Electronic Supplementary Material (ESI) for Green Chemistry

Supporting Information

On the Ethenolysis of Natural Rubber and Squalene

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General Experimental

All chemicals were purchased as reagent grade from commercial suppliers and used without further purification unless otherwise noted. Solvents were dried by passing over Al_2O_3 and/or by storing over molecular sieves unless otherwise noted. C_6D_6 was dried over molecular sieves and degassed by freeze-pump thaw cycles technique and stored under Argon. Column chromatography was performed using silica gel 60 (0.063–0.20 mesh ASTM) or with flash gel (15 - 40 μm). TLC was performed by using Fluka silica gel 60 F254 (0.2 mm) on alumina plates. NMR spectra were recorded on Bruker DRX500 and Bruker DRX300. The chemical shifts (δ) are given in ppm relative to TMS (0 ppm) or to the respective solvent signals, coupling constants are (J) in Hz. MS spectra were recorded on a Finnigan MAT95 spectrometer. GC experiments were done on a Clarus 500 GC with autosampler and FID detector. Column: Varian CP-Sil 8 CB (l = 15 m, diam. = 0.25 mm, d_F = 1.0 μm), N_2 (flow: 20 ml/min; split 1:9); Injector-temperature: 300 °C, detector temperature: 300 °C. Temperature program: isotherm 50°C for 3 min, heating to 200°C with 10°C/min, heating to 310°C with 25°C/min, isotherm for 12 min. Tetradecane was added as internal standard. HPLC experiments were done on a Merck-Hitachi L-6200 HPLC with a L-4250 UV/Vis detector (WL=203nm). Column: Merck Purospher RP 18e Hibar / 250 x 4.6 mm, solvent: methanol: acetonitrile 8:1, flow: 2 mL/min. GC/MS was done on a Thermoquest Trace GC 2000-series with Atas programmable injector optic 2 and a Finnigan Trace DSQ MS-detector. Column: restec RXI 5 fil (l = 30 m, diam. = 0.25 mm, d_F = 0.25 μm); Injector temperature: initial: 80°C heating to 320°C with 16°C/s. Temperature program: isotherm 40°C for 2 min, heating to 160°C with 20°C/min, heating to 310°C with 6°C/min, isotherm for 12 min. Natural rubber (SVR 3L, Vietnam) was obtained from Weber & Schaer GmbH & Co. KG, Hamburg, Germany. Liquid natural rubber was obtained from Aldrich.

For safety reasons the ethene cylinders were connected only prior to the reaction to adjust pressure in the reactor, then they were separated. A large reactor volume allows maintaining constant ethene pressure throughout the reaction. In contrast, in a small reactor the amount of ethene consumed in the ethenolysis reaction is not negligible relative to the reactor volume leading to pressure decrease during the reaction.

General procedure for squalene ethenolysis (catalyst screening)

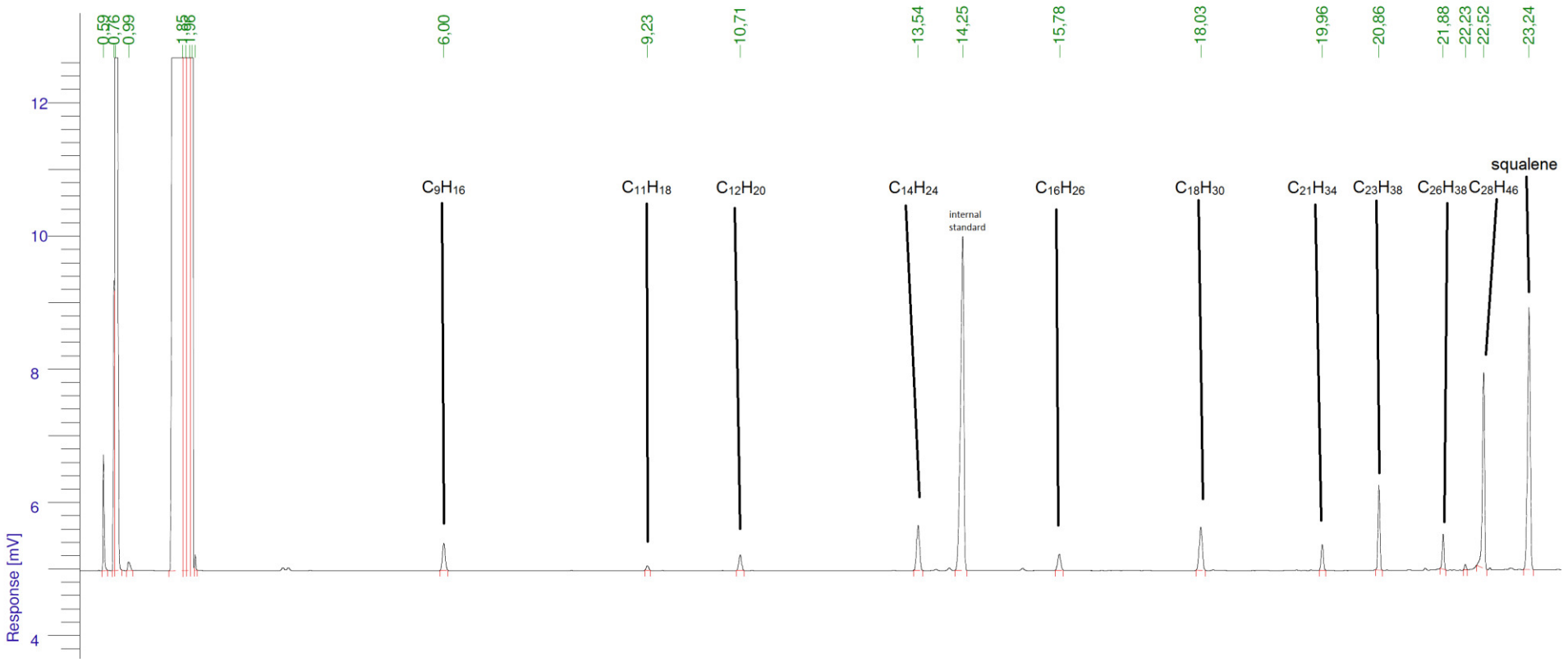
Squalene (100 mg) was dissolved in the respective solvent (5 mL) and the solution syringed into a 250 mL Büchi miniclave. To this solution was added a stock solution of the respective catalyst complex (0.005 – 0.1 mol% catalyst relative to the substrate) in toluene (0.005 – 0.1 mol% per mL toluene). The reactor was closed and purged with ethene for 5 min (alternatively for volatile solvents freeze-and-thaw cycles were employed). The ethene pressure in the reactor was then adjusted to 7 bar and the reactor heated to the respective reaction temperature during the designated reaction time. Then the reactor was allowed to rt, ethyl vinyl ether added (1 ml) and small samples analyzed by GC, GC/MS and HPLC.

Table SI-1. GC retention times of squalene ethenolysis products.

Product	Retention time [min] GC	Retention time [min] GC-MS
squalene	23.3	28.8
$C_{28}H_{46}$	22.5	27.0
$C_{26}H_{38}$	21.9	25.3
$C_{23}H_{38}$	20.9	21.8
$C_{21}H_{34}$	19.9	18.9
$C_{18}H_{30}$	18.0	15.1
$C_{16}H_{26}$	15.8	12.4
$C_{14}H_{24}$	13.5	10.4
$C_{12}H_{20}$	10.7	8.5
$C_{11}H_{18}$	9.2	7.6
C_9H_{16}	6.0	5.6
C_7H_{12}	5.75	

C_7H_{12} was identified via identical retention times of an authentic commercial sample.

Figure SI-1. Representative GC-trace from squalene ethenolysis using complex 1.



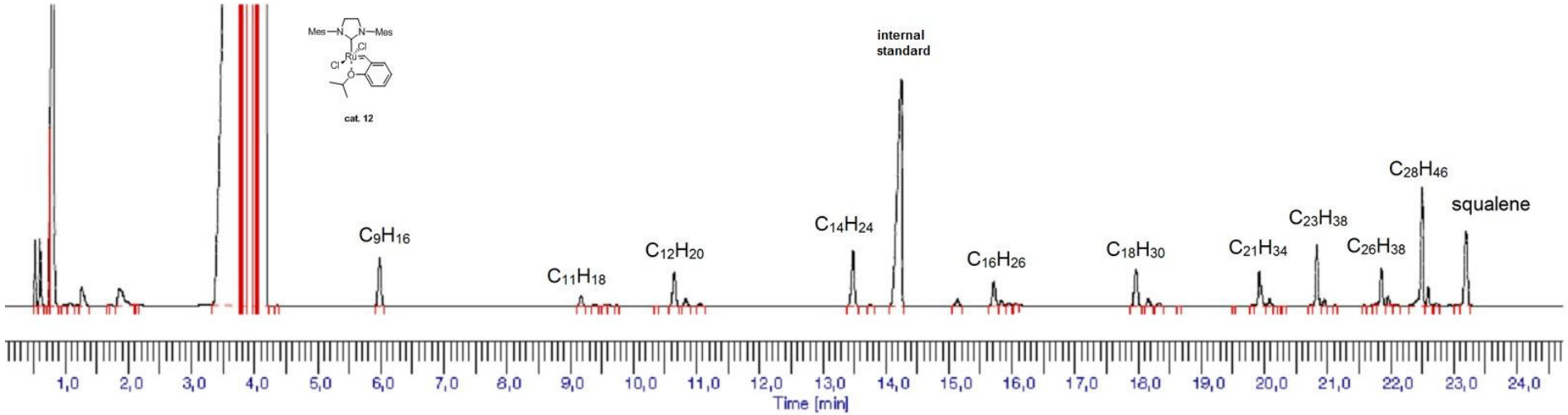


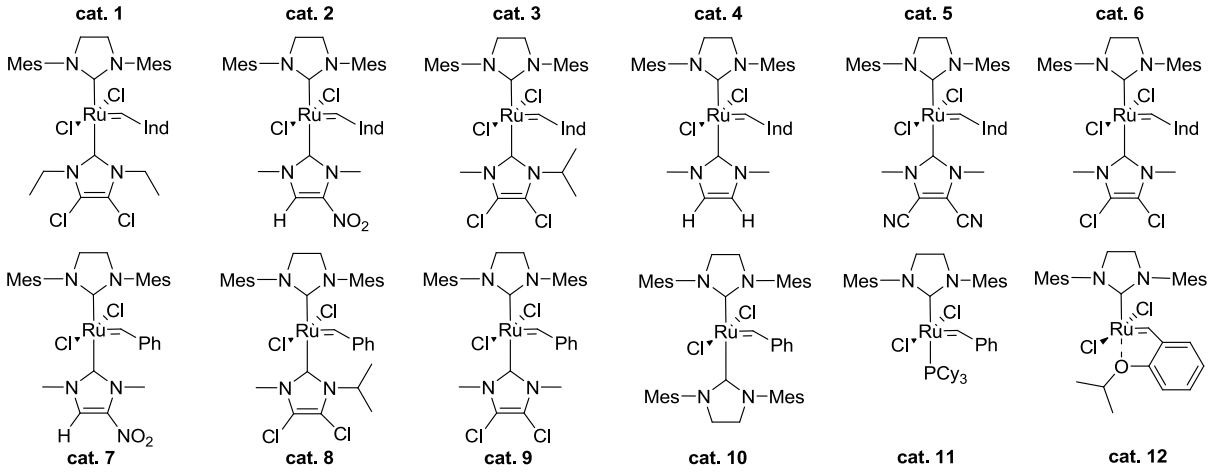
Figure SI-2. Representative GC-trace from squalene ethenolysis using Grubbs-Hoveyda complex **12**.

Extended table **SI-2**. Catalyst optimization (according to the general screening procedure)

	catalyst	catalyst (mol%)	conditions	conver. (rel. to internal n-tetradecane)	squalene	**$C_{28}H_{46}$**	$C_{26}H_{38}$	**$C_{23}H_{38}$**	$C_{21}H_{34}$	**$C_{18}H_{30}$**	$C_{16}H_{26}$	**$C_{14}H_{24}$**	$C_{12}H_{20}$	$C_{11}H_{18}$	**C_9H_{16}**
1	Cat. 1	0.1	100°C, 20h, C_6D_6	99%	-	1059	1387	886	3463	953	3739	2373	7709	2663	35343
2	Cat. 1	0.1	100°C, 6h, C_6D_6	99%	720	3337	4931	3146	10688	3799	12857	6678	20352	7992	7387
3	Cat. 1	0.05	100°C, 6h, C_6D_6	82%	18837	20904	5893	10772	7552	10395	7725	11880	8192	3007	9600
4	Cat. 1	0.01	100°C, 3h, C_6D_6	33%	66350	16680	1052	5854	787	4821	720	4351	597	196	3050
5	Cat. 1	0.01	100°C, 1h, C_6D_6	36%	43561	12554	850	4247	663	3493	596	3100	467	147	2111
6	Cat. 1	0.01	120°C, 3h, C_6D_6	65%	16376	10229	1527	3715	1267	3237	1156	3202	1050	317	1885
7	Cat. 1	0.01	120°C, 1h, C_6D_6	32%	66230	14612	756	5041	556	4080	480	3577	390	-	2449
8	Cat. 1	0.01	120°C, 3h, Toluene	65%	28165	19423	3410	7188	2660	6091	2311	5894	2055	641	3858
9	Cat. 1	0.005	120°C, 3h, Toluene	25%	76900	12226	463	4022	338	3186	295	2811	225	-	1800
10	Cat. 1	0.005	140°C, 3h, Toluene	20%	25079	2210	-	722	-	511	-	450	-	-	-
11	Cat. 1	0.005	120°C, 3h, Toluene, 8% wght. Squalene	35%	53057	14933	961	4770	694	3803	597	3357	482	139	2144
12	Cat. 1	0.005	120°C, 3h, Toluene 20%wght. squalene	45%	153975	64792	4428	22418	3664	18513	3090	17679	2748	-	11205
12	Cat. 1	0.005	120°C, 3h, CH_2Cl_2	3%	84285	1738	-	657	-	324	-	214	-	-	-
14	Cat. 1	0.005	120°C, 3h, Pentane	5%	47450	1437	-	580	-	312	-	267	-	-	-
15	Cat. 1	0.005	120°C, 3h, C_6H_{12}	1%		-	-	-	-	-	-	-	-	-	-
16	Cat. 5	0.005	120°C, 3h, Toluene	2%	89300	1601	-	631	-	-	-	-	-	-	-
17	Cat. 3	0.005	120°C, 3h, Toluene	3%	85475	1664	-	710	-	-	-	-	-	-	-
18	Cat. 3	0.01	120°C, 3h, Toluene	33%	60878	12912	685	4199	490	3332	384	2834	318	81	1837
19	Cat. 6	0.005	120°C, 3h, Toluene	1%		-	-	-	-	-	-	-	-	-	-
20	Cat. 2	0.01	120°C, 3h, Toluene	75%	30879	33561	9174	12659	7609	10847	6306	11282	5362	1504	6284
21	Cat. 4	0.01	120°C, 3h, Toluene	48%	65000	30633	3424	10519	2851	8059	2110	8719	1901	469	3766
22	Cat. 8	0.01	120°C, 3h, Toluene	33%	76827	18145	1030	5740	700	4651	614	4069	474	135	2499
23	Cat. 8	0.005	120°C, 3h, Toluene	6%	108480	6090	50	1943	-	1414	-	1250	-	-	-
24	Cat. 7	0.005	120°C, 3h, Toluene	6%	63440	1826	95	885	364	446	-	345	-	-	-
25	Cat. 9	0.01	120°C, 3h, Toluene	55%	52145	26556	3401	9075	2780	7091	2106	7589	1837	504	3479
26	Cat. 10	0.005	120°C, 3h, Toluene	1%		-	-	-	-	-	-	-	-	-	-
27	Cat. 10	0.005	140°C, 3h, Toluene	3%	92401	1602	-	632	130	327	-	237	-	-	152
28	Cat. 11	0.01	120°C, 3h, Toluene	37%	85270	13162	517	5242	-	3952	-	3496	-	-	1968
29	Cat. 12	0.005	120°C, 3h, Toluene	15%	81633	16173	745	5807	622	4591	557	4076	421	151	2508
30	Cat. 12	0.01	120°C, 3h, Toluene	85%	11117	13945	4039	7107	5079	6019	3846	8379	4914	1448	6862

The numbers in the columns correspond to the integrals of the respective peaks in the gc traces.

Figure SI-3. List of complexes employed in squalene ethenolysis (Mes = 2,4,6-trimethyl-phenyl and Ind = 3-phenyl-indene-ylidene)



General procedure for the ethenolysis of natural rubber and liquid natural rubber (catalyst screening)

NR or LNR (50 mg) was placed in a 250 mL Büchi miniclave and C_6D_6 (5 mL) added with stirring (dissolving natural rubber requires overnight treatment). To this mixture was added a stock solution of the respective catalyst complex (0.01 – 0.2 mol% catalyst relative to the substrate) in C_6D_6 (0.01 – 0.2 mol% per mL C_6D_6). The reactor was closed and degassed by three freeze-and-thaw cycles to replace all other gases by ethene. The ethene pressure in the reactor was adjusted to 7 bar and the reactor heated to the respective reaction temperature for the designated reaction time. After the reaction the reactor was allowed to rt and a small sample analyzed by ^{1}H NMR.

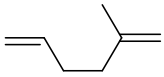
Ethenolysis of natural rubber (5 g scale)

NR or LNR (5 g) was place in a 250 mL Büchi miniclave and toluene (100 mL) added with stirring (dissolving natural rubber requires overnight treatment). Catalyst **1** (63.1 mg, 0.073 mmol, 0.1 mol% per double bond) was added. The reactor was purged with ethene for 5 min, the ethene pressure adjusted to 7 bar and the reactor heated to 120 ^{o}C for 3 h. After the reaction the reactor was allowed to rt and ethyl vinyl ether (0.5 ml) was added for catalyst deactivation. The reaction mixture was transferred to a millipore cell and filtered over a nanofiltration membrane (MWCO 500 Dalton, flow: 1ml/min, Δp = 3 bar) to remove residual polymers. After filtering off ca. 80 mL of the

solution, toluene (100 mL) was added and a volume of ca. 100 mL filtered through the membrane. The toluene was carefully removed from the permeate to obtain ca. 3.2 g of residue composed of the various oligoisoprenes (up to ca. n= 10), which were purified by column chromatography (silica, pentane).

2-Methyl-hexa-1,5-diene

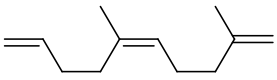
This compound is formed in the ethenolysis reaction, but could not be not isolated due to its high volatility); the spectrum listed here was obtained using commercially available 2-methyl-hexa-1,5-diene. The data are listed here only for comparison with the higher oligomers and to support NMR assignment in the higher oligomers.



^{1}H NMR (500 MHz, $CDCl_3$) δ 5.89 – 5.76 (m, 1H *(5)*), 5.03 (d, d *J*= 17 Hz, 5 Hz), 1H *(6)*), 4.96 (d, d *J*= 10 Hz, 5 Hz) 1H *(6)*), 4.71 (d, *J*= 10 Hz),m, 2H *(1)*), 2.25 – 2.05 (m, 4 H *(3,4-CH_2)*), 1.73 (m, 3H, *2*-methyl).

^{13}C NMR (126 MHz, $CDCl_3$) δ 145.50, 138.62, 114.60, 110.19, 37.27, 32.05, 22.58.

2,6-Dimethyl-deca-1,5,9-triene (n= 2)



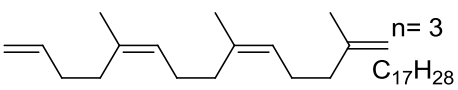
^{1}H NMR (500 MHz, $CDCl_3$) δ 5.90 – 5.78 (m, 1H *(9)*), 5.15 (d, *J*= 13 Hz), m, 1H *(5)*), 5.02 (d, *J*= 17 Hz), m, 1H *(10)*), 4.95 (d, *J*= 13 Hz),m, 1H *(10)*), 4.71 (d, *J*= 13 Hz),m, 2H *(1)*), 2.15 – 2.00 (m, 8 H *(3,4,7,8-CH_2)*), 1.72 (s, 3H, *2*-methyl), 1.69 (m, 3H, *6*-methyl).

^{13}C NMR (126 MHz, $CDCl_3$) δ 145.98, 138.85, 134.98, 125.25, 114.53, 109.98, 38.24, 32.41, 31.50, 26.25, 23.47, 22.60.

Due to the significant volatility of 2,6-dimethyldeca-1,5,9-triene only a small of amount of this compound was obtained after column chromatography (HPLC) with a purity of only ca. 80%.

GC-MS: $C_{12}H_{20}$ calc. 164.3, obsd. 164.8; (product too volatile for HR-MS)

2,6,10-Trimethyl-tetradeca-1,5,9,13-tetraene (n= 3)



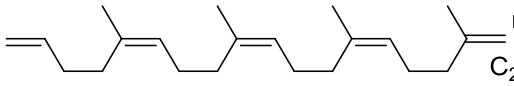
^{1}H NMR (500 MHz, $CDCl_3$) δ 5.89 – 5.76 (m, 1H *(13)*), 5.19 – 5.10 (m, 2H *(5,9)*), 5.02 (d, *J*= 17 Hz), m, 1H *(14)*), 4.95 (d, *J*= 13 Hz),m, 1H *(14)*), 4.70 (d, *J*= 13 Hz),m, 2H *(1)*), 2.17 – 2.00 (m, 12H *(3,4,7,8,11,12-CH_2)*), 1.73 (s, 3H, *2*-methyl), 1.70 (m, 3H, *6*-methyl), 1.69 (m, 3H *10*-methyl).

^{13}C NMR (126 MHz, $CDCl_3$) δ 145.98, 138.80, 135.37, 134.93, 125.48, 125.07, 114.54, 109.93, 38.26, 32.48, 32.41, 31.46, 26.53, 26.30, 23.56, 23.49, 22.64.

HR-MS: $C_{17}H_{28}$ calc. 232.2191; obsd. 232.22214.

purity after column chromatography (HPLC) > 93%

2,6,10,14-Tetramethyl-octadeca-1,5,9,13,17-pentaene (n= 4)



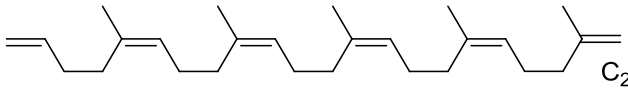
^{1}H NMR (500 MHz, $CDCl_3$) δ 5.86 – 5.77 (m, 1H *(17)*), 5.18 – 5.10 (m, 3H *(5,9,13)*), 5.03 (d, *J*= 17 Hz), m, 1H *(18)*), 4.95 (d, *J*= 13 Hz) m, 1H *(18)*), 4.70 (d, *J*= 13 Hz), m, 2H *(1)*), 2.15 – 2.01 (m, 16H *(3,4,7,8,11,12,15,16)*), 1.72 (s, 3H, *2*-methyl), 1.70 – 1.67 (m, 9H, *6,10,14*-methyl).

^{13}C NMR (126 MHz, $CDCl_3$) δ 145.99, 138.81, 135.41, 135.38, 134.92, 125.46, 125.27, 125.03, 114.54, 109.94, 38.26, 32.48, 32.41, 32.36, 31.48, 26.59, 26.53, 26.29, 23.58, 23.56, 23.49, 22.65.

HR-MS: $C_{22}H_{36}$ calcd. 300.2817; obsd. 300.2811.

purity after column chromatography (HPLC) > 94%

2,6,10,14,18-Pentamethyl-docosa-1,5,9,13,17,21-hexaene (n= 5)



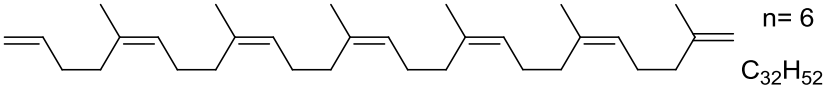
^{1}H NMR (500 MHz, $CDCl_3$) δ 5.87 – 5.76 (m, 1H *(21)*), 5.19 – 5.10 (m, 4H *(5,9,13,17)*), 5.03 (d, *J*= 17 Hz, m, 1H *(22)*), 4.95 (d, *J*= 13 Hz, m, 1H *(22)*), 4.70 (d, *J*= 13 Hz, m, 2H *(1)*), 2.16 – 2.01 (m, 20H *(3,4,7,8,11,12,15,16,19,20)*), 1.73 (s, 3H, *2*-methyl), 1.69 (s, 12H, *6,10,14,18*-methyl).

^{13}C NMR (126 MHz, $CDCl_3$) δ 145.98, 138.79, 135.40, 135.34, 134.90, 125.45, 125.24, 125.21, 125.01, 114.54, 109.93, 38.26, 32.49, 32.41, 32.37, 32.36, 31.47, 26.59, 26.58, 26.52, 26.28, 23.59, 23.57, 23.51, 22.66. Due to overlapping peaks the number of ^{13}C peaks is lower than the number of inequivalent carbon atoms.

HR-MS: $C_{27}H_{44}$ calcd. 368.3443; obsd. 368.3448.

purity after column chromatography (HPLC) > 96%.

2,6,10,14,18,22-Hexamethyl-hexacosa-1,5,9,13,17,21,25-heptaene (n= 6)



^{1}H NMR (500 MHz, $CDCl_3$) δ 5.87 – 5.76 (m, 1H *(25)*), 5.17 – 5.10 (m, 5H *(5,9,13,17, 21)*), 5.02 (d, *J*= 17 Hz, m, 1H *(22)*), 4.94 (d, *J*= 13 Hz), m, 1H *(26)*), 4.70 (d, *J*= 13 Hz, m, 2H *(1)*), 2.13 – 2.00 (m, 24H *(3,4,7,8,11,12,15,16,19,20,23,24)*), 1.72 (s, 3H, *2*-methyl), 1.69 (s, 15H, *6,10,14,18,22,*-methyl).

^{13}C NMR (126 MHz, $CDCl_3$) δ 145.99, 138.81, 135.42, 135.38, 135.34, 134.92, 125.46, 125.26, 125.21, 125.02, 114.54, 109.94, 38.26, 32.48, 32.41, 32.37, 31.47, 26.58, 26.51, 26.28, 23.58, 23.50, 22.65. Due to overlapping peaks the number of ^{13}C peaks is lower than the number of inequivalent carbon atoms.

HR-MS: $C_{32}H_{52}$ calc. 436.4069; obsd. 436.41079.

purity after column chromatography (HPLC) > 97%.

Figure S-3. HPLC trace of permeate after nanofiltration.

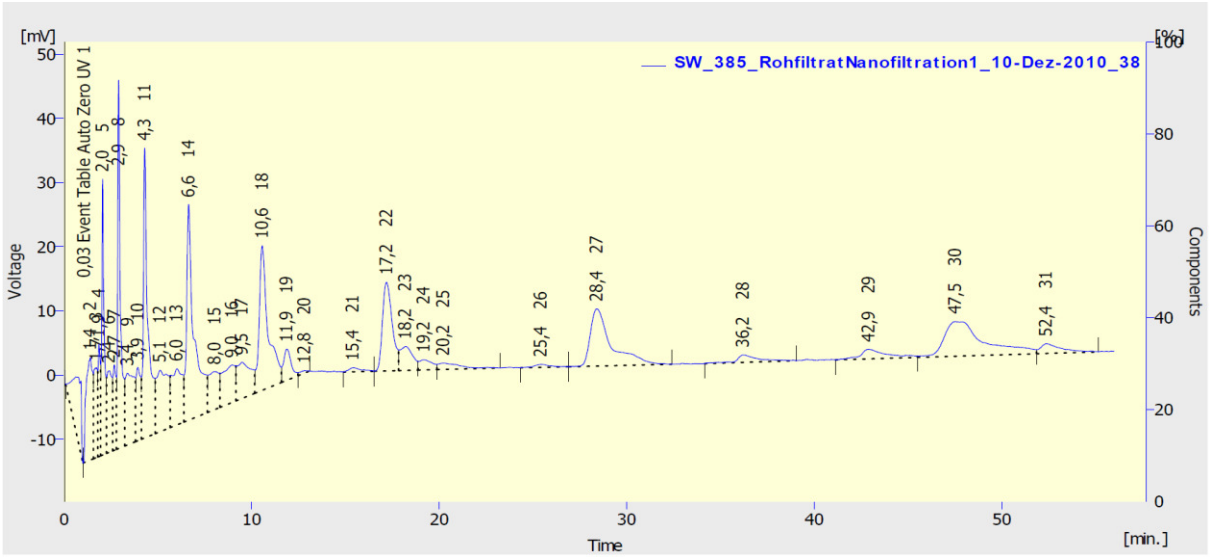
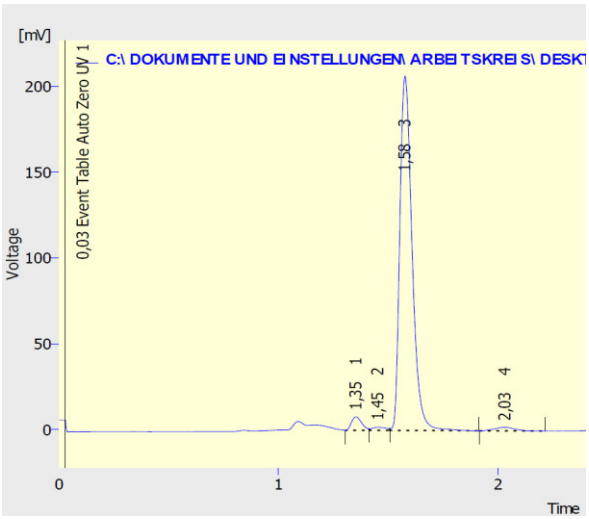


Figure S-4. HPLC traces of the oligoisoprenes including EI mass spectra and hi-res mass spectral data.

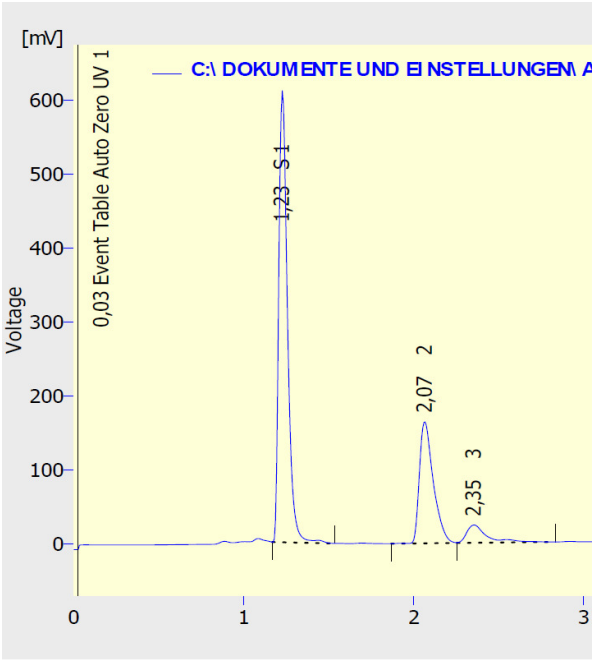
n=1 (commercial sample)



Result Table (Uncal - C:\DOKUMENTE UND EINSTELLUNGEN\ARBEITSKREIS\DESKTOP\SPALTUNG_NR_HPLC\2METHYL1,5HEXADIEN1_BEARBEITET)

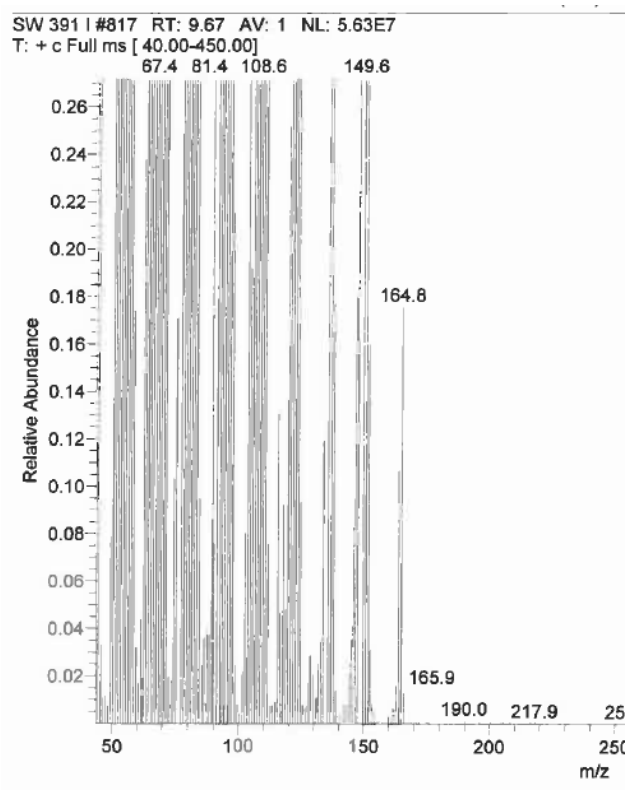
	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]
1	1,353	25,210	7,864	3,0	3,6
2	1,453	8,044	1,850	1,0	0,8
3	1,577	799,344	206,588	94,6	94,6
4	2,033	12,634	2,016	1,5	0,9
	Total	845,232	218,318	100,0	100,0

n=2

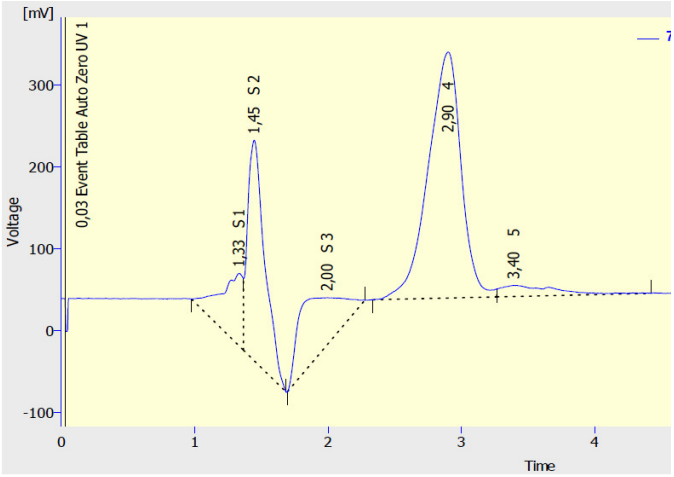


Result Table (Uncal - C:\DOKUMENTE UND EINSTELLUNGEN\ARBEITSKREIS\DESKTOP\SPALTUNG_NR_HPLC\SW_391_F74-82 _BEARBEITET)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]
2	2,067	930,038	164,089	82,2	87,2
3	2,353	202,015	24,160	17,8	12,8
	Total	1132,053	188,250	100,0	100,0



n=3



Result Table (Uncal - 73SW_394_F106-1301_20-JAN-2011_73)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]
4	2,903	5158,124	300,047	93,0	95,6
5	3,397	387,602	13,650	7,0	4,4
	Total	5545,726	313,698	100,0	100,0

Massenspektrometrie
Hochauflösung

Auftraggeber: WOLF

Probe: SW385F3

Aufnahme Nr.: 68878

Gemessene Masse: 232.22214

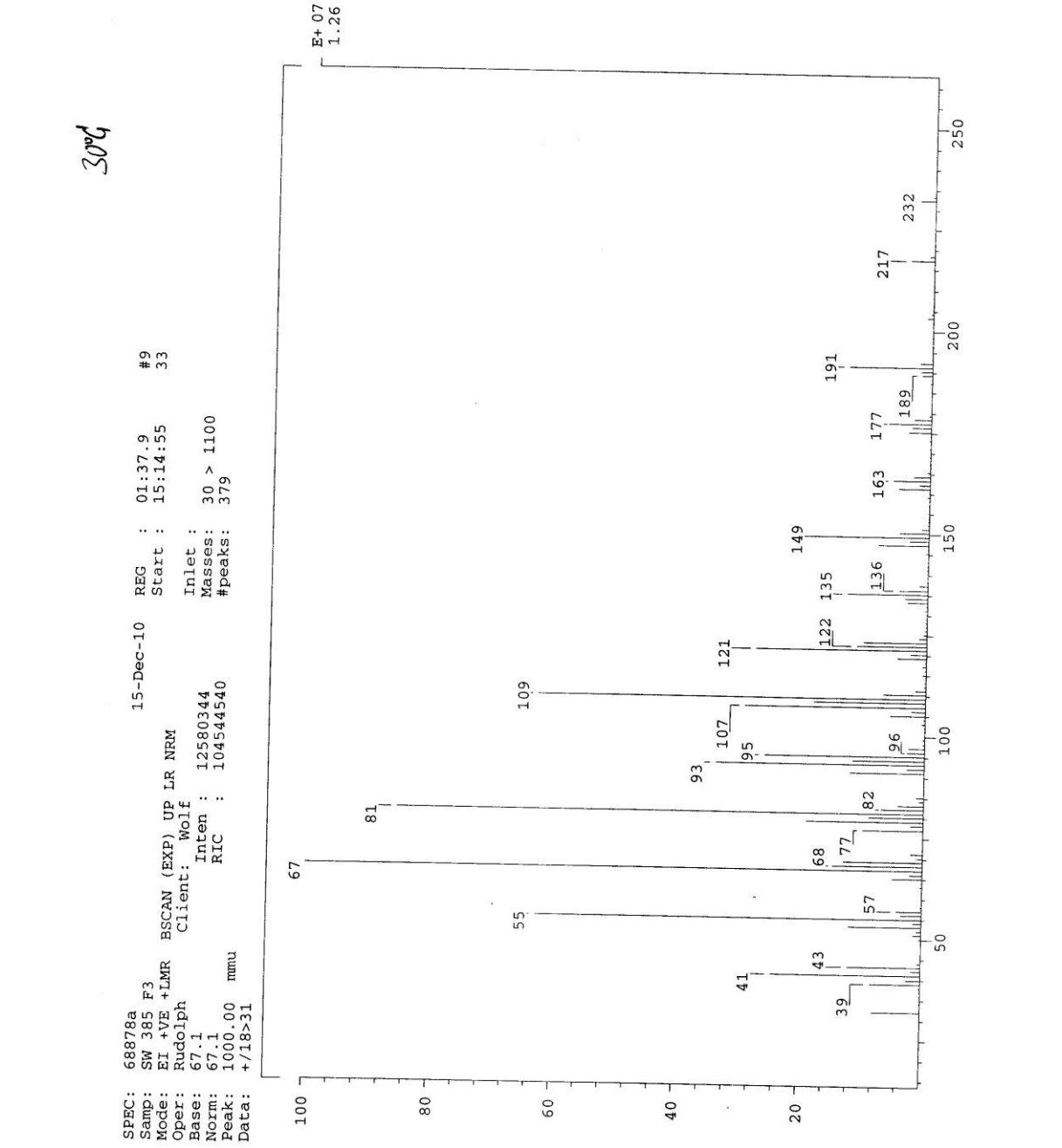
Zulässige Abweichung: 5 (mmu)

Berücksichtigte Elemente (max):
30 C, 5 N, 5 O,

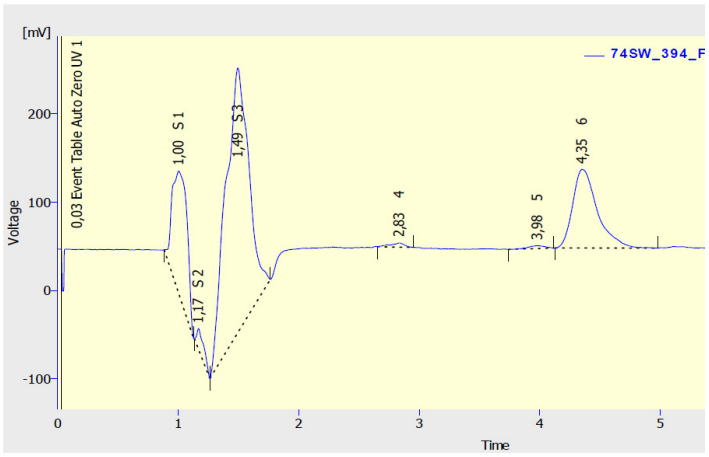
	Summenformel	Abweichung (mmu)	Ber. Masse
*	C11 H28 N4 O1	-4.3	232.2264
*	C17 H28	3	232.2191

22.12.10

f. Schimpf


SPEC: 68878a
Samp: SW 385 F3
Mode: EI +VE +LMR BSCAN (EXP) UP LR NRM
Oper: Rudolph Client: Wolf
Base: 67.1 Inten : 12580344
Norm: 67.1 RIC : 104544540
Peak: 1000.00 mmu
Data: +/18>31
15-Dec-10 REG : 01:37.9 #9
Start : 15:14:55 33
Inlet :
Masses: 30 > 1100
#peaks: 379
E+ 07
1.26
100
80
60
40
20
50
100
150
200
250
67
81
55
109
93
107
95
121
41
43
39
57
68
77
82
96
122
135
136
149
163
177
189
191
217
232

n=4



Result Table (Uncal - 74SW_394_F146-1661_20-JAN-2011_74)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]
4	2,833	38,996	4,744	2,9	4,9
5	3,980	30,954	3,294	2,3	3,4
6	4,350	1265,765	88,789	94,8	91,7
	Total	1335,715	96,828	100,0	100,0

Massenspektrometrie
Hochauflösung

Auftraggeber: WOLF

Probe: SW385F5

Aufnahme Nr.: 68879

Gemessene Masse: 300.28105

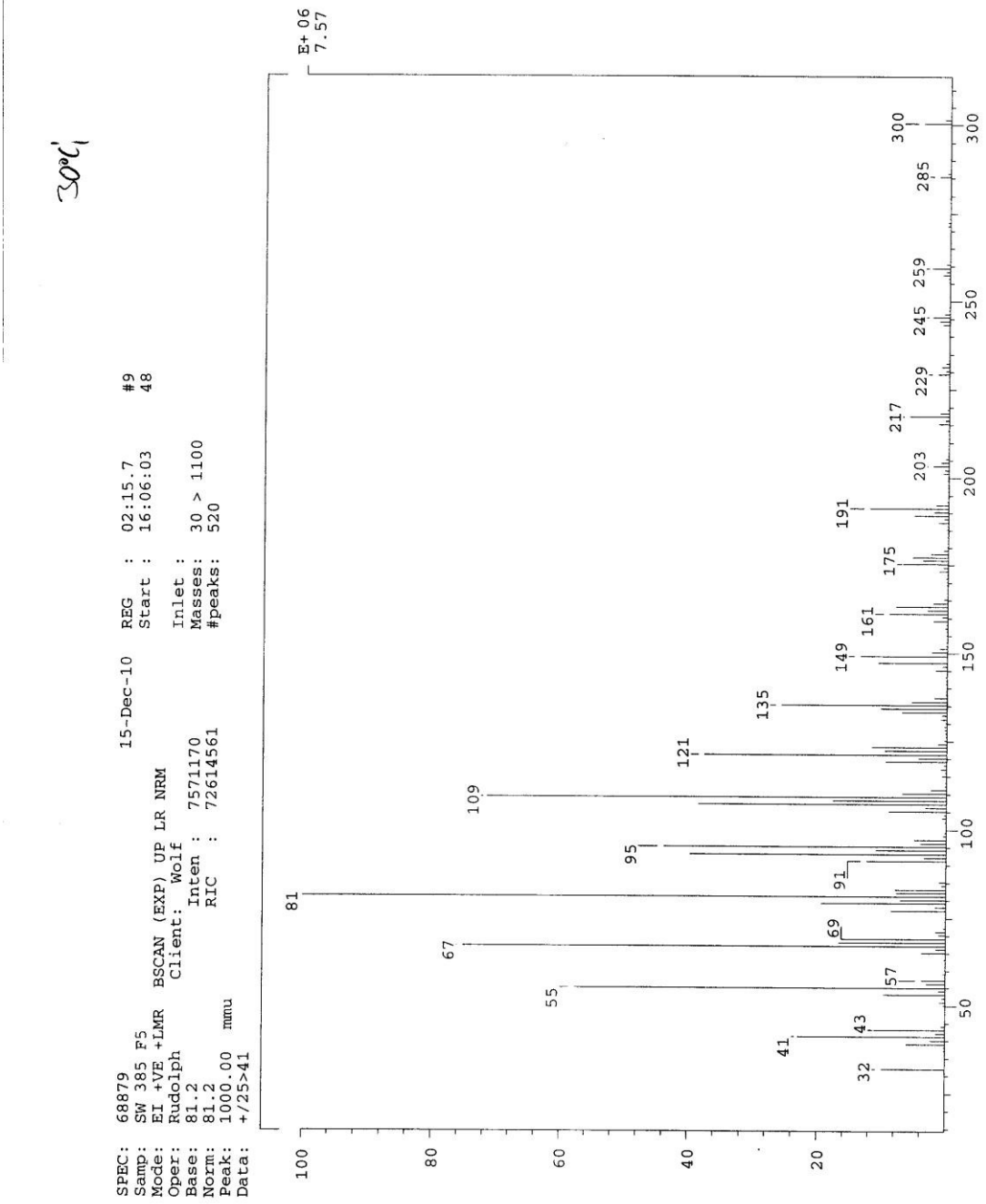
Zulässige Abweichung: 5 (mmu)

Berücksichtigte Elemente (max):
40 C, 5 N, 5 O,

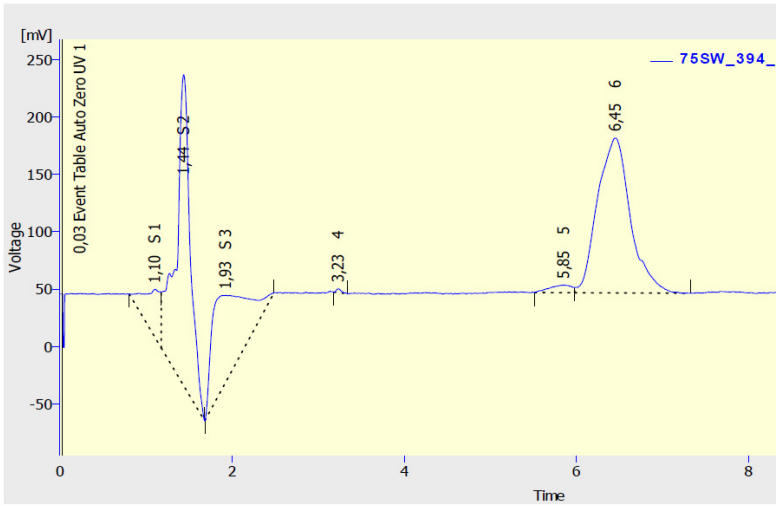
Summenformel	Abweichung (mmu)	Ber. Masse
C15 H34 N5 O1	4.6	300.2734
C17 H36 N2 O2	3.3	300.2777
* C22 H36	-0.7	300.2817 *

22.12.10

f. Schindler


SPEC: 68879
Samp: SW 385 F5
Mode: EI +VE +LMR
Oper: Rudolph
Base: 81.2
Norm: 81.2
Peak: 1000.00 mmu
Data: +/25>41
15-Dec-10
BSCAN (EXP) UP LR NRM
Client: Wolf
Inten : 7571170
RIC : 72614561
REG : 02:15.7 #9
Start : 16:06:03 48
Inlet :
Masses: 30 > 1100
#peaks: 520
E+ 06
7.57
81
67
55
109
95
121
135
149
161
175
191
203
217
229
245
259
285
300
41
43
57
69
91
32

n=5



Result Table (Uncal - 75SW_394_F220-2321_20-JAN-2011_75)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]
4	3,233	14,008	3,369	0,4	2,3
5	5,853	120,236	6,808	3,2	4,7
6	6,453	3581,832	134,865	96,4	93,0
	Total	3716,076	145,042	100,0	100,0

Massenspektrometrie
Hochauflösung

Auftraggeber: WOLF

Probe: SVV385F8

Aufnahme Nr.: 68800

Gemessene Masse: 368.34484

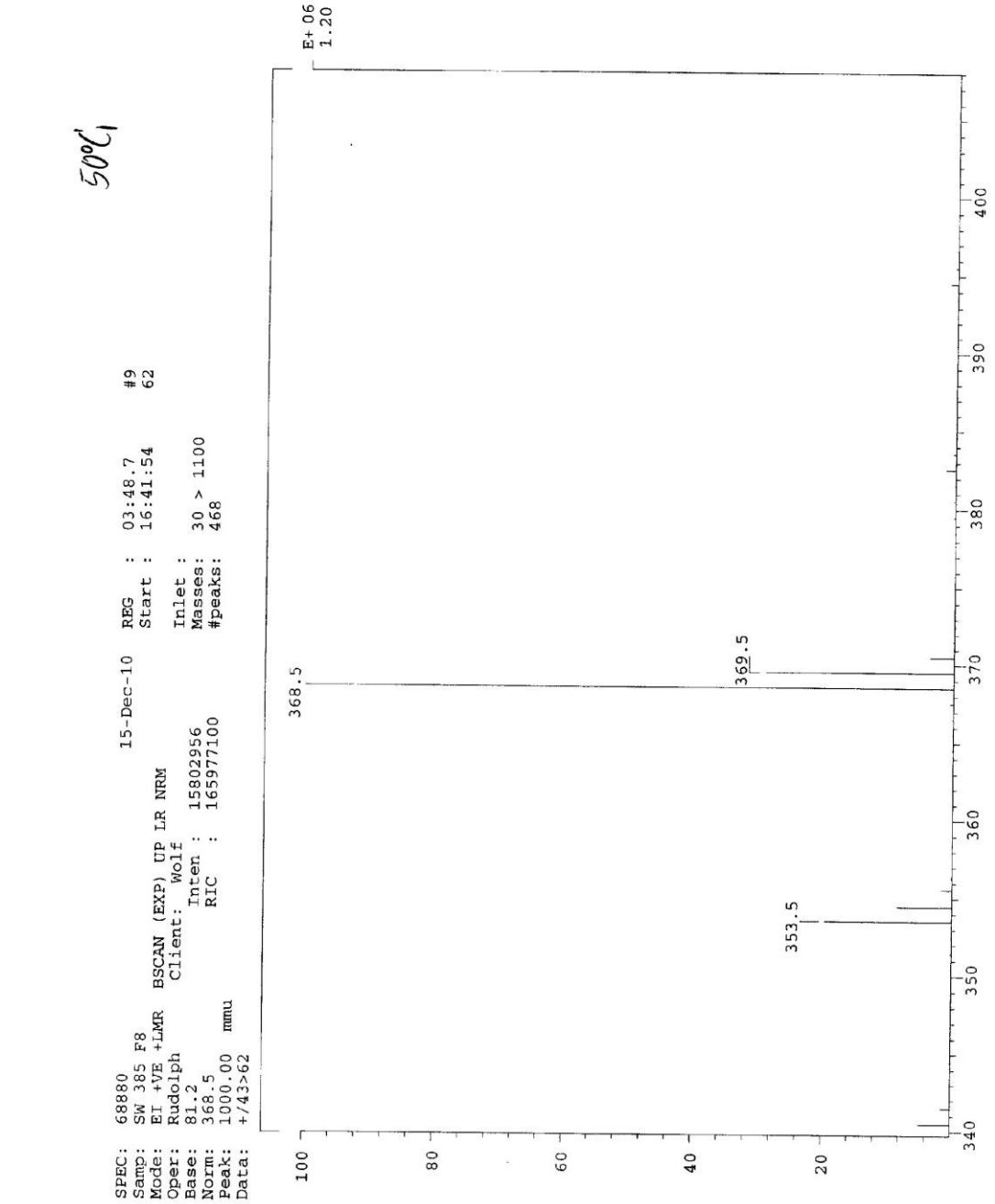
Zulässige Abweichung: 5 (mmu)

Berücksichtigte Elemente (max):
40 C, 5 N, 5 O,

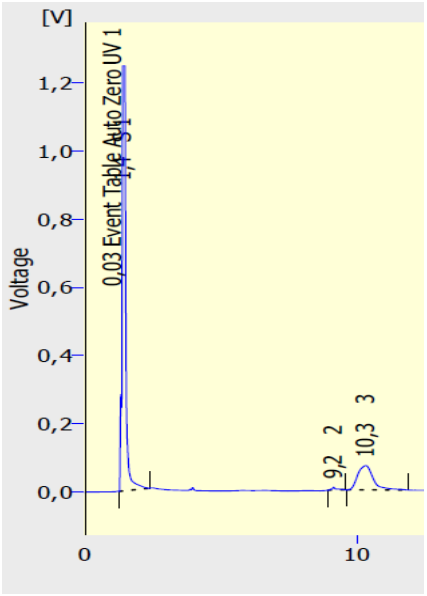
	Summenformel	Abweichung (mmu)	Ber. Masse
	C18 H48 N3 O4	-4	368.3488
	C22 H44 N2 O2	4.5	368.3403
*	C27 H44	0.5	368.3443 *

22.12.10

J. Salinas


50°C
SPEC: 68880
Samp: SW 385 F8
Mode: EI +VE +LMR BSCAN (EXP) UP LR NRM
Oper: Rudolph Client: Wolf
Base: 81.2 Inten : 15802956
Norm: 368.5 RIC : 165977100
Peak: 1000.00 mmu
Data: +/43>62
15-Dec-10 REG : 03:48.7 #9
Start : 16:41:54 62
Inlet :
Masses: 30 > 1100
#peaks: 468
E+06
1.20
368.5
369.5
353.5
100
80
60
40
20
340
350
360
370
380
390
400

n = 6



Result Table (Uncal - 71SW_394_F319-3271_18-JAN-2011_71)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]
2	9,150	94,777	8,604	3,2	10,7
3	10,335	2851,632	71,523	96,8	89,3
	Total	2946,410	80,128	100,0	100,0

Massenspektrometrie
Hochauflösung

Auftraggeber: Wolf

Probe: SW 384b6

Aufnahme Nr.: 69190

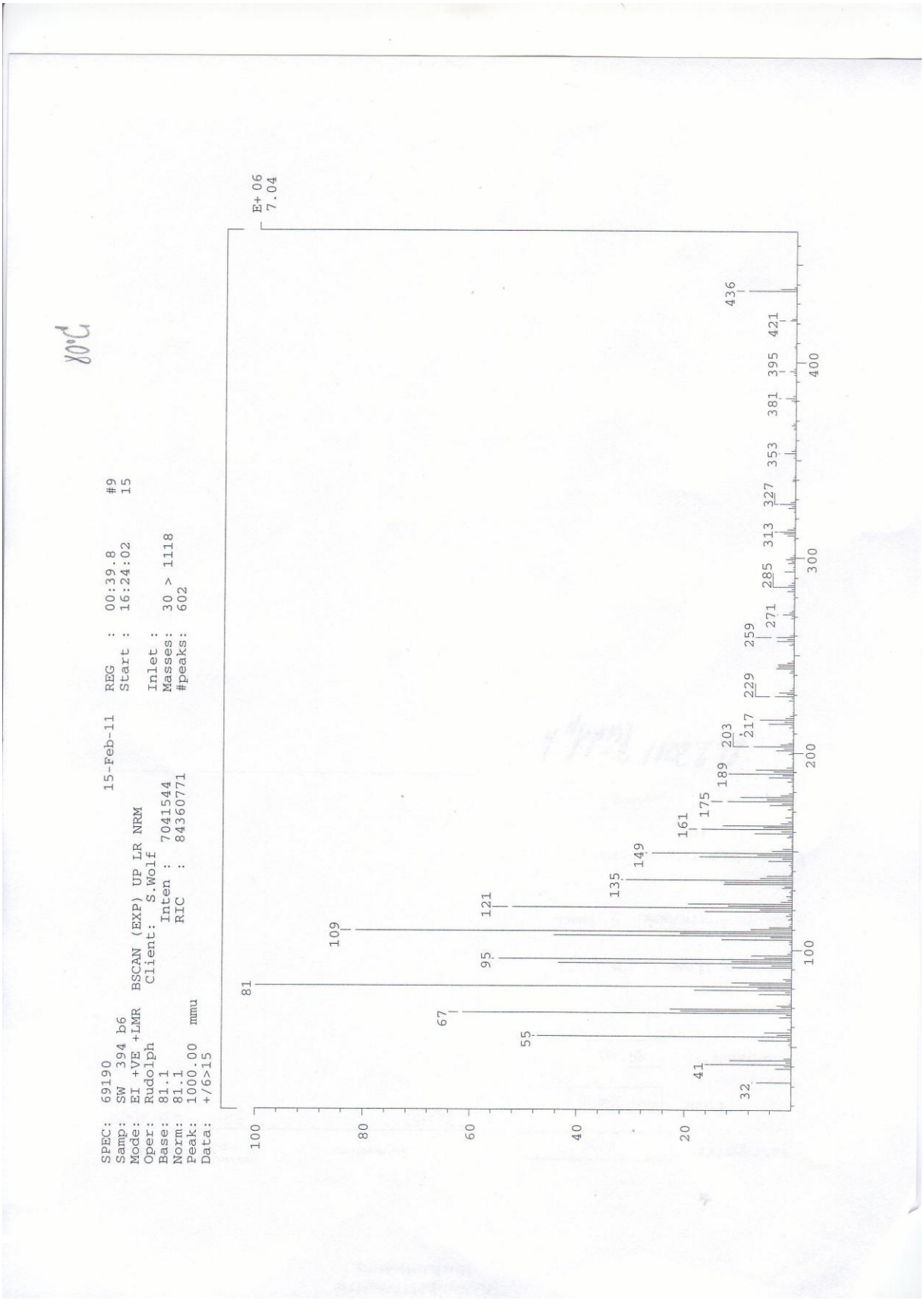
Gemessene Masse: 436.41079

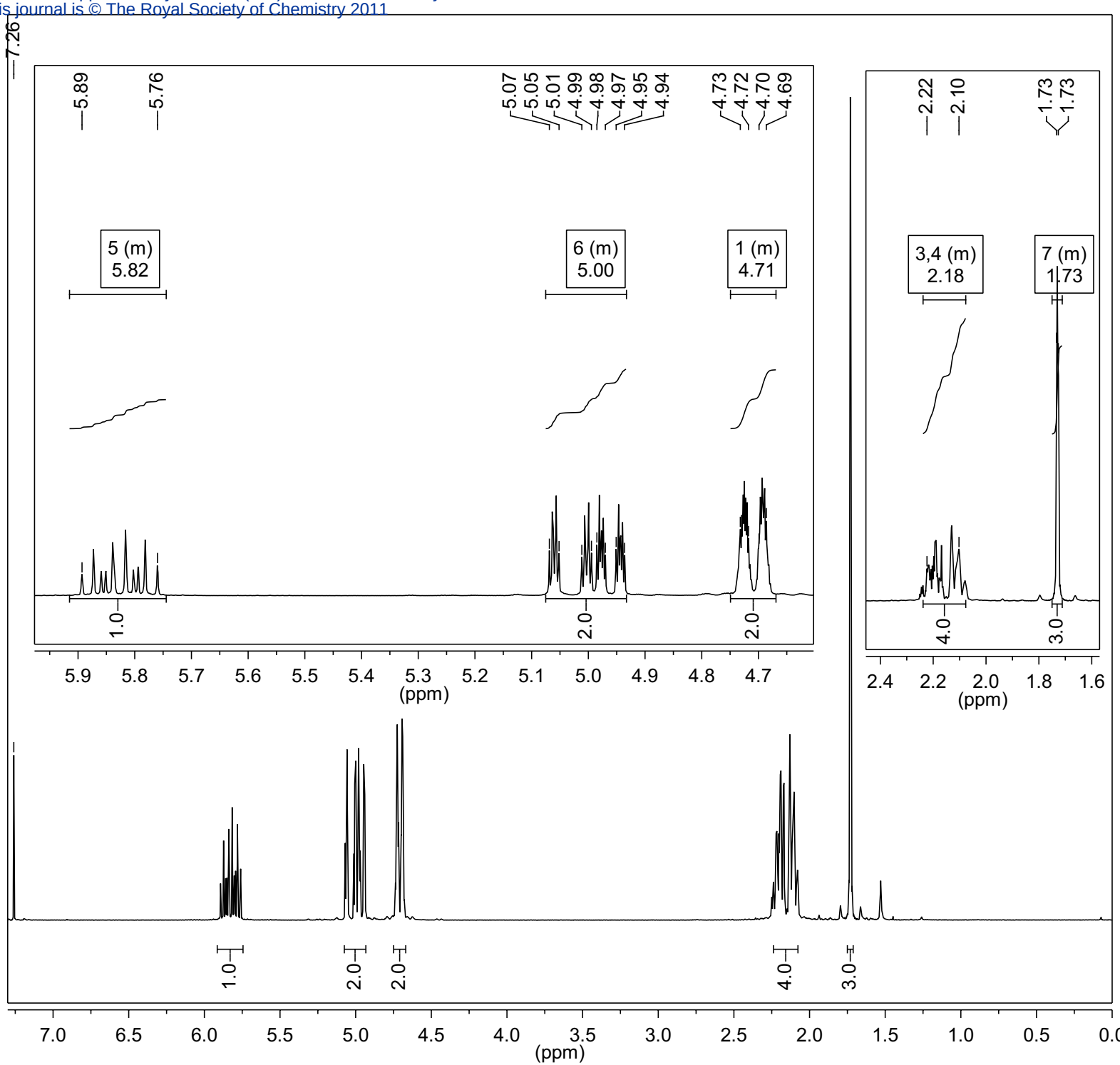
Zulässige Abweichung: 5 (mmu)

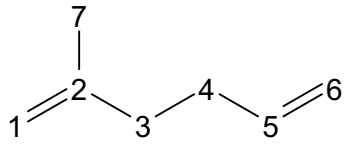
Berücksichtigte Elemente (max):
45 C.

21.2.2011 Rüdiger h

Summenformel	Abweichung (mmu)	Ber. Masse
C32 H52	3.9	436.4069

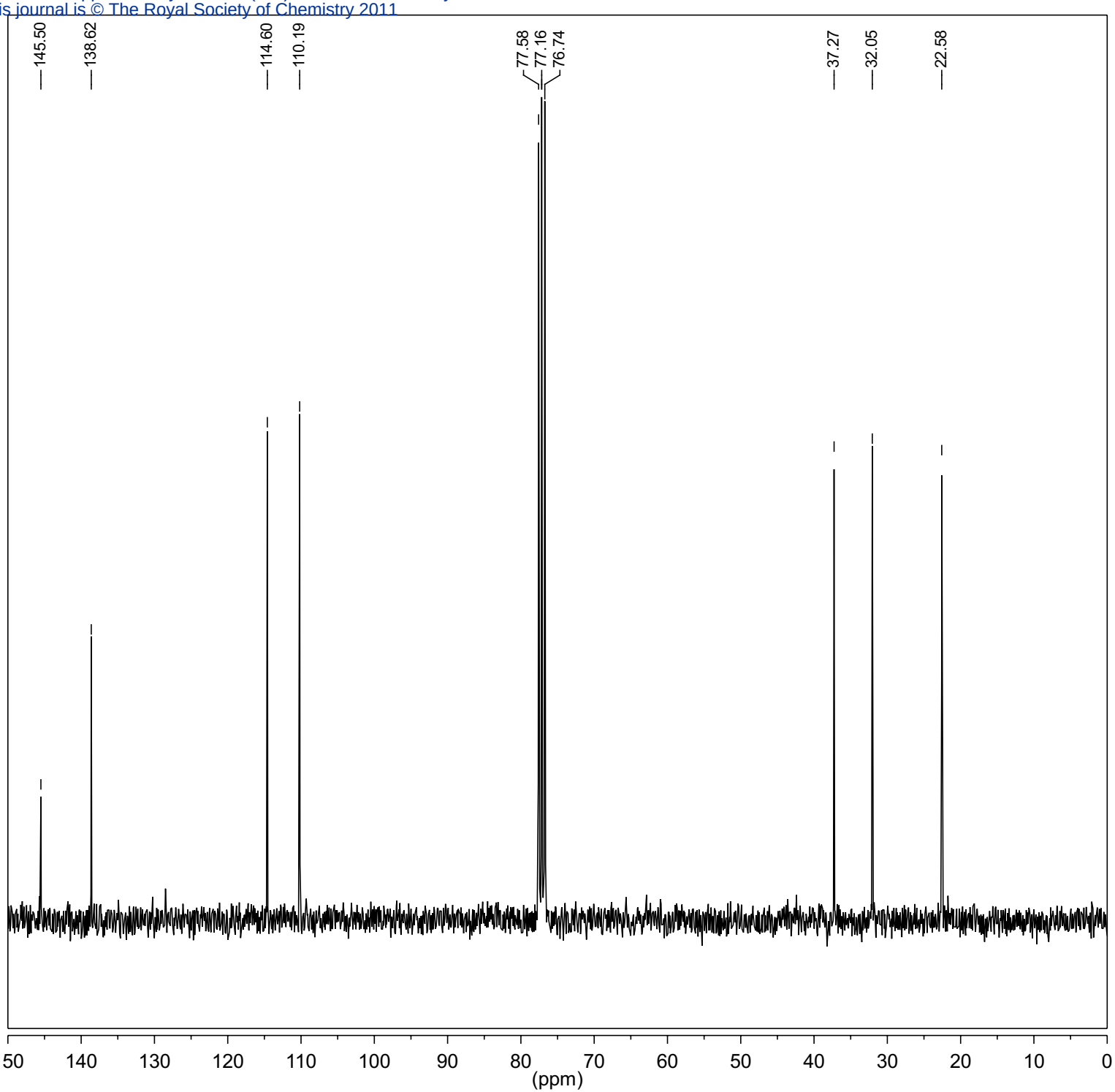

80°C
SPEC: 69190
Samp: SW 394 b6
Mode: EI +VE +LMR
Oper: Rudolph
Base: 81.1
Norm: 81.1
Peak: 1000.00 mmu
Data: +/6>15
15-Feb-11
BSCAN (EXP) UP LR NRM
Client: S.Wolf
Inten : 7041544
RIC : 84360771
REG : 00:39.8 #9
Start : 16:24:02 15
Inlet :
Masses: 30 > 1118
#peaks: 602
E+06
7.04
81
109
67
95
121
55
135
149
41
161
175
189
203
32
217
229
259
271
285
313
327
353
381
395
421
436
100
80
60
40
20
100
200
300
400

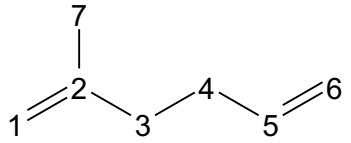




	Parameter	Value
1	Title	jun30ac
2	Comment	Wolf SW Methylhexadien
3	Origin	Bruker BioSpin GmbH
4	Solvent	CDCl3
5	Temperature	300.0
6	Pulse Sequence	zg30
7	Number of Scans	32
8	Receiver Gain	256
9	Relaxation Delay	1.0000
10	Pulse Width	9.7000
11	Acquisition Time	2.6477
12	Acquisition Date	2010-06-30T18:24:00
13	Spectrometer Frequency	299.90
14	Spectral Width	6188.1
15	Lowest Frequency	-1242.1
16	Nucleus	1H
17	Acquired Size	16384
18	Spectral Size	32768

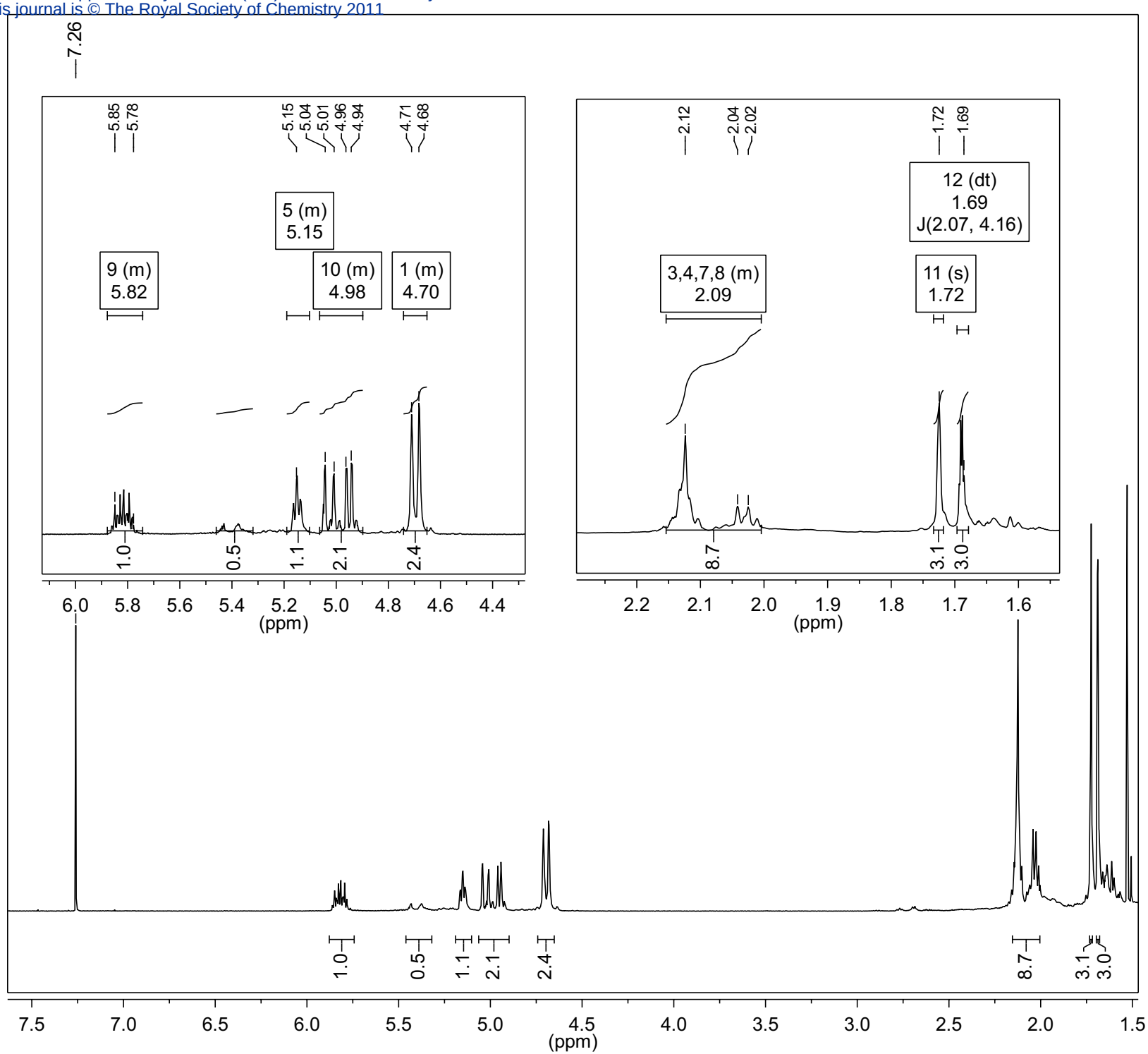
^{1}H NMR (300 MHz, $CDCl_3$) δ 5.92 – 5.74 (m, 1H), 5.08 – 4.93 (m, 2H), 4.75 – 4.67 (m, 2H), 2.24 – 2.08 (m, 4H), 1.75 – 1.71 (m, 3H).

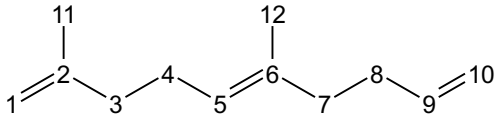




	Parameter	Value
1	Title	jun30ac
2	Comment	Wolf SW Methylhexadien
3	Origin	Bruker BioSpin GmbH
4	Solvent	CDCl3
5	Temperature	300.0
6	Pulse Sequence	zgpg30
7	Number of Scans	512
8	Receiver Gain	2050
9	Relaxation Delay	1.0000
10	Pulse Width	11.0000
11	Acquisition Time	0.7472
12	Acquisition Date	2010-06-30T18:41:00
13	Spectrometer Frequency	75.41
14	Spectral Width	21929.8
15	Lowest Frequency	-3411.0
16	Nucleus	13C
17	Acquired Size	16384
18	Spectral Size	32768

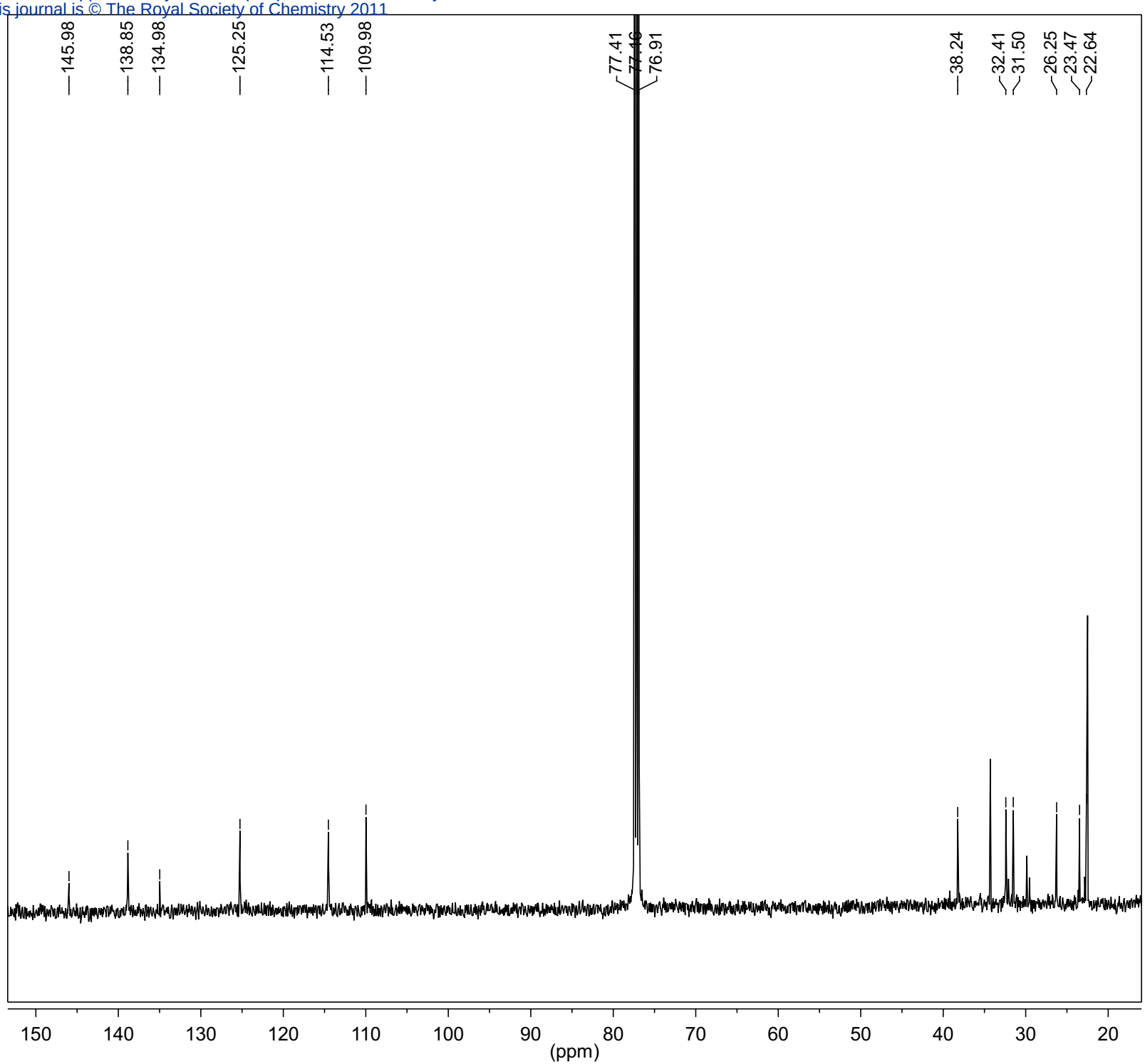
^{13}C NMR (75 MHz, $CDCl_3$) δ 145.50, 138.62, 114.60, 110.19, 37.27, 32.05, 22.58.

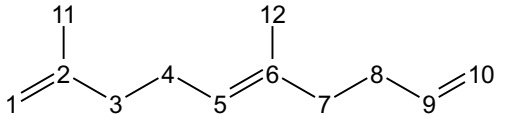




	Parameter	Value
1	Title	dez20av
2	Comment	Wolf 391-F74-82
3	Origin	Bruker BioSpin GmbH
4	Solvent	CDCl3
5	Temperature	300.0
6	Pulse Sequence	zg30
7	Number of Scans	64
8	Receiver Gain	228
9	Relaxation Delay	0.5000
10	Pulse Width	11.1000
11	Acquisition Time	3.1720
12	Acquisition Date	2010-12-21T06:56:00
13	Spectrometer Frequency	500.13
14	Spectral Width	10330.6
15	Lowest Frequency	-2088.9
16	Nucleus	1H
17	Acquired Size	32768
18	Spectral Size	65536

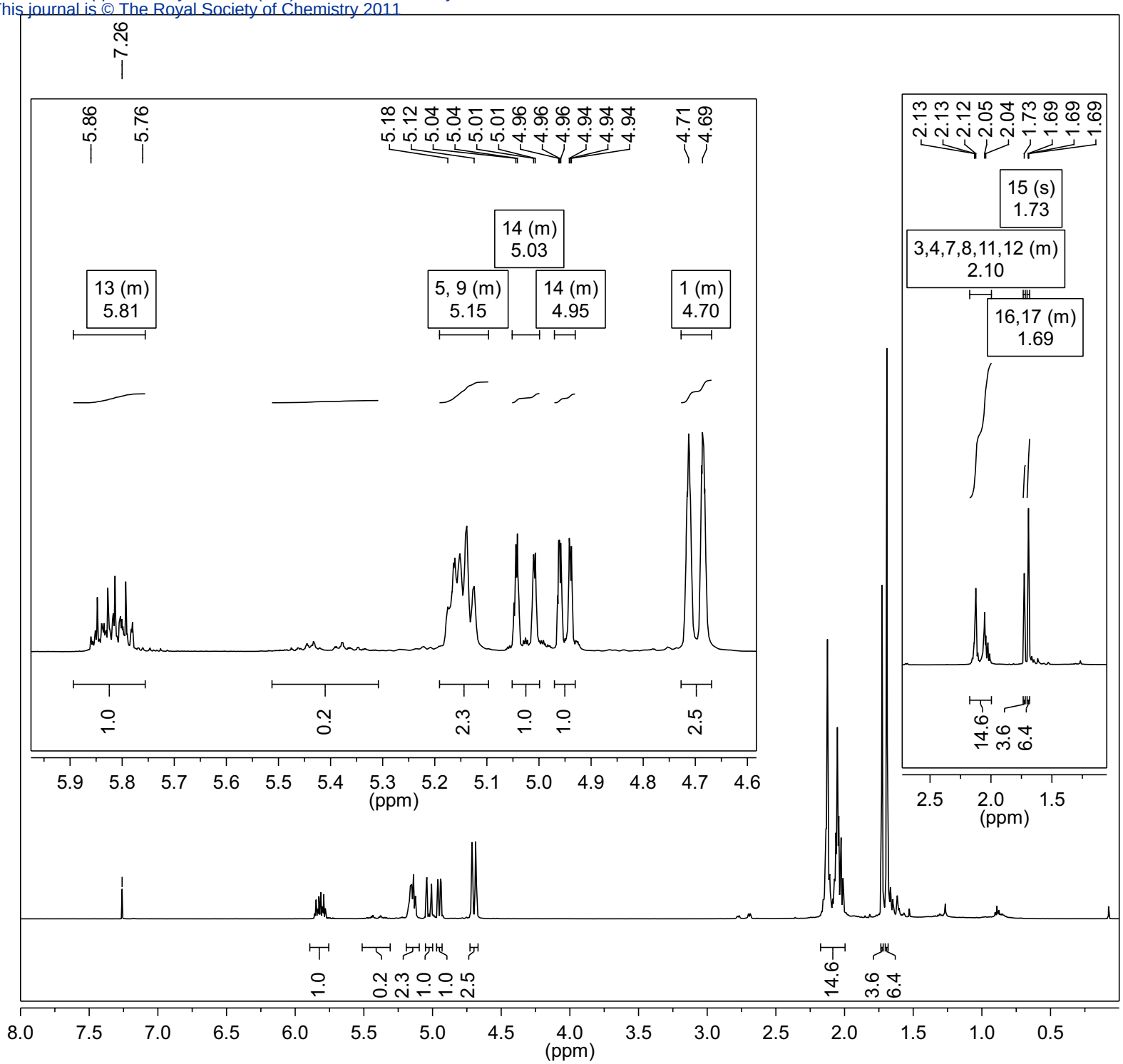
^{1}H NMR (500 MHz, $CDCl_3$) δ 5.88 – 5.74 (m, 1H), 5.19 – 5.10 (m, 1H), 5.06 – 4.90 (m, 2H), 4.74 – 4.65 (m, 2H), 2.15 – 2.00 (m, 8H), 1.72 (s, 3H), 1.69 (dt, *J* = 4.2, 2.1 Hz, 3H).

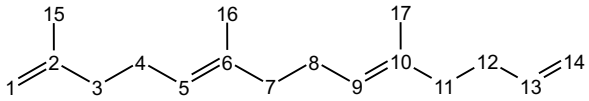




	Parameter	Value
1	Title	dez20av
2	Comment	Wolf 391-F74-82
3	Origin	Bruker BioSpin GmbH
4	Solvent	CDCl3
5	Temperature	300.0
6	Pulse Sequence	zgpg30
7	Number of Scans	2797
8	Receiver Gain	14596
9	Relaxation Delay	0.4000
10	Pulse Width	7.4000
11	Acquisition Time	0.8848
12	Acquisition Date	2010-12-21T07:59:00
13	Spectrometer Frequency	125.76
14	Spectral Width	37037.0
15	Lowest Frequency	-5923.0
16	Nucleus	13C
17	Acquired Size	32768
18	Spectral Size	65536

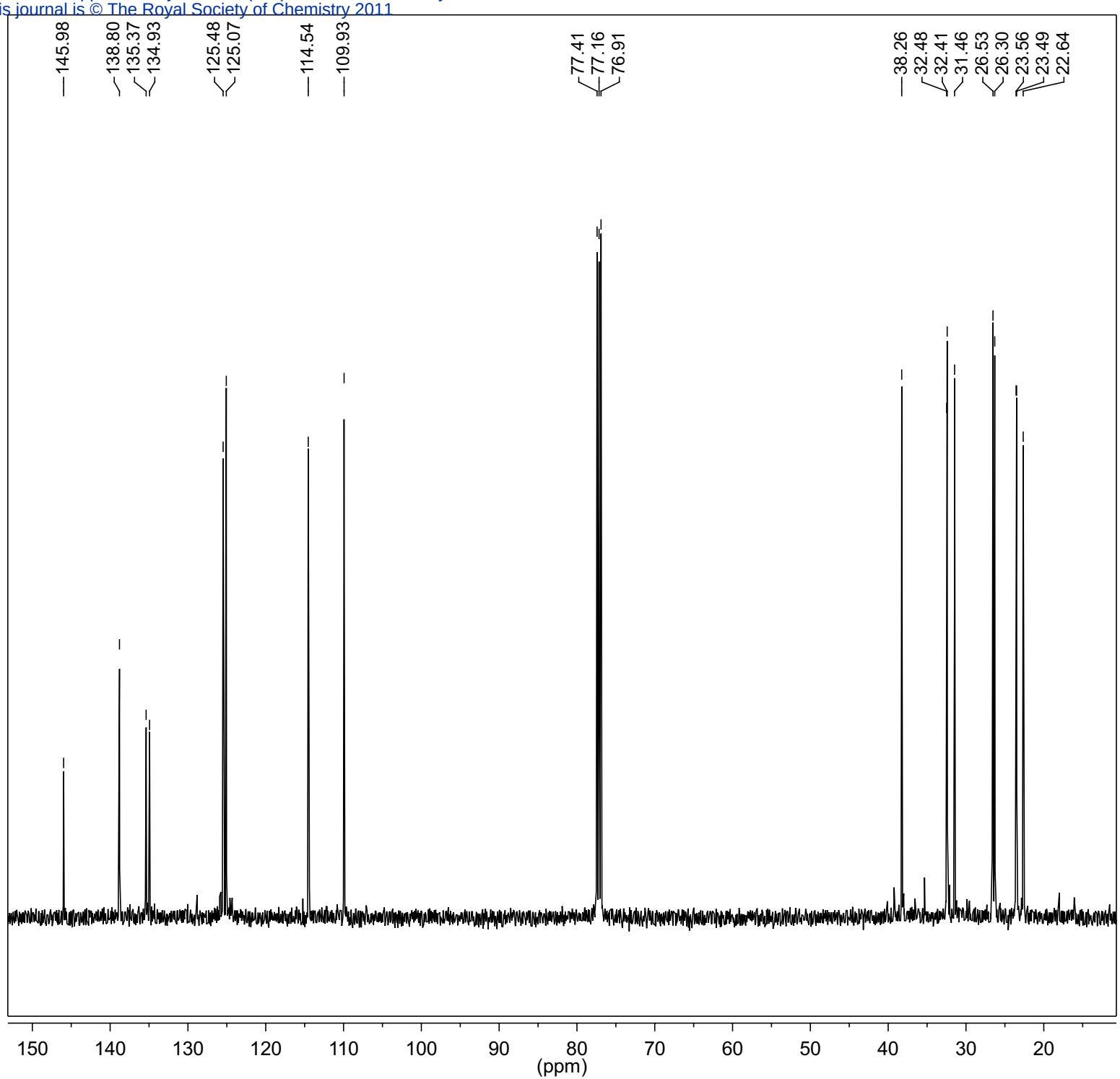
^{13}C NMR (126 MHz, $CDCl_3$) δ 145.98, 138.85, 134.98, 125.25, 114.53, 109.98, 38.24, 32.41, 31.50, 26.25, 23.47, 22.64.

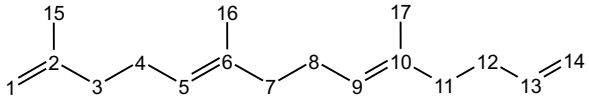




	Parameter	Value
1	Title	dez06av
2	Comment	Wolf 385-F3
3	Origin	Bruker BioSpin GmbH
4	Solvent	CDCl3
5	Temperature	300.0
6	Pulse Sequence	zg30
7	Number of Scans	32
8	Receiver Gain	72
9	Relaxation Delay	0.5000
10	Pulse Width	11.1000
11	Acquisition Time	3.1720
12	Acquisition Date	2010-12-06T13:40:00
13	Spectrometer Frequency	500.13
14	Spectral Width	10330.6
15	Lowest Frequency	-2088.8
16	Nucleus	1H
17	Acquired Size	32768
18	Spectral Size	65536

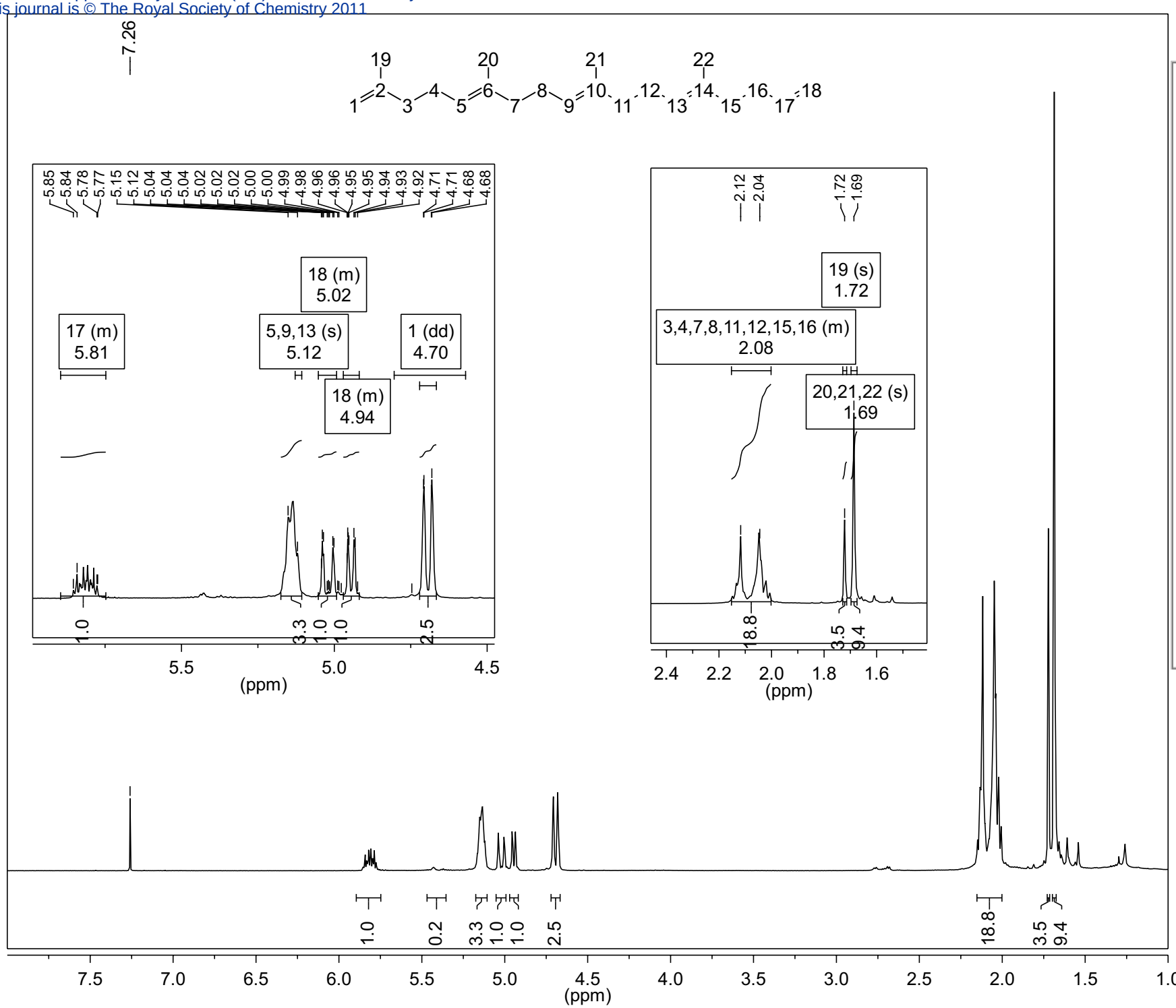
^{1}H NMR (500 MHz, $CDCl_3$) δ 5.89 – 5.76 (m, 1H), 5.19 – 5.10 (m, 2H), 5.05 – 5.00 (m, 1H), 4.97 – 4.93 (m, 1H), 4.73 – 4.67 (m, 2H), 2.17 – 2.00 (m, 12H), 1.73 (s, 3H), 1.70 – 1.68 (m, 6H).





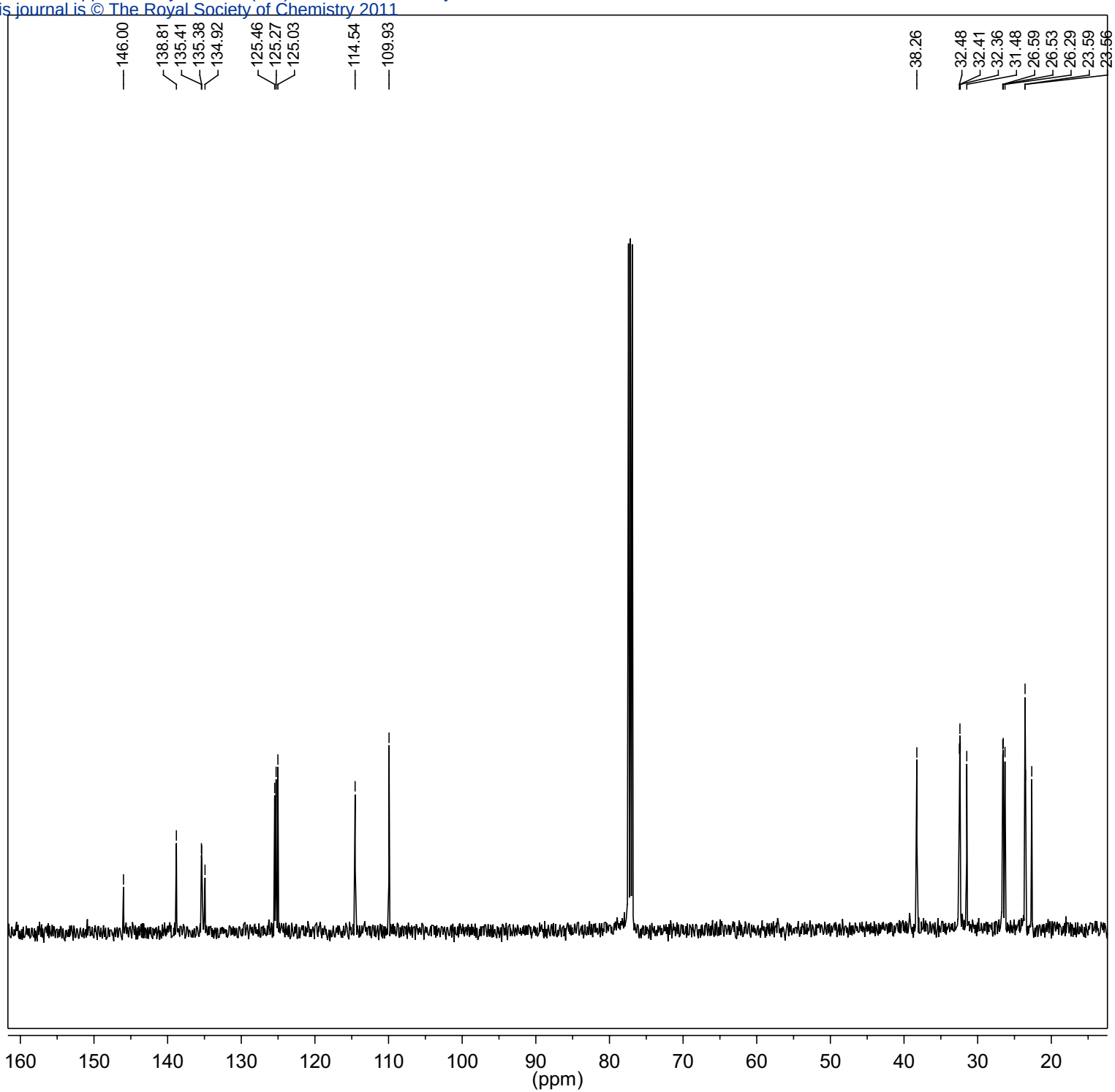
	Parameter	Value
1	Title	dez06av
2	Comment	Wolf 385-F3
3	Origin	Bruker BioSpin GmbH
4	Solvent	CDCl3
5	Temperature	300.0
6	Pulse Sequence	zgpg30
7	Number of Scans	512
8	Receiver Gain	4096
9	Relaxation Delay	0.4000
10	Pulse Width	7.4000
11	Acquisition Time	0.8848
12	Acquisition Date	2010-12-06T13:52:00
13	Spectrometer Frequency	125.76
14	Spectral Width	37037.0
15	Lowest Frequency	-5923.6
16	Nucleus	13C
17	Acquired Size	32768
18	Spectral Size	32768

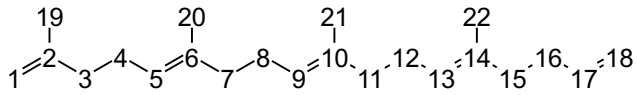
^{13}C NMR (126 MHz, $CDCl_3$) δ 145.98, 138.80, 135.37, 134.93, 125.48, 125.07, 114.54, 109.93, 77.41, 77.16, 76.91, 38.26, 32.48, 32.41, 31.46, 26.53, 26.30, 23.56, 23.49, 22.64.



	Parameter	Value
1	Title	jan24av
2	Comment	Wolf 394-b4
3	Origin	Bruker BioSpin GmbH
4	Solvent	CDCl3
5	Temperature	300.0
6	Pulse Sequence	zg30
7	Number of Scans	48
8	Receiver Gain	144
9	Relaxation Delay	0.5000
10	Pulse Width	11.1000
11	Acquisition Time	3.1720
12	Acquisition Date	2011-01-24T13:29:00
13	Spectrometer Frequency	500.13
14	Spectral Width	10330.6
15	Lowest Frequency	-2088.9
16	Nucleus	1H
17	Acquired Size	32768
18	Spectral Size	65536

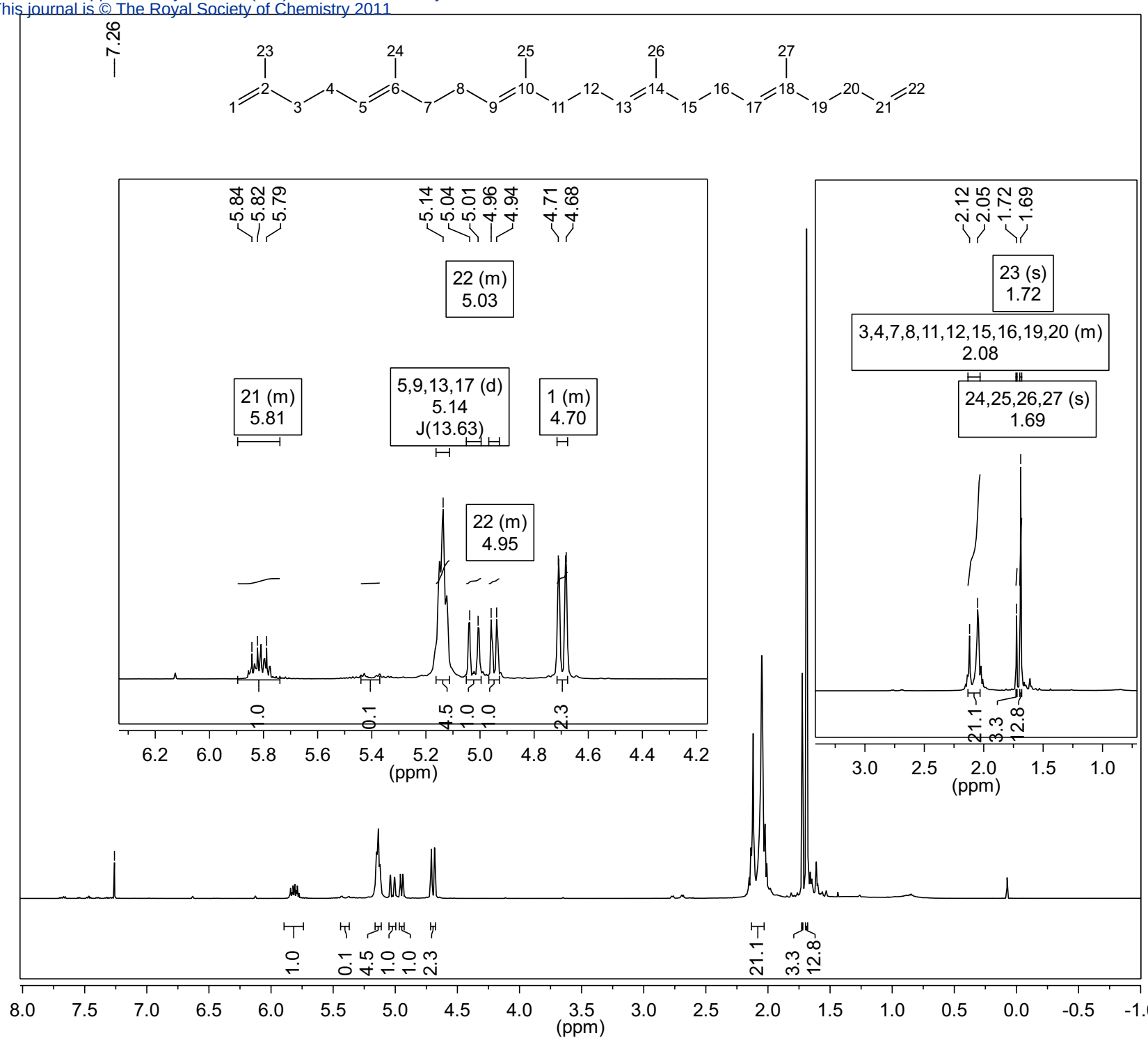
^{1}H NMR (500 MHz, $CDCl_3$) δ 5.90 – 5.75 (m, 1H), 5.17 – 5.11 (m, 3H), 5.05 – 4.99 (m, 1H), 4.97 – 4.92 (m, 1H), 4.69 (dd, *J* = 12.9, 0.7 Hz, 2H), 2.15 – 2.00 (m, 16H), 1.72 (s, 3H), 1.69 (s, 9H).





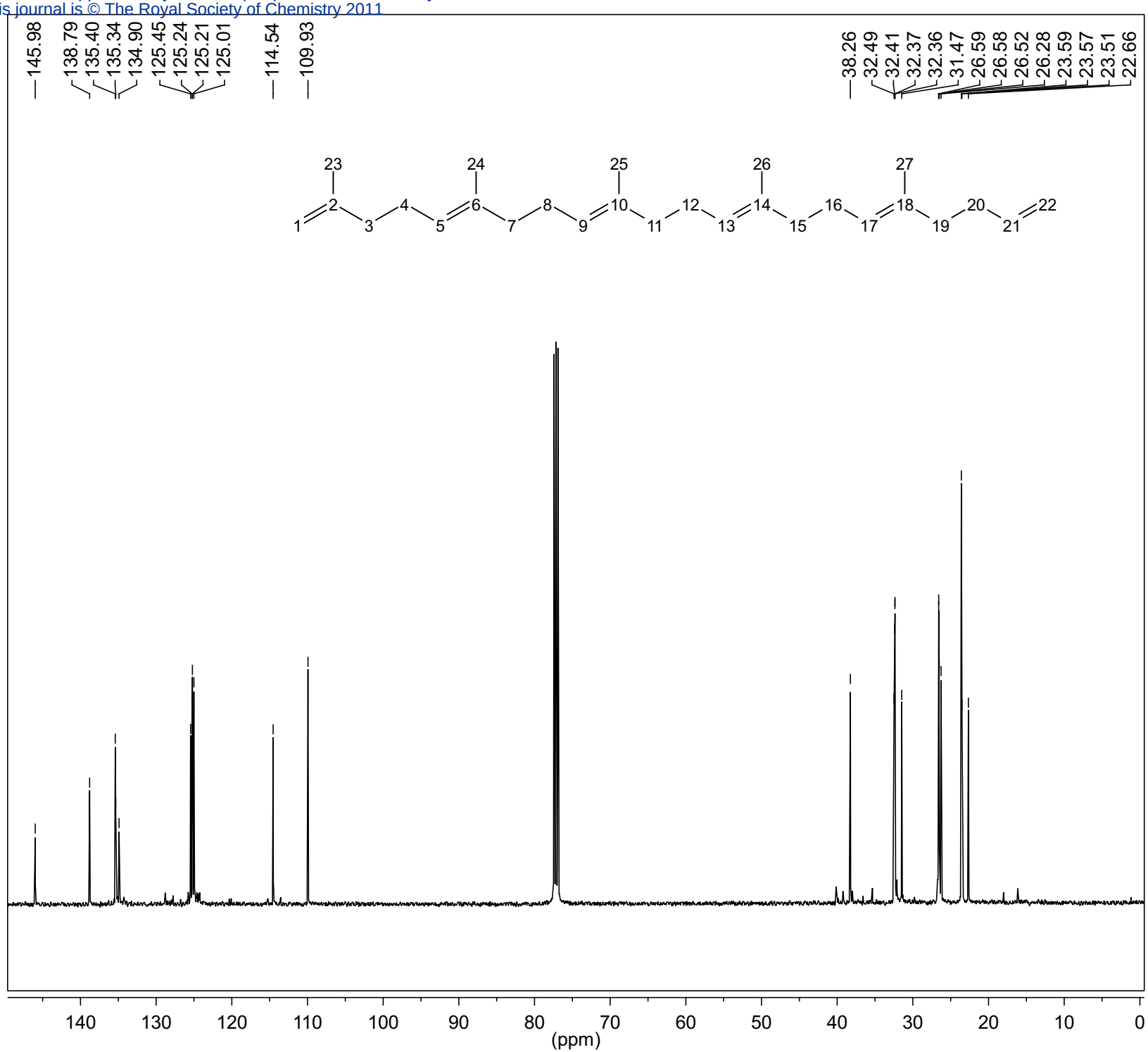
	Parameter	Value
1	Title	jan24av
2	Comment	Wolf 394-b4
3	Origin	Bruker BioSpin GmbH
4	Solvent	CDCl3
5	Temperature	300.0
6	Pulse Sequence	zgpg30
7	Number of Scans	700
8	Receiver Gain	14596
9	Relaxation Delay	0.4000
10	Pulse Width	7.4000
11	Acquisition Time	0.8848
12	Acquisition Date	2011-01-24T13:31:00
13	Spectrometer Frequency	125.76
14	Spectral Width	37037.0
15	Lowest Frequency	-5923.5
16	Nucleus	13C
17	Acquired Size	32768
18	Spectral Size	65536

^{13}C NMR (126 MHz, $CDCl_3$) δ 146.00, 138.81, 135.41, 135.38, 134.92, 125.46, 125.27, 125.03, 114.54, 109.93, 38.26, 32.48, 32.41, 32.36, 31.48, 26.59, 26.53, 26.29, 23.59, 23.56, 23.49, 22.65.



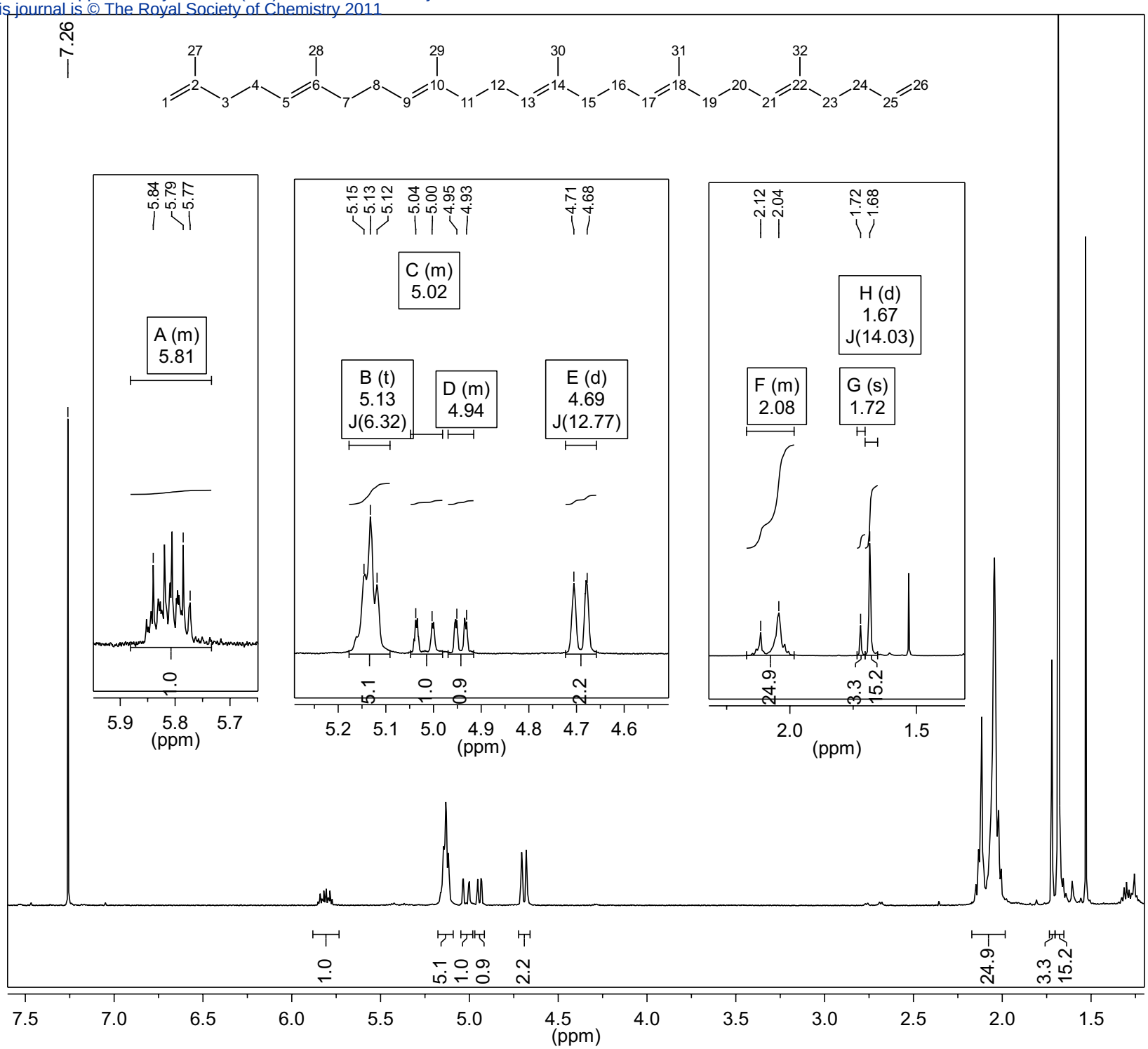
	Parameter	Value
1	Title	mai09av
2	Comment	Wolf SW-417-b5
3	Origin	Bruker BioSpin GmbH
4	Solvent	CDCl3
5	Temperature	343.6
6	Pulse Sequence	zg30
7	Number of Scans	80
8	Receiver Gain	90
9	Relaxation Delay	0.5000
10	Pulse Width	11.1000
11	Acquisition Time	3.1720
12	Acquisition Date	2011-05-09T18:24:00
13	Spectrometer Frequency	500.13
14	Spectral Width	10330.6
15	Lowest Frequency	-2088.8
16	Nucleus	1H
17	Acquired Size	32768
18	Spectral Size	65536

^{1}H NMR (500 MHz, $CDCl_3$) δ 5.90 – 5.74 (m, 1H), 5.14 (t, J = 6.8 Hz, 4H), 5.05 – 5.00 (m, 1H), 4.97 – 4.93 (m, 1H), 4.72 – 4.68 (m, 2H), 2.13 – 2.03 (m, 20H), 1.72 (s, 3H), 1.69 (s, 12H).



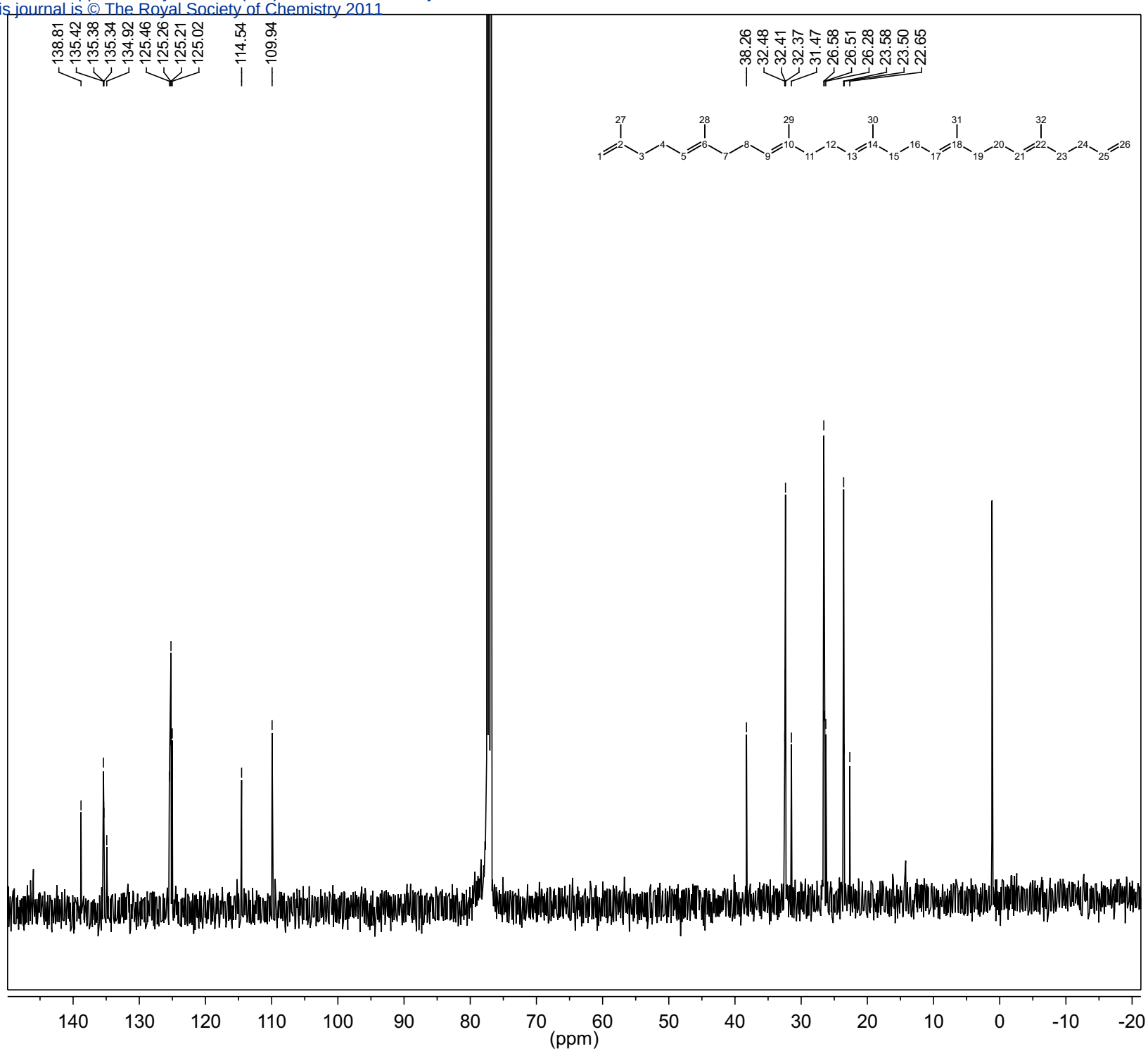
	Parameter	Value
1	Title	mai09av
2	Comment	Wolf SW-417-b5
3	Origin	Bruker BioSpin GmbH
4	Solvent	CDCl3
5	Temperature	342.2
6	Pulse Sequence	zgpg30
7	Number of Scans	5000
8	Receiver Gain	14596
9	Relaxation Delay	0.4000
10	Pulse Width	7.4000
11	Acquisition Time	0.8848
12	Acquisition Date	2011-05-09T18:29:00
13	Spectrometer Frequency	125.77
14	Spectral Width	37037.0
15	Lowest Frequency	-5923.7
16	Nucleus	13C
17	Acquired Size	32768
18	Spectral Size	65536

^{13}C NMR (126 MHz, $CDCl_3$) δ 145.98, 138.79, 135.40, 135.34, 134.90, 125.45, 125.24, 125.21, 125.01, 114.54, 109.93, 38.26, 32.49, 32.41, 32.37, 32.36, 31.47, 26.59, 26.58, 26.52, 26.28, 23.59, 23.57, 23.51, 22.66.



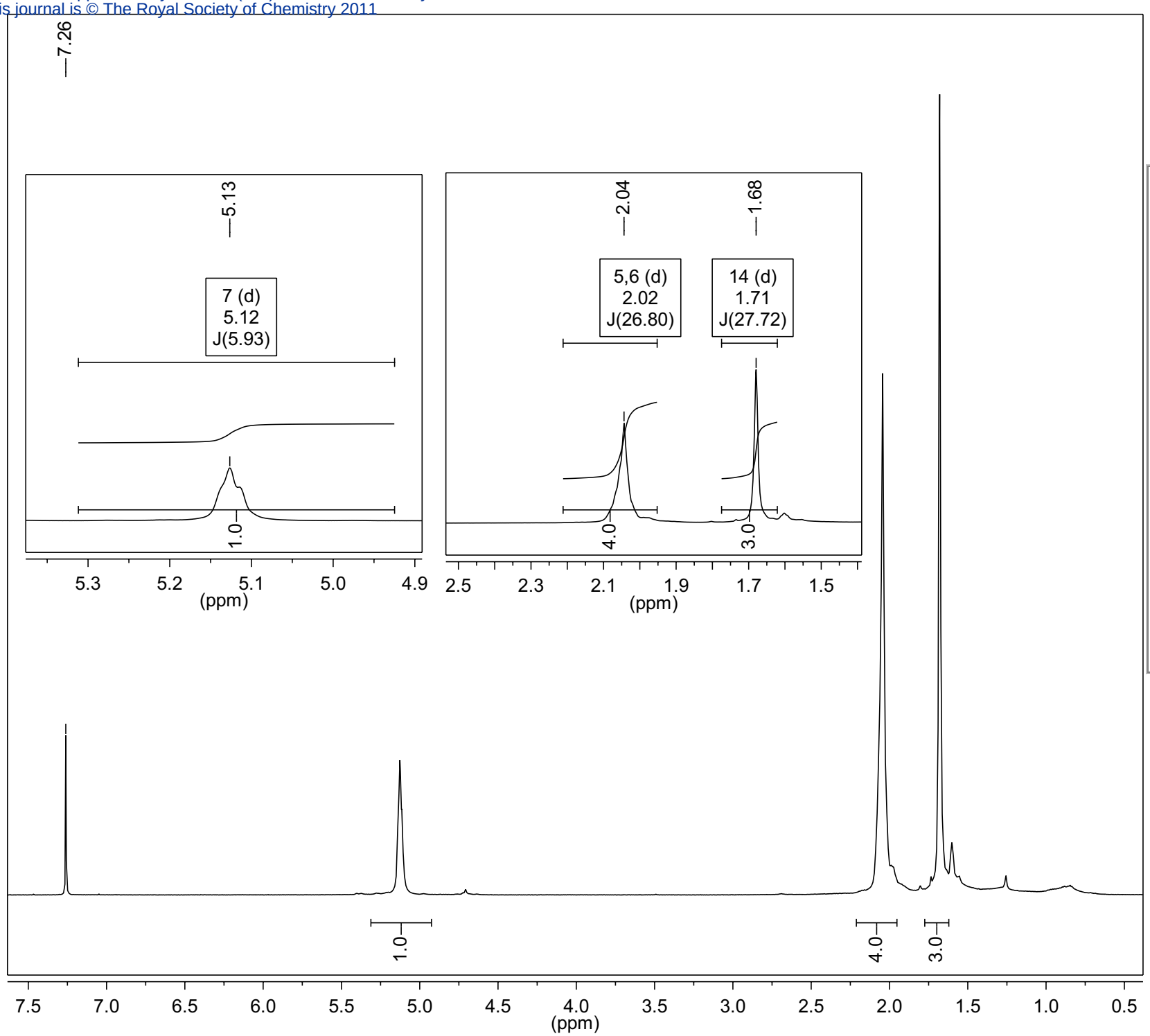
	Parameter	Value
1	Title	jan24av
2	Comment	Wolf 394-b6
3	Origin	Bruker BioSpin GmbH
4	Solvent	CDCl3
5	Temperature	300.0
6	Pulse Sequence	zg30
7	Number of Scans	64
8	Receiver Gain	406
9	Relaxation Delay	0.5000
10	Pulse Width	11.1000
11	Acquisition Time	3.1720
12	Acquisition Date	2011-01-24T17:20:00
13	Spectrometer Frequency	500.13
14	Spectral Width	10330.6
15	Lowest Frequency	-2088.8
16	Nucleus	1H
17	Acquired Size	32768
18	Spectral Size	65536

^{1}H NMR (500 MHz, $CDCl_3$) δ 5.88 – 5.73 (m, 1H), 5.13 (t, J = 6.3 Hz, 5H), 5.05 – 4.98 (m, 1H), 4.97 – 4.92 (m, 1H), 4.69 (d, J = 12.8 Hz, 2H), 2.17 – 1.98 (m, 24H), 1.72 (s, 3H), 1.67 (d, J = 14.0 Hz, 15H).



	Parameter	Value
1	Title	jan24av
2	Comment	Wolf 394-b6
3	Origin	Bruker BioSpin GmbH
4	Solvent	CDCl3
5	Temperature	300.0
6	Pulse Sequence	zgpg30
7	Number of Scans	6000
8	Receiver Gain	14596
9	Relaxation Delay	0.4000
10	Pulse Width	7.4000
11	Acquisition Time	0.8848
12	Acquisition Date	2011-01-24T19:35:00
13	Spectrometer Frequency	125.76
14	Spectral Width	37037.0
15	Lowest Frequency	-5923.4
16	Nucleus	13C
17	Acquired Size	32768
18	Spectral Size	65536

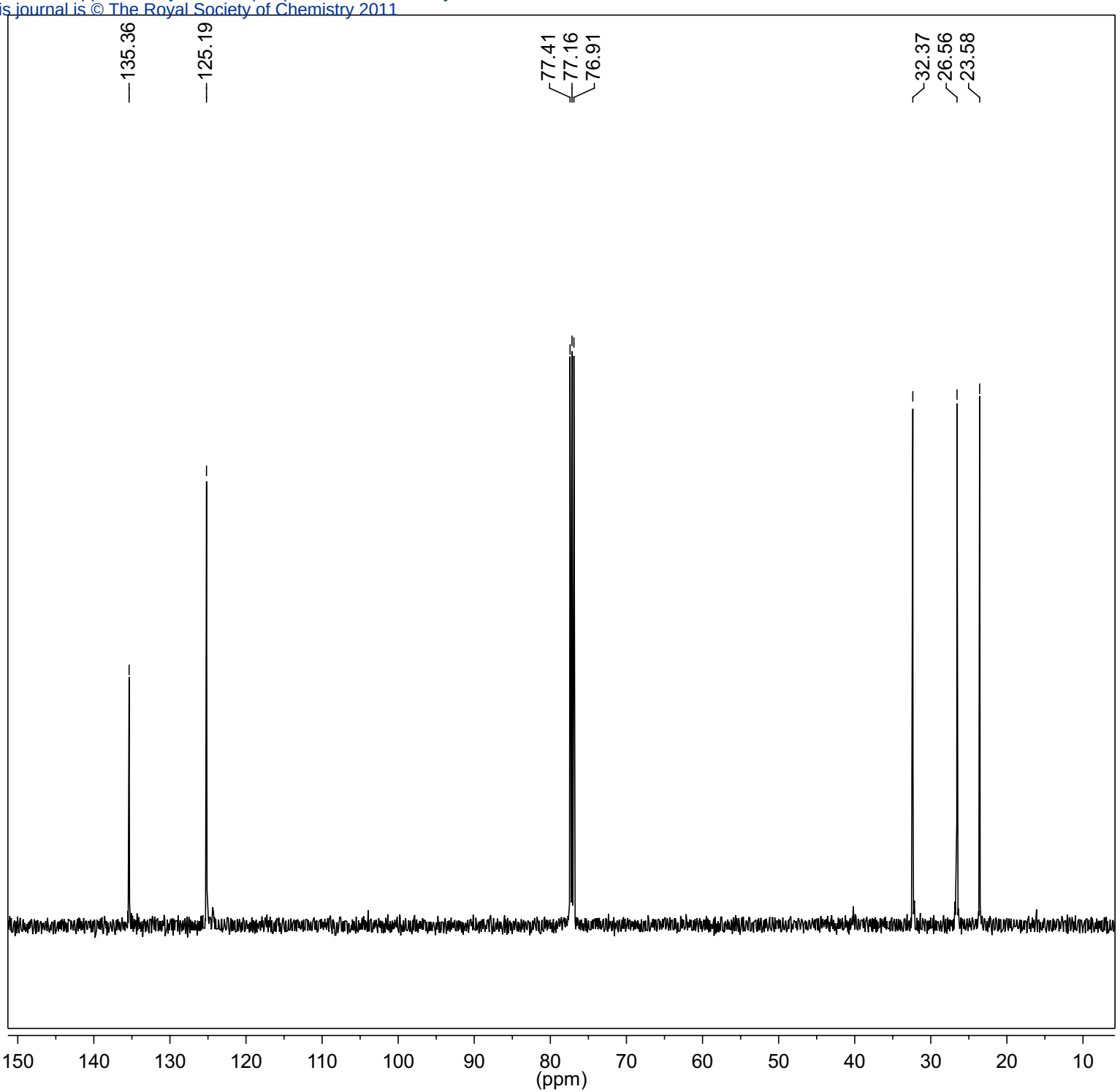
^{13}C NMR (126 MHz, $CDCl_3$) δ 138.81, 135.42, 135.38, 135.34, 134.92, 125.46, 125.26, 125.21, 125.02, 114.54, 109.94, 38.26, 32.48, 32.41, 32.37, 31.47, 26.58, 26.51, 26.28, 23.58, 23.50, 22.65.



Natural Rubber

	Parameter	Value
1	Title	jun14av
2	Comment	Wolf Polyisopren
3	Origin	Bruker Analytik GmbH
4	Solvent	CDCl3
5	Temperature	300.0
6	Pulse Sequence	zg30
7	Number of Scans	32
8	Receiver Gain	144
9	Relaxation Delay	0.5000
10	Pulse Width	11.1000
11	Acquisition Time	3.1720
12	Acquisition Date	2010-06-14T12:21:00
13	Spectrometer Frequency	500.13
14	Spectral Width	10330.6
15	Lowest Frequency	-2088.4
16	Nucleus	1H
17	Acquired Size	32768
18	Spectral Size	65536

^{1}H NMR (500 MHz, $CDCl_3$) δ 5.12 (d, J = 5.9 Hz, 1H), 2.02 (d, J = 26.8 Hz, 4H), 1.71 (d, J = 27.7 Hz, 3H).



Natural Rubber

	Parameter	Value
1	Title	jun14av
2	Comment	Wolf Polyisopren
3	Origin	Bruker Analytik GmbH
4	Solvent	CDCl3
5	Temperature	300.0
6	Pulse Sequence	zgpg30
7	Number of Scans	512
8	Receiver Gain	2896
9	Relaxation Delay	0.4000
10	Pulse Width	7.4000
11	Acquisition Time	0.8717
12	Acquisition Date	2010-06-14T12:33:00
13	Spectrometer Frequency	125.76
14	Spectral Width	37594.0
15	Lowest Frequency	-6252.2
16	Nucleus	13C
17	Acquired Size	32768
18	Spectral Size	65536

^{13}C NMR (126 MHz, $CDCl_3$) δ 135.36, 125.19, 32.37, 26.56, 23.58.