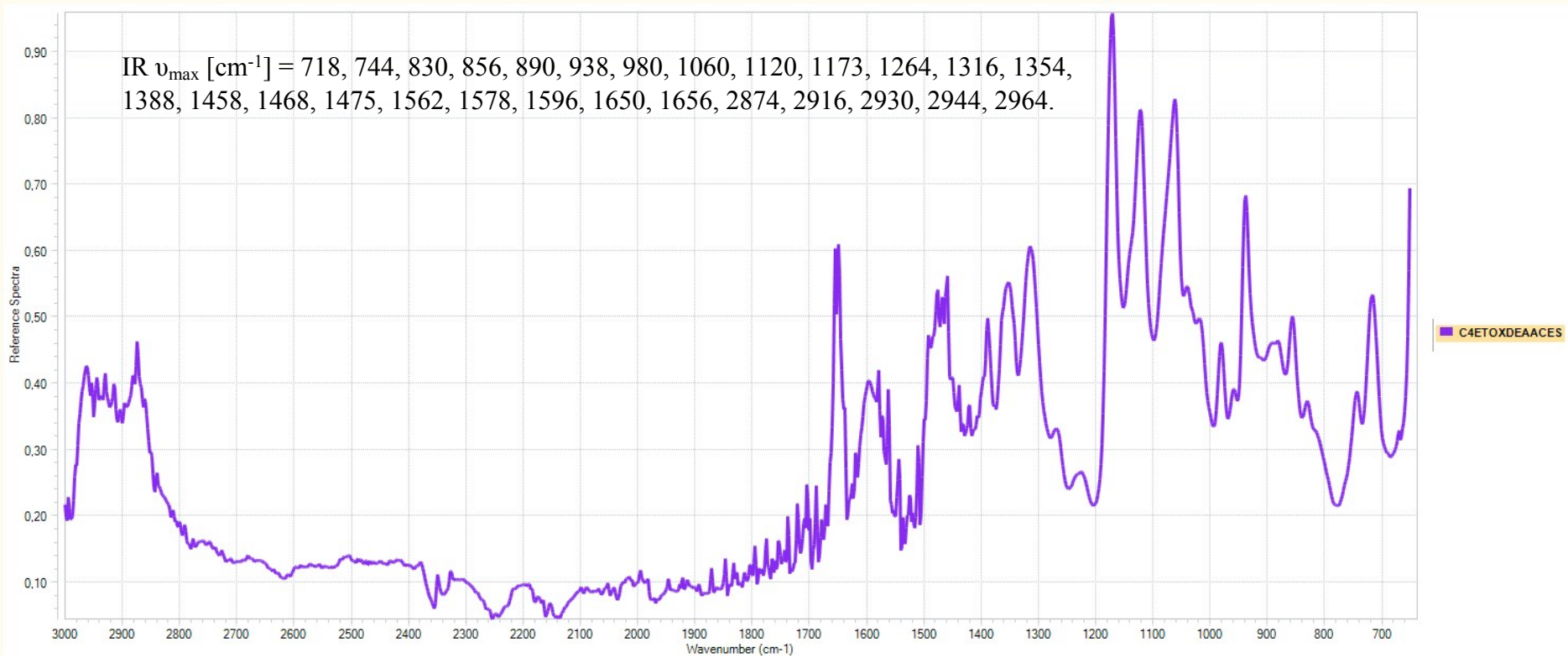


Figure S7. FT-IR spectrum of butyl(2-(2-hydroxyethoxy)ethyl)dimethylammonium acesulfame (**2**).

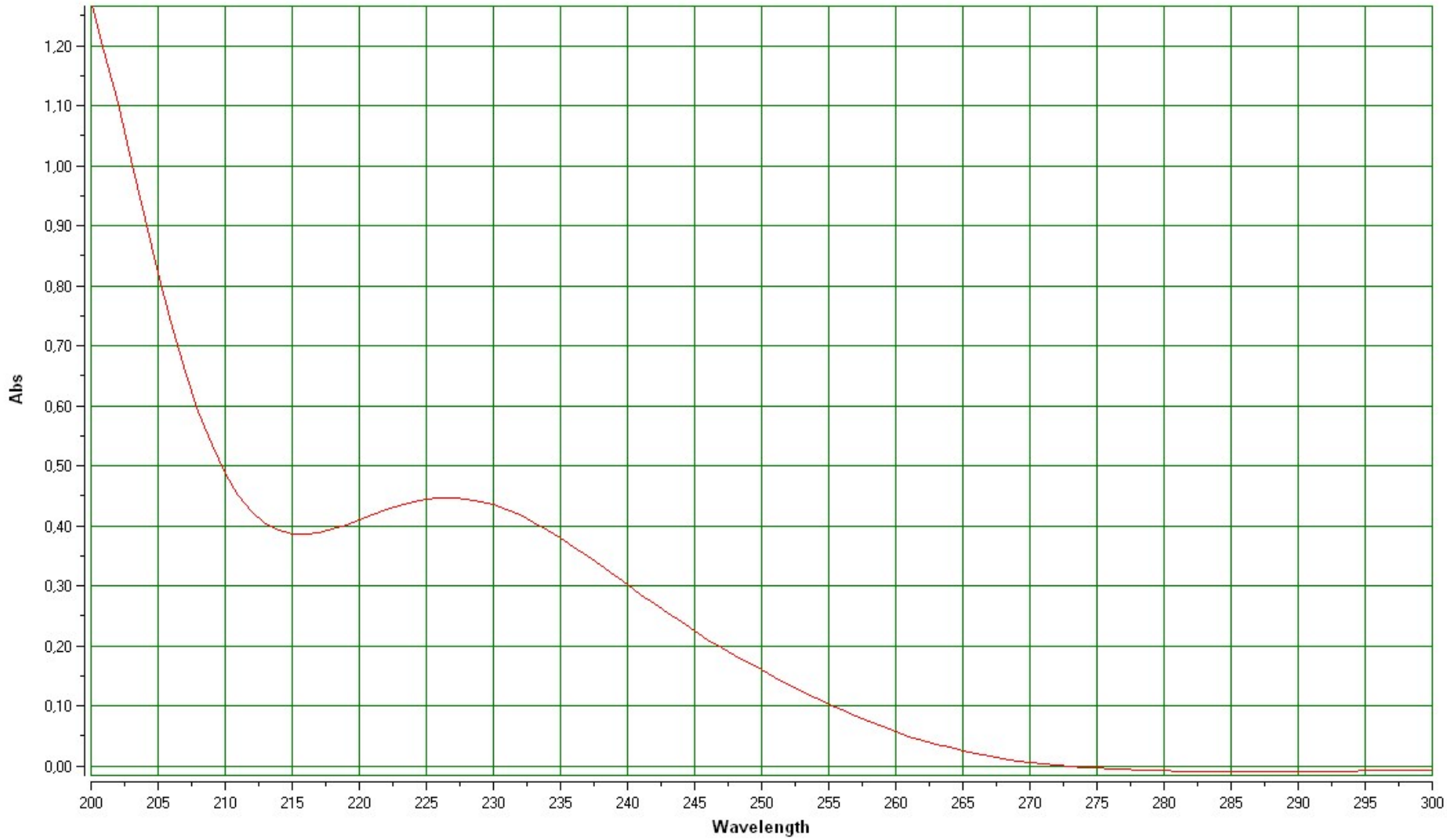


Figure S8. UV spectrum of butyl(2-(2-hydroxyethoxy)ethyl)dimethylammonium acesulfame (c $\approx 1{,}14 \times 10^{-4} \frac{mol}{dm^3}$) (**2**).

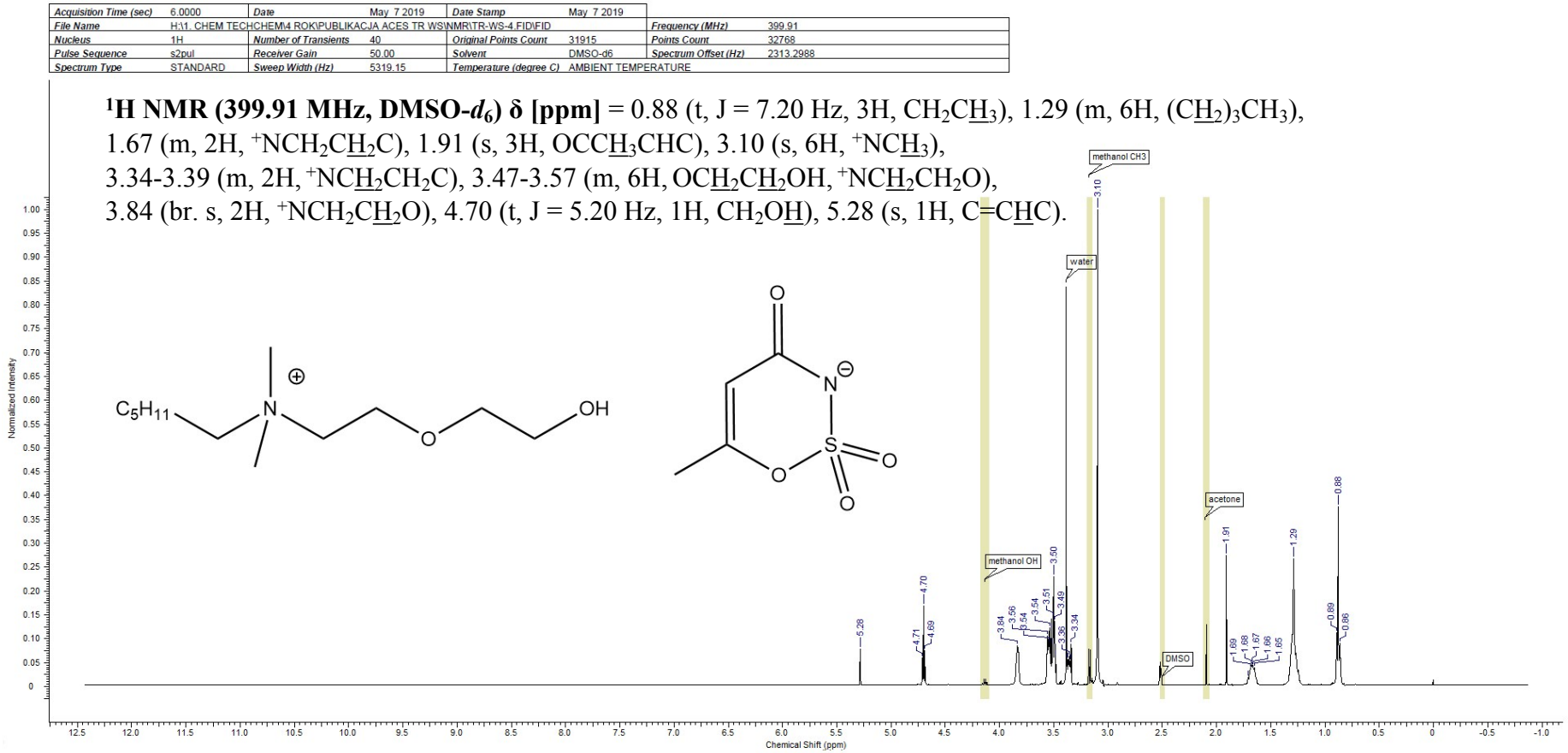


Figure S9. ^{1}H NMR spectrum of hexyl(2-(2-hydroxyethoxy)ethyl)dimethylammonium acesulfame (**3**).

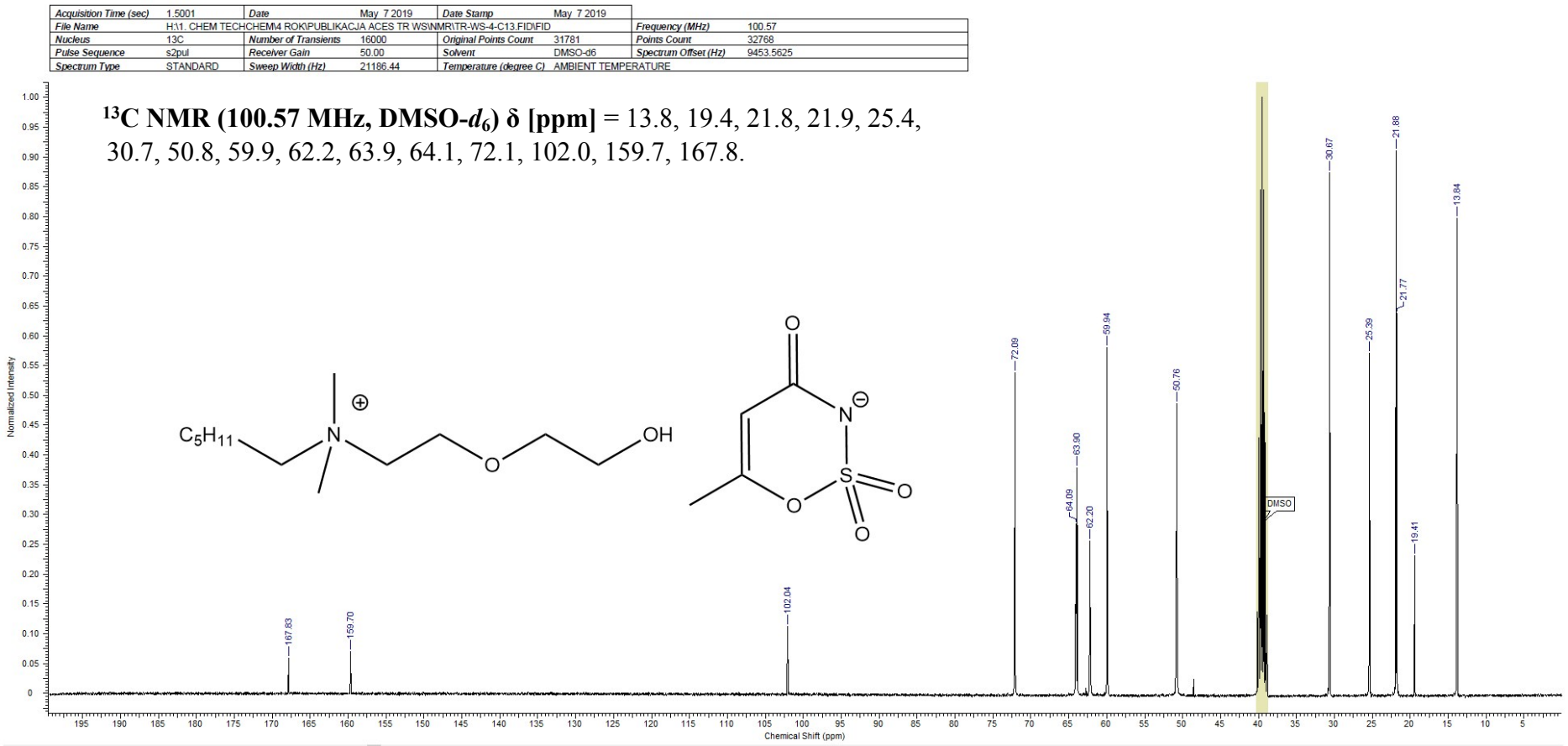


Figure S10. ^{13}C NMR spectrum of hexyl(2-(2-hydroxyethoxy)ethyl)dimethylammonium acesulfame (**3**).

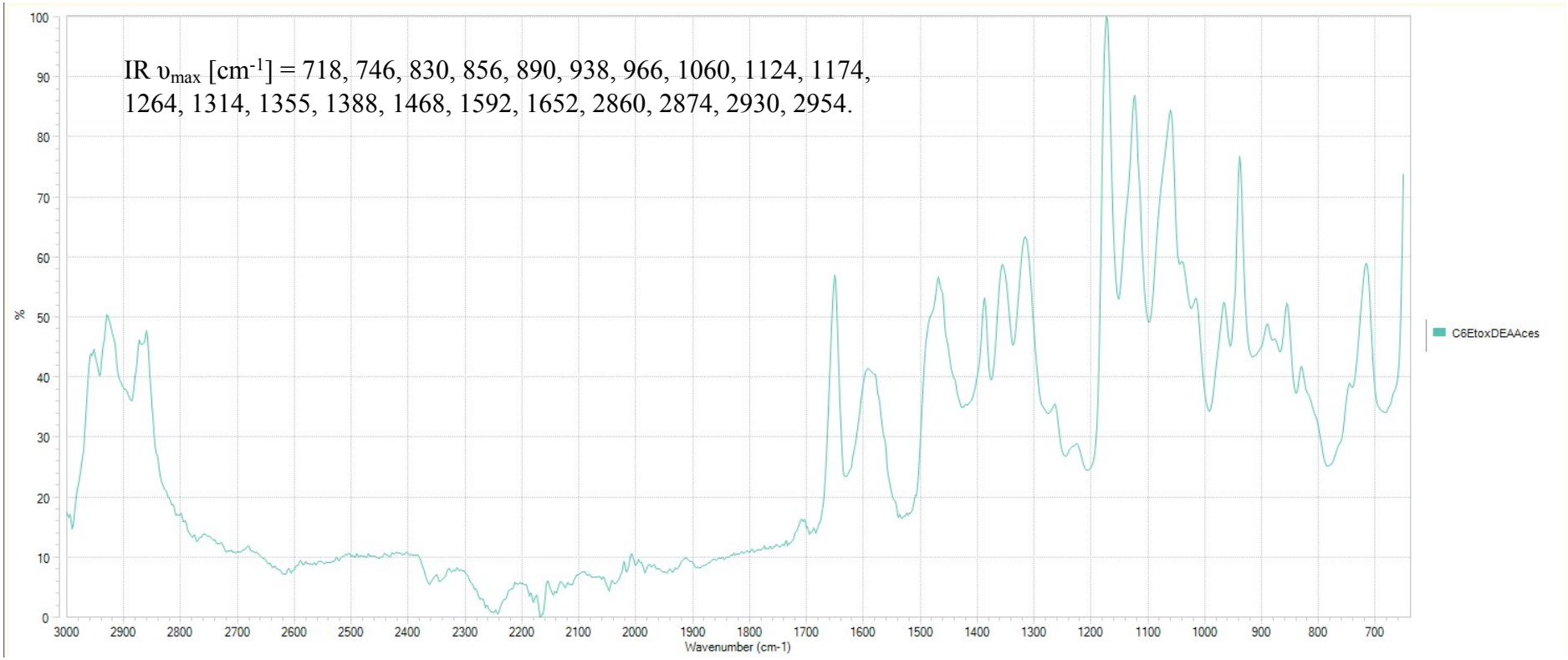


Figure S11. FT-IR spectrum of hexyl(2-(2-hydroxyethoxy)ethyl)dimethylammonium acesulfame (**3**).

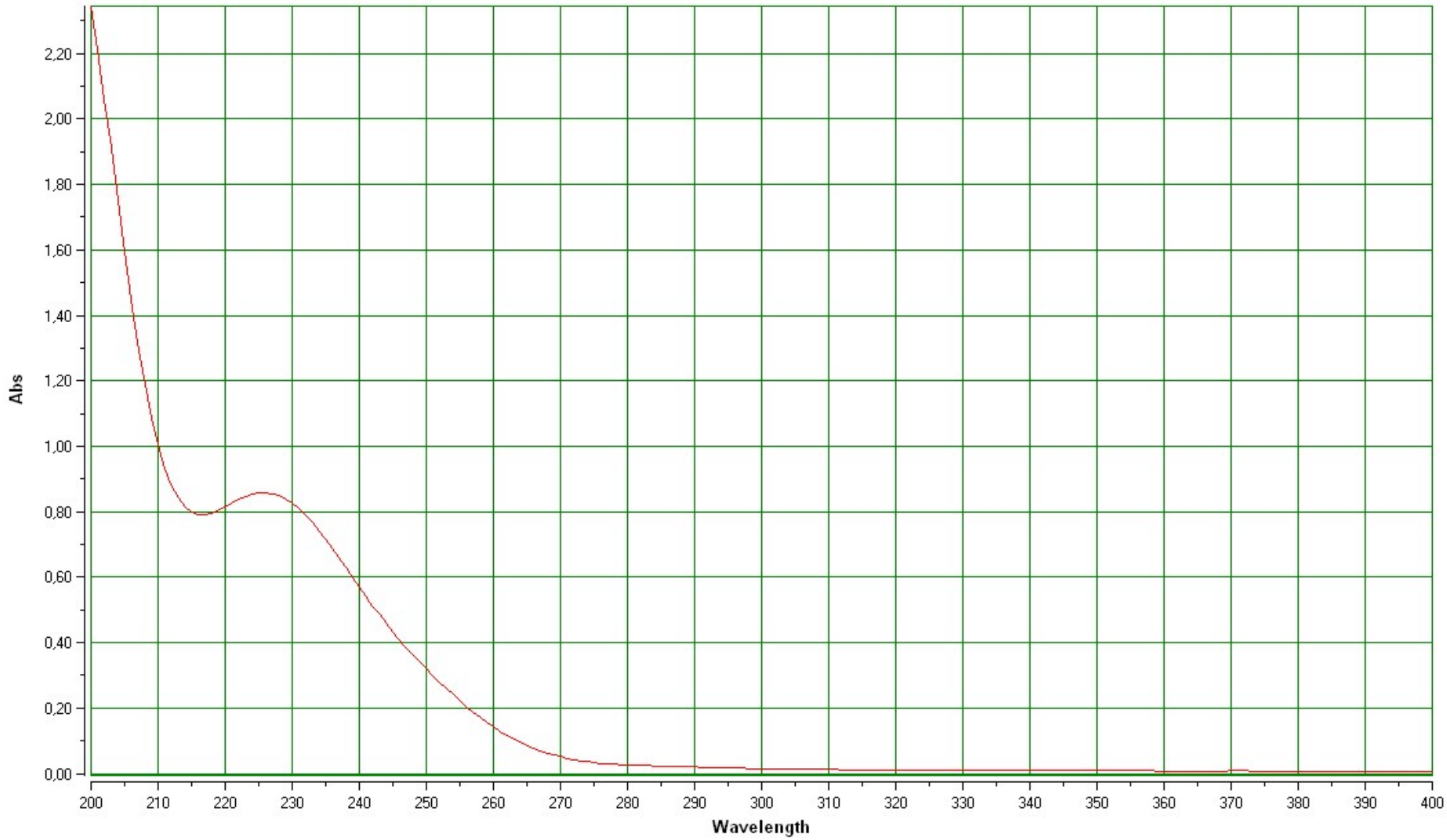
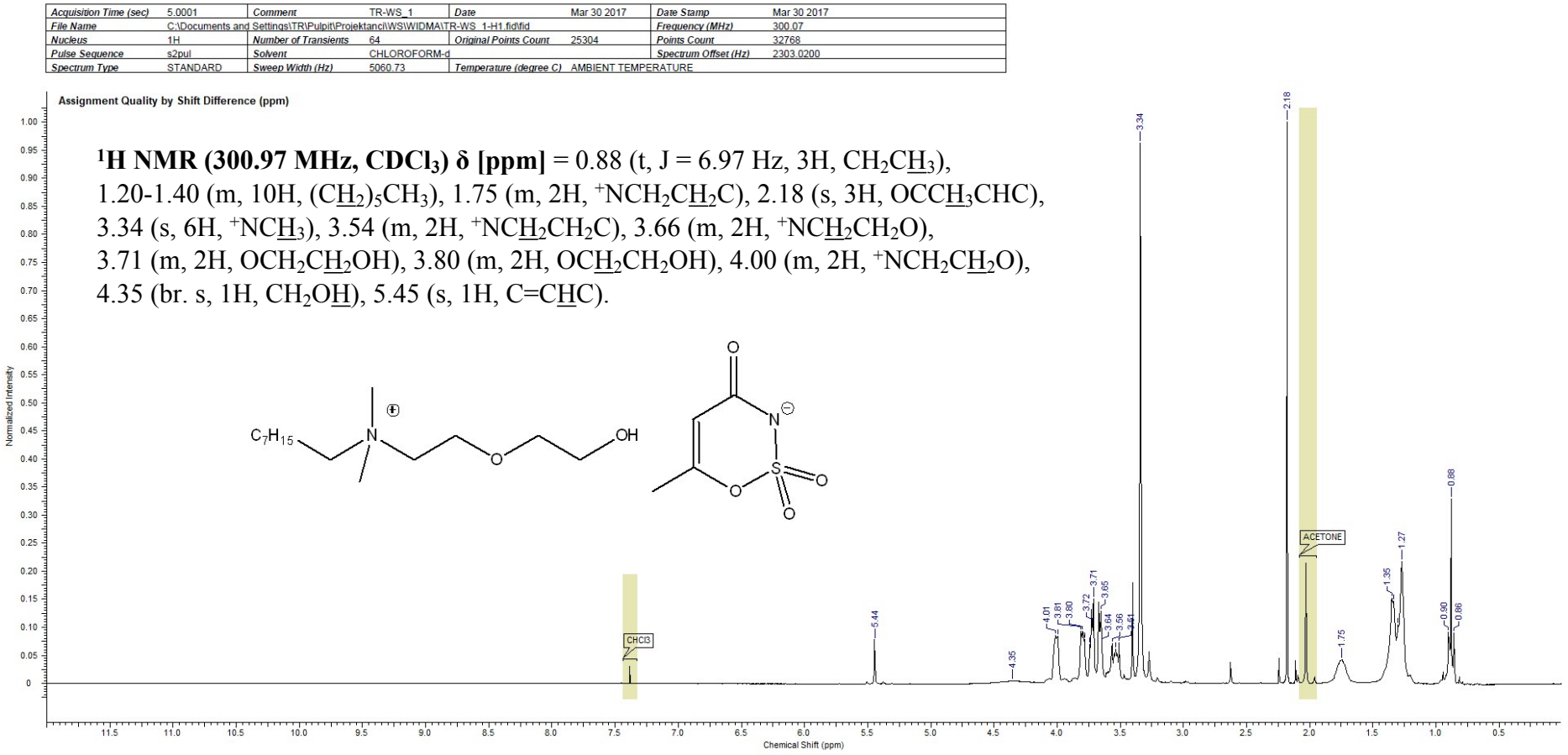


Figure S12. UV spectrum of hexyl(2-(2-hydroxyethoxy)ethyl)dimethylammonium acesulfame (c $\approx 2{,}42 \times 10^{-4} \frac{mol}{dm^3}$) (**3**).



^{1}H NMR (300.97 MHz, $CDCl_3$) δ [ppm] = 0.88 (t, J = 6.97 Hz, 3H, $CH_2C\underline{H}_3$), 1.20-1.40 (m, 10H, $(C\underline{H}_2)_5CH_3$), 1.75 (m, 2H, $^+NCH_2C\underline{H}_2C$), 2.18 (s, 3H, $OCC\underline{H}_3CHC$), 3.34 (s, 6H, $^+NC\underline{H}_3$), 3.54 (m, 2H, $^+NC\underline{H}_2CH_2C$), 3.66 (m, 2H, $^+NC\underline{H}_2CH_2O$), 3.71 (m, 2H, $OCH_2C\underline{H}_2OH$), 3.80 (m, 2H, $OC\underline{H}_2CH_2OH$), 4.00 (m, 2H, $^+NCH_2C\underline{H}_2O$), 4.35 (br. s, 1H, $CH_2O\underline{H}$), 5.45 (s, 1H, $C{=}C\underline{H}C$).

Figure S13. ^{1}H NMR spectrum of octyl(2-(2-hydroxyethoxy)ethyl)dimethylammonium acesulfame (**4**).

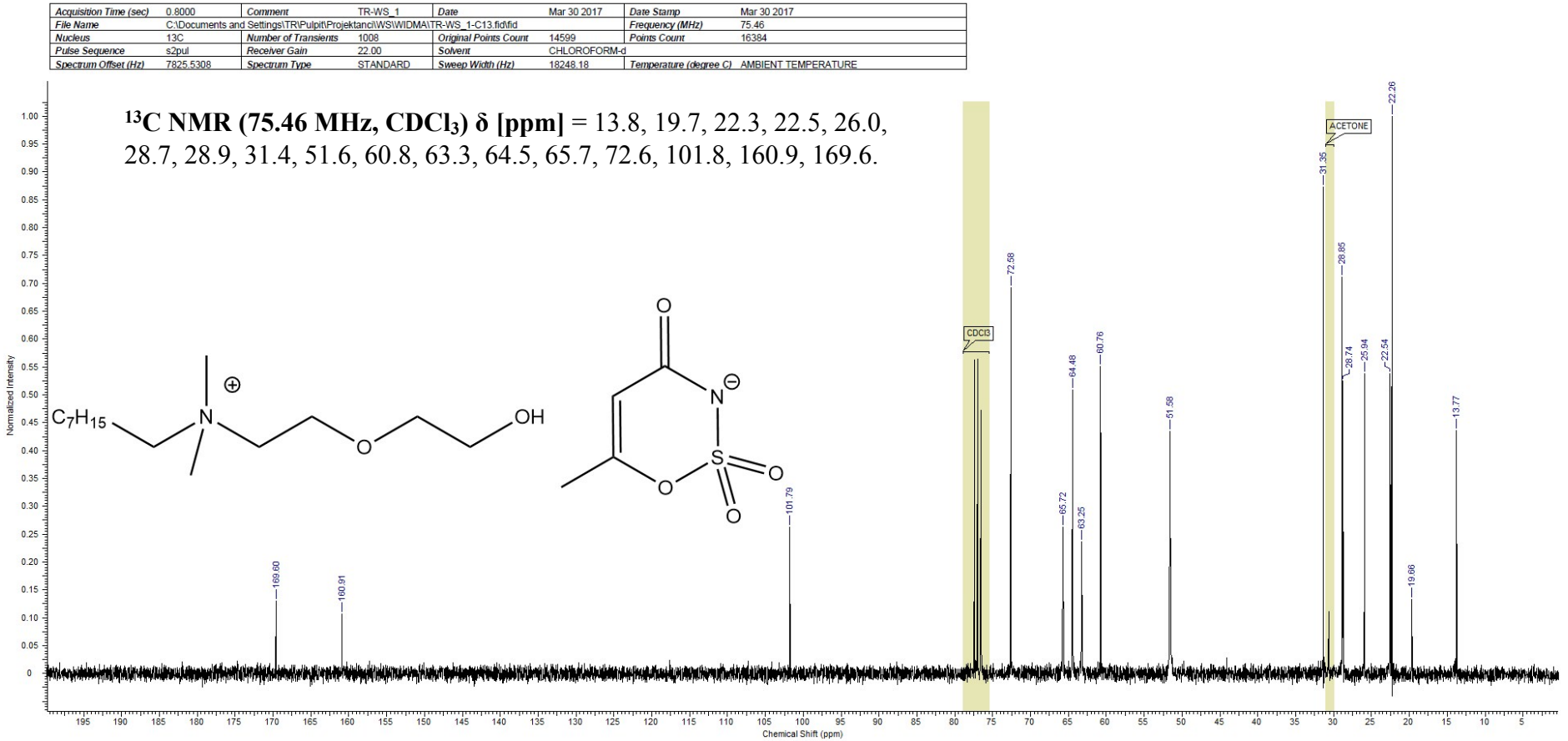


Figure S14. ^{13}C NMR spectrum of hexyl(2-(2-hydroxyethoxy)ethyl)dimethylammonium acesulfame (**4**).

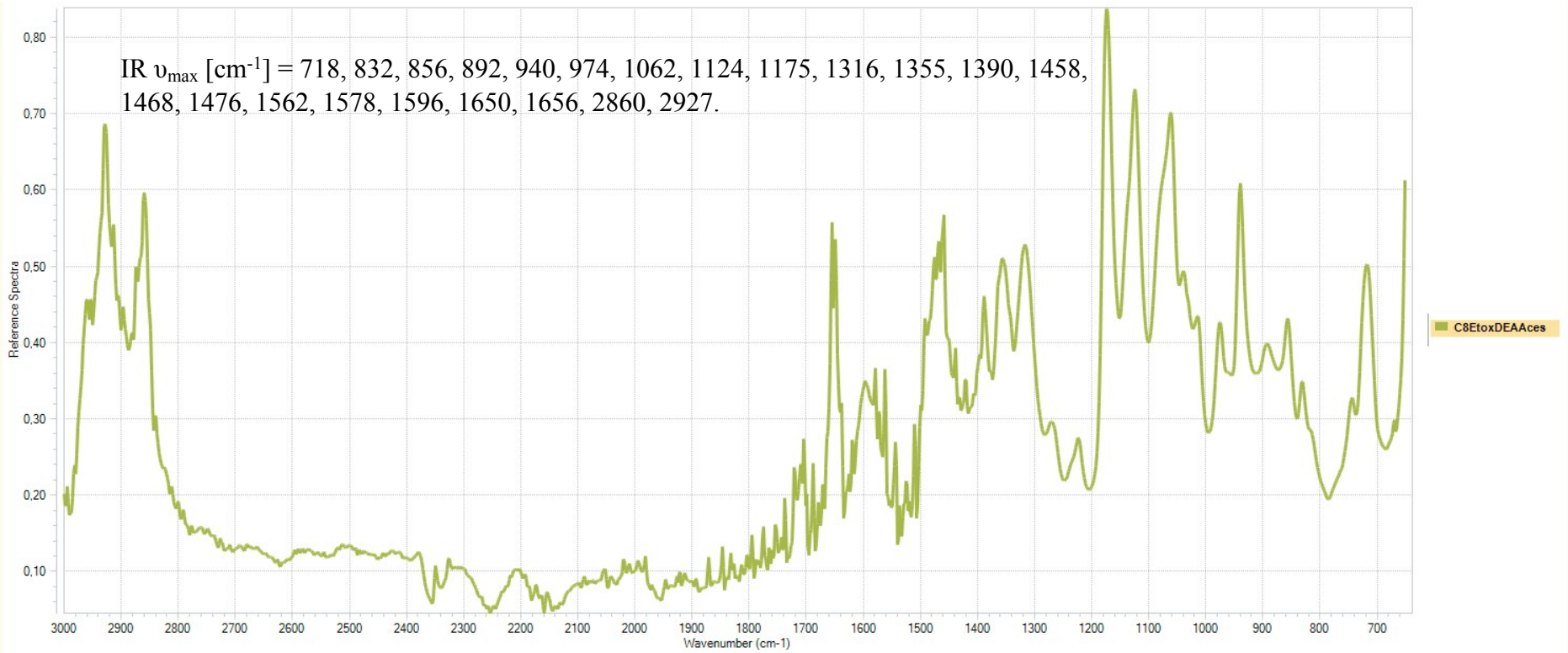


Figure S15. FT-IR spectrum of octyl(2-(2-hydroxyethoxy)ethyl)dimethylammonium acesulfame (**4**).

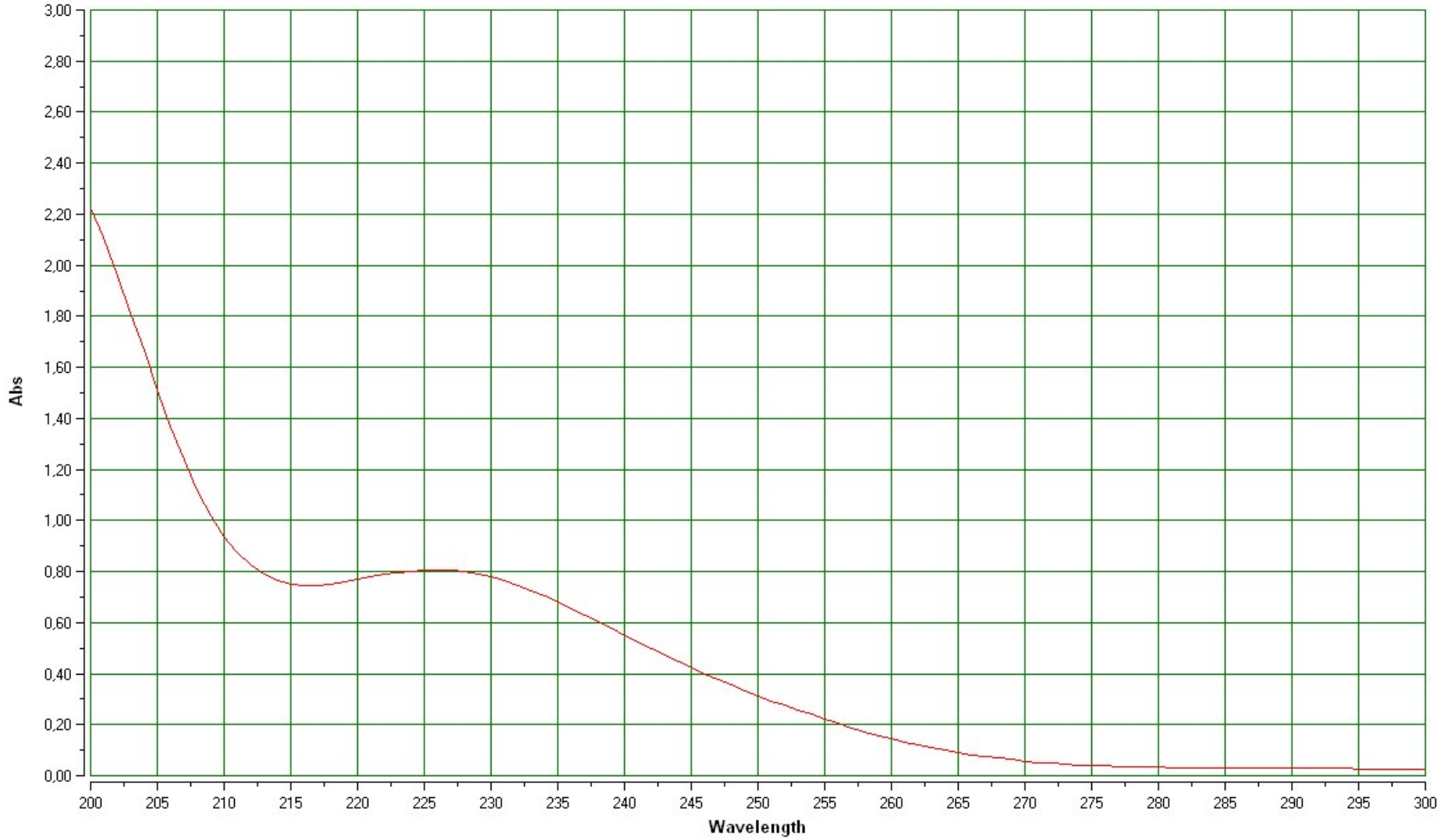


Figure S16. UV spectrum of octyl(2-(2-hydroxyethoxy)ethyl)dimethylammonium acesulfame (c $\approx 2{,}63 \times 10^{-4} \frac{mol}{dm^3}$) (**4**).

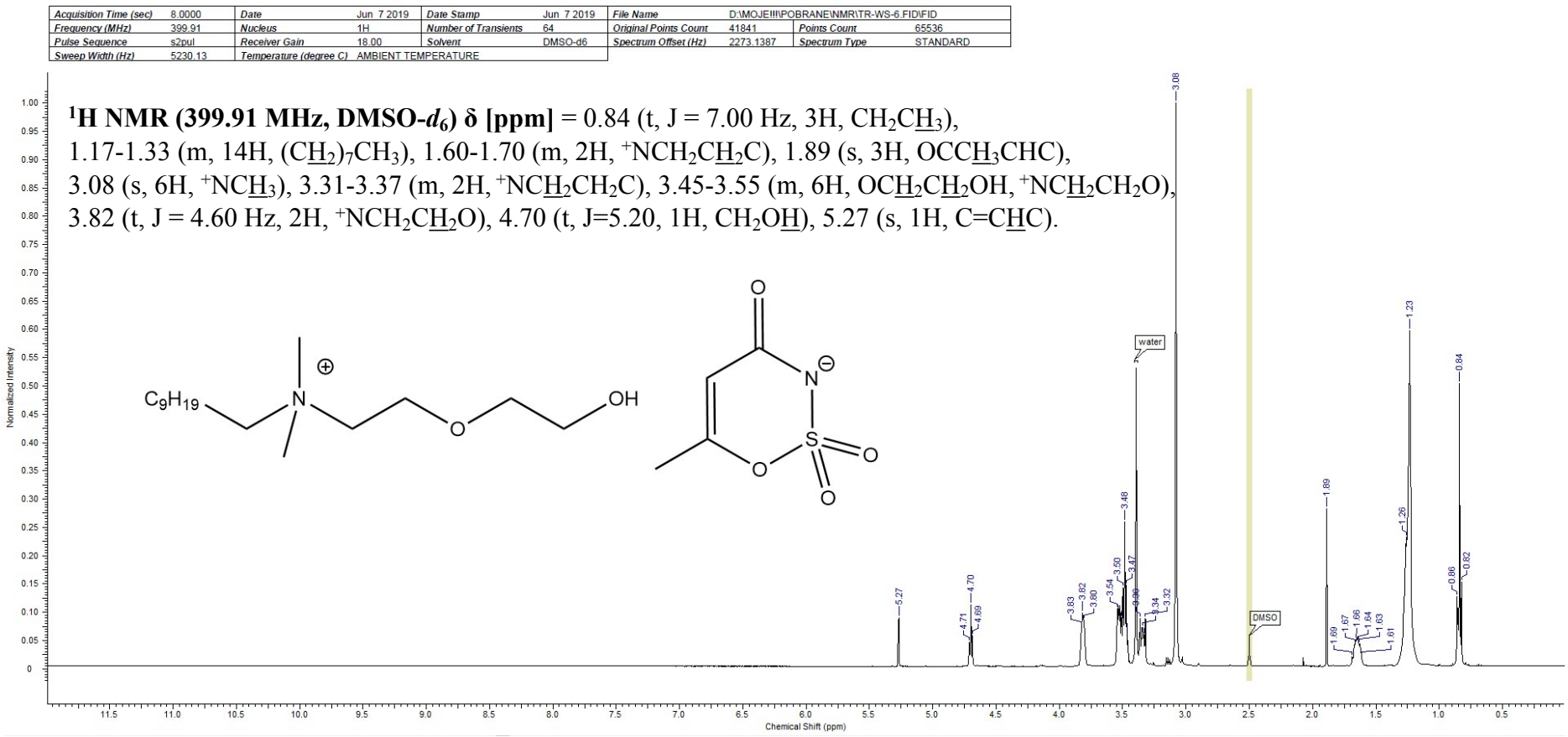


Figure S17. ^{1}H NMR spectrum of decyl(2-(2-hydroxyethoxy)ethyl)dimethylammonium acesulfame (**5**).

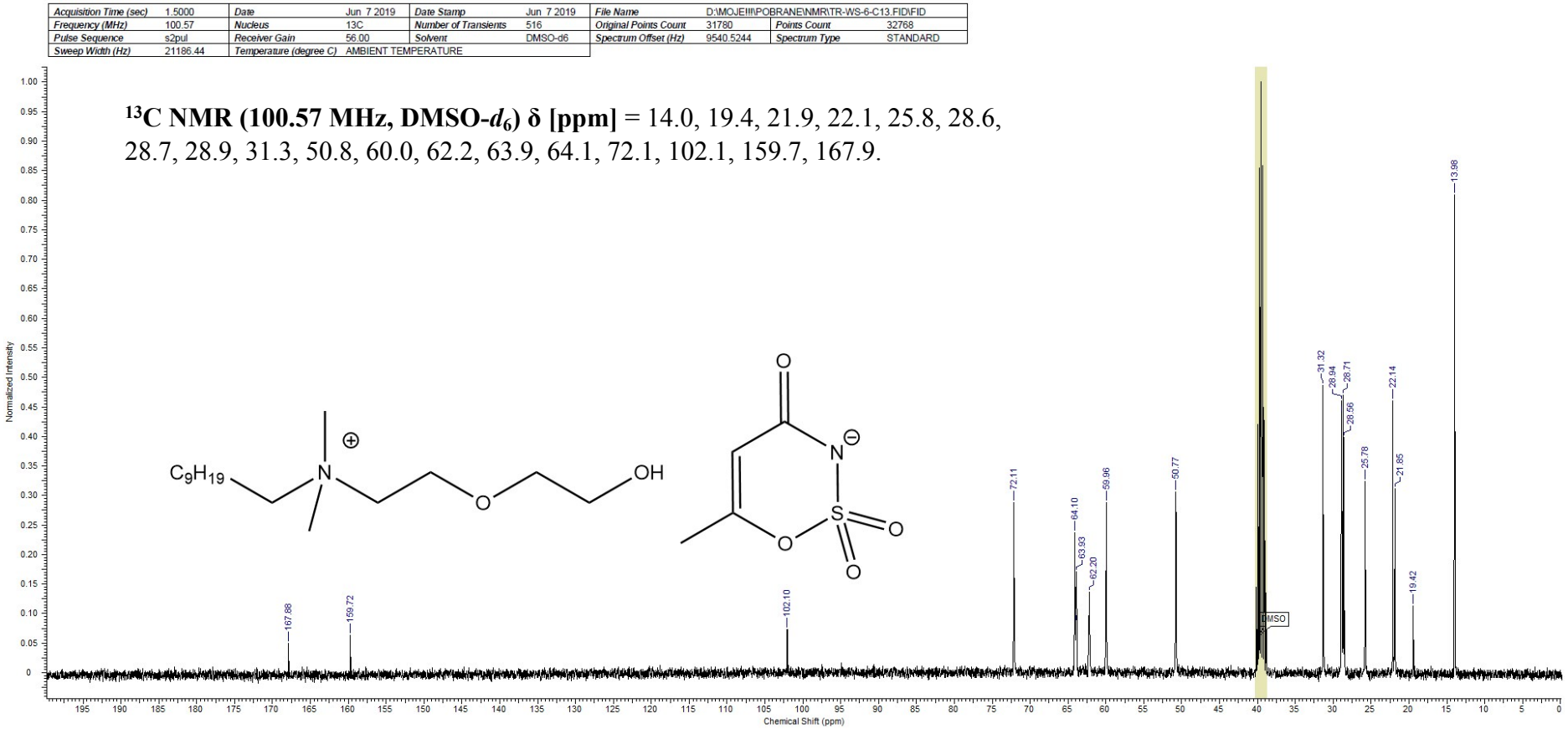


Figure S18. ^{13}C NMR spectrum of decyl(2-(2-hydroxyethoxy)ethyl)dimethylammonium acesulfame (**5**).

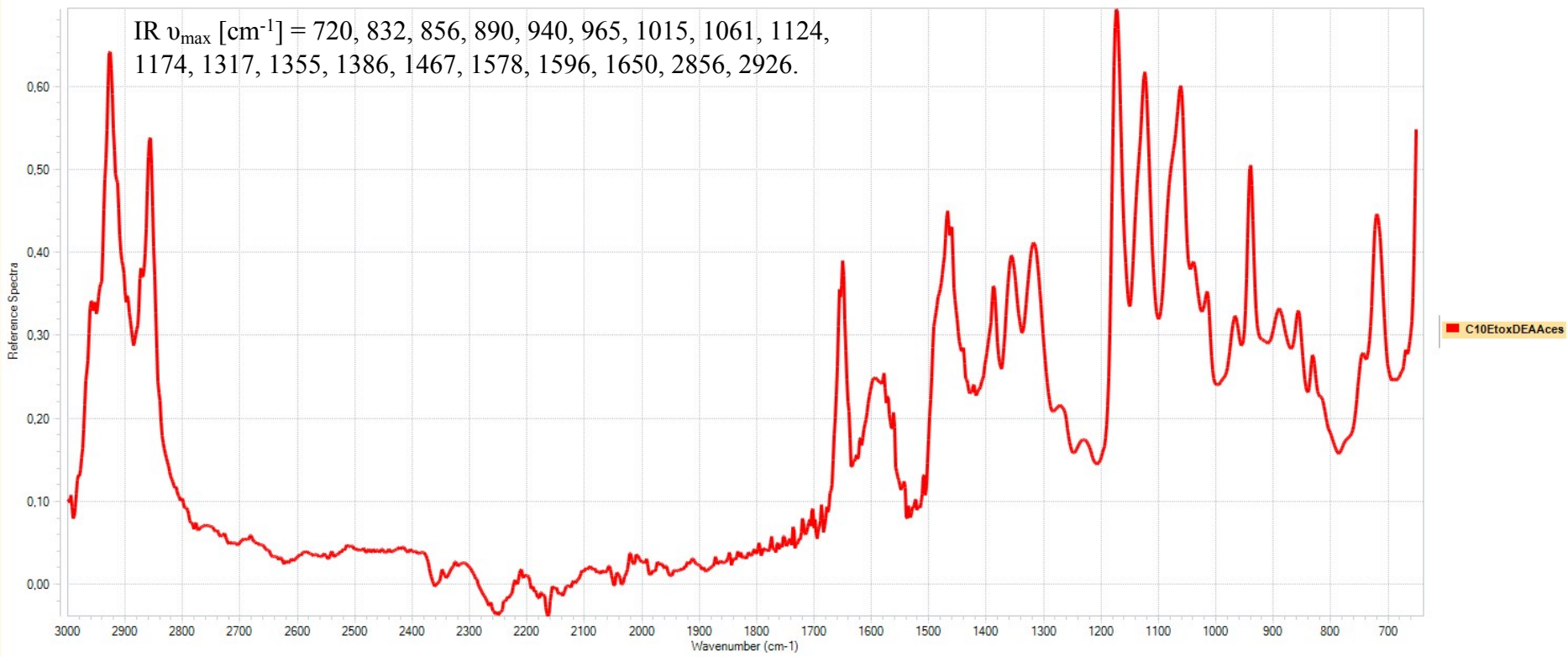


Figure S19. FT-IR spectrum of decyl(2-(2-hydroxyethoxy)ethyl)dimethylammonium acesulfame (**5**).

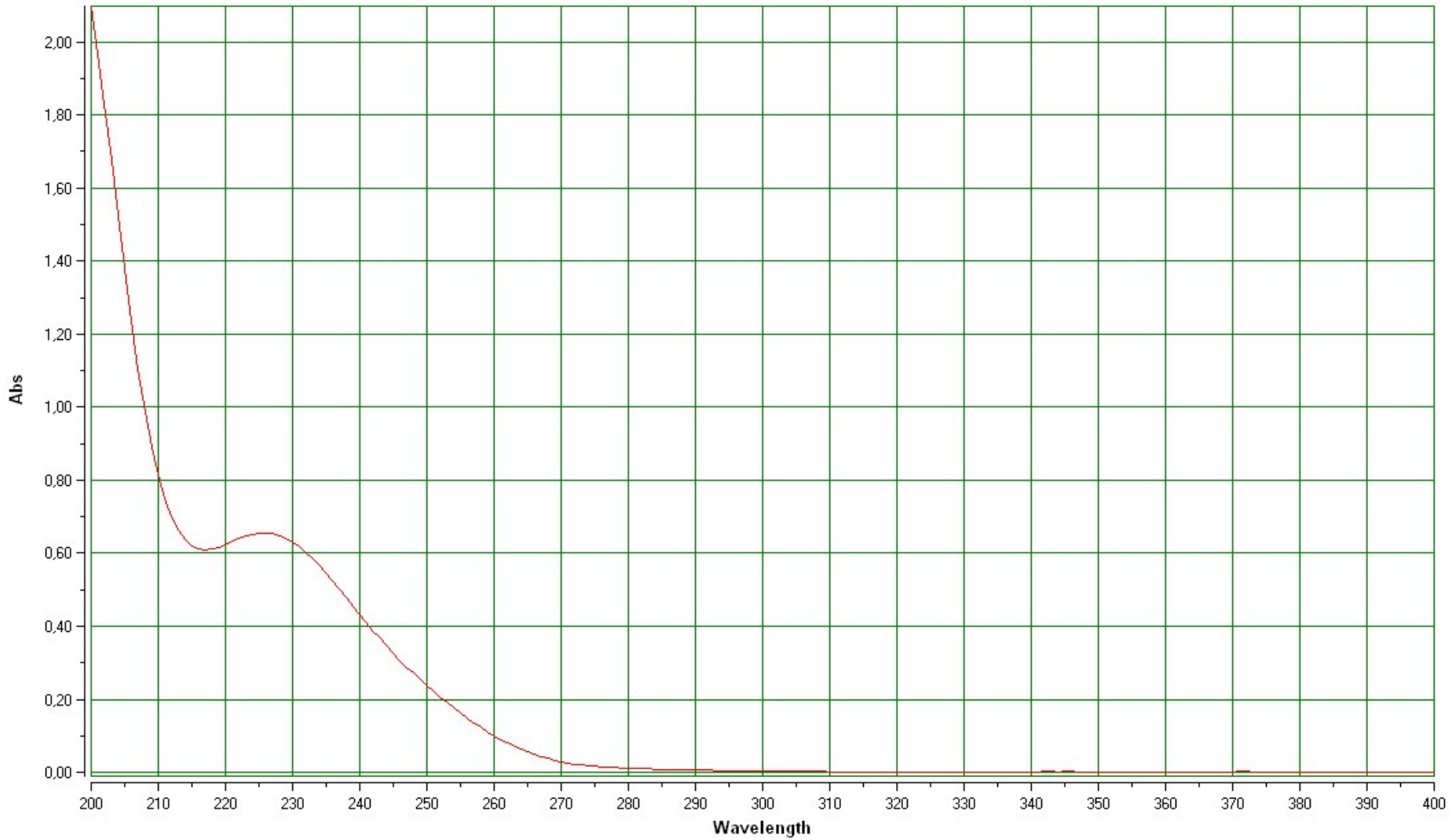

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Figure S20. UV spectrum of decyl(2-(2-hydroxyethoxy)ethyl)dimethylammonium acesulfame (c $\approx 2{,}14 \times 10^{-4} \frac{mol}{dm^3}$) (**5**).

Acquisition Time (sec)	5.0000	*Date*	Oct 30 2019	*Date Stamp*	Oct 30 2019		
File Name	D:\1. CHEM TECHCHEM\3 ROK\2. PUBLIKACJA ACES TR WS\1. NMR\[C12ETOXDEA][ACES]\TR-WS-10.FID\FID						
Frequency (MHz)	399.91	*Nucleus*	1H	*Number of Transients*	64	*Original Points Count*	31726
Points Count	32768	*Pulse Sequence*	s2pul	*Receiver Gain*	22.00	*Solvent*	DMSO-d6
Spectrum Offset (Hz)	2826.3457	*Spectrum Type*	STANDARD	*Sweep Width (Hz)*	6345.18	*Temperature (degree C)*	AMBIENT TEMPERATURE

^{1}H NMR (399.91 MHz, DMSO-d_6) δ [ppm] = 0.84 (t, J = 7.20 Hz, 3H, $CH_2C\underline{H}_3$),
1.20-1.30 (m, 18H, $(C\underline{H}_2)_9CH_3$), 1.60-1.70 (m, 2H, $^+NCH_2C\underline{H}_2C$), 1.89 (s, 3H, $OCC\underline{H}_3CHC$),
3.08 (s, 6H, $^+NC\underline{H}_3$), 3.31-3.37 (m, 2H, $^+NC\underline{H}_2CH_2C$),
3.45-3.55 (m, 6H, $OC\underline{H}_2C\underline{H}_2OH$, $^+NC\underline{H}_2CH_2O$), 3.82 (br. s, 2H, $^+NCH_2C\underline{H}_2O$),
4.69 (br. s, 1H, $CH_2O\underline{H}$), 5.26 (s, 1H, $C{=}C\underline{H}C$).

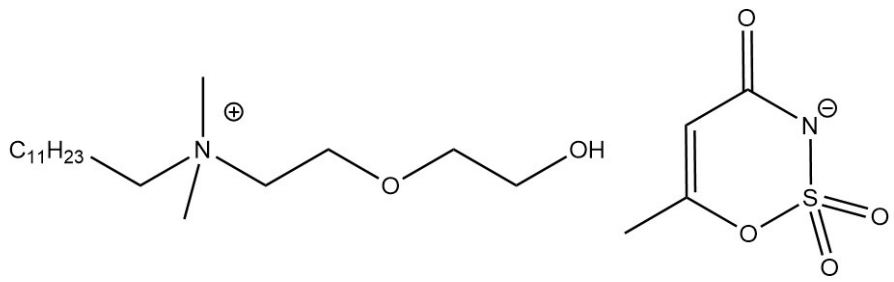

Figure S21. ^{1}H NMR spectrum of dodecyl(2-(2-hydroxyethoxy)ethyl)dimethylammonium acesulfame (**6**).

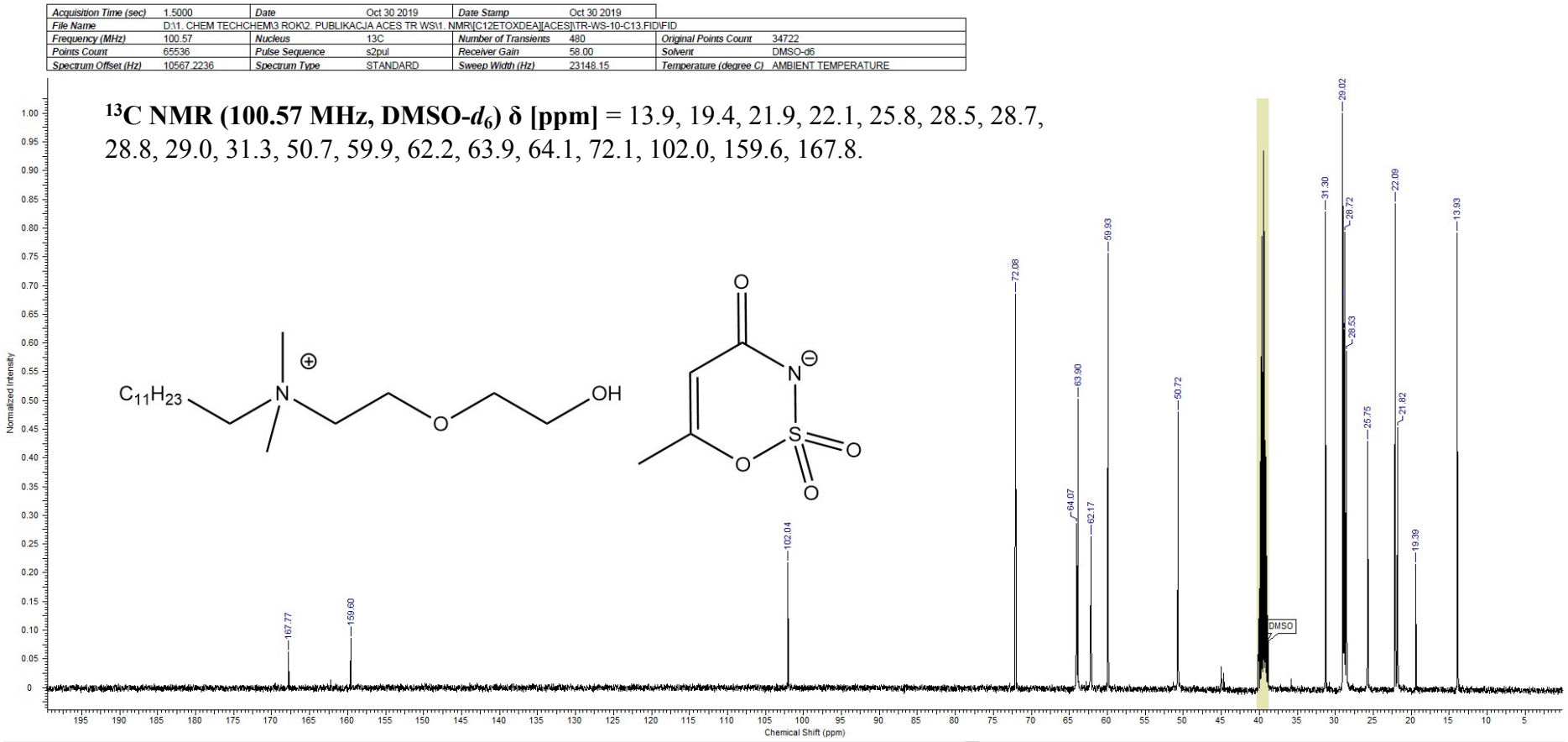

Figure S22. ^{13}C NMR spectrum of dodecyl(2-(2-hydroxyethoxy)ethyl)dimethylammonium acesulfame (**6**).

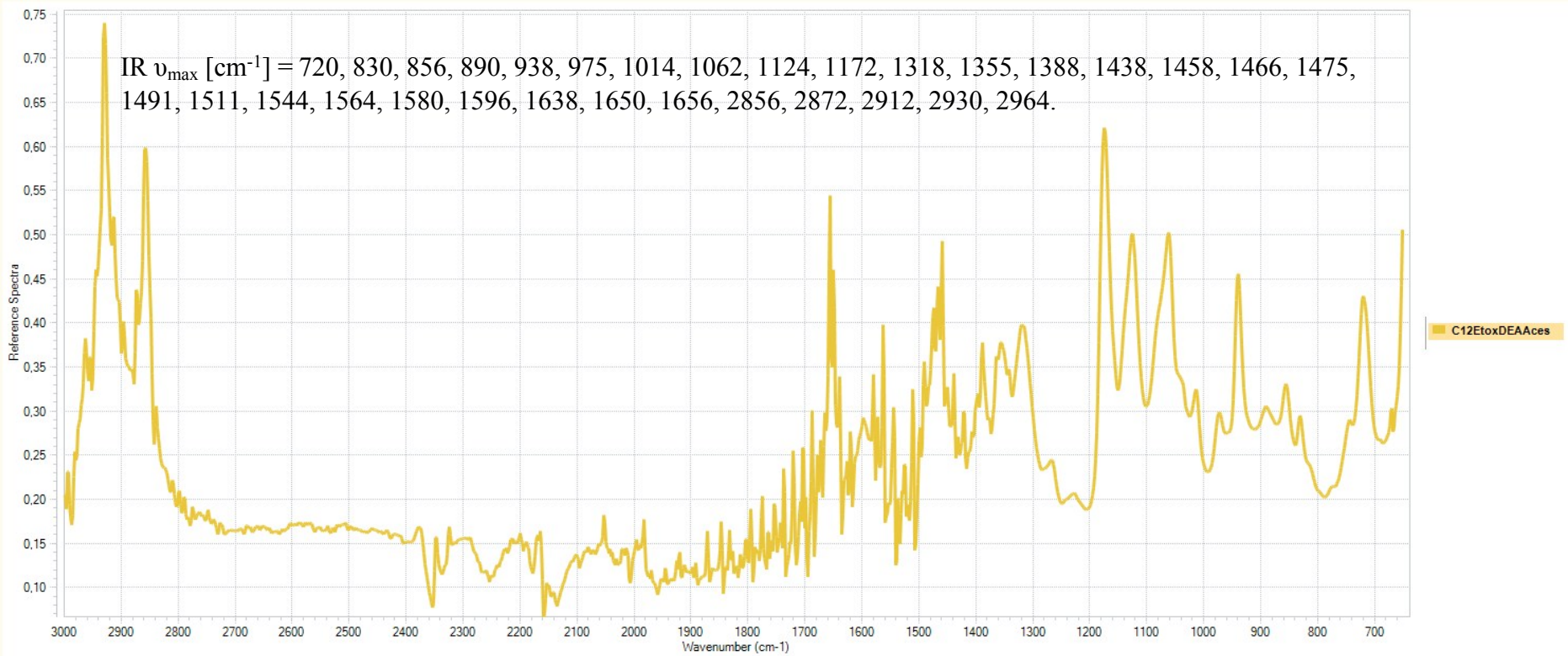

Figure S23. FT-IR spectrum of dodecyl(2-(2-hydroxyethoxy)ethyl)dimethylammonium acesulfame (**6**).

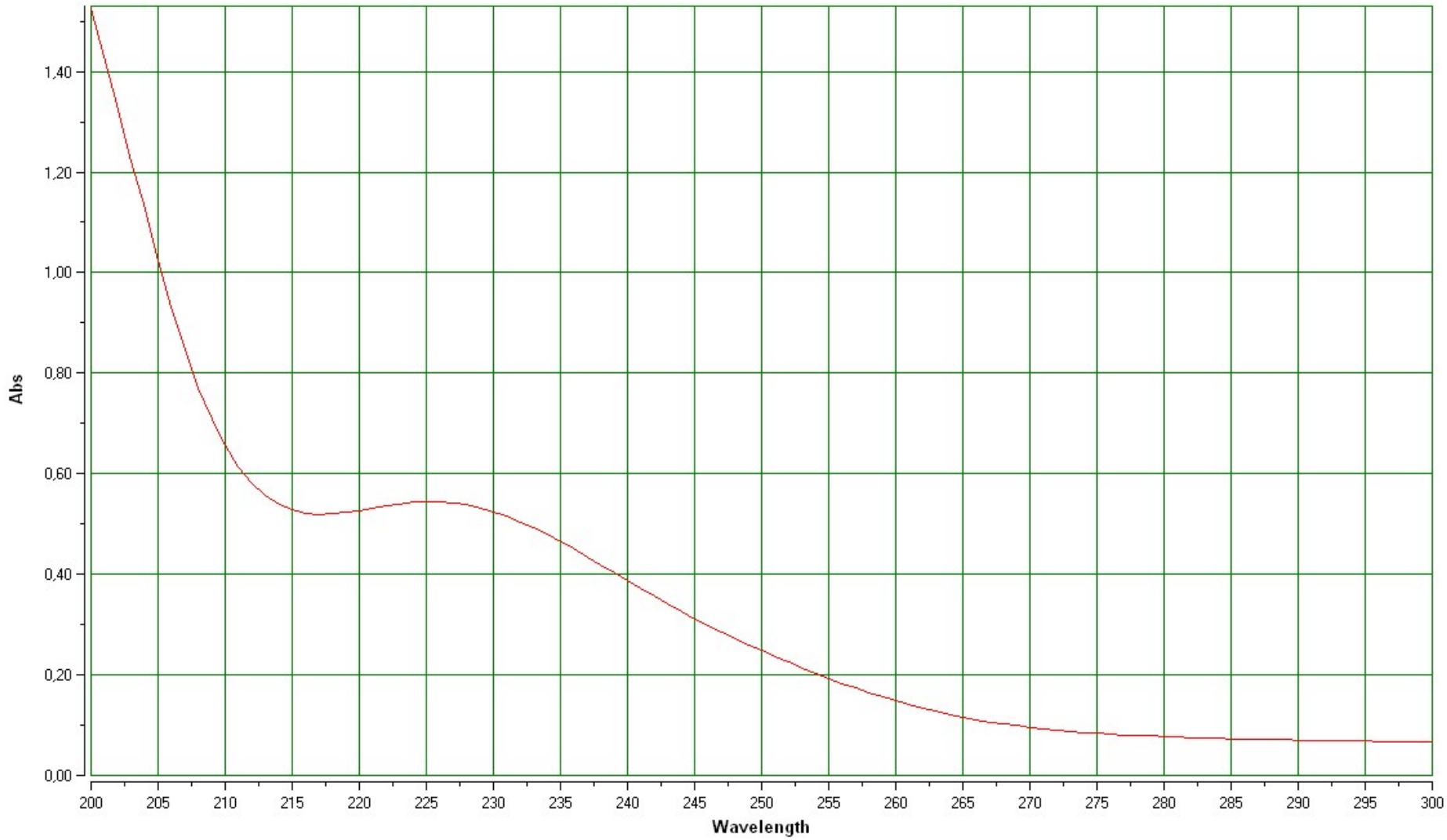

Figure S24. UV spectrum of dodecyl(2-(2-hydroxyethoxy)ethyl)dimethylammonium acesulfame (c $\approx 1{,}34 \times 10^{-4} \frac{mol}{dm^3}$) (**6**).

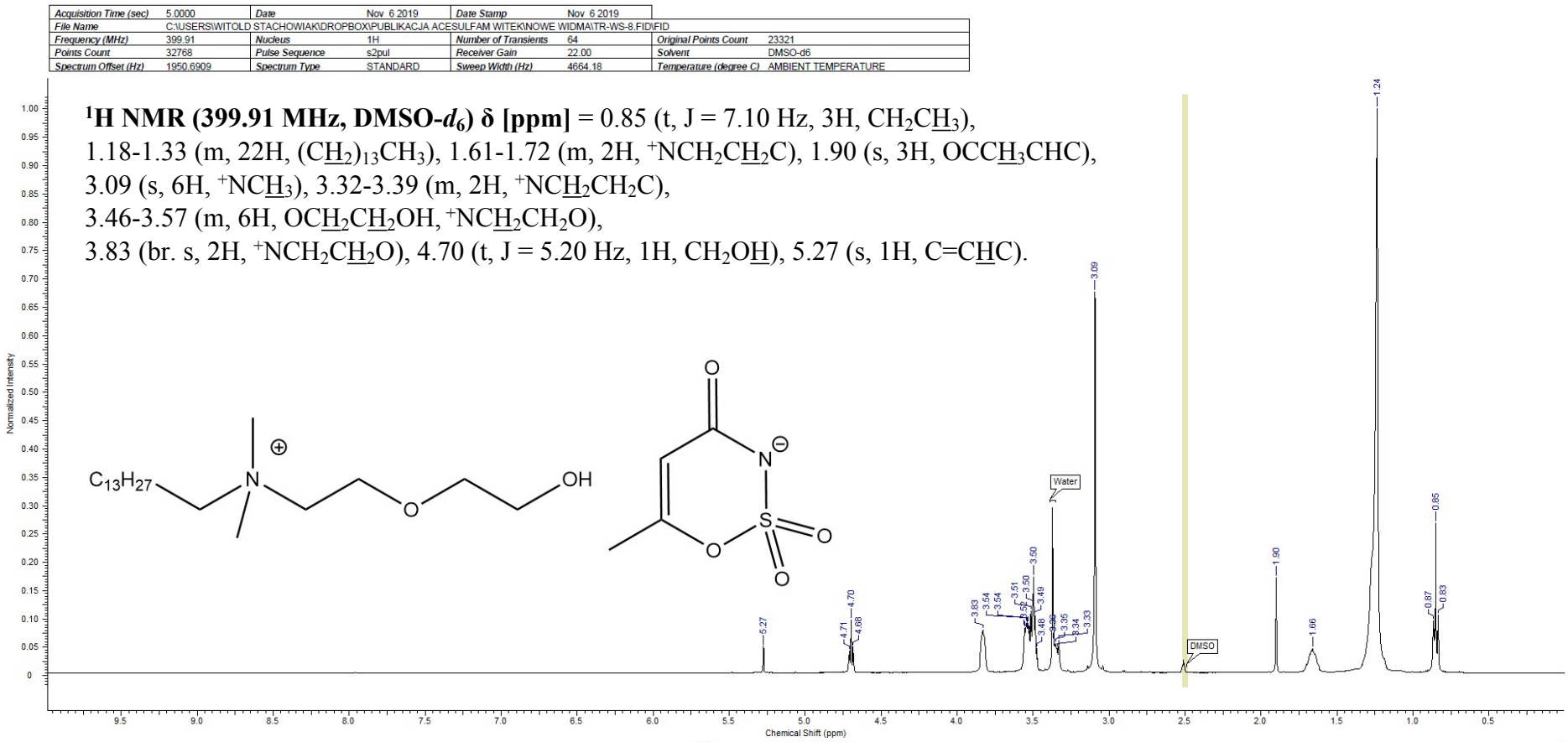

Figure S25. ^{1}H NMR spectrum of tetradecyl(2-(2-hydroxyethoxy)ethyl)dimethylammonium acesulfame (**7**).

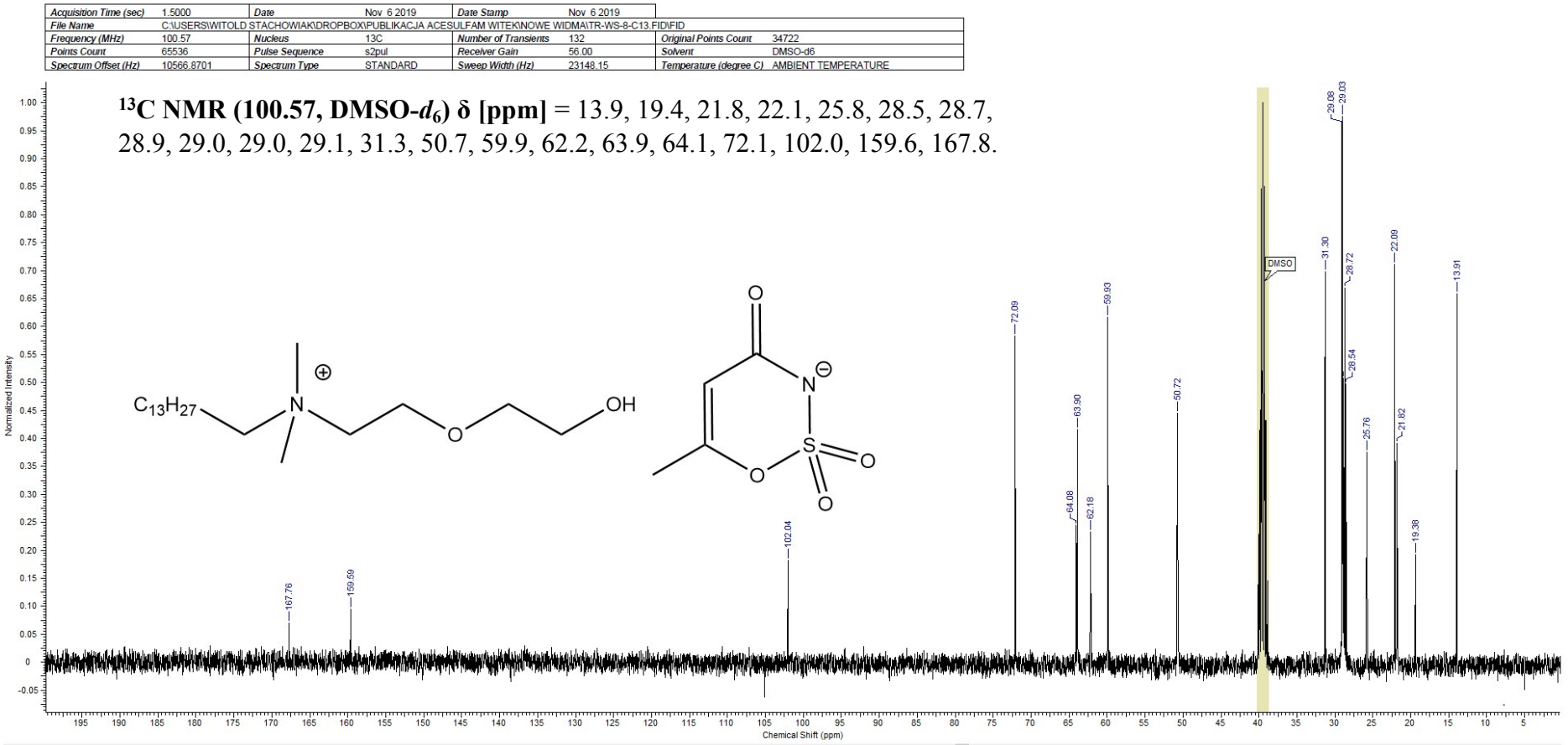

Figure S26. ^{13}C NMR spectrum of tetradecyl(2-(2-hydroxyethoxy)ethyl)dimethylammonium acesulfame (**7**).

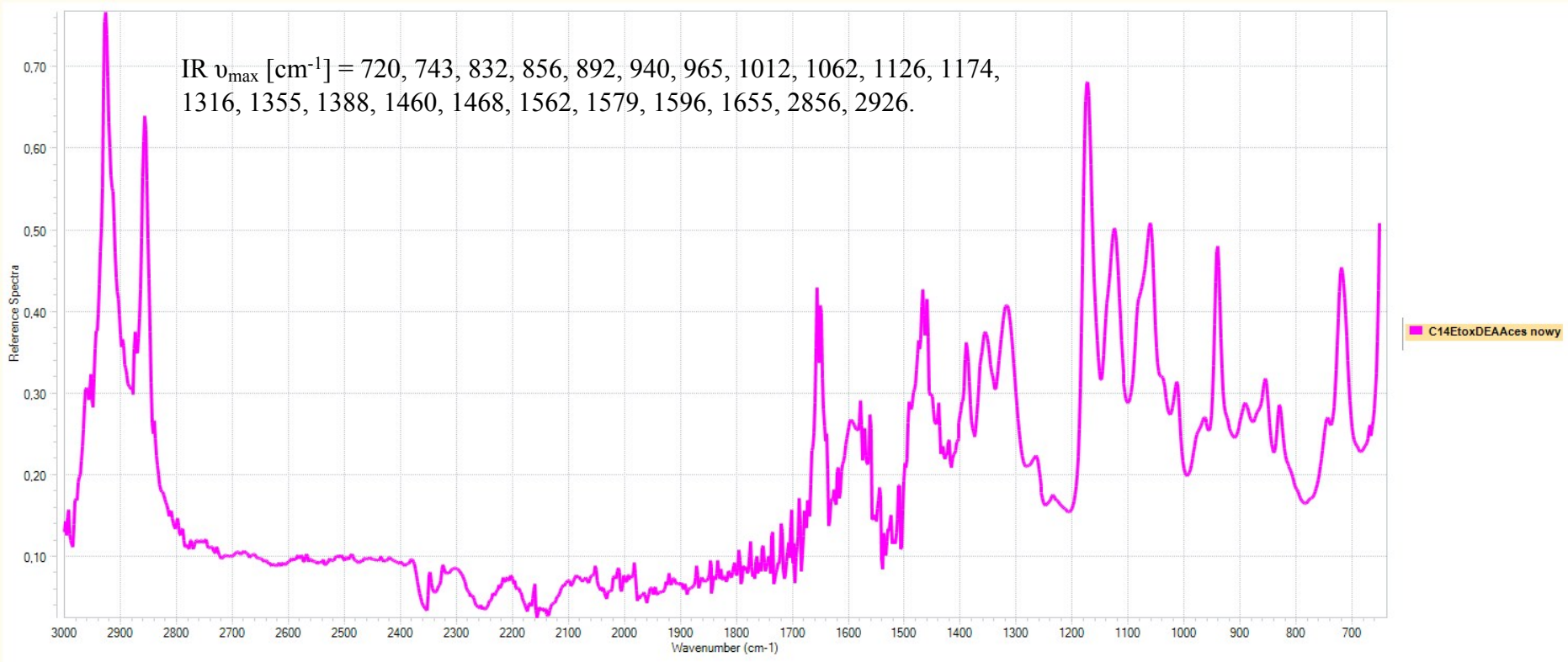

Figure S27. FT-IR NMR spectrum of tetradecyl(2-(2-hydroxyethoxy)ethyl)dimethylammonium acesulfame (**7**).

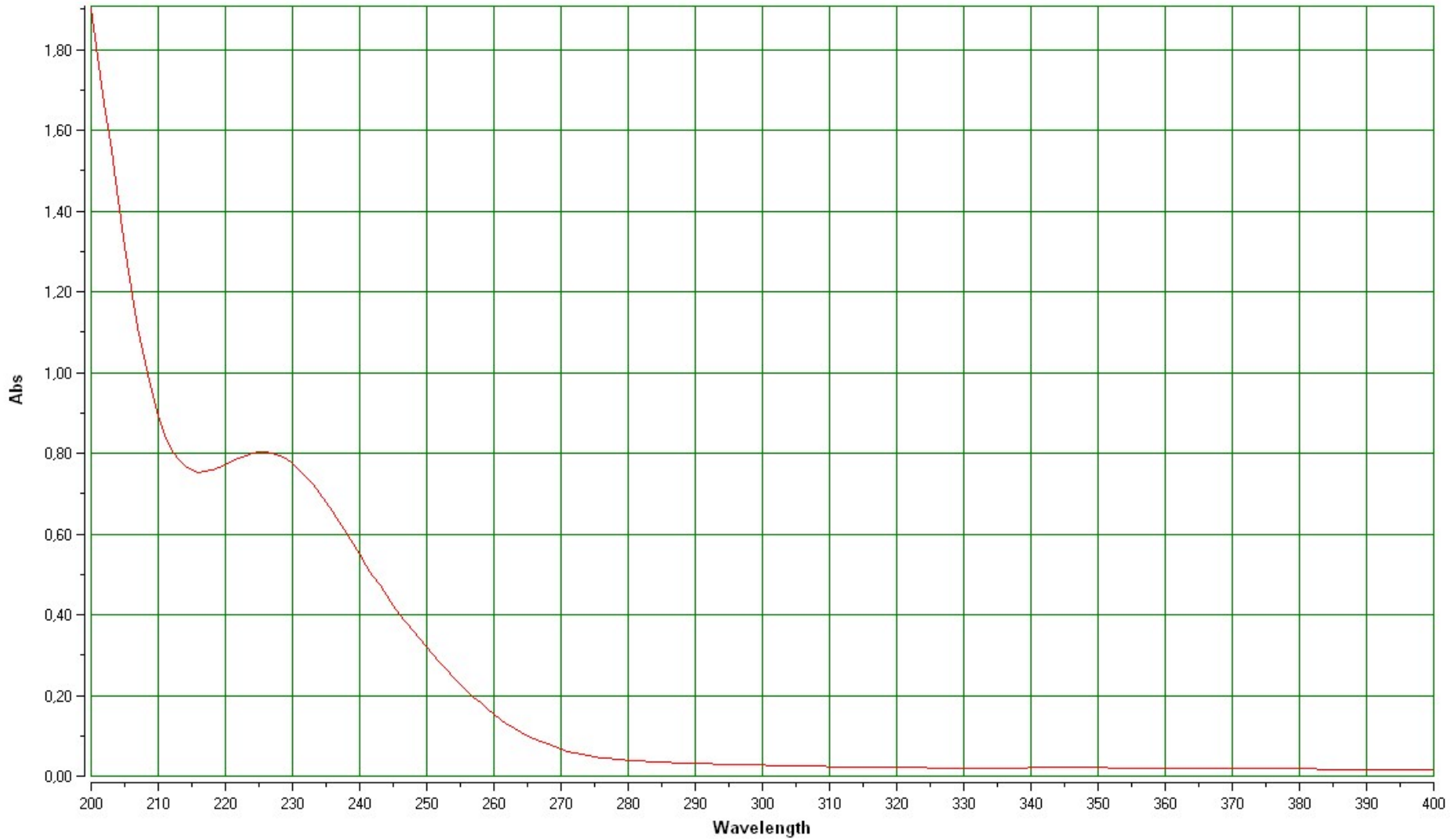
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Figure S28. UV spectrum of tetradecyl(2-(2-hydroxyethoxy)ethyl)dimethylammonium acesulfame (c $\approx 1{,}6 \times 10^{-4} \frac{mol}{dm^3}$) (**7**).

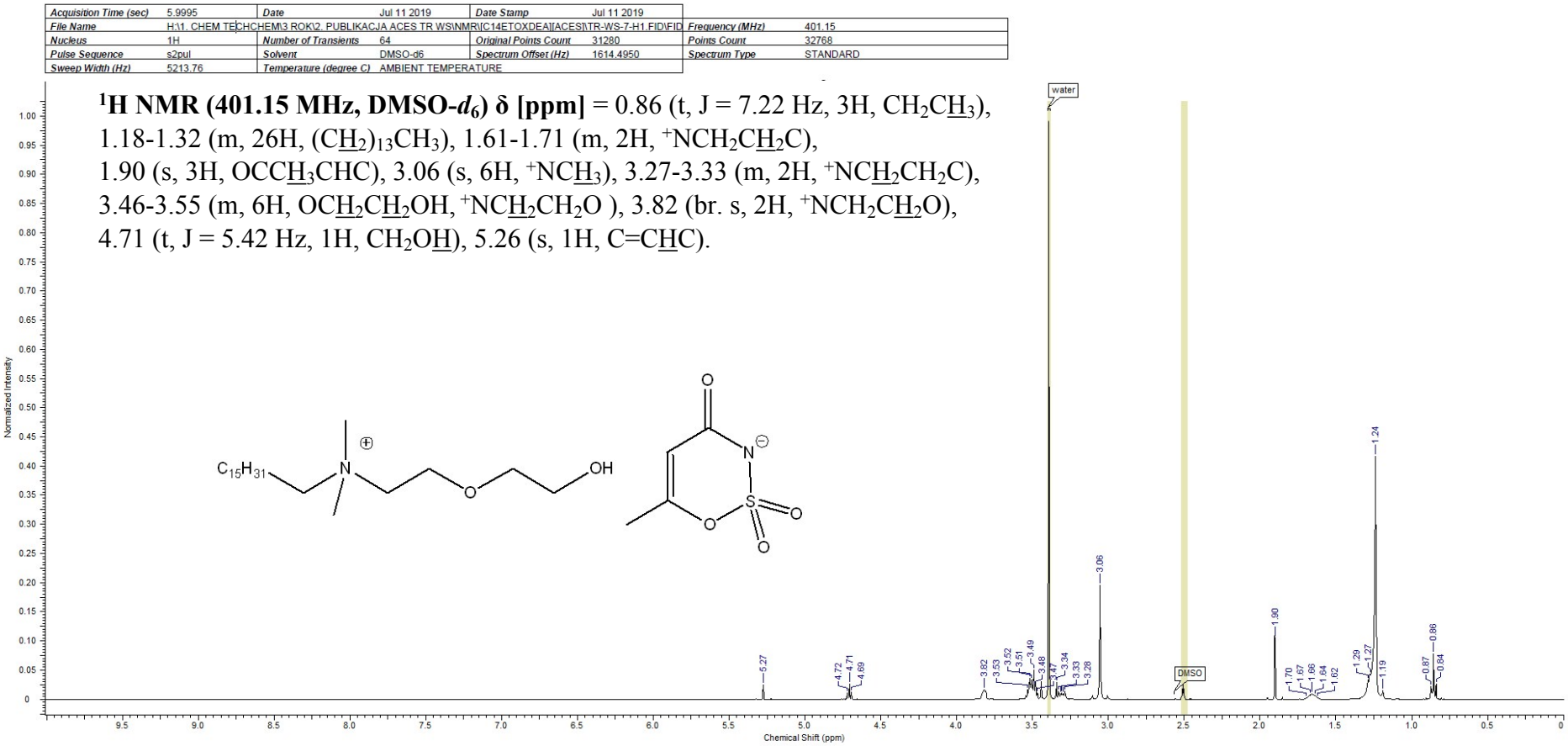

Figure S29. ^{1}H NMR spectrum of hexadecyl(2-(2-hydroxyethoxy)ethyl)dimethylammonium acesulfame (**8**).

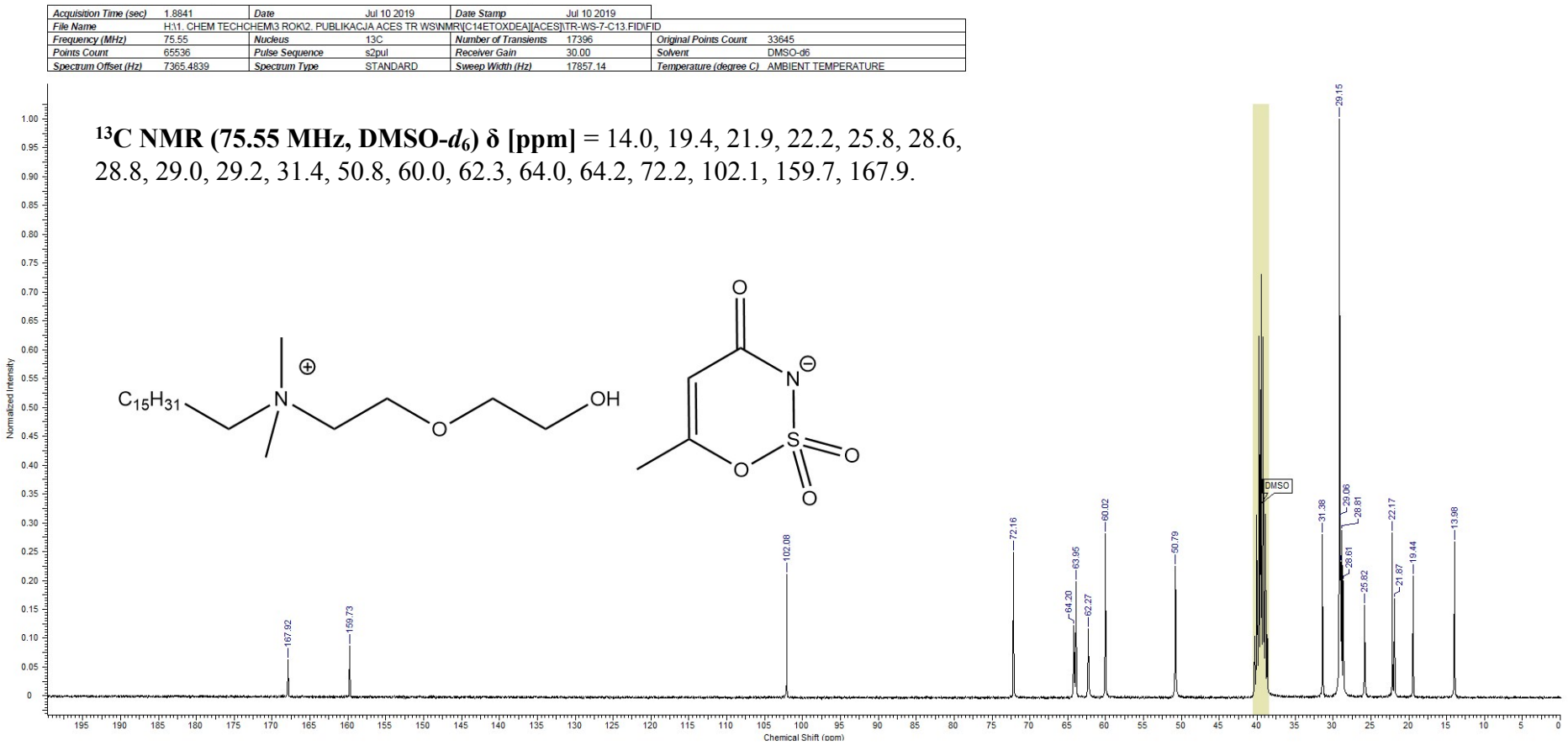

Figure S30. ^{13}C NMR spectrum of hexadecyl(2-(2-hydroxyethoxy)ethyl)dimethylammonium acesulfame (**8**).

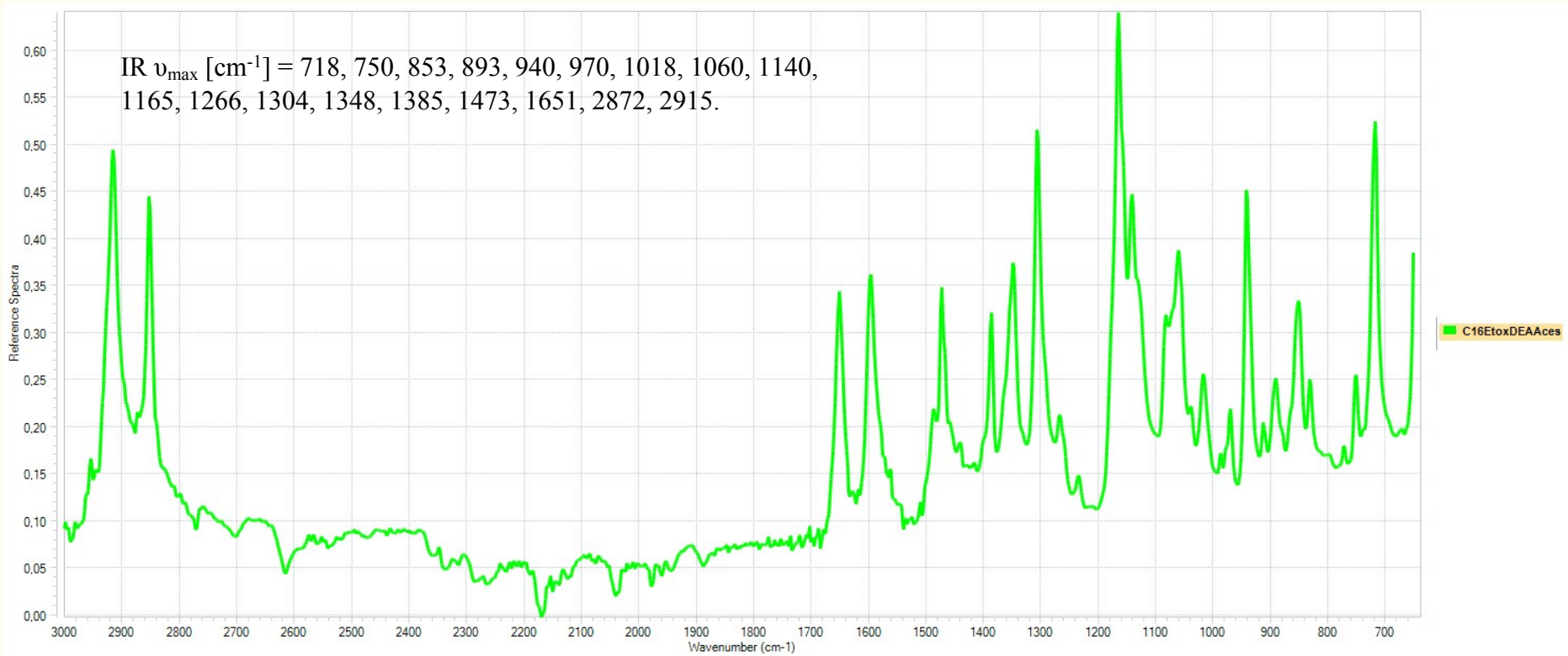

Figure S31. FT-IR spectrum of hexadecyl(2-(2-hydroxyethoxy)ethyl)dimethylammonium acesulfame (**8**).

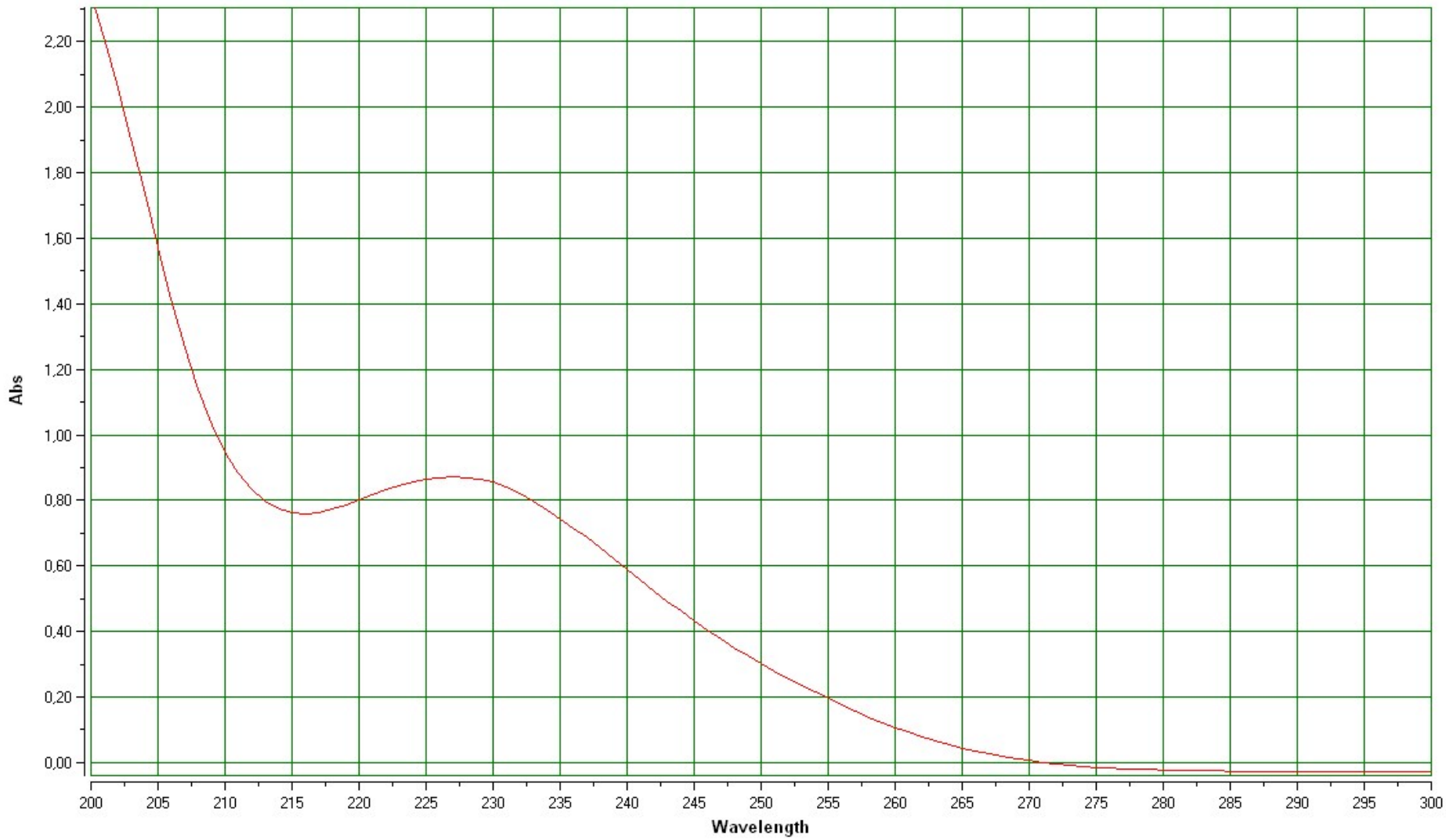

Figure S32. UV spectrum of hexadecyl(2-(2-hydroxyethoxy)ethyl)dimethylammonium acesulfame (c $\approx 1{,}1 \times 10^{-4} \frac{mol}{dm^3}$) (**8**).

Table S1. Density values (g cm^{-3}), molecular weight (M_w) and molecular volume (V_m) at 20 °C for ILs **1–5**.

IL	Temperature [°C]														M_w	V_m
	20	SD[a]	30	SD	40	SD	50	SD	60	SD	70	SD	80	SD	[g mol^{-1}]	[cm^3 mol^{-1}]
1	1.308	0.00003	1.30076	0.00003	1.29416	0.00004	1.28733	0.00002	1.28046	0.00004	1.27359	0.00002	1.26653	0.00002	296.34	235.78
2	1.244	0.00003	1.23747	0.00003	1.23097	0.00004	1.22435	0.00003	1.21766	0.00002	1.21097	0.00002	1.20426	0.00001	324.39	260.94
3	1.180	0.00001	1.17363	0.00003	1.16704	0.00004	1.16041	0.00002	1.15379	0.00002	1.14703	0.00004	1.14011	0.00002	352.44	298.63
4	1.149	0.00005	1.14243	0.00003	1.13602	0.00003	1.12951	0.00001	1.12281	0.00003	1.11613	0.00003	1.10953	0.00002	380.49	332.24
5	1.113	0.00002	1.10659	0.00002	1.10002	0.00004	1.09346	0.00003	1.08683	0.00002	1.08011	0.00002	1.07337	0.00004	408.54	367.01

[a] standard deviation

Table S2. Logarithmic regression data regarding influence between alkyl length and density or refractive index values at 20 °C for ILs **1–5**.

Property	Coefficient values for $y = a - bln(x + c)$			R^2
	a	b	c	
Density	1.598	0.193	2.499	0.99
Refractive index	1.649	0.052	9.030	0.98

Table S3. Linear regression data regarding influence between temperature and values of density for ILs **1–5**.

IL	Coefficient values for $y = ax + b$		R^2
	a	b	
1	-0.0007	1.3213	0.9999
2	-0.0007	1.2574	1.0000
3	-0.0007	1.1937	0.9999
4	-0.0007	1.1622	0.9999
5	-0.0007	1.1265	1.0000

Table S4. Refractive index values determined for ILs **1–5**.

IL	Temperature [°C]													
	20	SD[a]	30	SD	40	SD	50	SD	60	SD	70	SD	80	
1	1.52382	0.00003	1.52160	0.00001	1.51905	0.00002	1.51650	0.00001	1.51399	0.00003	1.51148	0.00001	1.50881	
2	1.51392	0.00002	1.51146	0.00004	1.50886	0.00001	1.50633	0.00001	1.50387	0.00001	1.50143	0.00001	1.49887	
3	1.50917	0.00001	1.50686	0.00001	1.50441	0.00001	1.50189	0.00001	1.49939	0.00001	1.49687	0.00002	1.49430	
4	1.49953	0.00001	1.49703	0.00002	1.49440	0.00003	1.49170	0.00002	1.48897	0.00004	1.48611	0.00003	1.48356	
5	1.49546	0.00002	1.49287	0.00001	1.49014	0.00001	1.48735	0.00002	1.48453	0.00002	1.48163	0.00001	1.47897	

[a] standard deviation

Table S5. Linear regression data regarding influence between temperature and values of refractive index for ILs **1–5**.

IL	Coefficient values for $y = ax + b$		R^2
	a	b	
1	-0.00025	1.52902	0.9995
2	-0.00025	1.51893	0.9999
3	-0.00025	1.51427	0.9998
4	-0.00027	1.50504	0.9997
5	-0.00028	1.50113	0.9998

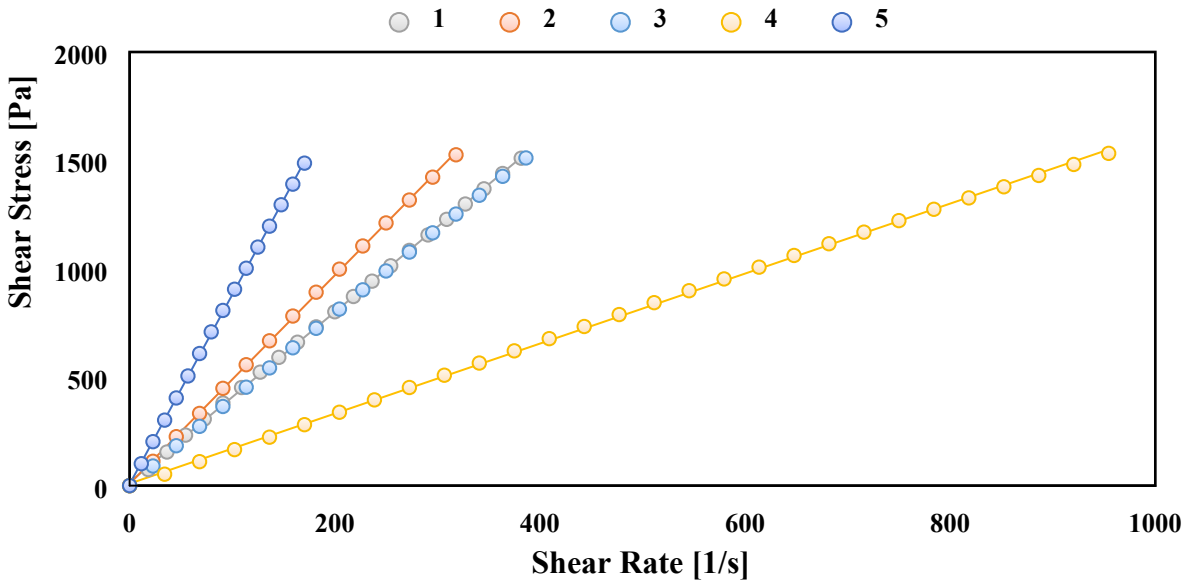

Figure S33. Shear stress versus shear rate for ILs **1–5**.

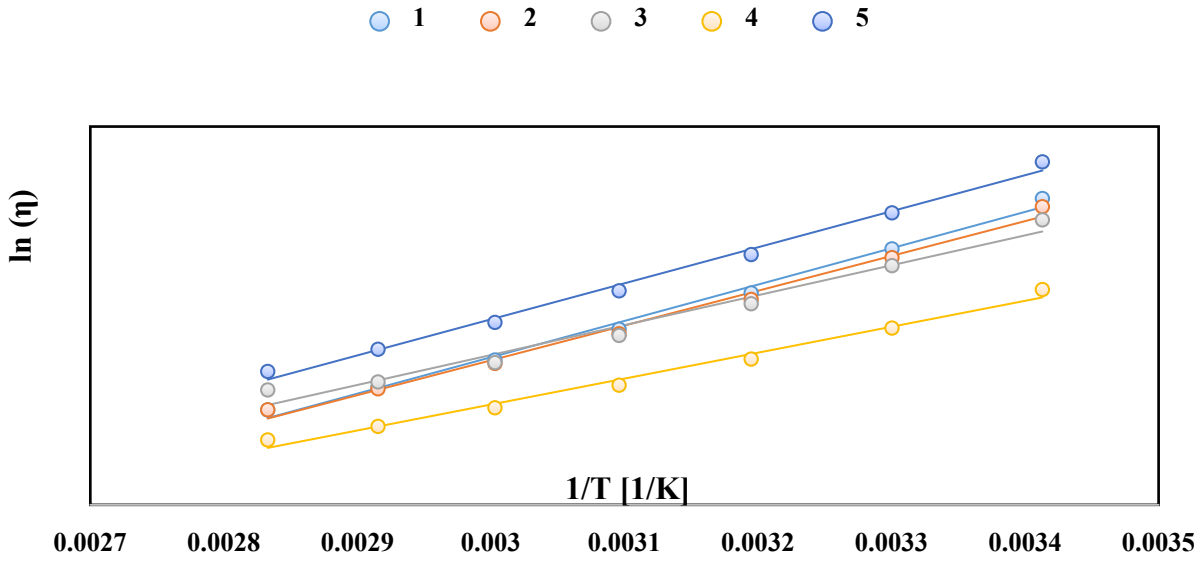

Fig. S34. Arrhenius plots for ILs **1–5**, where η is viscosity and T is temperature.

Table S6. Viscosity values (Pa s) and Arrhenius equation fitting parameters for ILs **1–5**.

IL	Temperature [°C]														E_a [kJ mol^{-1}]	$\ln\eta_\infty$ [b] [Pa s]	R^2
	20	**SD[a]**	**30**	**SD**	**40**	**SD**	**50**	**SD**	**60**	**SD**	**70**	**SD**	**80**	**SD**			
1	5.307	0.023	2.089	0.003	0.924	0.002	0.472	0.001	0.267	0.001	0.162	0.001	0.106	0.001	55.92	-21.45	0.99
2	4.557	0.020	1.776	0.003	0.820	0.001	0.434	0.001	0.251	0.001	0.157	0.001	0.106	0.001	53.55	-20.65	0.99
3	3.572	0.014	1.531	0.002	0.756	0.002	0.421	0.001	0.254	0.001	0.178	0.001	0.153	0.001	46.03	-17.84	0.98
4	0.984	0.001	0.482	0.001	0.272	0.001	0.168	0.001	0.110	0.001	0.078	0.001	0.061	0.001	39.89	-16.54	0.99
5	10.451	0.047	4.067	0.008	1.880	0.003	0.961	0.002	0.536	0.001	0.326	0.001	0.216	0.001	55.45	-20.58	0.99

[a] standard deviation; [b] According to Arrhenius-Guzmán equation: $\ln(\eta) = \ln\eta_\infty + Ea/(R \cdot T)$, where η is the dynamic viscosity, E_a is the activation energy for viscous flows, T is the temperature of the measurement, and $\ln\eta\infty$ is the natural logarithm of the viscosity at infinite temperature

Table S7. Octanol-water partition coefficient logarithms ($\log K_{OW}$) and cubic regression data regarding influence between alkyl length and $\log K_{OW}$ for ILs **1**–**8**.

IL	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**Coefficient values for** $y = ax^3 + bx^2 + cx + d$				R^2
									a	b	c	d	
$\log K_{OW}$	-1.064	-0.688	-0.329	0.340	1.059	1.231	1.577	1.775	-0.001	0.033	0.027	-1.250	0.98
SD[a]	0.011	0.042	0.054	0.006	0.013	0.007	0.011	0.016					

[a] standard deviation

Table S8. Relative (R), absolute (A) and total (T) deterrence coefficients regarding ILs' **1**–**8** activity toward tested organisms

IL	Granary weevil (*Sitophilus granarius* L.) Adults						Confused flour beetle (*Tribolium confusum* Duv.) Adults						Rice weevil (*Sitophilus oryzae* L.) Adults						Khapra beetle (*Trogoderma granarium* Ev.) Larvae					
	R		A		T		R		A		T		R		A		T		R		A		T	
1	70	a	-5	a	**64**	a	43	ab	19	a	**62**	ab	38	a	6	a	**44**	a	20	ab	-5	a	**15**	ab
2	76	a	19	abc	**95**	a	10	a	1	a	**11**	a	53	ab	8	a	**62**	ab	48	bc	4	ab	**52**	b
3	45	a	18	abc	**64**	a	82	b	2	a	**84**	ab	63	abc	5	a	**68**	abc	-19	a	-23	a	**-42**	a
4	56	a	9	ab	**65**	a	82	b	-5	a	**77**	ab	39	a	3	a	**42**	a	12	ab	7	ab	**19**	ab
5	95	a	32	abc	**127**	a	96	b	25	a	**121**	b	94	bc	16	ab	**110**	bcd	89	c	-1	a	**87**	bc
6	60	a	36	abc	**97**	a	99	b	20	a	**119**	b	98	c	20	abc	**118**	cd	99	c	63	bc	**162**	cd
7	79	a	48	bc	**127**	a	97	b	15	a	**112**	b	94	bc	51	bc	**145**	d	92	c	90	c	**182**	d
8	63	a	66	c	**129**	a	86	b	16	a	**102**	b	90	bc	52	c	**141**	d	95	c	92	c	**187**	d
$LSD_{0.05}$	62.3		53.8		80.5		65.2		42.6		79.0		44.6		36.7		57.8		68.2		64.8		85.0	
Azadirachtin (reference)	99		91		190		100		85		185		-		-		-		100		94		194	