Supporting Information for

Diverse acetals from stoichiometric amounts of aldehydes and alcohols under very mild conditions: a new twist to PPh_3-CCl_4 reagent combination

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Spectral data of the synthetized acetals are presented in the following format:

-Atom-numbering scheme used for NMR assignments
-Table of NMR data (chemical shift and coupling constant data from ^{1}H and ^{13}C NMR spectra with the observed HMBC and NOESY interactions)
-Scheme with key HMBC and NOESY interactions
-Analysis of ^{1}H-^{1}H coupling constants
-^{1}H NMR spectrum and the corresponding expansions with signal assignment
-^{1}H NMR spectra obtained in a series of ^{1}H selective homodecoupling experiments with signal assignment
-^{13}C NMR spectrum and the corresponding expansions (when needed) with signal assignment
-EI-MS spectrum

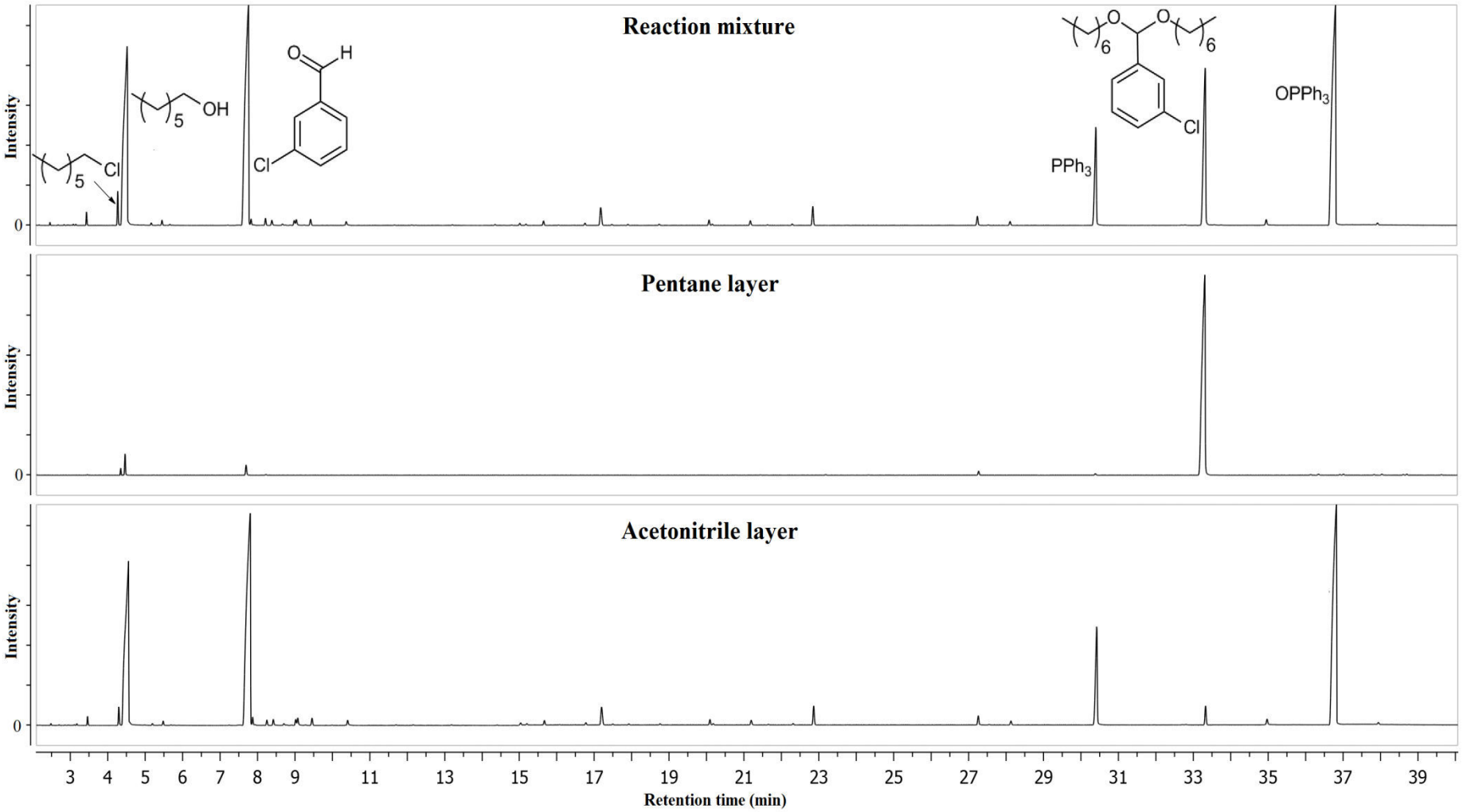


Figure S1. TIC chromatograms of the crude reaction mixture (top), and the pentane (middle) and MeCN (bottom) layers after a single pentane-MeCN partition

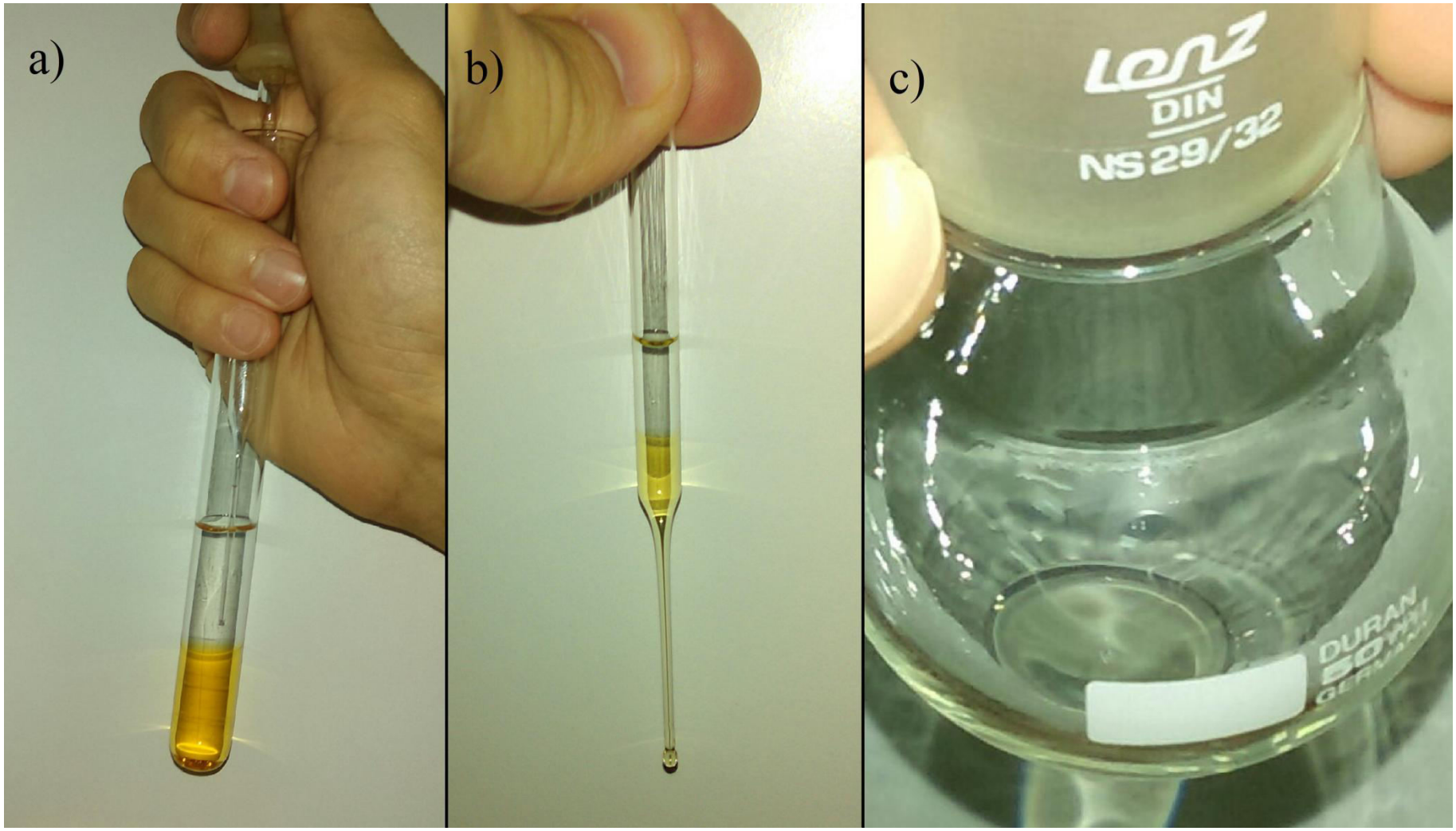


Figure S2. An efficient partition between pentane and acetonitrile at millimolar scale was effectuated in:
a) a test tube or b) a Pasteur pipette. Acetals were taken up by pentane (upper layer).
c) A reaction vessel with the product purified in this manner (after the removal of pentane). In this particular case, MeCN layer was yellow from the unreacted aldehyde (3-nitrobenzaldehyde), while the pentane layer contained acetal of this aldehyde and 1-heptanol

Experimental details regarding the measurement of NMR spectra and GC-MS analyses

The ^{1}H and ^{13}C NMR spectra were recorded on a Bruker Avance III 400 MHz NMR spectrometer (Fällanden, Switzerland; ^{1}H at 400 MHz, ^{13}C at 100.6 MHz), equipped with a 5-mm dual $^{13}C/^{1}H$ probe head. All NMR spectra were recorded at 20 °C in deuterated chloroform with TMS as internal standard. Chemical shifts (δ) are reported in parts per million and referenced to TMS (δ_H = 0.00 ppm) in ^{1}H and to the (residual) solvent signal in ^{13}C NMR and heteronuclear 2D spectra (residual $CHCl_3$ δ_H = 7.26 ppm and $^{13}CDCl_3$ δ_C = 77.16 ppm). Scalar couplings are reported in Hertz (Hz). Samples (*ca.* 20–30 mg of the acetals) were dissolved in 1 mL of deuterated chloroform, and 0.7 mL of the solution transferred into a 5 mm Wilmad, 528-TR-7 NMR tube. Details regarding the measurement of NMR spectra are provided in the Supplementary Information. The ^{1}H NMR spectra were recorded with 16 scans, 1 s relaxation delay, 4 s acquisition time, 0.125 Hz digital FID resolution, 51 280 FID size, with 6410 Hz spectral width, and an overall data point resolution of 0.0003 ppm. The ^{13}C NMR spectra were recorded with Waltz 161H broadband decoupling, 6400-12000 scans, 0.5 s relaxation delay, 1 s acquisition time, 0.5 Hz digital FID resolution, 65536 FID size, 31850 Hz spectral width, and an overall data point resolution of 0.005 ppm. Standard pulse sequences were used for 2D spectra. $^{1}H–^{1}H$ gDQCOSY and NOESY spectra were recorded at spectral widths of 5 kHz in both F2 and F1 domains; 1 K x 512 data points were acquired with 32 scans per increment and the relaxation delays of 2.0 s. The mixing time in NOESY experiments was 1 s. Data processing was performed on a 1 K x 1 K data matrix. Inverse detected 2D heteronuclear correlated spectra were measured over 512 complex points in F2 and 256 increments in F1, collecting 128 (gHMQC) or 256 ($^{1}H–^{13}C$ gHMBC) scans per increment with a relaxation delay of 1.0 s. The spectral widths were 5 and 27 kHz in F2 and F1 dimensions, respectively. The gHMQC experiments were optimized for C–H couplings of 165 Hz; the $^{1}H–^{13}C$ gHMBC experiments were optimized for long-range C–H couplings of 10 Hz. Fourier transforms were performed on a 512 x 512 data matrix. π/2 shifted sine-squared window functions were used along F1 and F2 axes for all 2D spectra.

In general, acetals are sensitive to a combination of an acid and a nucleophile. This usually represents an obstacle during their isolation and purification. The employment of standard SiO_2 columns is not only impaired by the duration of this step but also can result in their decomposition/hydrolysis. Additionally, traces of silica gel, which can be transferred to the “pure” sample even after careful filtration, can also catalyze the hydrolysis of acetals by ambient moisture during a longer period of time. Also any aqueous workup of acetal-containing reaction mixtures can be detrimental to the yield. During our initial measurements of NMR data of the obtained acetals in commercial $CDCl_3$, we noticed that the amount of the acetal declined, while the aldehyde amount increased, up to a certain level. We figured that there were traces of acid in the used deuterated chloroform and that the (small amount of) moisture present in the NMR tubes was sufficient to hydrolyze the acetals. The hydrolysis ceased when all of the water was used up. This situation could be dealt with in two ways: either to meticulously dry all of the glassware used or to remove the catalyst. A simple filtration of $CDCl_3$ through a small layer of anhydrous K_2CO_3 directly to the sample vial resolved this issue (the acetals were stable in $CDCl_3$ solution for at least 3-4 days).

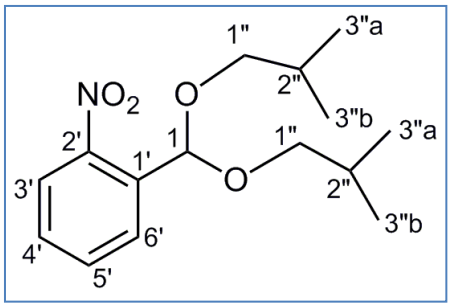
Gas chromatography/mass spectrometry (GC-MS) analyses were repeated three times for each sample using an HP 6890N gas chromatograph coupled with an HP 5975B mass-selective detector (Hewlett-Packard, Palo Alto, CA, USA). The gas chromatograph was equipped with a DB-5MS fused silica capillary column (5% phenylmethylsiloxane, 30 m × 0.25 mm, film thickness 0.25 µm; Agilent Technologies, Palo Alto, CA, USA). The oven temperature was raised linearly from 70 to 315 °C at a heating rate of 5 °C min^{-1} and then held isothermally for 10 min. Helium at a flow rate of 1 mL min^{-1} was used as carrier gas. The injector and interface were maintained at 250 and 320 °C respectively. The samples, 1 µL of the solutions of reaction mixtures or pure compounds in diethyl ether (*ca*. 1 mg in 1 mL of Et_2O), were injected in a pulsed split mode (the flow rate was 1.5 mL min^{-1} for the first 0.5 min and then set to 1 mL min^{-1} for the remainder of the analysis; split ratio 40:1). The mass-selective detector was operated at an ionization energy of 70 eV in the *m/z* 35–750 range with a scanning speed of 0.34 s/scan.

General remarks on the NMR spectra of the prepared acetals

Copies of ^{1}H- and ^{13}C- NMR and EI-MS spectra of all new acetals can be found in this very file. In addition, below, there are tables with the assigned NMR chemical shifts of ^{1}H and ^{13}C and an interpretation of the observed couplings in ^{1}H NMR spectra. HMBC and NOESY interactions are also summarized in tables, and the key ones used during the assignation are presented on appropriate schemes.

The signals in experimental data are listed as observed in the NMR spectra and the true splitting pattern is given in the mentioned tables in this supplementary data file. Values of coupling constants were precisely determined from a series of ^{1}H selective homodecoupled spectra. Coupling constant values lower than 0.5 Hz were usually observed as a broadening of the corresponding ^{1}H NMR signals.

All aromatic acetals (i.e. those derived from benzaldehydes) displayed long-range couplings of the acetal hydrogen and *ortho*- (0.4 - 0.6 Hz) and *para*- (≈ 0.3 - 0.4 Hz) hydrogens from the aromatic moiety, which were disclosed only in a series of selective ^{1}H homodecoupling experiments. Although, the *meta*-hydrogens also imparted partially to the broadening of the acetal hydrogen signal, the ^{5}J constant was lower in value and amounted in average to 0.2 Hz. The difference in chemical shifts of the diastereotopic -OCH_2- hydrogens, characteristic for achiral acetals, was dependent of the nature of the starting aldehyde. A more pronounced difference was noted in the case of aliphatic aldehydes, where it invariantly amounted to *ca.* 0.16 ppm, while the Δδ for the aromatic ones was much more spread out, Δδ = 0.03-0.11 ppm. It appears that the *ortho* regioisomer, probably due to steric reasons, shows a larger Δδ, especially in the case of the strongly anisotropic NO_2 group. Geminal coupling constant between these diastereotopic protons was proved to be $^{2}J_{1''a,1''b}$ = 8.9 – 9.5 Hz. The vicinal coupling constants for –OCH_2-CH- was found to be in the range 5.8 to 6.9 Hz, suggesting a relatively free rotation around this bond.



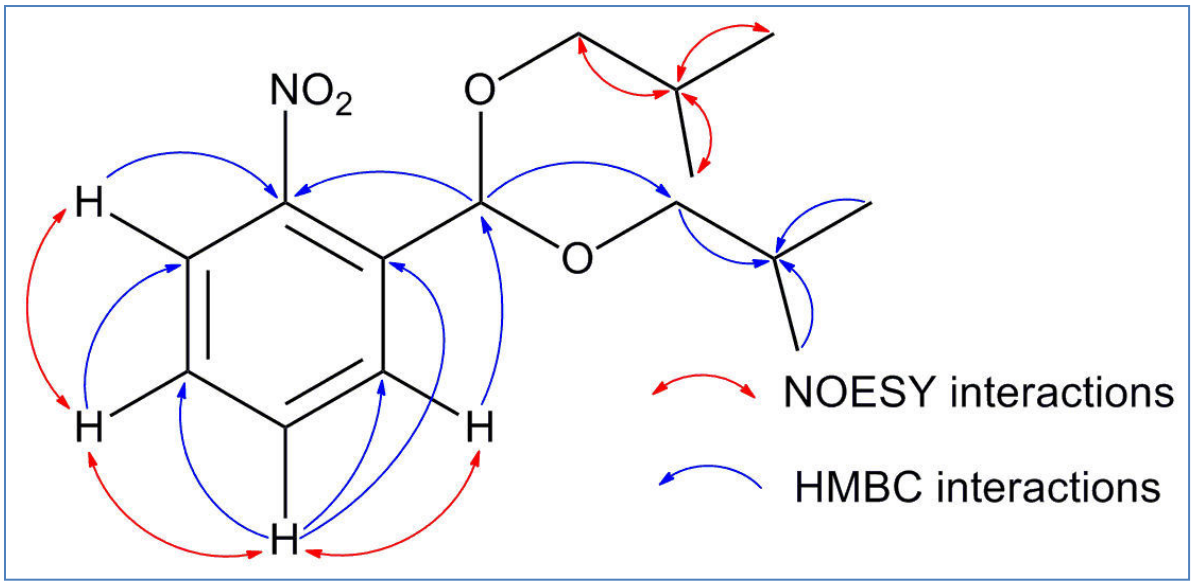
1-(diisobutoxymethyl)-2-nitrobenzene

Table of NMR data of 1-(diisobutoxymethyl)-2-nitrobenzene (chemical shift and coupling constant data from ^{1}H and ^{13}C NMR spectra with the observed HMBC and NOESY interactions)

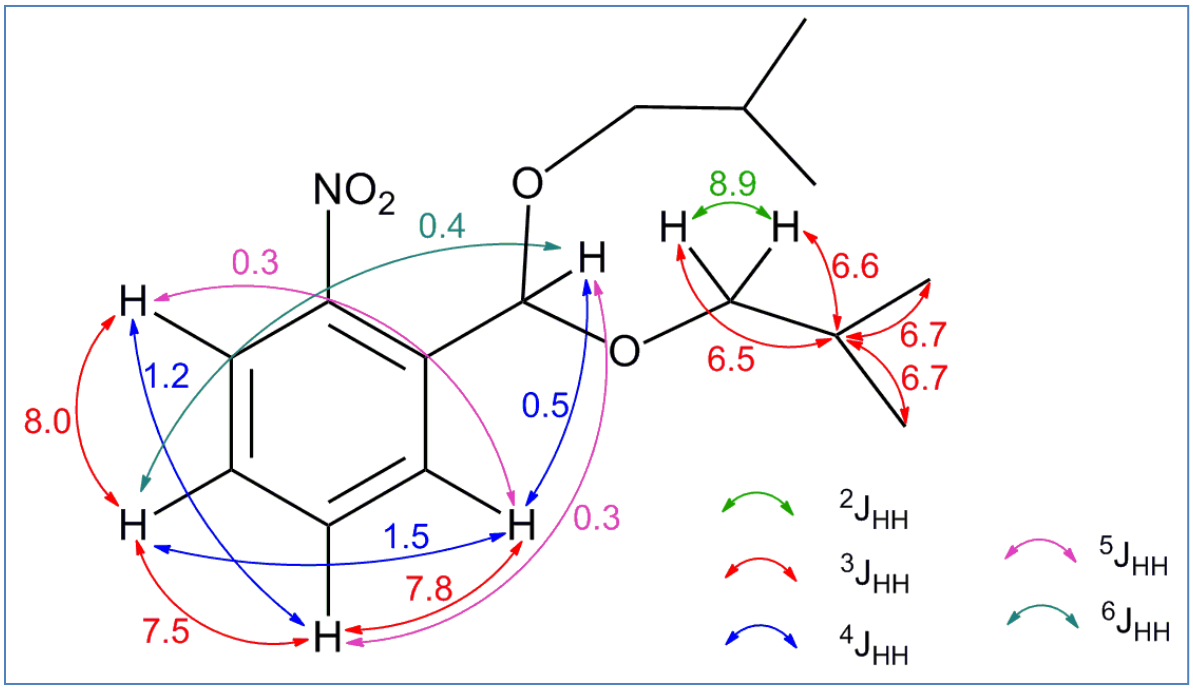
Position	δ_H (Integral, m, *J* (Hz))	δ_C (ppm) C-13 {H}	HMBC[c]	NOESY
1	6.04 (1 H, ddd, $^{4}J_{1,6'}$ = 0.5, $^{6}J_{1,4'}$ = 0.4, $^{5}J_{1,5'}$ = 0.3)[b]	98.5 (1 C)	2', 6', 1"	/
1'	/	133.8 (1 C)	/	/
2'	/	149.1 (1 C)	/	/
3'	7.80 (1 H, ddd, $^{3}J_{3',4'}$ = 8.0, $^{4}J_{3',5'}$ = 1.2, $J_{3',6'}$ = 0.3)[b]	124.3 (1 C)	5', 1'	4'
4'	7.45 (1 H, dddd, $^{3}J_{3',4'}$ = 8.0, $^{3}J_{4',5'}$ = 7.5, $^{4}J_{4',6'}$ = 1.5, $^{4}J_{1,4'}$ = 0.4)[b]	129.2 (1 C)	2', 3', 5', 6'	3', 5'
5'	7.59, (1 H, dddd, $^{3}J_{5',6'}$ = 7.8, $^{3}J_{4',5'}$ = 7.5, $^{4}J_{3',5'}$ = 1.2, $^{5}J_{1,5'}$ = 0.3)[b]	132.5 (1 C)	1', 3', 4', 6'	4', 6'
6'	7.84 (1 H, dddd, $^{3}J_{5',6'}$ = 7.8, $^{4}J_{4',6'}$ = 1.5, $^{4}J_{1,6'}$ = 0.5, $^{5}J_{3',6'}$ = 0.3)[b]	128.3 (1 C)	1, 2', 4'	5'
1"a	3.29 (2 H, dd, $^{2}J_{1"a,1"b}$ = 8.9, $^{3}J_{1"a,2"}$ = 6.6)	74.4 (2 C)	1, 2", 3"a, 3"b	2"
1"b	3.38 (2 H, dd, $^{2}J_{1"a,1"b}$ = 8.9, $^{3}J_{1"b,2"}$ = 6.5)			
2"	1.87 (2 H, septuplet of dd, $^{3}J_{2",3"}$ = 6.7, $^{3}J_{1"a,2"}$ = 6.6, $^{3}J_{1"b,2"}$ = 6.5)	28.7 (2 C)	1", 3"a, 3"b	1", 3"a, 3"b
3"a	0.92 (6 H, d, $^{3}J_{2",3a"}$ = 6.7)	19.6 (4 C)	1", 2", 3"b	2"
3"b	0.92 (6 H, d, $^{3}J_{2",3b"}$ = 6.7)		1", 2", 3"a	2"

[a]Correlation between the hydrogen in this row and the carbon in the listed position.

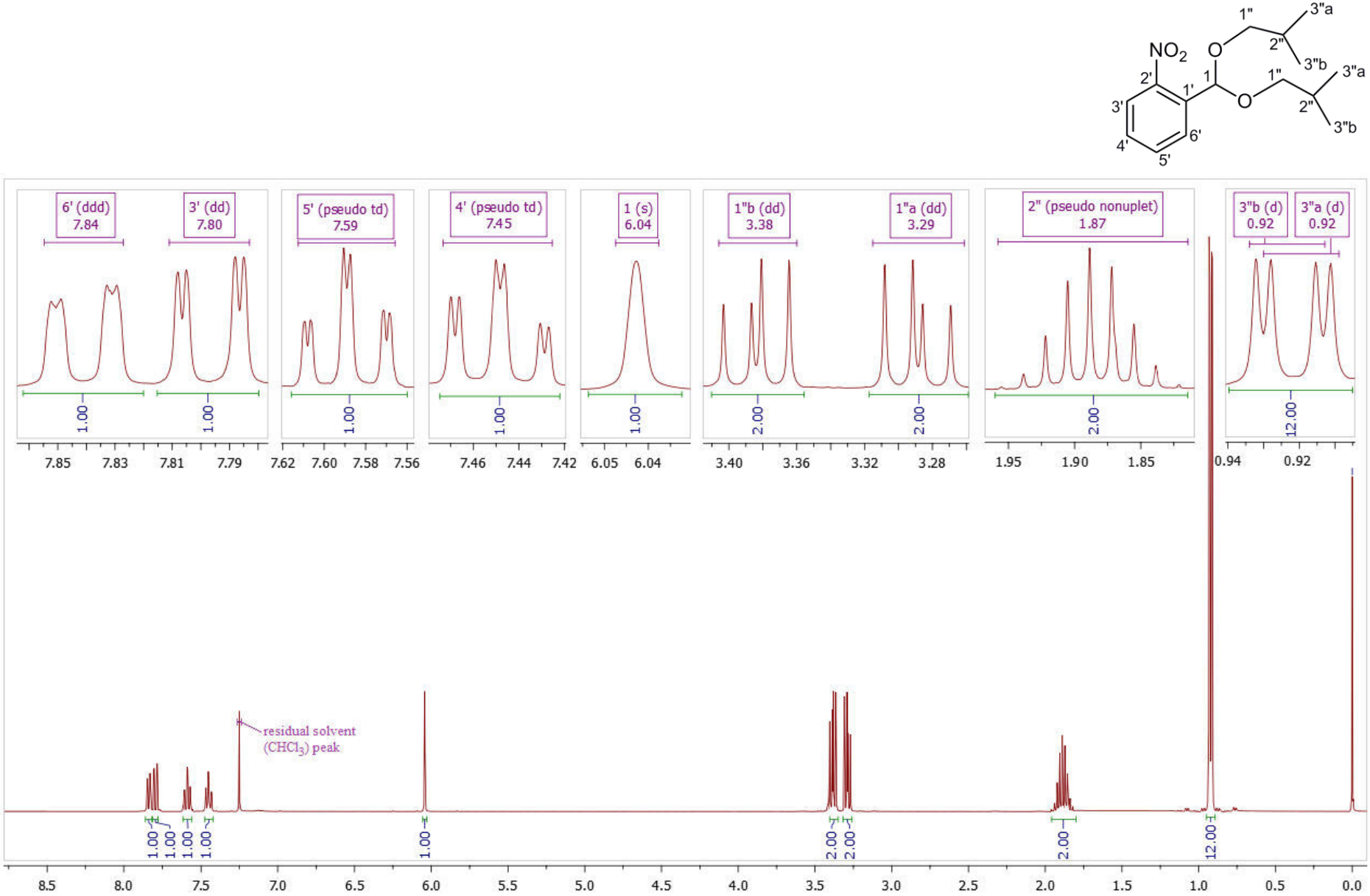
[b]Coupling constants (and multiplicity) were determined from appropriate ^{1}H selective homodecoupled spectra. Values of coupling constants lower than 0.5 Hz were usually observed as a broadening of the corresponding ^{1}H NMR signals; they were disclosed only in a series of selective homodecoupling experiments.



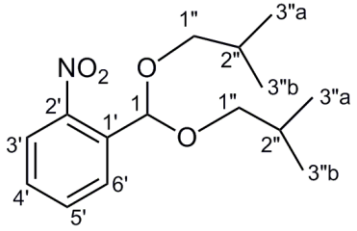
Scheme with key HMBC and NOESY interactions

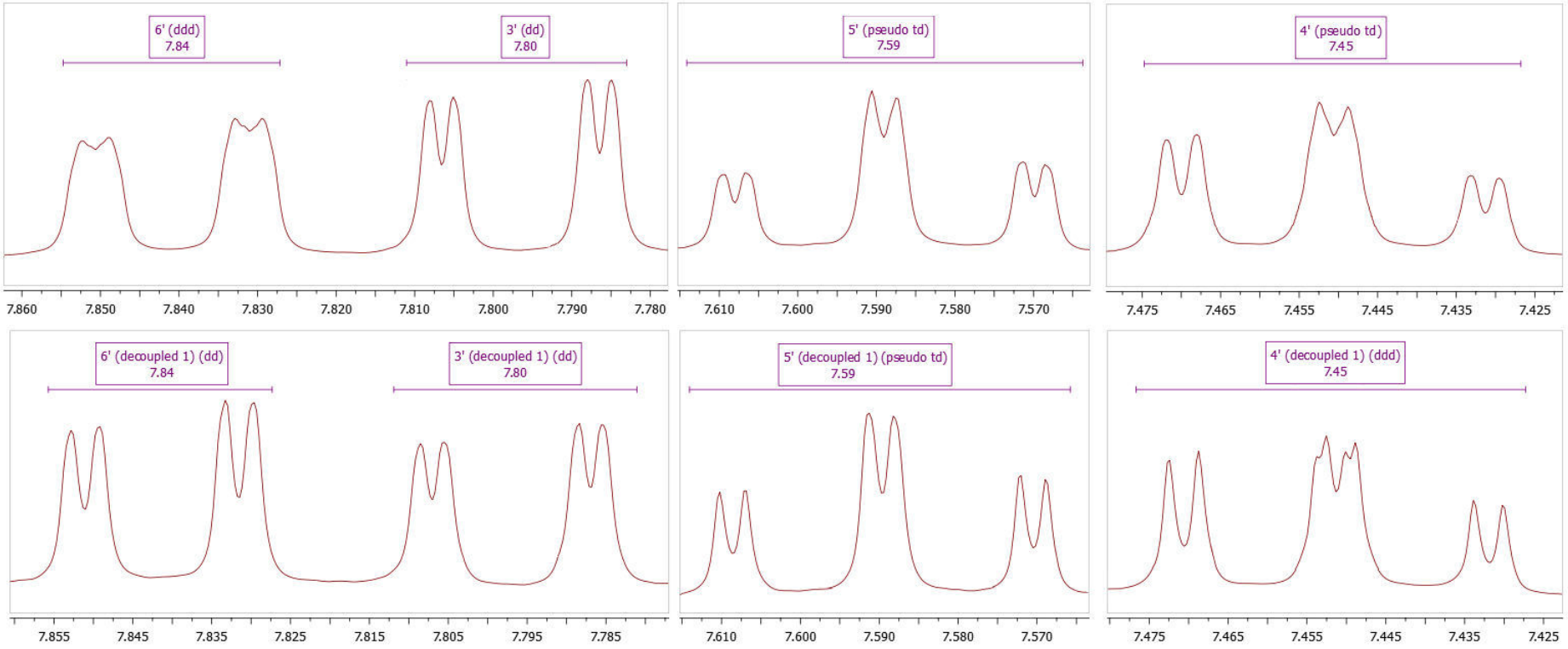


Analysis of ^{1}H-^{1}H coupling constants

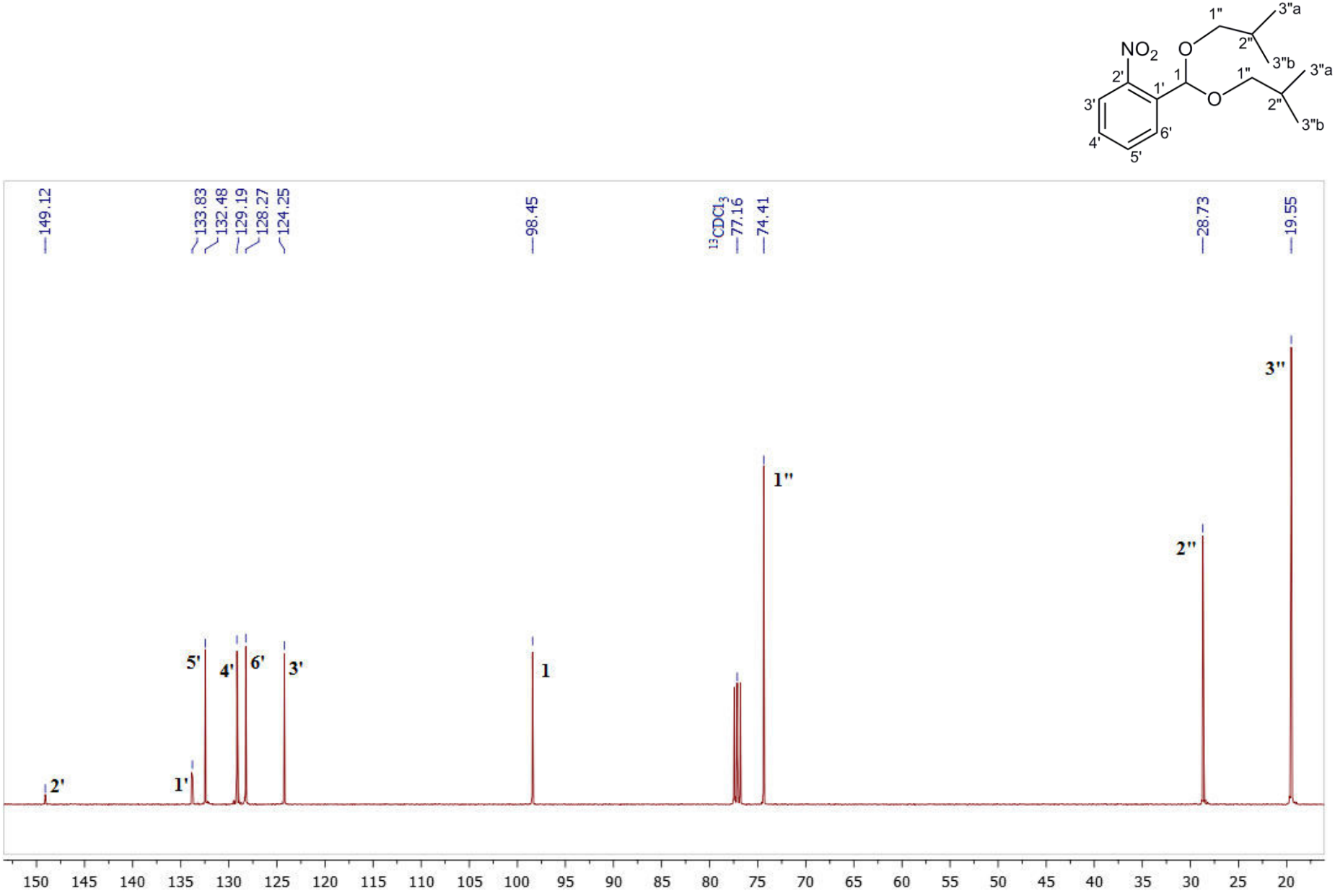


^{1}H NMR (400 MHz, $CDCl_3$) spectrum of (1-(diisobutoxymethyl)-2-nitrobenzene) and the corresponding expansions with signal assignment

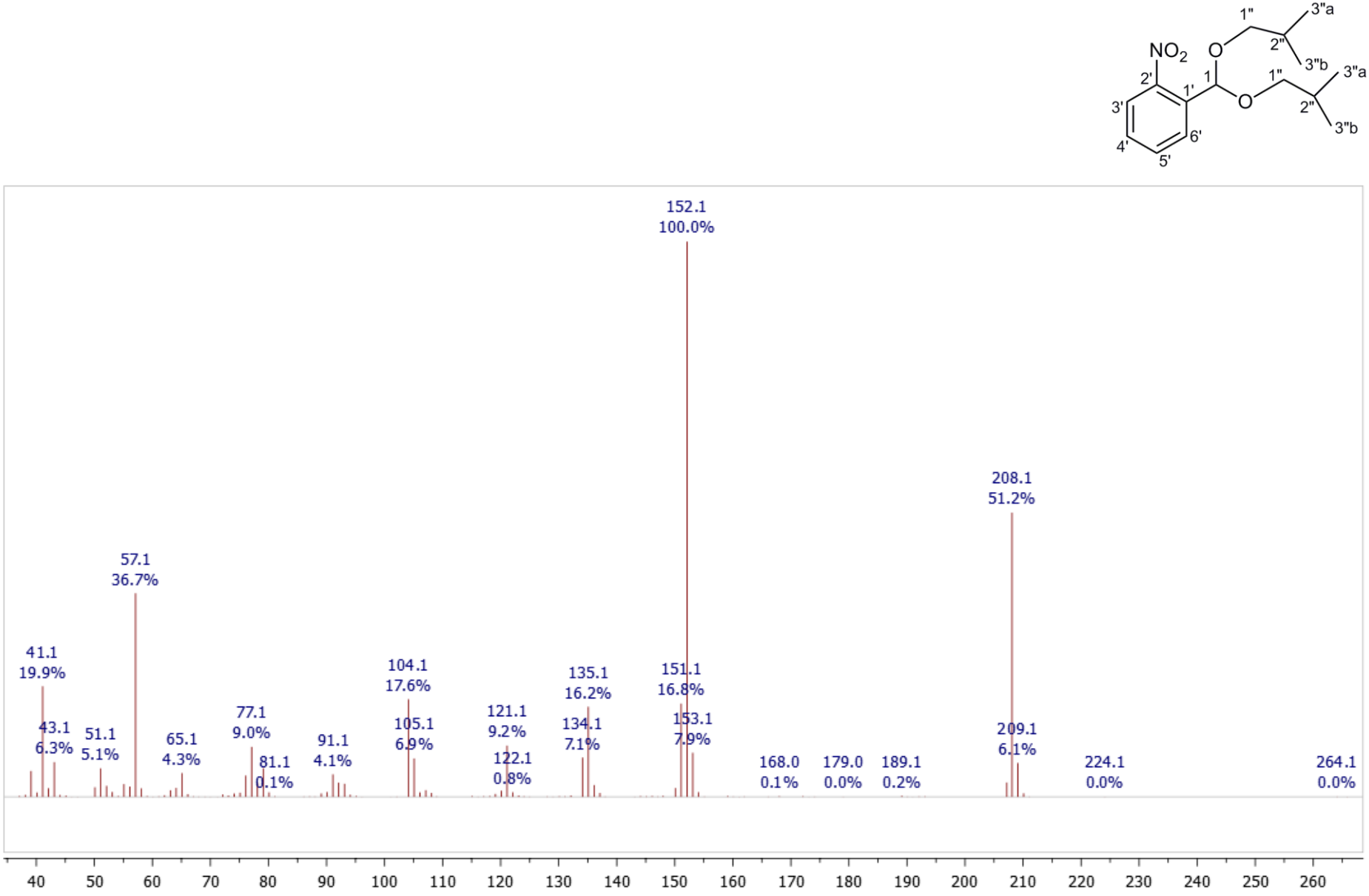




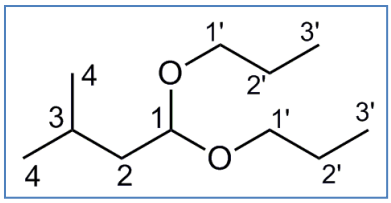
Selected expansions of ^{1}H-NMR spectrum (top) and
the corresponding expansions in ^{1}H-NMR spectrum during the decoupling of the acetal proton (bottom)



^{13}C-NMR (100.6 MHz, $CDCl_3$) spectrum of 1-(diisobutoxymethyl)-2-nitrobenzene with signal assignment



EI-MS spectrum of 1-(diisobutoxymethyl)-2-nitrobenzene

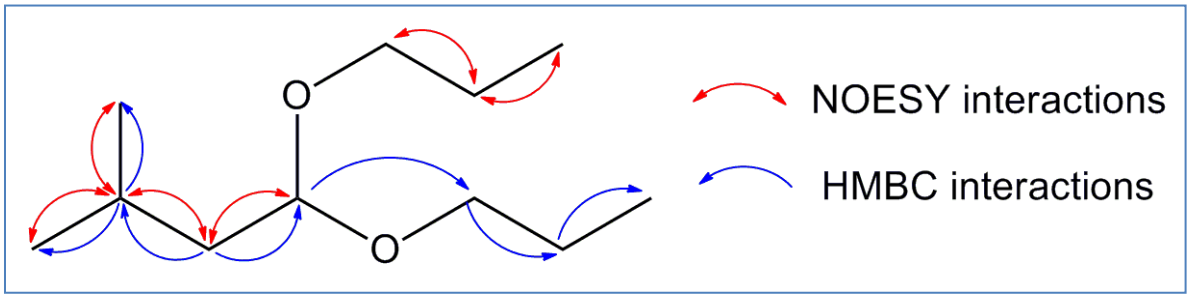


3-methyl-1,1-dipropoxybutan

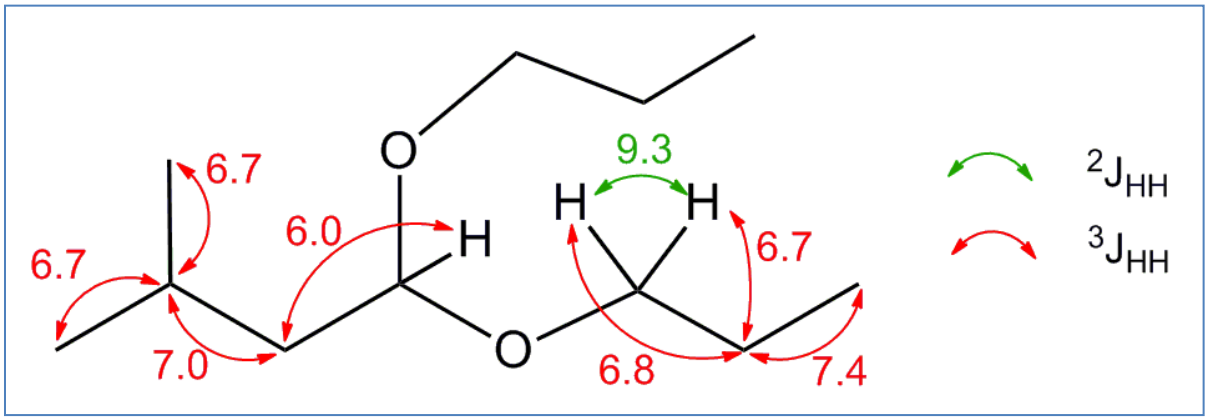
Table of NMR data of 3-methyl-1,1-dipropoxybutan (chemical shift and coupling constant data from ^{1}H and ^{13}C NMR spectra with the observed HMBC and NOESY interactions)

Position	δ_H (Integral, m, *J* (Hz))	δ_C (ppm) C-13 {H}	HMBC[a]	NOESY
1	4.56 (1 H, t, $^{3}J_{1,2}$ = 6.0)	101.9 (1 C)	3, 1’	2
2	1.50 (2 H, dd, $^{3}J_{2,3}$ = 7.0, $^{3}J_{1,2}$ = 6.0)	42.4 (1 C)	1, 3, 4	1, 3
3	1.73 (1 H, t of septuplet, $^{3}J_{2,3}$ = 7.0, $^{3}J_{3,4}$ = 6.7)[b]	24.5 (1 C)	1, 2, 4	2, 4
4	0.92 (6 H, d, $^{3}J_{3,4}$ = 6.7 Hz)	22.9 (1 C)	2, 3	3
1’a	3.38 (2 H, dt, $^{2}J_{1'a,1'b}$ = 9.3, $^{3}J_{1'a,2'}$ = 6.8)	67.2 (2 C)	1, 2’, 3’	2’
1’b	3.54 (2 H, dt, $^{2}J_{1'a,1'b}$ = 9.3, $^{3}J_{1'b,2'}$ = 6.7)			
2’	1.59 (4 H, pseudo sextuplet, $^{3}J_{2',3'}$ = 7.4, $^{3}J_{1'a,2'}$ = 6.8, $^{3}J_{1'b,2'}$ = 6.7)[b]	23.3 (2 C)	1’, 3’	1’, 3’
3’	0.93 (6 H, t, $^{3}J_{2',3'}$ = 7.4)	10.9 (2 C)	1’, 2’	2’

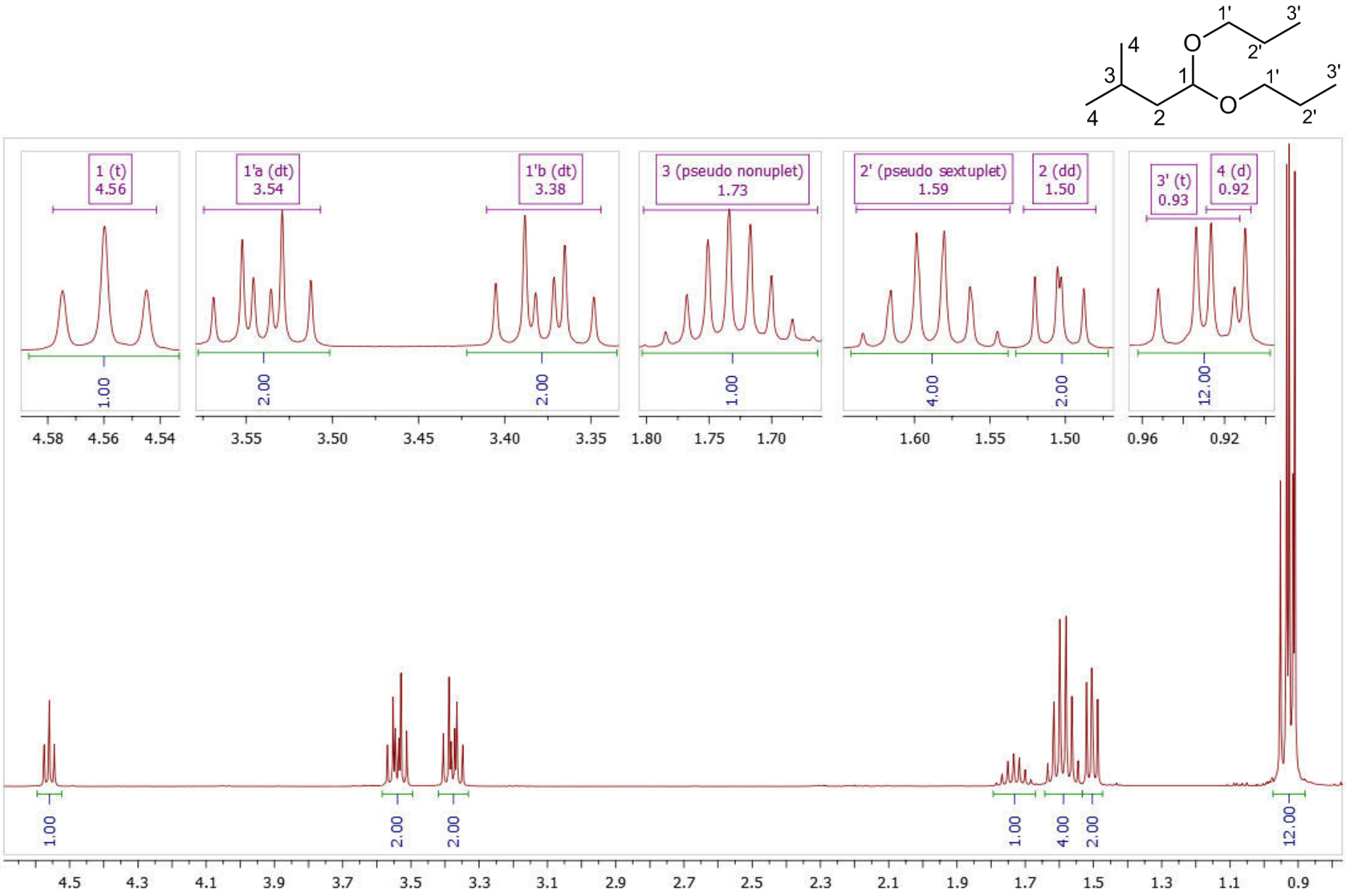
[a]Correlation between the hydrogen in this row and the carbon in the listed position.
[b]Coupling constants (and multiplicity) were determined from appropriate ^{1}H selective homodecoupled spectra.



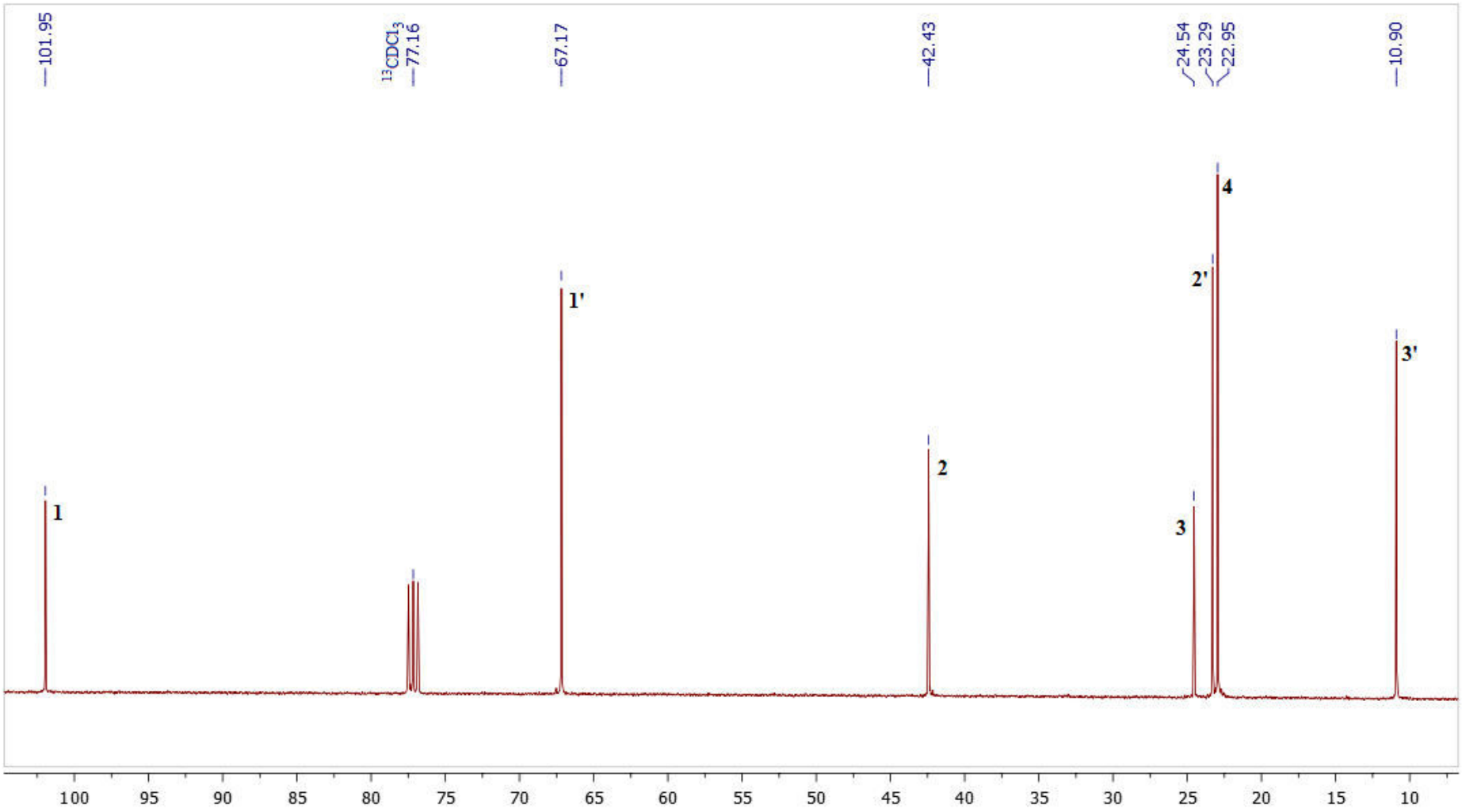
Scheme with key HMBC and NOESY interactions



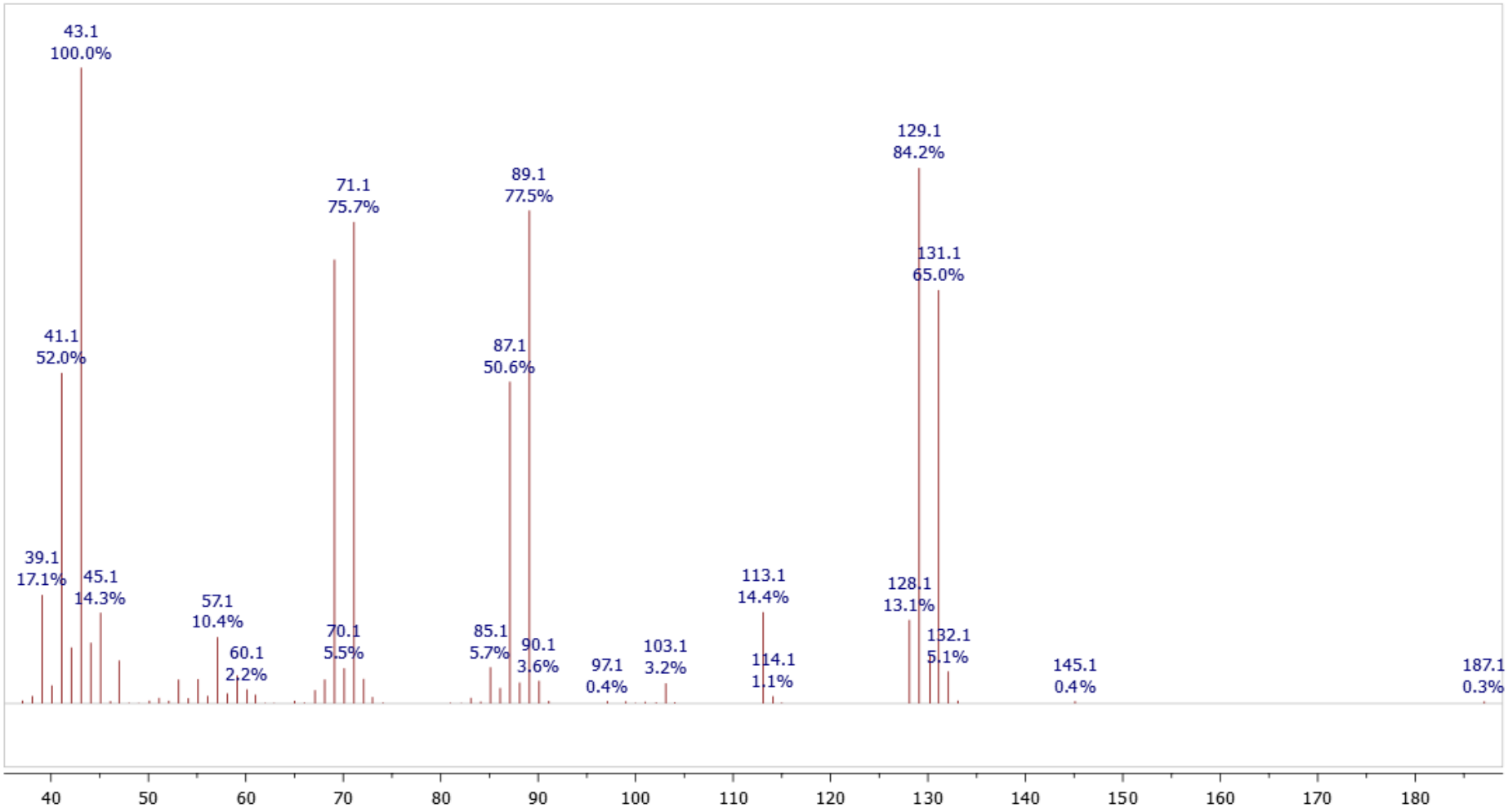
Analysis of ^{1}H-^{1}H coupling constants



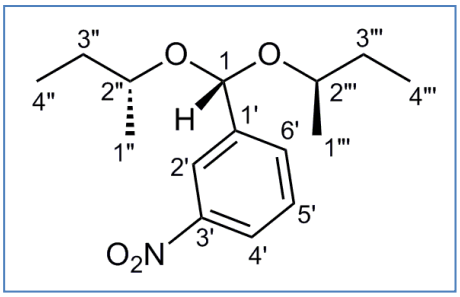
^{1}H-NMR (400 MHz, $CDCl_3$) spectrum of 3-methyl-1,1-dipropoxybutan and the corresponding expansions with signal assignment



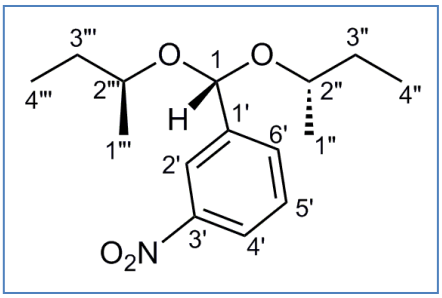
^{13}C-NMR (100.6 MHz, $CDCl_3$) spectrum of 3-methyl-1,1-dipropoxybutan with signal assignment



EI-MS spectrum of 3-methyl-1,1-dipropoxybutane



1-(di((*R*)-*sec*-butoxy)methyl)-3-nitrobenzene (compound 3a)



1-(di((*S*)-*sec*-butoxy)methyl)-3-nitrobenzene (compound 3b)

Table of NMR data of 1-(di((*R*/*S*)-*sec*-butoxy)methyl)-3-nitrobenzene (chemical shift and coupling constant data from ^{1}H and ^{13}C NMR spectra with the observed HMBC and NOESY interactions). This enantiomeric pair made up 50% of the reaction products.

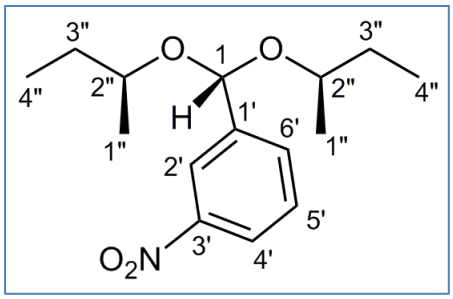
Position	δ_H (Integral, m, *J* (Hz))	δ_C (ppm) C-13 {H}	HMBC[a]	NOESY
1	5.60 (1 H, ddd, $^{4}J_{1,6'}$ = 0.6, $^{4}J_{1,2'}$ = 0.5, $^{6}J_{1,4'}$ = 0.4)[b]	98.75[c] (1 C)	2', 6', 2"	/
1'	/	143.03 (1 C)	/	/
2'	8.34 (1 H, dddd, $^{4}J_{2',4'}$ = 2.3, $^{4}J_{2',6'}$ = 1.5, $^{4}J_{1,2'}$ = 0.5, $^{5}J_{2',5'}$ = 0.2)[b]	121.87 (1 C)	1, 3', 4', 6'	/
3'	/	148.09 (1 C)	/	/
4'	8.18 (1 H, dddd, $^{3}J_{4',5'}$ = 8.2, $^{4}J_{2',4'}$ = 2.3, $^{4}J_{4',6'}$ = 1.0, $^{4}J_{1,4'}$ = 0.3)[b]	123.20 (1 C)	2', 3', 6'	5'
5'	7.54 (1 H, ddd, $^{3}J_{4',5'}$ = 8.2, $^{3}J_{5',6'}$ = 7.7, $^{5}J_{2',5'}$ = 0.2)[b]	129.14 (1 C)	1', 3', 4', 6'	4', 6'
6'	7.83 (1 H, dddd, $^{3}J_{5',6'}$ = 7.7, $^{4}J_{2',6'}$ = 1.5, $^{4}J_{4',6'}$ = 1.0, $^{4}J_{1,6'}$ = 0.6)[b]	132.96 (1 C)	1, 2', 4', 5'	5'
1"	1.14 (3 H, d, $^{3}J_{1"a,2"}$ = 6.2)	19.66 (1 C)	2", 3"	2"
2"	3.75 (1 H, tq, $^{3}J_{2",3"}$ = 6.4, $^{3}J_{1",2"}$ = 6.2)[b]	73.73 (1 C)	1", 3", 4"	1, 3"ab
3"a	1.63[d]	29.23 (1 C)	2", 4"	2", 3"b, 4"
3"b	1.52[d]		2", 4"	2", 3"a, 4"
4"	0.95 (3 H, t, $^{3}J_{3"a,4"}$ = $^{3}J_{3"b,4"}$ = 7.5)	9.84 (1 C)	3"	3"ab
1"'	1.21 (3 H, d, $^{3}J_{1"',2"'}$ = 6.2)[b]	20.03 (1 C)	2"', 3"'	2"'
2"'	3.68 (1 H, tq, $^{3}J_{2"',3"'}$ = 6.4, $^{3}J_{1"',2"'}$ = 6.2)[b]	74.31 (1 C)	1"', 3"', 4"'	1, 3"'ab
3"'a	1.59[d]	29.86 (1 C)	2"', 4"'	2"', 3"'b, 4"'
3"'b	1.49[d]		2"', 4"'	2"', 3"'a, 4"'
4"'	0.86 (3 H, t, $^{3}J_{3"'a,4"'}$ = $^{3}J_{3"'b,4"'}$ = 7.5)	9.49 (1 C)	3"'	3"'ab

[a]Correlation between the hydrogen in this row and the carbon in the listed position.

[b]Coupling constants (and multiplicity) were determined from appropriate ^{1}H selective homodecoupled spectra. Values of coupling constants lower than 0.5 Hz were usually observed as a broadening of the corresponding ^{1}H NMR signals; they were disclosed only in a series of selective homodecoupling experiments. For overlapping signals of the isomers, assignation was performed with the aid of spin simulation, and the comparison with related acetals of 3-nitrobenzadehyde.

[c]Due to a small difference in chemical shift values, these are presented on two decimal points.

[d]Overlapped signals (range: 1.42-1.67 ppm, 4 H). Chemical shifts were determined from HSQC and HMBC spectra.



1-((*r*)-(*R*)-*sec*-butoxy((*S*)-*sec*-butoxy)methyl)-3-nitrobenzene (compound 4)

Table of NMR data of 1-((*r*)-(*R*)-*sec*-butoxy((*S*)-*sec*-butoxy)methyl)-3-nitrobenzene (chemical shift and coupling constant data from ^{1}H and ^{13}C NMR spectra with the observed HMBC and NOESY interactions). This *meso* compound made up 29% of the reaction products.

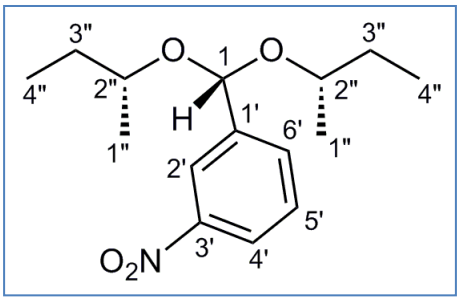
Position	δ_H (m, *J* (Hz), Integral)	δ_C (ppm) C-13 {H}	HMBC[a]	NOESY
1	5.61 (1 H, ddd, $^{4}J_{1,6'}$ = 0.6, $^{4}J_{1,2'}$ = 0.5, $^{6}J_{1,4'}$ = 0.4)[b]	98.16[c] (1 C)	2', 6', 2"	/
1'	/	143.25 (1 C)	/	/
2'	8.34 (1 H, dddd, $^{4}J_{2',4'}$ = 2.3, $^{4}J_{2',6'}$ = 1.5, $^{4}J_{1,2'}$ = 0.5, $^{5}J_{2',5'}$ = 0.2)[b]	121.89 (1 C)	1, 3', 4', 6'	/
3'	/	148.09 (1 C)	/	/
4'	8.18 (1 H, dddd, $^{3}J_{4',5'}$ = 8.2, $^{4}J_{2',4'}$ = 2.3, $^{4}J_{4',6'}$ = 1.0, $^{4}J_{1,4'}$ = 0.3)[b]	123.16 (1 C)	2', 3', 6'	5'
5'	7.54 (1 H, ddd, $^{3}J_{4',5'}$ = 8.2, $^{3}J_{5',6'}$ = 7.7, $^{5}J_{2',5'}$ = 0.2)[b]	129.12 (1 C)	1', 3', 4', 6'	4', 6'
6'	7.83 (1 H, dddd, $^{3}J_{5',6'}$ = 7.7, $^{4}J_{2',6'}$ = 1.5, $^{4}J_{4',6'}$ = 1.0, $^{4}J_{1,6'}$ = 0.6)[b]	132.97 (1 C)	1, 2', 4', 5'	5'
1"	1.14 (3 H, d, $^{3}J_{1",2"}$ = 6.2)	19.62 (2 C)	2", 3"	2"
2"	3.75 (1 H, tq, $^{3}J_{2",3"}$ = 6.4, $^{3}J_{1",2"}$ = 6.2)[b]	73.93 (2 C)	1", 3", 4"	1, 3"ab
3"a	1.62[d]	29.88 (2 C)	2", 4"	2", 3"b, 4"
3"b	1.50[d]		2", 4"	2", 3"a, 4"
4"	0.94 (3 H, t, $^{3}J_{3"a,4"}$ = $^{3}J_{3"b,4"}$ = 7.5)	9.85 (2 C)	3"	3"ab

[a]Correlation between the hydrogen in this row and the carbon in the listed position.

[b]Coupling constants (and multiplicity) were determined from appropriate ^{1}H selective homodecoupled spectra. Values of coupling constants lower than 0.5 Hz were usually observed as a broadening of the corresponding ^{1}H NMR signals; they were disclosed only in a series of selective homodecoupling experiments. For overlapping signals of the isomers, assignation was performed with the aid of spin simulation, and the comparison with related acetals of 3-nitrobenzadehyde.

[c]Due to a small difference in chemical shift values, these are presented on two decimal points.

[d]Overlapped signals (range: 1.42-1.67 ppm, 4 H). Chemical shifts were determined from HSQC and HMBC spectra.



1-((*s*)-(*R*)-*sec*-butoxy((*S*)-*sec*-butoxy)methyl)-3-nitrobenzene (compound 5)

Table of NMR data of 1-((*s*)-(*R*)-*sec*-butoxy((*S*)-*sec*-butoxy)methyl)-3-nitrobenzene (chemical shift and coupling constant data from ^{1}H and ^{13}C NMR spectra with the observed HMBC and NOESY interactions). This *meso* compound made up 21% of the reaction products.

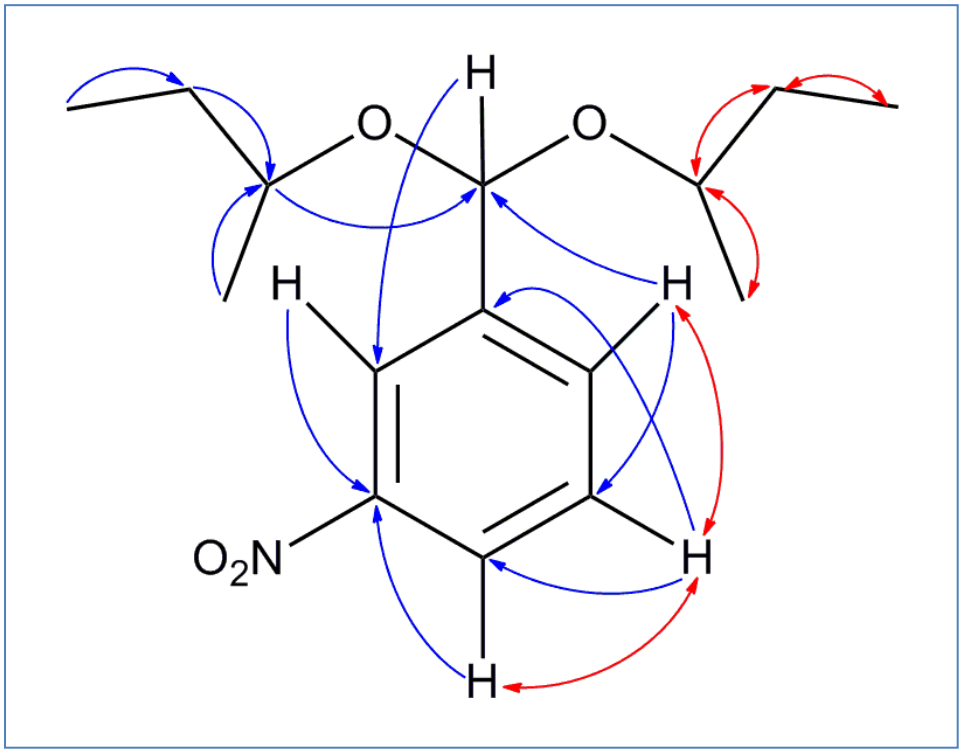
Position	δ_H (m, *J* (Hz), Integral)	δ_C (ppm) C-13 {H}	HMBC[a]	NOESY
1	5.58 (1 H, ddd, $^{4}J_{1,6'}$ = 0.6, $^{4}J_{1,2'}$ = 0.5, $^{6}J_{1,4'}$ = 0.4)[b]	99.45[c] (1 C)	2', 6', 2"	/
1'	/	142.86 (1 C)	/	/
2'	8.34 (1 H, dddd, $^{4}J_{2',4'}$ = 2.3, $^{4}J_{2',6'}$ = 1.5, $^{4}J_{1,2'}$ = 0.5, $^{5}J_{2',5'}$ = 0.2)[b]	121.85 (1 C)	1, 3', 4', 6'	/
3'	/	148.09 (1 C)	/	/
4'	8.18 (1 H, dddd, $^{3}J_{4',5'}$ = 8.2, $^{4}J_{2',4'}$ = 2.3, $^{4}J_{4',6'}$ = 1.0, $^{4}J_{1,4'}$ = 0.3)[b]	123.23 (1 C)	2', 3', 6'	5'
5'	7.54 (1 H, ddd, $^{3}J_{4',5'}$ = 8.2, $^{3}J_{5',6'}$ = 7.7, $^{5}J_{2',5'}$ = 0.2)[b]	129.16 (1 C)	1', 3', 4', 6'	4', 6'
6'	7.83 (1 H, dddd, $^{3}J_{5',6'}$ = 7.7, $^{4}J_{2',6'}$ = 1.5, $^{4}J_{4',6'}$ = 1.0, $^{4}J_{1,6'}$ = 0.6)[b]	132.95 (1 C)	1, 2', 4', 5'	5'
1"	1.21 (3 H, d, $^{3}J_{1''',2'''}$ = 6.2)[b]	20.06 (2 C)	2", 3"	2"
2"	3.68 (1 H, pseudo sextuplet, $^{3}J_{2",3"}$ = 6.4, $^{3}J_{1",2"}$ = 6.2)	74.29 (2 C)	1", 3", 4"	1, 3"ab
3"a	1.59[d]	29.25 (2 C)	2", 4"	2", 3"b, 4"
3"b	1.49[d]		2", 4"	2", 3"a, 4"
4"	0.86 (3 H, t, $^{3}J_{3"a,4"}$ = $^{3}J_{3"b,4"}$ = 7.5)	9.51 (2 C)	3"	3"ab

[a]Correlation between the hydrogen in this row and the carbon in the listed position.

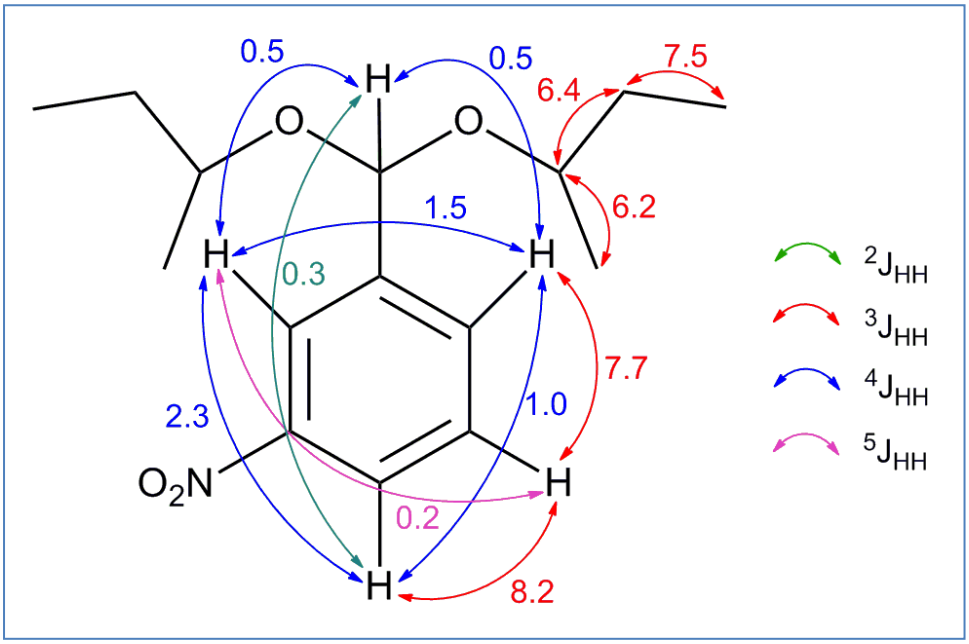
[b]Coupling constants (and multiplicity) were determined from appropriate ^{1}H selective homodecoupled spectra. Values of coupling constants lower than 0.5 Hz were usually observed as a broadening of the corresponding ^{1}H NMR signals; they were disclosed only in a series of selective homodecoupling experiments. For overlapping signals of the isomers, assignation was performed with the aid of spin simulation, and the comparison with related acetals of 3-nitrobenzadehyde.

[c]Due to a small difference in chemical shift values, these are presented on two decimal points.

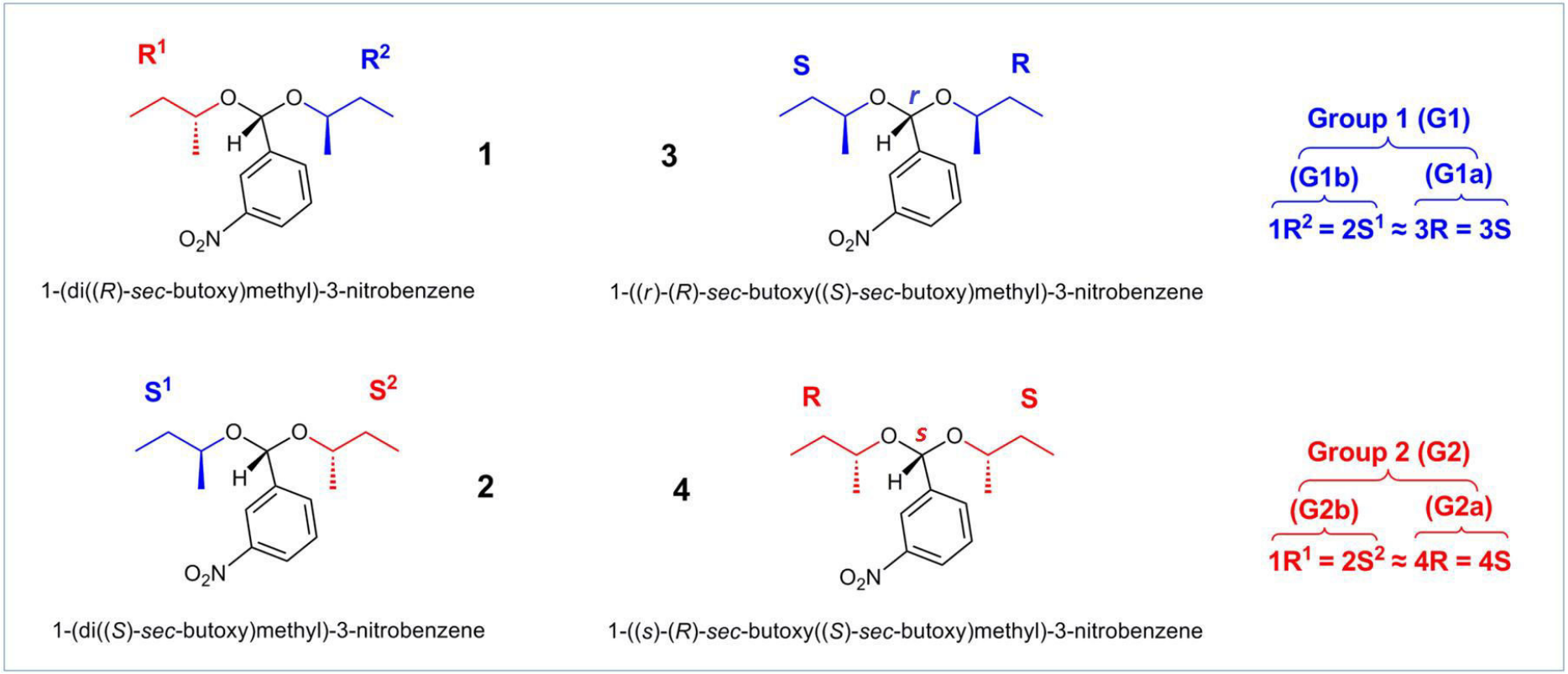
[d]Overlapped signals (range: 1.42-1.67 ppm, 4 H). Chemical shifts were determined from HSQC and HMBC spectra.



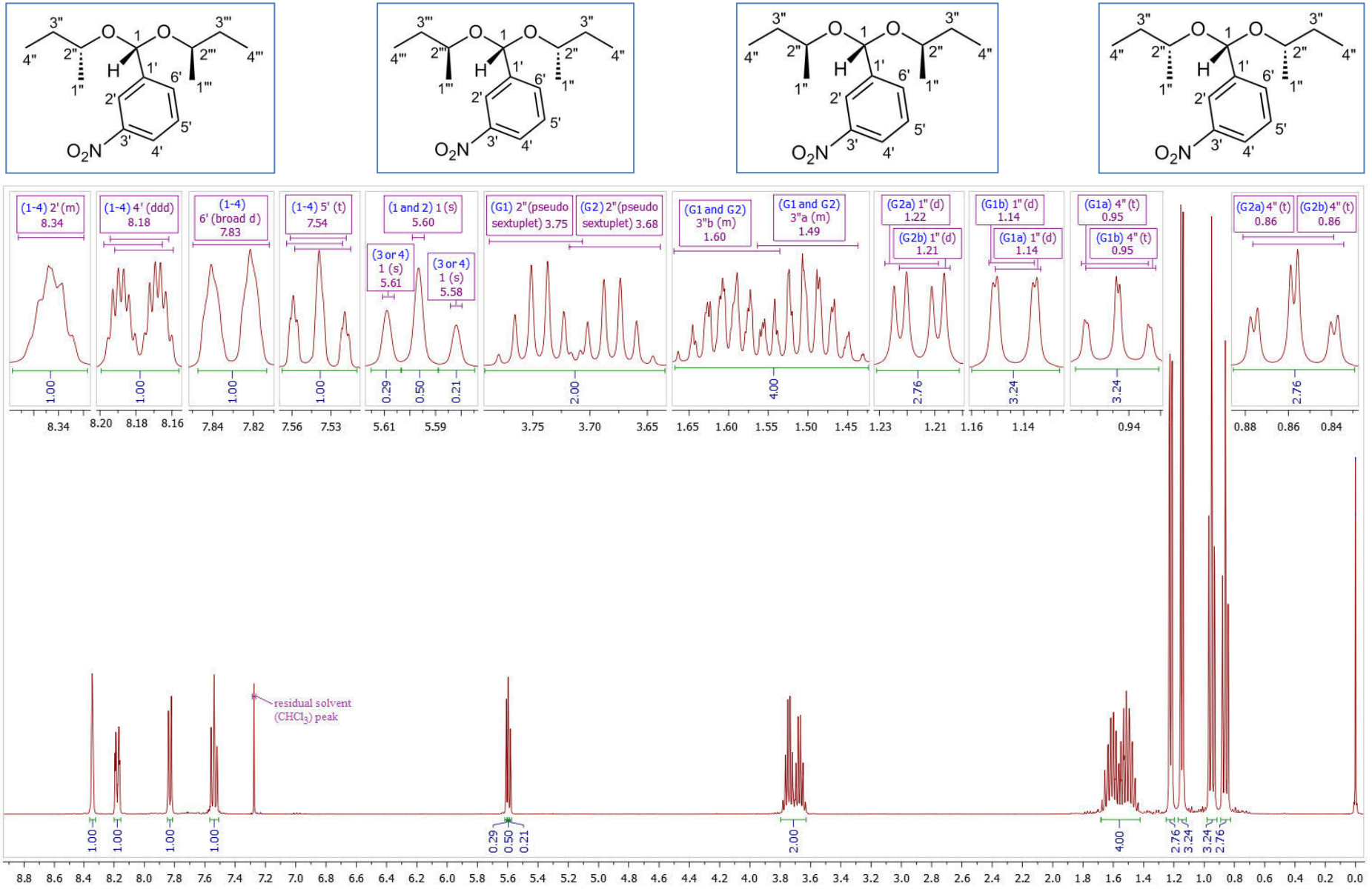
Scheme with key HMBC and NOESY interactions



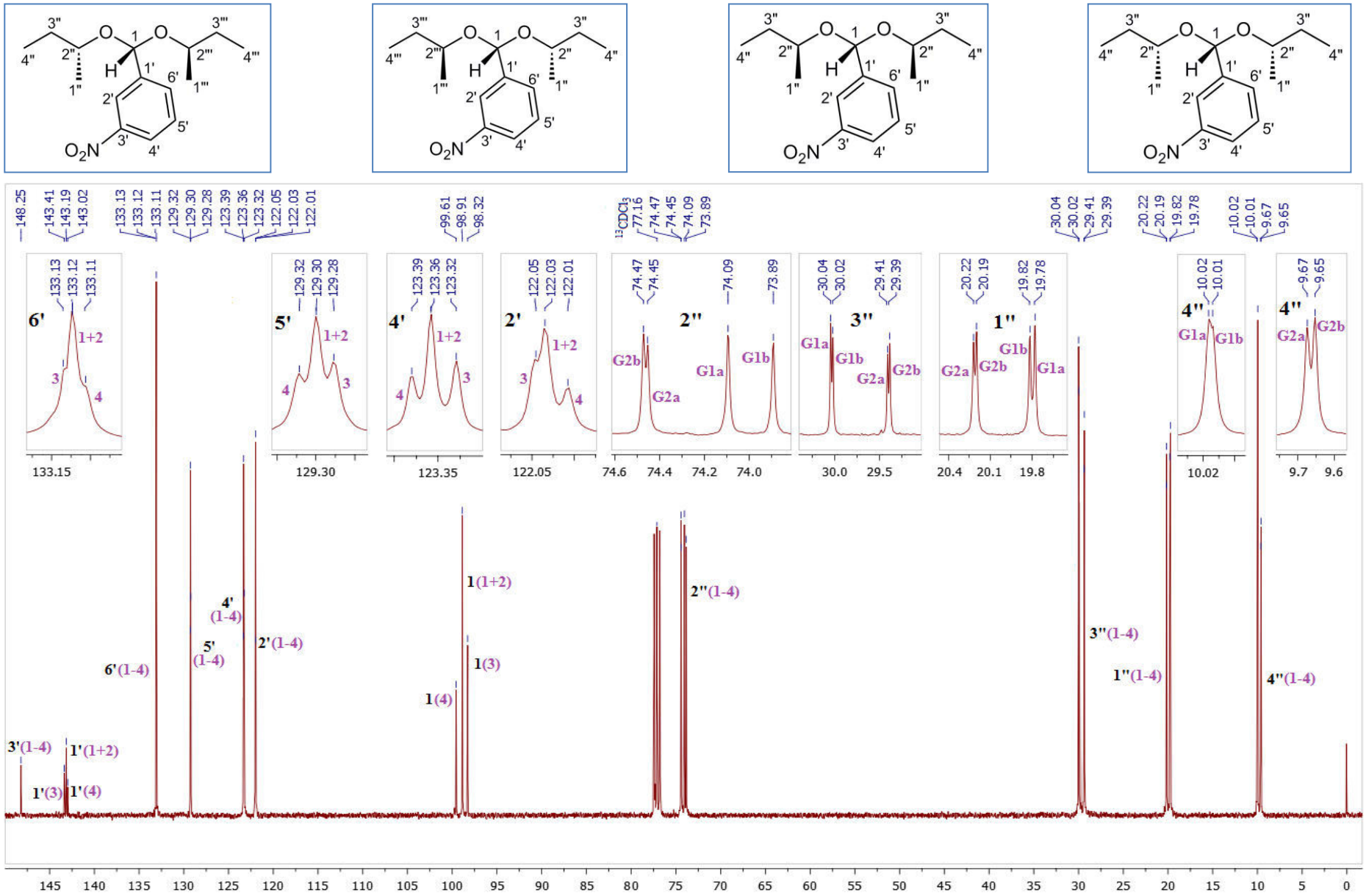
Analysis of ^{1}H-^{1}H coupling constants



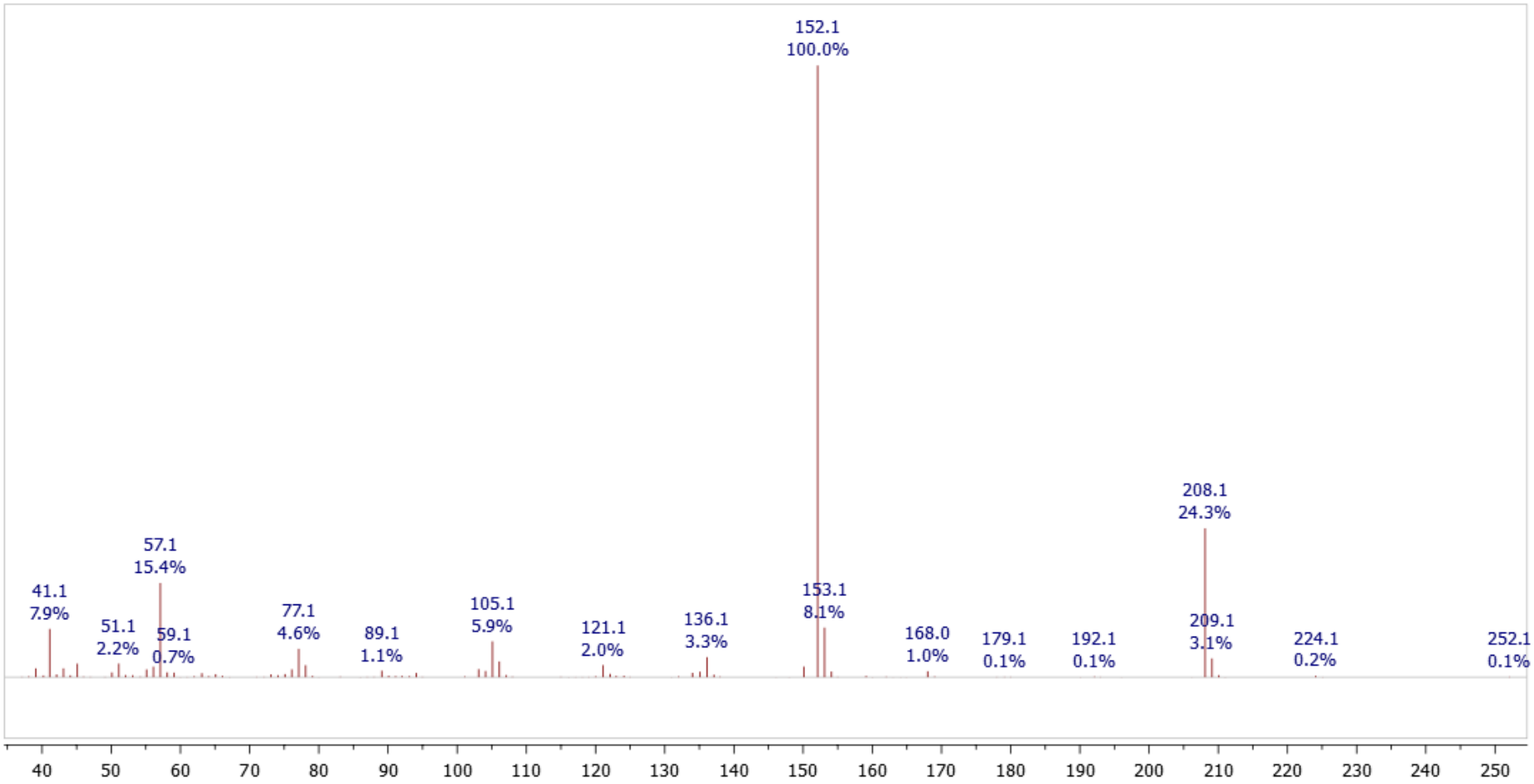
Possible stereoisomers of 1-(di(*sec*-butoxy)methyl)-3-nitrobenzene. Designations used for assignation of signals in spectra.



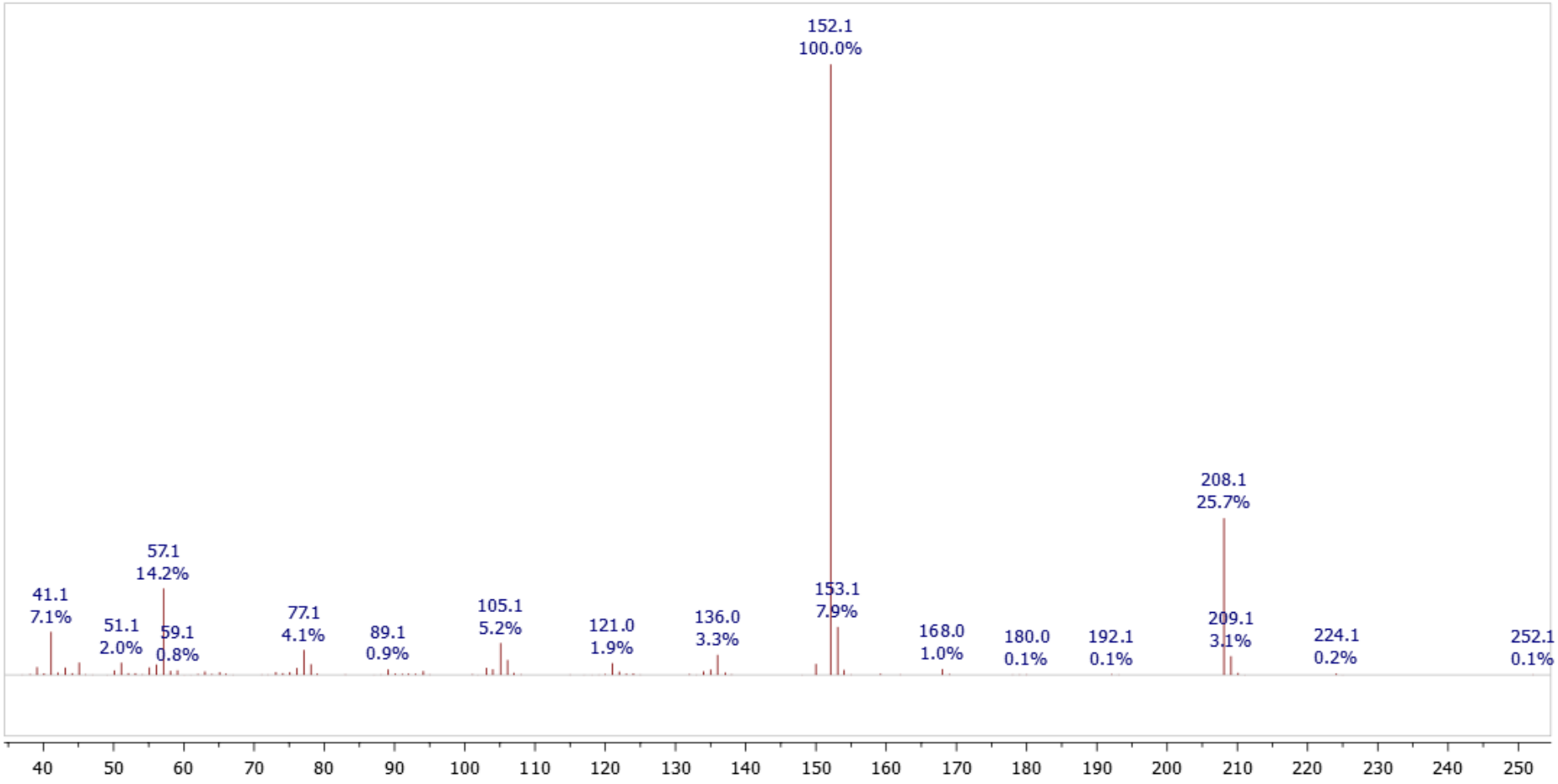
^{1}H-NMR (400 MHz, $CDCl_3$) spectrum of the mixture of diastereoisomeric 1-(di(*sec*-butoxy)methyl)-3-nitrobenzenes and the corresponding expansions with signal assignment (*r-meso* : *s-meso* : enantiomeric pair = 29 : 21 : 50)



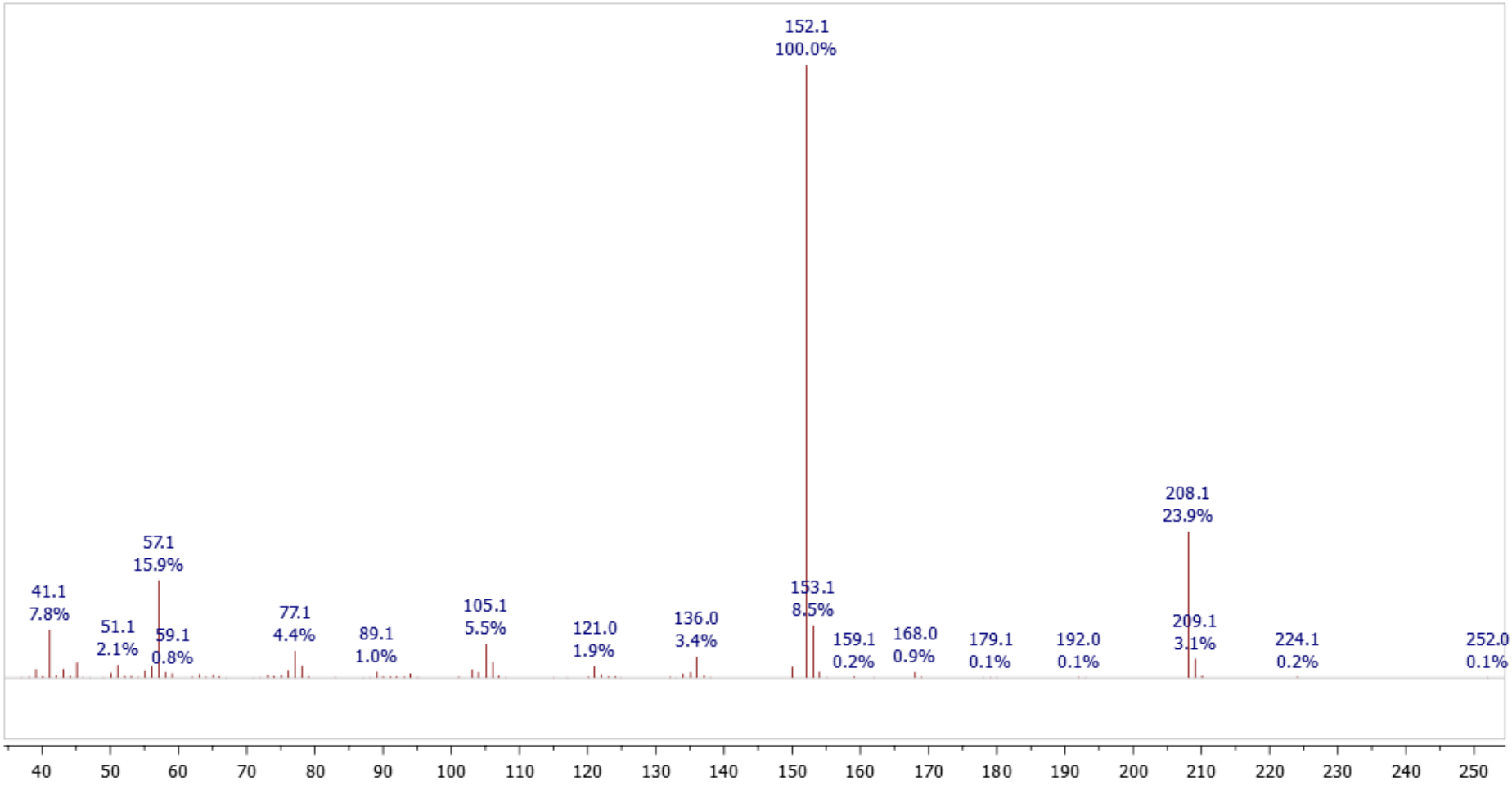
^{13}C-NMR (100.6 MHz, $CDCl_3$) spectrum of the mixture of diastereoisomeric 1-(di(*sec*-butoxy)methyl)-3-nitrobenzenes and the corresponding expansions with signal assignment (*r-meso* : *s-meso* : enantiomeric pair = 29 : 21 : 50)



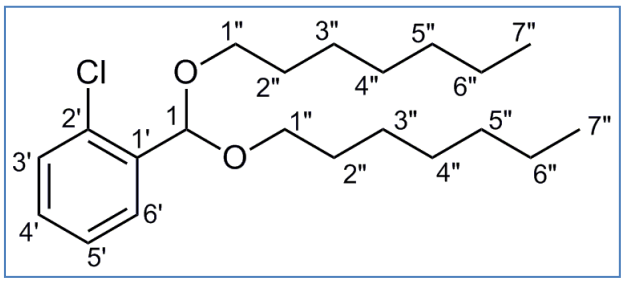
MS spectrum of 1-(di((*R*)-*sec*-butoxy)methyl)-3-nitrobenzene and 1-(di((*S*)-*sec*-butoxy)methyl)-3-nitrobenzene



MS spectrum of 1-((*r*)-(*R*)-*sec*-butoxy((*S*)-*sec*-butoxy)methyl)-3-nitrobenzene



MS spectrum of 1-((*s*)-(*R*)-*sec*-butoxy((*S*)-*sec*-butoxy)methyl)-3-nitrobenzene



1-(bis(heptyloxy)methyl)-2-chlorobenzene

Table of NMR data of 1-(bis(heptyloxy)methyl)-2-chlorobenzene (chemical shift and coupling constant data from ^{1}H and ^{13}C NMR spectra with the observed HMBC and NOESY interactions)

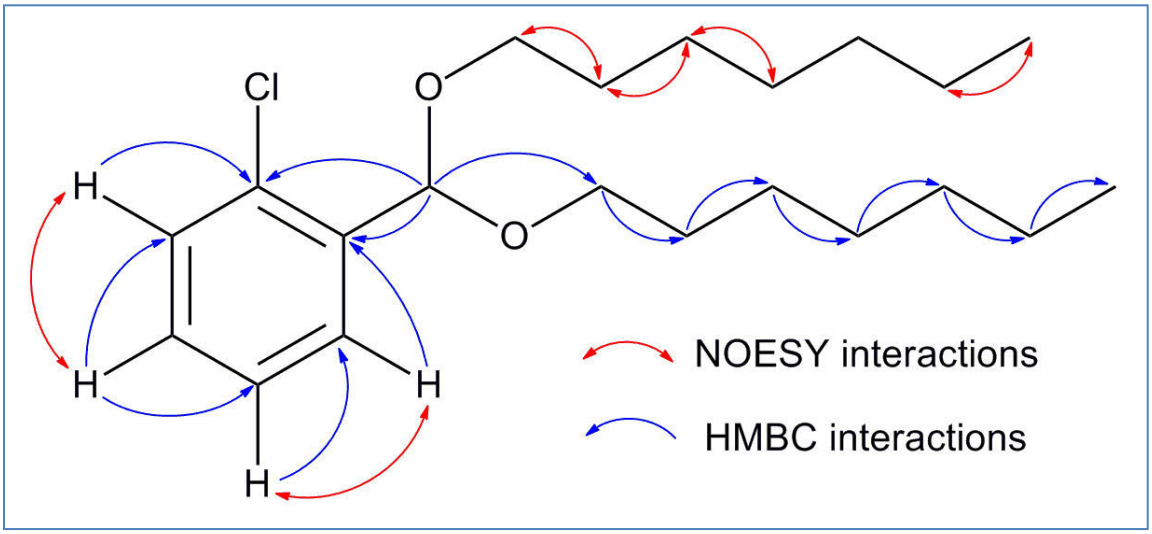
Position	δ_H (Integral, m, *J* (Hz))	δ_C (ppm) C-13 {H}	HMBC[a]	NOESY
1	5.72 (1 H, dd, $^{4}J_{1,6'} = 0.5$, $^{4}J_{1,4'} = 0.4$)[b]	99.5 (1 C)	1', 6', 1"	/
1'	/	133.3 (1 C)	/	/
2'	/	136.6 (1 C)	/	/
3'	7.34 (1 H, m, ABCD)[d]	129.6 (1 C)	1', 2', 4'	4'
4'	7.27 (1 H, m, ABCD, $J_{1,4'} = 0.4$)[d]	126.7 (1 C)	2', 3', 5'	3'
5'	7.25 (1 H, m, ABCD)[d]	129.6 (1 C)	1', 4', 6'	6'
6'	7.66 (1 H, m, ABCD, $^{4}J_{1,6'} = 0.5$)[d]	128.3 (1 C)	1, 1', 4', 5'	5'
1"a	3.49 (2 H, dt, $^{2}J_{1"a,1"b} = 9.3$, $^{3}J_{1"a,2"} = 6.6$)[b]	66.9 (2 C)	1, 2", 3"	2"
1"b	3.57 (2 H, dt, $^{2}J_{1"a,1"b} = 9.3$, $^{3}J_{1"b,2"} = 6.7$)[b]		1, 2", 3"	2"
2"	1.61 (4 H, tdd, $^{3}J_{2",3"} = 6.9$, $^{3}J_{1"b,2"} = 6.7$, $^{3}J_{1"a,2"} = 6.6$)[b]	29.9 (2 C)	1", 3", 4"	1", 3"
3"	1.35[e]	26.3 (2 C)	2", 4"	2", 4"
4"	1.28[e]	29.2 (2 C)	3", 5"	3"
5"	1.26[e]	32.0 (2 C)	4", 6", 7"	/
6"	1.28[e]	22.8 (2 C)	5", 7"	7"
7"	0.87 (6 H, t, $^{3}J_{6",7"} = 6.9$)[b]	14.2 (2 C)	5", 6"	6"

[a]Correlation between the hydrogen in this row and the carbon in the listed position.

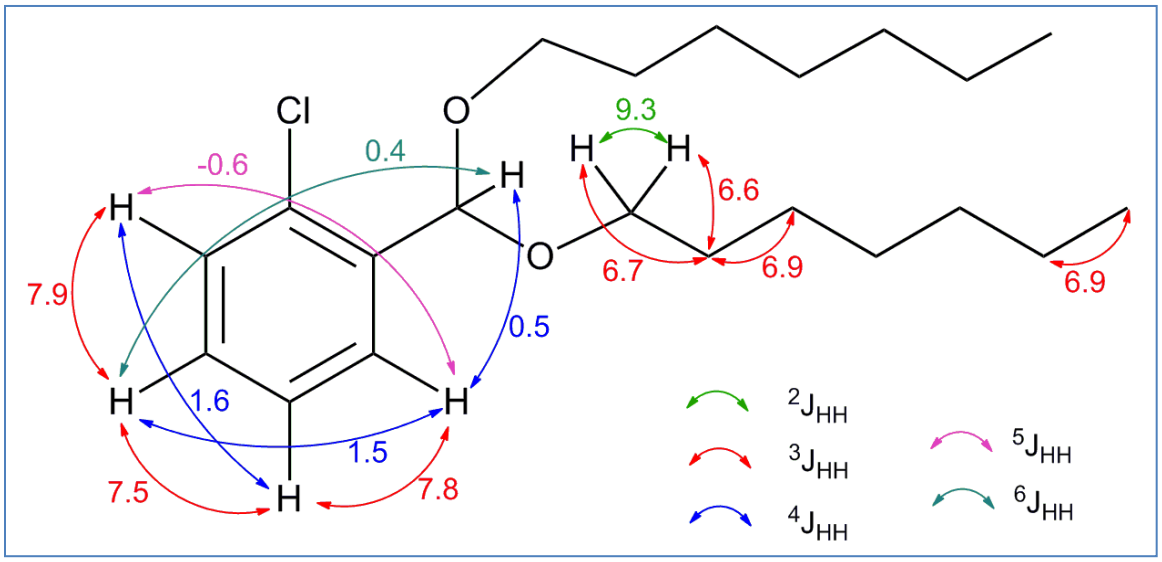
[b]Coupling constants (and multiplicity) were determined from appropriate ^{1}H selective homodecoupled spectra. Values of coupling constants lower than 0.5 Hz were usually observed as a broadening of the corresponding ^{1}H NMR signals; they were disclosed only in a series of selective homodecoupling experiments.

[c] $^{3}J_{AB} = 7.5$ Hz, $^{4}J_{AC} = 1.6$ Hz, $^{3}J_{AD} = 7.8$ Hz, $^{3}J_{BC} = 7.9$ Hz, $^{4}J_{BD} = 1.5$ Hz, $^{5}J_{CD} = -0.6$ Hz. The data from the spectrum obtained in a selective homodecoupling experiment (acetal proton decoupled) and "WinDNMR" simulation were used for the determination of the coupling constants. More accurate values of δ_H: 7.2451 (A/5'), 7.2732 (B/4'), 7.3440 (C/3'), 7.6555 (D/6').

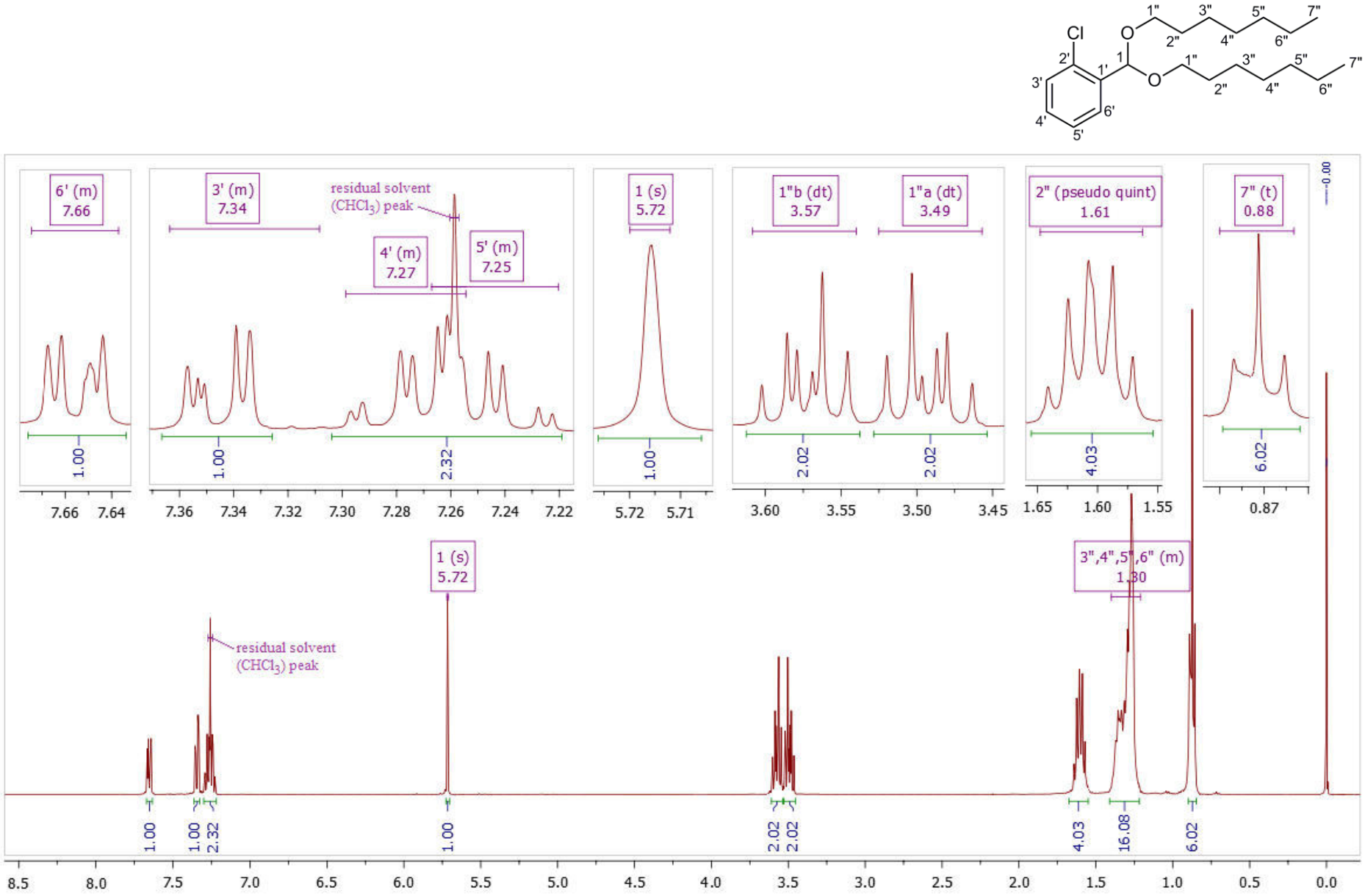
[e]Overlapped signals (range: 1.23-1.43 ppm, 16 H). Chemical shifts were determined from HSQC and HMBC spectra.



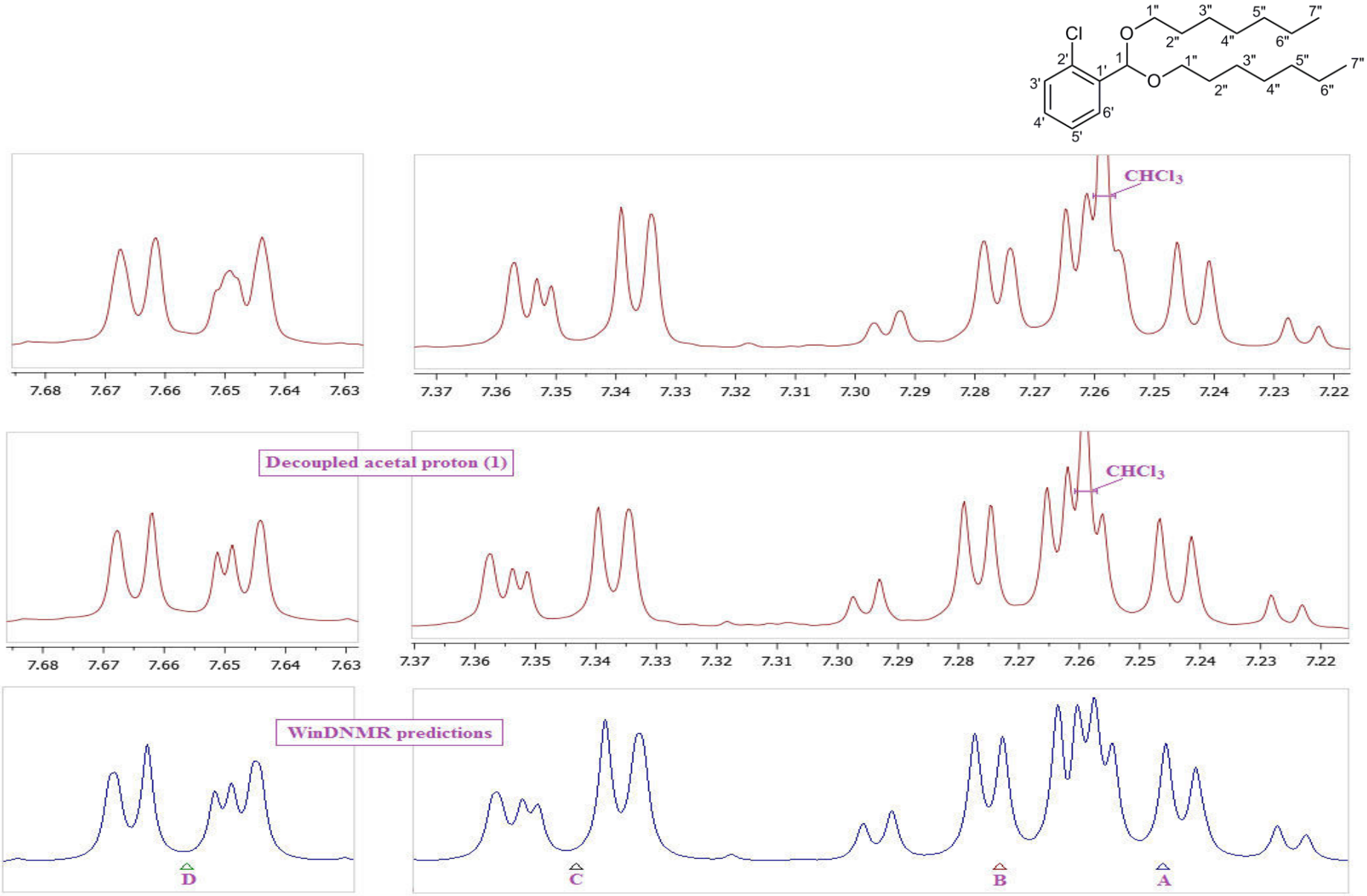
Scheme with key HMBC and NOESY interactions



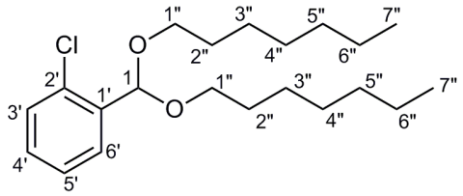
Analysis of ^{1}H-^{1}H coupling constants

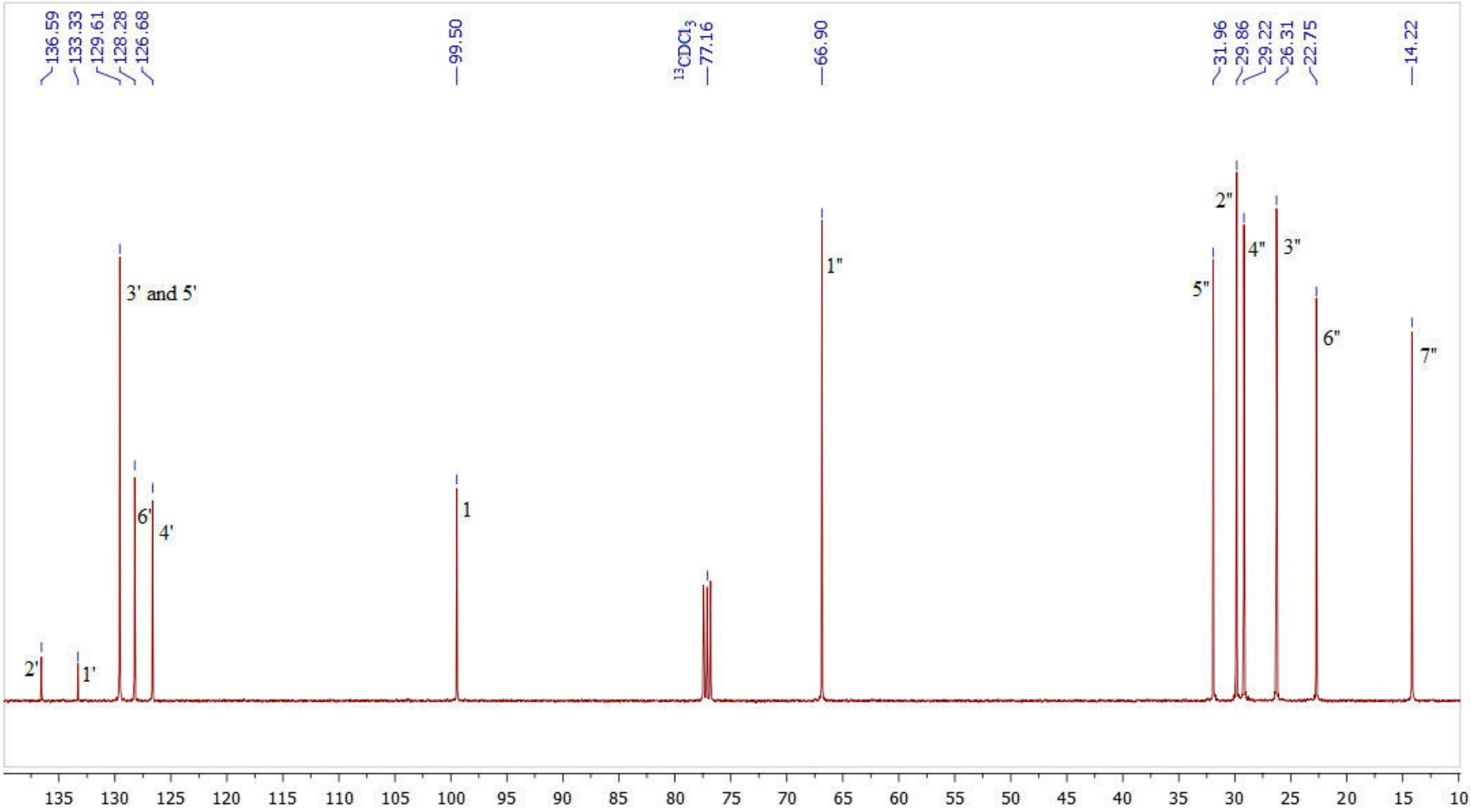


^{1}H-NMR (400 MHz, $CDCl_3$) spectrum of 1-(bis(heptyloxy)methyl)-2-chlorobenzene and the corresponding expansions with signal assignment

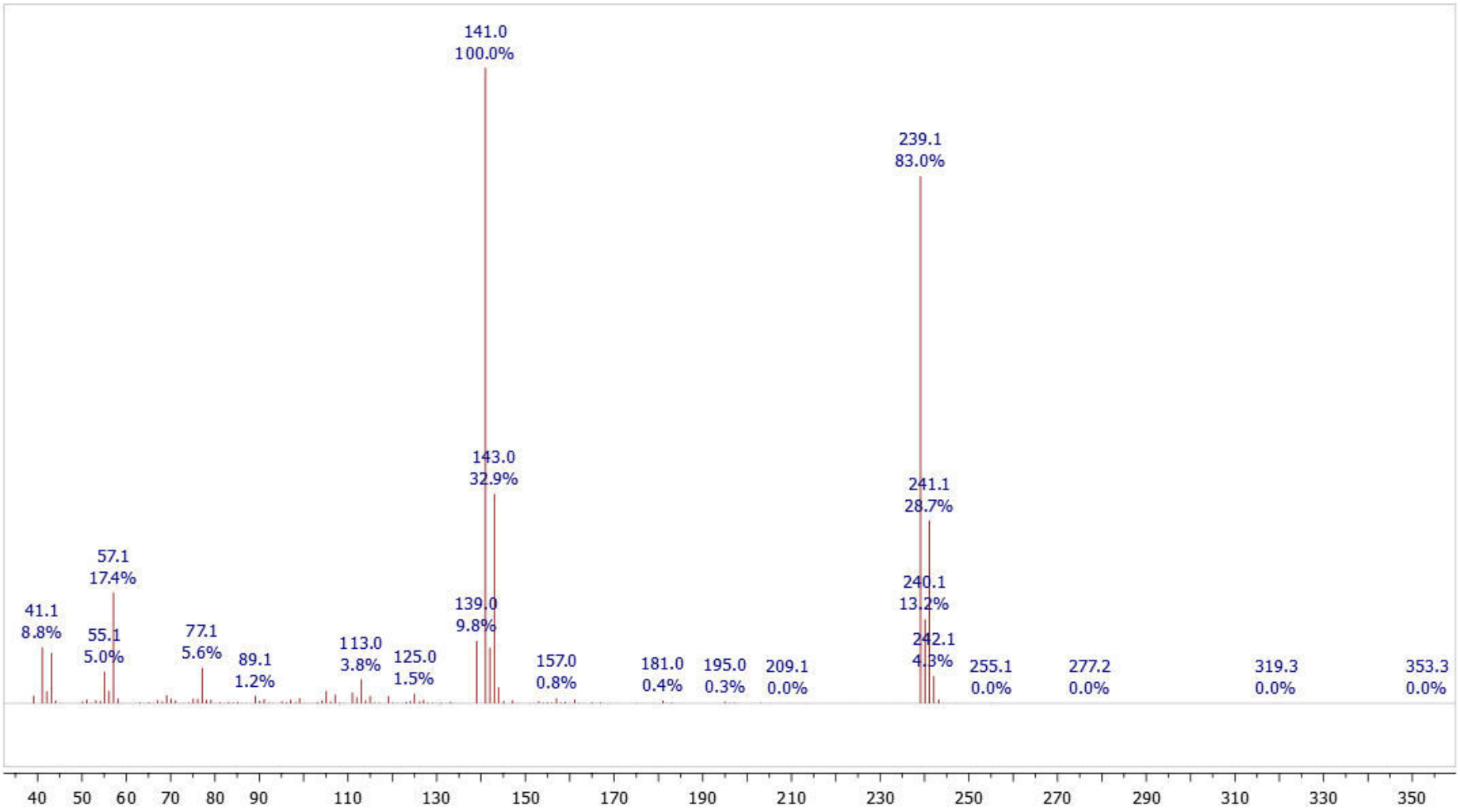


Expansions of ^{1}H-NMR spectrum of 1-(bis(heptyloxy)methyl)-2-chlorobenzene corresponding to protons attached to the aromatic ring (top); the corresponding expansions obtained in homodecoupling experiment (acetal proton decoupled; middle), and WinDNMR simulation of the mentioned decoupled spectrum (bottom)

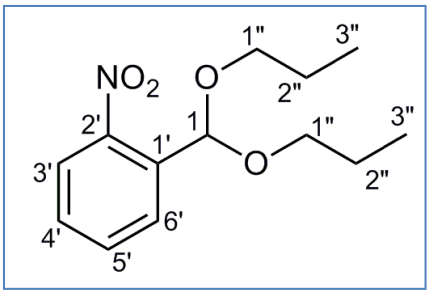




^{13}C-NMR (100.6 MHz, $CDCl_3$) spectrum of 1-(bis(heptyloxy)methyl)-2-chlorobenzene with signal assignment



EI-MS spectrum of 1-(bis(heptyloxy)methyl)-2-chlorobenzene



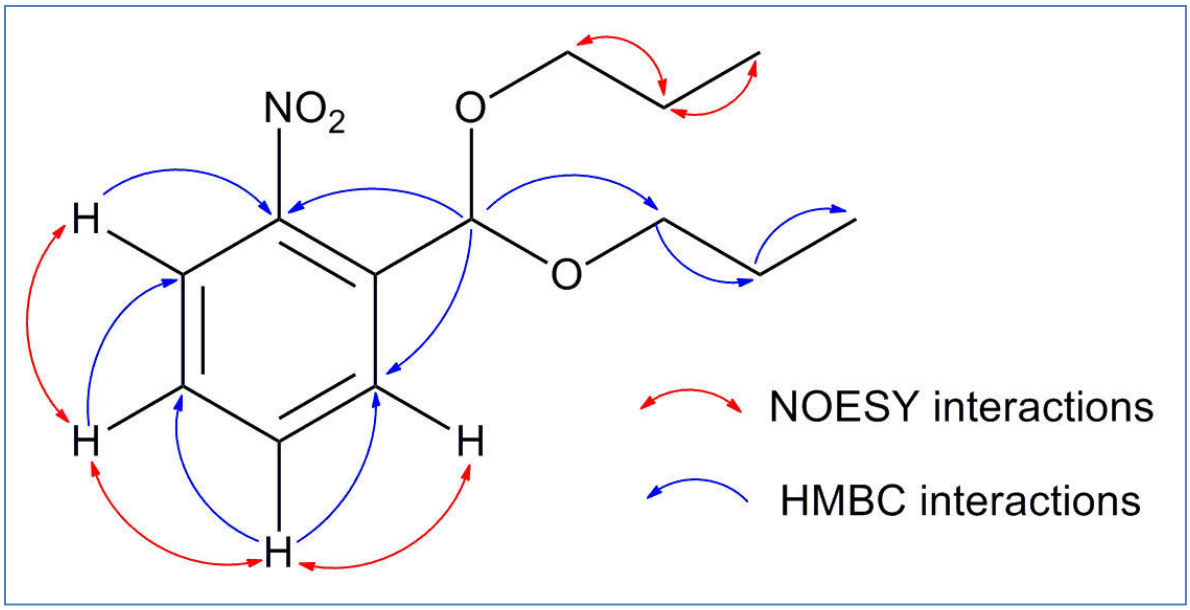
1-(dipropoxymethyl)-2-nitrobenzene

Table of NMR data of 1-(dipropoxymethyl)-2-nitrobenzene (chemical shift and coupling constant data from ^{1}H and ^{13}C NMR spectra with the observed HMBC and NOESY interactions)

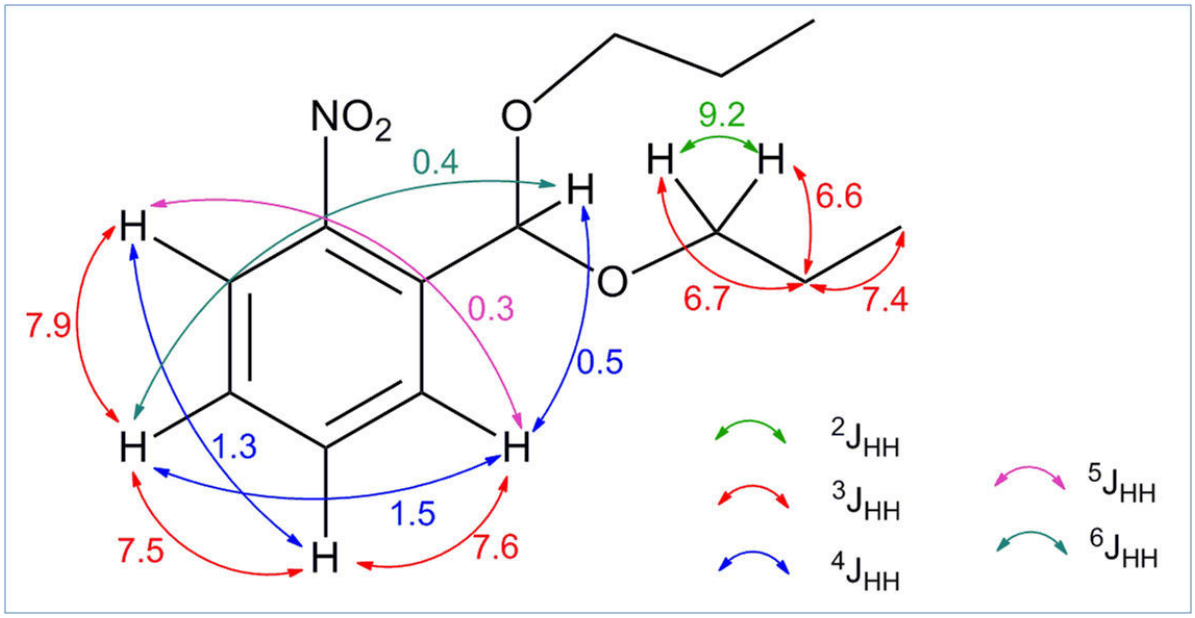
Position	δ_H (Integral, m, *J* (Hz))	δ_C (ppm) C-13 {H}	HMBC[c]	NOESY
1	6.04 (1 H, ddd, $^{4}J_{1,6'}$= 0.5, $^{6}J_{1,4'}$= 0.4, $^{5}J_{1,5'}$ = 0.3)[b]	98.6 (1 C)	2', 6', 1"	/
1'	/	133.9 (1 C)	/	/
2'	/	149.1 (1 C)	/	/
3'	7.81 (1 H, ddd, $^{3}J_{3',4'}$ =7.5, $^{4}J_{3',5'}$=1.3, $^{5}J_{3',6'}$= 0.3)[b]	124.3 (1 C)	5', 2'	4'
4'	7.45, (1 H, dddd, $^{3}J_{4',5'}$ = 7.9, $^{3}J_{3',4'}$ = 7.5, $^{4}J_{4',6'}$ = 1.5, $^{4}J_{1,4'}$= 0.4, 1H)[b]	129.2 (1 C)	2', 3', 5', 6'	3', 5'
5'	7.59 (1 H, ddd, $^{3}J_{4',5'}$ = 7.9, $^{3}J_{5',6'}$ = 7.6, $^{4}J_{3',5'}$ = 1.3, $^{5}J_{1,5'}$ = 0.3)[b]	132.6 (1 C)	1', 3', 4', 6'	4', 6'
6'	7.84, (1 H, dddd, $^{3}J_{5',6'}$ = 7.6, $^{4}J_{4',6'}$ = 1.5, $^{4}J_{1,6'}$= 0.5, $^{5}J_{3',6'}$= 0.3)[b]	128.2 (1 C)	2', 4'	5'
1"a	3.49 (2 H, dt, $^{2}J_{1"a,1"b}$ = 9.2, $^{3}J_{1"a,2"}$ = 6.7)	69.7 (2 C)	1, 2", 3"	2"
1"b	3.60 (2 H, dt, $^{2}J_{1"a,1"b}$ = 9.2, $^{3}J_{1"b,2"}$ = 6.6)			
2"	1.63 (4 H, qdd, $^{3}J_{2",3"}$ = 7.4, $^{3}J_{1"a,2"}$ = 6.7, $^{3}J_{1"b,2"}$ = 6.6)[b]	23.1 (2 C)	1", 3"	1", 3"
3"	0.94 (6 H, t, $^{3}J_{2",3"}$ = 7.4)	10.8 (2 C)	1", 2"	2"

[a]Correlation between the hydrogen in this row and the carbon in the listed position.

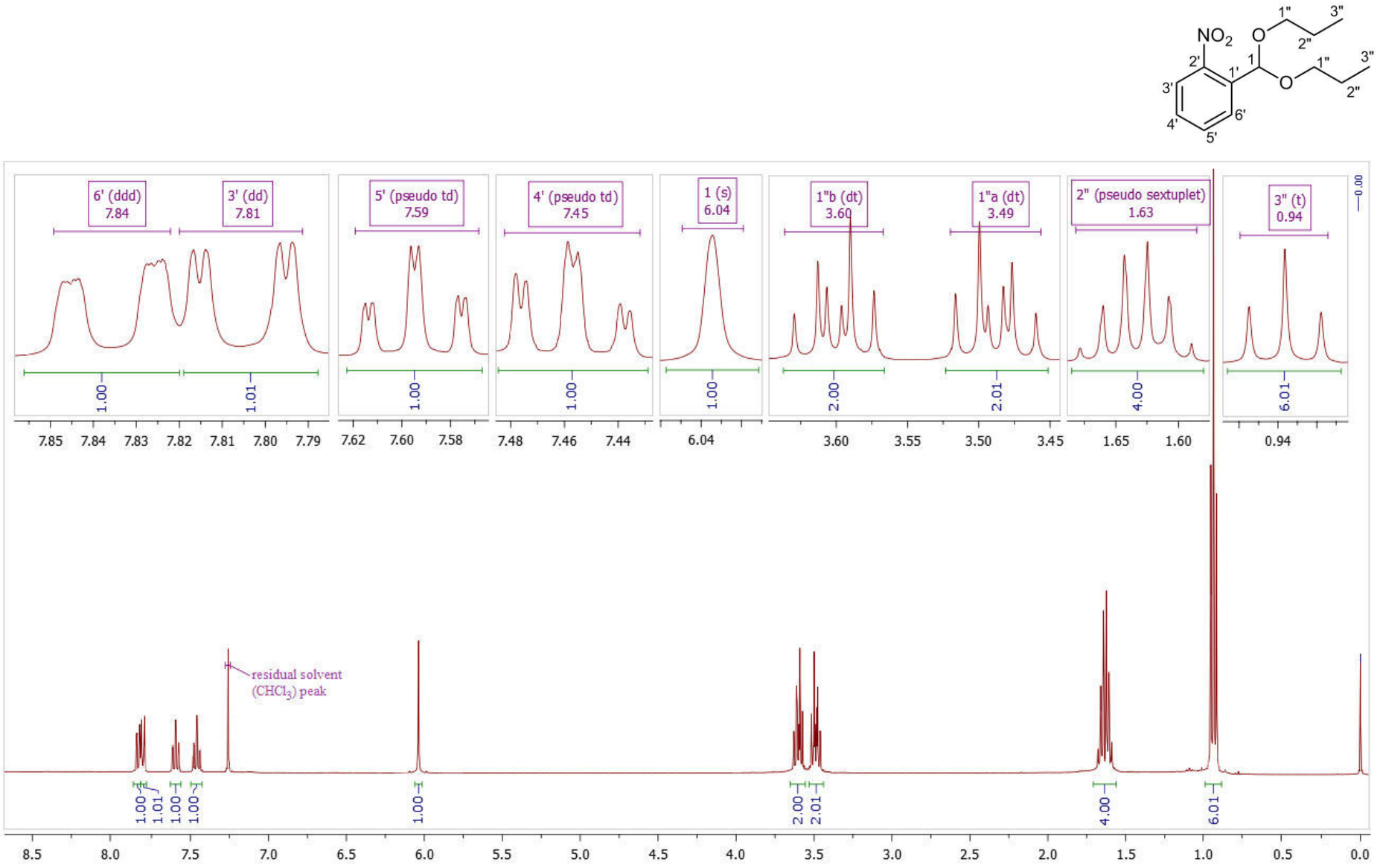
[b]Coupling constants (and multiplicity) were determined from appropriate ^{1}H selective homodecoupled spectra. Values of coupling constants lower than 0.5 Hz were usually observed as a broadening of the corresponding ^{1}H NMR signals; they were disclosed only in a series of selective homodecoupling experiments.



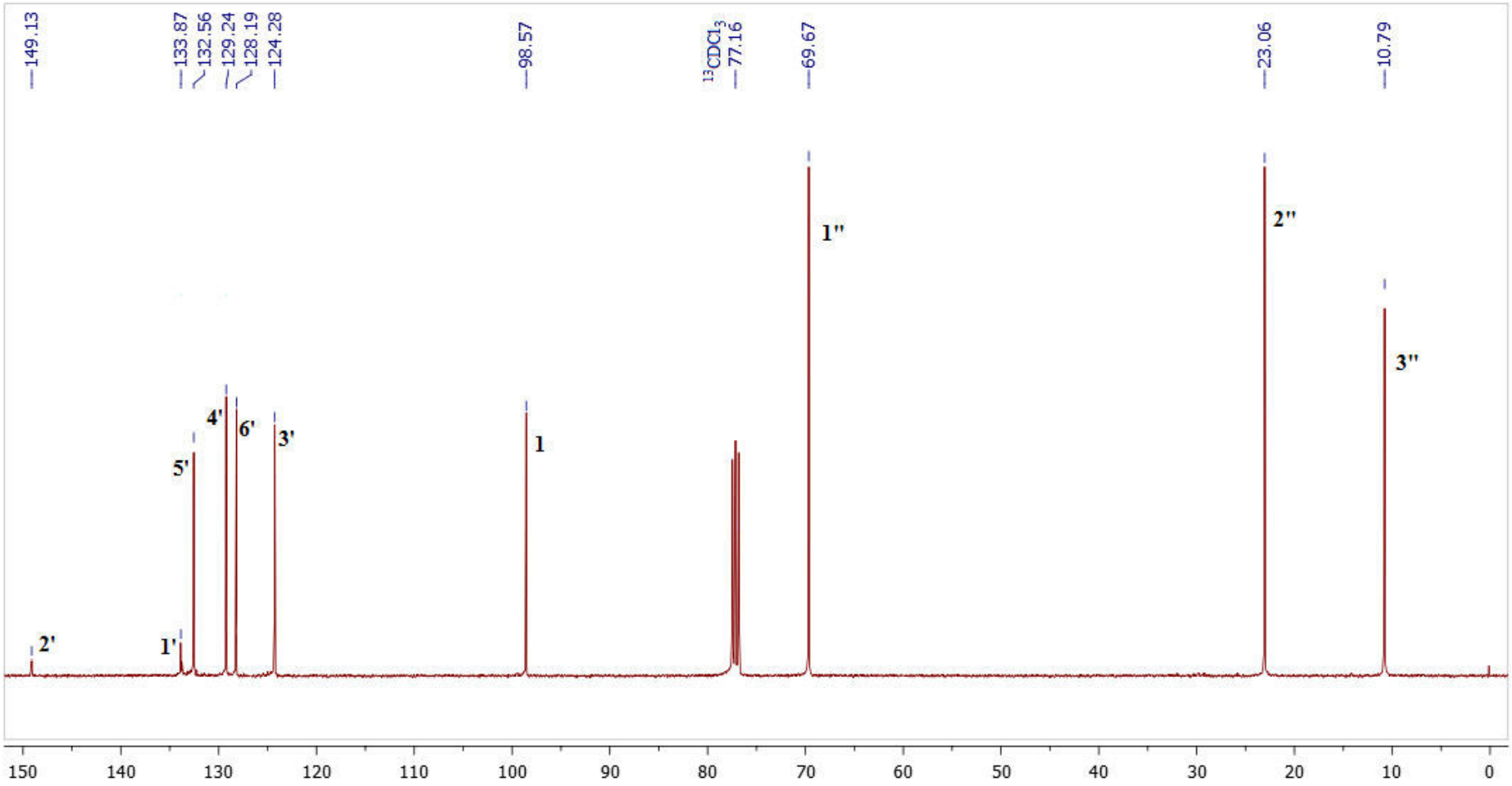
Scheme with key HMBC and NOESY interactions



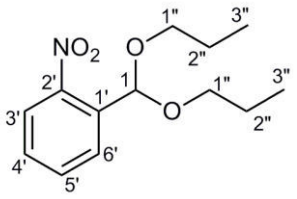
Analysis of ^{1}H-^{1}H coupling constants

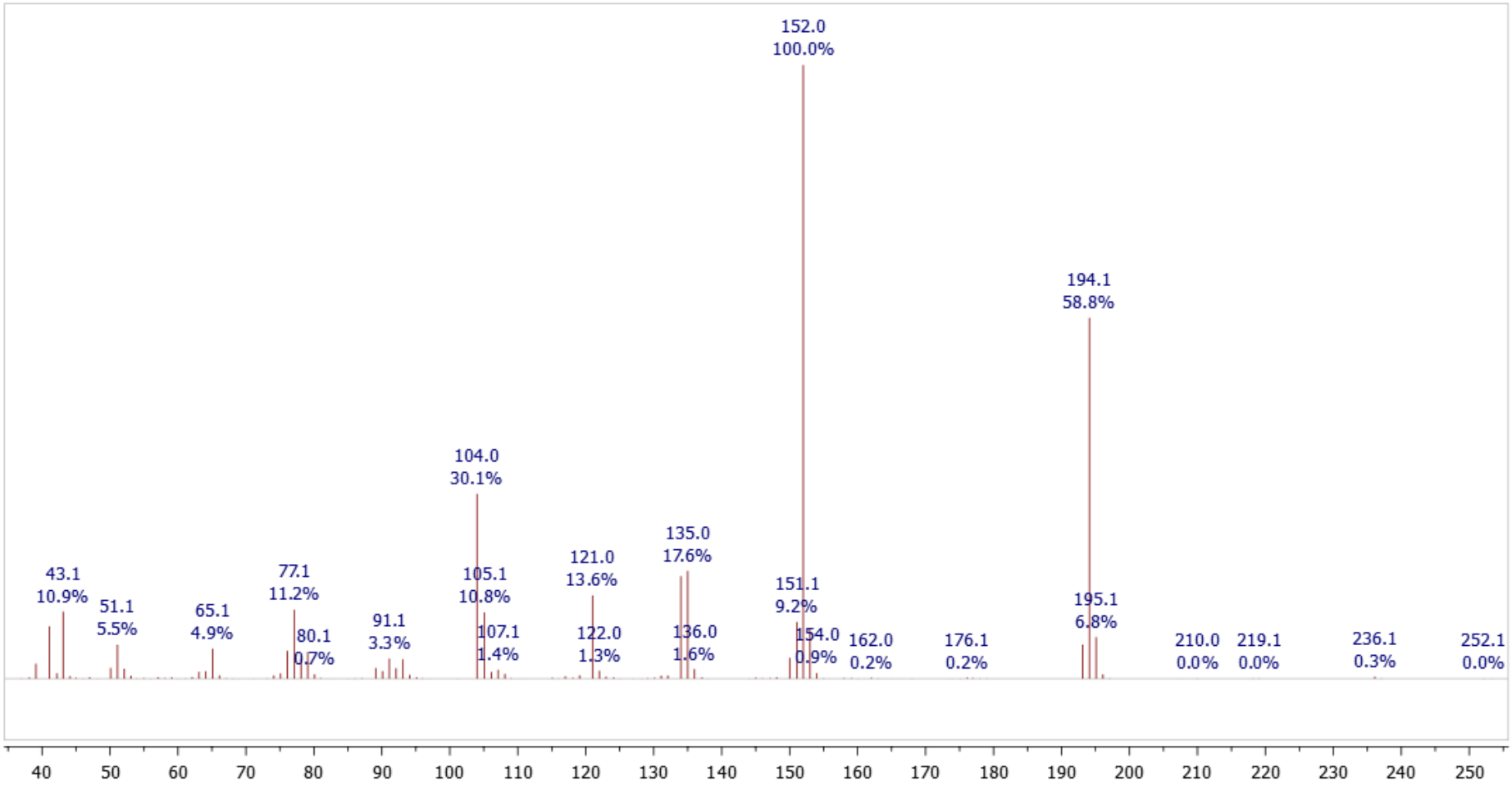


^{1}H-NMR (400 MHz, $CDCl_3$) spectrum of 1-(dipropoxymethyl)-2-nitrobenzene and the corresponding expansions with signal assignment

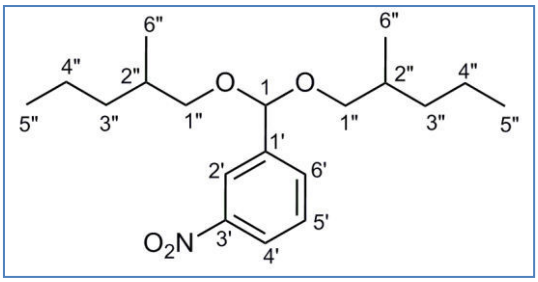


^{13}C-NMR (100.6 MHz, $CDCl_3$) spectrum of 1-(dipropoxymethyl)-2-nitrobenzene with signal assignment





EI-MS spectrum of 1-(dipropoxymethyl)-2-nitrobenzene



1-(bis((2-methylpentyl)oxy)methyl)-3-nitrobenzene

This product was a mixture of 4 diastereomers: two of them enantiomers (compounds 8a and 8b), and two *meso* compounds (9 and 10). These were obtained in an almost statistical ratio, 1 : 2 : 1 (*r-meso* : enantiomeric pair : *s-meso*; see scheme on page 34). ^{1}H NMR signals of these isomers were in a number of cases insufficiently resolved, hence hindering assignation.

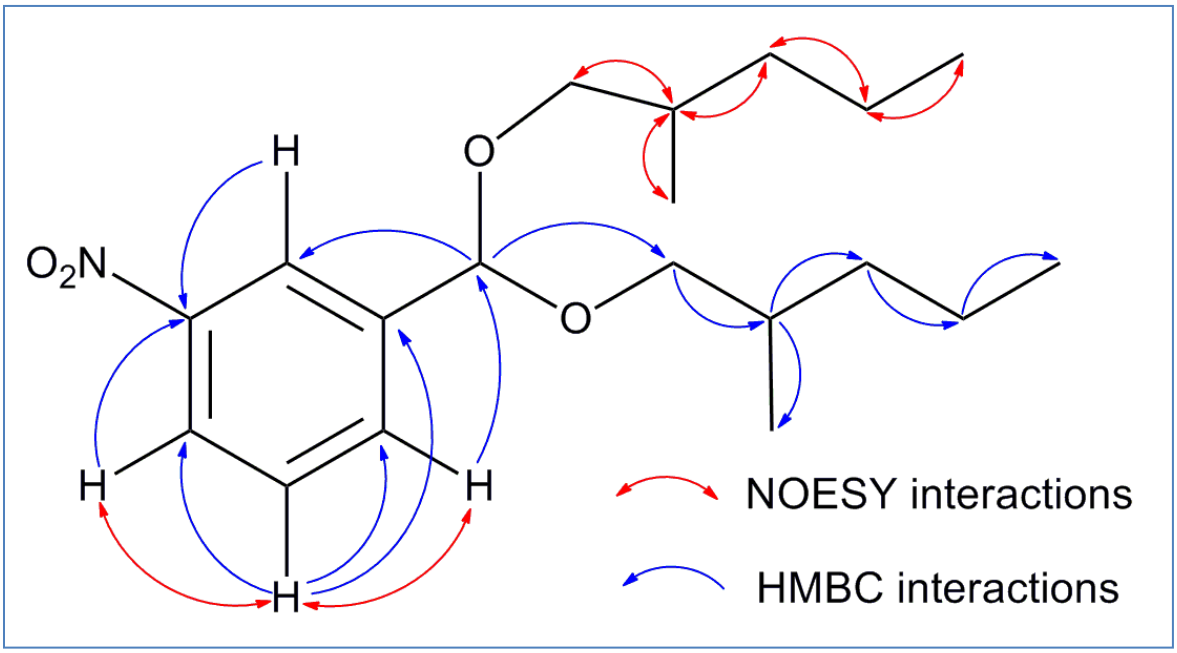
Table of NMR data of 1-(bis((2-methylpentyl)oxy)methyl)-3-nitrobenzene (chemical shift and coupling constant data from ^{1}H and ^{13}C NMR spectra with the observed HMBC and NOESY interactions).

Position	δ_H (Integral, m, *J* (Hz))	δ_C (ppm) C-13 {H}	HMBC[a]	NOESY
1	5.57/5.58 (1 H, ddd, $^{4}J_{1,6'}$ = 0.6, $^{4}J_{1,2'}$ = 0.5, $^{6}J_{1,4'}$ = 0.3)[b]	100.1/100.2/ /100.4 (1 C)	2', 6', 1"	/
1'	/	141.6 (1 C)	/	/
2'	8.35 (1 H, dddd, $^{4}J_{2',4'}$ = 2.3, $^{4}J_{2',6'}$ = 1.5, $^{4}J_{1,2'}$ = 0.5, $^{5}J_{2',5'}$ = 0.2)[b]	122.1 (1 C)	1, 3', 4', 6'	/
3'	/	148.4 (1 C)	/	/
4'	8.19 (1 H, dddd, $^{3}J_{4',5'}$ = 8.2, $^{4}J_{2',4'}$ = 2.3, $^{4}J_{4',6'}$ = 1.0, $^{4}J_{1,4'}$ = 0.3)[b]	123.3 (1 C)	2', 3', 6'	5'
5'	7.55 (1 H, ddd, $^{3}J_{4',5'}$ = 8.2, $^{3}J_{5',6'}$ = 7.7, $^{5}J_{2',5'}$ = 0.2)[b]	129.2 (1 C)	1', 3', 4', 6'	4', 6'
6'	7.81 (1 H, dddd, $^{3}J_{5',6'}$ = 7.7, $^{4}J_{2',6'}$ = 1.5, $^{4}J_{4',6'}$ = 1.0, $^{4}J_{1,6'}$ = 0.5)[b]	133.1 (1 C)	1, 2', 3', 4'	5'
1"a	3.37/3.39 (2 H, dd, $^{2}J_{1"a,1"b}$ = 9.4, $^{3}J_{1"a,2"}$ = 5.8)[b]	70.9/71.1 (2 C)	1, 2", 3", 6"	2"
1"b	3.26/3.29 (2 H, dd, $^{2}J_{1"a,1"b}$ = 9.4, $^{3}J_{1"b,2"}$ = 6.8)[b]			
2"	1.77 (4 H, dtqd, $^{3}J_{1"b,2"}$ = 6.8, $^{3}J_{2",3"}$ = 6.7, $^{3}J_{2",6"}$ = 6.4, $^{3}J_{1"a,2"}$ = 5.8)	33.3 (2 C)	1", 3", 4", 6"	1"ab, 3"ab, 6"
3"a	1.12[c]	36.0 (2 C)	1", 2", 4", 6"	2", 3"b
3"b	1.40[c]		1", 2", 4", 6"	2", 4"a, 3"a
4"a	1.27[c]	20.2 (2 C)	2", 3", 5"	3"b, 5"
4"b	1.37[c]		2", 3", 5"	5"
5"	0.89/0.90 (6 H, t, $^{3}J_{4",5"}$ = 7.1)	14.4 (2 C)	3", 4"	4"ab
6"	0.95 (6 H, d, $^{3}J_{2",6"}$ = 6.4)	17.4 (2 C)	1", 2", 3"	2"

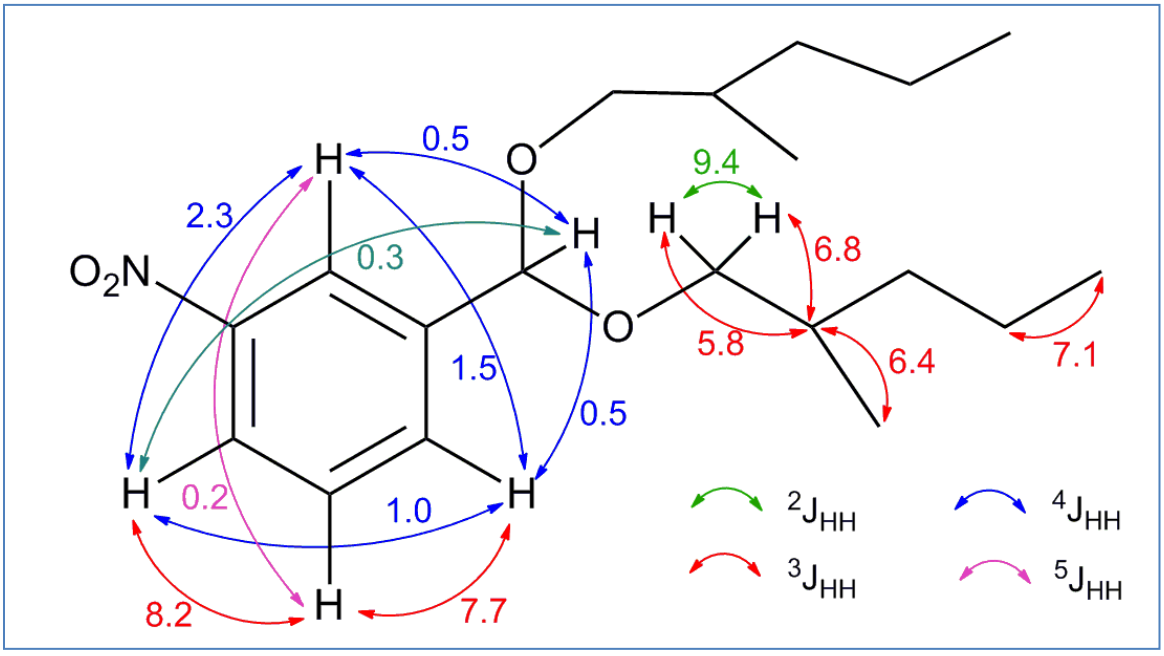
[a]Correlation between the hydrogen in this row and the carbon in the listed position.

[b]Coupling constants (and multiplicity) were determined from appropriate ^{1}H selective homodecoupled spectra. Values of coupling constants lower than 0.5 Hz were usually observed as a broadening of the corresponding ^{1}H NMR signals; they were disclosed only in a series of selective homodecoupling experiments.

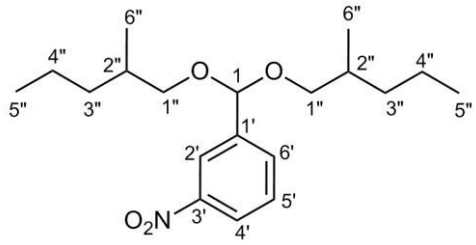
[c]Overlapped signals (range: 1.06-1.46 ppm, 8 H). Chemical shifts were determined from HSQC and HMBC spectra.



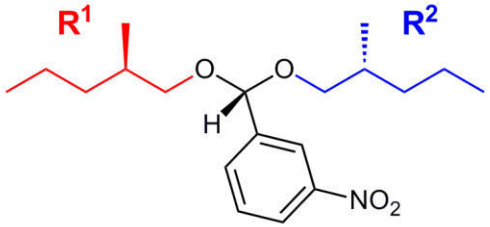
Scheme with key HMBC and NOESY interactions



Analysis of ^{1}H-^{1}H coupling constants



Group 1 (G1)
$8aR^1 = 8bS^2 \approx 10R = 10S$



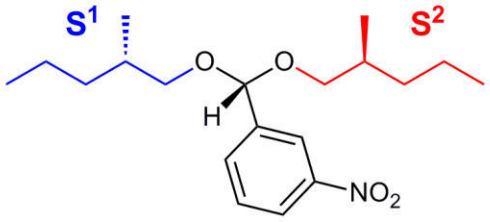
8a

1-(bis(((*R*)-2-methylpentyl)oxy)methyl)-3-nitrobenzene

Group 1 (G1)
$8aR^2 = 8bS^1 \approx 9R = 9S$

9

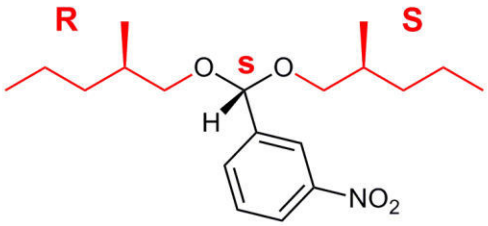
1-((*r*)-(((*R*)-2-methylpentyl)oxy)(((*S*)-2-methylpentyl)oxy)methyl)-3-nitrobenzene



8b

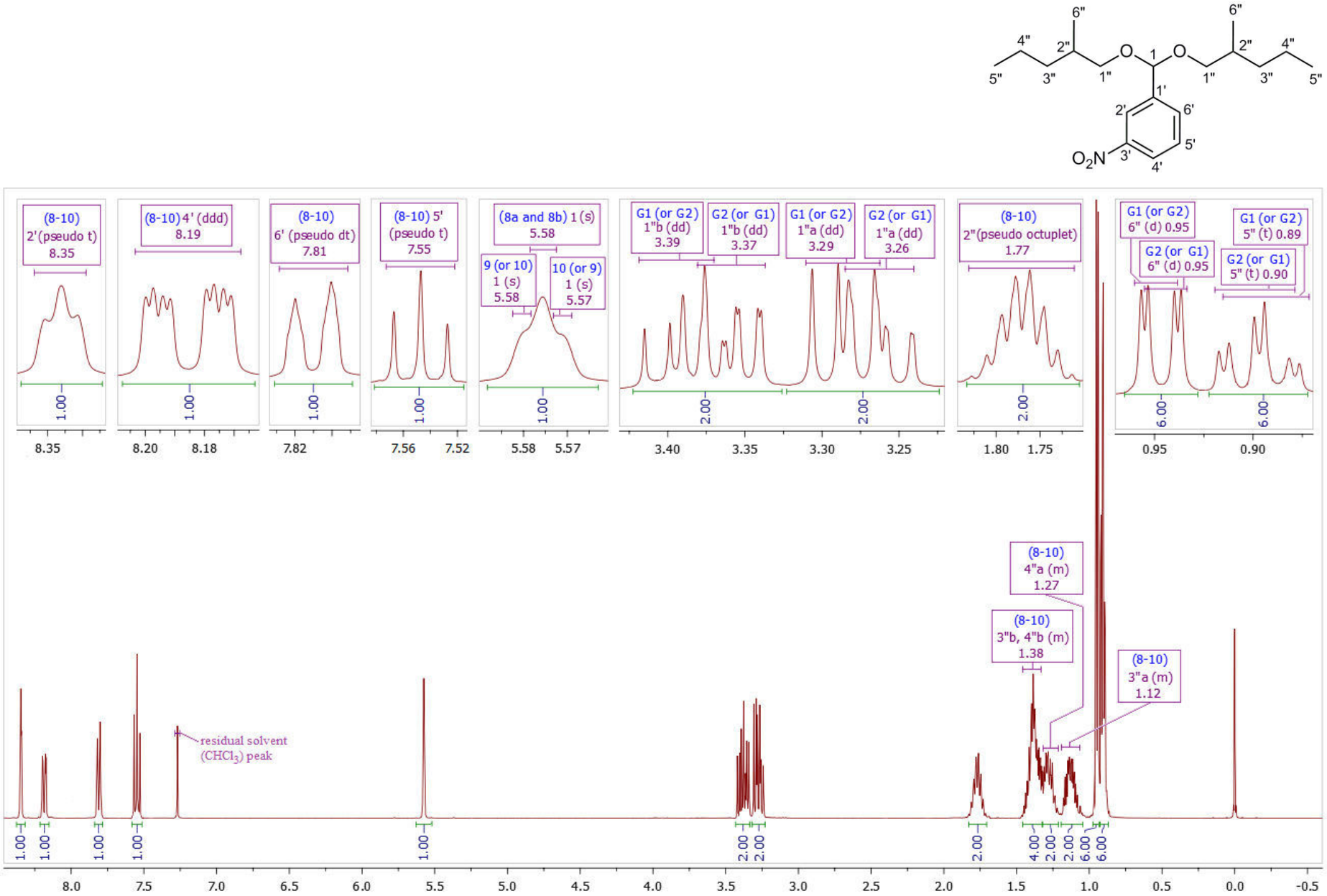
1-(bis(((*S*)-2-methylpentyl)oxy)methyl)-3-nitrobenzene

10

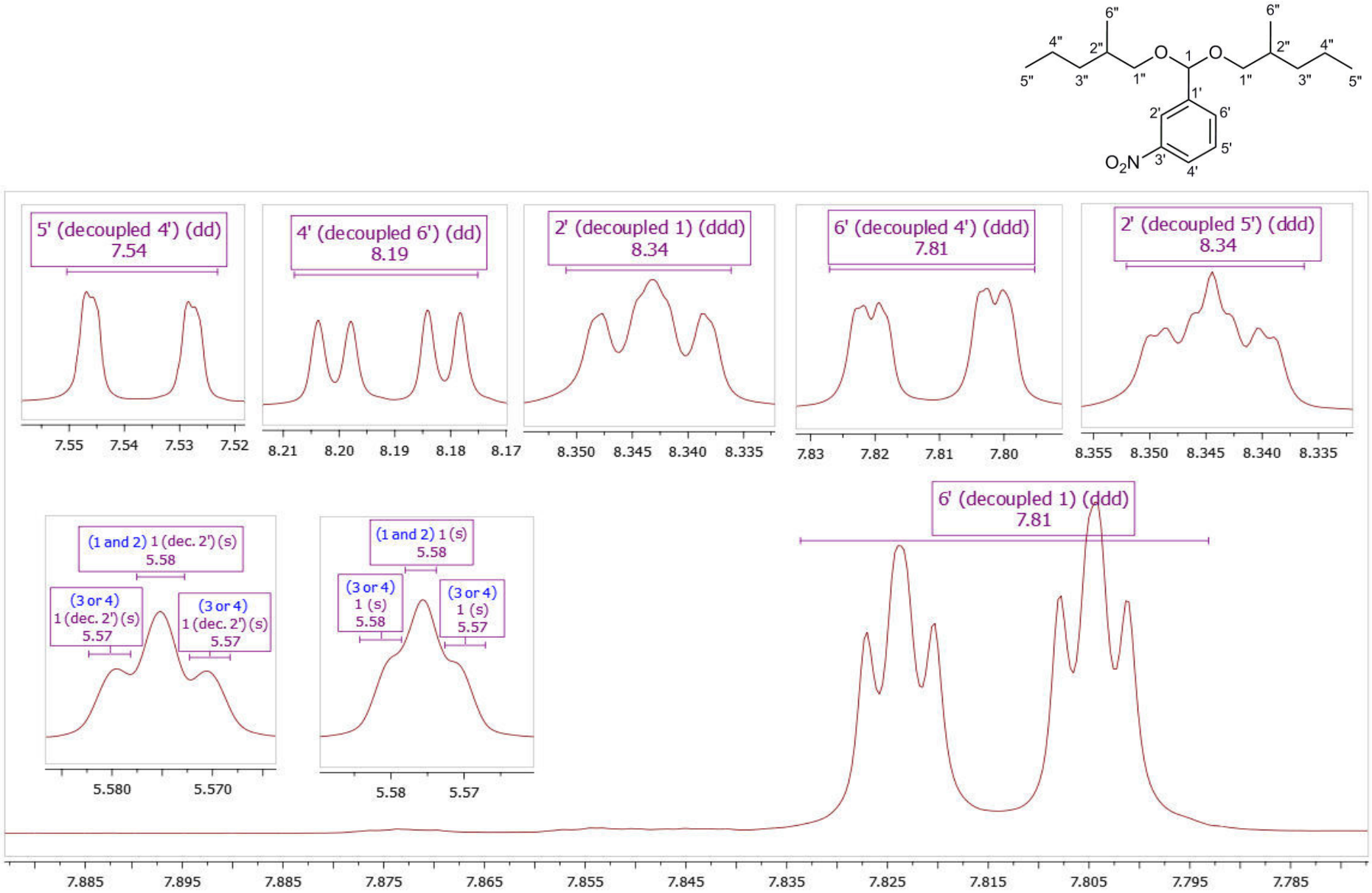


1-((*s*)-(((*R*)-2-methylpentyl)oxy)(((*S*)-2-methylpentyl)oxy)methyl)-3-nitrobenzene

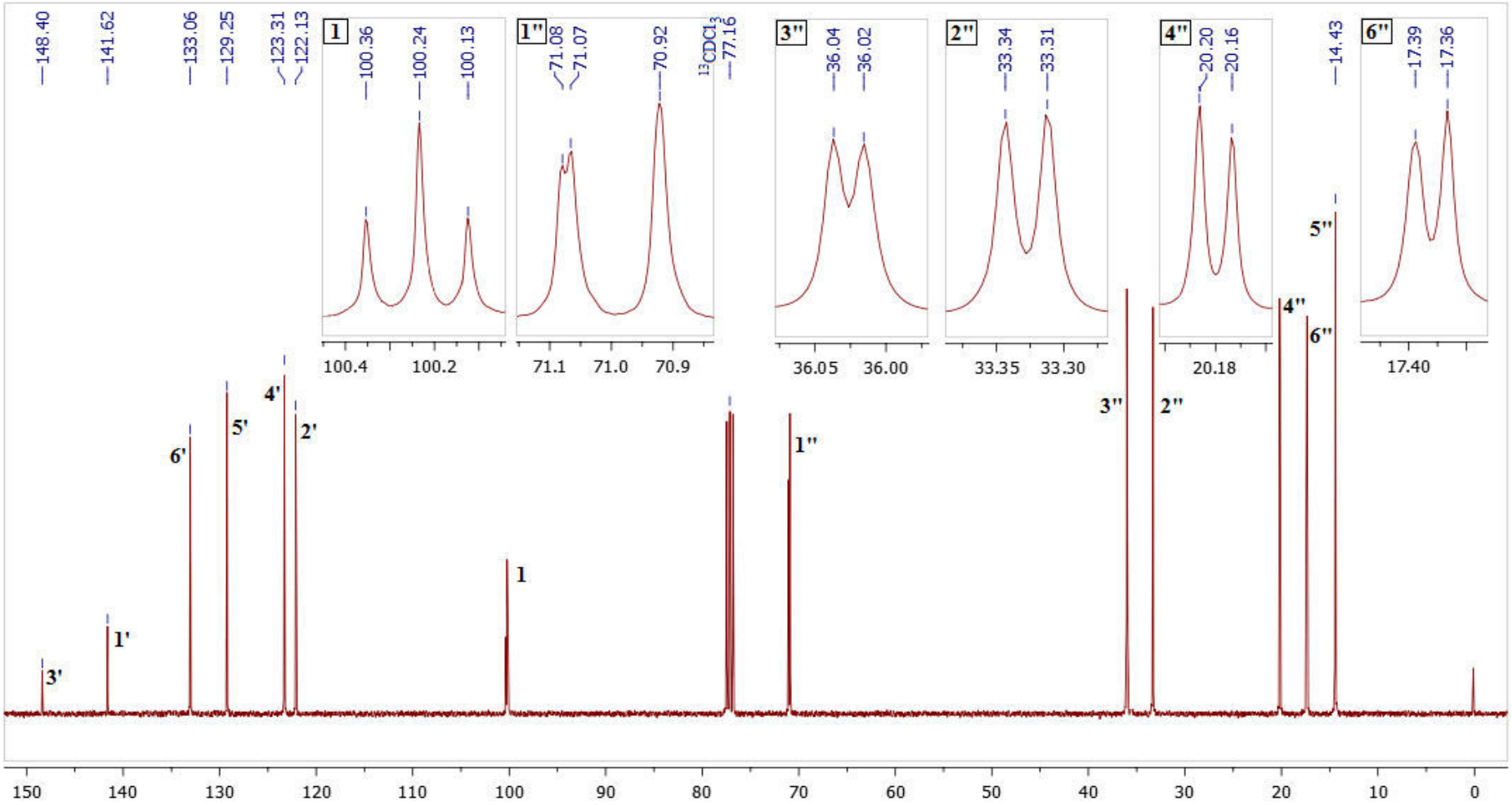
Possible stereoisomers of 1-(bis((2-methylpentyl)oxy)methyl)-3-nitrobenzene. Designations used for assignation of signals in spectra.



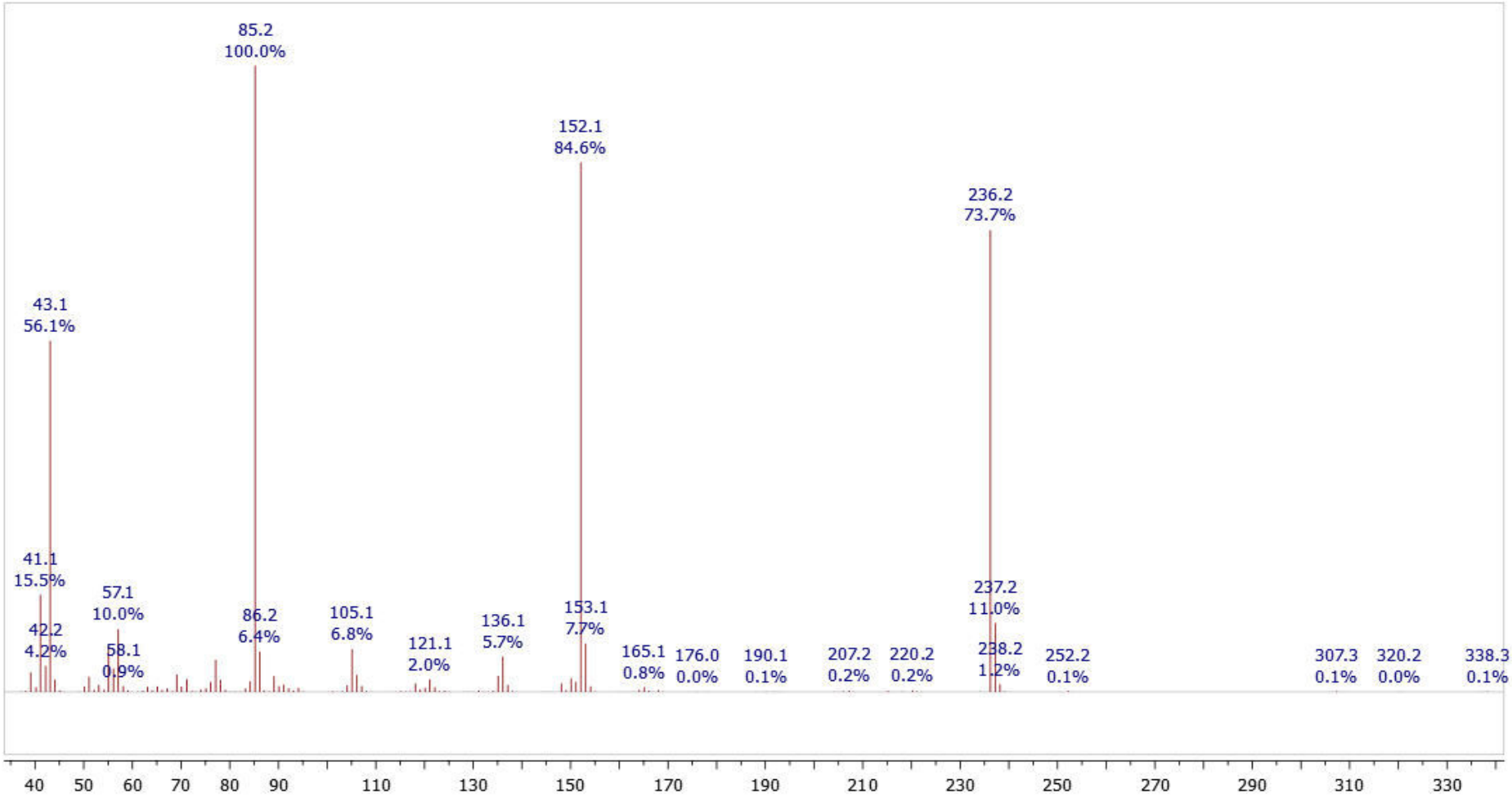
^{1}H-NMR (400 MHz, $CDCl_3$) spectrum of 1-(bis((2-methylpentyl)oxy)methyl)-3-nitrobenzene and the corresponding expansions with signal assignment



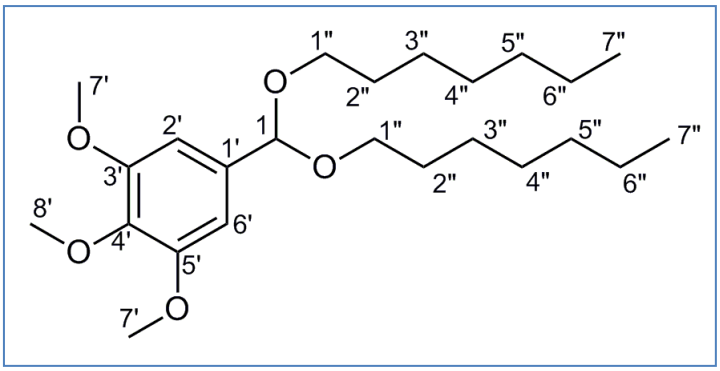
^{1}H NMR spectra obtained in a series of ^{1}H selective homodecoupling experiments with signal assignment



^{13}C-NMR (100.6 MHz, $CDCl_3$) spectrum of 1-(bis((2-methylpentyl)oxy)methyl)-3-nitrobenzene with signal assignment and the corresponding expansions



EI-MS spectrum of 1-(bis((2-methylpentyl)oxy)methyl)-3-nitrobenzene



5-(bis(heptyloxy)methyl)-1,2,3-trimethoxybenzene

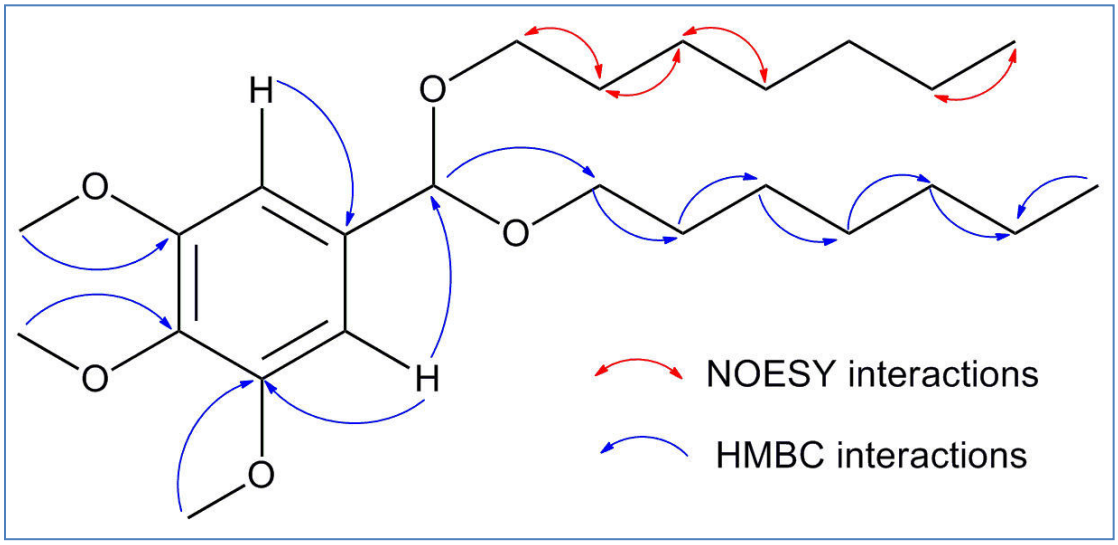
Table of NMR data of 5-(bis(heptyloxy)methyl)-1,2,3-trimethoxybenzene (chemical shift and coupling constant data from ^{1}H and ^{13}C NMR spectra with the observed HMBC and NOESY interactions)

Position	δ_H (Integral, m, *J* (Hz))	δ_C (ppm) C-13 {H}	HMBC[a]	NOESY
1	5.42 (1 H, t, $^{4}J_{1,2'} = {}^{4}J_{1,6'} = 0.5$)[b]	101.8 (1 C)	2', 6', 1"	/
1'	/	135.0 (1 C)	/	/
2', 6'	6.71 (2 H, d, $^{4}J_{1,2'/6'} = 0.5$)[b]	103.5 (2 C)	1, 1', 3', 5', 4'	/
3', 5'	/	137.7 (2 C)	/	/
4'	/	153.2 (1 C)	/	/
7'	3.87 (6 H, s)	56.1 (1 C)	4'	/
8'	3.84 (3 H, s)	61.0 (1 C)	3'	/
1"a	3.49 (2 H, dt, $^{2}J_{1"a,1"b} = 9.4$, $^{3}J_{1"a,2"} = 6.6$)	65.9 (2 C)	1, 2", 3"	2"
1"b	3.57 (2 H, dt, $^{2}J_{1"a,1"b} = 9.4$, $^{3}J_{1"b,2"} = 6.7$)		1, 2", 3"	2"
2"	1.57 (4 H, tdd, $^{3}J_{2",3"} = 6.9$, $^{3}J_{1"b,2"} = 6.7$, $^{3}J_{1"a,2"} = 6.6$)[b]	29.9 (2 C)	1"a, 1"a, 3", 4"	1"a, 1"b, 3"
3"	1.37[c]	26.4 (2 C)	4", 2"	2", 4"
4"	1.30[c]	29.3 (2 C)	5"	3"
5"	1.28[c]	32.0 (2 C)	6"	/
6"	1.29[c]	22.8 (2 C)	7"	7"
7"	0.88 (6 H, t, $^{3}J_{6",7"} = 6.9$)	14.2 (2 C)	5", 6"	6"

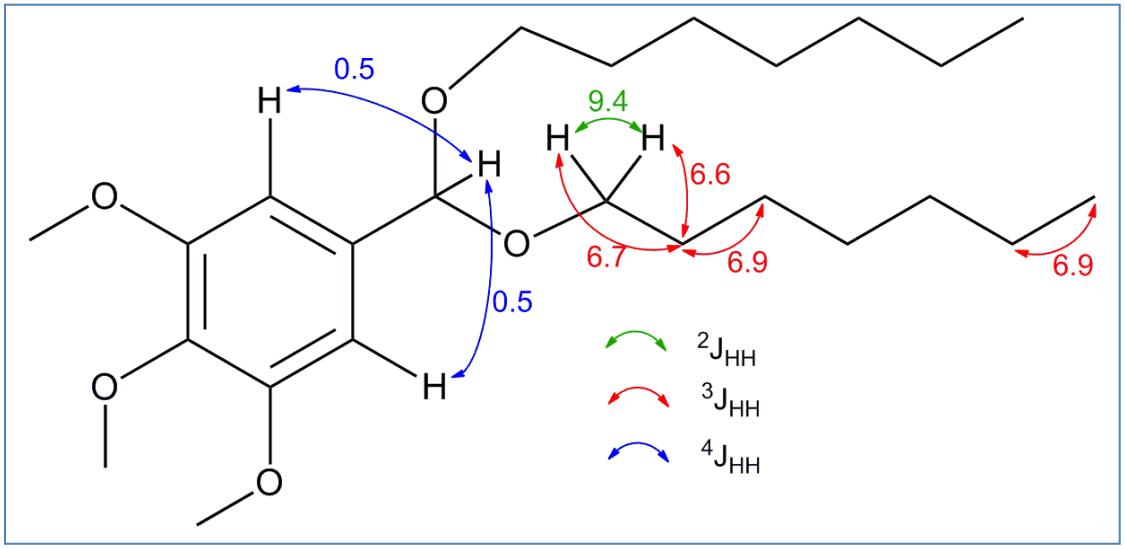
[a]Correlation between the hydrogen in this row and the carbon in the listed position.

[b]Coupling constants (and multiplicity) were determined from appropriate ^{1}H selective homodecoupled spectra. Values of coupling constants lower than 0.5 Hz were usually observed as a broadening of the corresponding ^{1}H NMR signals; they were disclosed only in a series of selective homodecoupling experiments.

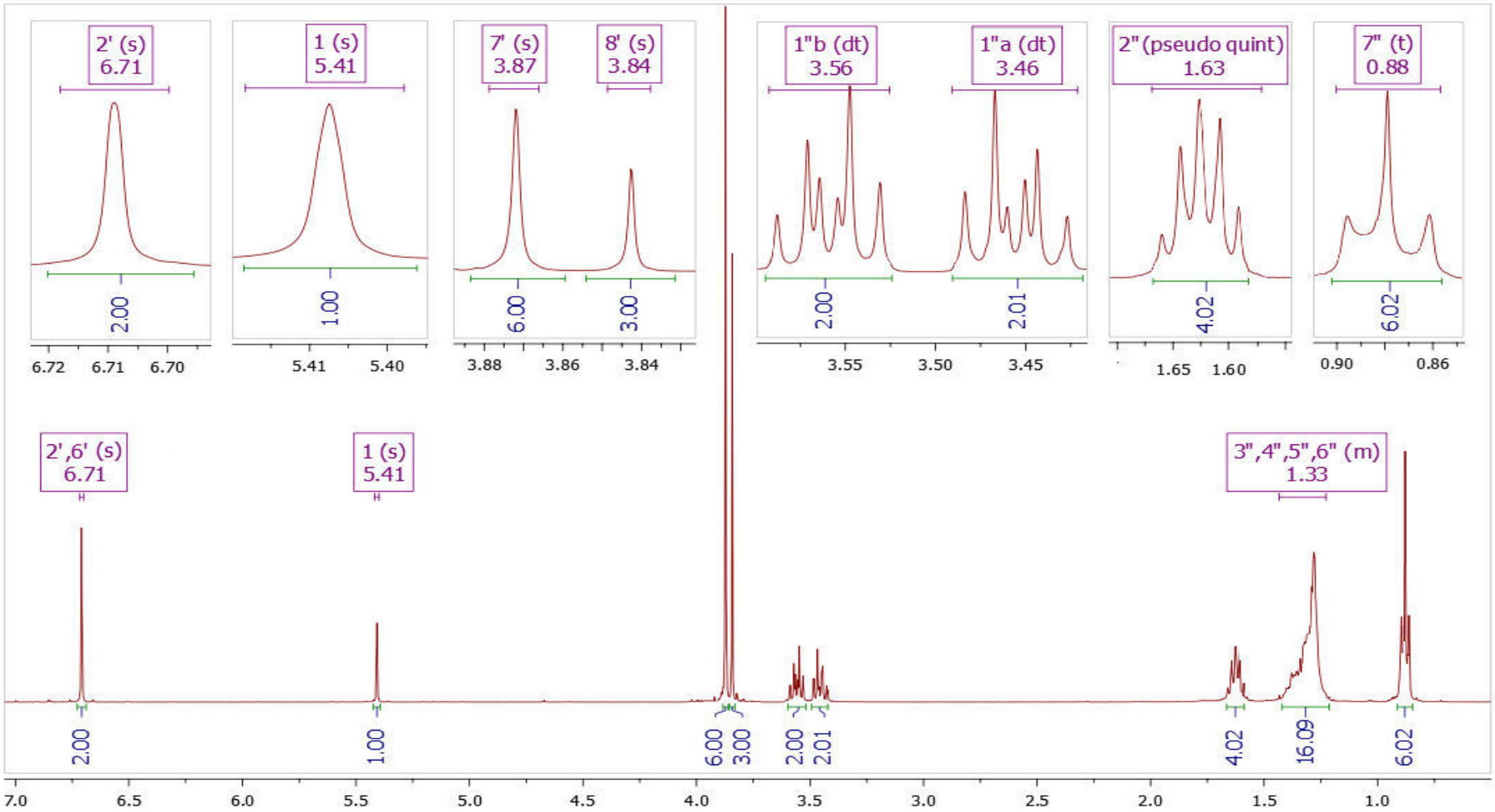
[c]Overlapped signals (range: 1.23-1.42 ppm, 16 H). Chemical shifts were determined from HSQC and HMBC spectra.



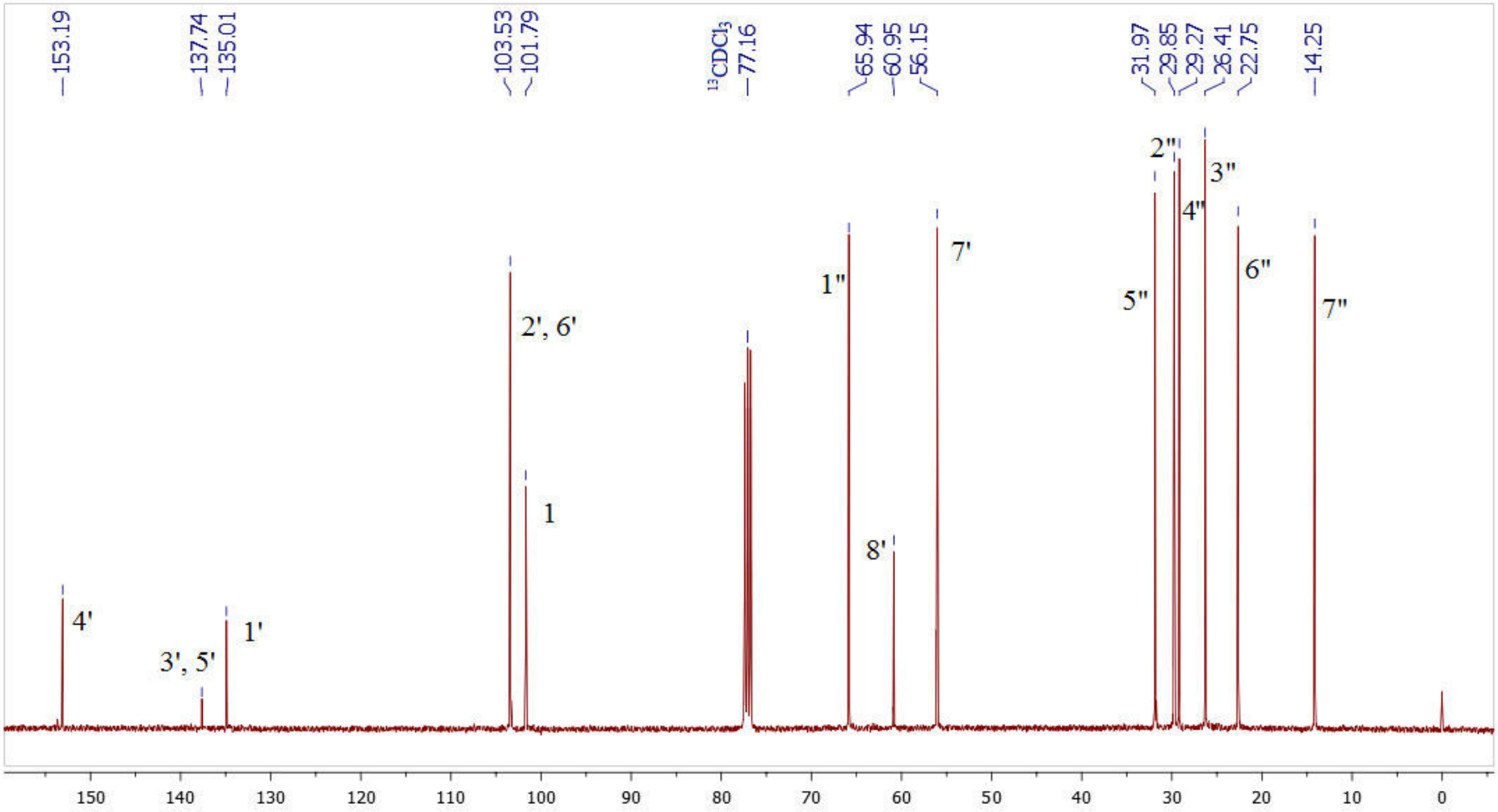
Scheme with key HMBC and NOESY interactions



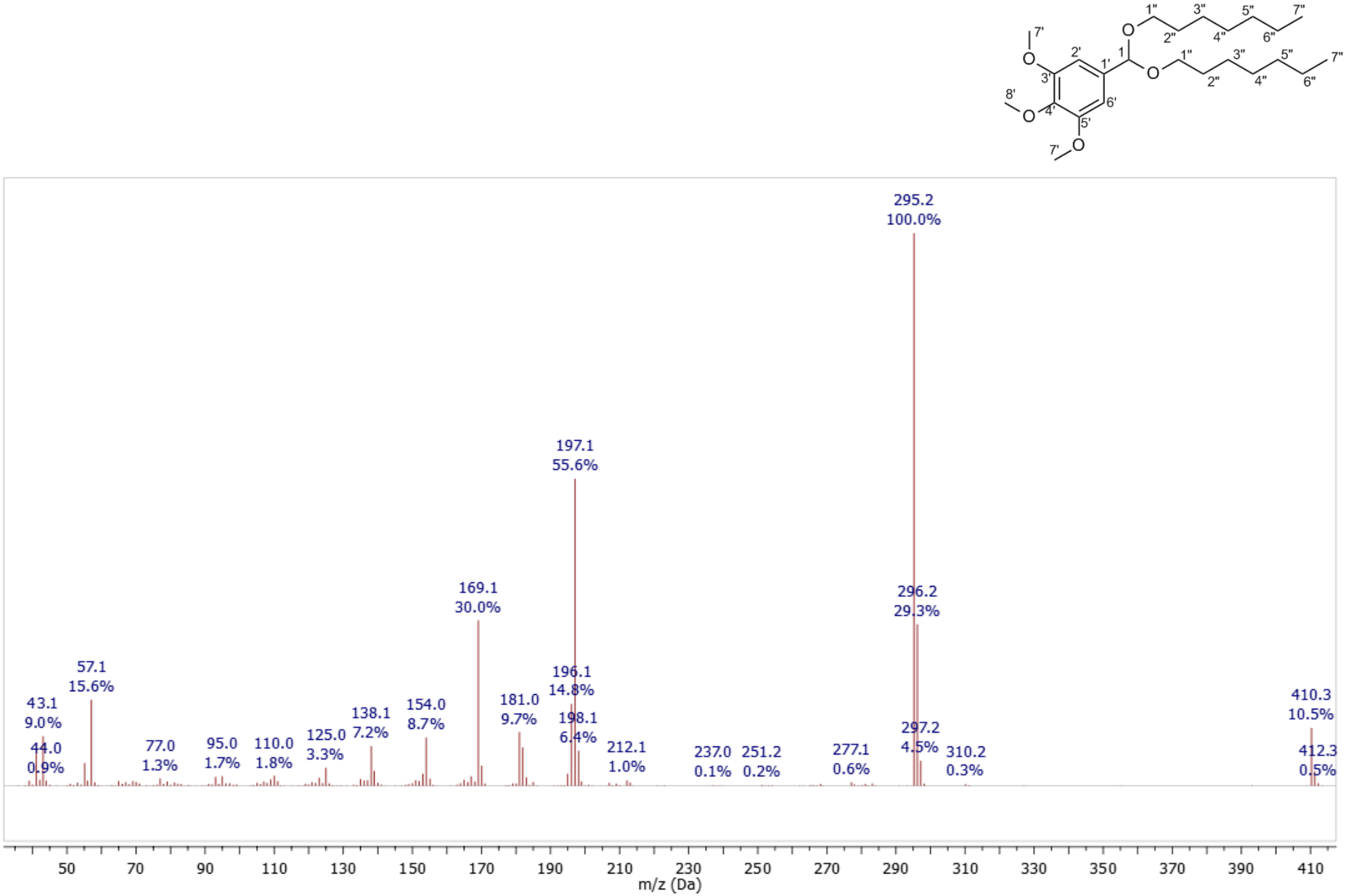
Analysis of ^{1}H-^{1}H coupling constants



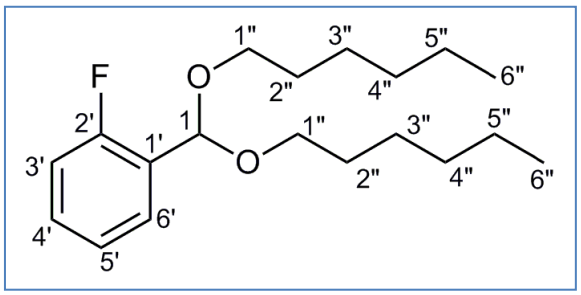
^{1}H-NMR (400 MHz, $CDCl_3$) spectrum of 5-(bis(heptyloxy)methyl)-1,2,3-trimethoxybenzene and the corresponding expansions with signal assignment



^{13}C-NMR (100.6 MHz, $CDCl_3$) spectrum of 5-(bis(heptyloxy)methyl)-1,2,3-trimethoxybenzene with signal assignment



EI-MS spectrum of 5-(bis(heptyloxy)methyl)-1,2,3-trimethoxybenzene



1-(bis(hexyloxy)methyl)-2-fluorobenzene

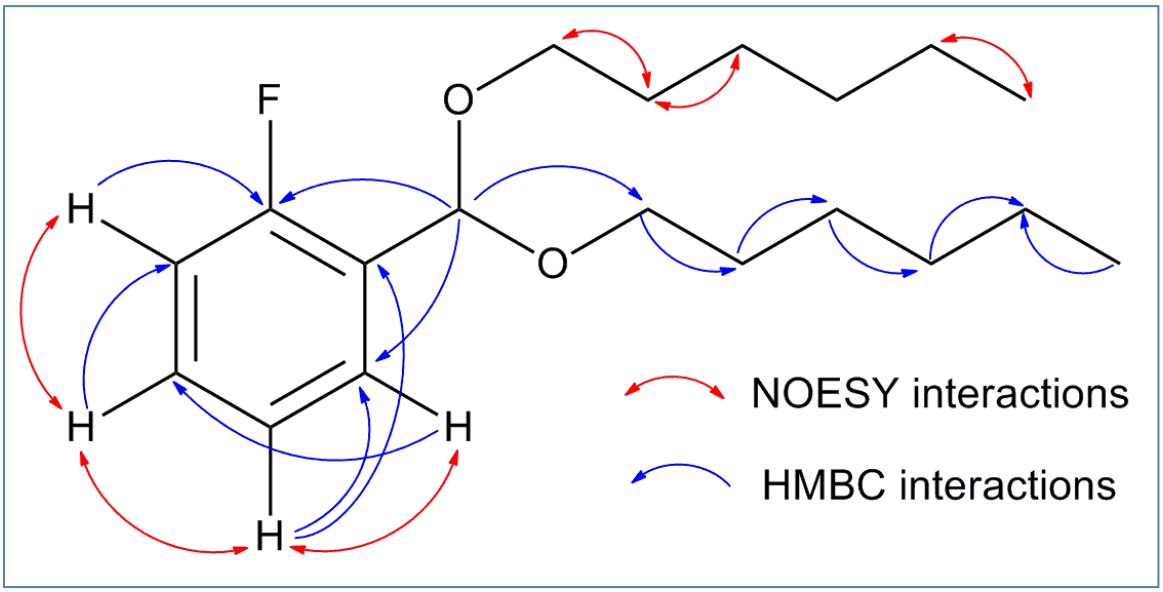
Table of NMR data of 1-(bis(hexyloxy)methyl)-2-fluorobenzene (chemical shift and coupling constant data from ^{1}H and ^{13}C NMR spectra with the observed HMBC and NOESY interactions)

Position	δ_H (Integral, m, *J* (Hz))	δ_C (m, *J* (Hz)), C-13 {H}	HMBC[a]	NOESY
1	5.72 (1 H, tdd, $^{4}J_{1,6'} = {}^{4}J_{1,F} = 0.4$, $^{5}J_{1,5'}$=0.3, $^{6}J_{1,4'}$=0.2)[b]	97.0 (1 C, d, $^{3}J_{CF} = 3.4$)	2', 6', 1"	/
1'	/	126.6 (1 C, d, $^{2}J_{CF} = 12.7$)	/	/
2'	/	160.6 (1 C, d, $^{1}J_{CF} = 248.1$)	/	/
3'	7.03 (1 H, dddd, $^{3}J_{3',F} = 10.2$, $^{3}J_{3',4'}$ =8.2, $^{4}J_{3',5'} = 1.0$, $^{5}J_{3',6'} = 0.2$)[b]	115.4 (1 C, d, $^{2}J_{CF} = 21.6$)	1', 2', 5'	4'
4'	7.29 (1 H, ddddd, $^{3}J_{3',4'}$ =8.2, $^{3}J_{4',5'} = 7.3$, $^{4}J_{4',F} = 5.3$, $^{4}J_{4',6'} = 1.8$, $^{6}J_{1,4'}$=0.2)[b]	130.0 (1 C, d, $^{3}J_{CF} = 8.2$)	2', 3', 6'	3', 5'
5'	7.14 (1 H, dddd, $^{3}J_{5',6'} = 7.5$, $^{3}J_{4',5'} = 7.3$, $^{4}J_{3',5'} = 1.0$, $^{5}J_{1,5'} = 0.3$)[b]	123.9 (1 C, d, $^{4}J_{CF} = 3.6$)	1', 3', 6'	4', 6'
6'	7.60 (1 H, ddddd, $^{3}J_{5',6'} = 7.5$, $^{4}J_{6',F'} = 7.1$, $^{4}J_{4',6'} = 1.8$, $^{4}J_{1,6'}$=0.4, $^{5}J_{3',6'}$=0.2)[b]	128.2 (1 C, d, $^{3}J_{CF} = 4.0$)	1, 2', 4'	5'
1"a	3.49 (2 H, dt, $^{2}J_{1"a,1"b} = 9.3$, $^{3}J_{1"a,2"} = 6.6$)	66.7 (2 C, s)	1, 2", 3"	2"
1"b	3.58 (2 H, dt, $^{2}J_{1"a,1"b} = 9.3$, $^{3}J_{1"b,2"} = 6.7$)		1, 2", 3"	2"
2"	1.60 (4 H, tdd, $^{3}J_{2",3"} = 6.9$, $^{3}J_{1"b,2"} = 6.7$, $^{3}J_{1"a,2"} = 6.6$)[b]	29.8 (2 C, s)	1", 3", 4"	1", 3"
3"	1.35[c]	26.0 (2 C, s)	1", 2", 4", 5"	2"
4"	1.27[c]	31.8 (2 C, s)	5", 6"	/
5"	1.30[c]	22.7 (2 C, s)	4", 6"	6"
6"	0.88 (6 H, t, $^{3}J_{6",7"} = 6.9$)	14.2 (2 C, s)	4", 5"	5"

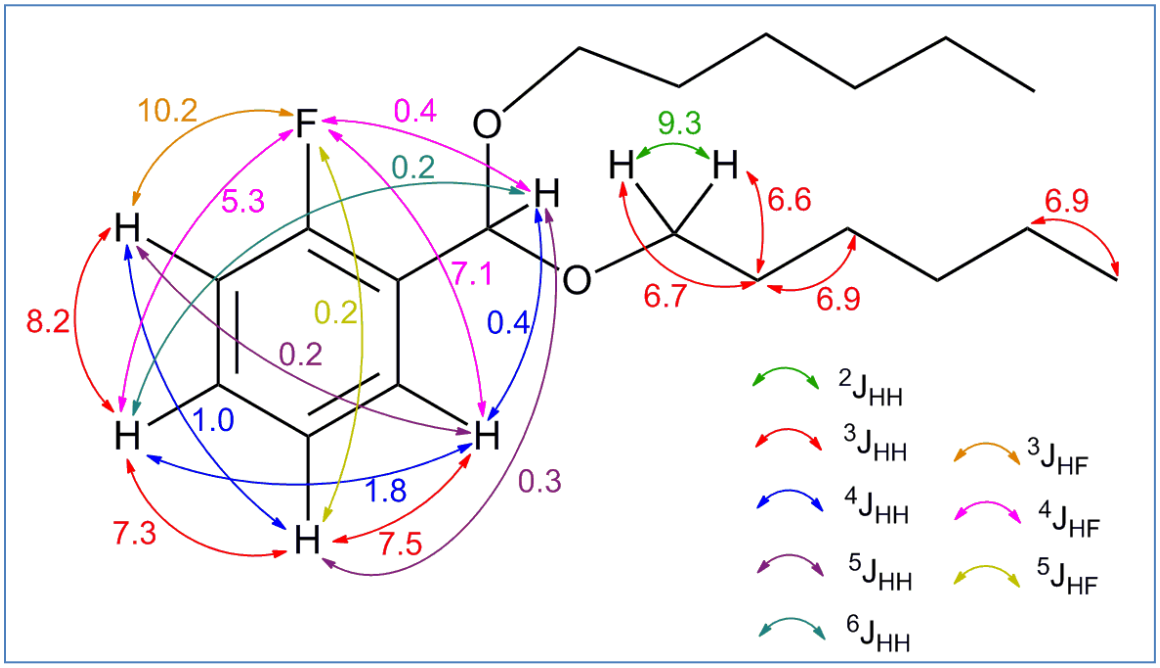
[a]Correlation between the hydrogen in this row and the carbon in the listed position.

[b]Coupling constants (and multiplicity) were determined from appropriate ^{1}H selective homodecoupled spectra. Values of coupling constants lower than 0.5 Hz were usually observed as a broadening of the corresponding ^{1}H NMR signals; they were disclosed only in a series of selective homodecoupling experiments.

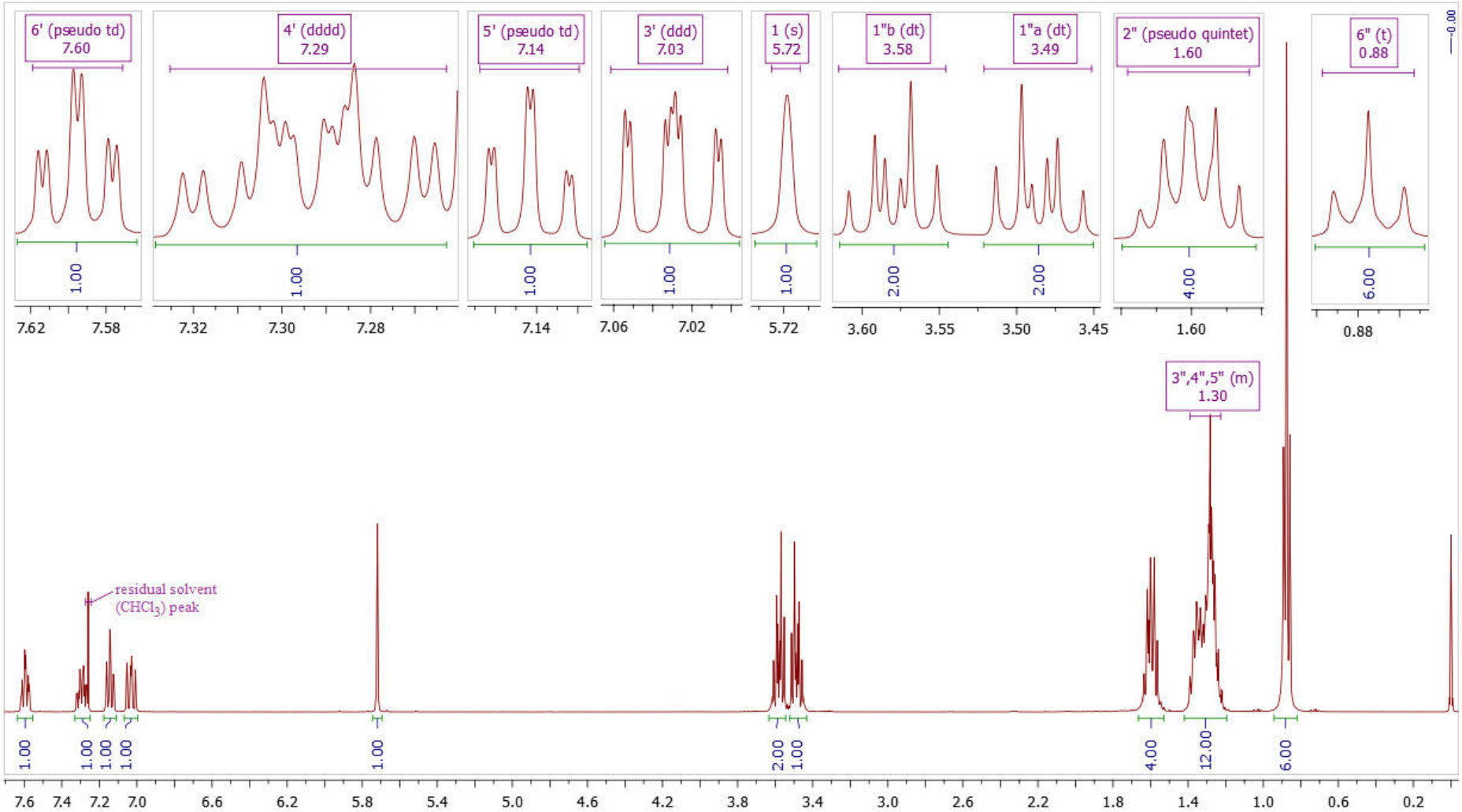
[c]Overlapped signals (range: 1.23-1.43 ppm, 12 H). Chemical shifts were determined from HSQC and HMBC spectra.



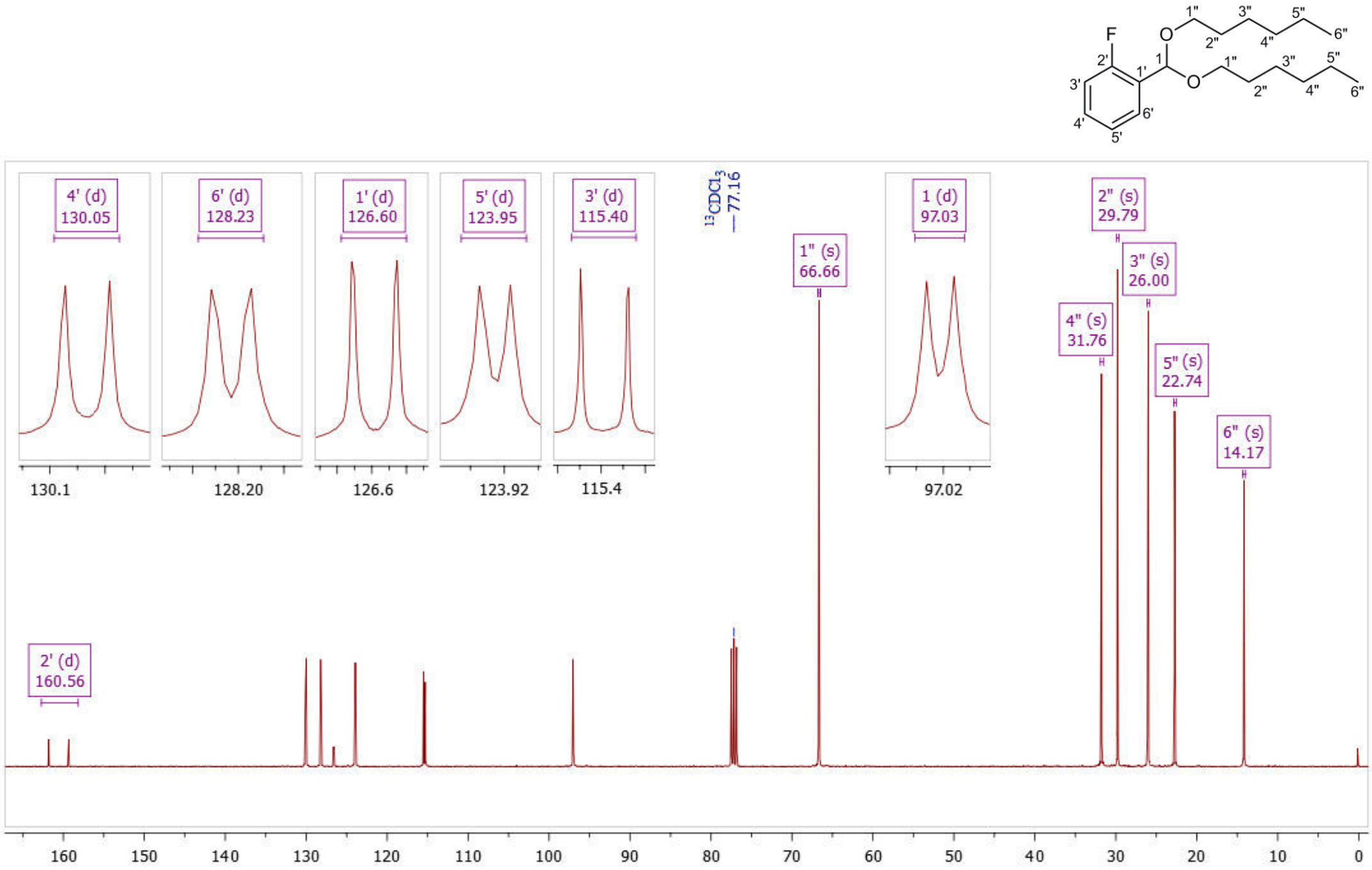
Scheme with key HMBC and NOESY interactions



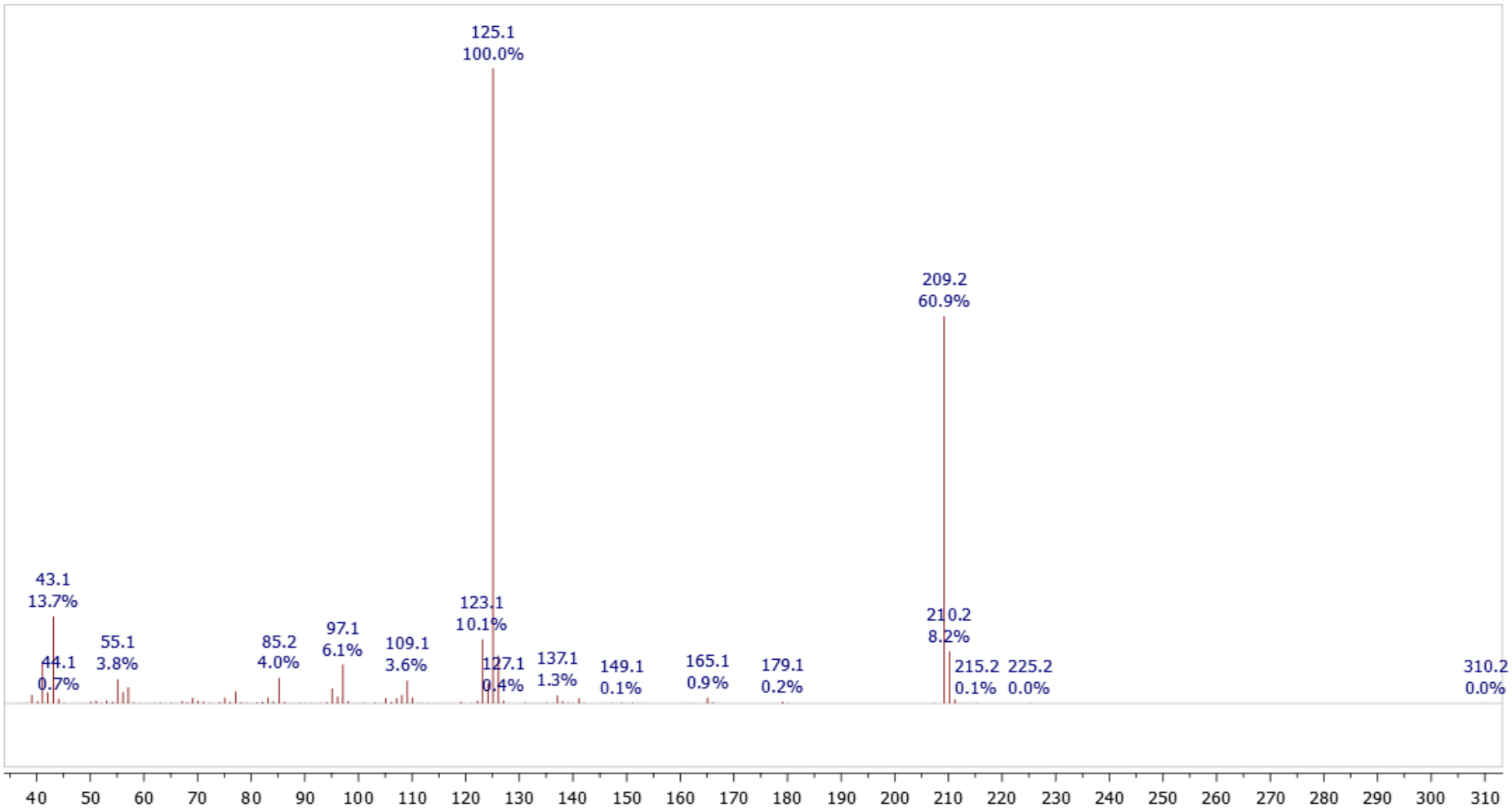
Analysis of ^{1}H-^{1}H coupling constants



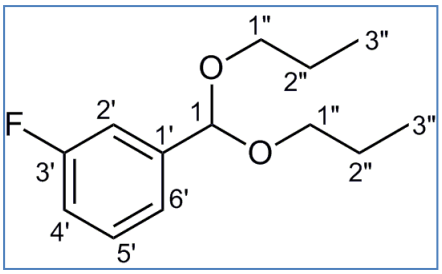
^{1}H-NMR (400 MHz, $CDCl_3$) spectrum of 1-(bis(hexyloxy)methyl)-2-fluorobenzene and the corresponding expansions with signal assignment



^{13}C-NMR (100.6 MHz, $CDCl_3$) spectrum of 1-(bis(hexyloxy)methyl)-2-fluorobenzene and the corresponding expansions with signal assignment



EI-MS spectrum of 1-(bis(hexyloxy)methyl)-2-fluorobenzene



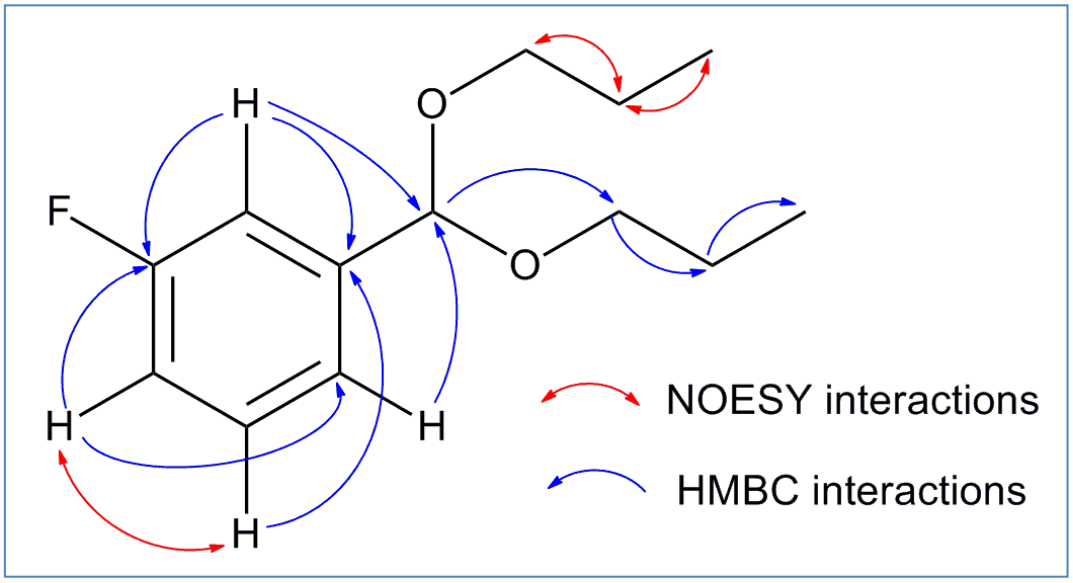
1-(dipropoxymethyl)-3-fluorobenzene

Table of NMR data of 1-(dipropoxymethyl)-3-fluorobenzene (chemical shift and coupling constant data from ^{1}H and ^{13}C NMR spectra with the observed HMBC and NOESY interactions)

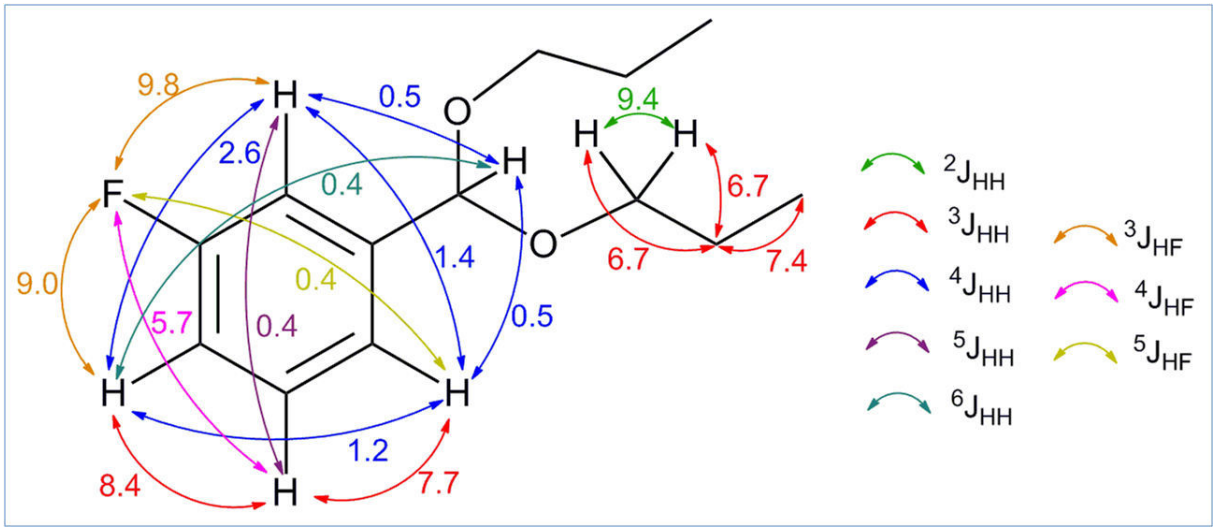
Position	δ_H (Integral, m, *J* (Hz))	δ_C (m, *J* (Hz)), C-13 {H}	HMBC[a]	NOESY
1	5.50 (1 H, td, $^{4}J_{1,2'} = {}^{4}J_{1,6'} = 0.5$, $^{6}J_{1,4'} = 0.4$)[b]	100.7 (1 C, d, $^{4}J_{CF} = 2.0$ Hz)	2', 6', 1"	/
1'	/	142.0 (1 C, d, $^{3}J_{CF} = 6.8$ Hz)	/	/
2'	7.20 (1 H, ddddd, $^{3}J_{2',F} = 9.8$, $^{4}J_{2',4'} = 2.6$, $^{4}J_{2',6'} = 1.4$, $^{4}J_{1,2'} = 0.5$, $^{5}J_{2',5'} = 0.4$)[b]	113.9 (1 C, d, $^{2}J_{CF} = 22.2$ Hz)	1, 1', 3', 6'	/
3'	/	163.0 (1 C, d, $^{1}J_{CF} = 245.5$ Hz)	/	/
4'	7.00 (1 H, ddddd, $^{3}J_{4',F} = 9.0$, $^{3}J_{4',5'} = 8.4$, $^{4}J_{2',4'} = 2.6$, $^{4}J_{4',6'} = 1.0$, $^{6}J_{1,4'} = 0.4$)[b]	115.2 (1 C, d, $^{2}J_{CF} = 21.3$ Hz)	2', 3', 6'	5'
5'	7.32 (1 H, dddd, $J_{4',5'} = 8.4$, $J_{5',6'} = 7.7$, $^{4}J_{5',F} = 5.7$, $^{5}J_{2',5'} = 0.4$)[b]	129.8 (1 C, d, $^{3}J_{CF} = 8.3$ Hz)	1', 3'	4'
6'	7.25 (1 H, ddd, $^{3}J_{5',6'} = 7.7$, $^{4}J_{2',6'} = 1.4$, $^{4}J_{4',6'} = 1.0$, $^{4}J_{1,6'} = 0.5$)[b]	122.5 (1 C, d, $^{4}J_{CF} = 2.8$ Hz)	1, 2', 4'	/
1"a	3.43 (2 H, dt, $^{2}J_{1"a,1"b} = 9.4$, $^{3}J_{1"a,2"} = 6.7$)	67.3 (2 C, s)	1, 1", 2"	2"
1"b	3.50 (2 H, dt, $^{2}J_{1"a,1"b} = 9.4$, $^{3}J_{1"b,2"} = 6.7$)		1, 1", 2"	2"
2"	1.64 (4 H, qt, $^{3}J_{2",3"} = 7.4$, $^{3}J_{1"a,2"} = {}^{3}J_{1"b,2"} = 6.7$)[b]	23.1 (2 C, s)	1", 3"	1", 3"
3"	0.95 (6 H, t, $J_{2",3"} = 7.4$)	10.9 (2 C, s)	1", 2"	2"

[a]Correlation between the hydrogen in this row and the carbon in the listed position.

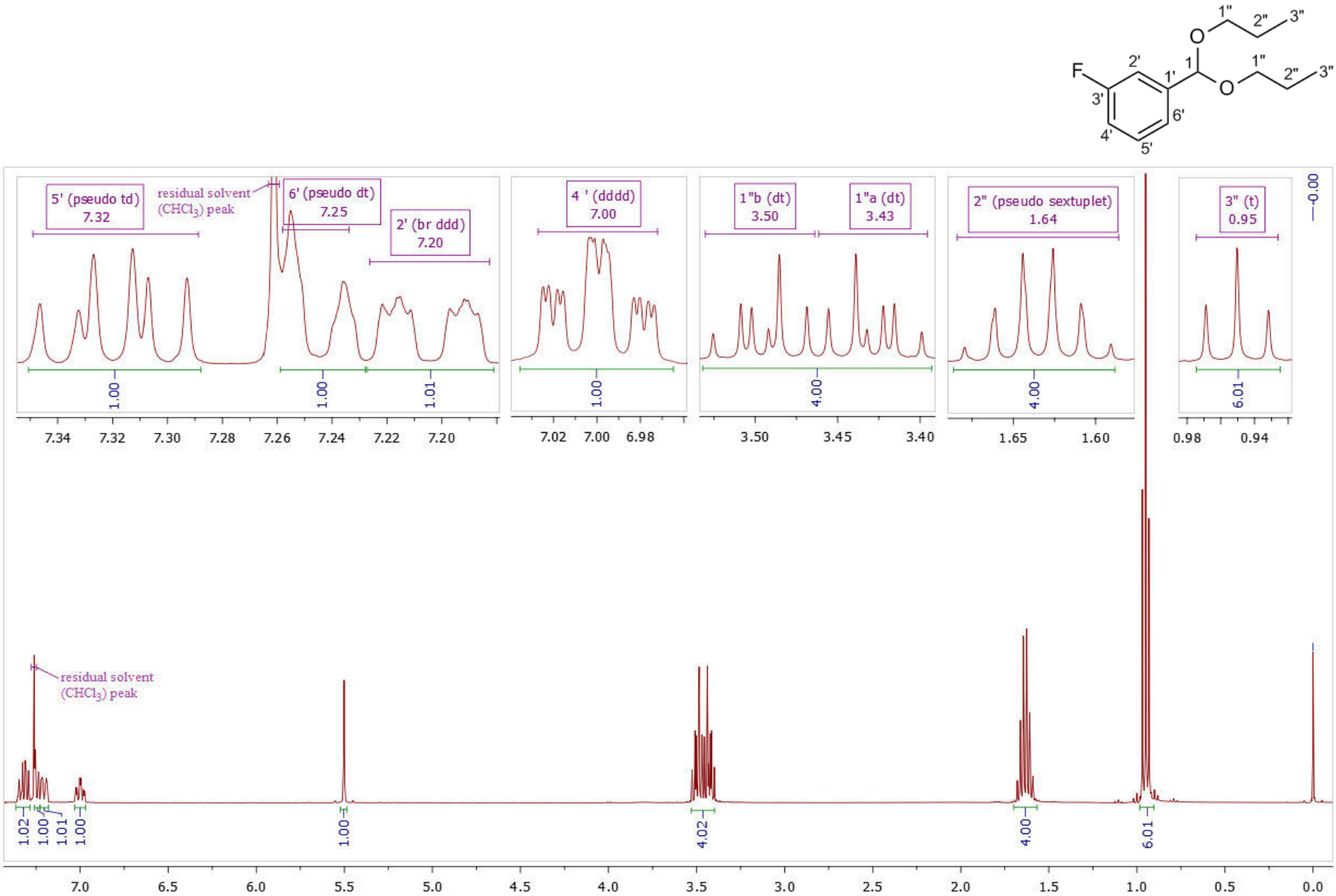
[b]Coupling constants (and multiplicity) were determined from appropriate ^{1}H selective homodecoupled spectra. Values of coupling constants lower than 0.5 Hz were usually observed as a broadening of the corresponding ^{1}H NMR signals; they were disclosed only in a series of selective homodecoupling experiments.



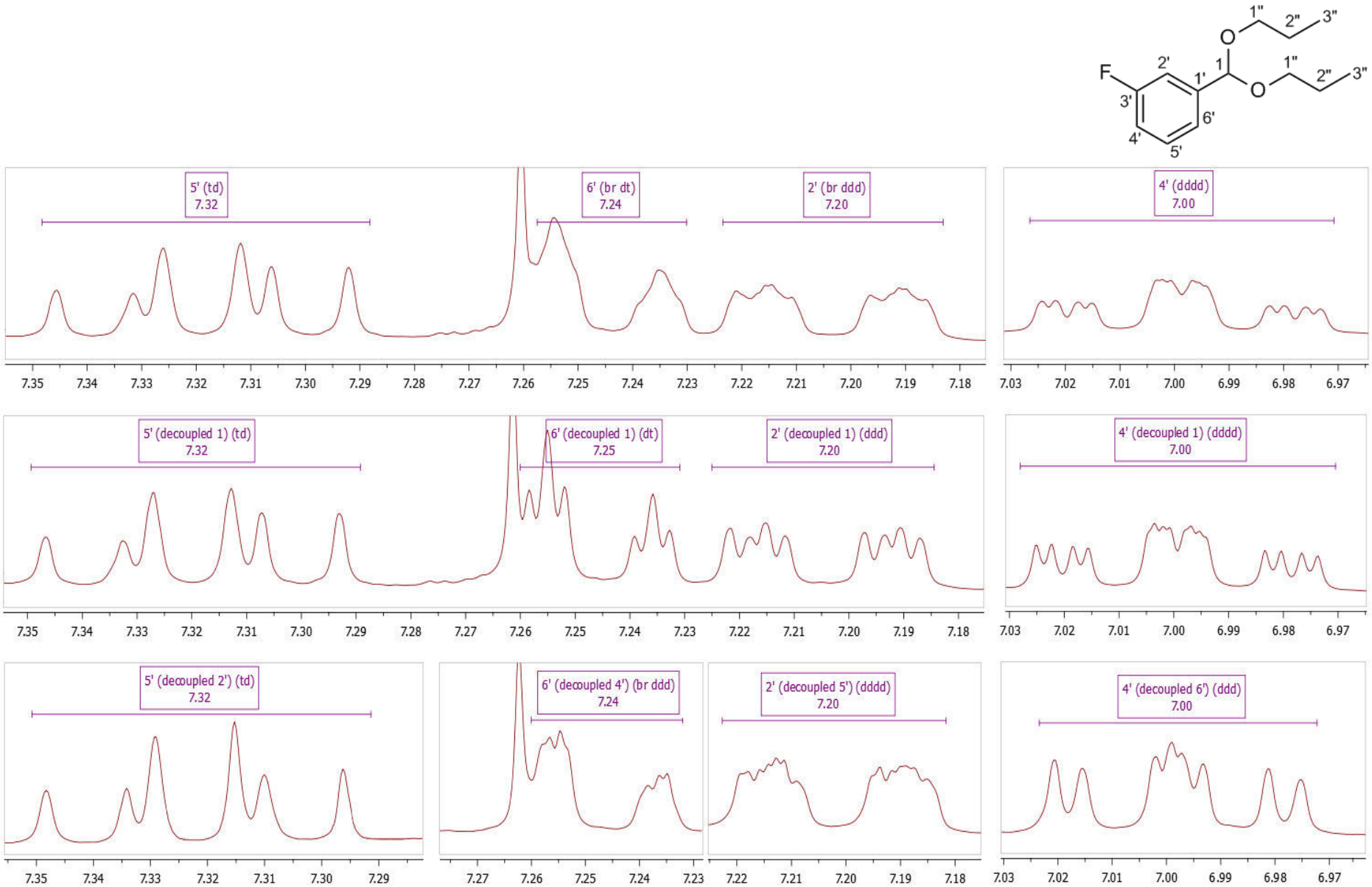
Scheme with key HMBC and NOESY interactions



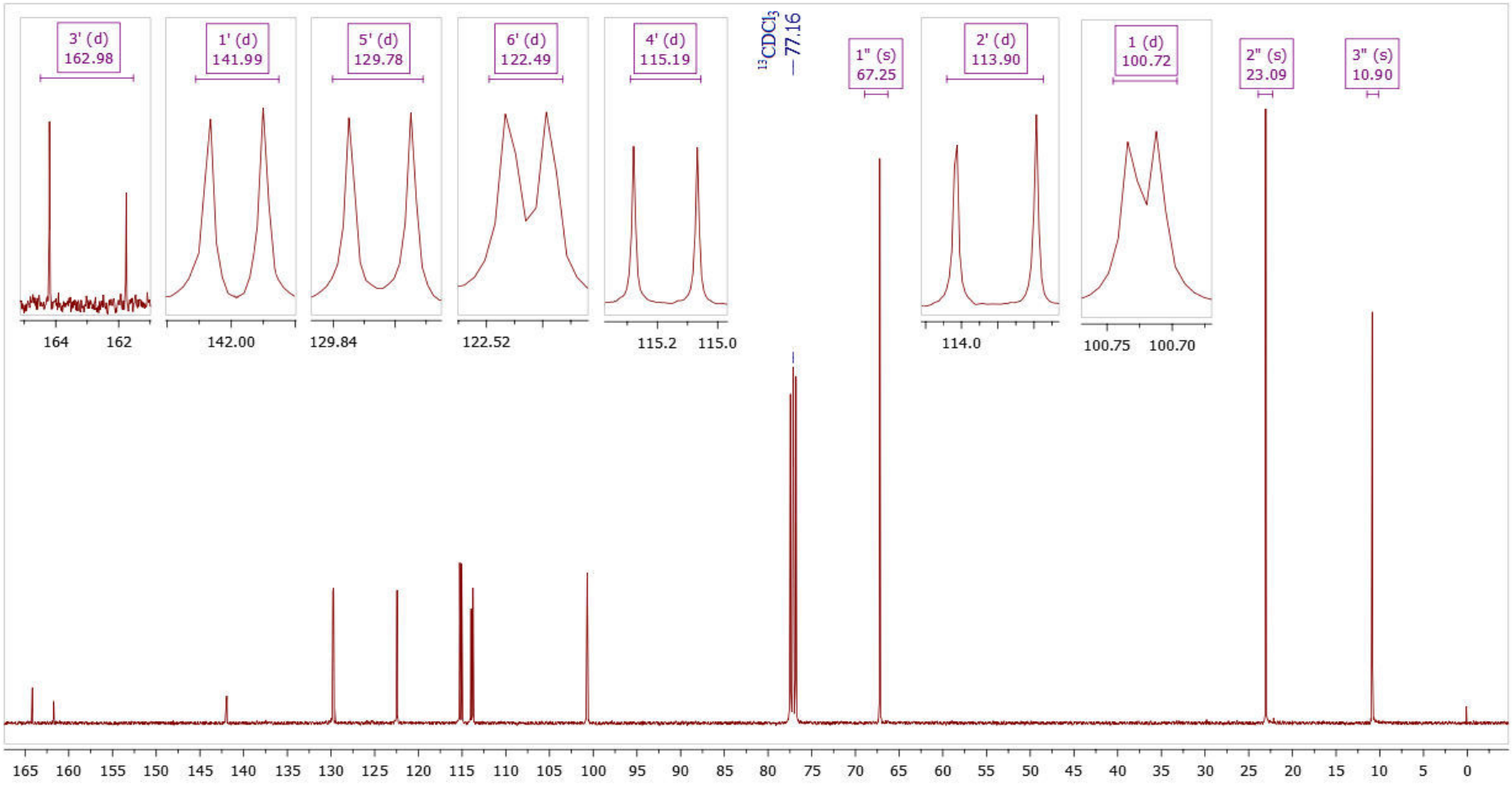
Analysis of ^{1}H-^{1}H coupling constants



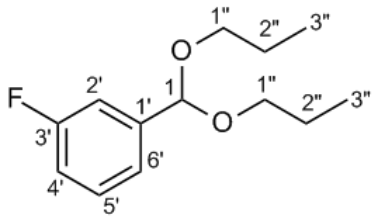
^{1}H-NMR (400 MHz, $CDCl_3$) spectrum of 1-(dipropoxymethyl)-3-fluorobenzene and the corresponding expansions with signal assignment

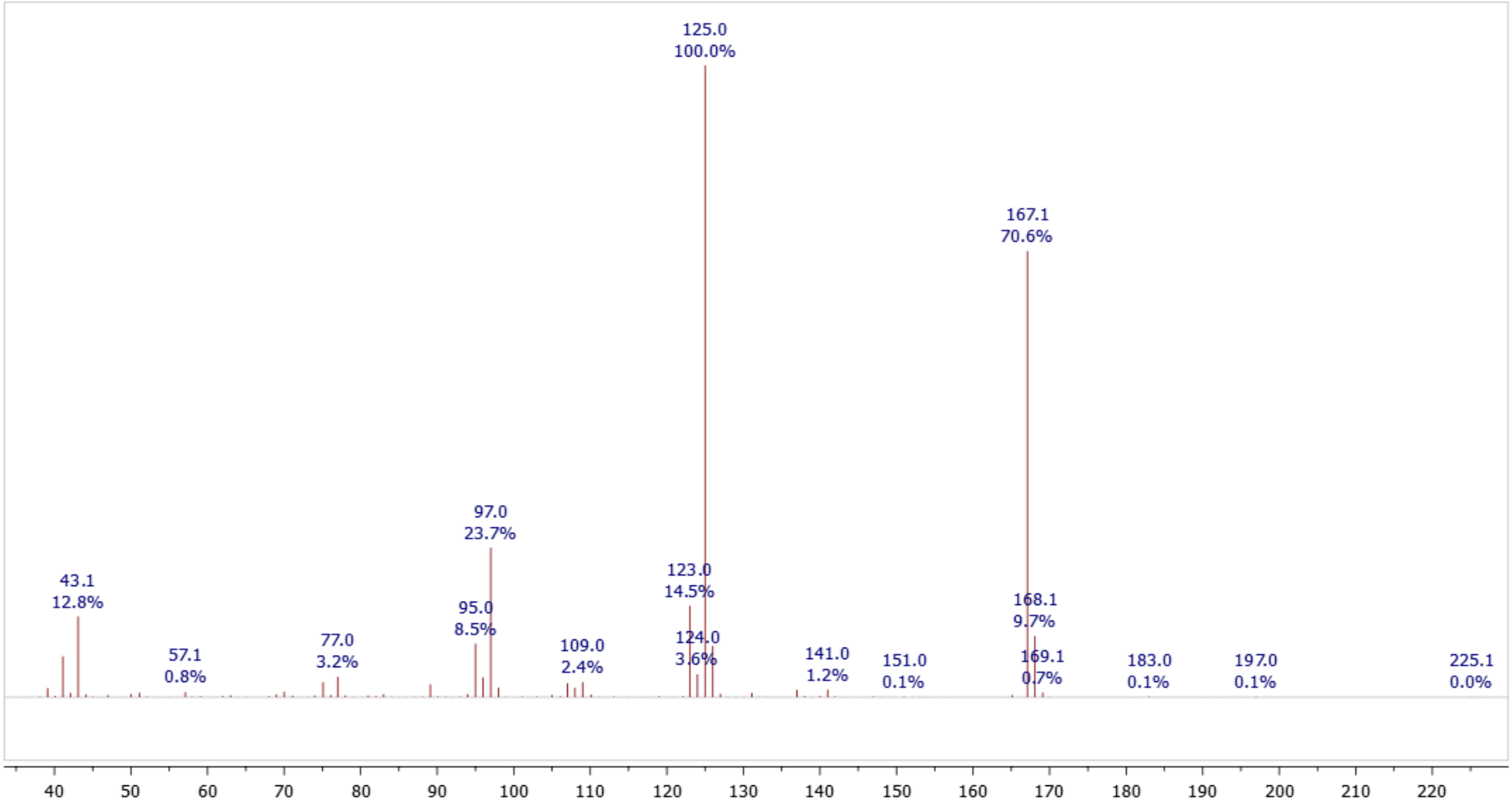


Selected expansions of ^{1}H-NMR spectrum (top) and the corresponding expansions in ^{1}H-NMR spectra obtained in a series of ^{1}H selective homodecoupling experiments (middle and bottom)

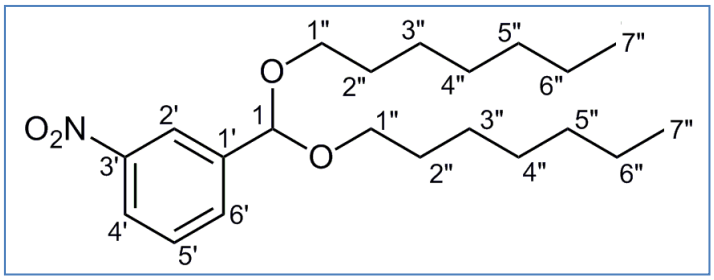


^{13}C-NMR (100.6 MHz, $CDCl_3$) spectrum of 1-(dipropoxymethyl)-3-fluorobenzene and the corresponding expansions with signal assignment





EI-MS spectrum of 1-(dipropoxymethyl)-3-fluorobenzene



1-(bis(heptyloxy)methyl)-3-nitrobenzene

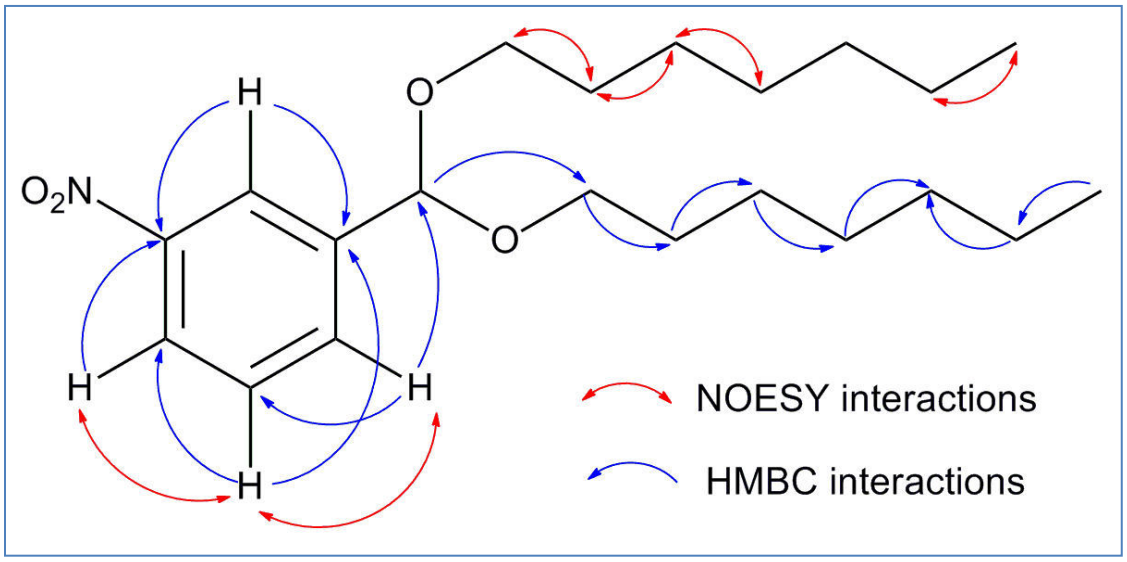
Table of NMR data of 1-(bis(heptyloxy)methyl)-3-nitrobenzene (chemical shift and coupling constant data from ^{1}H and ^{13}C NMR spectra with the observed HMBC and NOESY interactions)

Position	δ_H (Integral, m, *J* (Hz))	δ_C (ppm) C-13 {H}	HMBC[a]	NOESY
1	5.57 (1 H, ddd, $^{4}J_{1,2'} = 0.6$, $^{4}J_{1,6'} = 0.5$, $^{6}J_{1,4'} = 0.4$)[b]	100.2 (1 C)	2', 1"	/
1'	/	141.6 (1 C)	/	/
2'	8.34 (1 H, dddd, $^{4}J_{2',4'} = 2.3$, $^{4}J_{2',6'} = 1.5$, $^{4}J_{1,2'} = 0.6$, $^{5}J_{2',5'} = 0.5$)[b]	122.1 (1 C)	1, 1', 3', 4'	/
3'	/	148.4 (1 C)	/	/
4'	8.18 (1 H, ddd, $^{3}J_{4',5'} = 8.2$, $^{4}J_{2',4'} = 2.3$, $^{4}J_{4',6'} = 1.0$, $^{4}J_{1,4'} = 0.4$)[b]	123.4 (1 C)	2', 3'	5'
5'	7.54 (1 H, ddd, $^{3}J_{4',5'} = 8.2$, $^{3}J_{5',6'} = 7.7$, $^{5}J_{2',5'} = 0.5$)[b]	129.3 (1 C)	3', 1', 3', 4'	4', 6'
6'	7.81 (1 H, dddd, $^{3}J_{5',6'} = 7.7$, $^{4}J_{2',6'} = 1.5$, $^{4}J_{4',6'} = 1.0$, $^{4}J_{1,6'} = 0.5$)[b]	133.0 (1 C)	1, 4', 5'	5'
1"a	3.49 (2 H, dt, $^{2}J_{1"a,1"b} = 9.4$, $^{3}J_{1"a,2"} = 6.6$)	65.9 (2 C)	1, 2", 3"	2"
1"b	3.53 (2 H, dt, $^{2}J_{1"a,1"b} = 9.4$, $^{3}J_{1"b,2"} = 6.7$)			
2"	1.62 (4 H, pseudo quintet, $^{3}J_{1"a,2"} \approx {}^{3}J_{1"b,2"} \approx {}^{3}J_{2",3"} \approx 6.7$)	29.8 (2 C)	1", 3", 4"	1", 3"
3"	1.35[c]	26.3 (2 C)	1", 2", 4"	2", 4"
4"	1.28[c]	29.2 (2 C)	5"	3"
5"	1.27[c]	31.9 (2 C)	3", 6"	/
6"	1.28[c]	22.7 (2 C)	7", 5"	7"
7"	0.88 (6 H, t, $^{3}J_{6",7"} = 6.9$)	14.2 (2 C)	5", 6"	6"

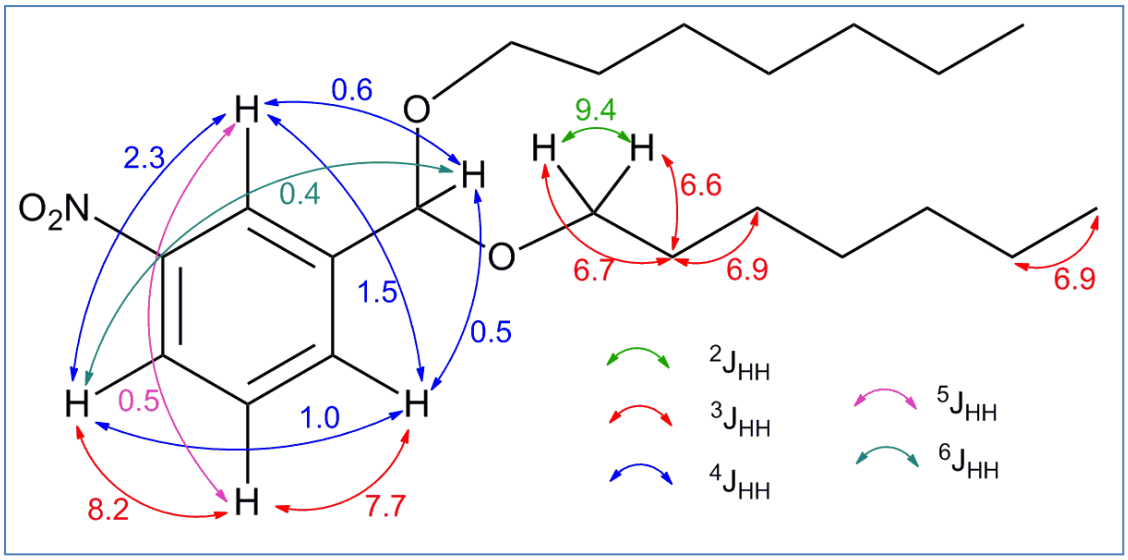
[a]Correlation between the hydrogen in this row and the carbon in the listed position.

[b]Coupling constants (and multiplicity) were determined from appropriate ^{1}H selective homodecoupled spectra. Values of coupling constants lower than 0.5 Hz were usually observed as a broadening of the corresponding ^{1}H NMR signals; they were disclosed only in a series of selective homodecoupling experiments.

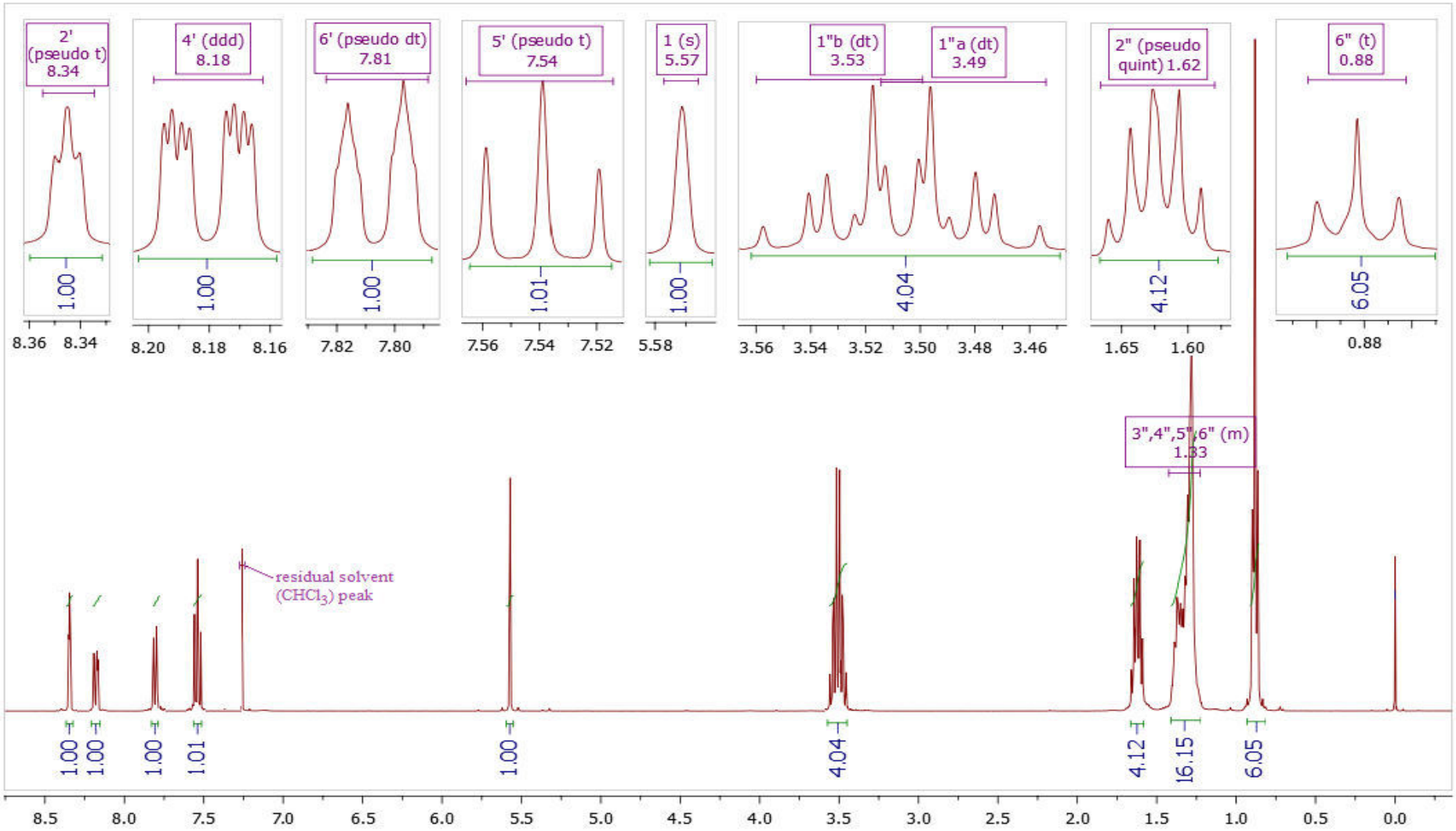
[c]Overlapped signals (range: 1.23-1.43 ppm, 16 H). Chemical shifts were determined from HSQC and HMBC spectra.



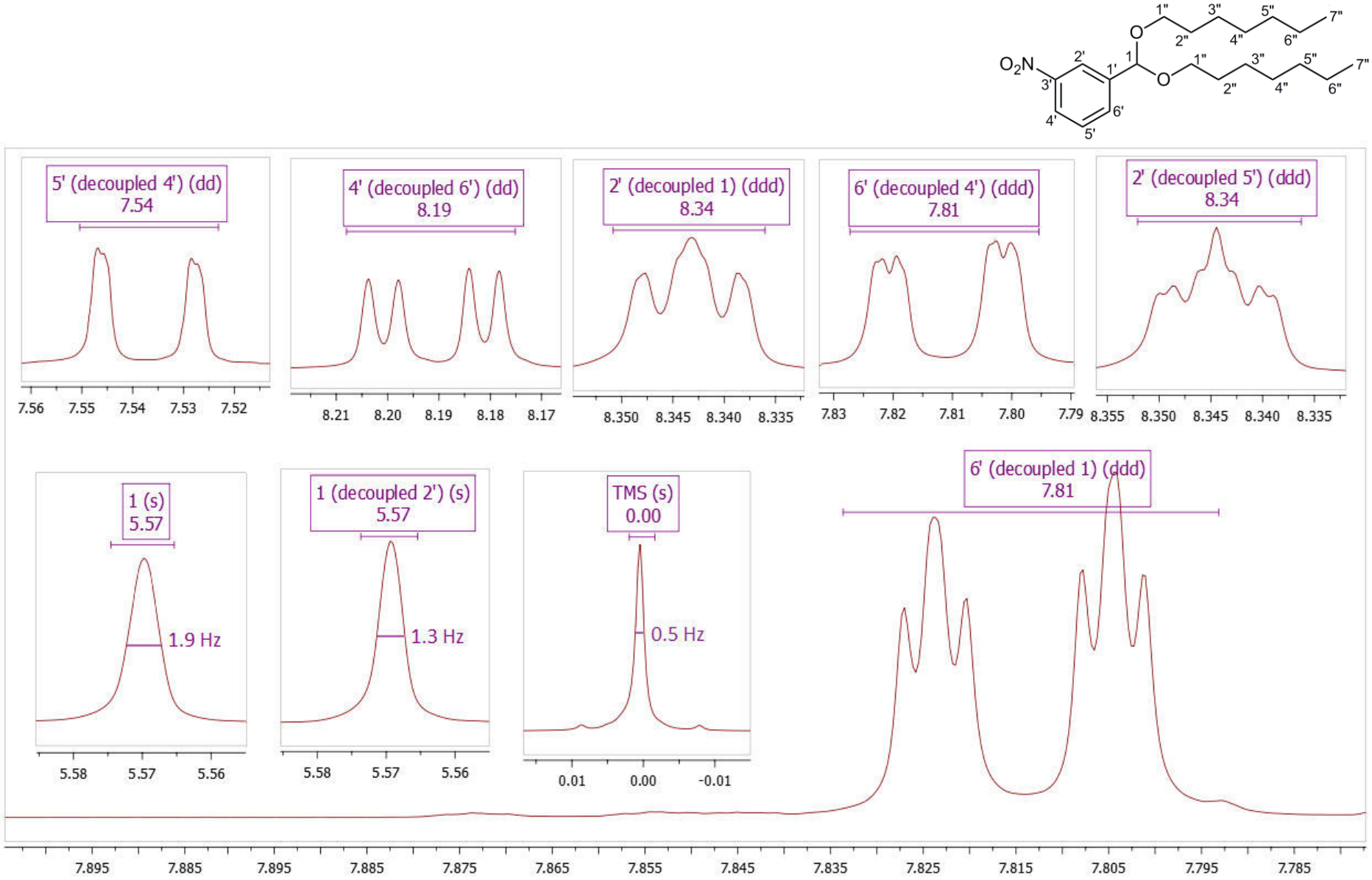
Scheme with key HMBC and NOESY interactions



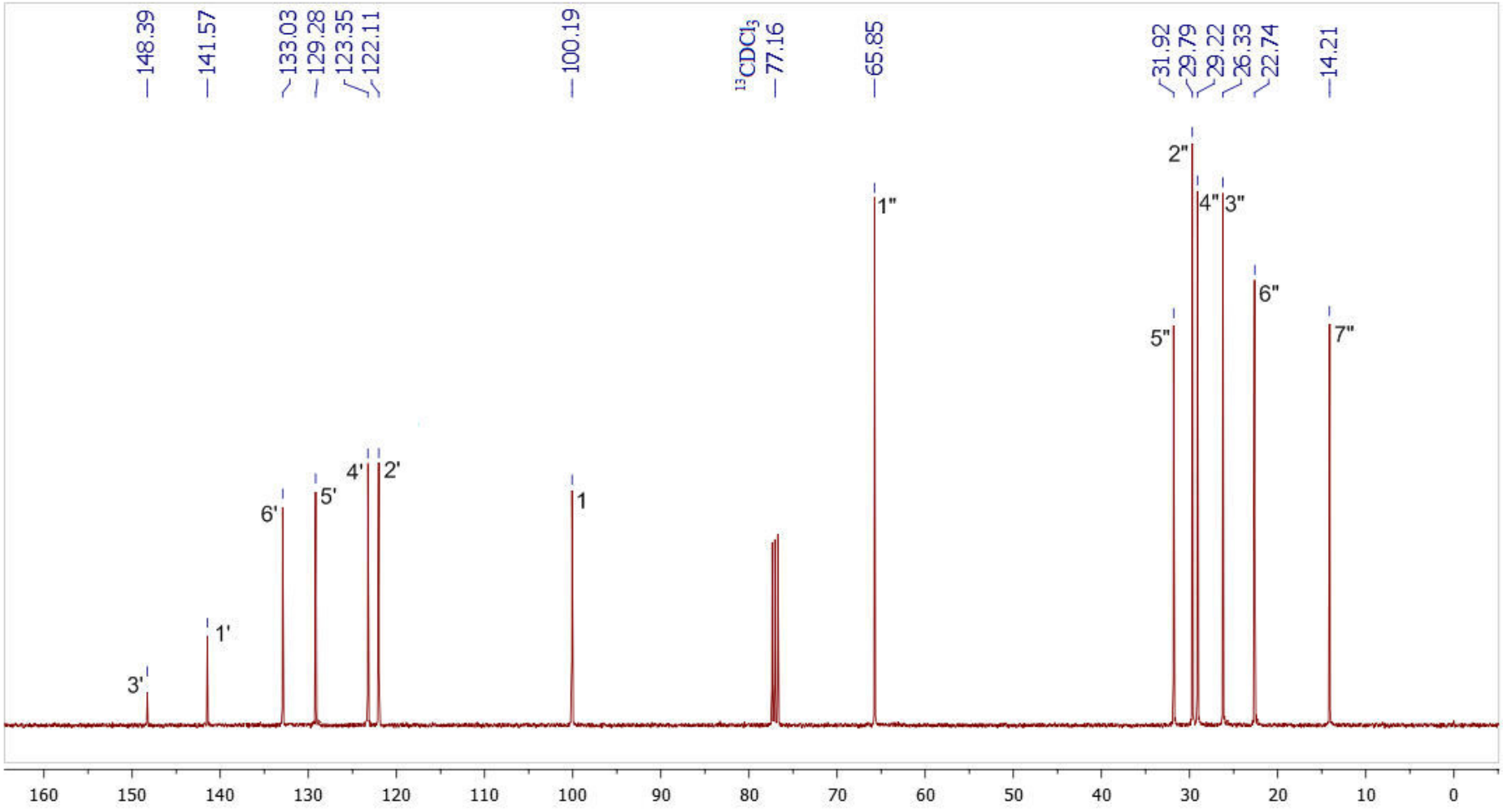
Analysis of ^{1}H-^{1}H coupling constants



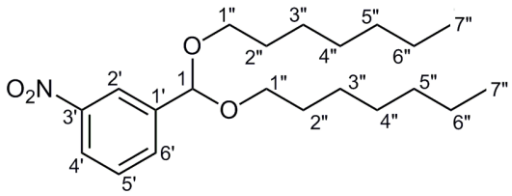
^{1}H-NMR (400 MHz, $CDCl_3$) spectrum of 1-(bis(heptyloxy)methyl)-3-nitrobenzene and the corresponding expansions with signal assignment

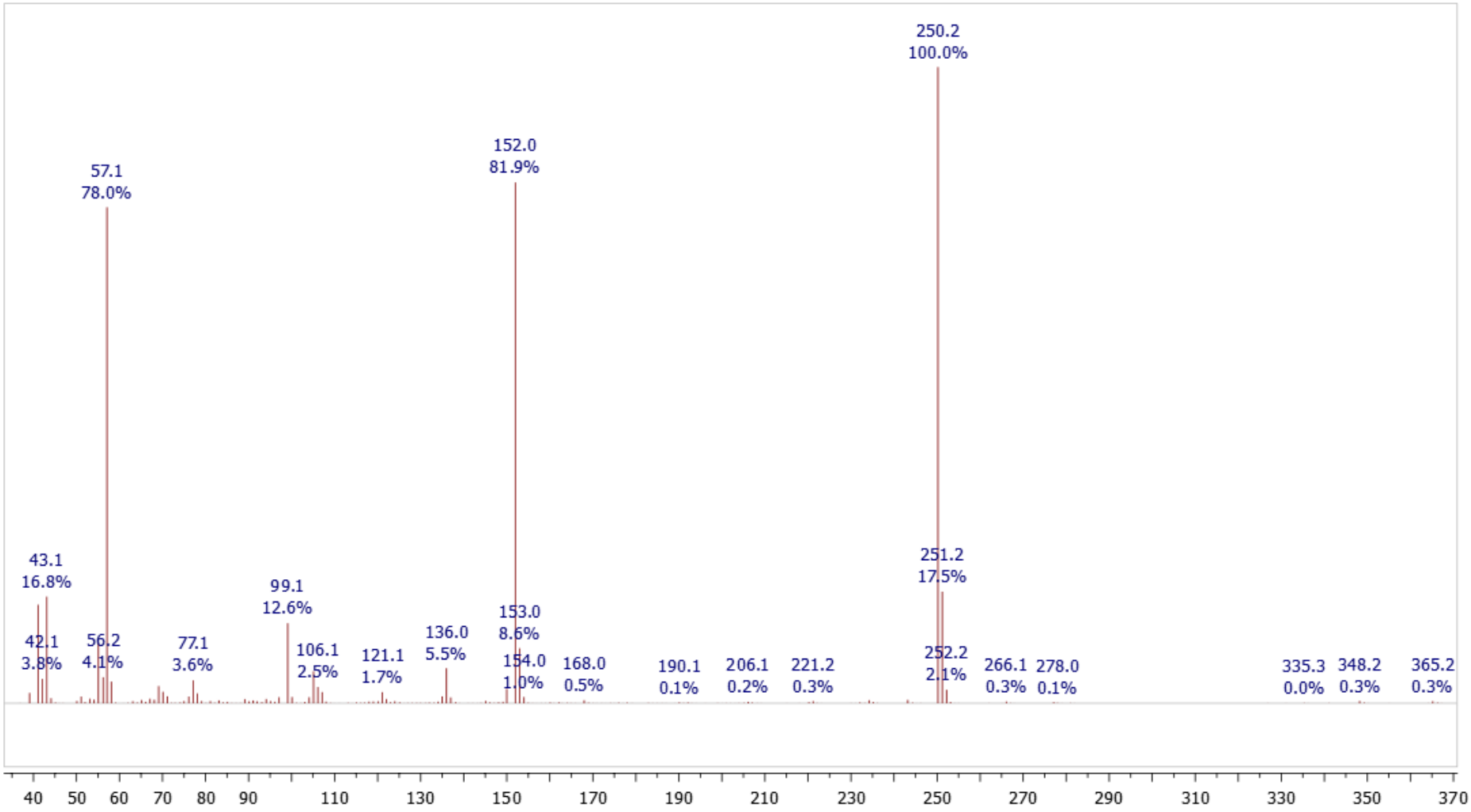


^{1}H NMR spectra obtained in a series of ^{1}H selective homodecoupling experiments with signal assignment

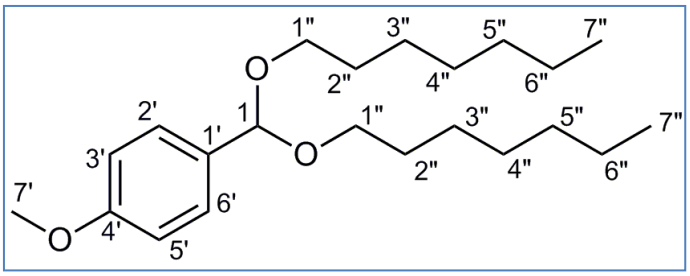


^{13}C-NMR (100.6 MHz, $CDCl_3$) spectrum of 1-(bis(heptyloxy)methyl)-3-nitrobenzene with signal assignment





EI-MS spectrum of 1-(bis(heptyloxy)methyl)-3-nitrobenzene



1-(bis(heptyloxy)methyl)-4-methoxybenzene

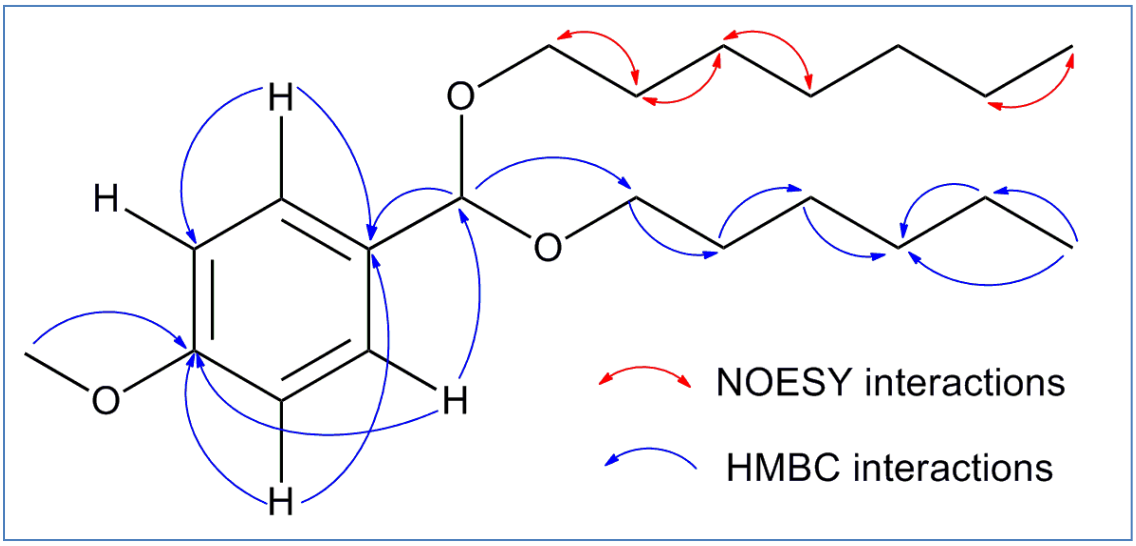
Table of NMR data of 1-(bis(heptyloxy)methyl)-4-methoxybenzene (chemical shift and coupling constant data from ^{1}H and ^{13}C NMR spectra with the observed HMBC and NOESY interactions)

Position	δ_H (Integral, m, *J* (Hz))	δ_C (ppm) C-13 {H}	HMBC[a]	NOESY
1	5.46 (1 H, t, $^{4}J_{1,2'} = {}^{4}J_{1,6'} = 0.5$)[b]	101.5 (1 C)	2', 6', 1"	/
1'	/	131.6 (1 C)	/	/
2', 6' (AA')	7.38 (2 H, m, AA'BB') ($^{3}J_{AB} = 8.6$, $^{4}J_{AA'} = 2.4$, $^{4}J_{BB'} = 2.6$, $^{5}J_{AB'/A'B} = 0.3$, $^{4}J_{AA',1} = 0.5$)[b]	128.0 (2 C)	1, 1', 3', 4', 5' (6'/2')	3'/5'
3', 5' (BB')	6.88 (2 H, m, AA'BB') ($^{3}J_{AB} = 8.6$, $^{4}J_{AA'} = 2.4$, $^{4}J_{BB'} = 2.6$, $^{5}J_{AB'/A'B} = 0.3$)[b]	113.5 (2 C)	1', 4', (5'/3')	2'/6'
4'	/	159.6 (1 C)	/	/
7'	3.80 (3 H, s)	55.3 (1 C)	4'	/
1"a	3.43 (2 H, dt, $^{2}J_{1"a,1"b} = 9.4$, $^{3}J_{1"a,2"} = 6.6$)	65.4 (2 C)	1, 2", 3"	2"
1"b	3.51 (2 H, dt, $^{2}J_{1"a,1"b} = 9.4$, $^{3}J_{1"b,2"} = 6.7$)		1, 2", 3"	2"
2"	1.60 (4 H, tdd, $^{3}J_{2",3"} = 6.9$, $^{3}J_{1"b,2"} = 6.7$, $^{3}J_{1"a,2"} = 6.6$)	29.9 (2 C)	1"a, 1"b, 3", 4"	1"a, 1"b, 3"
3"	1.36[c]	26.4 (2 C)	2", 4"	2", 4"
4"	1.30[c]	29.3 (2 C)	5"	3"
5"	1.28[c]	32.0 (2 C)	3"	/
6"	1.29[c]	22.7 (2 C)	5", 7"	7"
7"	0.88 (6 H, t, $^{3}J_{6",7"} = 6.9$)	14.2 (2 C)	5", 6"	6"

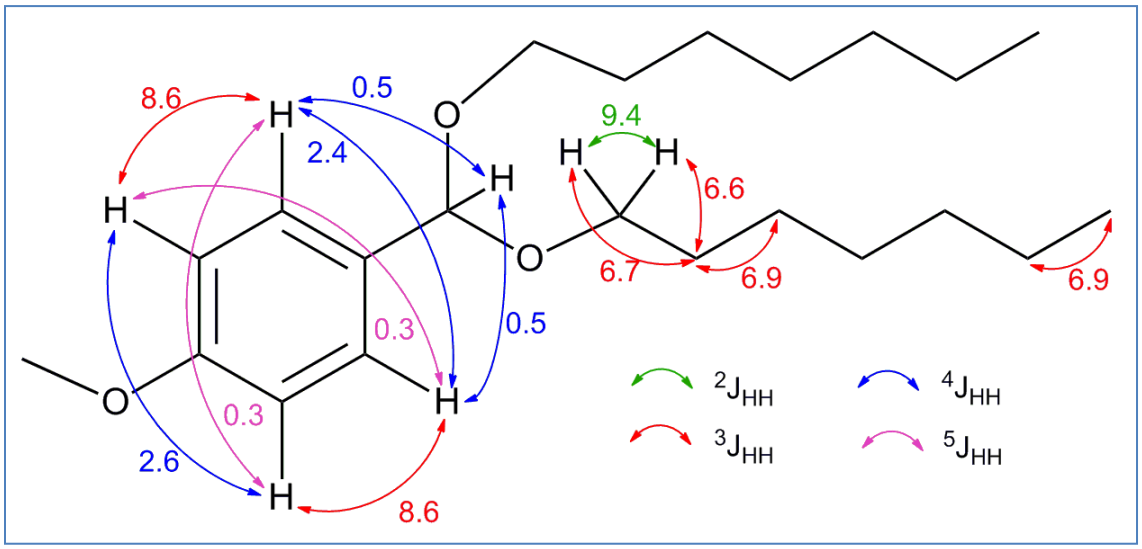
[a]Correlation between the hydrogen in this row and the carbon in the listed position.

[b]Coupling constants (and multiplicity) were determined from appropriate ^{1}H selective homodecoupled spectra. Values of coupling constants lower than 0.5 Hz were usually observed as a broadening of the corresponding ^{1}H NMR signals; they were disclosed only in a series of selective homodecoupling experiments. Values of coupling constants of higher-order spin systems were determined with the aid of "WinDNMR" simulations.

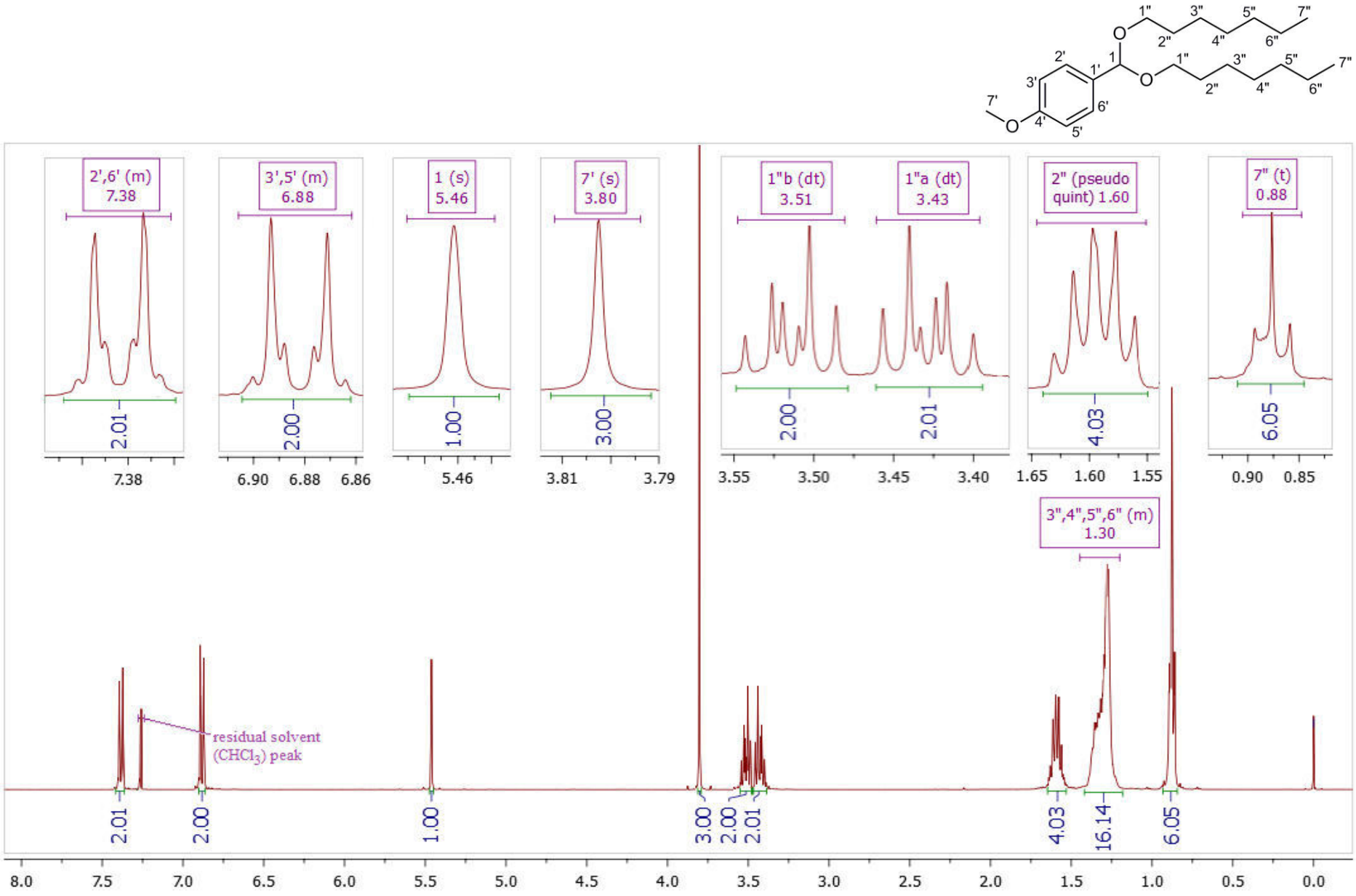
[c]Overlapped signals (range: 1.23-1.40 ppm, 16 H). Chemical shifts were determined from HSQC and HMBC spectra.



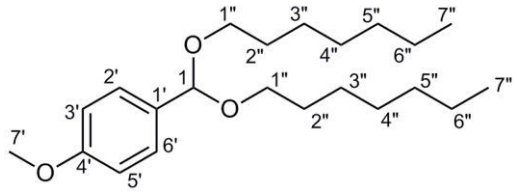
Scheme with key HMBC and NOESY interactions

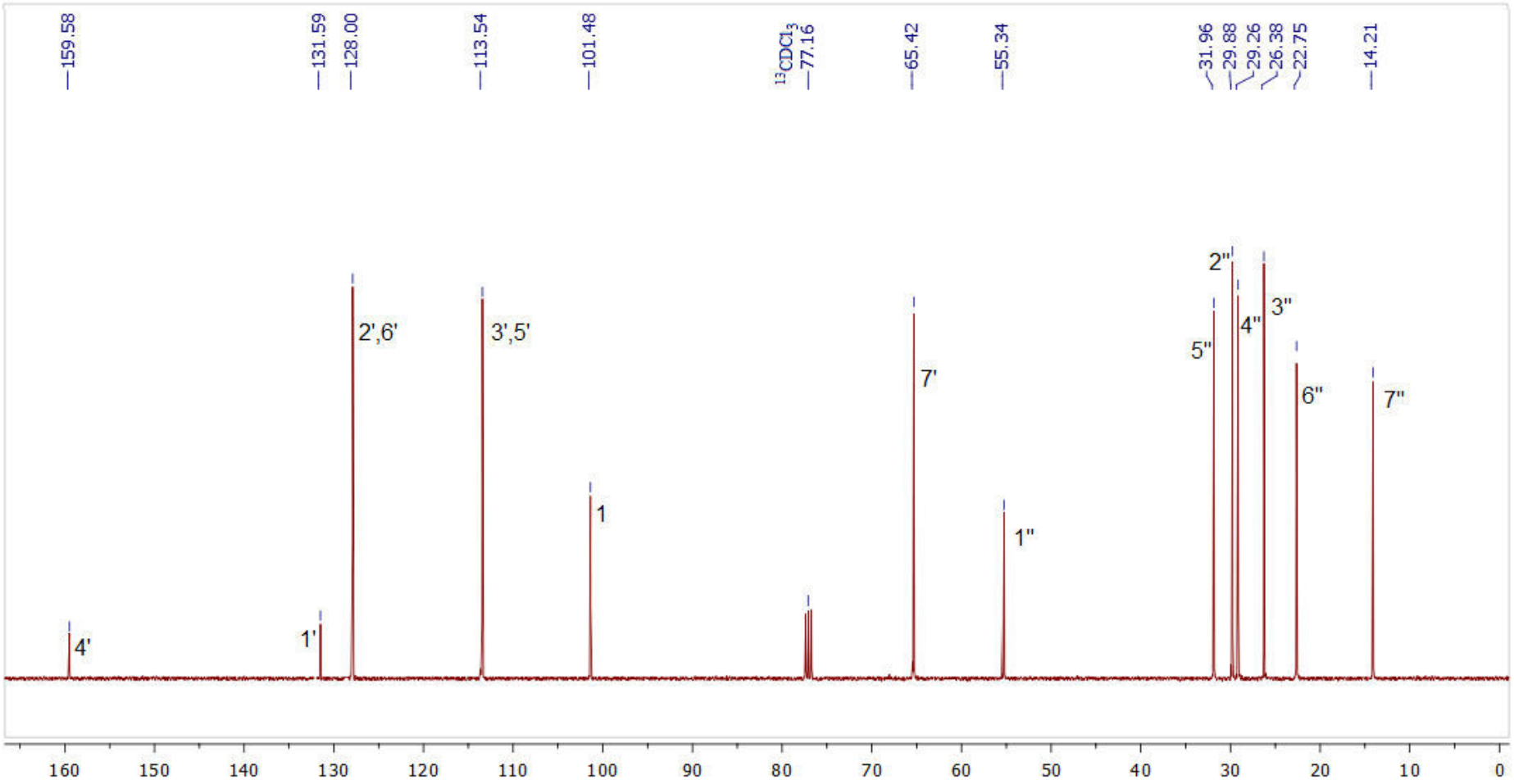


Analysis of ^{1}H-^{1}H coupling constants

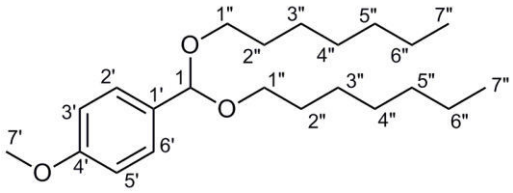


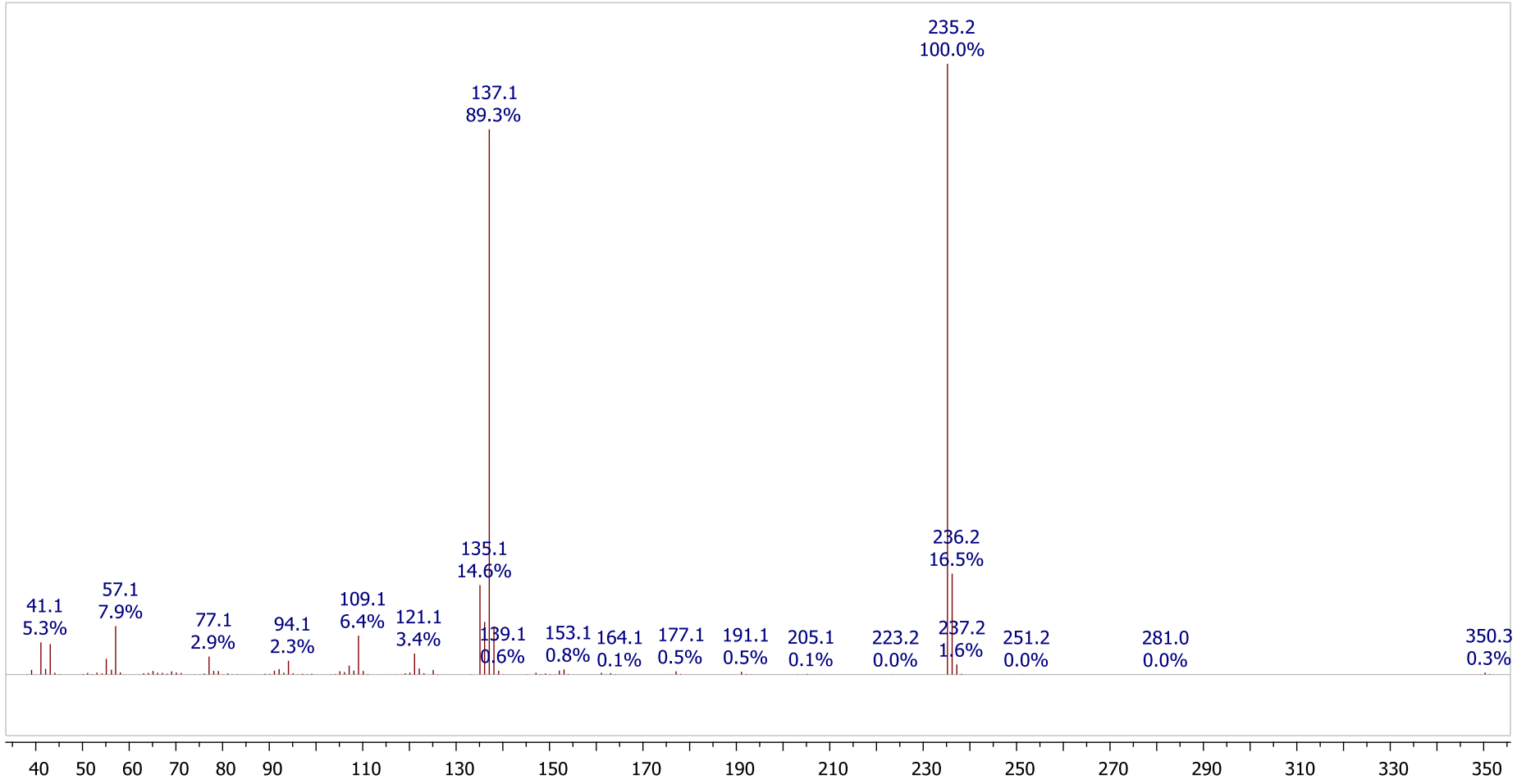
^{1}H-NMR (400 MHz, $CDCl_3$) spectrum of 1-(bis(heptyloxy)methyl)-4-methoxybenzene and the corresponding expansions with signal assignment



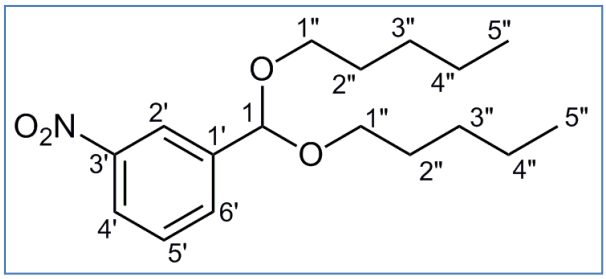


^{13}C-NMR (100 MHz, $CDCl_3$) spectrum of 1-(bis(heptyloxy)methyl)-4-methoxybenzene with signal assignment





EI-MS spectrum of 1-(bis(heptyloxy)methyl)-4-methoxybenzene



1-(bis(pentoxy)methyl)-3-nitrobenzene

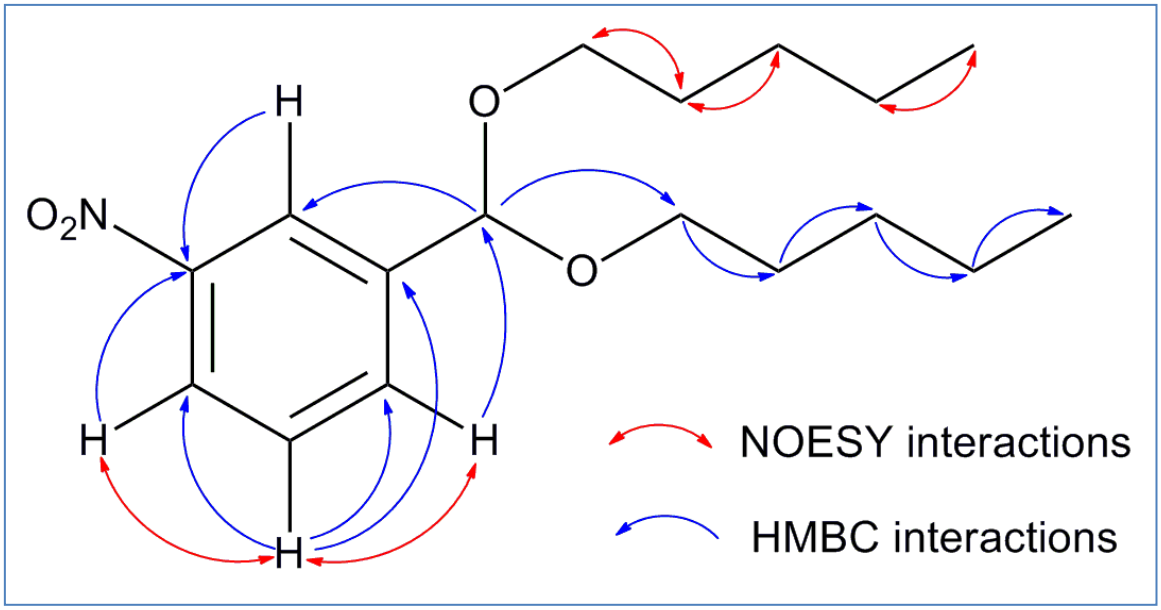
Table of NMR data of 1-(bis(pentoxy)methyl)-3-nitrobenzene (chemical shift and coupling constant data from ^{1}H and ^{13}C NMR spectra with the observed HMBC and NOESY interactions)

Position	δ_H (Integral, m, *J* (Hz))	δ_C (ppm) C-13 {H}	HMBC[a]	NOESY
1	5.58 (1 H, ddd, $^{4}J_{1,6}$ = 0.6, $^{4}J_{1,2'}$ = 0.5, $^{6}J_{1,4'}$ = 0.4)[b]	100.1 (1 C)	2', 6', 1"	/
1'	/	141.5 (1 C)	/	/
2'	8.35 (1 H, ddt, $^{4}J_{2',4'}$ = 2.3, $^{4}J_{2',6'}$ = 1.5, $^{4}J_{1,2'}$ = 0.5, $^{5}J_{2',5'}$ = 0.5)[b]	122.1 (1 C)	1, 3', 4', 6'	/
3'	/	148.3 (1 C)	/	/
4'	8.19 (1 H, dddd, $^{3}J_{4',5'}$ = 8.2, $^{4}J_{2',4'}$ = 2.3, $^{4}J_{4',6'}$ = 1.0, $^{4}J_{1,4'}$ = 0.4)[b]	123.3 (1 C)	2', 3', 6'	5'
5'	7.55 (1 H, ddd, $^{3}J_{4',5'}$ = 8.2, $^{3}J_{5',6'}$ = 7.7, $^{5}J_{2',5'}$ = 0.5)[b]	129.3 (1 C)	1', 3', 4', 6'	4', 6'
6'	7.82 (1 H, dddd, $^{3}J_{5',6'}$ = 7.7, $^{4}J_{2',6'}$ = 1.5, $^{4}J_{4',6'}$ = 1.0, $^{4}J_{1,6'}$ = 0.5)[b]	133.0 (1 C)	1, 2', 4'	5'
1"a	3.49 (2 H, dt, $^{2}J_{1"a,1"b}$ = 9.3, $^{3}J_{1"a,2"}$ = 6.6, 2H)	65.8 (2 C)	1, 2", 3"	2"
1"b	3.53 (2 H, dt, $^{2}J_{1"a,1"b}$ = 9.3, $^{3}J_{1"b,2"}$ = 6.7, 2H)			
2"	1.64 (4 H, qd, $^{3}J_{1"b,2"}$ = $^{3}J_{2",3"}$ = 6.7, $^{3}J_{1"a,2"}$ = 6.6)	29.5 (2 C)	1", 3", 4"	1", 3"
3"	1.36 (m, $^{3}J_{2",3"}$ = 6.7, $^{3}J_{3",4"}$ = 6.7)[b,c]	28.5 (2 C)	1", 2", 4"	2"
4"	1.34 (m, $^{3}J_{4",5"}$ = 7.1, $^{3}J_{3",4"}$ = 6.7)[b,c]	22.6 (2 C)	3", 5"	5"
5"	0.91 (6 H, t, $^{3}J_{4",5"}$ = 7.1)	14.1 (2 C)	3", 4"	4"

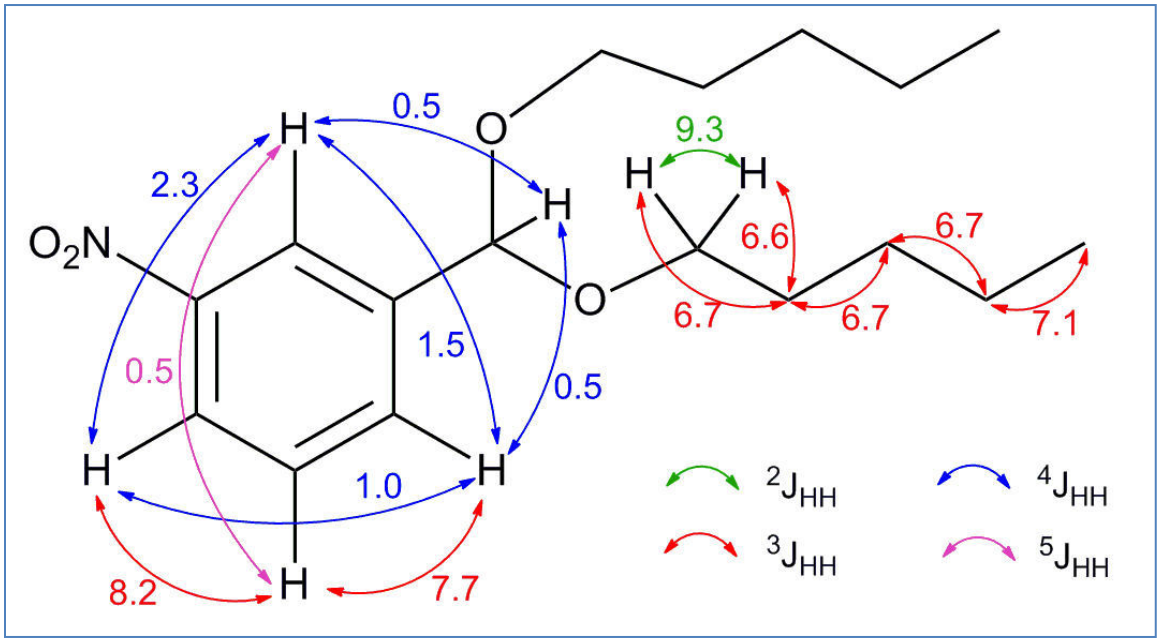
[a]Correlation between the hydrogen in this row and the carbon in the listed position.

[b]Coupling constants (and multiplicity) were determined from appropriate ^{1}H selective homodecoupled spectra. Values of coupling constants lower than 0.5 Hz were usually observed as a broadening of the corresponding ^{1}H NMR signals; they were disclosed only in a series of selective homodecoupling experiments.

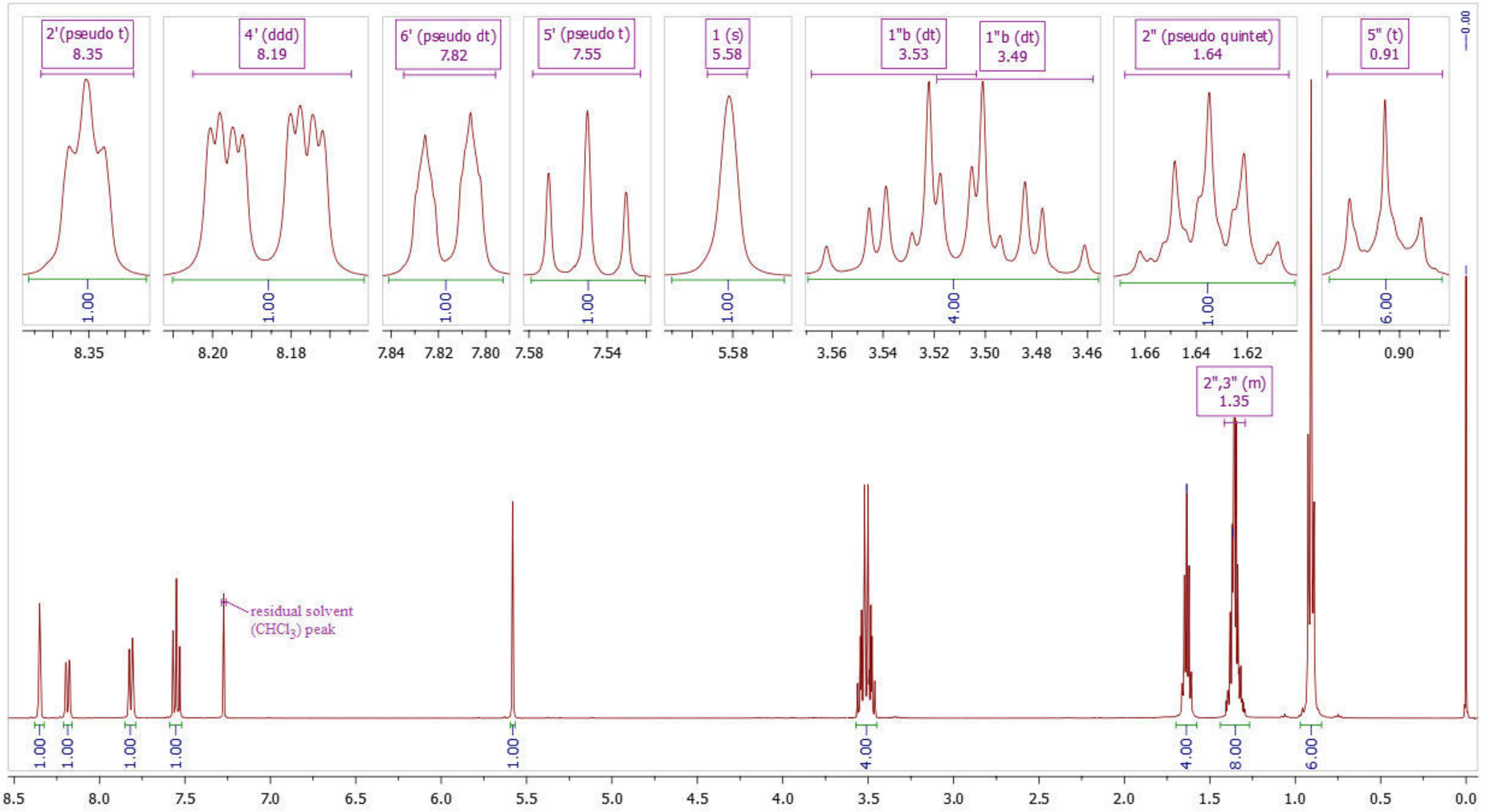
[c]Overlapped signals (range: 1.29-1.42 ppm, 8 H). Chemical shifts were determined from HSQC and HMBC spectra. Values of coupling constants of higher-order spin systems were determined with the aid of "WinDNMR" simulations.



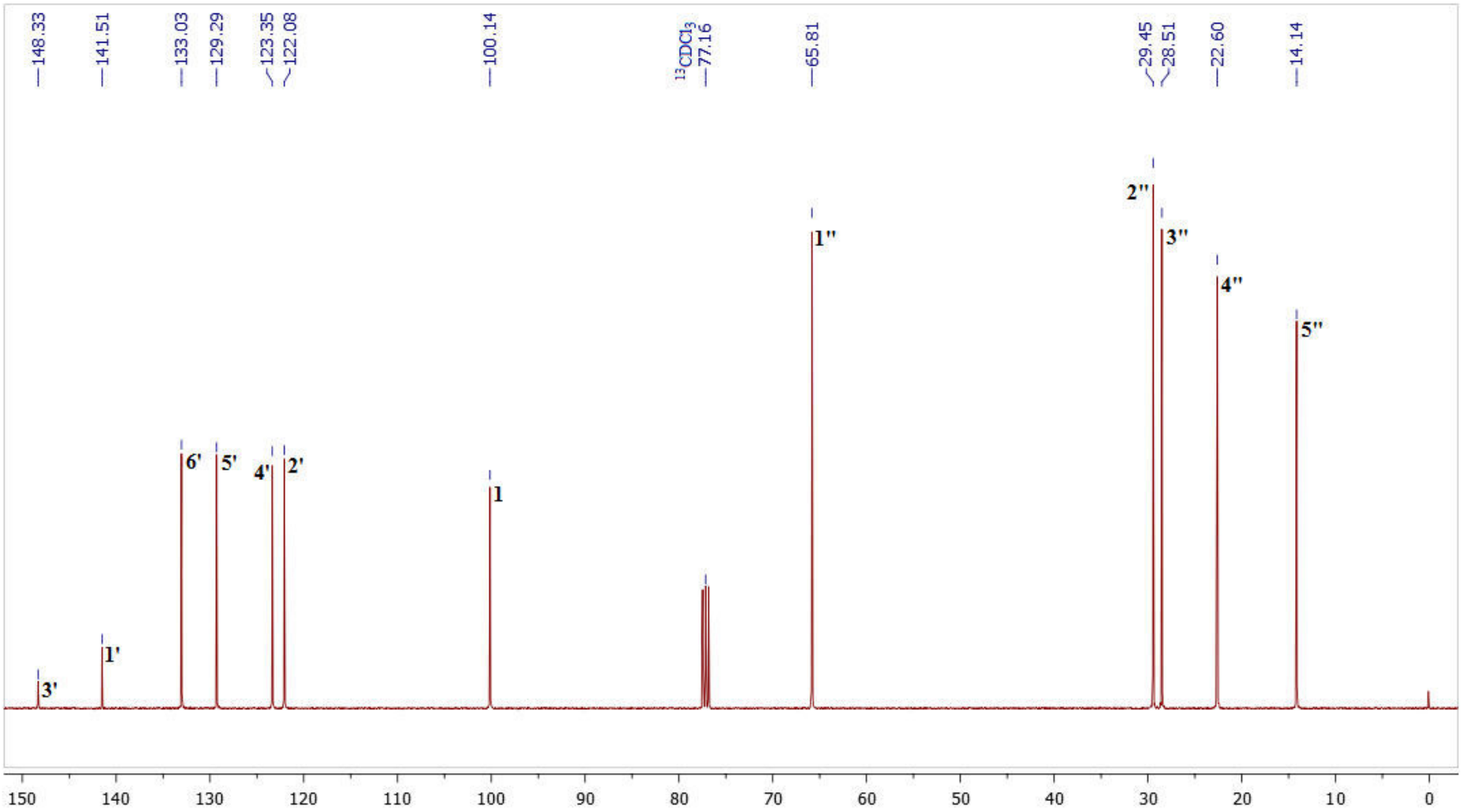
Scheme with key HMBC and NOESY interactions



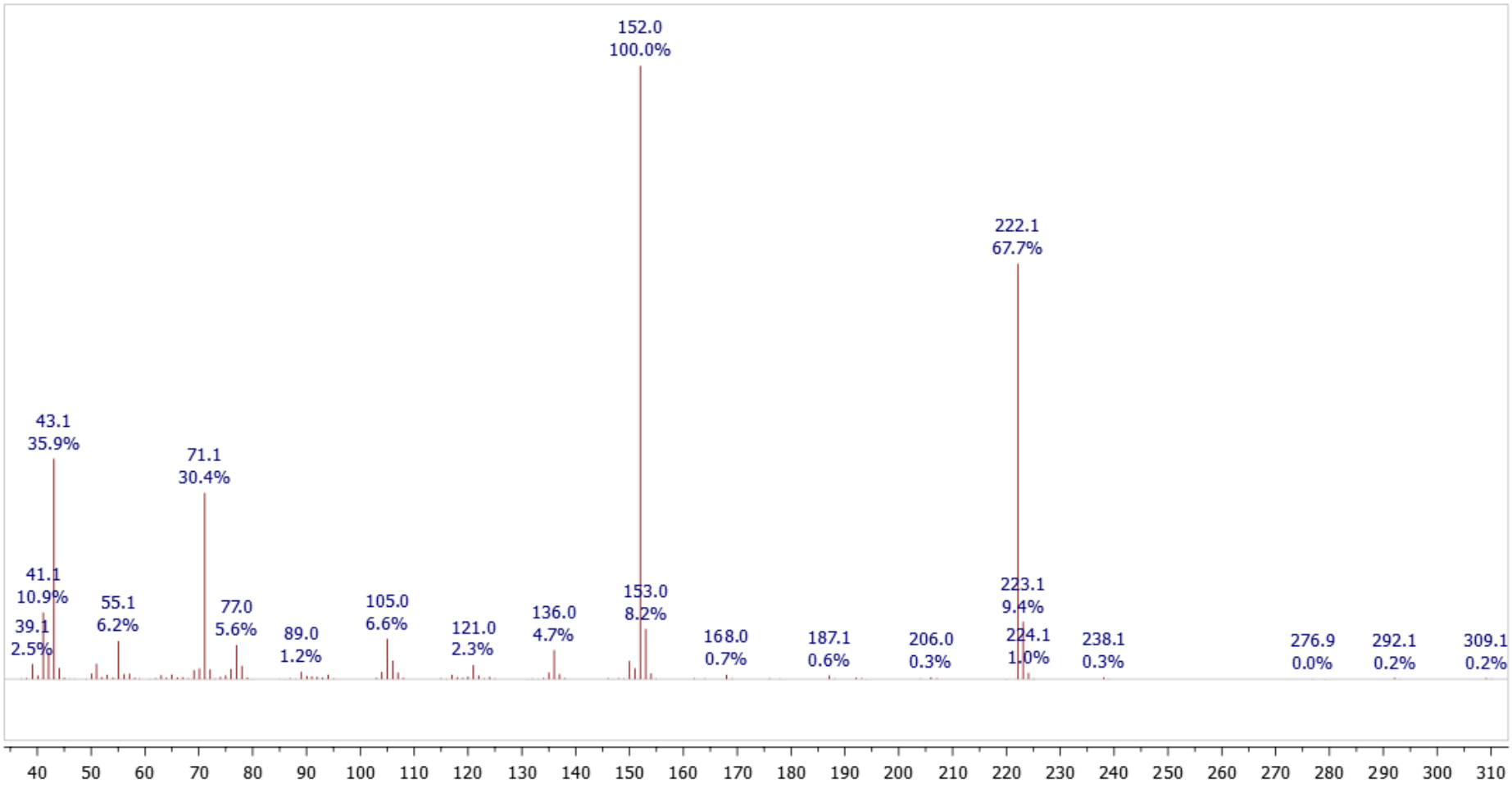
Analysis of ^{1}H-^{1}H coupling constants



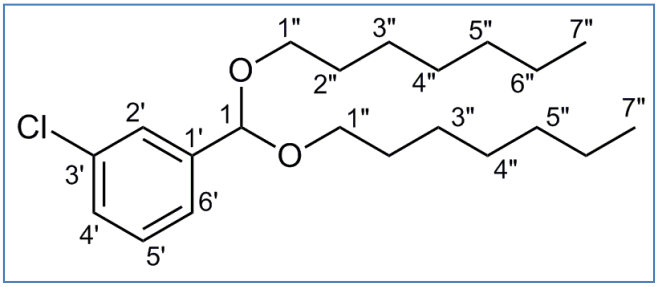
^{1}H-NMR (400 MHz, $CDCl_3$) spectrum of 1-(bis(pentoxy)methyl)-3-nitrobenzene and the corresponding expansions with signal assignment



^{13}C-NMR (400 MHz, $CDCl_3$) spectrum of 1-(bis(pentoxy)methyl)-3-nitrobenzene with signal assignment



EI-MS spectrum of 1-(bis(pentoxy)methyl)-3-nitrobenzene



1-(bis(heptyloxy)methyl)-3-chlorobenzene

Table of NMR data of 1-(bis(heptyloxy)methyl)-3-chlorobenzene (chemical shift and coupling constant data from ^{1}H and ^{13}C NMR spectra with the observed HMBC and NOESY interactions)

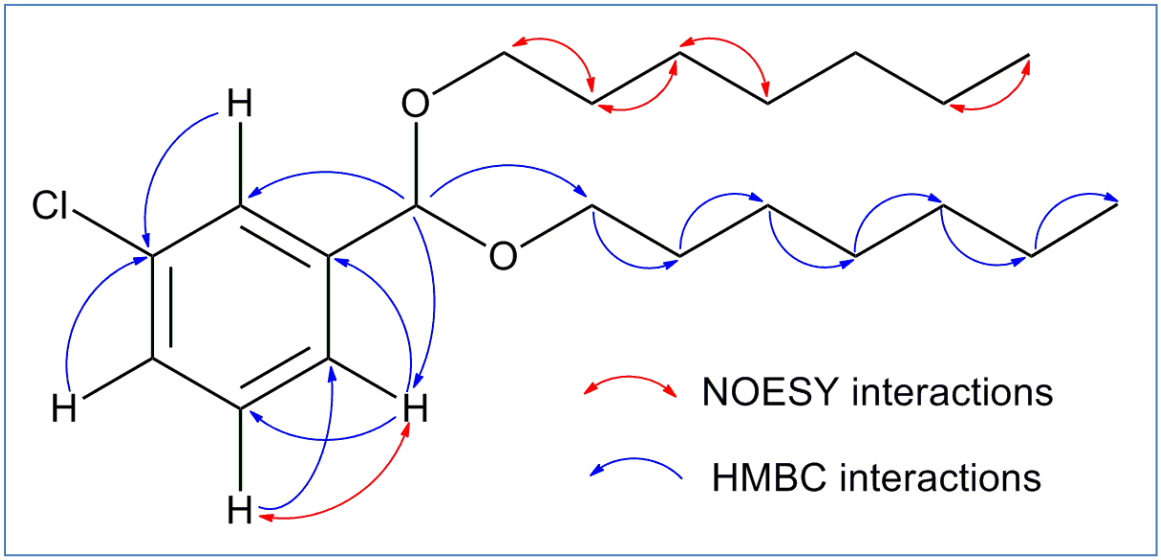
Position	δ_H (Integral, m, *J* (Hz))	δ_C (ppm) C-13 {H}	HMBC[c]	NOESY
1	5.47 (1 H, ddd, $^{4}J_{1,6'}$ = 0.5, $^{4}J_{1,2'}$ = 0.4, $^{6}J_{1,4'}$ = 0.3)[b]	100.7 (1 C)	1', 6', 1"	/
1'	/	134.5 (1 C)	/	/
2'	7.47 (1 H, m, ABCD, $^{4}J_{1,2'}$ = 0.4)[c]	127.1 (1 C)	1, 1', 4', 6'	/
3'	/	141.4 (1 C)	/	/
4'	7.281 (1 H, m, ABCD, $^{6}J_{1,4'}$ = 0.3)[c]	129.6 (1 C)	3', 6'	/
5'	7.284 (1 H, m, ABCD)[c]	128.5 (1 C)	1', 3', 6'	6'
6'	7.34 (1 H, m, ABCD, $^{4}J_{1,6'}$ = 0.5)[c]	125.1 (1 C)	1, 5'	5'
1"a	3.44 (2 H, dt, $^{2}J_{1"a,1"b}$ = 9.4, $^{3}J_{1"a,2"}$ = 6.6)	65.6 (2 C)	1, 2", 3"	2"
1"b	3.51 (2 H, dt, $^{2}J_{1"a,1"b}$ = 9.4, $^{3}J_{1"b,2"}$ = 6.7)			
2"	1.60 (4 H, tdd, $^{3}J_{1"b,2"}$ = 6.9, $^{3}J_{2",3"}$ = 6.7, $^{3}J_{1"a,2"}$ =6.6)	29.8 (2 C)	1", 3", 4"	1", 3"
3"	1.35[d]	26.4 (2 C)	1", 2", 4"	2", 4"
4"	1.28[d]	29.3 (2 C)	3", 5"	3"
5"	1.27[d]	32.0 (2 C)	6"	/
6"	1.28[d]	22.8 (2 C)	5", 7"	7"
7"	0.88 (6 H, t, $^{3}J_{6",7"}$ = 6.7)	14.2 (2 C)	5", 6"	6"

[a]Correlation between the hydrogen in this row and the carbon in the listed position.

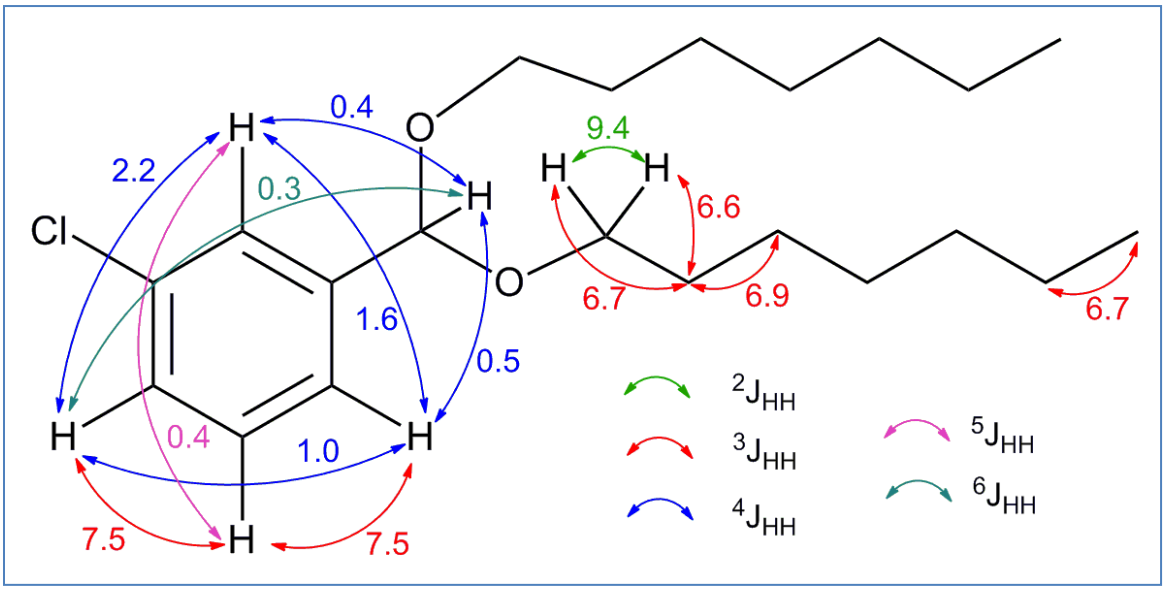
[b]Coupling constants (and multiplicity) were determined from appropriate ^{1}H selective homodecoupled spectra. Values of coupling constants lower than 0.5 Hz were usually observed as a broadening of the corresponding ^{1}H NMR signals; they were disclosed only in a series of selective homodecoupling experiments.

[c]J_{AB} = 7.5 Hz, J_{AC} = 1.0 Hz, J_{AD} = 2.2 Hz, J_{BC} = 7.5 Hz, J_{BD} = 0.4 Hz, J_{CD} = 1.6 Hz. The data from the spectrum obtained in a selective homodecoupling experiment (acetal proton decoupled) and "WinDNMR" simulation were used for the determination of the coupling constants. More accurate values of δ_H: 7.2808 (A/4'), 7.2835 (B/5'), 7.3360 (C/6'), 7.4692 (D/2').

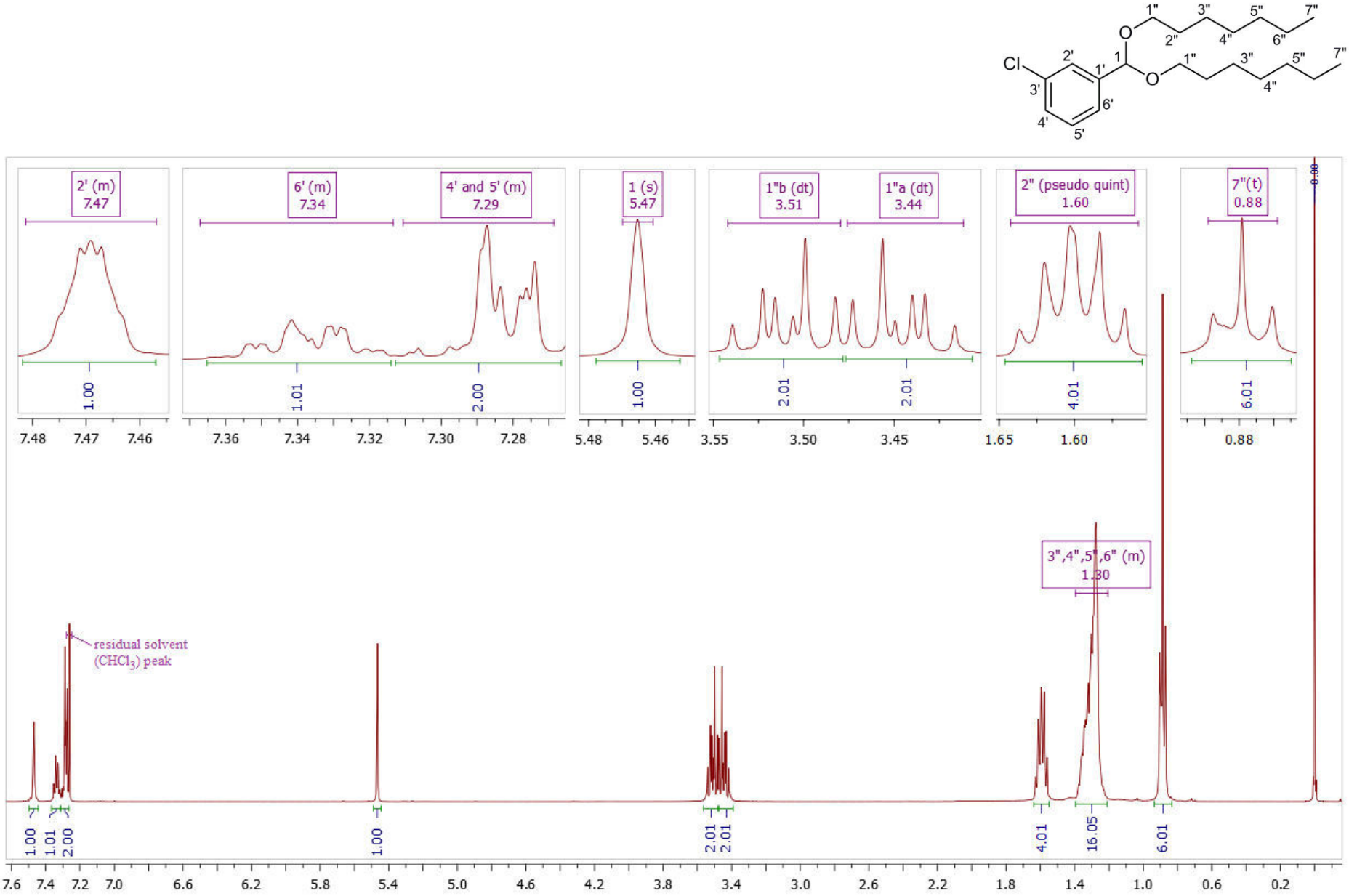
[d]Overlapped signals (range: 1.21-1.43 ppm, 16 H). Chemical shifts were determined from HSQC and HMBC spectra.



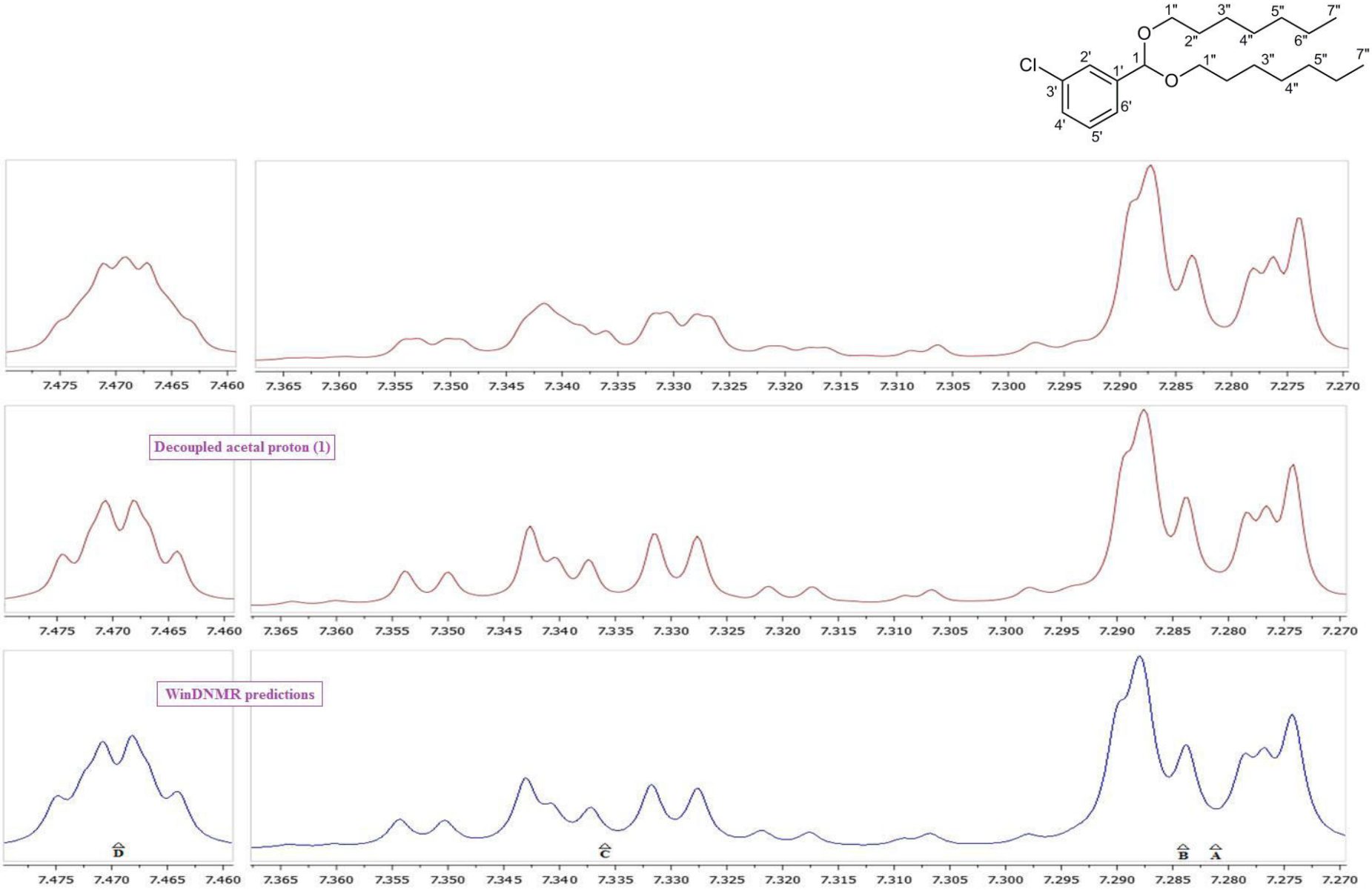
Scheme with key HMBC and NOESY interactions



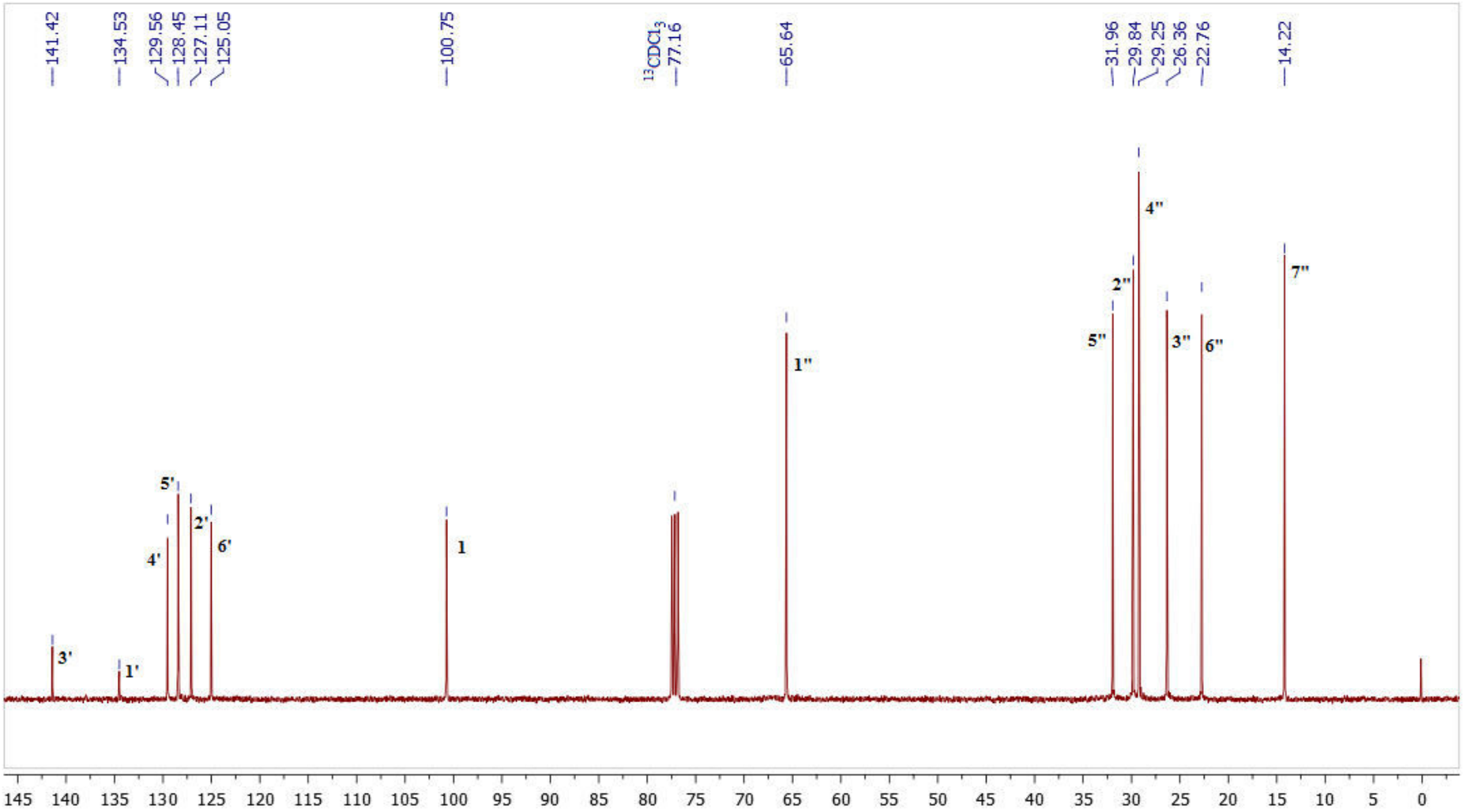
Analysis of ^{1}H-^{1}H coupling constants



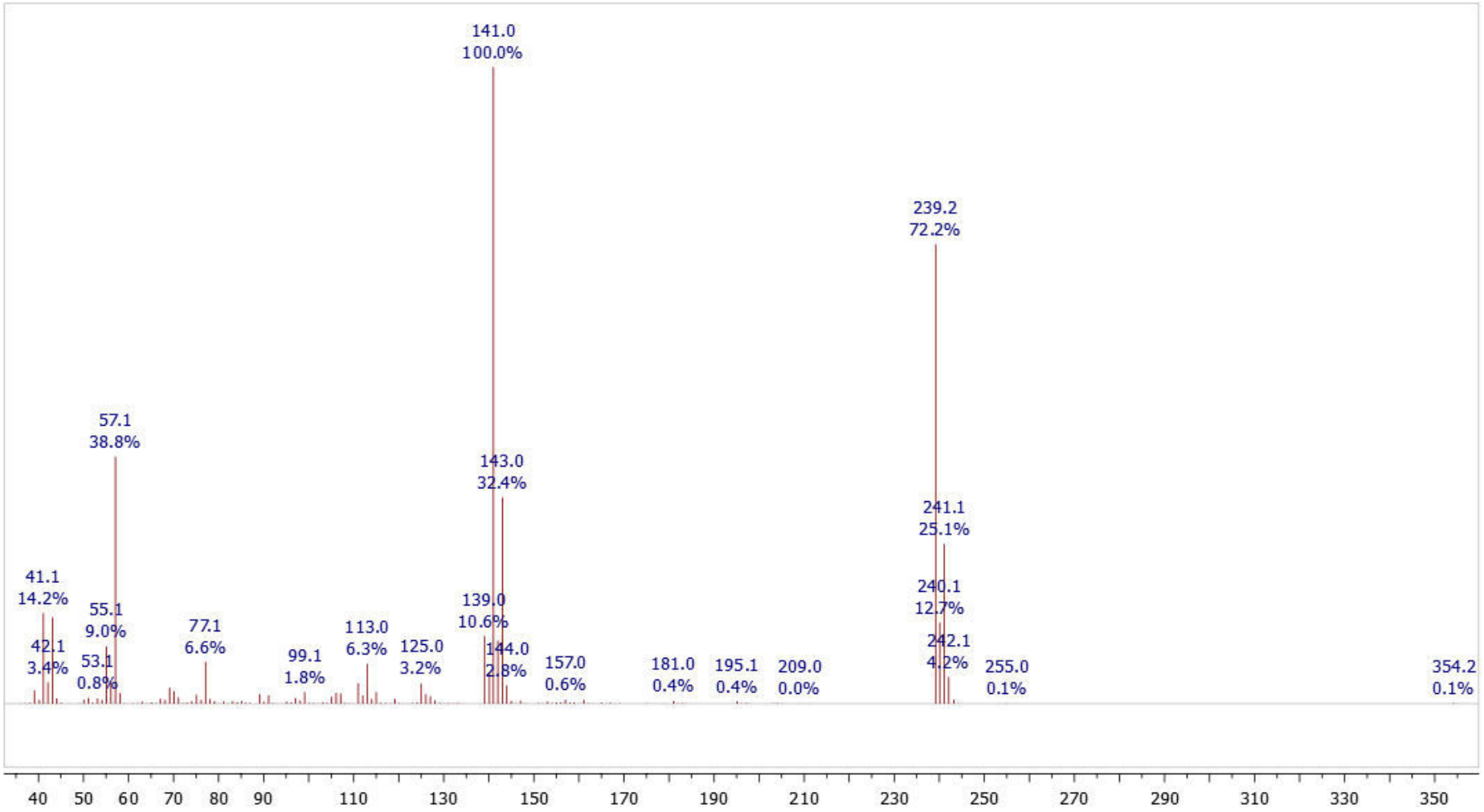
^{1}H-NMR (400 MHz, $CDCl_3$) spectrum of 1-(bis(heptyloxy)methyl)-3-chlorobenzene and the corresponding expansions with signal assignment



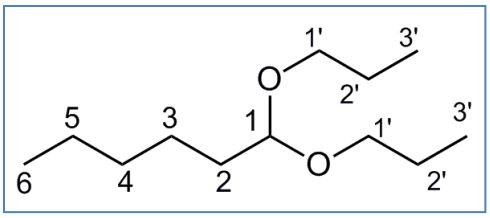
Expansions of ^{1}H-NMR spectrum of 1-(bis(heptyloxy)methyl)-3-chlorobenzene corresponding to protons attached to the aromatic ring (top); the corresponding expansions obtained in homodecoupling experiments (acetal proton decoupled; middle), and WinDNMR simulation of the mentioned decoupled spectrum (bottom)



^{13}C-NMR (100.6 MHz, $CDCl_3$) spectrum of 1-(bis(heptyloxy)methyl)-3-chlorobenzene with signal assignment



EI-MS spectrum of 1-(bis(heptyloxy)methyl)-3-chlorobenzene

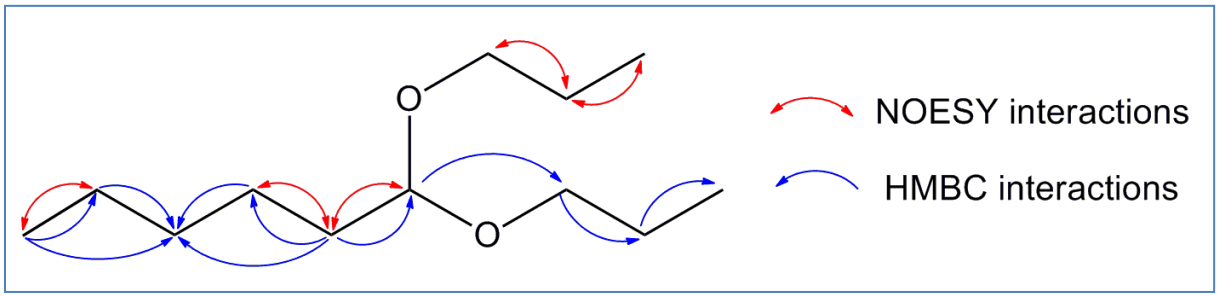


1,1-dipropoxyhexane

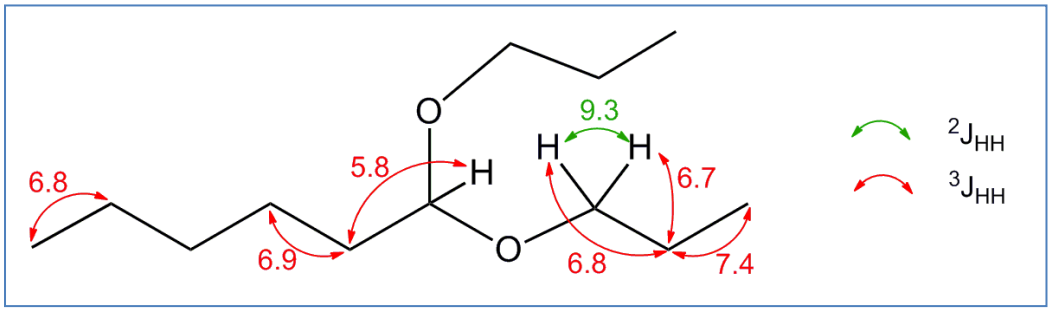
Table of NMR data of 1,1-dipropoxyhexane (chemical shift and coupling constant data from ^{1}H and ^{13}C NMR spectra with the observed HMBC and NOESY interactions)

Position	δ_H (Integral, m, *J* (Hz))	δ_C (ppm) C-13 {H}	HMBC[a]	NOESY
1	4.48 (1 H, t, $^{3}J_{1,2}$ = 5.8)	103.3 (1 C)	3, 1'	2
2	1.60 (2 H, td, $^{3}J_{2,3}$ = 6.7, $^{3}J_{1,2}$ = 5.8)[b]	33.6 (1 C)	1, 3, 4	1, 3
3	1.34[c]	24.6 (1 C)	1, 2, 4	2
4	1.30[c]	31.8 (1 C)	3, 5, 6	/
5	1.32[c]	22.7 (1 C)	4, 6	6
6	0.89 (3 H, t, $^{3}J_{5,6}$ = 6.8)	14.2 (1 C)	4, 5	5
1'a	3.38 (2 H, dt, $^{2}J_{1'a,1'b}$ = 9.3, $^{3}J_{1'a,2'}$ = 6.8)	67.2 (2 C)	1, 2', 3'	2'
1'b	3.54 (2 H, dt, $^{2}J_{1'a,1'b}$ = 9.3, $^{3}J_{1'b,2'}$ = 6.7)			
2'	1.59 (4 H, qt, $^{3}J_{2',3'}$ = 7.4, $^{3}J_{1'a,2'}$ = 6.8, $^{3}J_{1'b,2'}$ = 6.7)[b]	23.3 (2 C)	1', 3'	1', 3'
3'	0.94 (6 H, t, $^{3}J_{2',3'}$ = 7.4)	10.9 (2 C)	1', 2'	2'

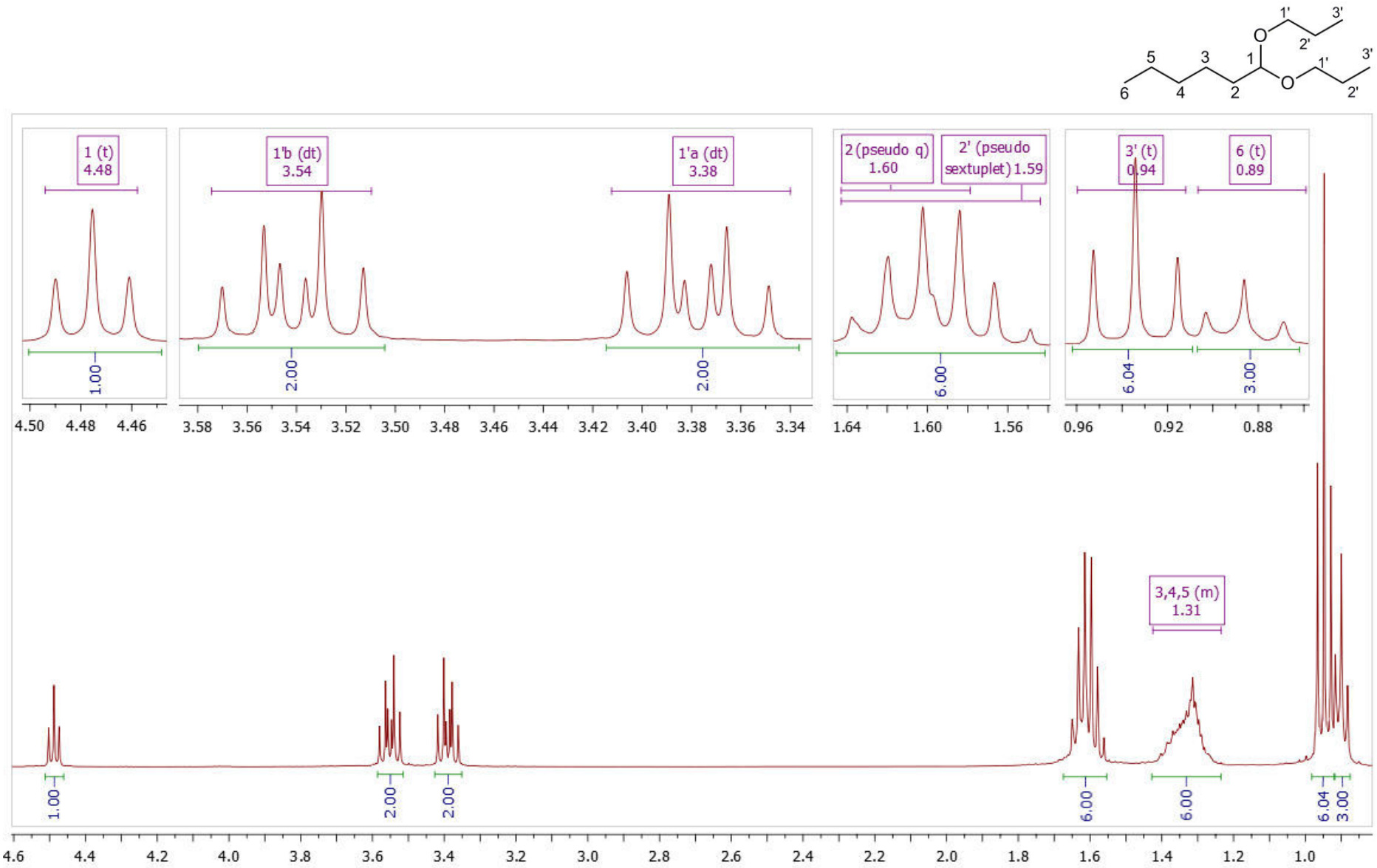
[a]Correlation between the hydrogen in this row and the carbon in the listed position.
[b]Overlapped signals (range: 1.54-1.65 ppm, 6 H). Chemical shifts were determined from HSQC and HMBC spectra. Coupling constants were determined from the appropriate decoupled spectra.
[c]Overlapped signals (range: 1.25-1.43 ppm, 6 H). Chemical shifts were determined from HSQC and HMBC spectra.



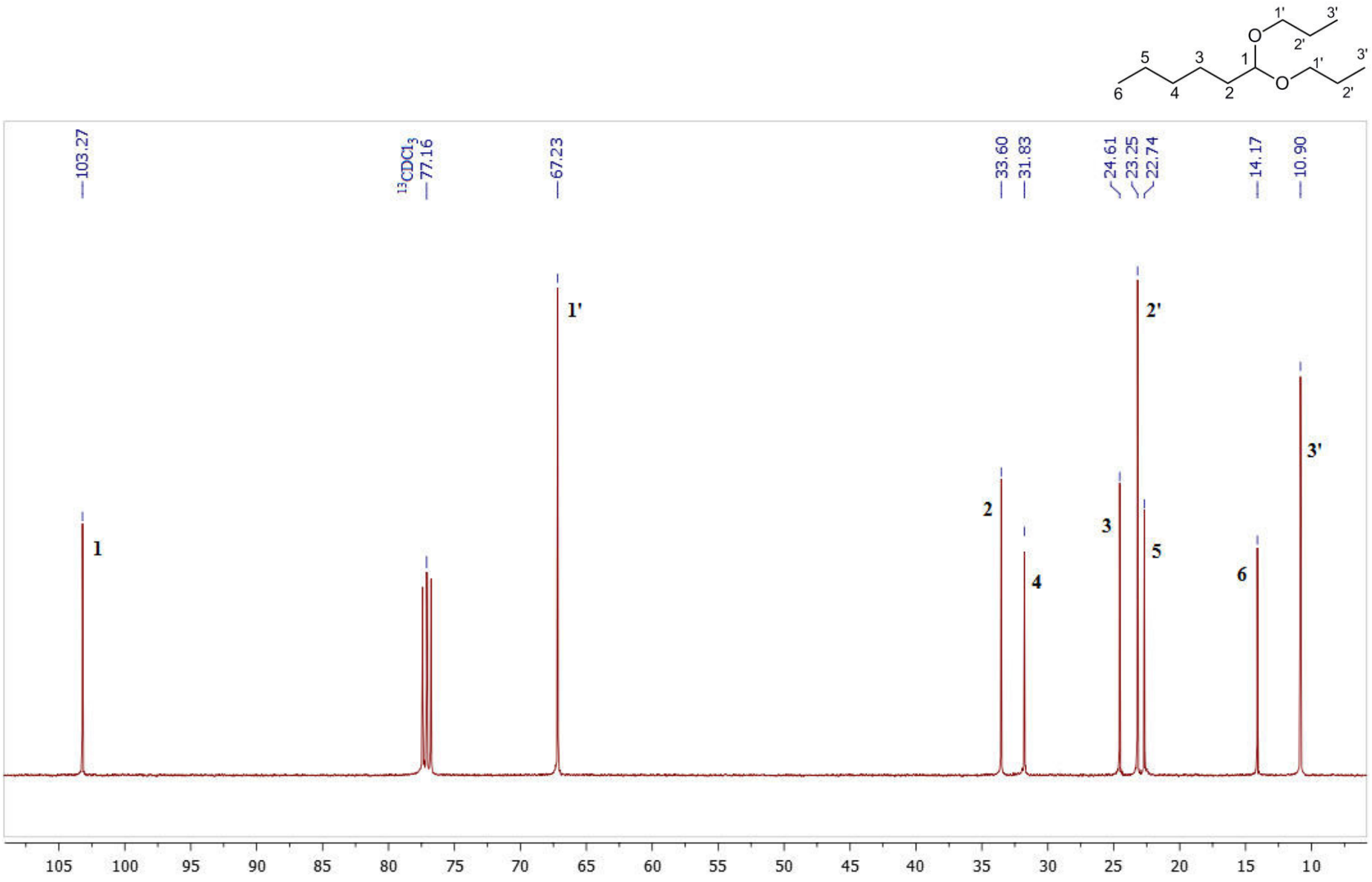
Scheme with key HMBC and NOESY interactions



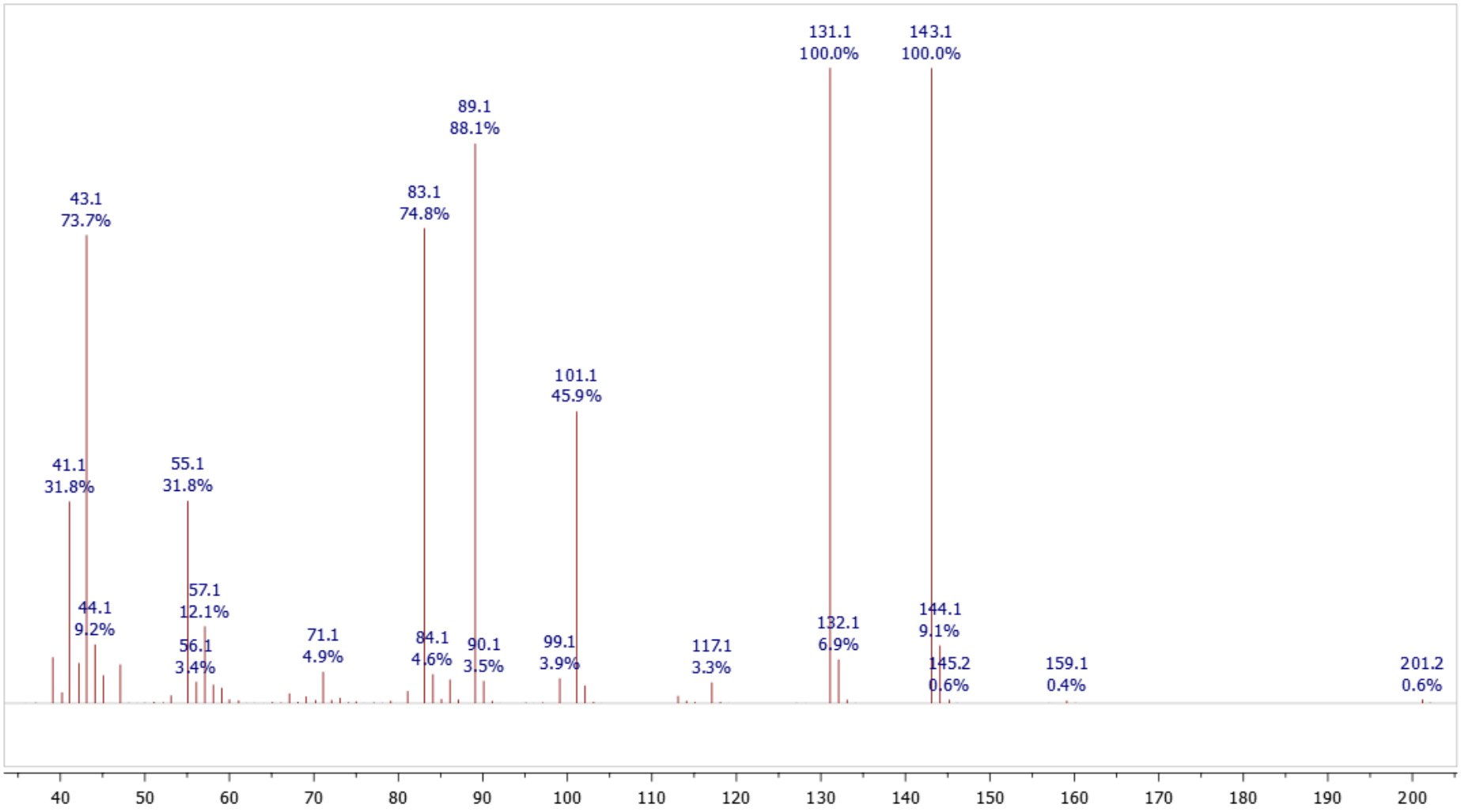
Analysis of ^{1}H-^{1}H coupling constants



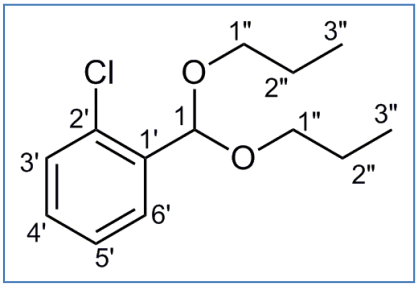
^{1}H-NMR (400 MHz, $CDCl_3$) spectrum of 1,1-dipropoxyhexane and the corresponding expansions with signal assignment



^{13}C-NMR (100 MHz, $CDCl_3$) spectrum of 1,1-dipropoxyhexane with signal assignment



EI-MS spectrum of 1,1-dipropoxyhexane



1-(bis(propoxy)methyl)-2-chlorobenzene

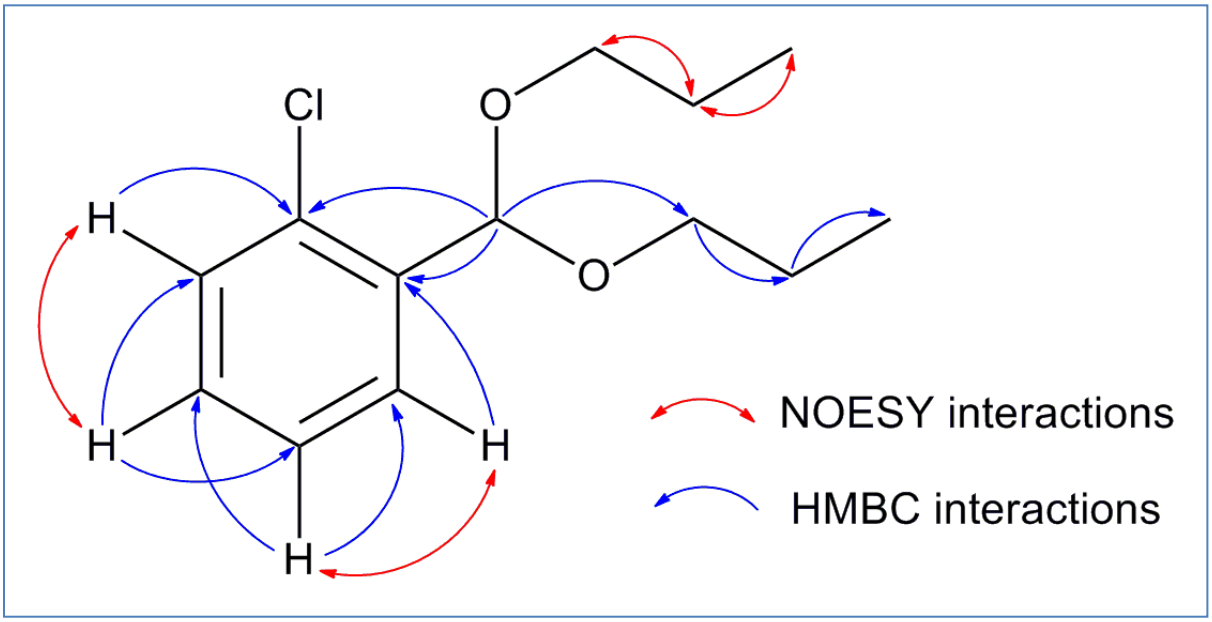
Table of NMR data of 1-(bis(propoxy)methyl)-2-chlorobenzene (chemical shift and coupling constant data from ^{1}H and ^{13}C NMR spectra with the observed HMBC and NOESY interactions)

Position	δ_H (Integral, m, *J* (Hz))	δ_C (ppm) C-13 {H}	HMBC[a]	NOESY
1	5.72 (1 H, dd, $^{4}J_{1,6'} = 0.5$, $^{6}J_{1,4'} = 0.4$)[b]	99.4 (1 C)	1', 6', 1"	/
1'	/	133.3 (1 C)	/	/
2'	/	136.5 (1 C)	/	/
3'	7.35 (1 H, m, AB<u>C</u>D)[c]	129.60 (1 C)	1', 2', 4'	4'
4'	7.28 (1 H, m, A<u>B</u>CD, $^{6}J_{1,4'} = 0.4$)[c]	126.7 (1 C)	2', 3', 5'	3'
5'	7.25 (1 H, m, <u>A</u>BCD)[c]	129.64 (1 C)	1', 4', 6'	6'
6'	7.67 (1 H, m, ABC<u>D</u>, $^{4}J_{1,6'} = 0.5$)[c]	128.2 (1 C)	1, 1', 5'	5'
1"a	3.49 (2 H, dt, $^{2}J_{1"a,1"b} = 9.2$, $^{3}J_{1"a,2"} = 6.7$)	68.5 (2 C)	1, 2", 3"	2"
1"b	3.57 (2 H, dt, $^{2}J_{1"a,1"b} = 9.2$, $^{3}J_{1"b,2"} = 6.7$)			
2"	1.63 (4 H, qt, $^{3}J_{2",3"} = 7.4$, $^{3}J_{1"b,2"} = {}^{3}J_{1"a,2"} = 6.7$)[b]	23.1 (2 C)	1", 3"	1", 3"
3"	0.94 (6 H, t, $^{3}J_{2",3"} = 7.4$)	10.9 (2 C)	1", 2"	2"

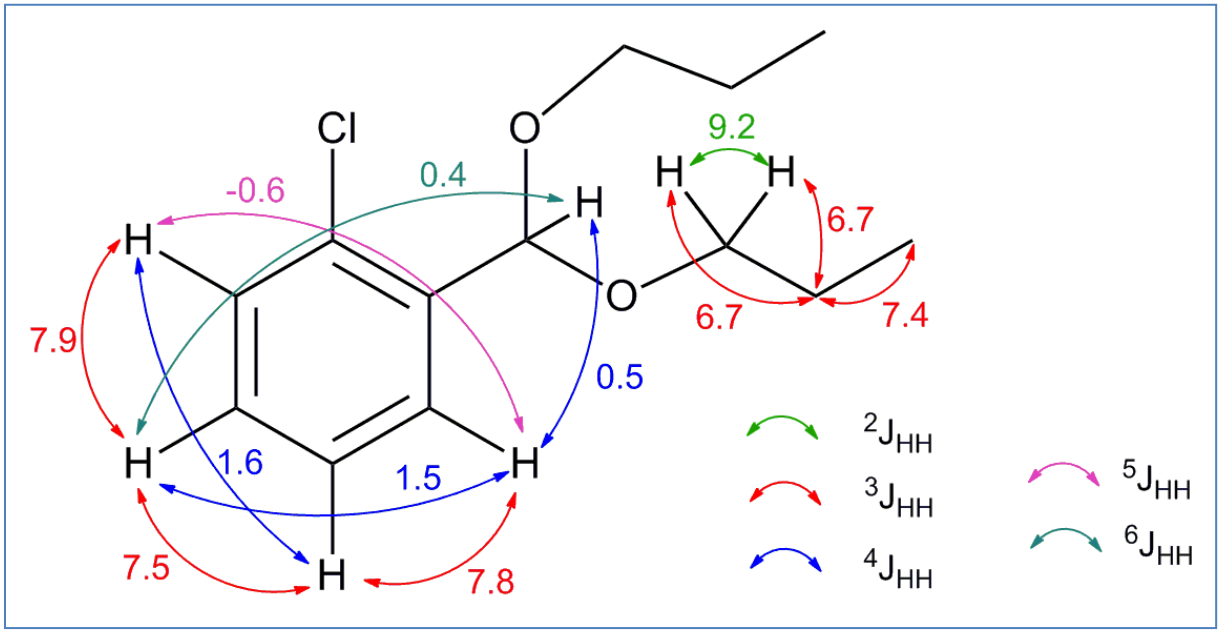
[a]Correlation between the hydrogen in this row and the carbon in the listed position.

[b]Coupling constants (and multiplicity) were determined from appropriate ^{1}H selective homodecoupled spectra. Values of coupling constants lower than 0.5 Hz were usually observed as a broadening of the corresponding ^{1}H NMR signals; they were disclosed only in a series of selective homodecoupling experiments.

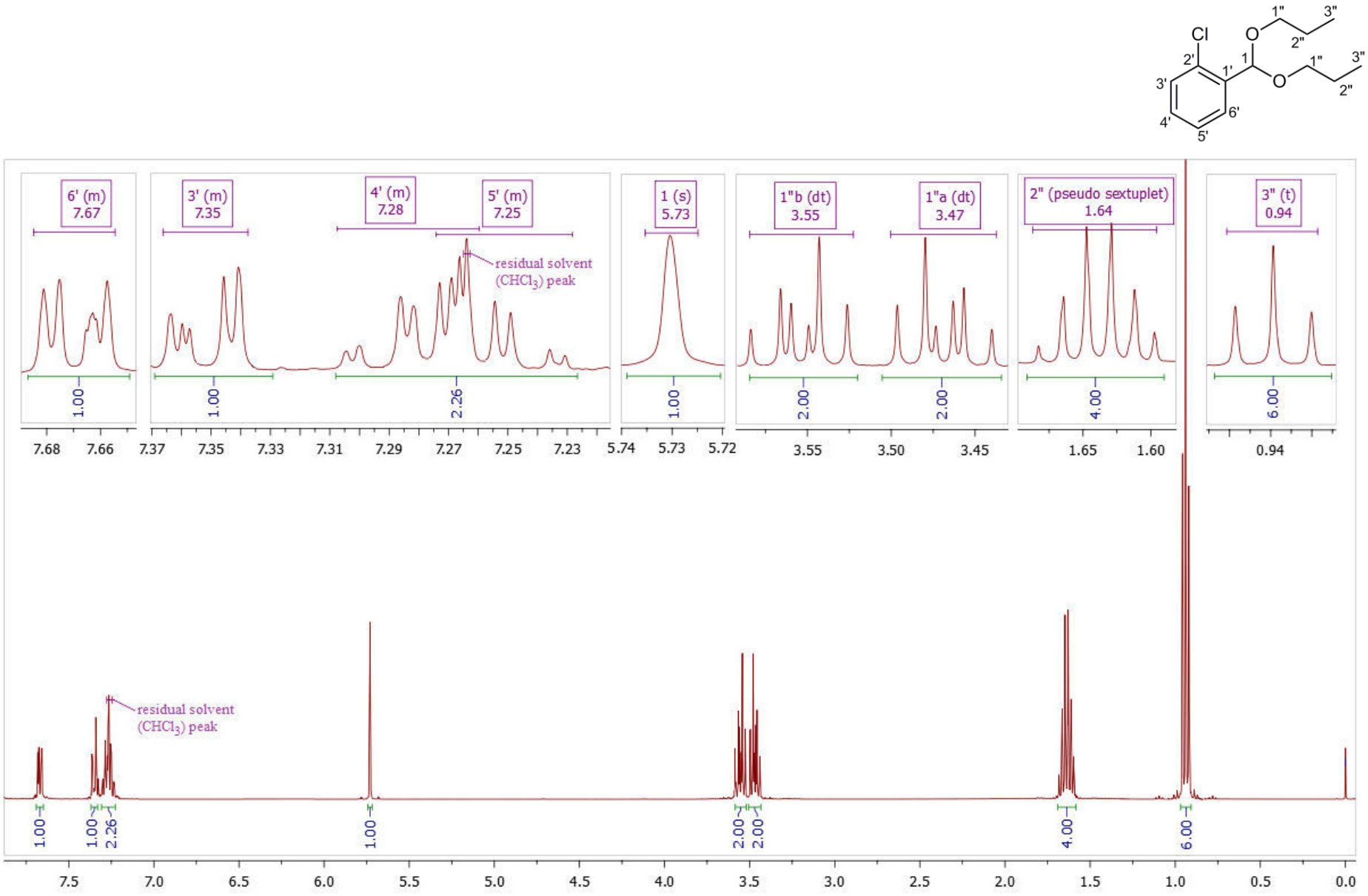
[c]$J_{AB} = 7.5$ Hz, $J_{AC} = 1.6$ Hz, $J_{AD} = 7.8$ Hz, $J_{BC} = 7.9$ Hz, $J_{BD} = 1.5$ Hz, $J_{CD} = -0.6$ Hz. The data from the spectrum obtained in a selective homodecoupling experiment (acetal proton decoupled) and "WinDNMR" simulation were used for the determination of the coupling constants. More accurate values of δ_H: 7.2543 (A/5'), 7.2825 (B/4'), 7.3495 (C/3'), 7.669 (D/6').



Scheme with key HMBC and NOESY interactions

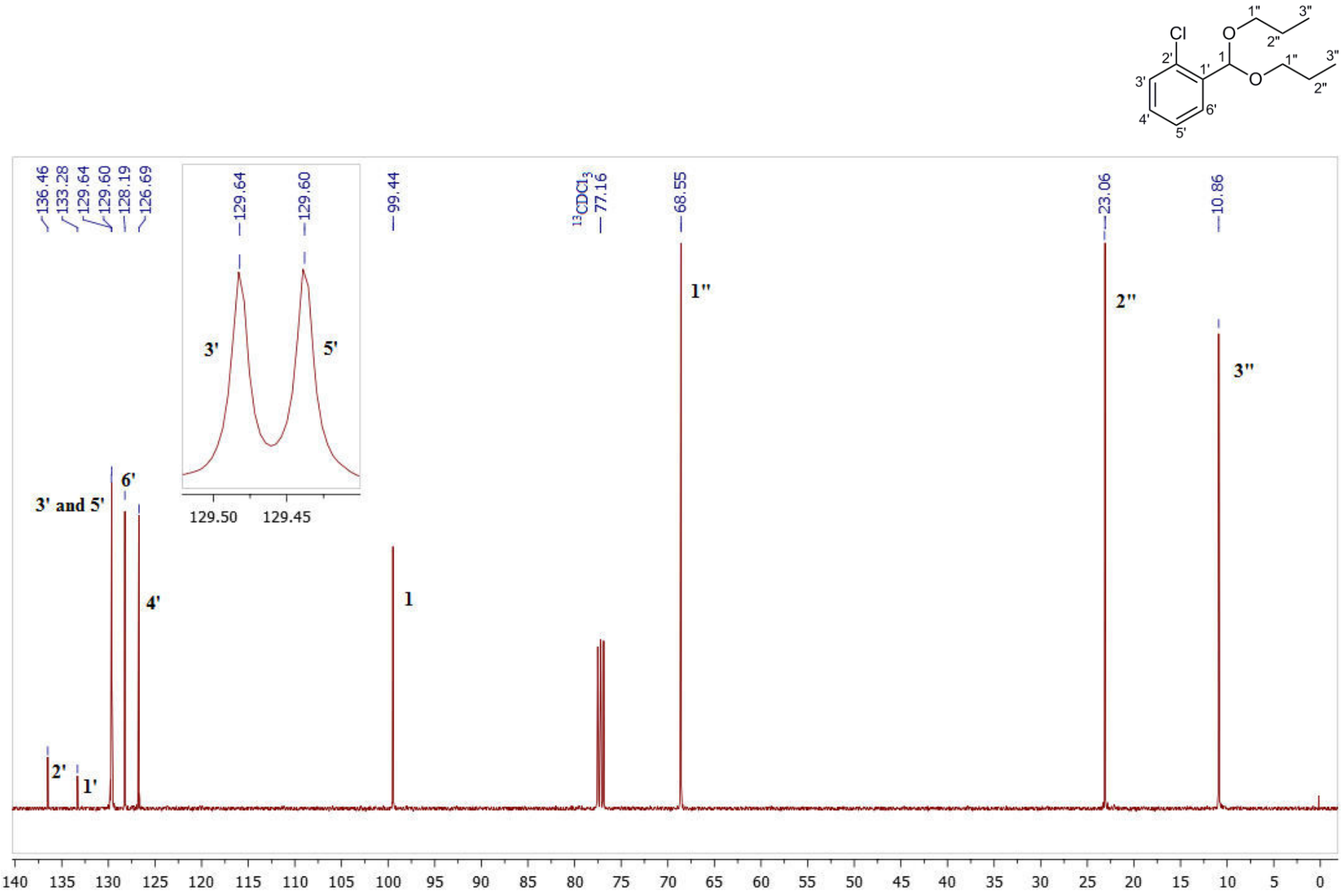


Analysis of ^{1}H-^{1}H coupling constants

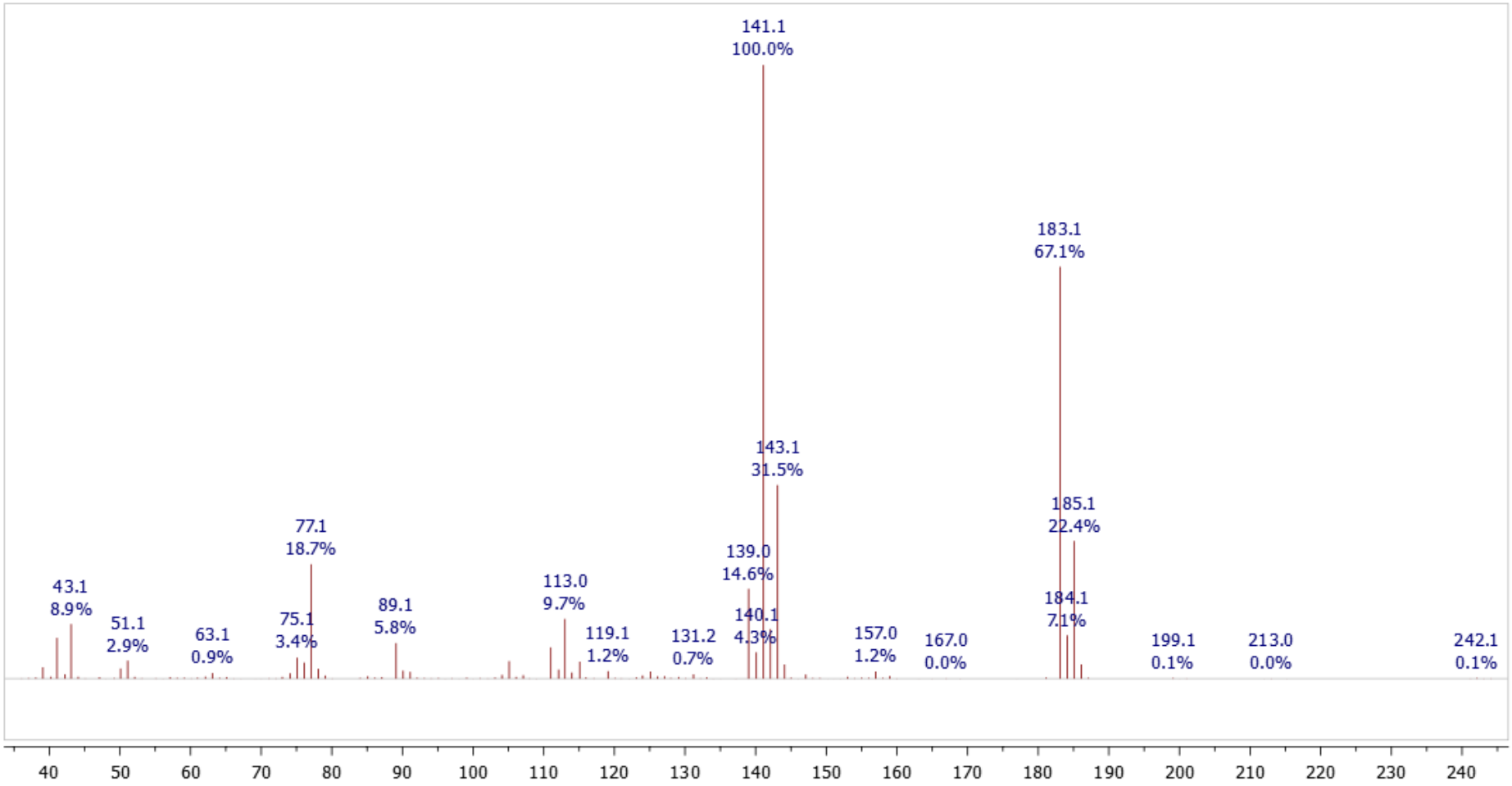


^{1}H-NMR (400 MHz, $CDCl_3$) spectrum of 1-(bis(propoxy)methyl)-2-chlorobenzene and the corresponding expansions with signal assignment

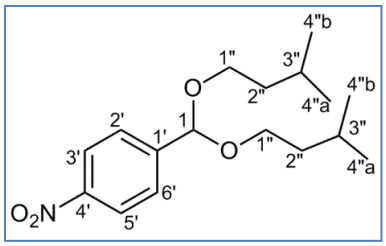
Expansions of ^{1}H-NMR spectrum of 1-(bis(propoxy)methyl)-2-chlorobenzene corresponding to protons attached to the aromatic ring (top); the corresponding expansions obtained in homodecoupling experiments (acetal proton decoupled; middle), and WinDNMR simulation of the mentioned decoupled spectrum (bottom)



^{13}C-NMR (100 MHz, $CDCl_3$) spectrum of 1-(bis(propoxy)methyl)-2-chlorobenzene with signal assignment and the corresponding expansion



EI-MS spectrum of 1-(bis(propoxy)methyl)-2-chlorobenzene



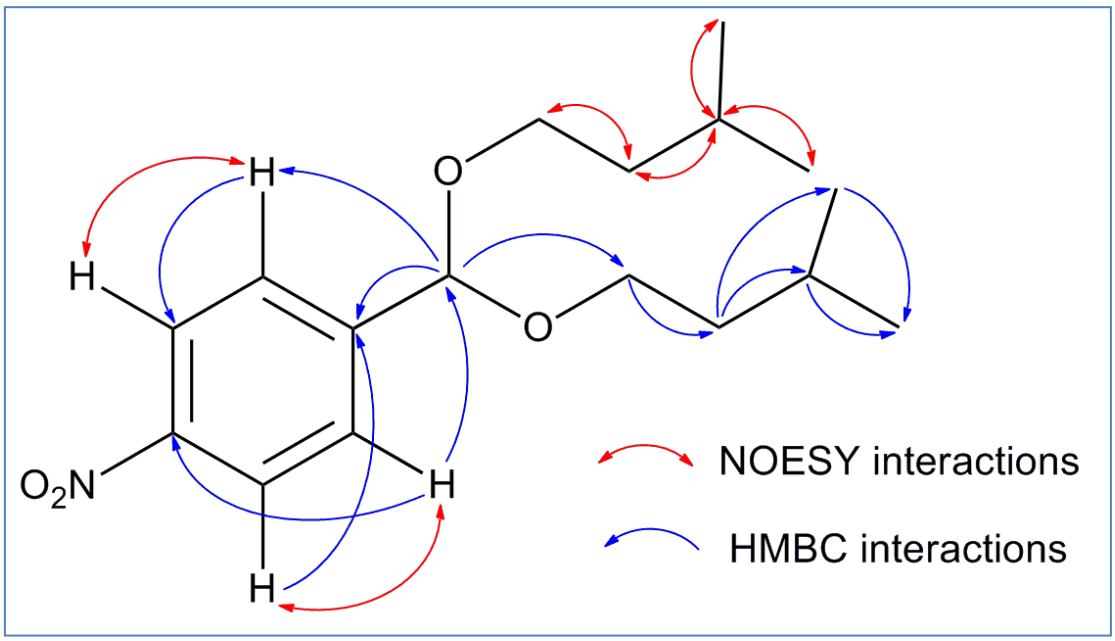
1-(bis(isopentyloxy)methyl)-4-nitrobenzene

Table of NMR data of 1-(bis(isopentyloxy)methyl)-4-nitrobenzene (chemical shift and coupling constant data from ^{1}H and ^{13}C NMR spectra with the observed HMBC and NOESY interactions)

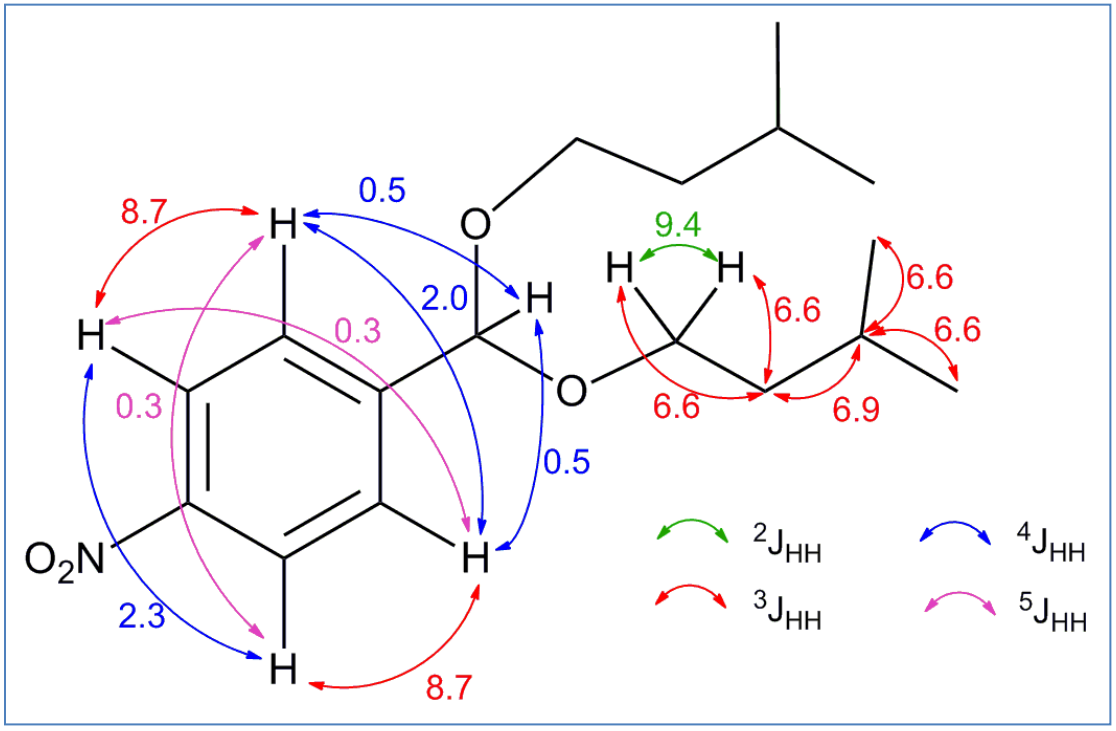
Position	δ_H (Integral, m, *J* (Hz))	δ_C (ppm) C-13 {H}	HMBC[a]	NOESY
1	5.57 (1 H, t, $^{4}J_{1,2'} = {}^{4}J_{1,6'} = 0.5$)[b]	100.4 (1 C)	2', 6', 1"	/
1'	/	146.3 (1 C)	/	/
2', 6' (AA')	7,65 (2 H, m, AA'BB') ($^{3}J_{AB} = 8.7$, $^{4}J_{AA'} = 2.0$, $^{4}J_{BB'} = 2.3$, $^{5}J_{AB'/A'B} = 0.3$, $^{4}J_{AA',1} = 0.5$)[b]	127.9 (2 C)	1, 3'/5', 4', (6'/2')	3'/5'
3', 5' (BB')	8,22 (2 H, m, AA'BB') ($^{3}J_{AB} = 8.7$, $^{4}J_{AA'} = 2.0$, $^{4}J_{BB'} = 2.3$, $^{5}J_{AB'/A'B} = 0.3$)[b]	123.5 (2 C)	1', 4', (5'/3')	2'/6'
4'	/	148.0 (1 C)	/	/
1"a	3.51 (2 H, dt, $^{2}J_{1"a,1"b} = 9.4$, $^{3}J_{1"a,2"} = 6.6$)	64.2 (2 C)	1, 2", 3"	2"
1"b	3.54 (2 H, dt, $^{2}J_{1"a,1"b} = 9.4$, $^{3}J_{1"b,2"} = 6.6$)			
2"	1.51 (4 H, dt, $^{3}J_{2",3"} = 6.9$, $^{3}J_{2",1"a} = {}^{3}J_{2",1"b} = 6.6$)[b]	38.6 (2 C)	1", 3", 4"	1"a, 1"b, 3"
3"	1.74 (2 H, t of septuplet, $^{3}J_{3",2"} = 6.9$, $^{3}J_{3",4"a} = {}^{3}J_{3",4"b} = 6.6$, 2H)[b]	25.2 (2 C)	1", 2", 4"	2", 4"a, 4"b
4"a	0.91 (6 H, d, $^{3}J_{4"a,3"} = 6.6$)	22.8 (2 C)	2", 3", 4"b	3"
4"b	0.90 (6 H, d, $^{3}J_{4"b,3"} = 6.6$)	22.7 (2 C)	2", 3", 4"a	3"

[a]Correlation between the hydrogen in this row and the carbon in the listed position.

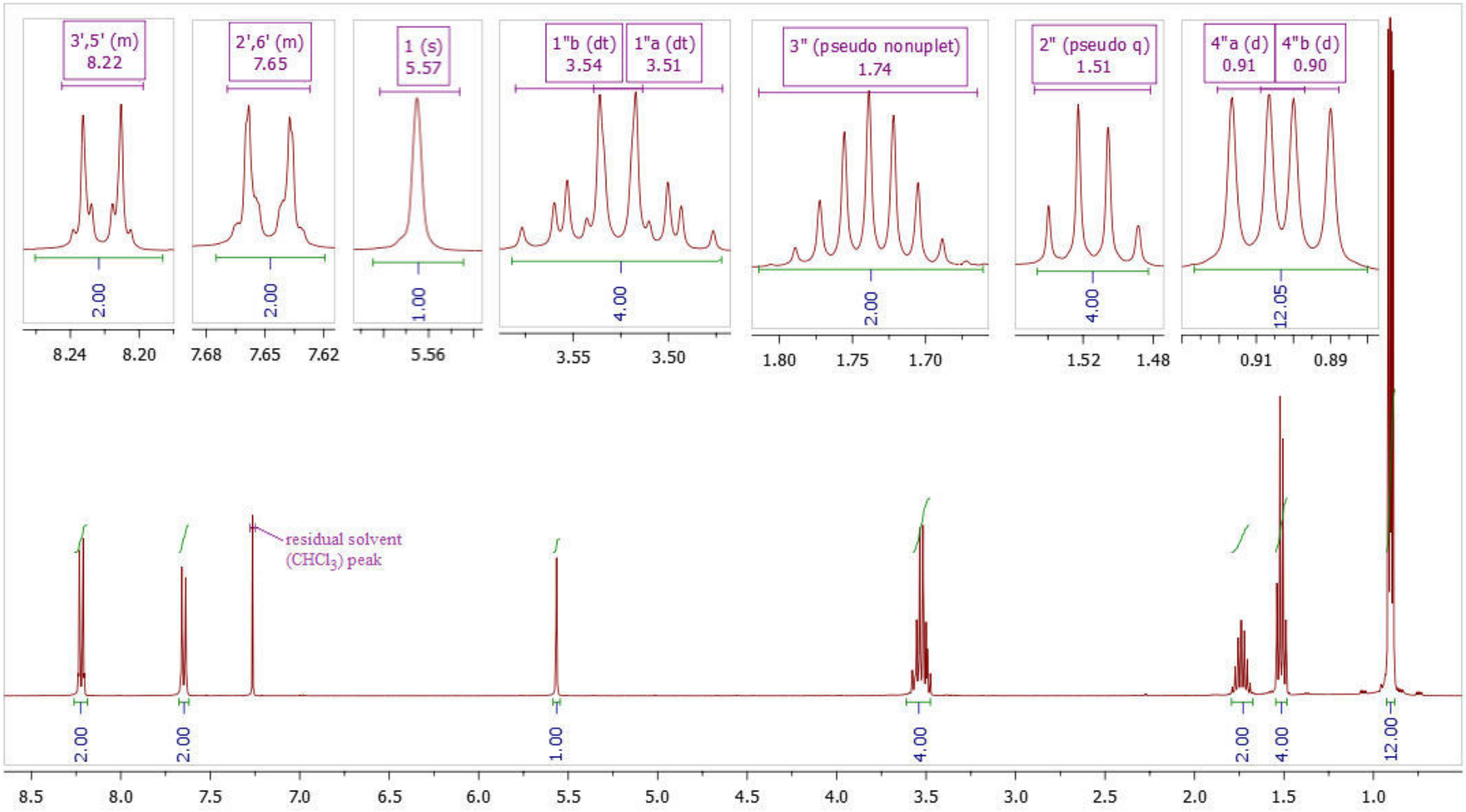
[b]Coupling constants (and multiplicity) were determined from appropriate ^{1}H selective homodecoupled spectra. Values of coupling constants lower than 0.5 Hz were usually observed as a broadening of the corresponding ^{1}H NMR signals; they were disclosed only in a series of selective homodecoupling experiments.



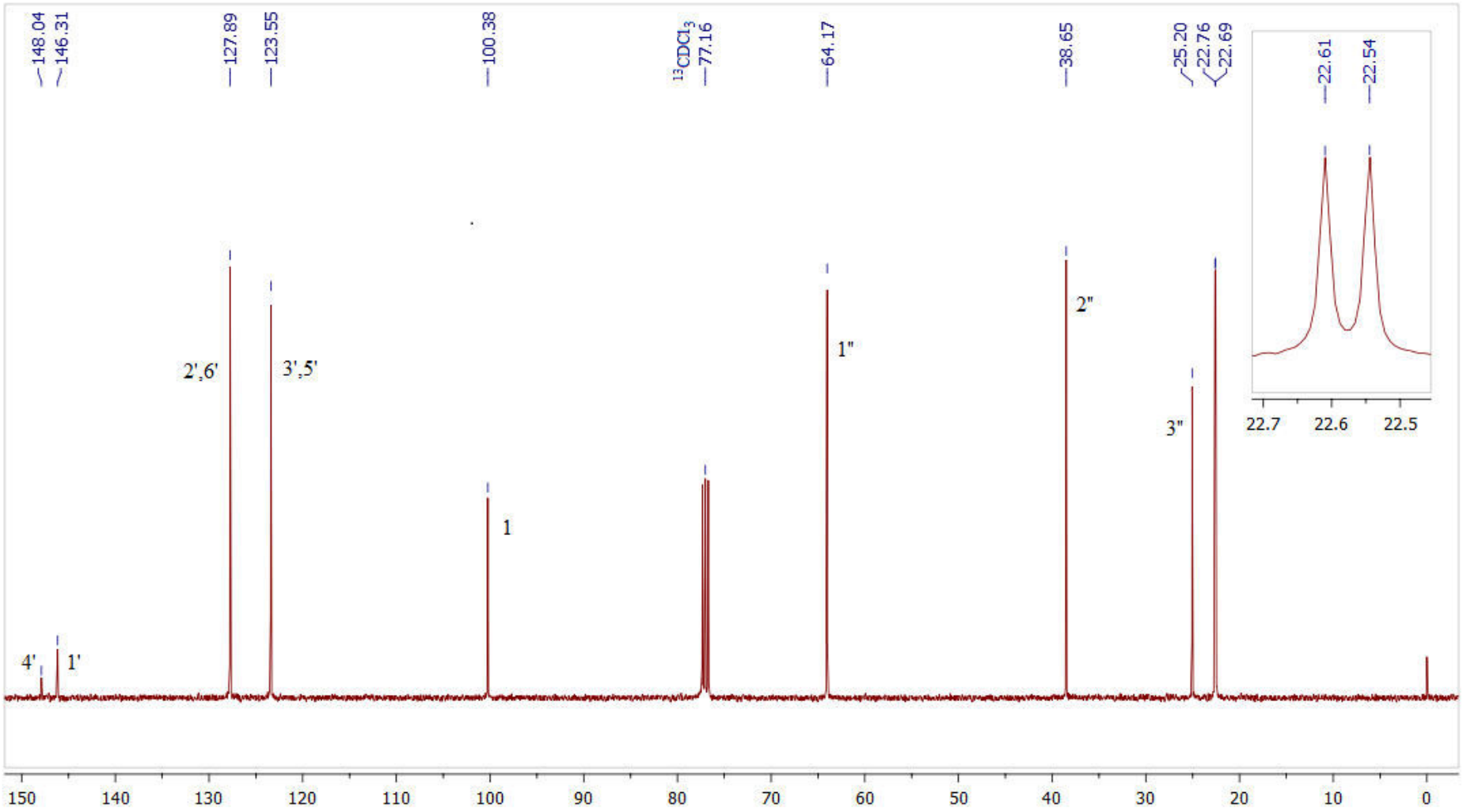
Scheme with key HMBC and NOESY interactions



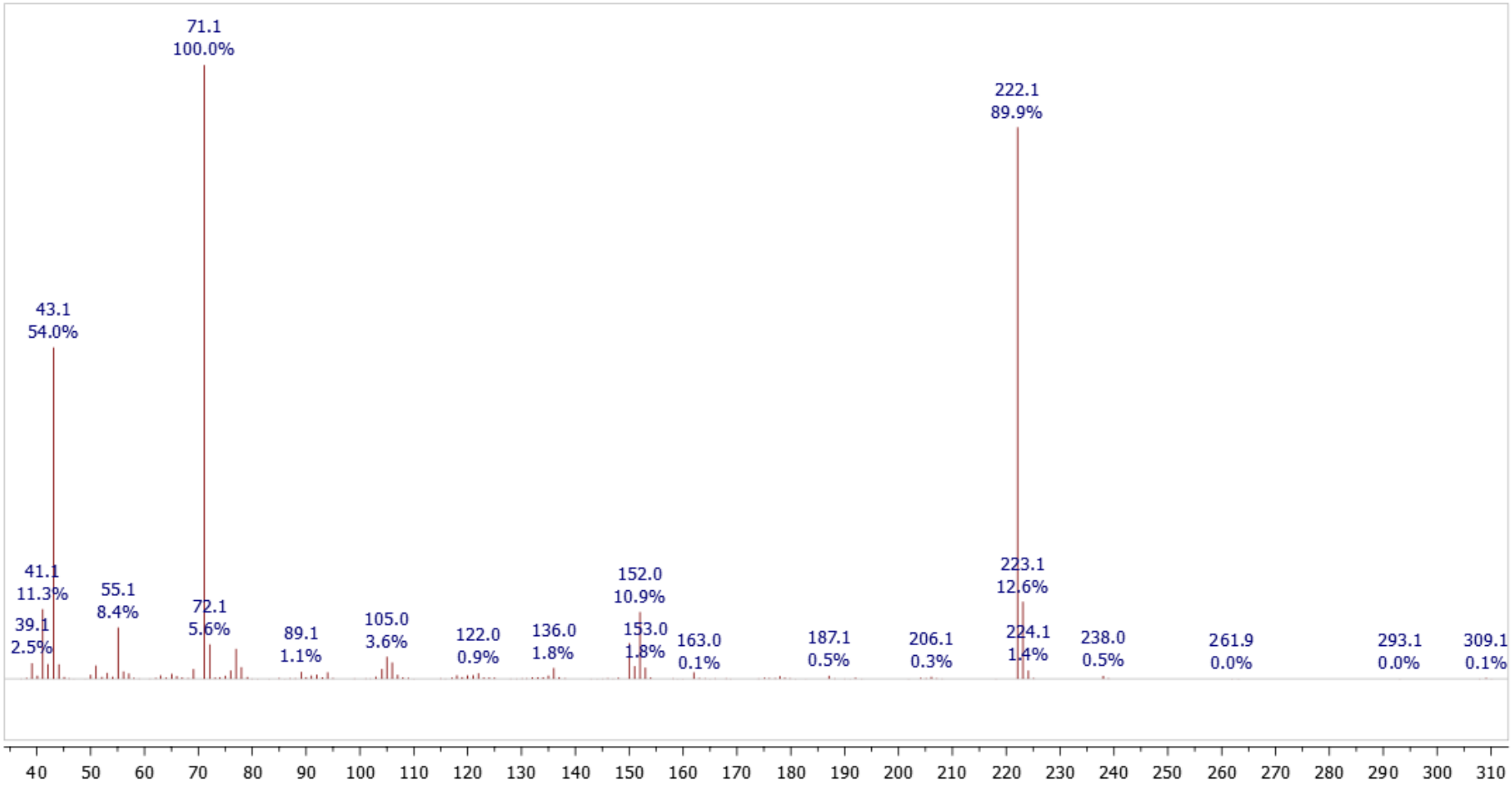
Analysis of ^{1}H-^{1}H coupling constants



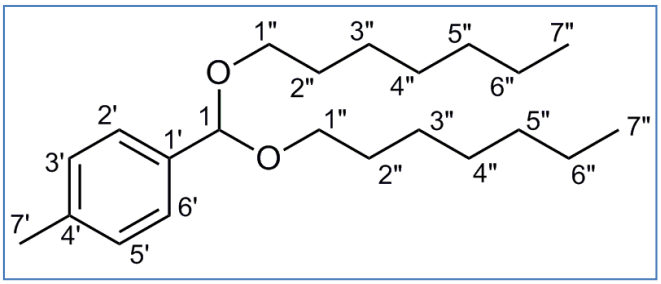
^{1}H-NMR (400 MHz, $CDCl_3$) spectrum of 1-(bis(isopentyloxy)methyl)-4-nitrobenzene and the corresponding expansions with signal assignment



^{13}C-NMR (100 MHz, $CDCl_3$) spectrum of 1-(bis(isopentyloxy)methyl)-4-nitrobenzene with signal assignment and the corresponding expansion



EI-MS spectrum of 1-(bis(isopentyloxy)methyl)-4-nitrobenzene



1-(bis(heptyloxy)methyl)-4-methylbenzene

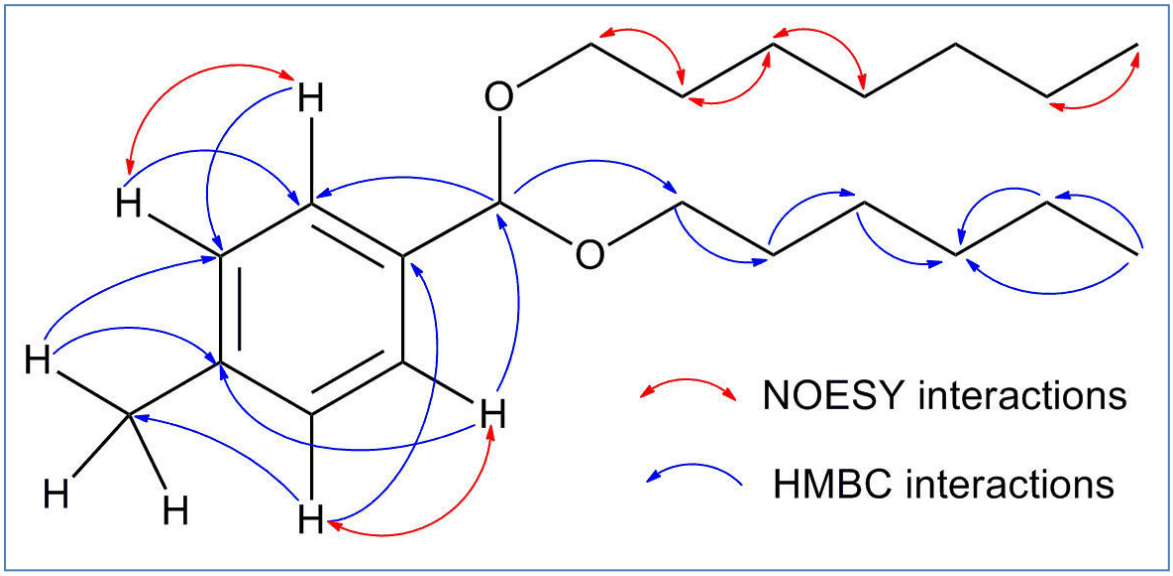
Table of NMR data of 1-(bis(heptyloxy)methyl)-4-methylbenzene (chemical shift and coupling constant data from ^{1}H and ^{13}C NMR spectra with the observed HMBC and NOESY interactions)

Position	δ_H (Integral, m, *J* (Hz))	δ_C (ppm) C-13 {H}	HMBC[a]	NOESY
1	5.48 (1 H, t, $^{4}J_{1,2'} = {}^{4}J_{1,6'} = 0.5$, 1H)[b]	101.6 (1 C)	2', 6', 1"	/
1'	/	136.3 (1 C)	/	/
2', 6' (AA')	7.35 (2 H, m, AA'BB') ($^{3}J_{AB} = 7.9$, $^{4}J_{AA'} = 1.7$, $^{4}J_{BB'} = 2.0$, $^{5}J_{AB'/A'B} = 0.3$, $^{4}J_{AA',1} = 0.5$)[b]	126.7 (2 C)	1, 4',3'/5', (6'/2')	3'/5'
3', 5' (BB')	7.16 (2 H, m, AA'BB') ($^{3}J_{AB} = 7.9$, $^{4}J_{AA'} = 1.7$, $^{4}J_{BB'} = 2.0$, $^{5}J_{AB'/A'B} = 0.3$, $^{4}J_{BB',7} = 0.3$)[b]	128.9 (2 C)	1',7', 2'/6', (5'/3')	2'/6'
4'	/	138.0 (1 C)	/	/
7'	2.35 (3 H, t, $^{4}J_{7',3'} = {}^{4}J_{7',5'} = 0.3$)[b]	21.3 (1 C)	3', 4', 5'	/
1"a	3.44 (2 H, dt, $^{2}J_{1"a,1"b} = 9.4$, $^{3}J_{1"a,2"} = 6.6$)	65.4 (2 C)	1, 2", 3"	2"
1"b	3.52 (2 H, dt, $^{2}J_{1"a,1"b} = 9.4$, $^{3}J_{1"b,2"} = 6.7$)		1, 2", 3"	2"
2"	1.60 (4 H, tdd, $^{3}J_{2",3"} = 6.9$, $^{3}J_{1"b,2"} = 6.7$, $^{3}J_{1"a,2"} = 6.6$)	29.9 (2 C)	1", 3", 4"	1", 3"
3"	1.35[c]	26.4 (2 C)	4"	2", 4"
4"	1.28[c]	29.3 (2 C)	5"	3"
5"	1.27[c]	32.0 (2 C)	6"	/
6"	1.28[c]	22.8 (2 C)	5", 7"	7"
7"	0.88 (6 H, t, $^{3}J_{6",7"} = 6.9$)	14.2 (2 C)	5", 6"	6"

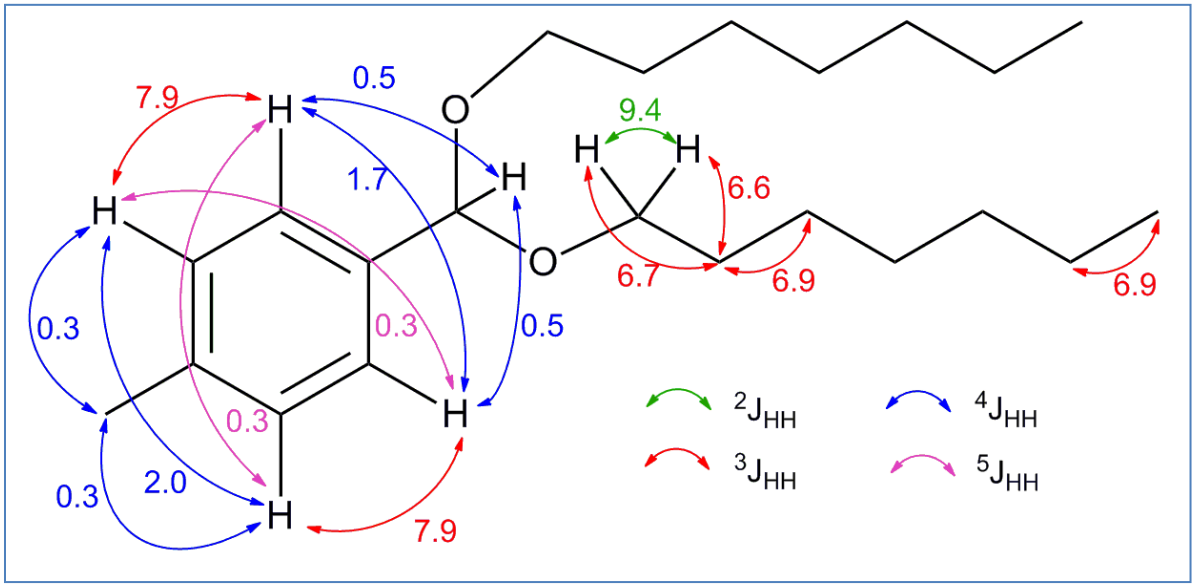
[a]Correlation between the hydrogen in this row and the carbon in the listed position.

[b]Coupling constants (and multiplicity) were determined from appropriate ^{1}H selective homodecoupled spectra. Values of coupling constants lower than 0.5 Hz were usually observed as a broadening of the corresponding ^{1}H NMR signals; they were disclosed only in a series of selective homodecoupling experiments.

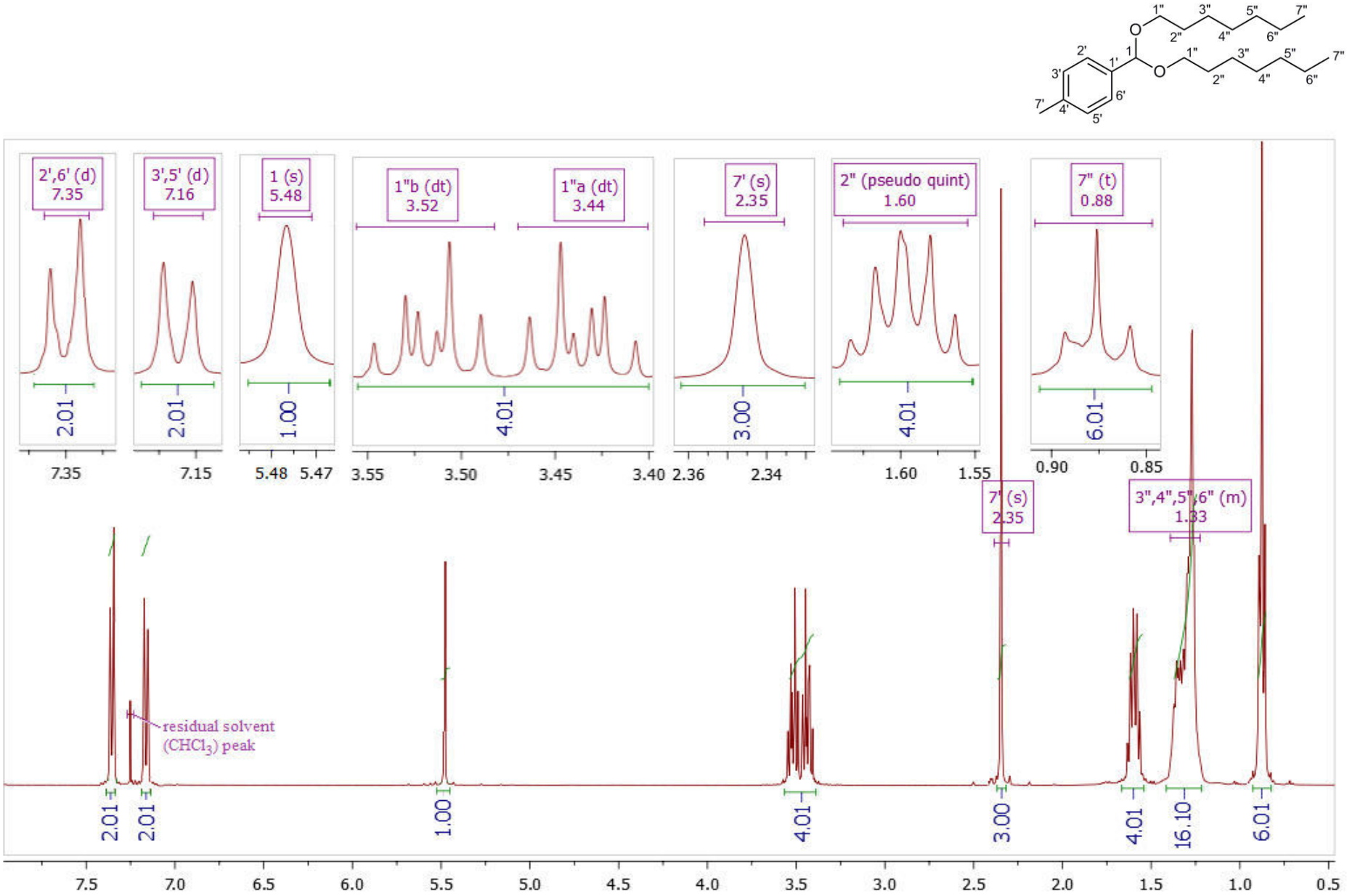
[c]Overlapped signals (range: 1.23-1.40 ppm, 16 H). Chemical shifts were determined from HSQC and HMBC spectra.



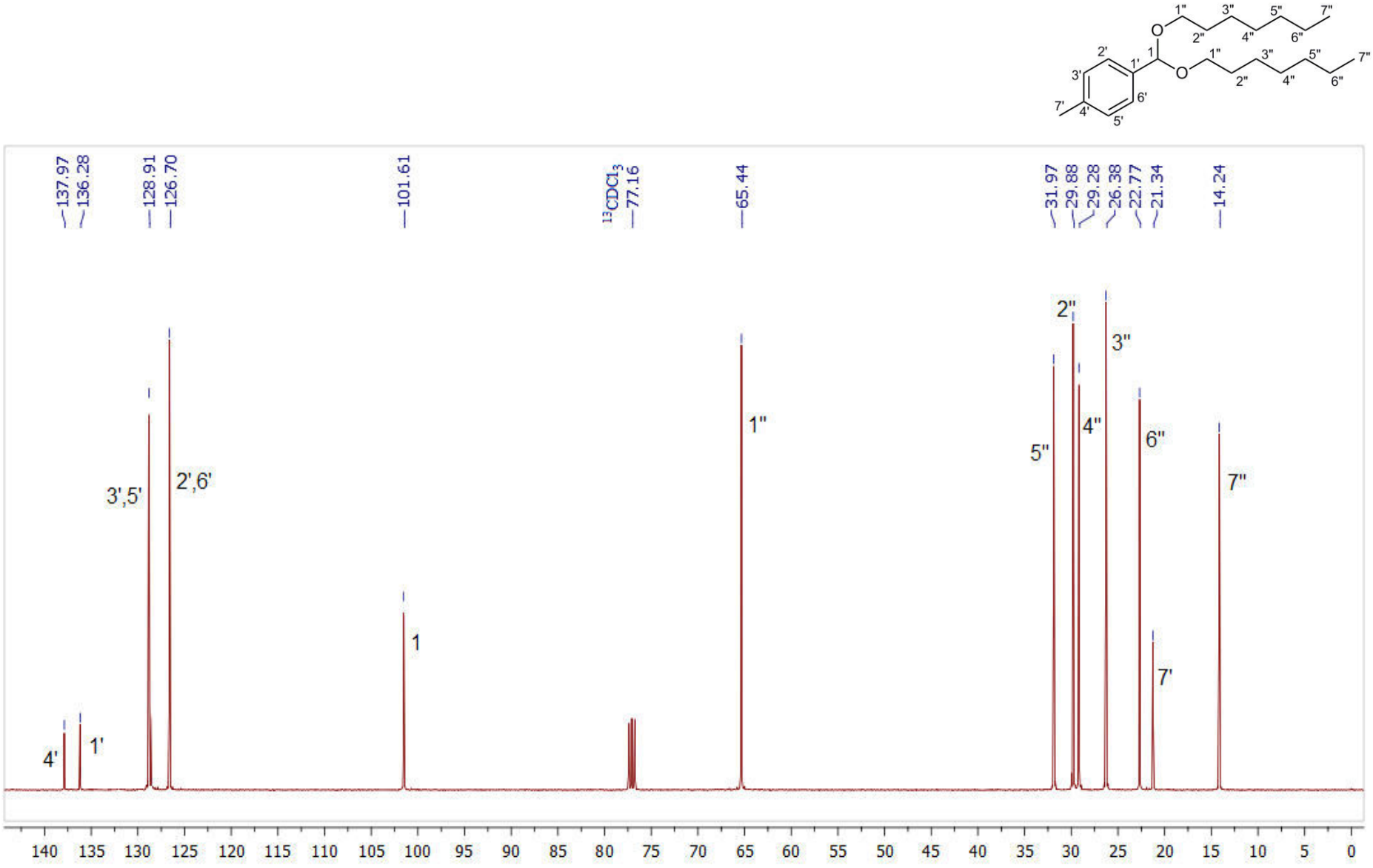
Scheme with key HMBC and NOESY interactions



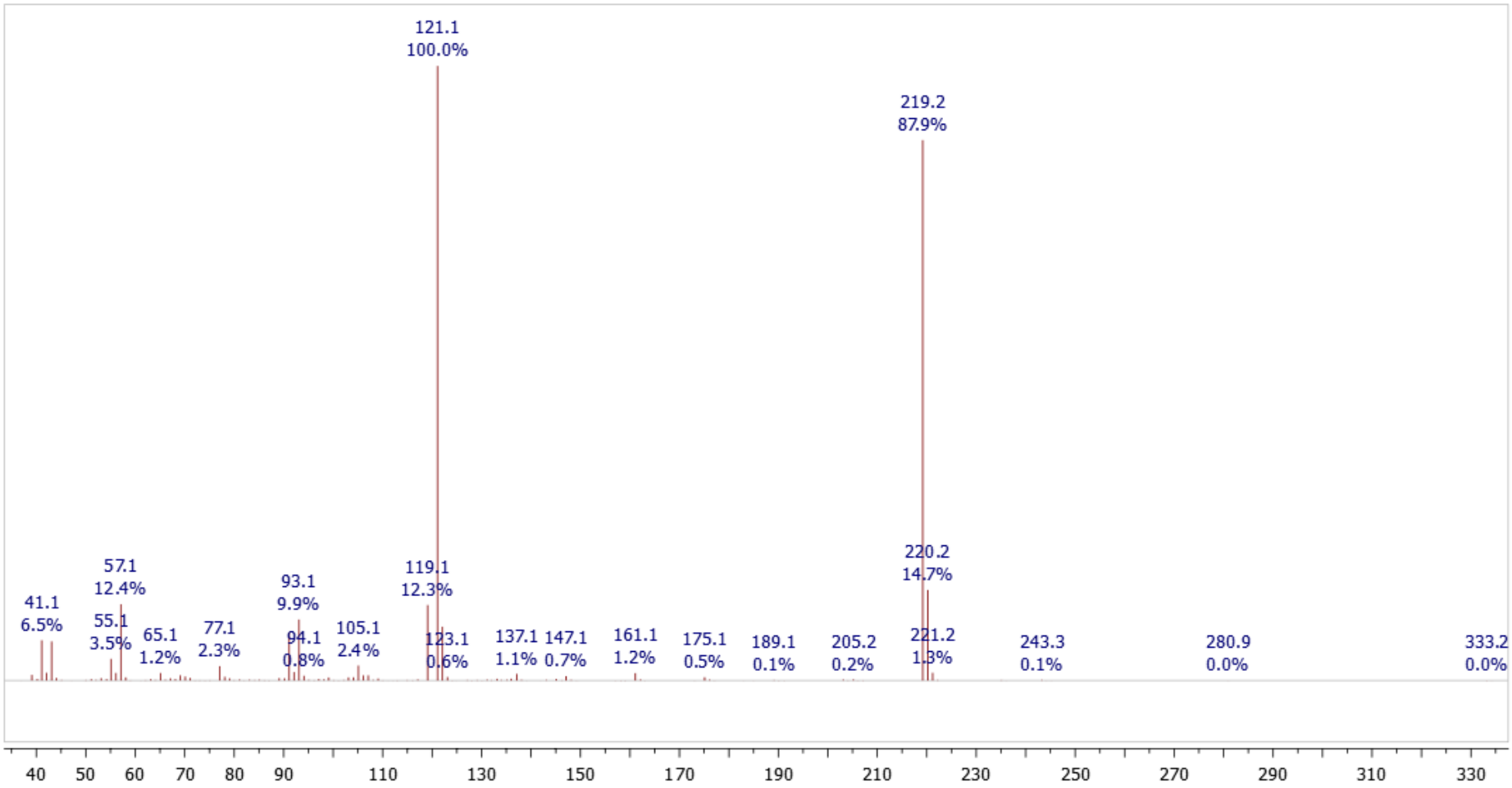
Analysis of ^{1}H-^{1}H coupling constants



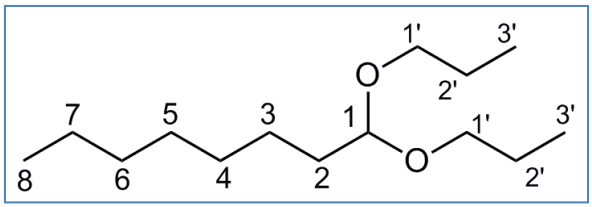
^{1}H-NMR (400 MHz, $CDCl_3$) spectrum of 1-(bis(heptyloxy)methyl)-4-methylbenzene and the corresponding expansions with signal assignment



^{13}C-NMR (100 MHz, $CDCl_3$) spectrum of 1-(bis(heptyloxy)methyl)-4-methylbenzene with signal assignment



EI-MS spectrum of 1-(bis(heptyloxy)methyl)-4-methylbenzene



1,1-dipropoxyoctane

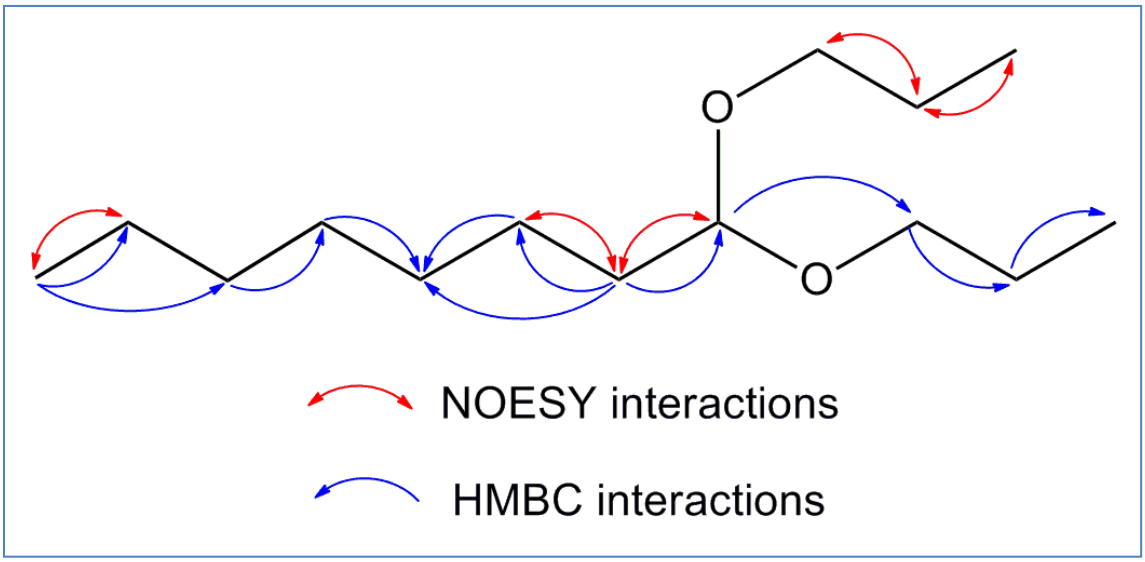
Table of NMR data of 1,1-dipropoxyoctane (chemical shift and coupling constant data from ^{1}H and ^{13}C NMR spectra with the observed HMBC and NOESY interactions)

Position	δ_H (Integral, m, *J* (Hz))	δ_C (ppm) C-13 {H}	HMBC[a]	NOESY
1	4.48 (1 H, t, $^{3}J_{1,2}$ = 5.8)	103.3 (1 C)	3, 1’	2
2	1.61 (2 H, td, $^{3}J_{2,3}$ = 6.7, $^{3}J_{1,2}$ = 5.8)[b]	33.6 (1 C)	1, 3, 4	1, 3
3	1.34[c]	24.9 (1 C)	1, 2, 4	2
4	1.30[c]	29.6 (1 C)	3	/
5	1.28[c]	29.4 (1 C)	6	/
6	1.27[c]	31.9 (1 C)	5, 7, 8	/
7	1.28[c]	22.8 (1 C)	6, 8	8
8	0.88 (3 H, t, $^{3}J_{7,8}$ = 6.7)	14.2 (1 C)	6, 7	7
1’a	3.38 (2 H, dt, $^{2}J_{1'a,1'b}$ = 9.2, $^{3}J_{1'a,2'}$ = 6.8)	67.2 (2 C)	1, 2’, 3’	2’
1’b	3.54 (2 H, dt, $^{2}J_{1'a,1'b}$ = 9.2, $^{3}J_{1'b,2'}$ = 6.7)			
2’	1.59 (4 H, qdd, $^{3}J_{2',3'}$ = 7.4, $^{3}J_{1'a,2'}$ = 6.8, $^{3}J_{1'b,2'}$ = 6.7)[b]	23.3 (2 C)	1’, 3’	1’, 3’
3’	0.94 (6 H, t, $^{3}J_{2',3'}$ = 7.4)	10.9 (2 C)	1’, 2’	2’

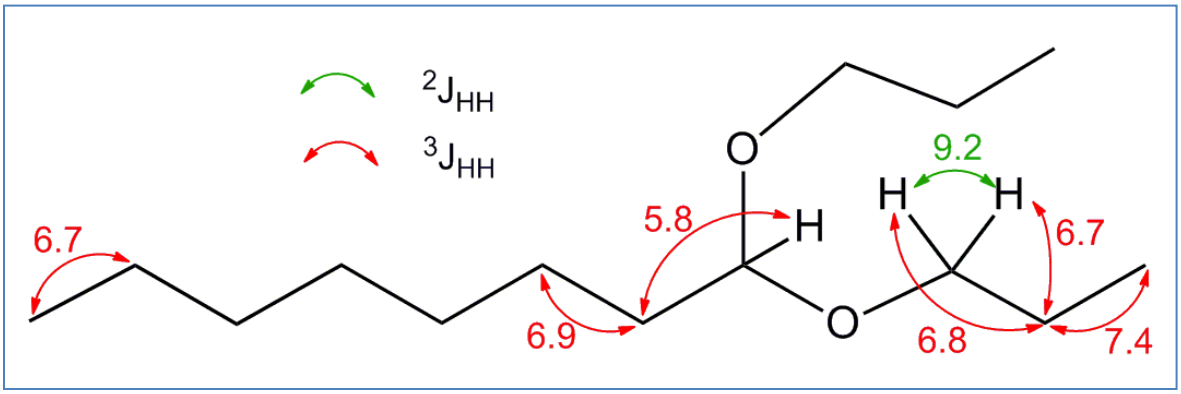
[a]Correlation between the hydrogen in this row and the carbon in the listed position.

[b]Overlapped signals (range: 1.54-1.65 ppm, 6 H). Chemical shifts were determined from HSQC and HMBC spectra. Coupling constants were determined from the appropriate decoupled spectra.

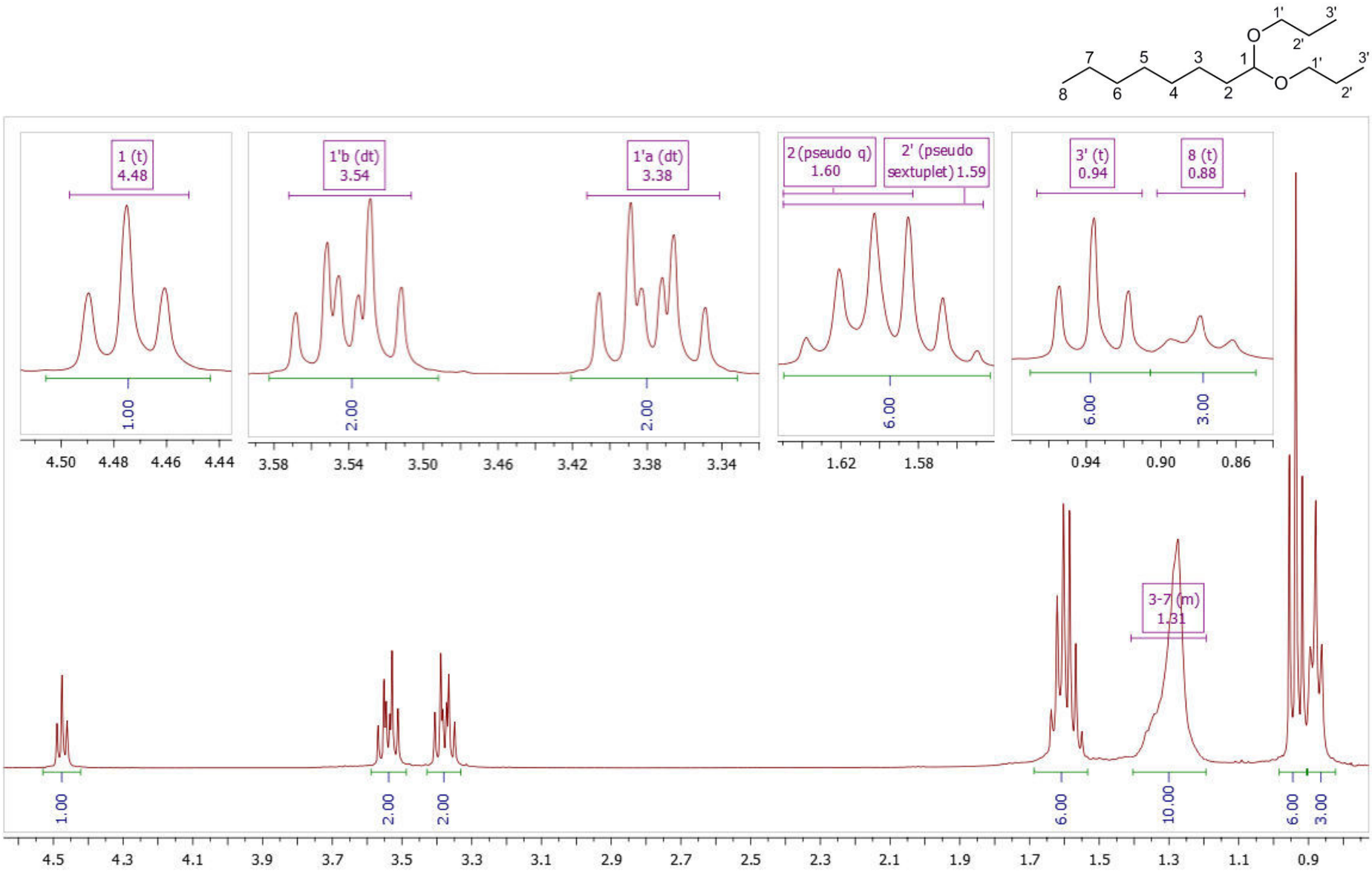
[c]Overlapped signals (range: 1.20-1.40 ppm, 10 H). Chemical shifts were determined from HSQC and HMBC spectra.



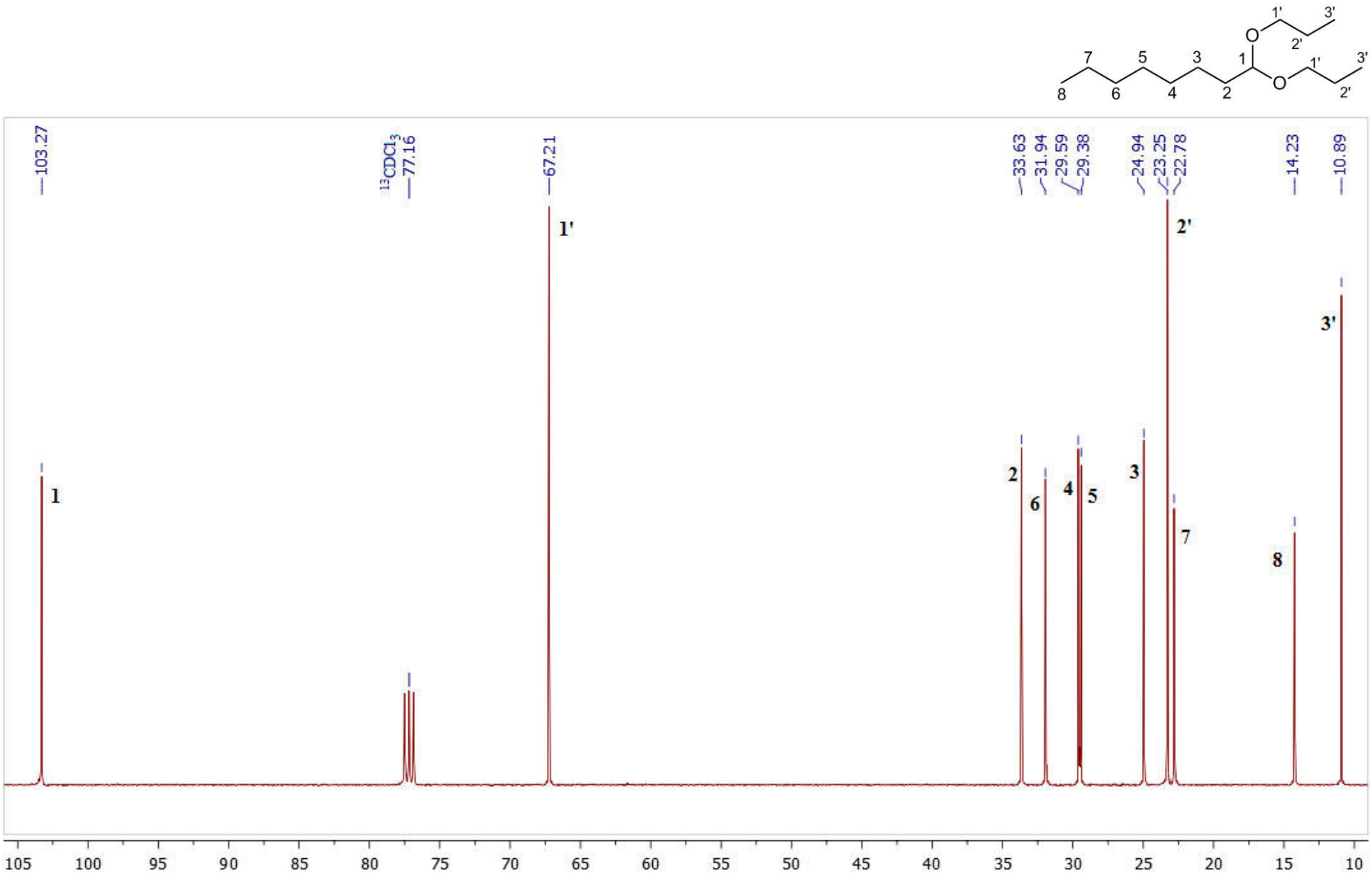
Scheme with key HMBC and NOESY interactions



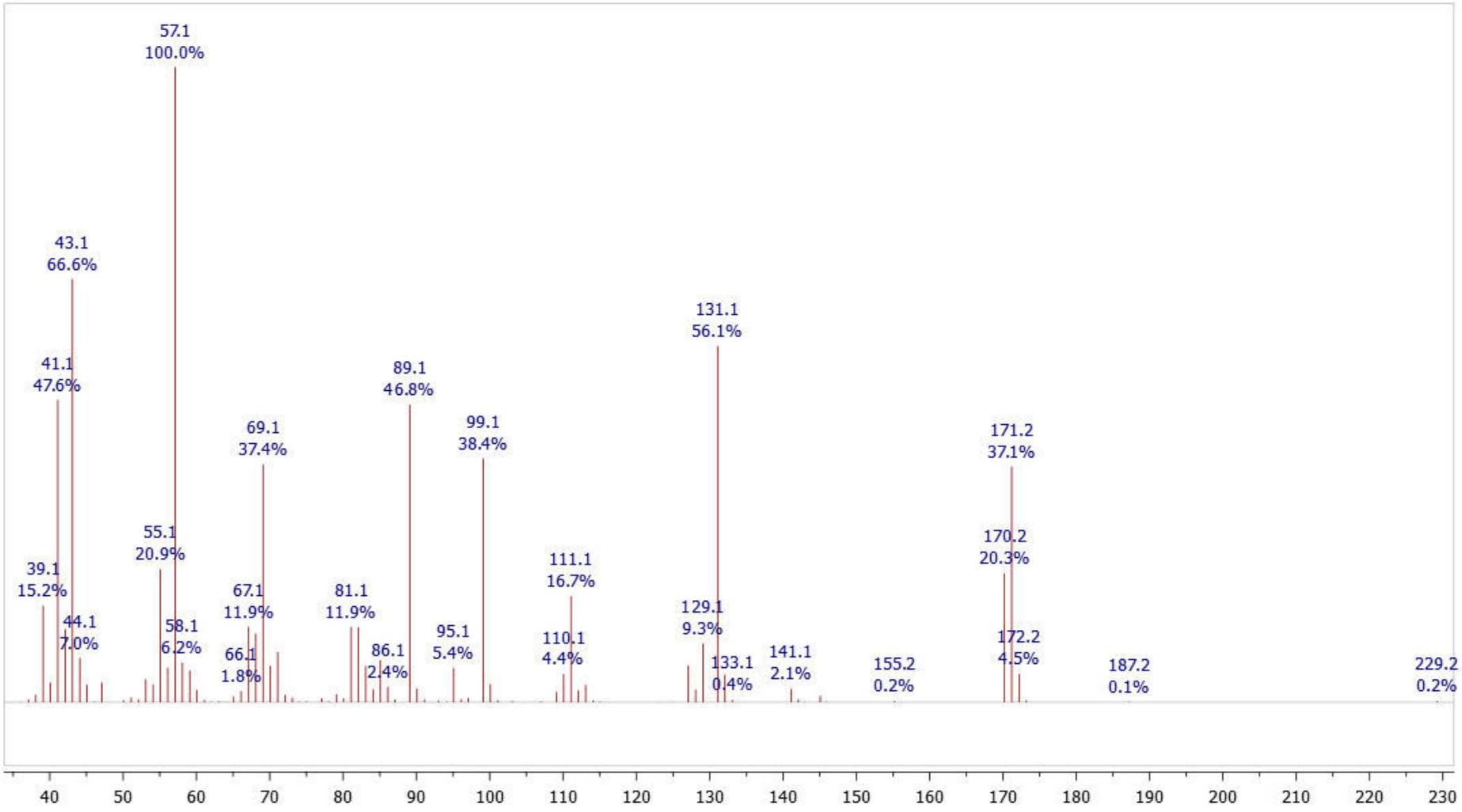
Analysis of ^{1}H-^{1}H coupling constants



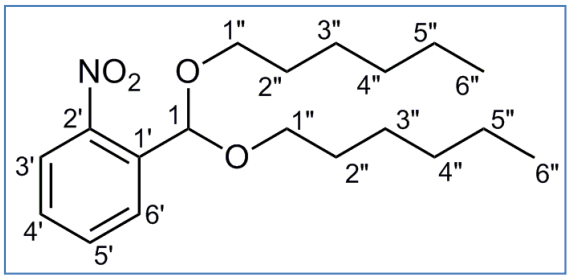
^{1}H-NMR (400 MHz, $CDCl_3$) spectrum of 1,1-dipropoxyoctane and the corresponding expansions with signal assignment



^{13}C-NMR (100 MHz, $CDCl_3$) spectrum of 1,1-dipropoxyoctane with signal assignment



EI-MS spectrum of 1,1-dipropoxyoctane



1-(bis(hexyloxy)methyl)-2-nitrobenzene

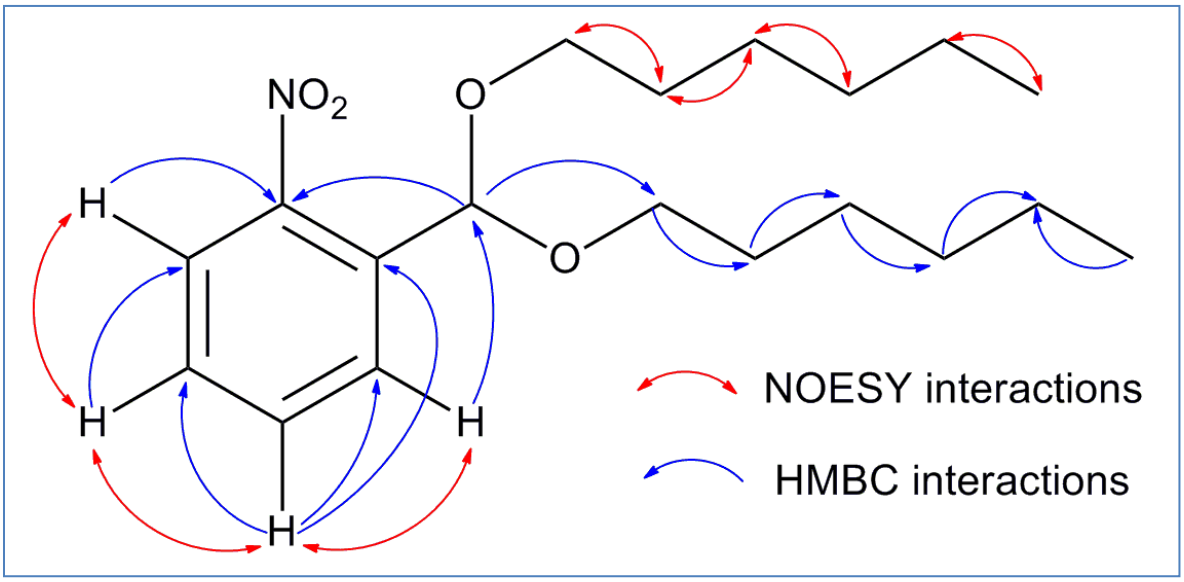
Table of NMR data of 1-(bis(hexyloxy)methyl)-2-nitrobenzene (chemical shift and coupling constant data from ^{1}H and ^{13}C NMR spectra with the observed HMBC and NOESY interactions)

Position	δ_H (Integral, m, *J* (Hz))	δ_C (ppm) C-13 {H}	HMBC[c]	NOESY
1	6.02 (1 H, dd, $^{4}J_{1,6'}$= 0.5, $^{6}J_{1,4'}$= 0.4)[b]	98.6 (1 C)	2', 6', 1"	/
1'	/	133.9 (1 C)	/	/
2'	/	149.2 (1 C)	/	/
3'	7.80 (1 H, ddd, $^{3}J_{3',4'}$ = 7.5, $^{4}J_{3',5'}$=1.2, $^{5}J_{3',6'}$= 0.3)[b]	124.2 (1 C)	1', 2', 5'	4'
4'	7.45 (1 H, dddd, $^{3}J_{4',5'}$ = 7.9, $^{3}J_{3',4'}$ = 7.5, $^{4}J_{4',6'}$ = 1.5, $^{6}J_{1,4'}$= 0.4)[b]	129.2 (1 C)	2', 3', 5', 6'	3', 5'
5'	7.59 (1 H, ddd, $^{3}J_{4',5'}$ = 7.9, $^{3}J_{5',6'}$ = 7.7, $^{4}J_{3',5'}$ = 1.2)[b]	132.5 (1 C)	1', 3', 4',6'	4', 6'
6'	7.83 (1 H, dddd, $^{3}J_{5',6'}$ = 7.7, $^{4}J_{4',6'}$ = 1.5, $^{4}J_{1,6'}$= 0.5, $^{5}J_{3',6'}$= 0.3)[b]	128.2 (1 C)	1, 2', 4'	5'
1"a	3.52 (2 H, dt, $^{2}J_{1"a,1"b}$ = 9.2, $^{3}J_{1"a,2"}$ = 6.7)	68.0 (2 C)	1, 2", 3"	2"
1"b	3.62 (2 H, dt, $^{2}J_{1"a,1"b}$ = 9.2, $^{3}J_{1"b,2"}$ = 6.6)			
2"	1.60 (4 H, qdd, $^{3}J_{2",3"}$ = 6.9, $^{3}J_{1"a,2"}$ = 6.7, $^{3}J_{1"b,2"}$ = 6.6)	29.8 (2 C)	1", 3", 4"	1", 3"
3"	1.38[c]	25.9 (2 C)	1", 2", 4", 5"	2", 4"
4"	1.30[c]	31.7 (2 C)	5", 6"	3"
5"	1.31[c]	22.7 (2 C)	4", 6"	6"
6"	0.88 (6 H, t, $^{3}J_{5",6"}$ = 6.9)	14.2 (2 C)	4", 5"	5"

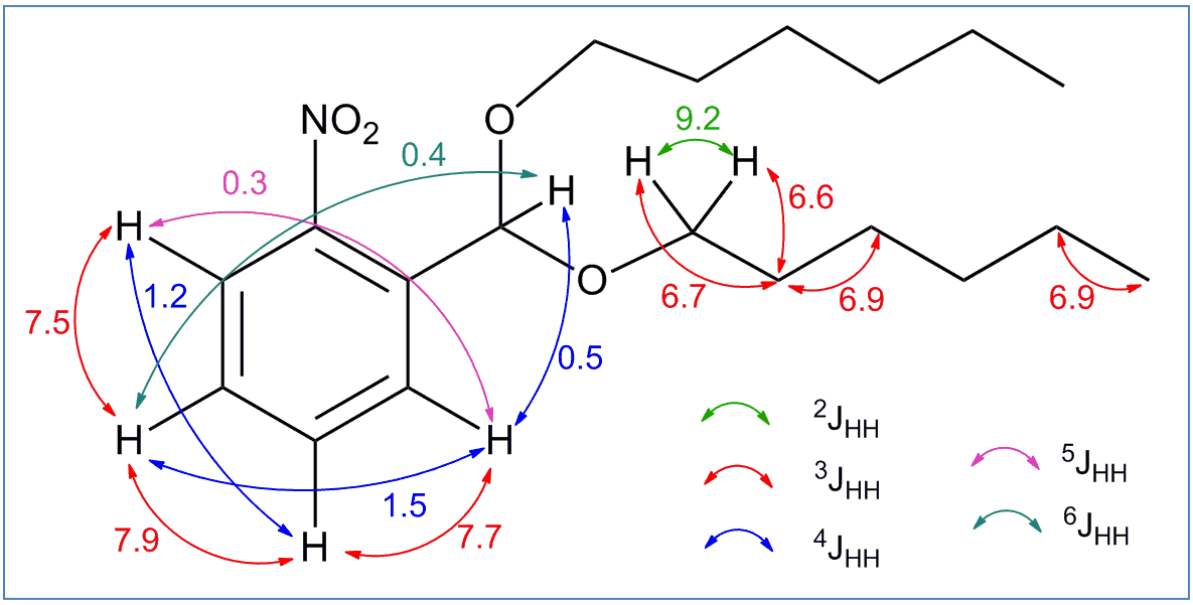
[a]Correlation between the hydrogen in this row and the carbon in the listed position.

[b]Coupling constants (and multiplicity) were determined from appropriate ^{1}H selective homodecoupled spectra. Values of coupling constants lower than 0.5 Hz were usually observed as a broadening of the corresponding ^{1}H NMR signals; they were disclosed only in a series of selective homodecoupling experiments.

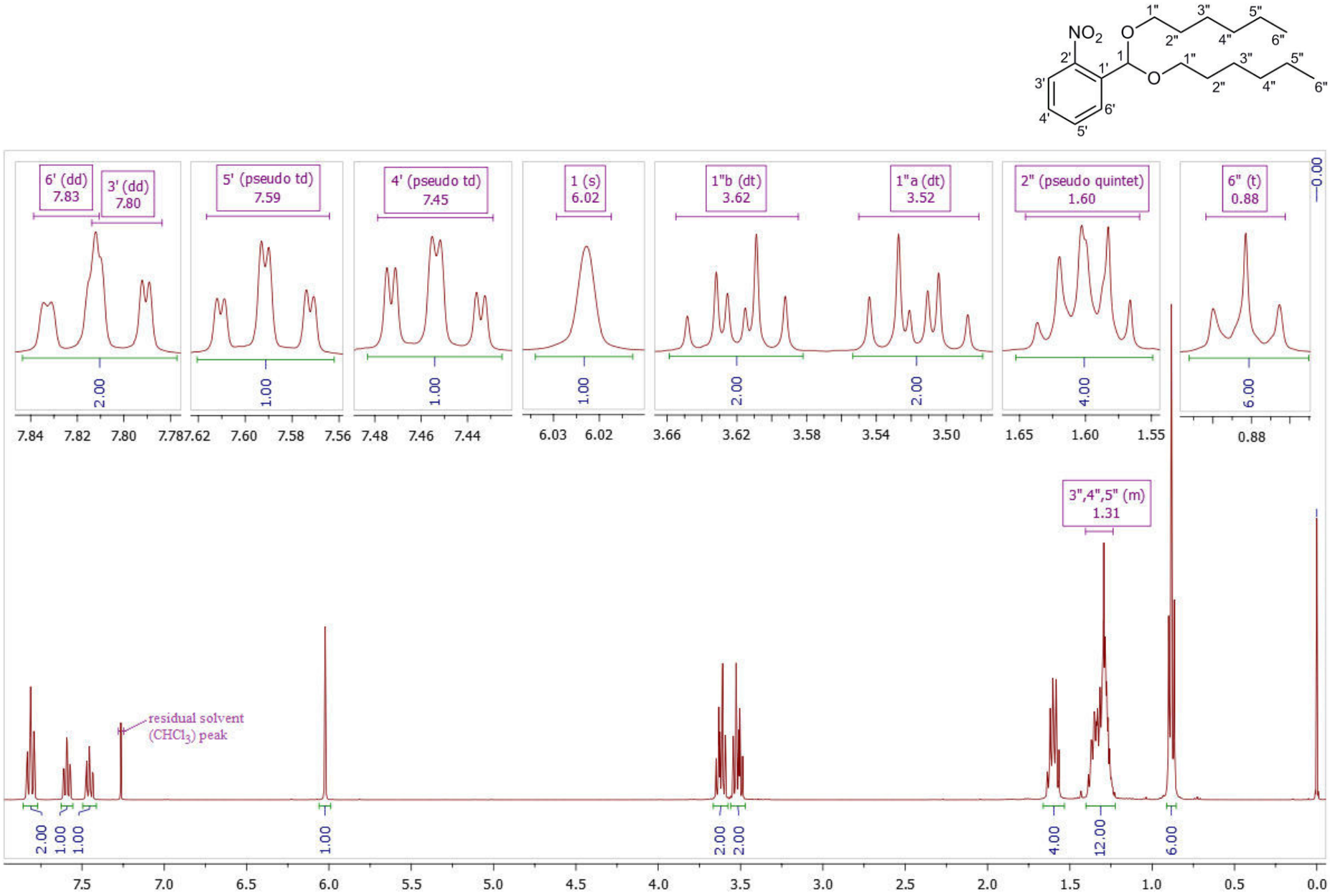
[c]Overlapped signals (range: 1.22-1.40 ppm, 12 H). Chemical shifts were determined from HSQC and HMBC spectra.



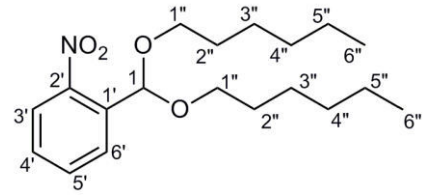
Scheme with key HMBC and NOESY interactions

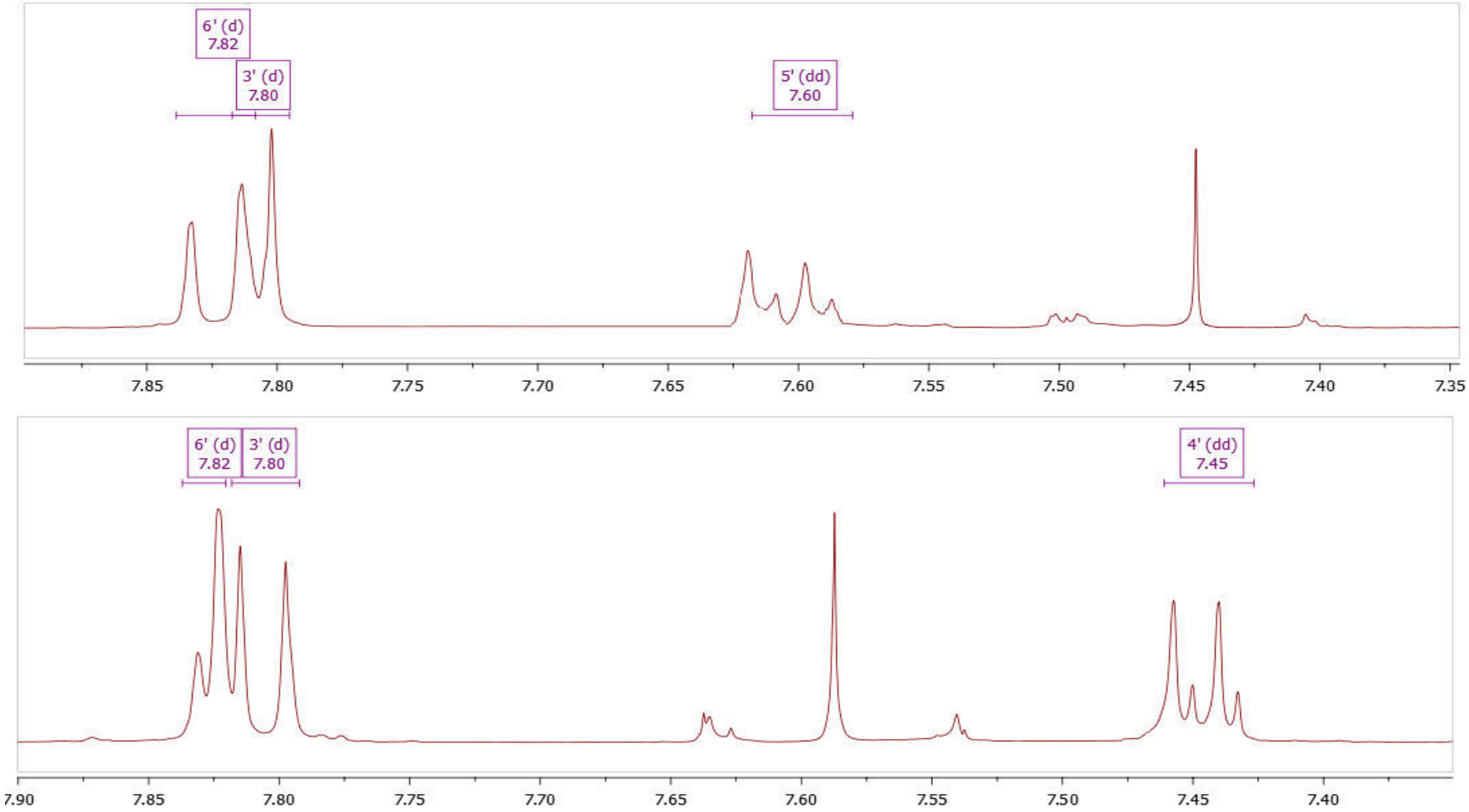


Analysis of ^{1}H-^{1}H coupling constants

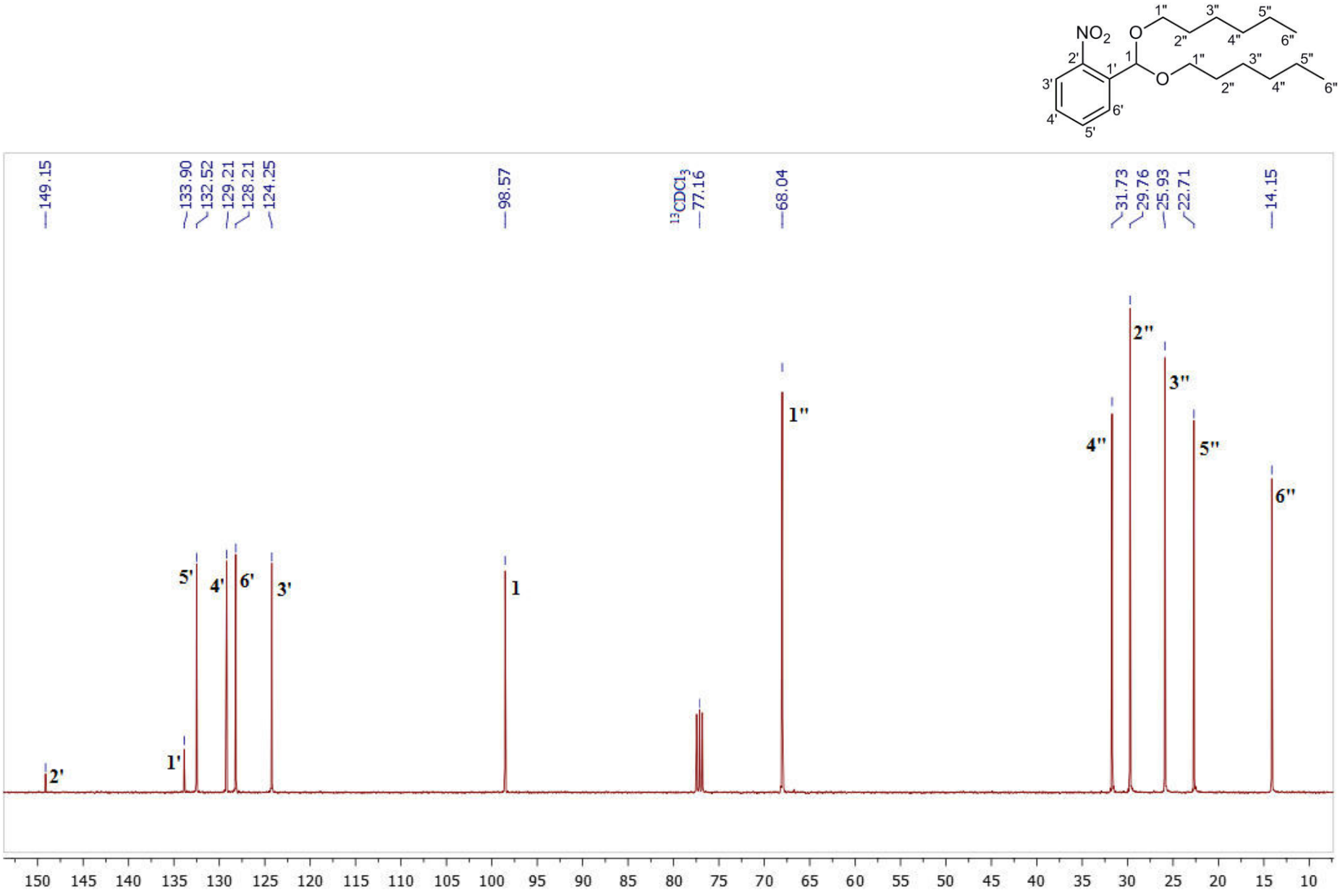


^{1}H-NMR (400 MHz, $CDCl_3$) spectrum of 1-(bis(hexyloxy)methyl)-2-nitrobenzene and the corresponding expansions with signal assignment

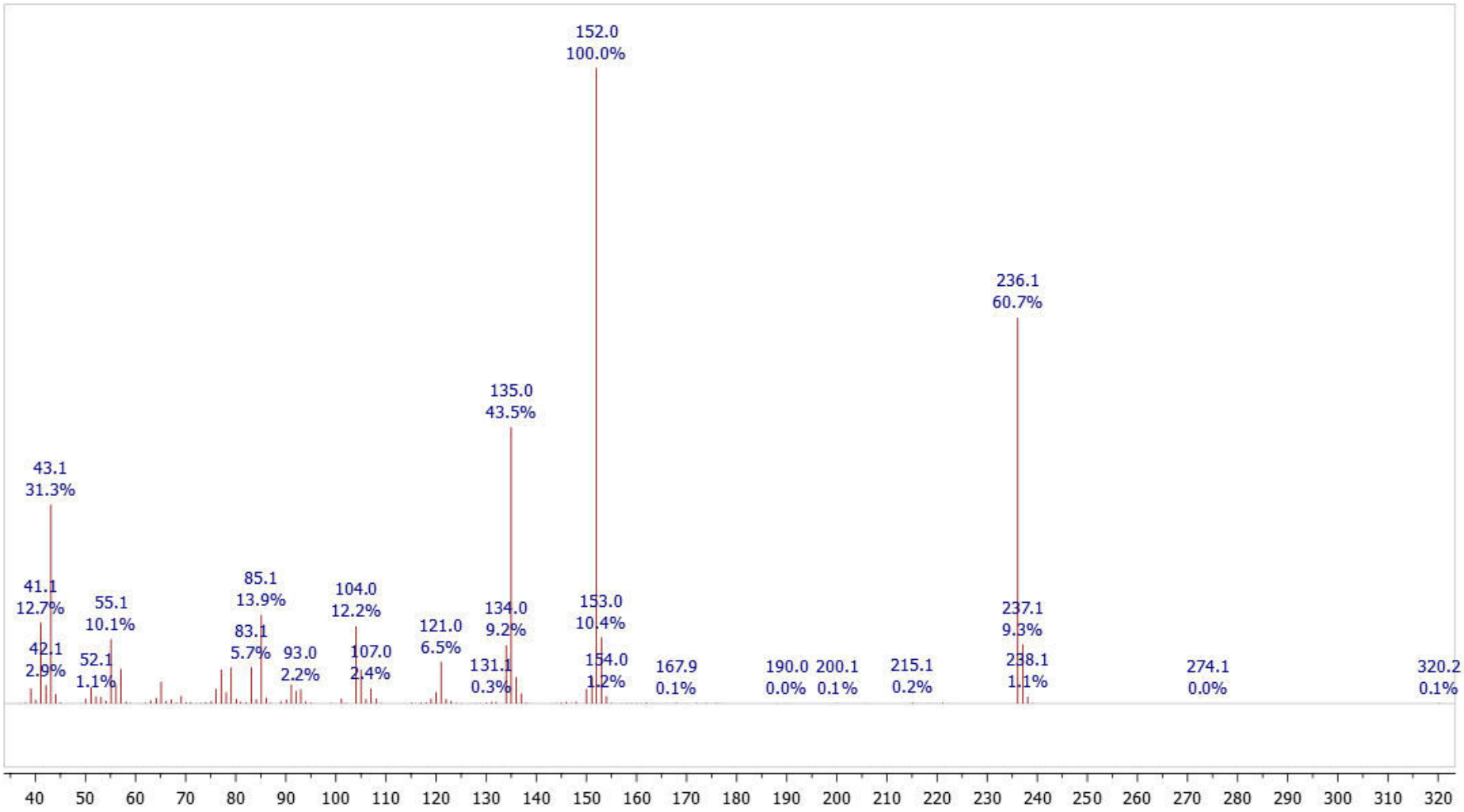




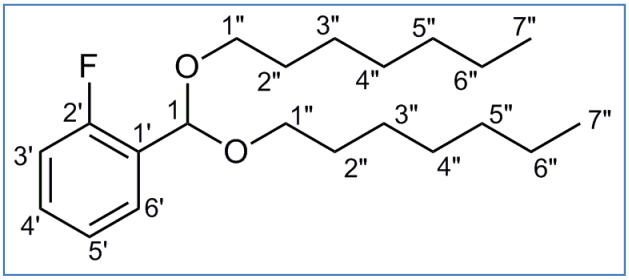
^{1}H NMR spectra obtained in a series of ^{1}H selective homodecoupling experiments with signal assignment



^{13}C-NMR (100.6 MHz, $CDCl_3$) spectrum of 1-(bis(hexyloxy)methyl)-2-nitrobenzene with signal assignment



EI-MS spectrum of 1-(bis(hexyloxy)methyl)-2-nitrobenzene



1-(bis(heptyloxy)methyl)-2-fluorobenzene

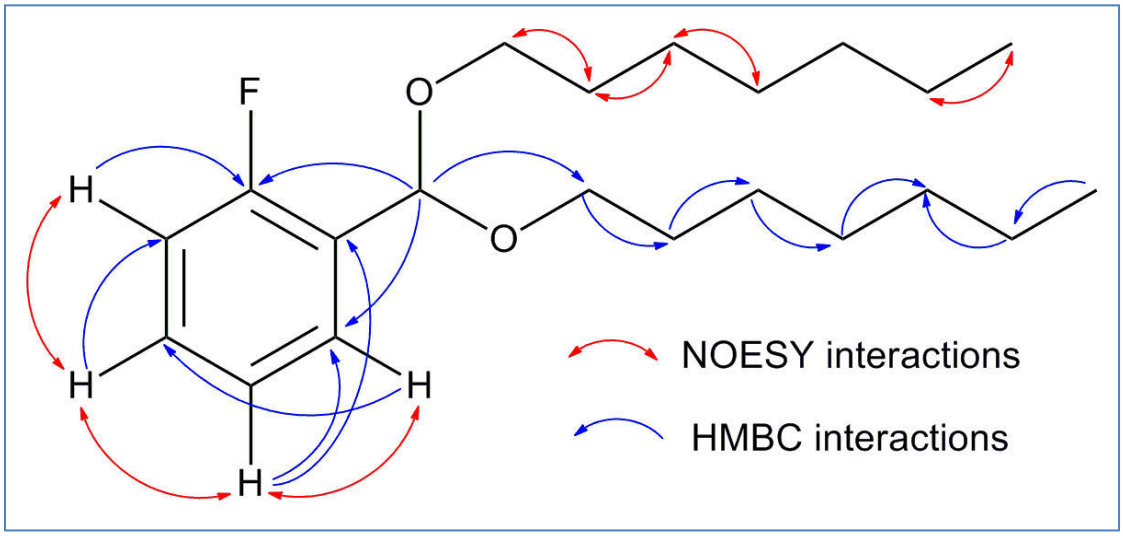
Table of NMR data of 1-(bis(heptyloxy)methyl)-2-fluorobenzene (chemical shift and coupling constant data from ^{1}H and ^{13}C NMR spectra with the observed HMBC and NOESY interactions)

Position	δ_H (Integral, m, *J* (Hz))	δ_C (m, *J* (Hz)), C-13 {H}	HMBC[a]	NOESY
1	5.71 (1 H, t, $^{4}J_{1,6'}$ = 0.5, $^{4}J_{1,F}$ = 0.5)[b]	97.0 (1 C, d, $^{3}J_{CF}$ = 3.5)	2', 6', 1"	/
1'	/	126.6 (1 C, d, $^{2}J_{CF}$ = 12.8)	/	/
2'	/	160.6 (1 C, d, $^{1}J_{CF}$ = 248.1)	/	/
3'	7.03 (1 H, ddd, $^{3}J_{3',F}$ = 10.2, $^{3}J_{3',4'}$ = 8.1, $^{4}J_{3',5'}$ = 1.2, 1H)[b]	115.4 (1 C, d, $^{2}J_{CF}$ = 21.4)	1', 2', 5'	4'
4'	7.29 (1 H, dddd, $^{3}J_{3',4'}$ = 8.1, $^{3}J_{4',5'}$ = 7.3, $^{4}J_{4',F}$ = 5.4, $^{4}J_{4',6'}$ = 1.8)[b]	130.0 (1 C, d, $^{3}J_{CF}$ = 8.4)	2', 3', 6'	3', 5'
5'	7.14 (1 H, dddd, $^{3}J_{5',6'}$ = 7.5, $^{3}J_{4',5'}$ = 7.3, $^{4}J_{3',5'}$ = 1.2, $^{5}J_{5',F}$ = 0.3)[b]	123.9 (1 C, d, $^{4}J_{CF}$ = 3.5)	1', 3', 6'	4', 6'
6'	7.59 (1 H, ddd, $^{3}J_{5',6'}$ = 7.5, $^{4}J_{6',F'}$ = 7.1, $^{4}J_{4',6'}$ = 1.8, $^{4}J_{1,6'}$ = 0.5)[b]	128.2 (1 C, d, $^{3}J_{CF}$ = 3.9)	1, 2', 4'	5'
1"a	3.47 (2 H, dt, $^{2}J_{1"a,1"b}$ = 9.4, $^{3}J_{1"a,2"}$ = 6.6)	66.7 (2 C, s)	1, 2", 3"	2"
1"b	3.55 (2 H, dt, $^{2}J_{1"a,1"b}$ = 9.4, $^{3}J_{1"b,2"}$ = 6.7)		1, 2", 3"	2"
2"	1.57 (4 H, qdd, $^{3}J_{2",3"}$ = 6.9, $^{3}J_{1"b,2"}$ = 6.7, $^{3}J_{1"a,2"}$ = 6.6)	29.8 (2 C, s)	1", 3", 4"	1", 3"
3"	1.35[c]	26.3 (2 C, s)	1", 4"	2", 4"
4"	1.28[c]	29.2 (2 C, s)	5"	3"
5"	1.26[c]	32.0 (2 C, s)	3", 6"	/
6"	1.28[c]	22.8 (2 C, s)	5", 7"	7"
7"	0.87 (6 H, t, $^{3}J_{6",7"}$ = 6.9)	14.2 (2 C, s)	5", 6"	6"

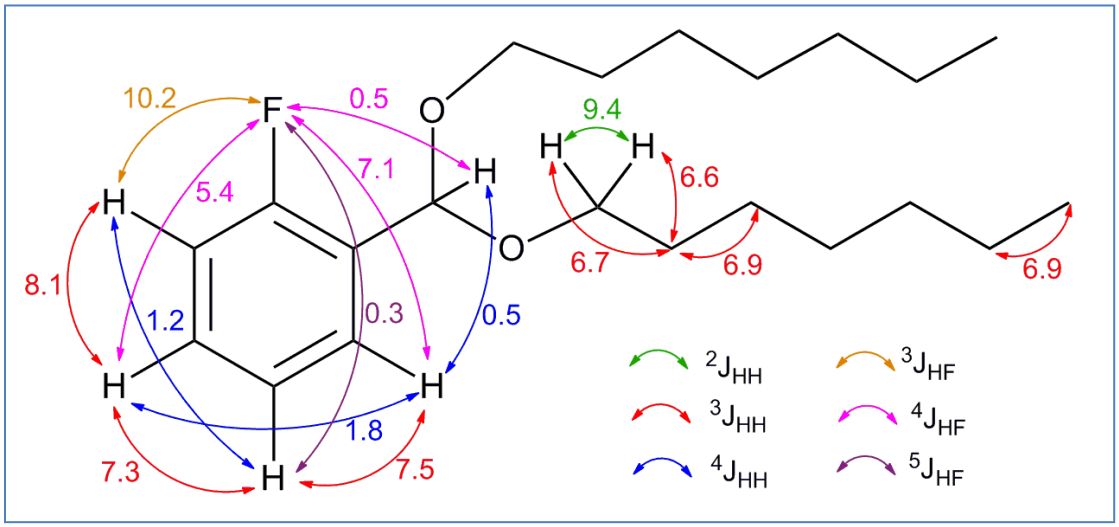
[a]Correlation between the hydrogen in this row and the carbon in the listed position.

[b]Coupling constants (and multiplicity) were determined from appropriate ^{1}H selective homodecoupled spectra. Values of coupling constants lower than 0.5 Hz were usually observed as a broadening of the corresponding ^{1}H NMR signals; they were disclosed only in a series of selective homodecoupling experiments.

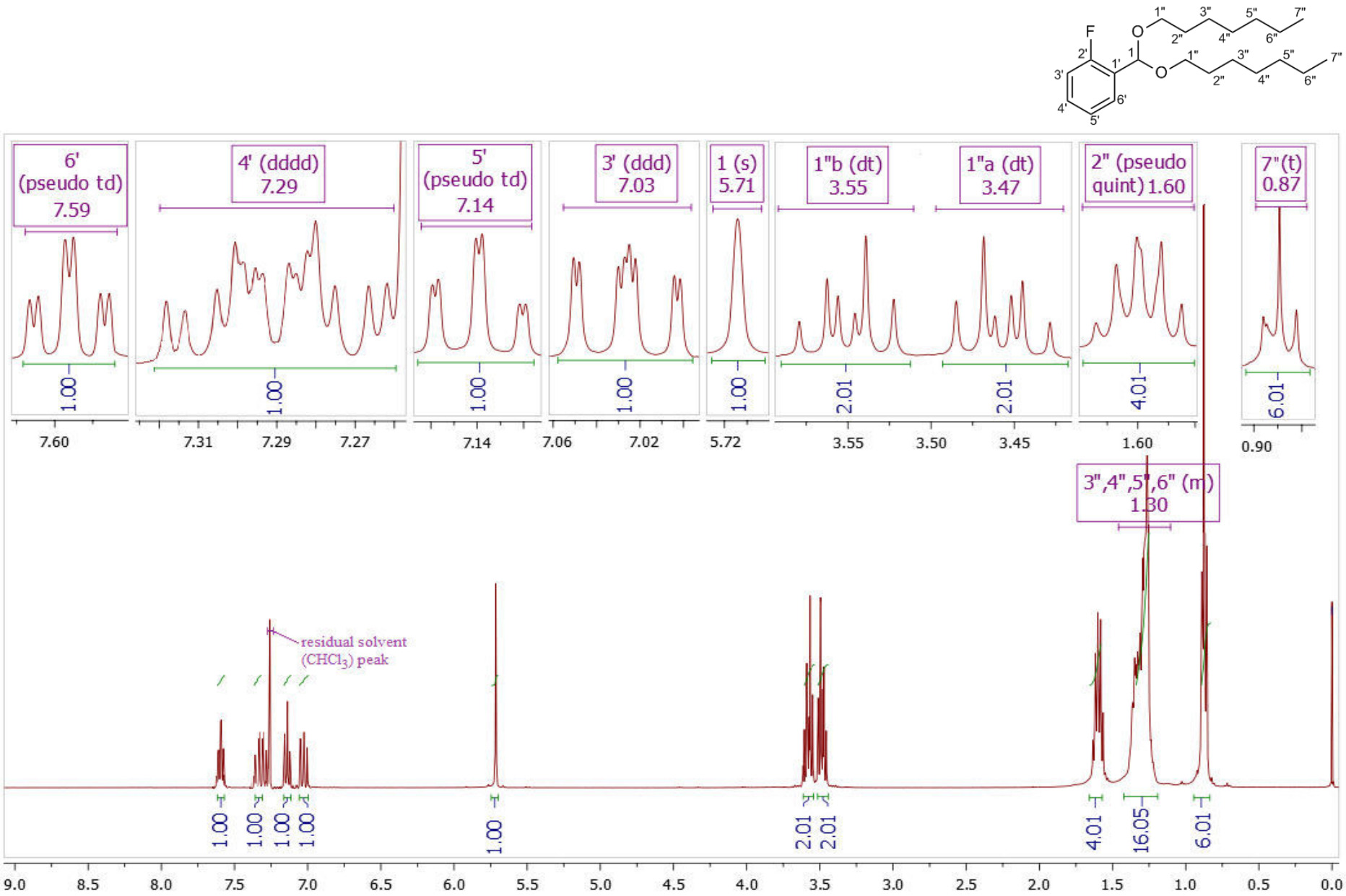
[c]Overlapped signals (range: 1.23-1.43 ppm, 16 H). Chemical shifts were determined from HSQC and HMBC spectra.



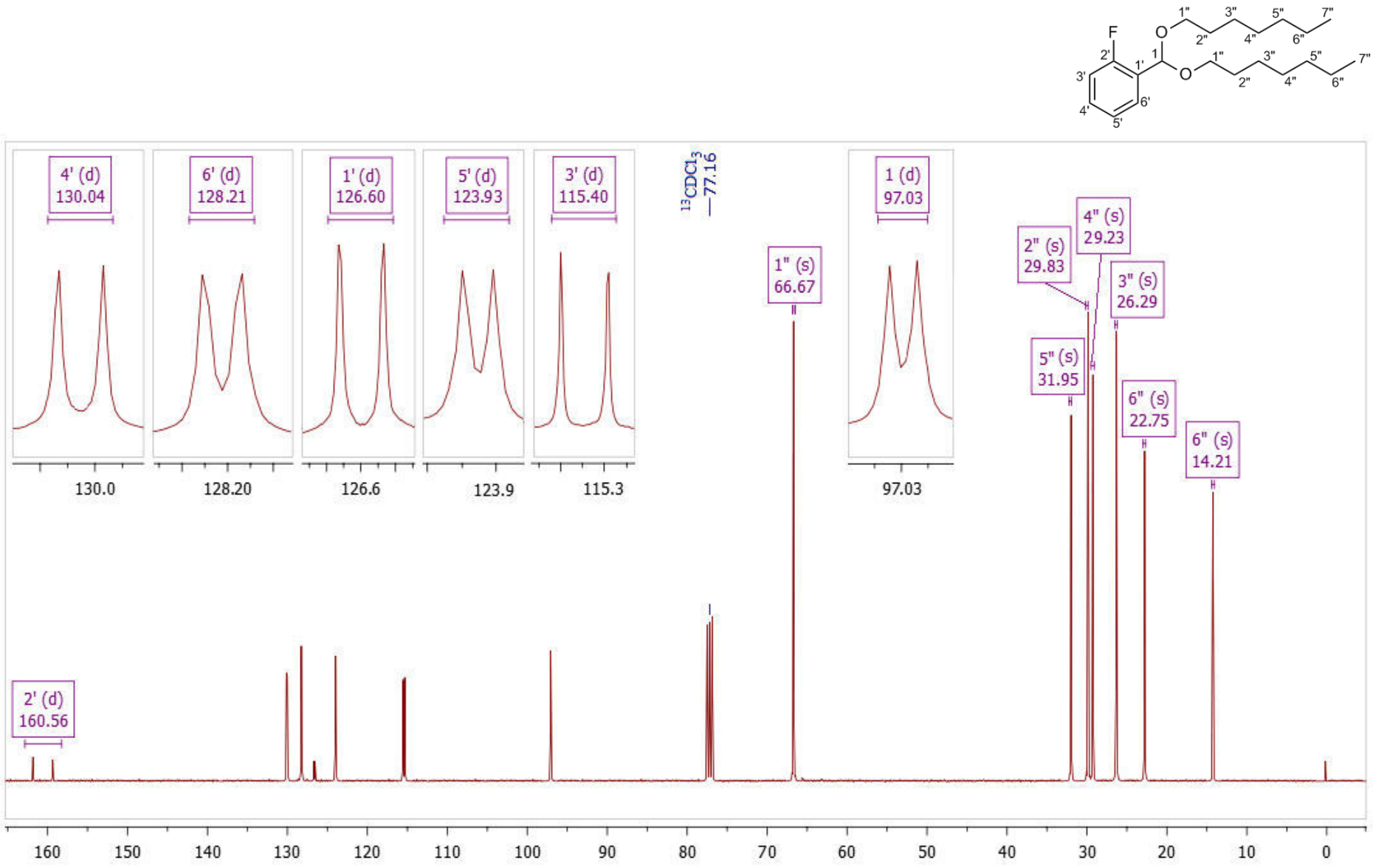
Scheme with key HMBC and NOESY interactions



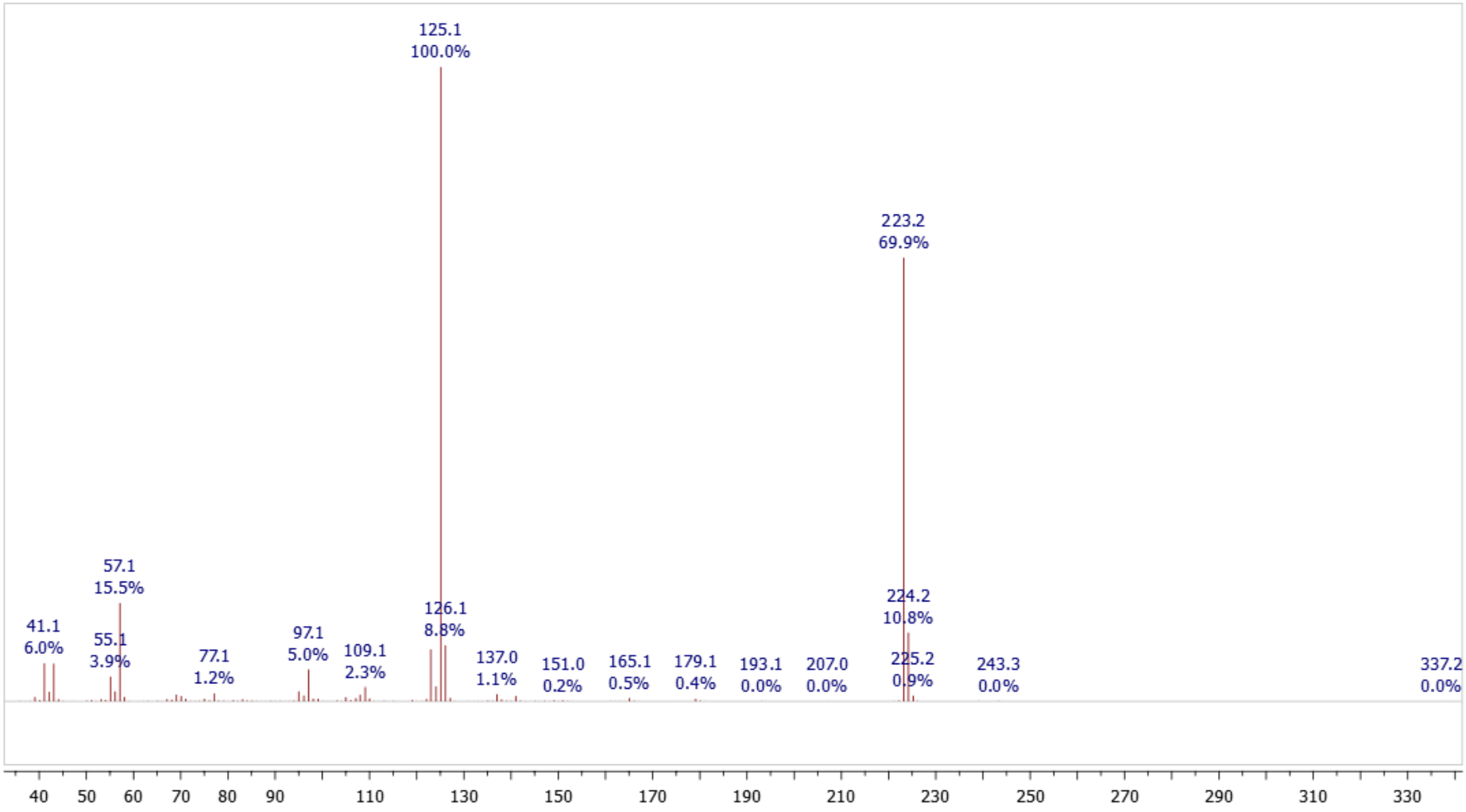
Analysis of ^{1}H-^{1}H coupling constants



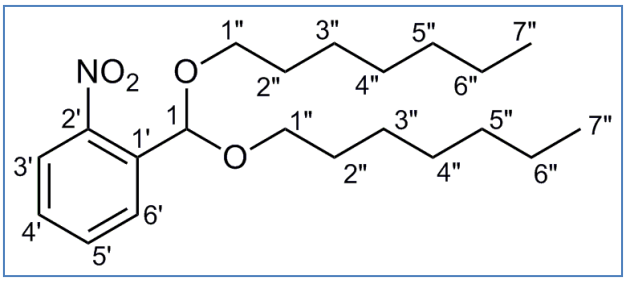
^{1}H-NMR (400 MHz, $CDCl_3$) spectrum of 1-(bis(heptyloxy)methyl)-2-fluorobenzene and the corresponding expansions with signal assignment



^{13}C-NMR (100.6 MHz, $CDCl_3$) spectrum of 1-(bis(heptyloxy)methyl)-2-fluorobenzene and the corresponding expansions with signal assignment



EI-MS spectrum of 1-(bis(heptyloxy)methyl)-2-fluorobenzene



1-(bis(heptyloxy)methyl)-2-nitrobenzene

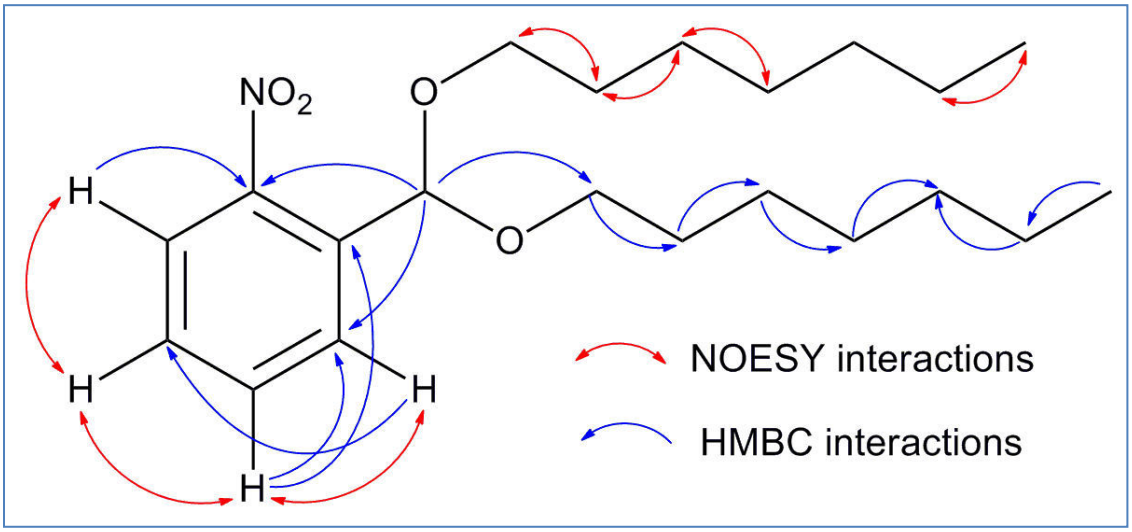
Table of NMR data of 1-(bis(heptyloxy)methyl)-2-nitrobenzene (chemical shift and coupling constant data from ^{1}H and ^{13}C NMR spectra with the observed HMBC and NOESY interactions)

Position	δ_H (Integral, m, *J* (Hz))	δ_C (ppm) C-13 {H}	HMBC[c]	NOESY
1	6.02 (1 H, dd, $^{4}J_{1,6'}$ = 0.5, $^{6}J_{1,4'}$ = 0.4)[b]	98.6 (1 C)	2', 6', 1"	/
1'	/	133.9 (1 C)	/	/
2'	/	149.2 (1 C)	/	/
3'	7.80 (1 H, ddd, $^{3}J_{3',4'}$ = 7.5, $^{4}J_{3',5'}$ = 1.2, $^{5}J_{3',6'}$ = 0.3)[b]	124.3 (1 C)	5'	4'
4'	7.45 (1 H, dddd, $^{3}J_{4',5'}$ = 7.9, $^{3}J_{3',4'}$ = 7.5, $^{4}J_{4',6'}$ = 1.5, $^{6}J_{1,4'}$ = 0.4)[b]	129.2 (1 C)	2', 6'	3', 5'
5'	7.59 (1 H, ddd, $^{3}J_{4',5'}$ = 7.9, $^{3}J_{5',6'}$ = 7.7, $^{4}J_{3',5'}$ = 1.2)[b]	132.5 (1 C)	1', 3'	4', 6'
6'	7.82 (1 H, dddd, $^{3}J_{5',6'}$ = 7.7, $^{4}J_{4',6'}$ = 1.5, $^{4}J_{1,6'}$ = 0.5, $^{5}J_{3',6'}$ = 0.3)[b]	128.2 (1 C)	1, 2', 4'	5'
1"a	3.51 (2 H, dt, $^{2}J_{1"a,1"b}$ = 9.2, $^{3}J_{1"a,2"}$ = 6.7)	68.1 (2 C)	1, 2", 3"	2"
1"b	3.62 (2 H, dt, $^{2}J_{1"a,1"b}$ = 9.2, $^{3}J_{1"b,2"}$ = 6.6)			
2"	1.60 (4 H, qdd, $^{3}J_{2",3"}$ = 6.9, $^{3}J_{1"a,2"}$ = 6.7, $^{3}J_{1"b,2"}$ = 6.6)	29.8 (2 C)	1", 3", 4"	1", 3"
3"	1.35[c]	26.2 (2 C)	1", 2", 4"	2", 4"
4"	1.28[c]	29.2 (2 C)	5"	3"
5"	1.27[c]	31.9 (2 C)	6"	/
6"	1.28[c]	22.7 (2 C)	5", 7"	7"
7"	0.88 (6 H, t, $^{3}J_{6",7"}$ = 6.9)	14.2 (2 C)	5", 6"	6"

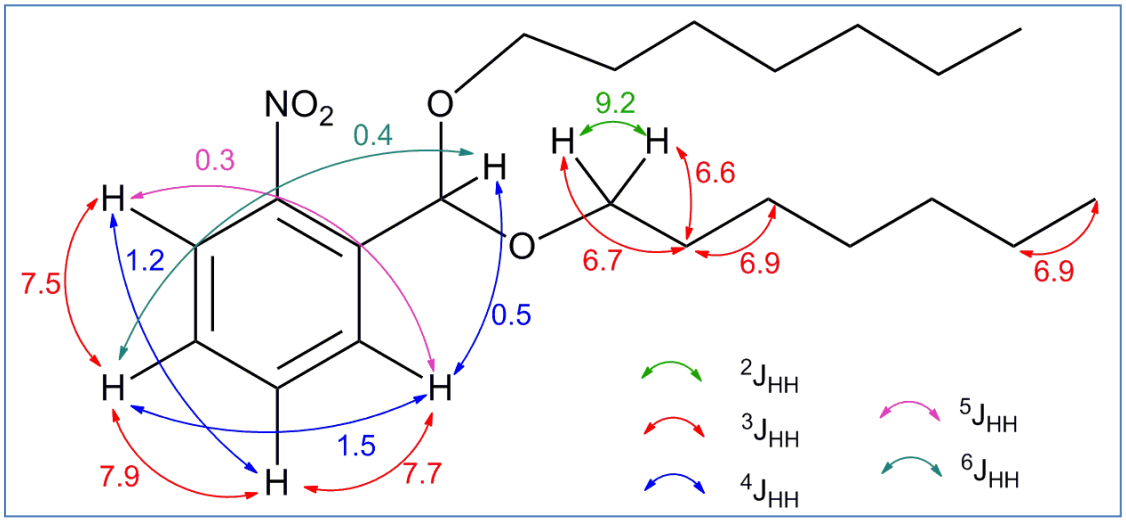
[a]Correlation between the hydrogen in this row and the carbon in the listed position.

[b]Coupling constants (and multiplicity) were determined from appropriate ^{1}H selective homodecoupled spectra. Values of coupling constants lower than 0.5 Hz were usually observed as a broadening of the corresponding ^{1}H NMR signals; they were disclosed only in a series of selective homodecoupling experiments.

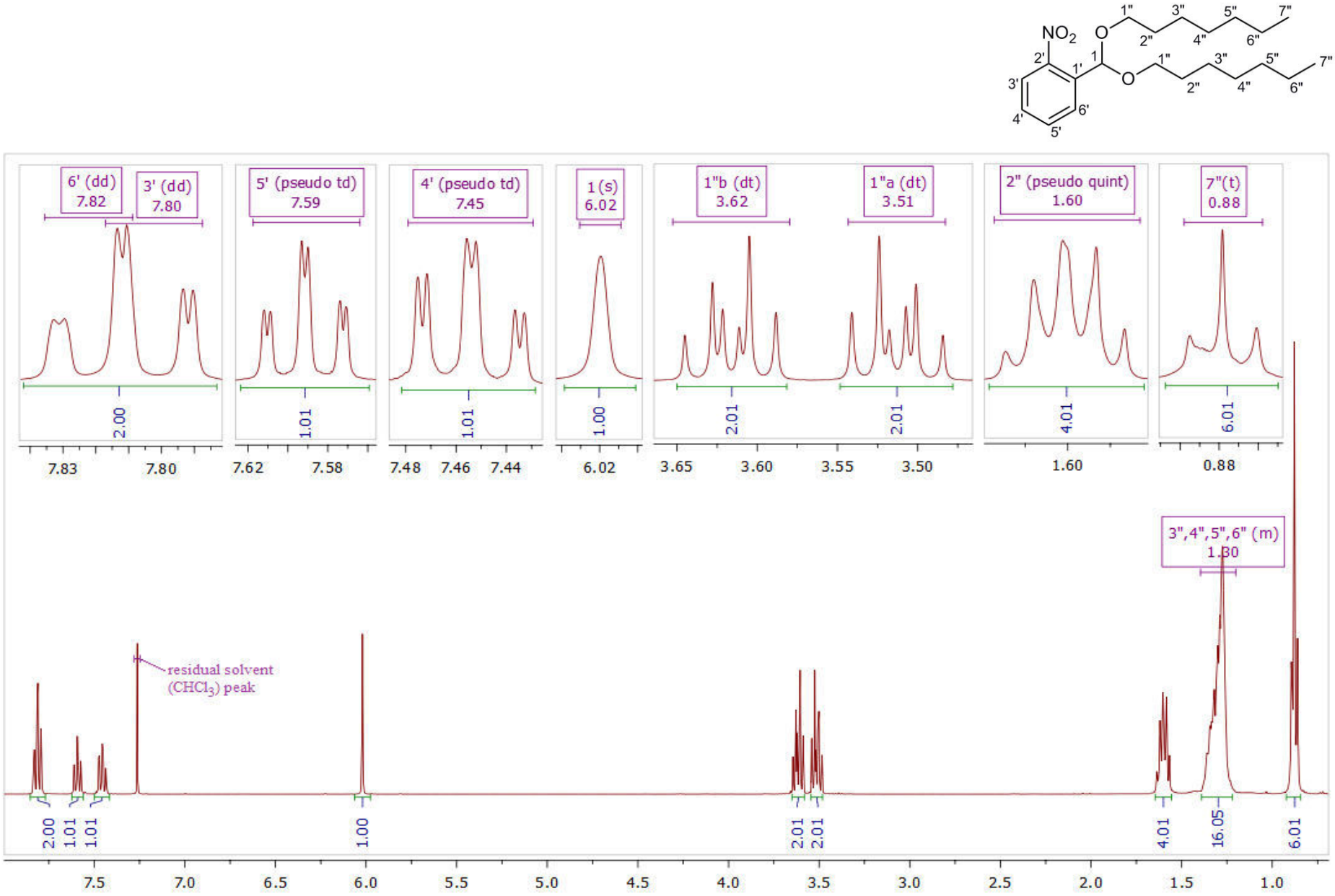
[c]Overlapped signals (range: 1.20-1.40 ppm, 16 H). Chemical shifts were determined from HSQC and HMBC spectra.



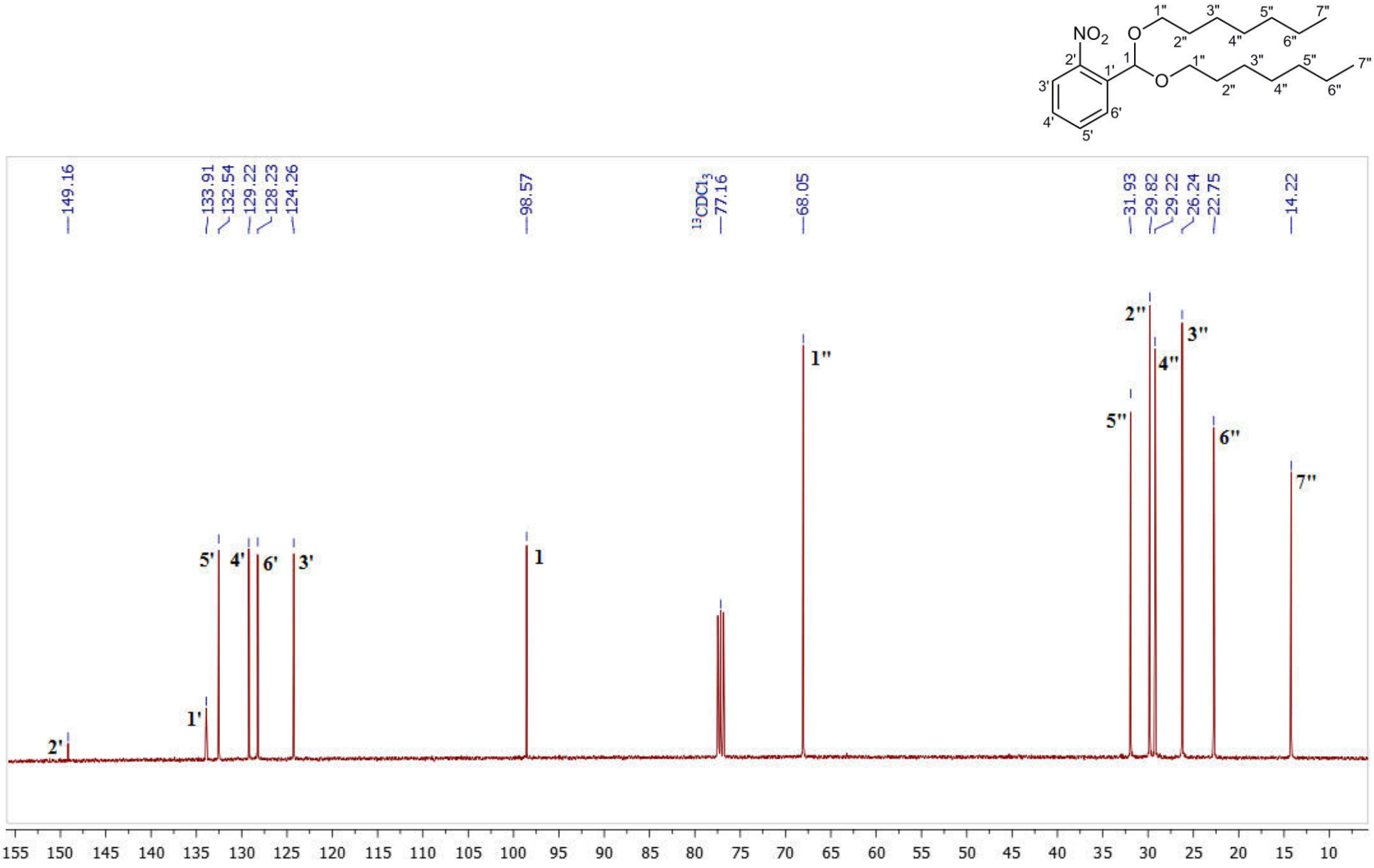
Scheme with key HMBC and NOESY interactions



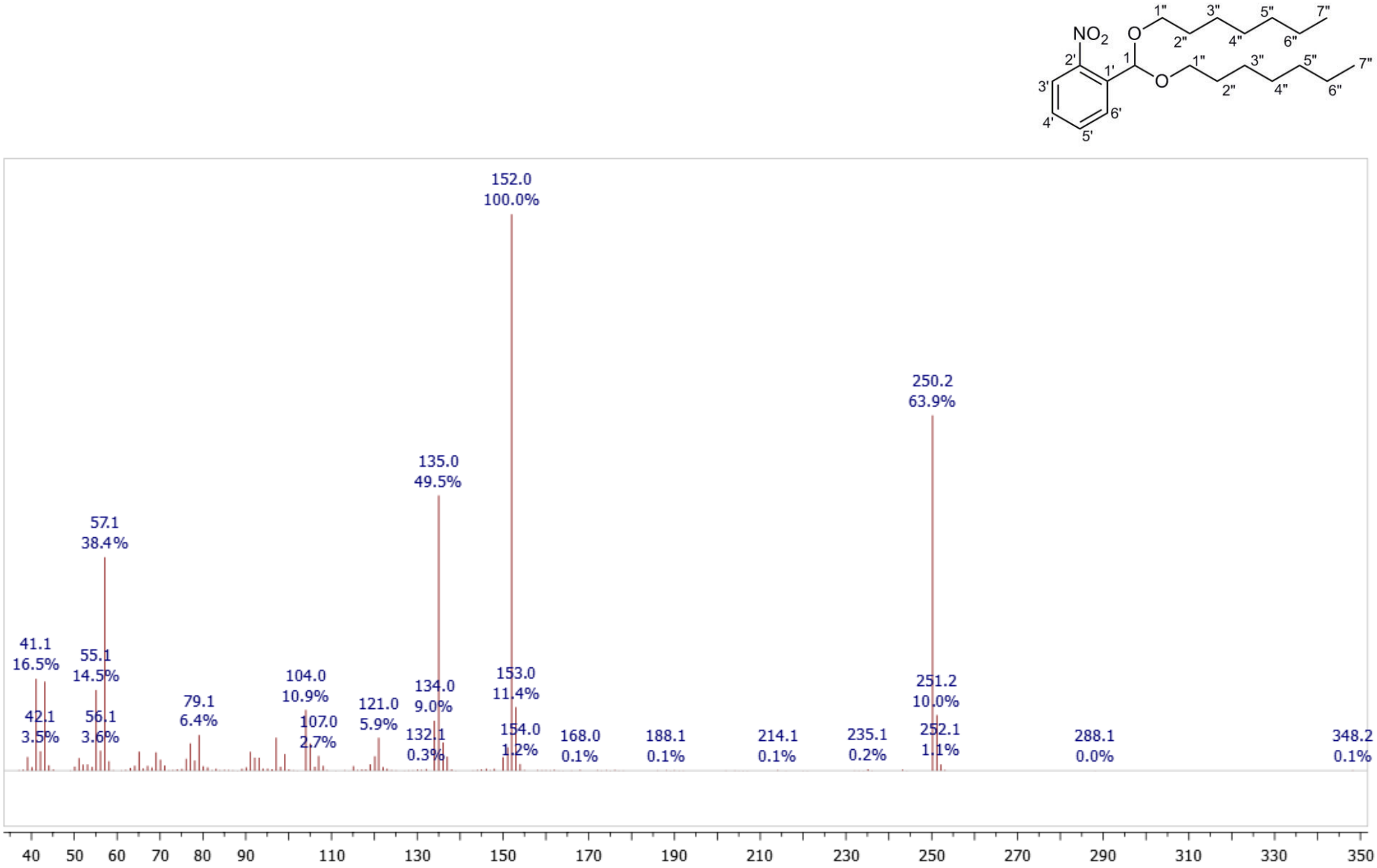
Analysis of ^{1}H-^{1}H coupling constants



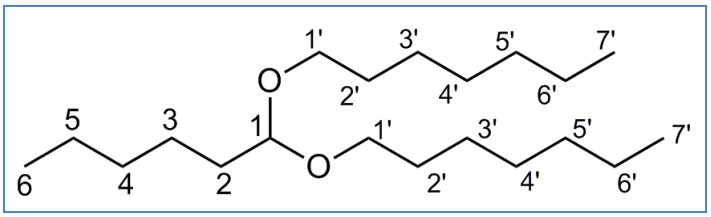
^{1}H-NMR (400 MHz, $CDCl_3$) spectrum of 1-(bis(heptyloxy)methyl)-2-nitrobenzeneand the corresponding expansions with signal assignment



^{13}C-NMR (100 MHz, $CDCl_3$) spectrum of 1-(bis(heptyloxy)methyl)-2-nitrobenzene with signal assignment



EI-MS spectrum of 1-(bis(heptyloxy)methyl)-2-nitrobenzene



1,1-diheptoxyhexane

Table of NMR data of 1,1-diheptoxyhexane (chemical shift and coupling constant data from ^{1}H and ^{13}C NMR spectra with the observed HMBC and NOESY interactions)

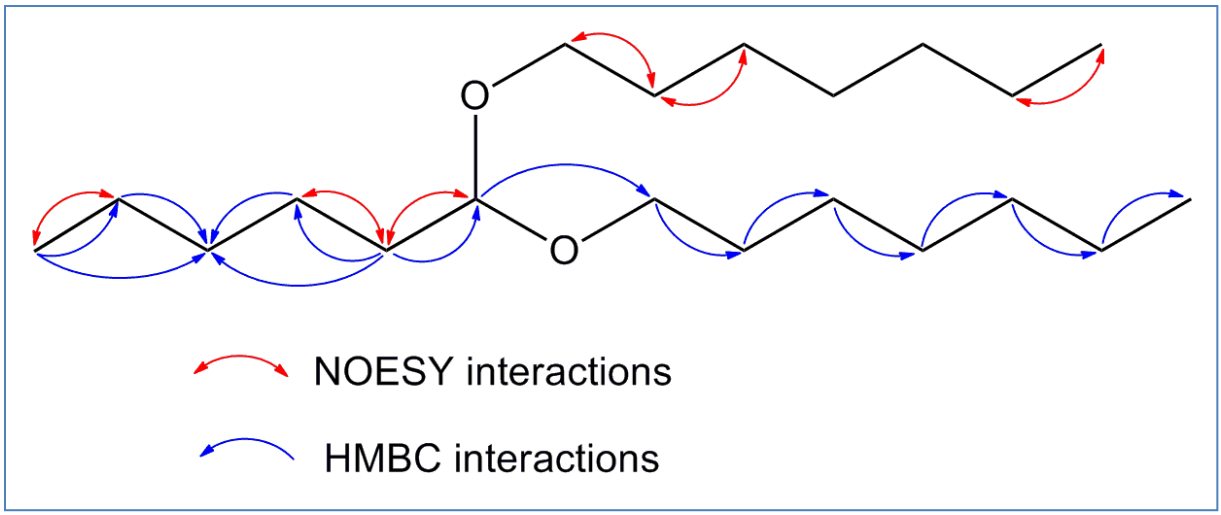
Position	δ_H (Integral, m, *J* (Hz))	δ_C (ppm) C-13 {H}	HMBC[a]	NOESY
1	4.46 (1 H, t, $^{3}J_{1,2}$ = 5.8)	103.29 (1 C)	2, 3, 1’	2
2	1.60 (2 H, td, $^{3}J_{2,3}$ = 6.7, $^{3}J_{1,2}$ = 5.8)[b]	33.58 (1 C)	1, 3, 4	1, 3
3	1.34[c]	24.63 (1 C)	1, 2, 4	2
4	1.30[c]	31.84 (1 C)	3, 5, 6	/
5	1.32[c]	22.75 (1 C)	4, 6	6
6	0.89 (t, $^{3}J_{5,6}$ = 6.9)[d]	14.13 (1 C)	4, 5	5
1’a	3.40 (2 H, $^{2}J_{1'a,1'b}$ = 9.3, $^{3}J_{1'a,2'}$ = 6.7)	65.56 (2 C)	1, 2’, 3’	2’, 1’b
1’b	3.56 (2 H, dt, $^{2}J_{1'a,1'b}$ = 9.3, $^{3}J_{1'b,2'}$ = 6.6)		1, 2’, 3’	2’, 1’a
2’	1.60 (4 H, qd, $^{3}J_{1'a,2'}$ = $^{3}J_{2',3'}$ = 6.7, $^{3}J_{1'b,2'}$ = 6.6)[b]	30.08 (2 C)	1’, 3’, 4’	1’, 3’
3’	1.35[c]	26.40 (2 C)	1’, 2’, 4’	2’
4’	1.28[c]	29.29 (2 C)	3’, 5’	/
5’	1.27[c]	31.99 (2 C)	6’	/
6’	1.28[c]	22.76 (2 C)	5’, 7’	7’
7’	0.88 (t, $^{3}J_{6',7'}$ = 6.9)[d]	14.20 (2 C)	5’, 6’	6’

[a]Correlation between the hydrogen in this row and the carbon in the listed position.

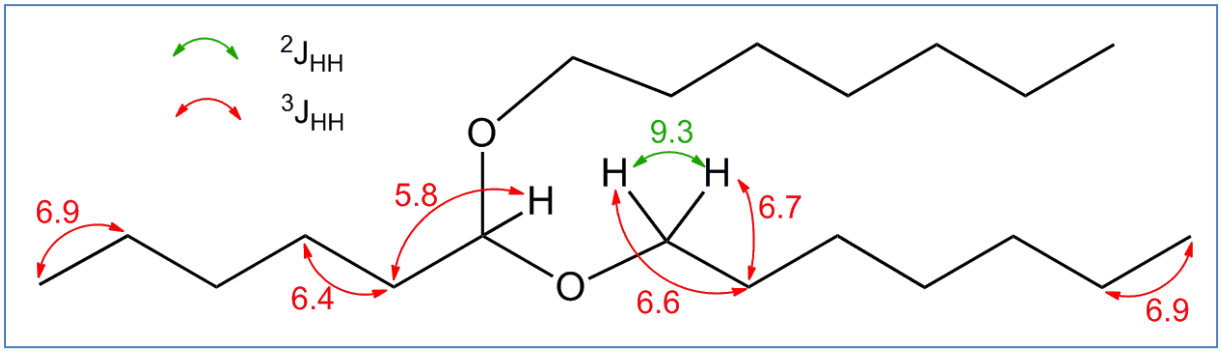
[b]Overlapped signals (range: 1.52-1.63 ppm, 6 H). Chemical shifts were determined from HSQC and HMBC spectra. Coupling constants were determined from the appropriate decoupled spectra.

[c]Overlapped signals (range: 1.24-1.40 ppm, 22 H). Chemical shifts were determined from HSQC and HMBC spectra.

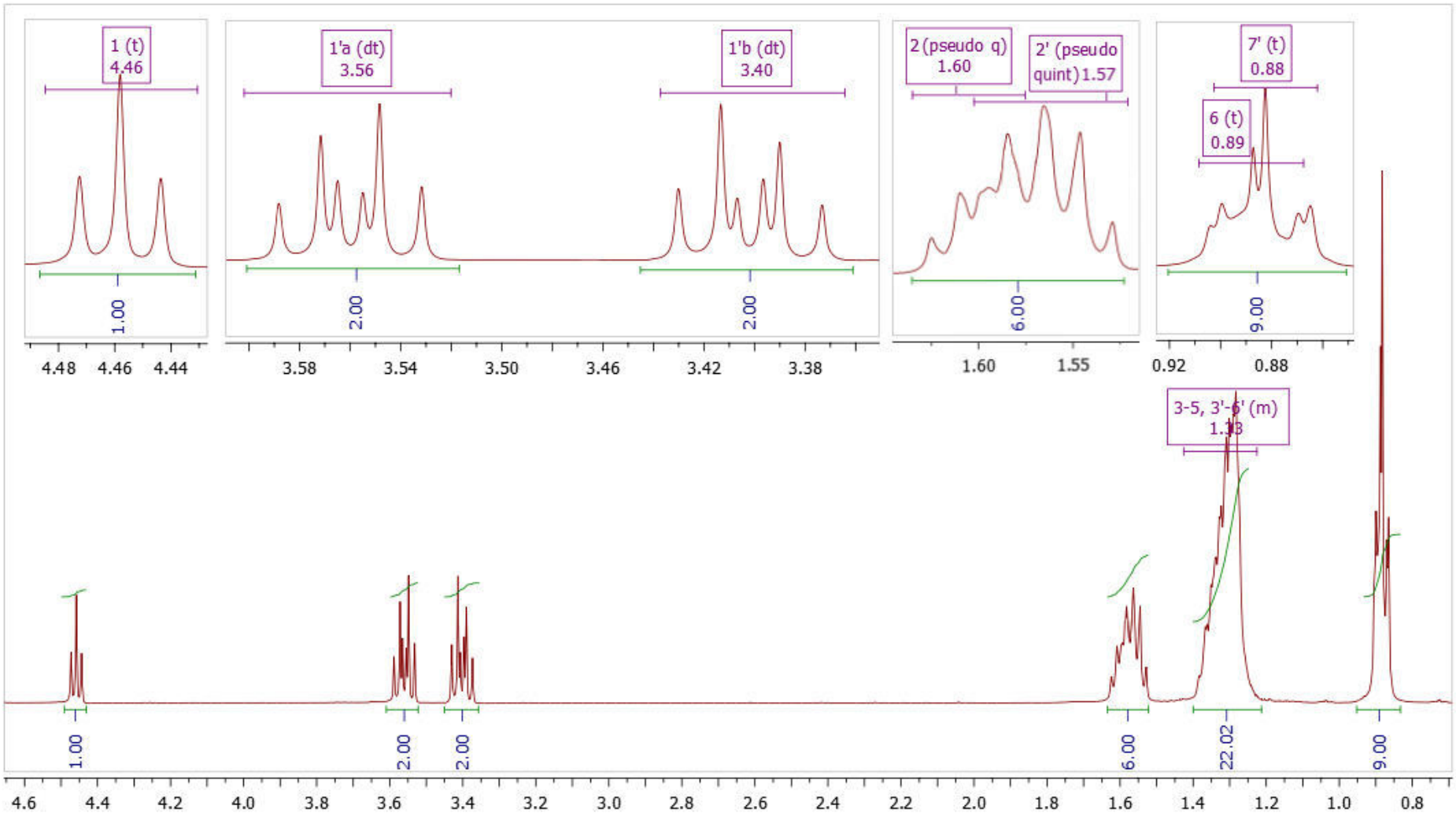
[d]Overlapped signals (range: 0.85-0.92 ppm). Total integral 9 H.



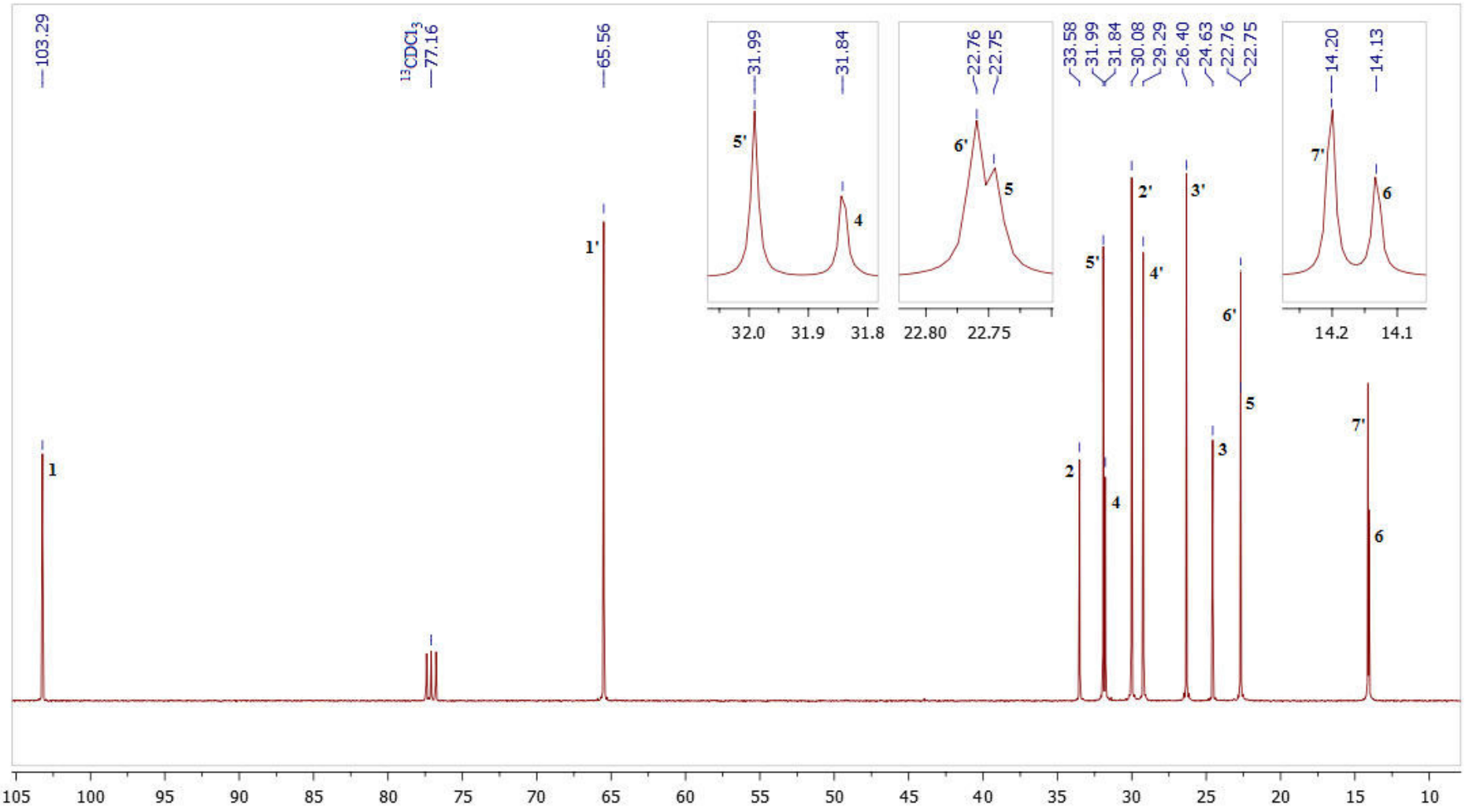
Scheme with key HMBC and NOESY interactions



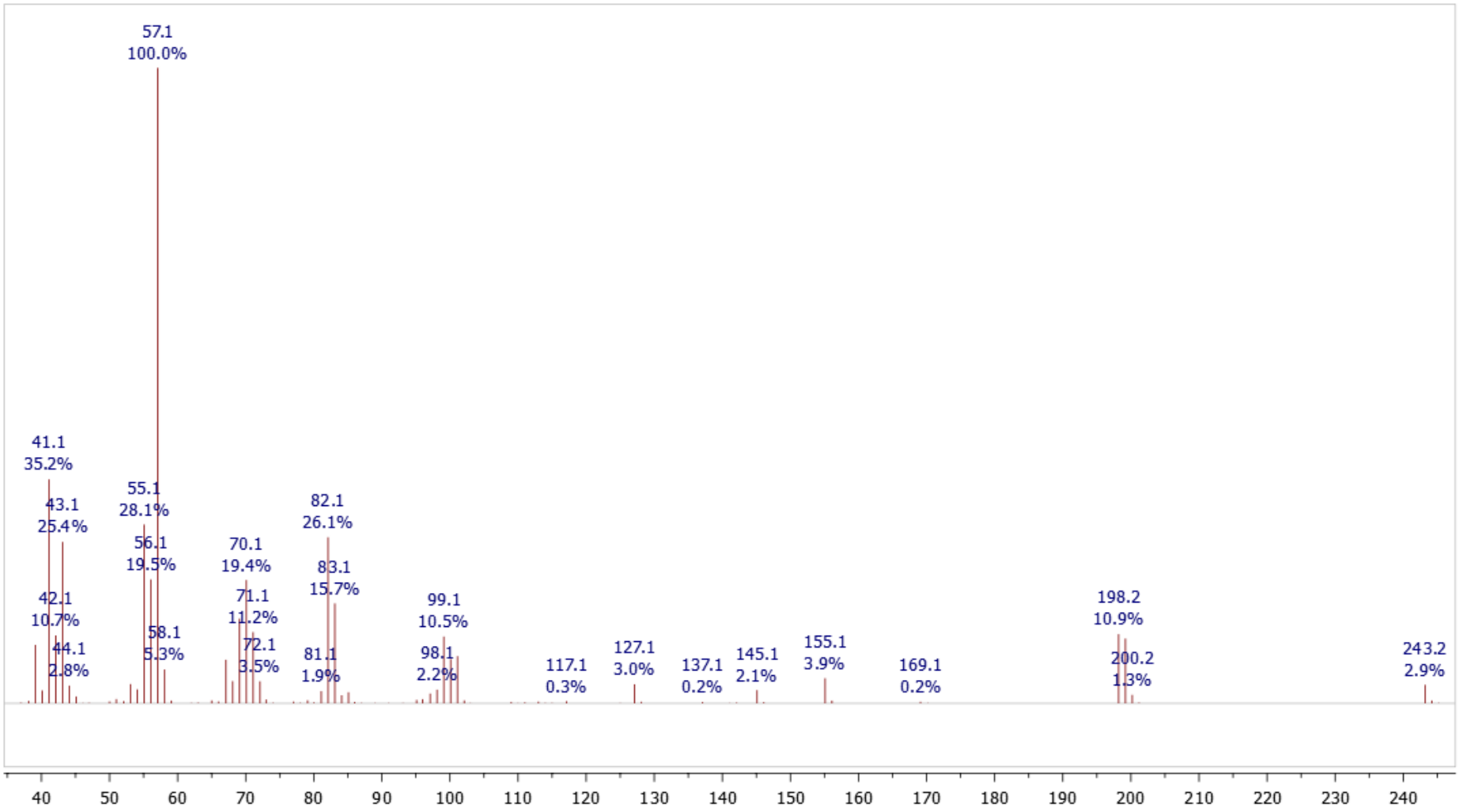
Analysis of ^{1}H-^{1}H coupling constants



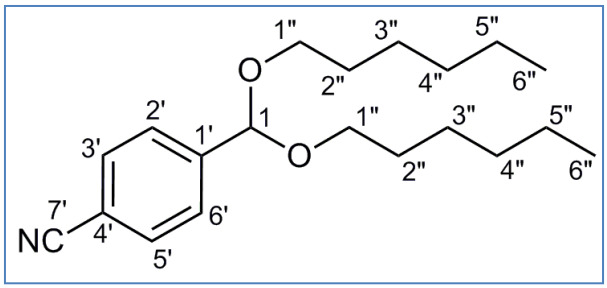
^{1}H-NMR (400 MHz, $CDCl_3$) spectrum of 1,1-diheptoxyhexane and the corresponding expansions with signal assignment



^{13}C-NMR (400 MHz, $CDCl_3$) spectrum of 1,1-diheptoxyhexane with signal assignment and the corresponding expansions



EI-MS spectrum of 1,1-diheptoxyhexane



4-(bis(hexyloxy)methyl)benzonitrile

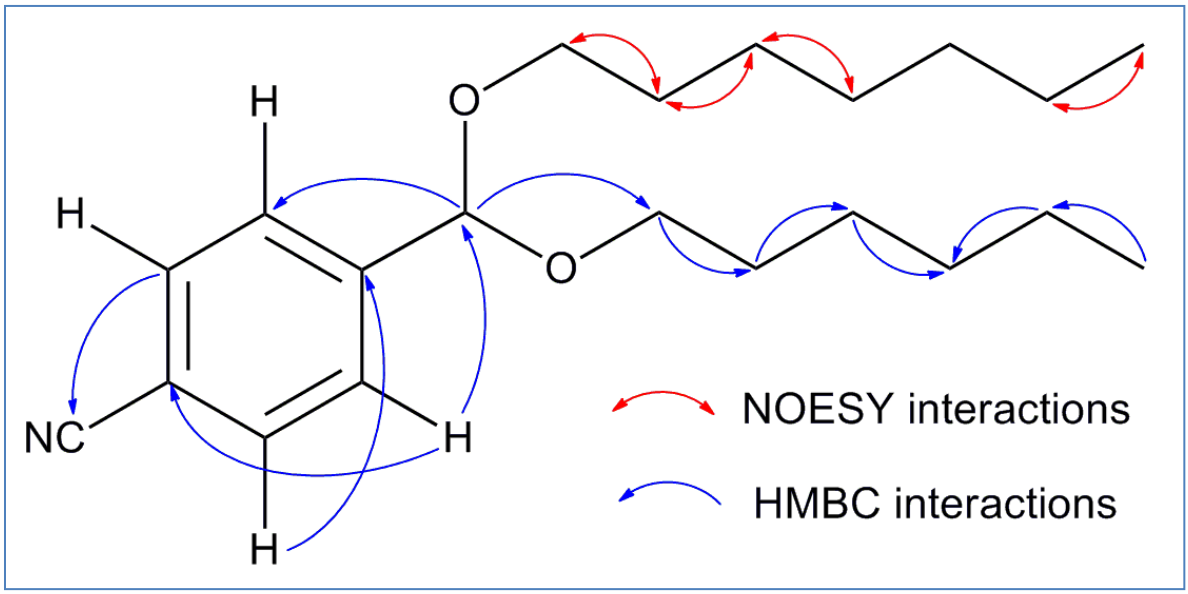
Table of NMR data of 4-(bis(hexyloxy)methyl)benzonitrile (chemical shift and coupling constant data from ^{1}H and ^{13}C NMR spectra with the observed HMBC and NOESY interactions)

Position	δ_H (Integral, m, *J* (Hz))	δ_C (ppm) C-13 {H}	HMBC[a]	NOESY
1	5.52 (1 H, t, $^{4}J_{1,2'/6'} = 0.4$)[b]	100.4 (1 C)	2', 6', 1"	/
1'	/	144.4 (1 C)	/	/
2', 6' (AA')	7.59 (2 H, m, AA'BB') ($^{3}J_{AB} = 8.3$, $^{4}J_{AA'} = 1.7$, $^{4}J_{BB'} = 1.6$, $^{5}J_{AB'/A'B} = 0.5$, $^{4}J_{AA',1} = 0.4$)[b]	127.7 (2 C)	1, 3'/5', 4', (6'/2')	3'/5'
3', 5' (BB')	7.66 (2 H, m, AA'BB') ($^{3}J_{AB} = 8.3$, $^{4}J_{AA'} = 1.7$, $^{4}J_{BB'} = 1.6$, $^{5}J_{AB'/A'B} = 0.5$)[b]	132.2 (2 C)	1', (5'/3'), 7'	2'/6'
4'	/	112.2 (1 C)	/	/
7'	/	118.9 (1 C)	/	/
1"a	3.46 (2 H, dt, $^{2}J_{1"a,1"b} = 9.4$, $^{3}J_{1"a,2"} = 6.6$)	65.8 (2 C)	1, 2", 3"	2"
1"b	3.50 (2 H, dt, $^{2}J_{1"a,1"b} = 9.4$, $^{3}J_{1"b,2"} = 6.7$)			
2"	1.60 (4 H, qd, $^{3}J_{2",3"} = {}^{3}J_{1"b,2"} = 6.7$, $^{3}J_{1"a,2"} = 6.6$)	29.8 (2 C)	1", 3", 4"	1", 3"
3"	1.38[c]	26.0 (2 C)	1", 2", 4", 5"	2"
4"	1.30[c]	31.8 (2 C)	5", 6"	/
5"	1.31[c]	22.7 (2 C)	4", 6"	6"
6"	0.88 (6 H, t, $^{3}J_{6",7"} = 6.9$)	14.2 (2 C)	4", 5"	5"

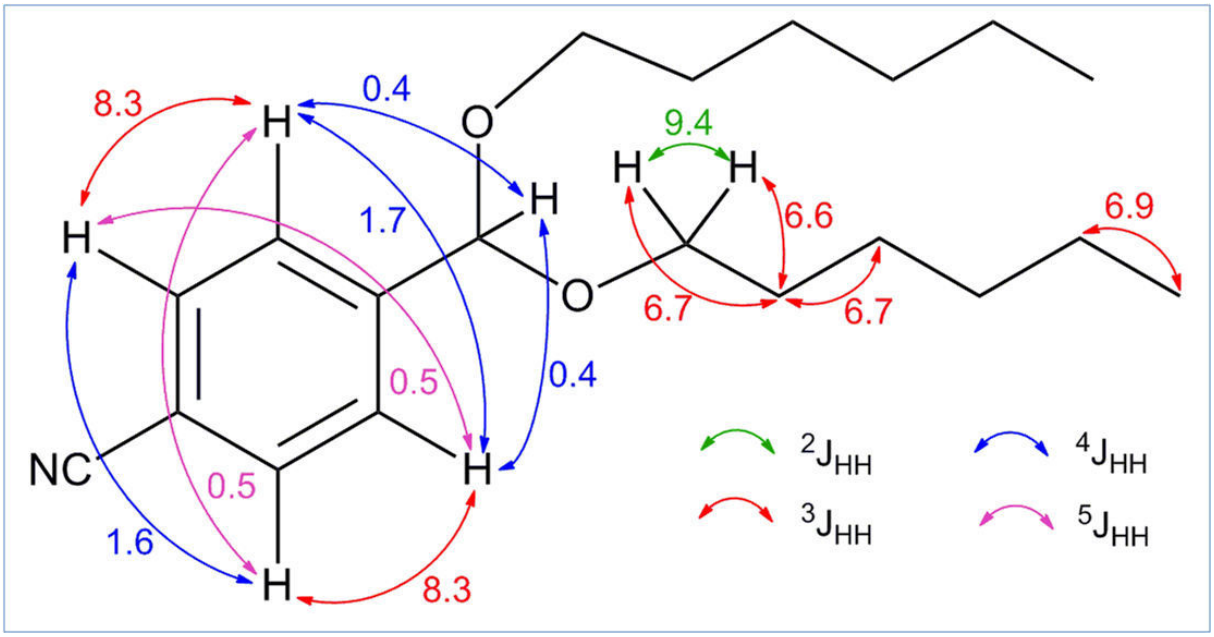
[a]Correlation between the hydrogen in this row and the carbon in the listed position.

[b]Coupling constants (and multiplicity) were determined from appropriate ^{1}H selective homodecoupled spectra. Values of coupling constants lower than 0.5 Hz were usually observed as a broadening of the corresponding ^{1}H NMR signals; they were disclosed only in a series of selective homodecoupling experiments.

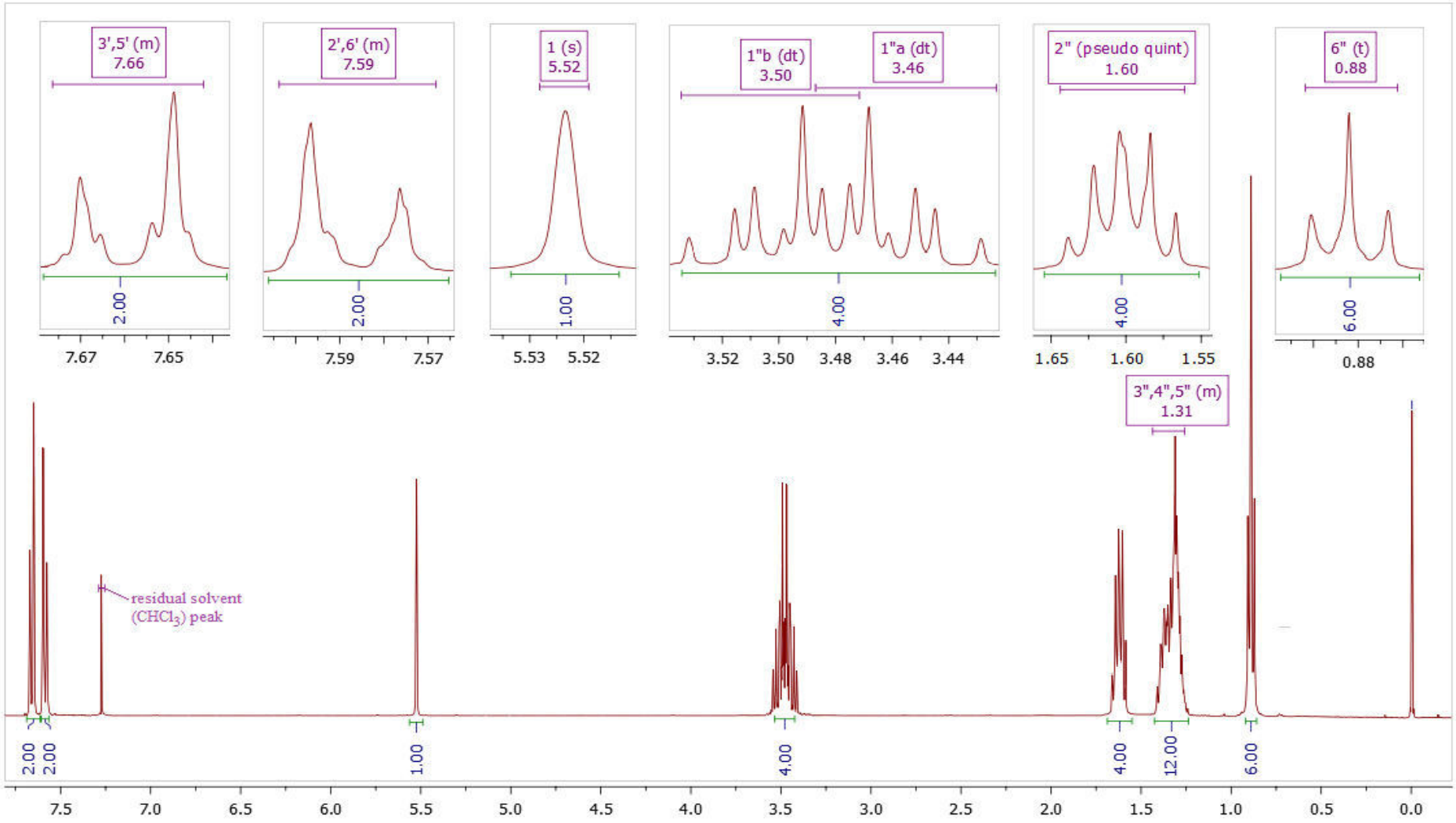
[c]Overlapped signals (range: 1.22-1.42 ppm, 12 H). Chemical shifts were determined from HSQC and HMBC spectra.



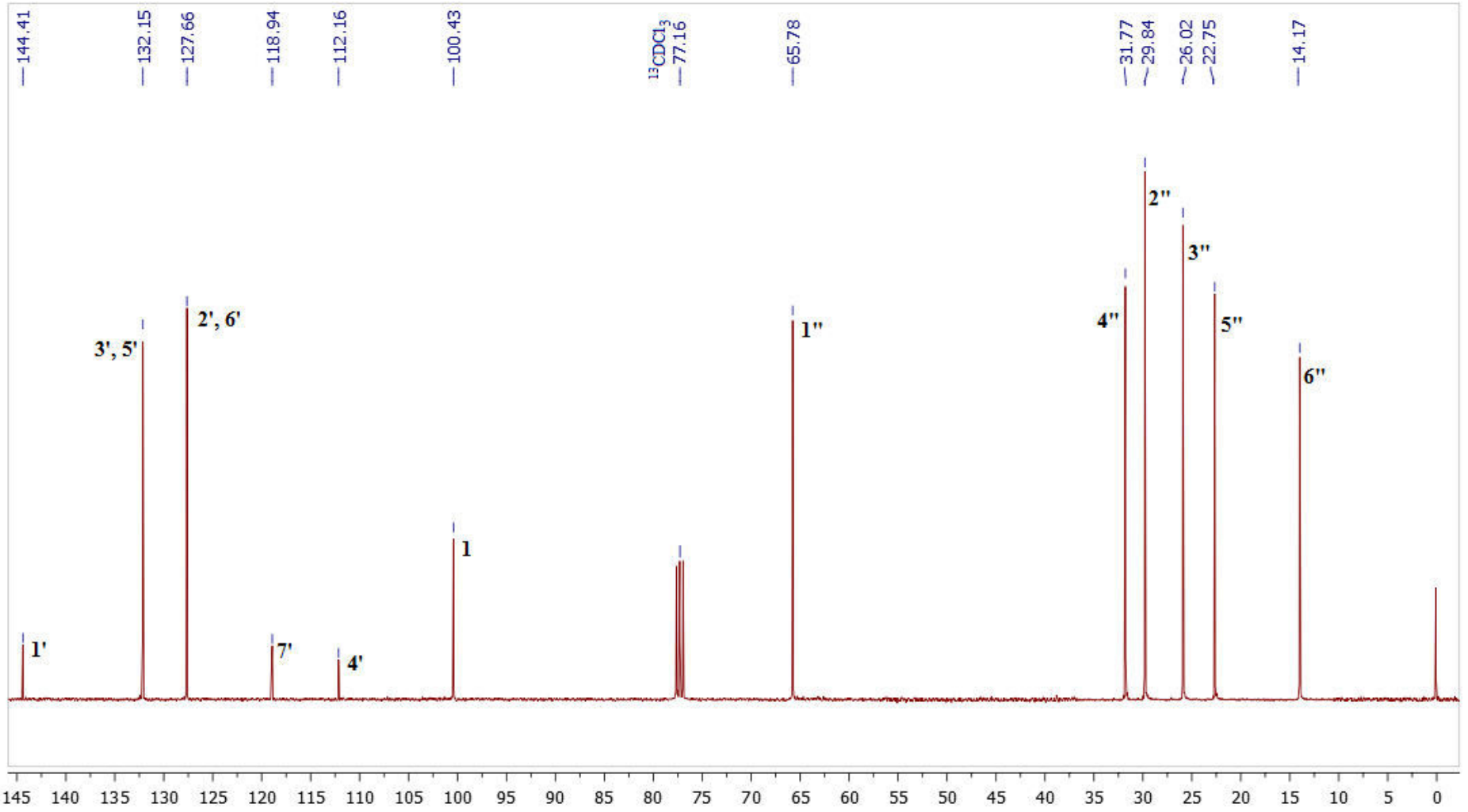
Scheme with key HMBC and NOESY interactions



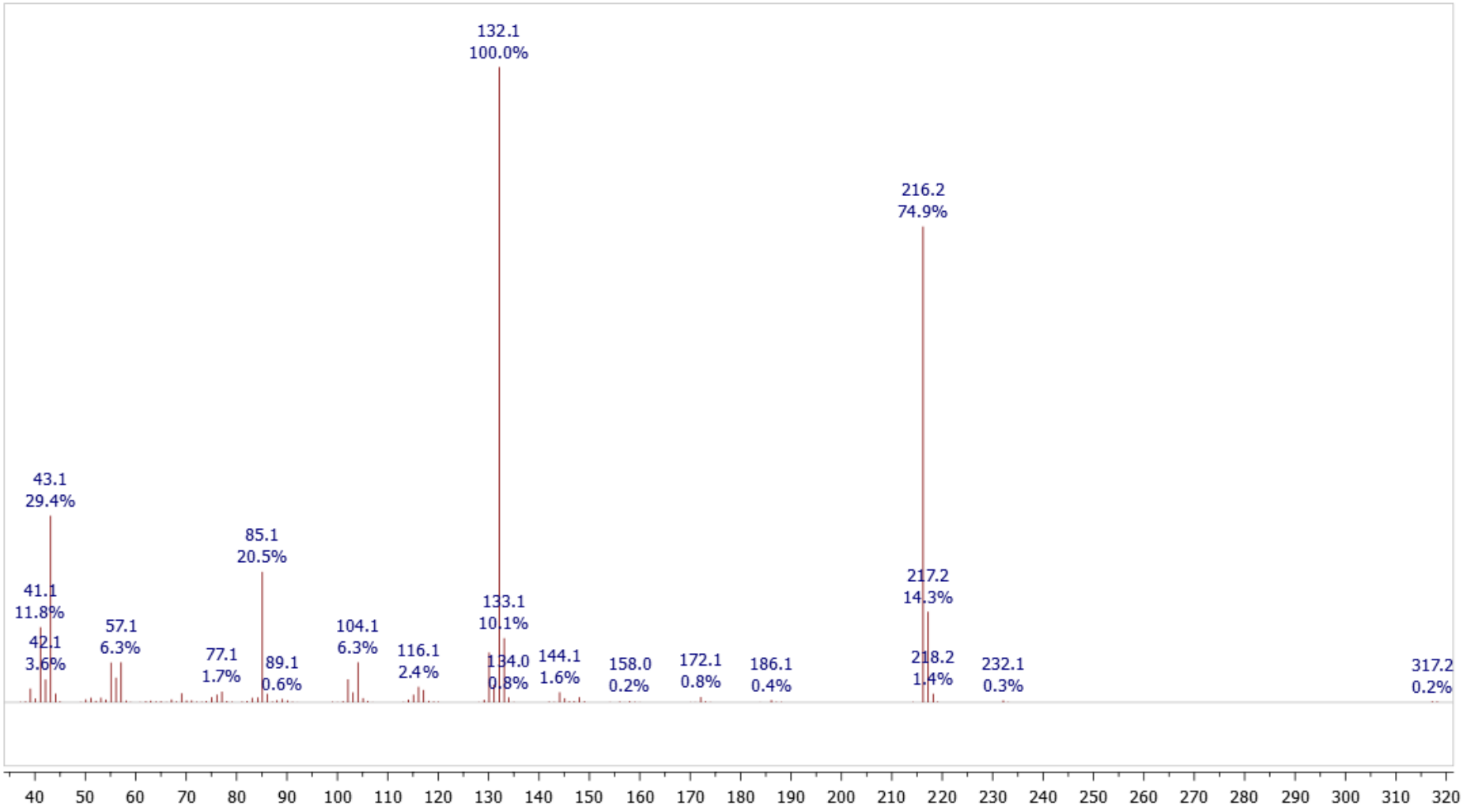
Analysis of ^{1}H-^{1}H coupling constants



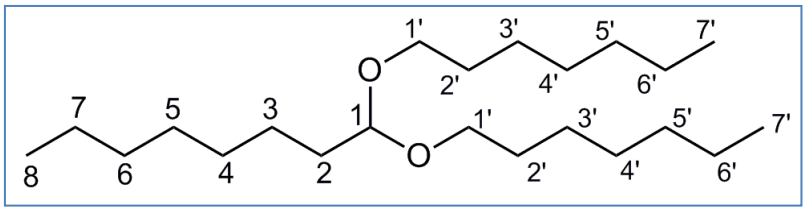
^{1}H-NMR (400 MHz, $CDCl_3$) spectrum of 4-(bis(hexyloxy)methyl)benzonitrile and the corresponding expansions with signal assignment



^{13}C-NMR (100.6 MHz, $CDCl_3$) spectrum of 4-(bis(hexyloxy)methyl)benzonitrile with signal assignment



EI-MS spectrum of 4-(bis(hexyloxy)methyl)benzonitrile



1,1-diheptoxyoctane

Table of NMR data of 1,1-diheptoxyoctane (chemical shift and coupling constant data from ^{1}H and ^{13}C NMR spectra with the observed HMBC and NOESY interactions)

Position	δ_H (Integral, m, *J* (Hz))	δ_C (ppm) C-13 {H}	HMBC[a]	NOESY
1	4.46 (1 H, t, $^{3}J_{1,2}$ = 5.8)	103.30 (1 C)	3, 1’	2
2	1.60 (2 H, td, $^{3}J_{2,3}$ = 6.7, $^{3}J_{1,2}$ = 5.8)[b]	33.62 (1 C)	1, 3, 4	1, 3
3	1.34[c]	24.96 (1 C)	1, 2, 4[d]	2
4	1.31[c]	29.60 (1 C)	3, 5	/
5	1.29[c]	29.39 (1 C)	3, 4, 6[d]	/
6	1.27[c]	31.95 (1 C)	7[d], 8[d]	/
7	1.29[c]	22.79 (1 C)	6[d], 8[d]	8[d]
8	0.88 (t, $^{3}J_{5,6}$ = 6.7)	14.22 (1 C)	6[d], 7[d]	7[d]
1’a	3.40 (2 H, dt, $^{2}J_{1'a,1'b}$ = 9.3, $^{3}J_{1'a,2'}$ = 6.7)	65.58 (2 C)	1, 2’, 3’	2’, 1’b
1’b	3.56 (2 H, dt, $^{2}J_{1'a,1'b}$ = 9.3, $^{3}J_{1'b,2'}$ = 6.7)		1, 2’, 3’	2’, 1’a
2’	1.57 (4 H, tt, $^{3}J_{2',3'}$ = 6.8[b], $^{3}J_{1'a,2'}$ = $^{3}J_{1'b,2'}$ = 6.7)	30.08 (2 C)	1’, 3’, 4’	1’, 3’
3’	1.35[c]	26.40 (2 C)	1’, 2’, 4’[d]	2’
4’	1.29[c]	29.30 (2 C)	3’, 5’[d]	/
5’	1.27[c]	32.00 (2 C)	6’[d], 7’[d]	/
6’	1.29[c]	22.77 (2 C)	5’[d], 7’[d]	7’[d]
7’	0.88 (t, $^{3}J_{6',7'}$ = 6.9)[d]	14.22 (2 C)	5’[d], 6’[d]	6’[d]

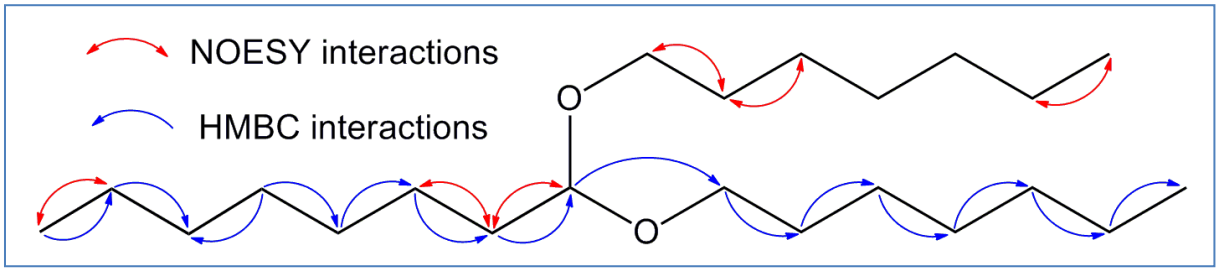
[a]Correlation between the hydrogen in this row and carbon in the listed position.

[b]Overlapped signals (range: 1.52-1.63 ppm, 6 H). Chemical shifts were determined from HSQC and HMBC spectra. Coupling constants were determined from the appropriate decoupled spectra.

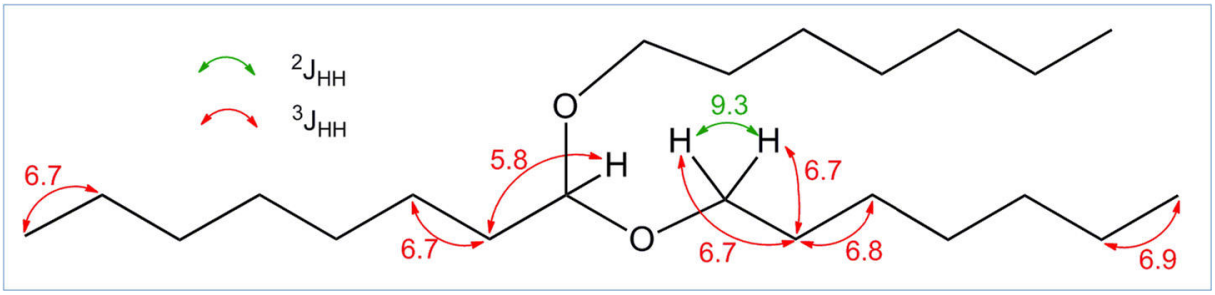
[c]Overlapped signals (range: 1.24-1.40 ppm, 26 H). Chemical shifts were determined from HSQC and HMBC spectra, and comparing with spectra of other, appropriate acetals.

[d]Signal isochronicity prevented a clear cut assignment of the particular HMBC interactions to a specific alkyl chain/both chains.

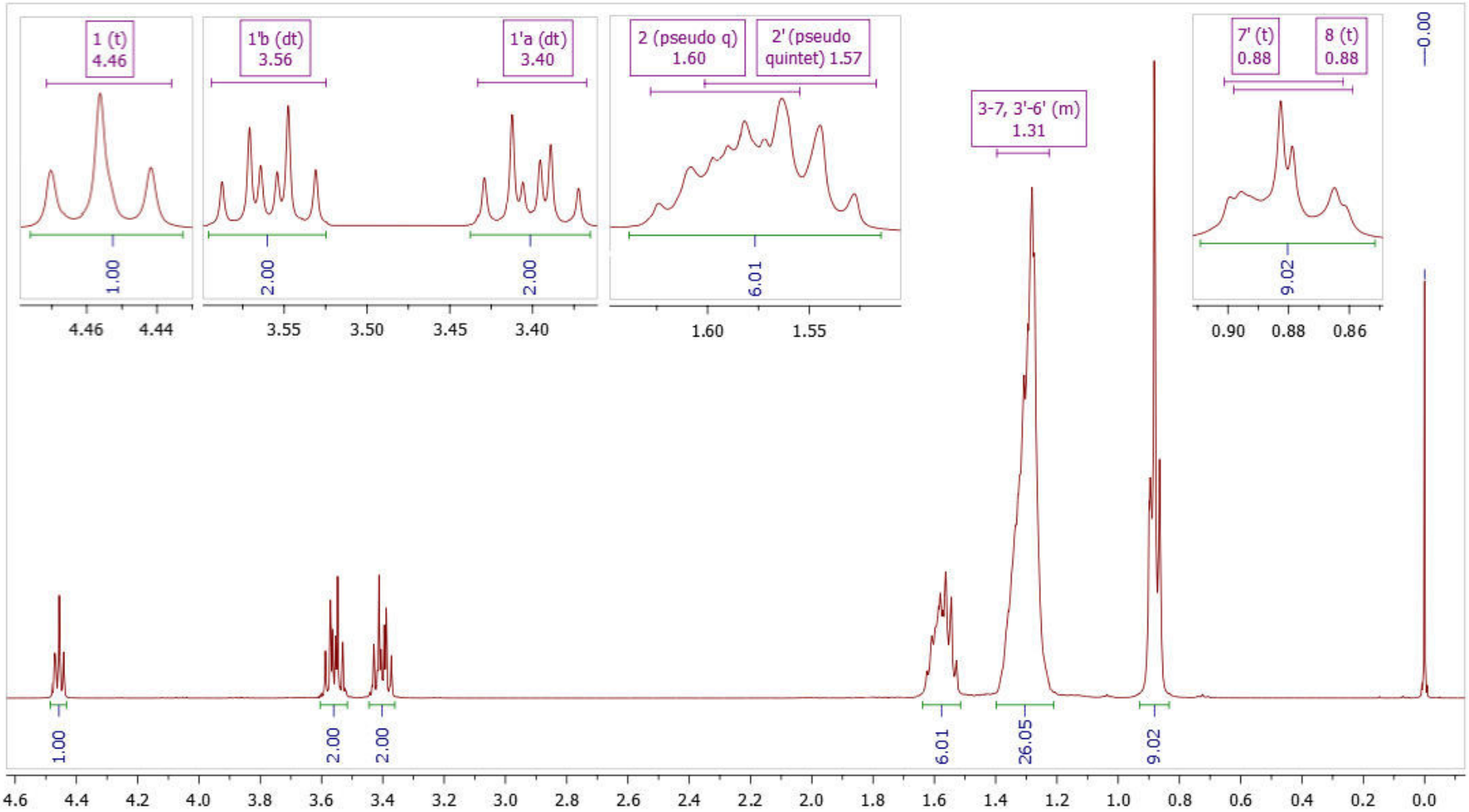
[d]Overlapped signals (range: 0.85-0.91 ppm). Total integral 9 H.



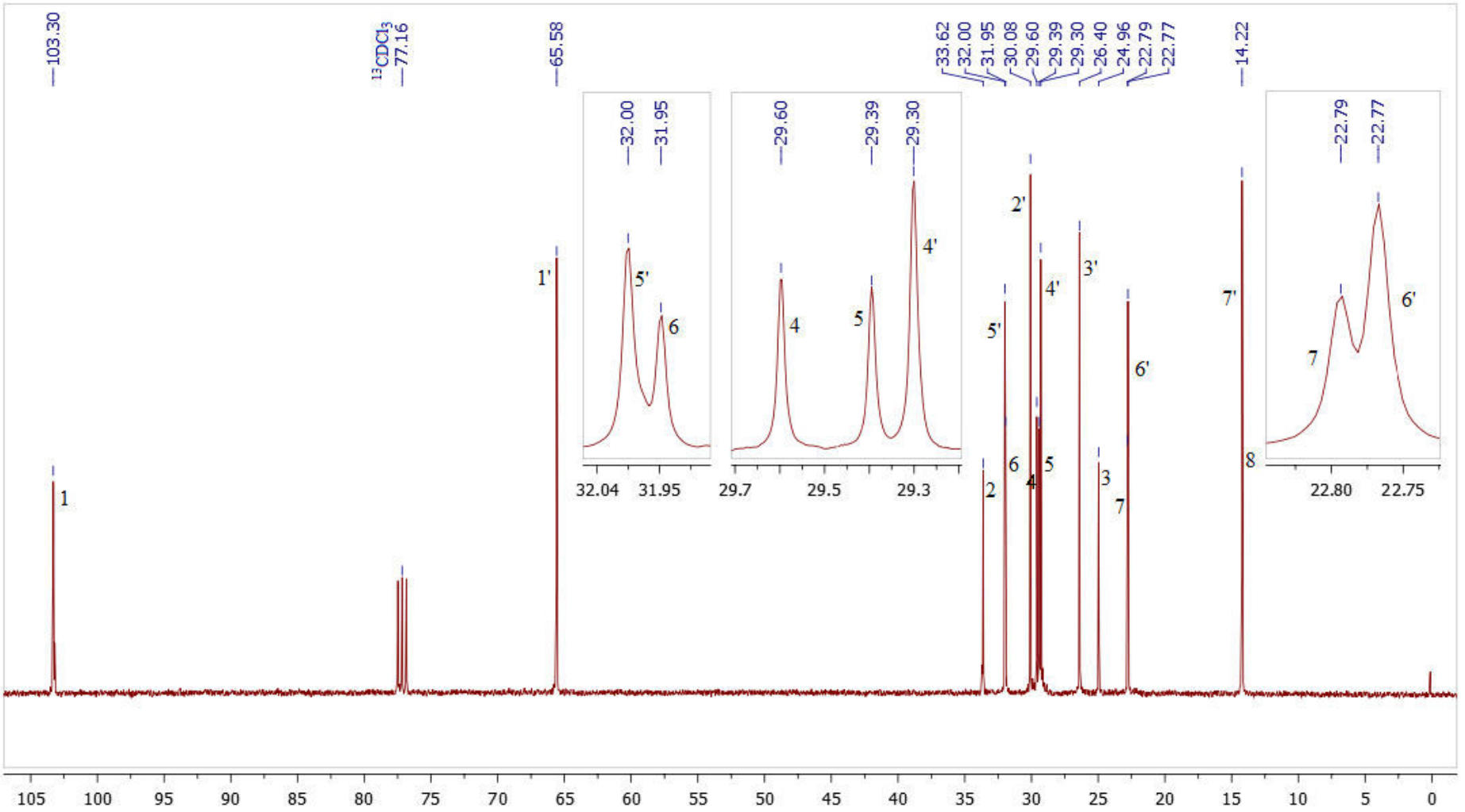
Scheme with key HMBC and NOESY interactions



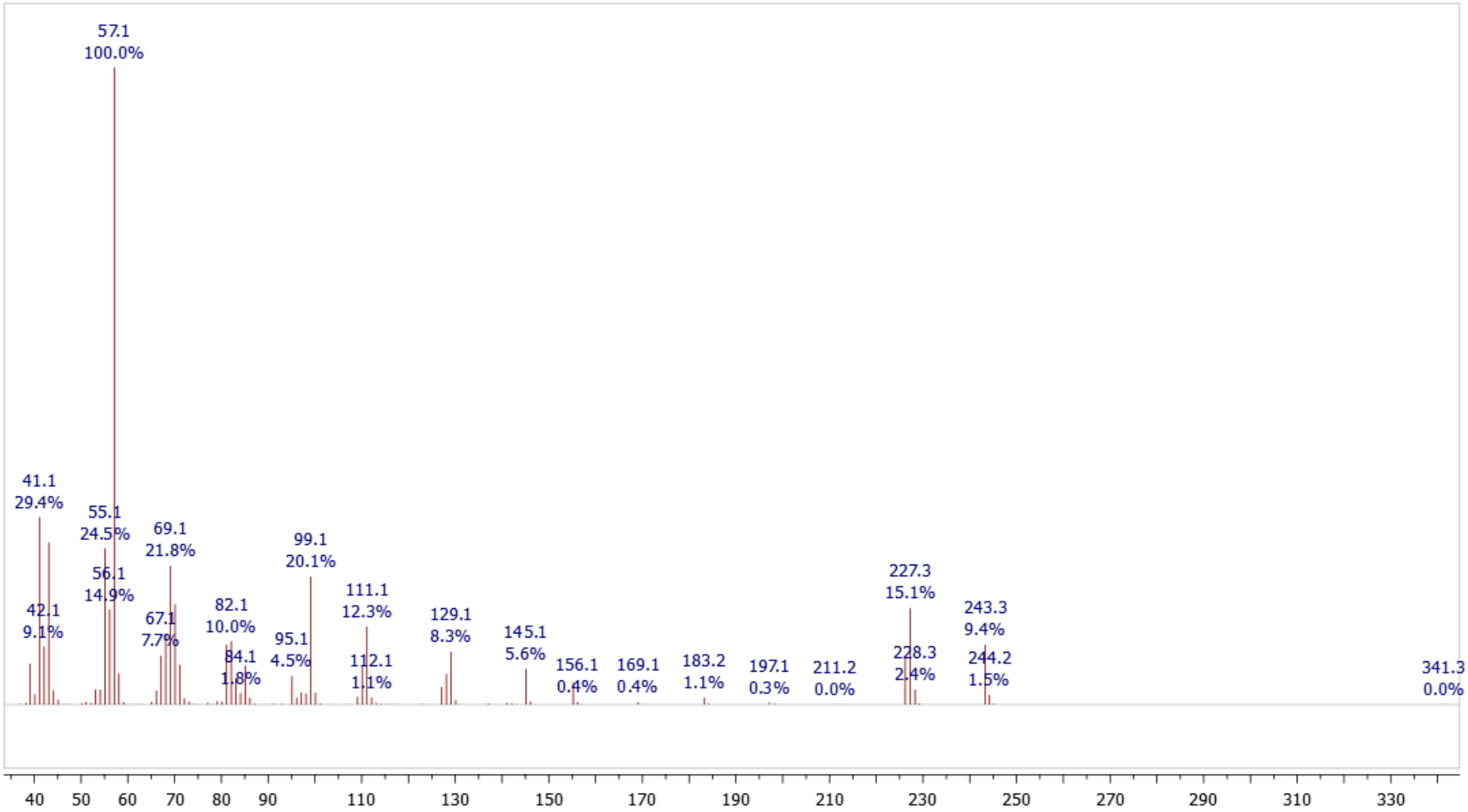
Analysis of ^{1}H-^{1}H coupling constants



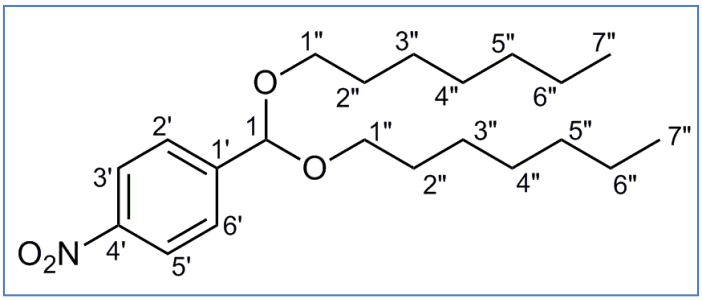
^{1}H-NMR (400 MHz, $CDCl_3$) spectrum of 1,1-diheptoxyoctane and the corresponding expansions with signal assignment



^{13}C-NMR (100.6 MHz, $CDCl_3$) spectrum of 1,1-diheptoxyoctane with signal assignment and the corresponding expansions



EI-MS spectrum of 1,1-diheptoxyoctane



1-(bis(heptyloxy)methyl)-4-nitrobenzene

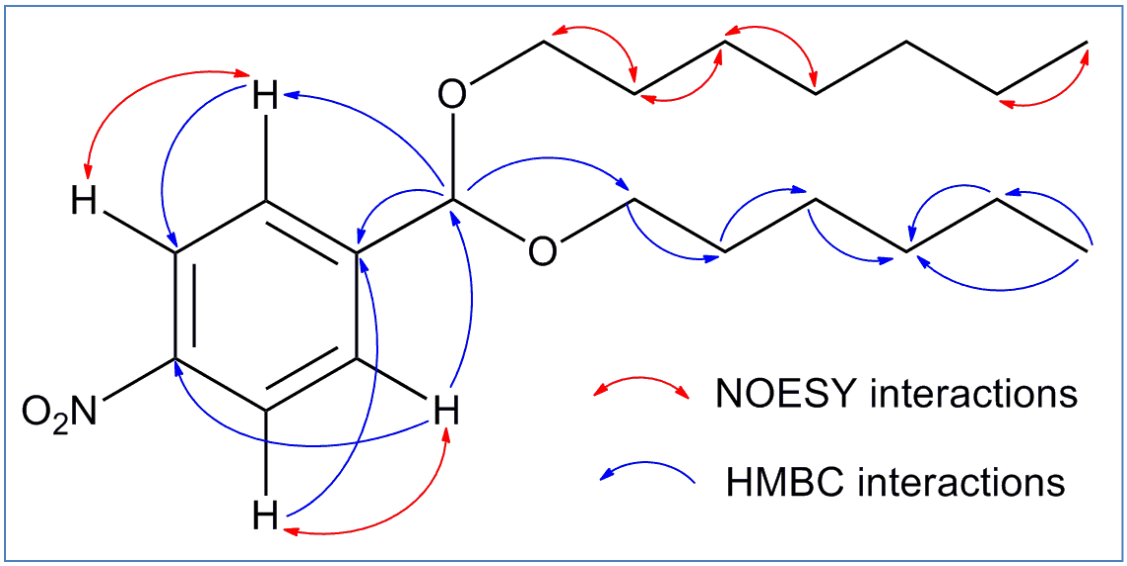
Table of NMR data of 1-(bis(heptyloxy)methyl)-4-nitrobenzene (chemical shift and coupling constant data from ^{1}H and ^{13}C NMR spectra with the observed HMBC and NOESY interactions)

Position	δ_H (Integral, m, *J* (Hz))	δ_C (ppm) C-13 {H}	HMBC[a]	NOESY
1	5.57 (1 H, t, $^{4}J_{1,2'} = {}^{4}J_{1,6'} = 0.5$)[b]	100.3 (1 C)	2', 6', 1"	/
1'	/	146.3 (1 C)	/	/
2', 6' (AA')	7.65 (2 H, m, AA'BB') ($^{3}J_{AB} = 8.7$, $^{4}J_{AA'} = 2.0$, $^{4}J_{BB'} = 2.3$, $^{5}J_{AB'/A'B} = 0.3$, $^{4}J_{AA',1} = 0.5$)[b]	127.9 (2 C)	1, 1', 3'/5', 4', (6'/2')	3'/5'
3', 5' (BB')	8,22 (2 H, m, AA'BB') ($^{3}J_{AB} = 8.7$, $^{4}J_{AA'} = 2.0$, $^{4}J_{BB'} = 2.3$, $^{5}J_{AB'/A'B} = 0.3$)[b]	123.5 (2 C)	1', 4', (5'/3')	2'/6'
4'	/	148.0 (1 C)	/	/
1"a	3.48 (2 H, dt, $^{2}J_{1"a,1"b} = 9.4$, $^{3}J_{1"a,2"} = 6.7$)	65.8 (2 C)	1, 2", 3"	2"
1"b	3.51 (2 H, dt, $^{2}J_{1"a,1"b} = 9.4$, $^{3}J_{1"b,2"} = 6.6$)			
2"	1.60 (4 H, qd, $^{3}J_{2",3"} = 6.9$, $^{3}J_{1"b,2"} = 6.7$, $^{3}J_{1"a,2"} = 6.6$)	29.8 (2 C)	1"a, 1"b, 3", 4"	1"a, 1"b, 3"
3"	1.36[c]	26.3 (2 C)	2", 4", 5"	2", 4"
4"	1.30[c]	29.2 (2 C)	3", 5"	3"
5"	1.28[c]	31.9 (2 C)	3"	/
6"	1.29[c]	22.7 (2 C)	5", 7"	7"
7"	0.88 (6 H, t, $^{3}J_{6",7"} = 6.9$)	14.2 (2 C)	5", 6"	6"

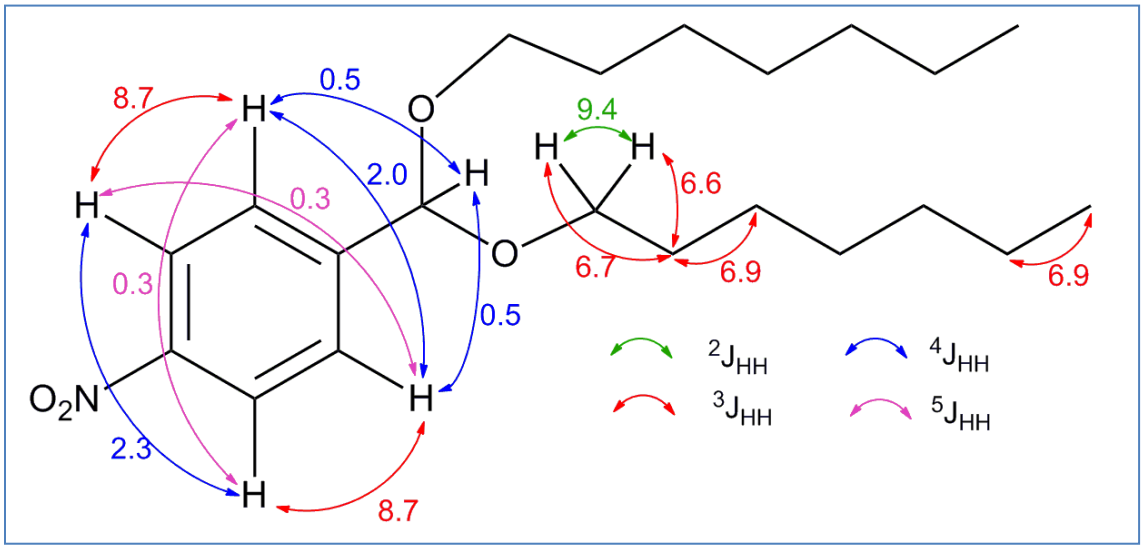
[a]Correlation between the hydrogen in this row and the carbon in the listed position.

[b]Coupling constants (and multiplicity) were determined from appropriate ^{1}H selective homodecoupled spectra. Values of coupling constants lower than 0.5 Hz were usually observed as a broadening of the corresponding ^{1}H NMR signals; they were disclosed only in a series of selective homodecoupling experiments.

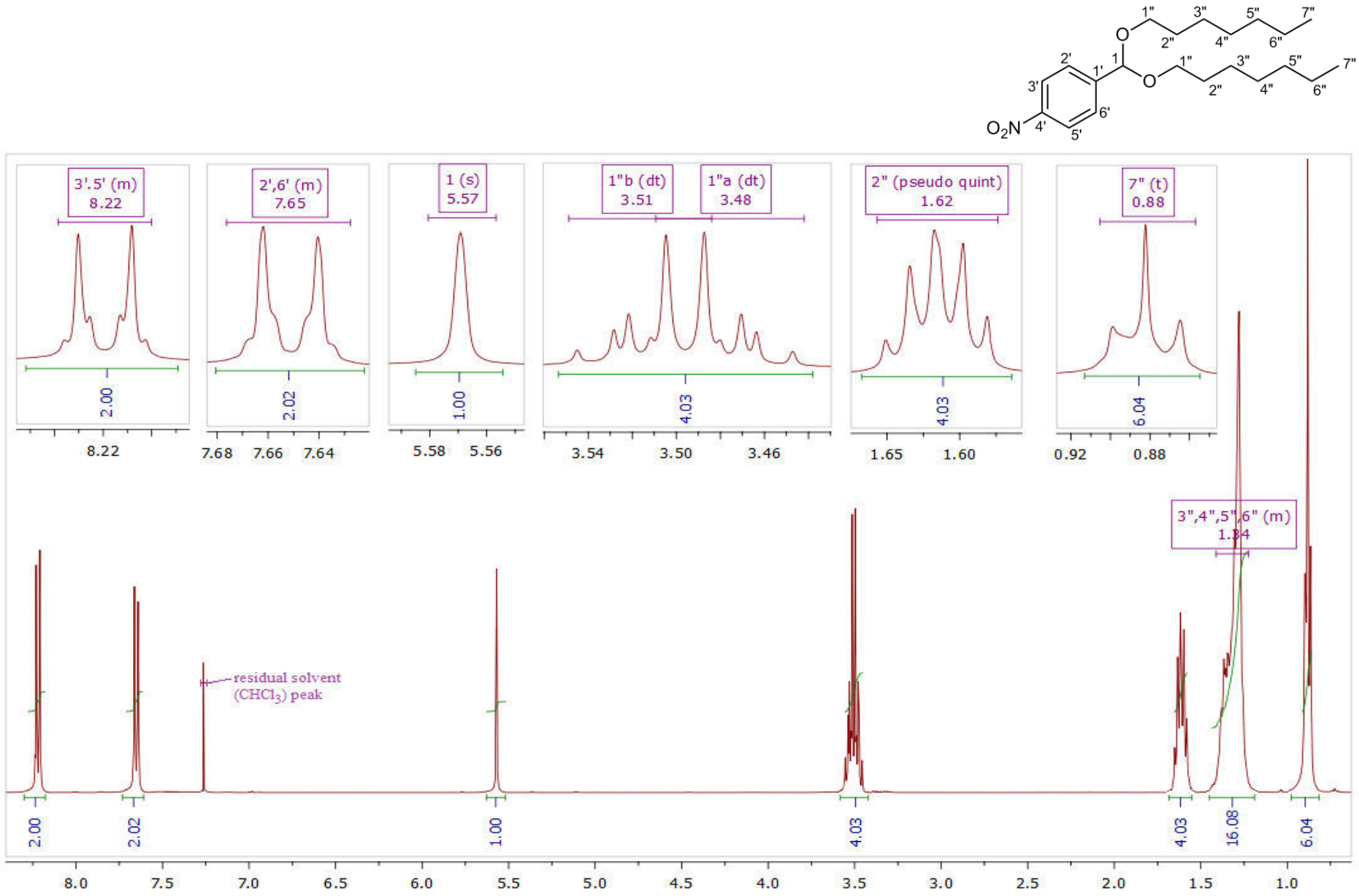
[c]Overlapped signals (range: 1.23-1.43 ppm, 16 H). Chemical shifts were determined from HSQC and HMBC spectra.



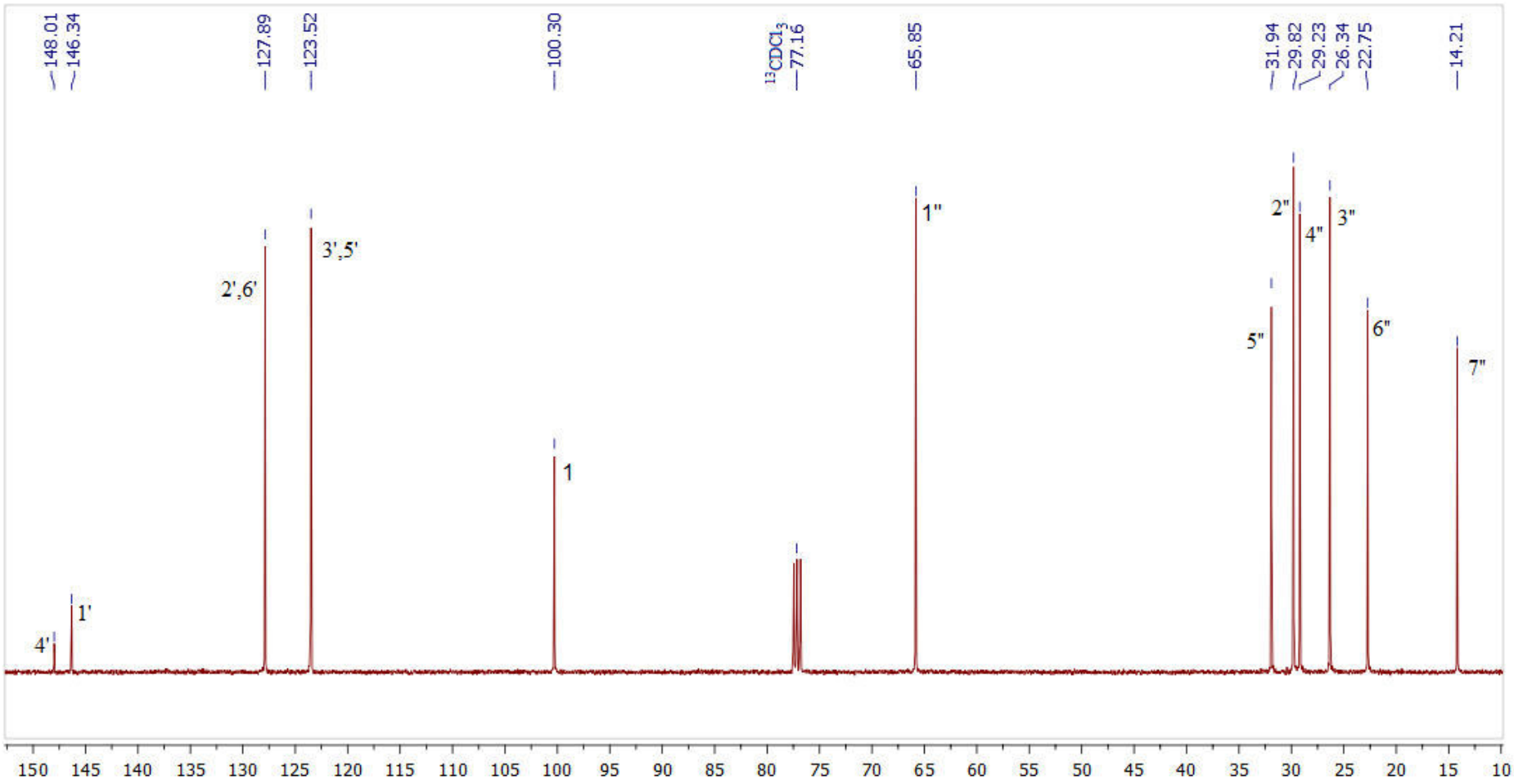
Scheme with key HMBC and NOESY interactions



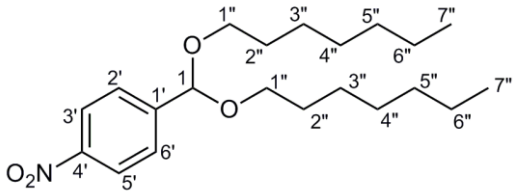
Analysis of ^{1}H-^{1}H coupling constants

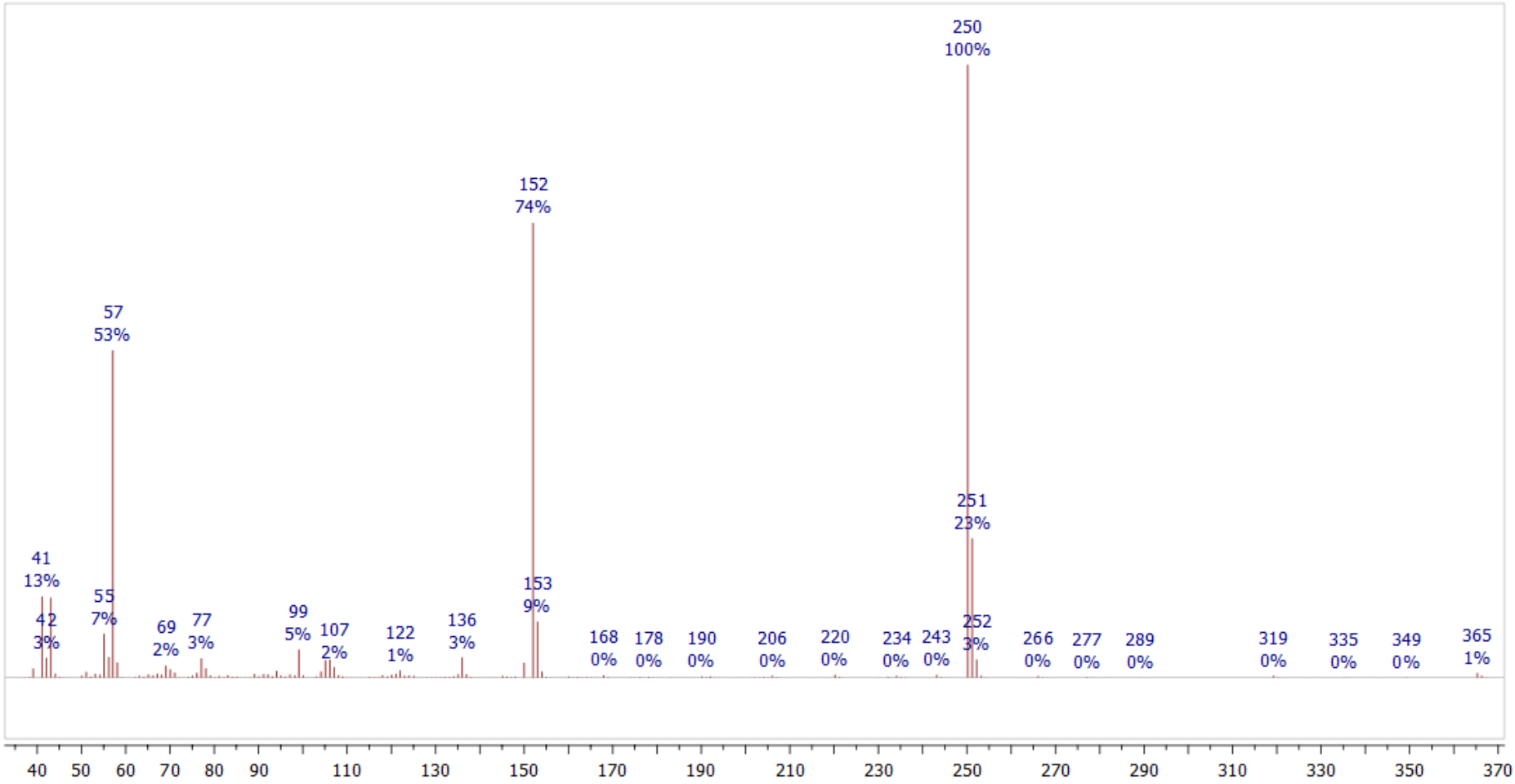


^{1}H-NMR (400 MHz, $CDCl_3$) spectrum of 1-(bis(heptyloxy)methyl)-4-nitrobenzene and the corresponding expansions with signal assignment

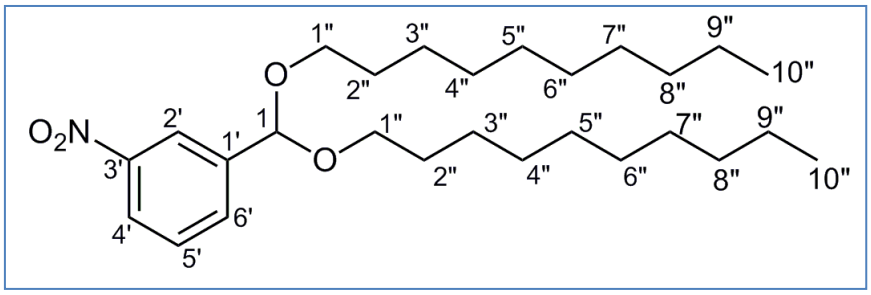


^{13}C-NMR (100.6 MHz, $CDCl_3$) spectrum of 1-(bis(heptyloxy)methyl)-4-nitrobenzene with signal assignment





EI-MS spectrum of 1-(bis(heptyloxy)methyl)-4-nitrobenzene

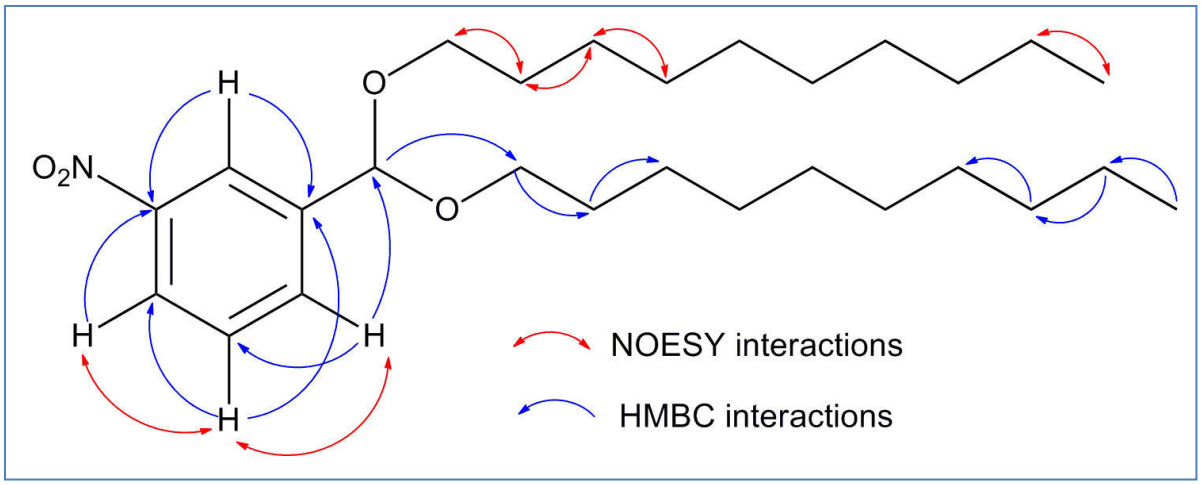


1-(bis(decyloxy)methyl)-3-nitrobenzene

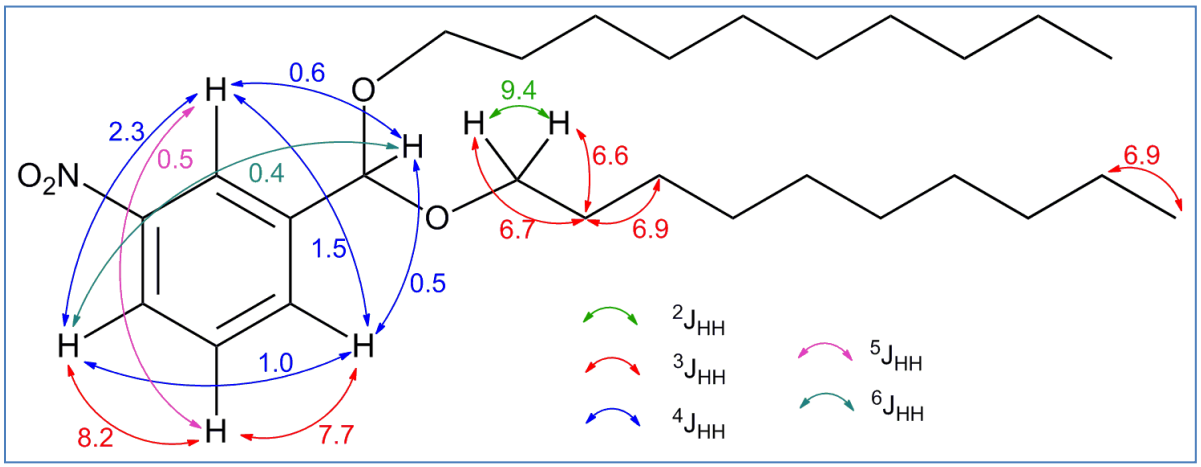
Table of NMR data of 1-(bis(decyloxy)methyl)-3-nitrobenzene (chemical shift and coupling constant data from ^{1}H and ^{13}C NMR spectra with the observed HMBC and NOESY interactions)

Position	δH (Integral, m, *J* (Hz))	δ_C (ppm) C-13 {H}	HMBC[a]	NOESY
1	5.57 (1 H, ddd, $^{4}J_{1,2'}$ = 0.6, $^{4}J_{1,6'}$ = 0.5, $^{6}J_{1,4'}$ = 0.4)[b]	100.2 (1 C)	2', 1"	/
1'	/	141.6 (1 C)	/	/
2'	8.34 (1 H, dddd, $^{4}J_{2',4'}$ = 2.3, $^{4}J_{2',6'}$ = 1.5, $^{4}J_{1,2'}$ = 0.6, $^{5}J_{2',5}$ = 0.5)[b]	122.1 (1 C)	1, 1', 3', 4'	/
3'	/	148.4 (1 C)	/	/
4'	8.18 (1 H, dddd, $^{3}J_{4',5'}$ = 8.2, $^{4}J_{2',4'}$ = 2.3, $^{4}J_{4',6'}$ = 1.0, $^{6}J_{1,4'}$ = 0.4)[b]	123.4 (1 C)	2', 3'	5'
5'	7.54 (1 H, ddd, $^{3}J_{4',5'}$ = 8.2, $^{3}J_{5',6'}$ = 7.7, $^{5}J_{2',5'}$ = 0.5)[b]	129.3 (1 C)	3', 1', 3', 4'	4', 6'
6'	7.81 (1 H, dddd, $^{3}J_{5',6'}$ = 7.7, $^{4}J_{2',6'}$ = 1.5, $^{4}J_{4',6'}$ = 1.0, $^{4}J_{1,6'}$ = 0.5)[b]	133.0 (1 C)	1, 4', 5'	5'
1"a	3.48 (2 H, dt, $^{2}J_{1"a,1"b}$ = 9.4, $^{3}J_{1"a,2"}$ = 6.6)	65.9 (2 C)	1, 2", 3"	2"
1"b	3.53 (2 H, dt, $^{2}J_{1"a,1"b}$ = 9.4, $^{3}J_{1"b,2"}$ = 6.7)			
2"	1.62 (4 H, tdd, $^{3}J_{2",3"}$ = 6.9, $^{3}J_{1"b,2"}$ = 6.7, $^{3}J_{1"a,2"}$ = 6.6)	29.8 (2 C)	1", 3", (4"-6")	1", 3"
3"	1.35[c]	26.4 (2 C)	1", 2", (4"-6")	2", 4"
4", 5", 6"[e]	1.28[c]	29.57 (2 C)[d] 29.72 (2 C) 29.73 (2 C)	3", 7"	/
7"	1.28[c]	29.5 (2 C)	8", 9"	6"
8"	1.27[c]	32.0 (2 C)	9"	/
9"	1.28[c]	22.8 (2 C)	8", 10"	7"
10"	0.88 (6 H, t, $^{3}J_{6",7"}$ = 6.9)	14.2 (2 C)	8", 9"	6"

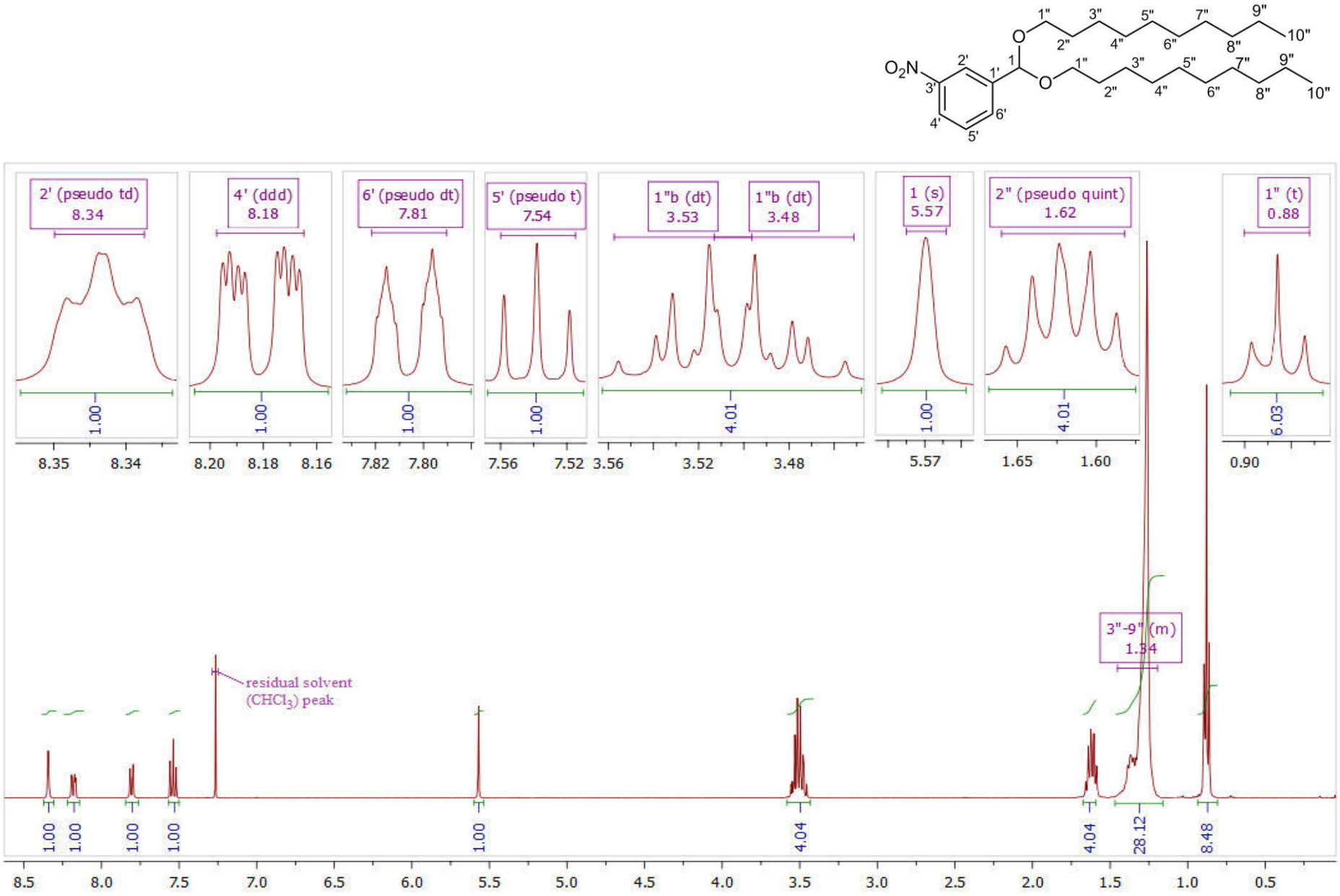
[a]Correlation between the hydrogen in this row and the carbon in the listed position.
[b]Coupling constants (and multiplicity) were determined from appropriate ^{1}H selective homodecoupled spectra. Values of coupling constants lower than 0.5 Hz were usually observed as a broadening of the corresponding ^{1}H NMR signals; they were disclosed only in a series of selective homodecoupling experiments.
[c]Overlapped signals (range: 1.23-1.43 ppm, 28 H). Chemical shifts were determined from HSQC and HMBC spectra. Diheptyl acetal of 3-nitrobenzaldehyde were used as a reference.
[d]Overlapped HSQC peaks. Signals in ^{13}C NMR spectrum are very close in value.



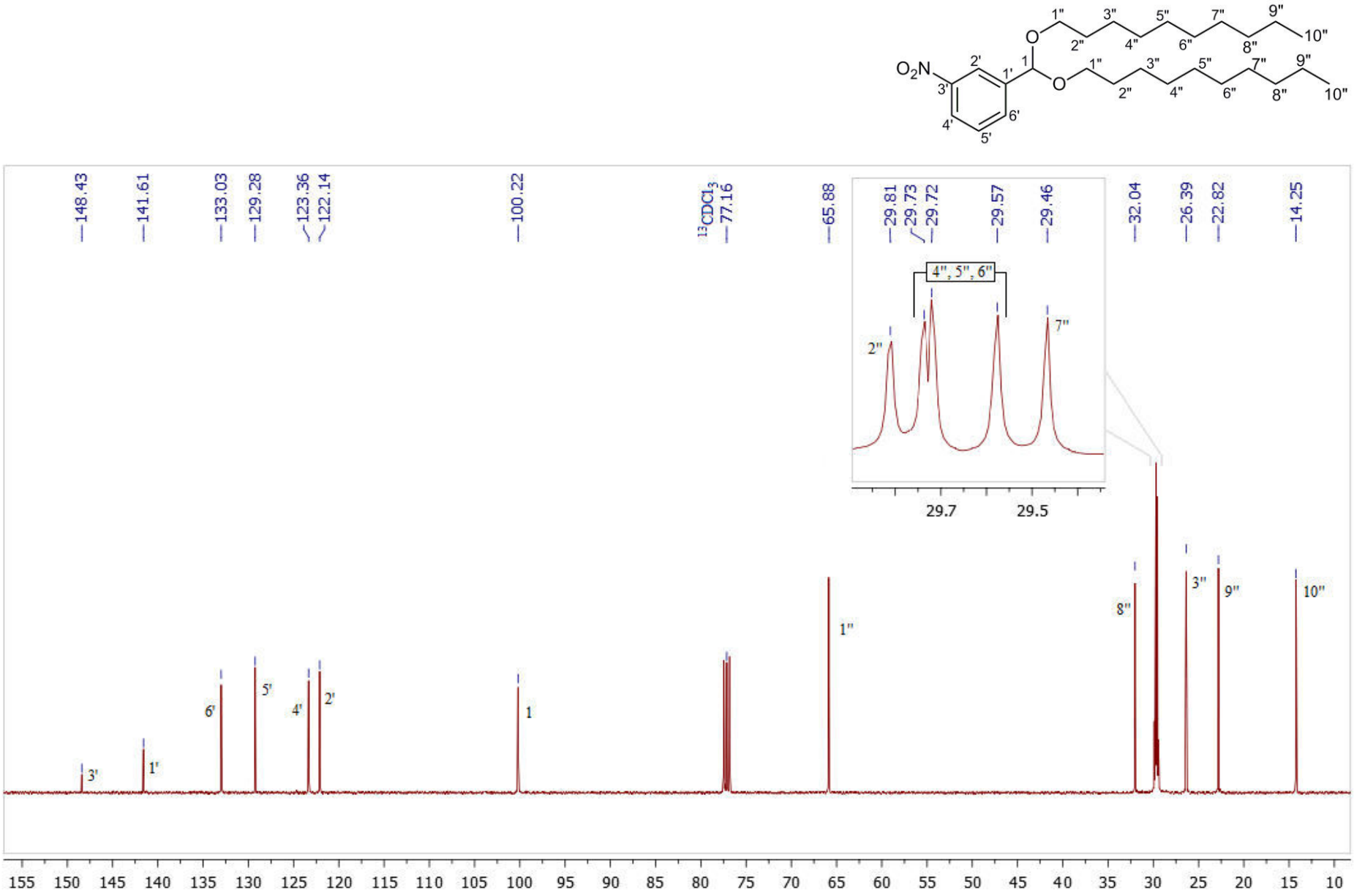
Scheme with key HMBC and NOESY interactions



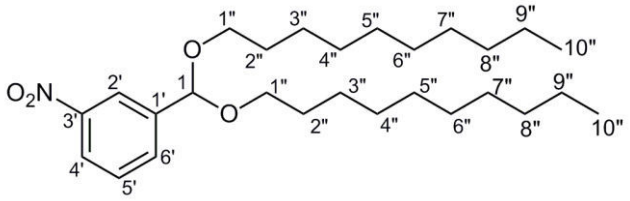
Analysis of ^{1}H-^{1}H coupling constants

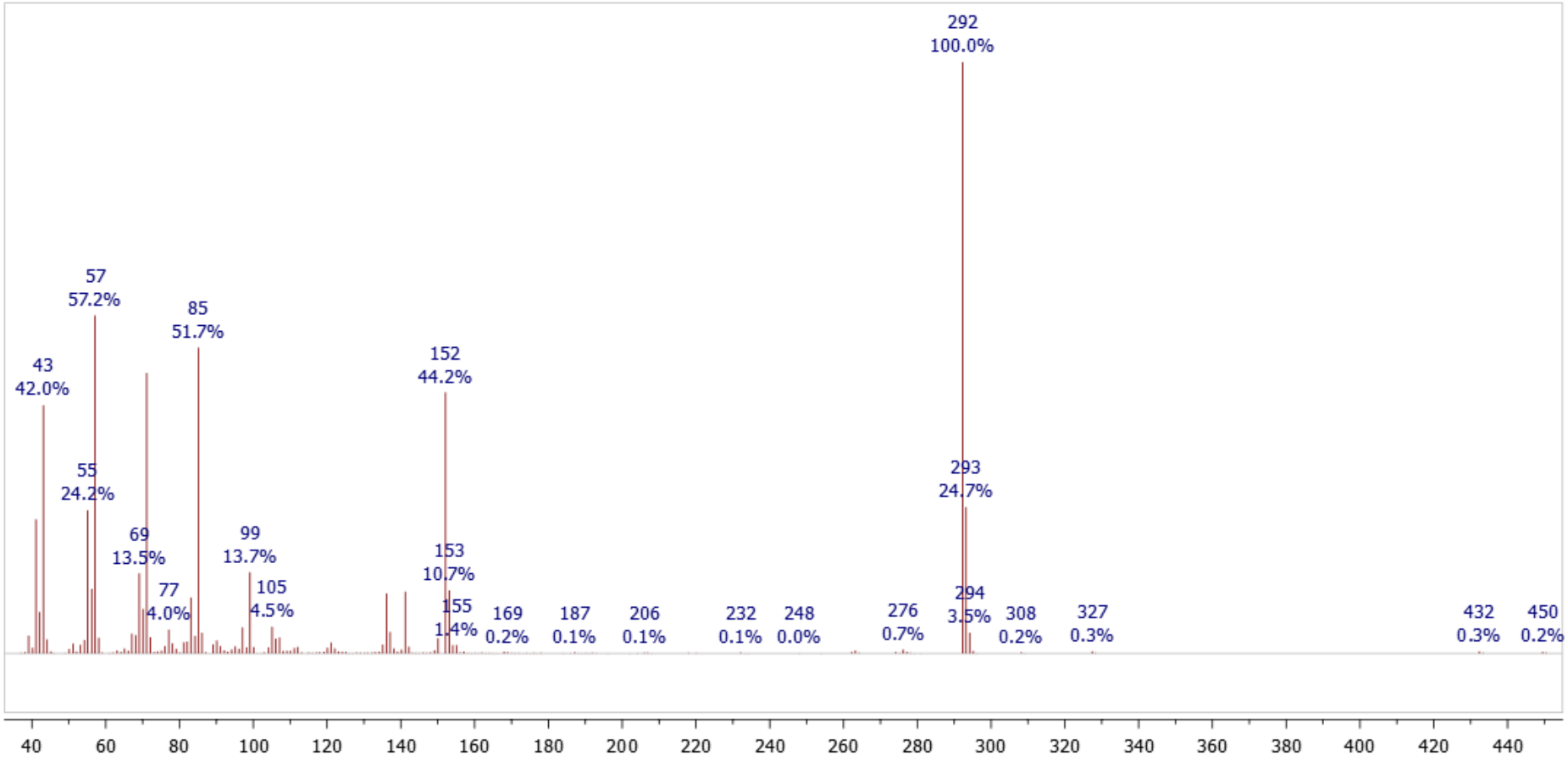


^{1}H-NMR (400 MHz, $CDCl_3$) spectrum of 1-(bis(decyloxy)methyl)-3-nitrobenzene and the corresponding expansions with signal assignment

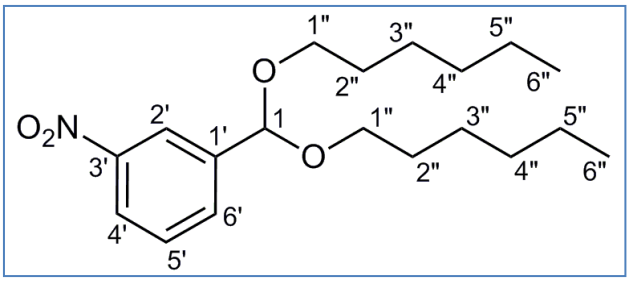


^{13}C-NMR (100.6 MHz, $CDCl_3$) spectrum of 1-(bis(decyloxy)methyl)-3-nitrobenzene with signal assignment and the corresponding expansion





EI-MS spectrum of 1-(bis(decyloxy)methyl)-3-nitrobenzene



1-(bis(hexyloxy)methyl)-3-nitrobenzene

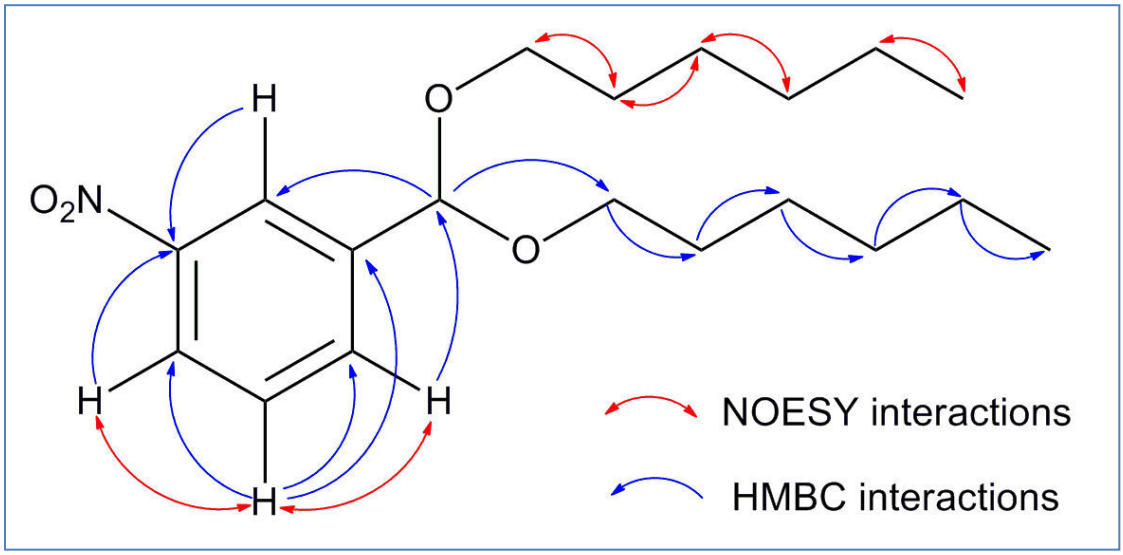
Table of NMR data of 1-(bis(hexyloxy)methyl)-3-nitrobenzene (chemical shift and coupling constant data from ^{1}H and ^{13}C NMR spectra with the observed HMBC and NOESY interactions)

Position	δ_H (Integral, m, *J* (Hz))	δ_C (ppm) C-13 {H}	HMBC[a]	NOESY
1	5.57 (1 H, ddd, $^{4}J_{1,2'}$ = 0.6, $^{4}J_{1,6'}$ = 0.5, $^{6}J_{1,4'}$ = 0.4)[b]	100.2 (1 C)	2', 1"	/
1'	/	141.6 (1 C)	/	/
2'	8.34 (1 H, dddd, $^{4}J_{2',4'}$ = 2.3, $^{4}J_{2',6'}$ = 1.5, $^{4}J_{1,2'}$ = 0.6, $^{5}J_{2',5'}$ = 0.5)[b]	122.1 (1 C)	1, 1', 3', 4'	/
3'	/	148.4 (1 C)	/	/
4'	8.18 (1 H, dddd, $^{3}J_{4',5'}$ = 8.2, $^{4}J_{2',4'}$ = 2.3, $^{4}J_{4',6'}$ = 1.0, $^{4}J_{1,4'}$ = 0.4)[b]	123.4 (1 C)	2', 3'	5'
5'	7.54 (1 H, ddd, $^{3}J_{4',5'}$ = 8.2, $^{3}J_{5',6'}$ = 7.7, $^{5}J_{2',5'}$ = 0.5)[b]	129.3 (1 C)	1', 3', 4'	4', 6'
6'	7.81 (1 H, dddd, $^{3}J_{5',6'}$ = 7.7, $^{4}J_{2',6'}$ = 1.5, $^{4}J_{4',6'}$ = 1.0, $^{4}J_{1,6'}$ = 0.5)[b]	133.0 (1 C)	1, 4', 5'	5'
1"a	3.49 (2 H, dt, $^{2}J_{1"a,1"b}$ = 9.4, $^{3}J_{1"a,2"}$ = 6.6)	65.9 (2 C)	1, 2", 3"	2"
1"b	3.53 (2 H, dt, $^{2}J_{1"a,1"b}$ = 9.4, $^{3}J_{1"b,2"}$ = 6.7)			
2"	1.62 (4 H, tdd, $^{3}J_{2",3"}$ = 6.9, $^{3}J_{1"b,2"}$ = 6.7, $^{3}J_{1"a,2"}$ = 6.6)	29.8 (2 C)	1", 3", 4"	1", 3"
3"	1.38[c]	26.1 (2 C)	1", 2", 4"	2", 4"
4"	1.30[c]	31.8 (2 C)	3", 6"	/
5"	1.31[c]	22.7 (2 C)	4", 6"	7"
6"	0.89 (6 H, t, $^{3}J_{5",6"}$ = 6.9)	14.2 (2 C)	4", 5"	6"

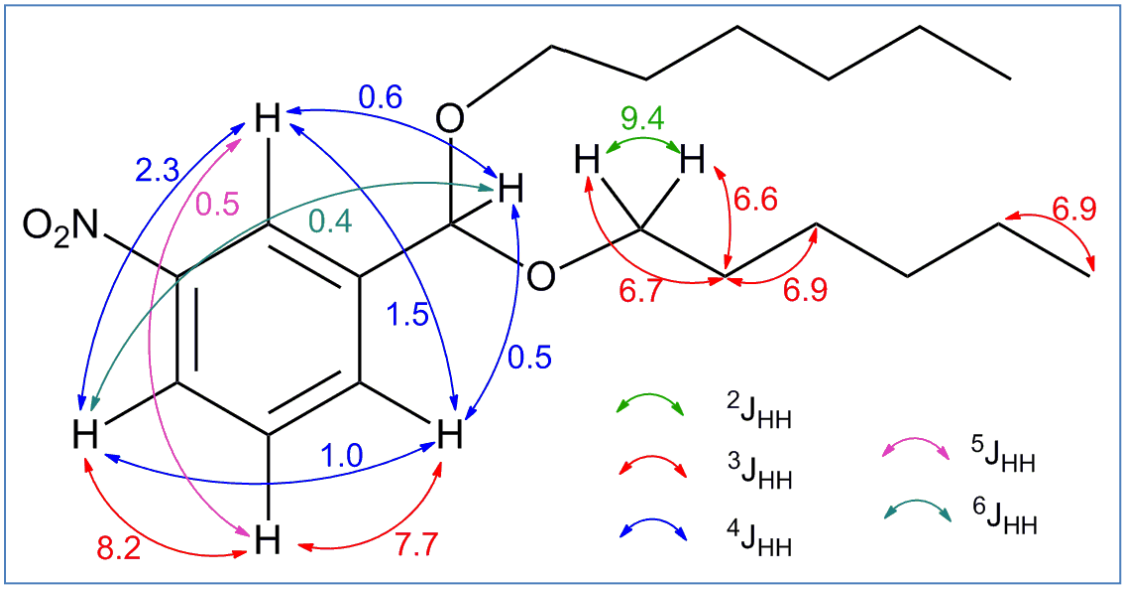
[a]Correlation between the hydrogen in this row and the carbon in the listed position.

[b]Coupling constants (and multiplicity) were determined from appropriate ^{1}H selective homodecoupled spectra. Values of coupling constants lower than 0.5 Hz were usually observed as a broadening of the corresponding ^{1}H NMR signals; they were disclosed only in a series of selective homodecoupling experiments.

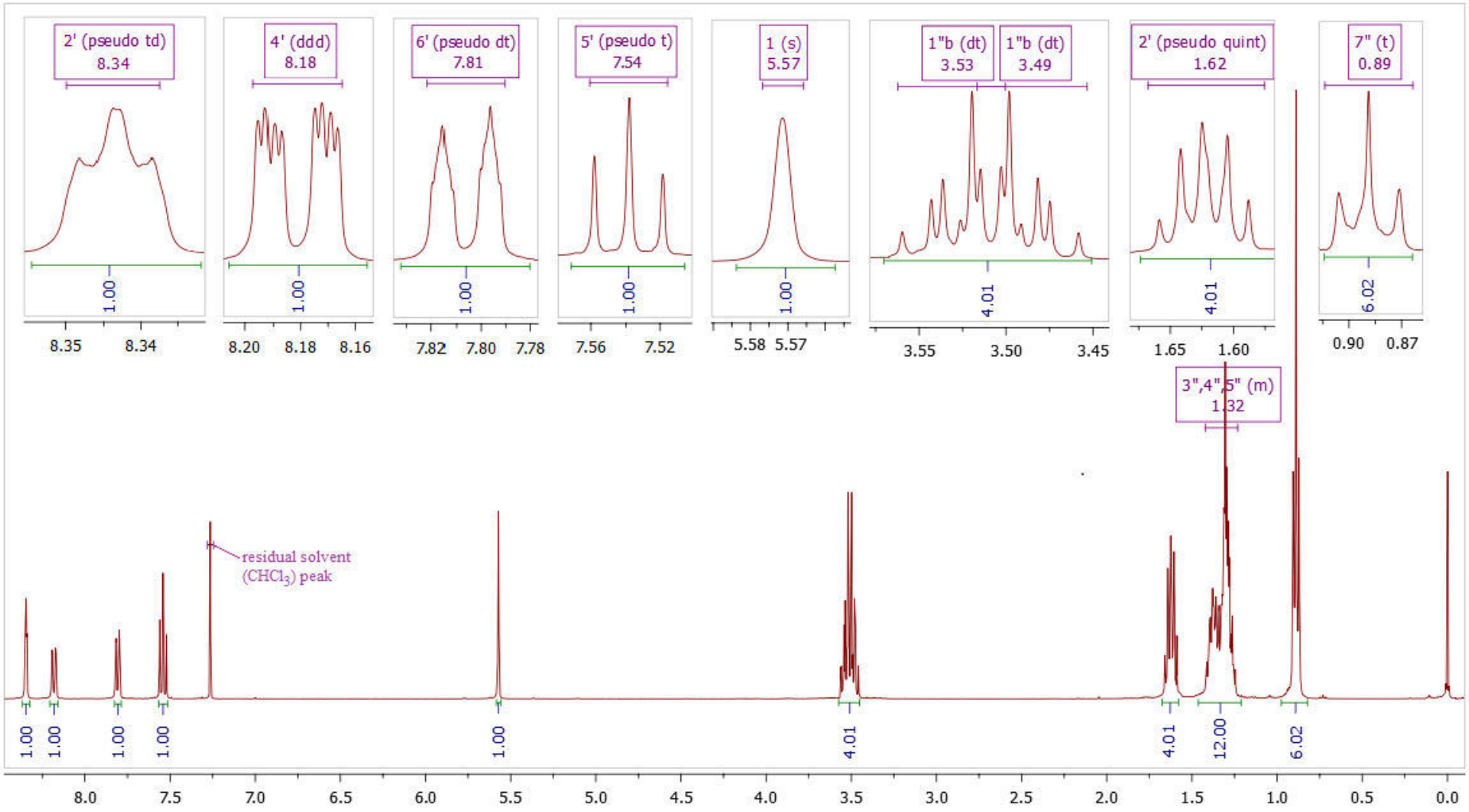
[c]Overlapped signals (range: 1.26-1.45 ppm, 12 H). Chemical shifts were determined from HSQC and HMBC spectra.



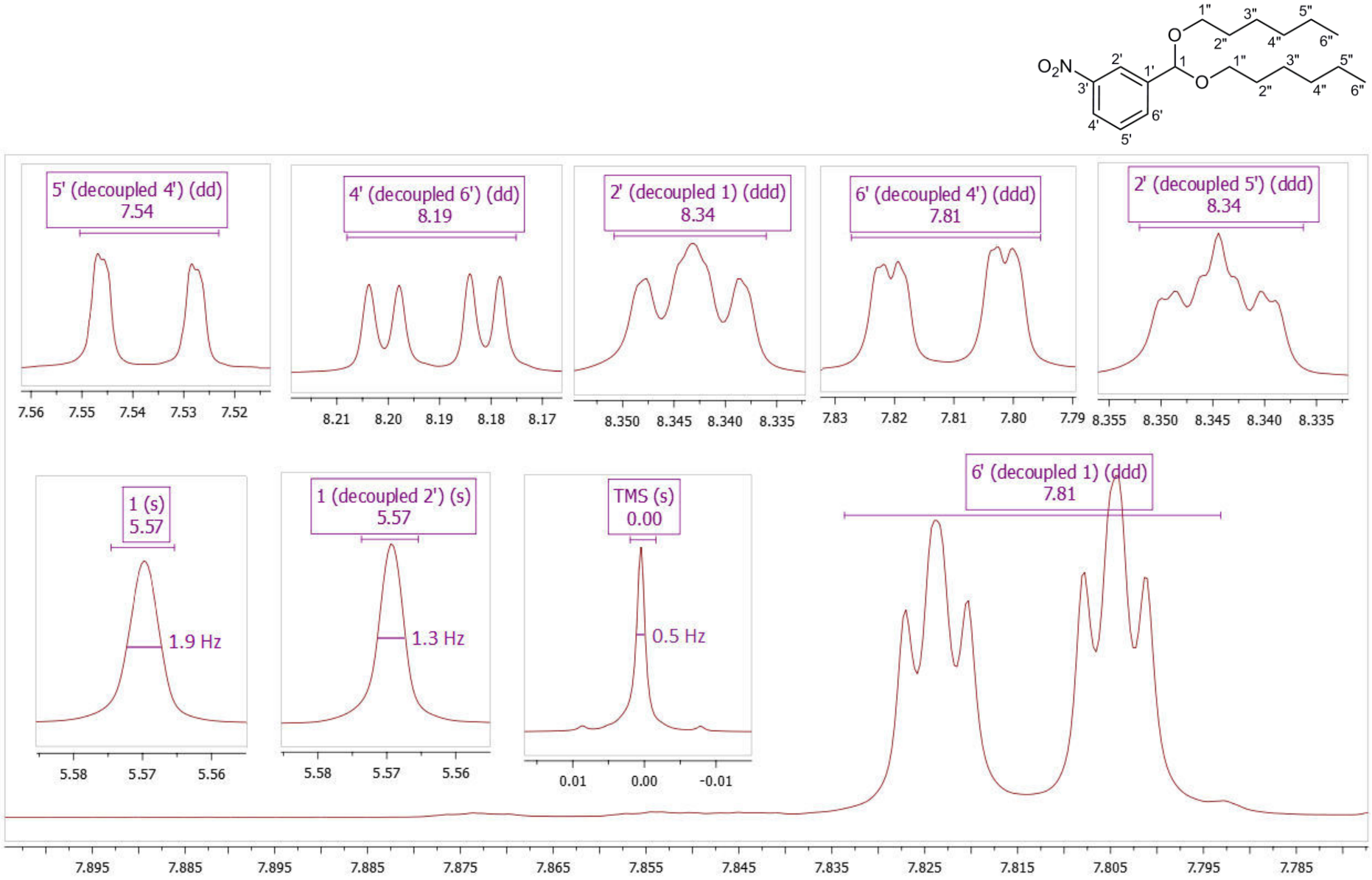
Scheme with key HMBC and NOESY interactions



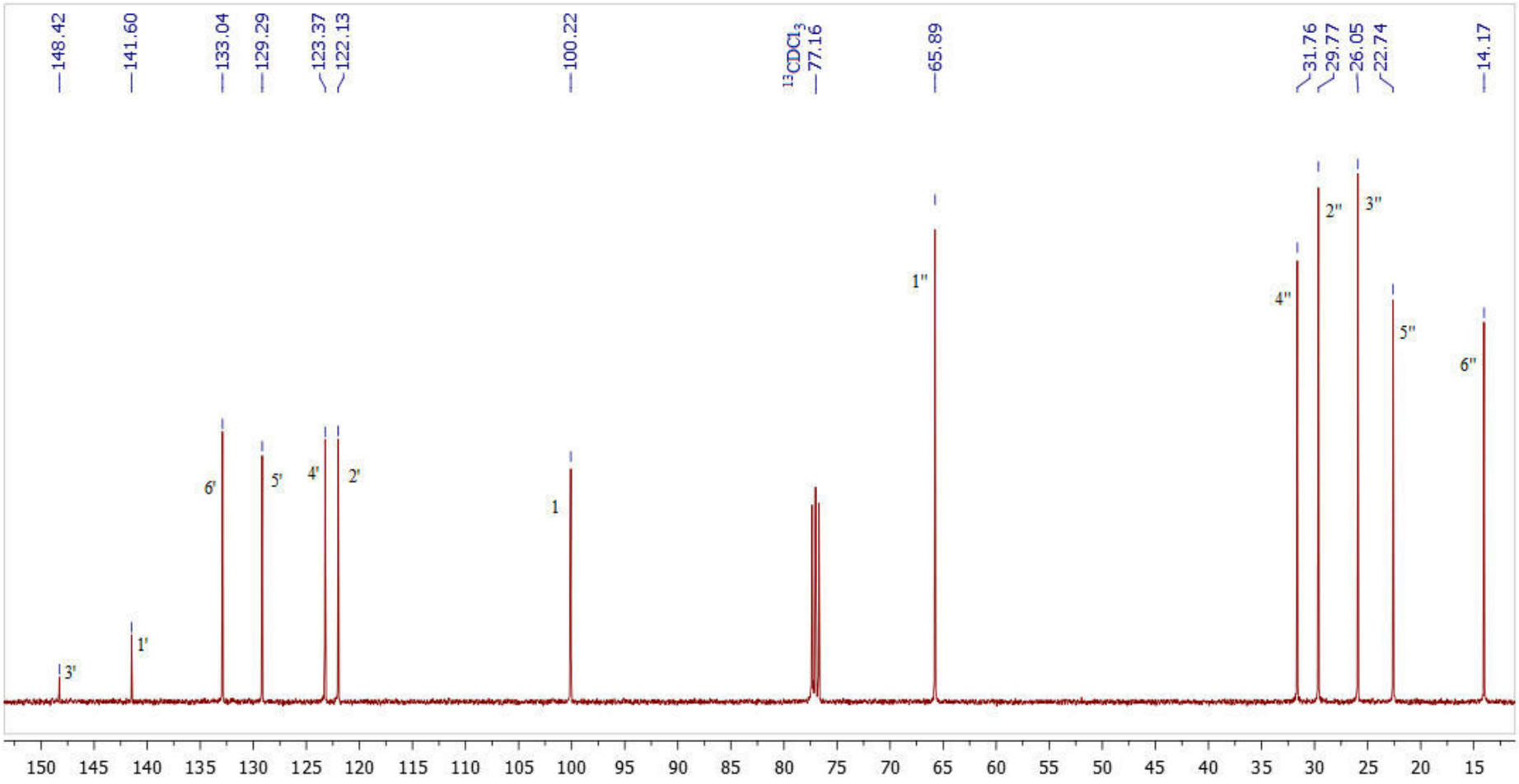
Analysis of ^{1}H-^{1}H coupling constants



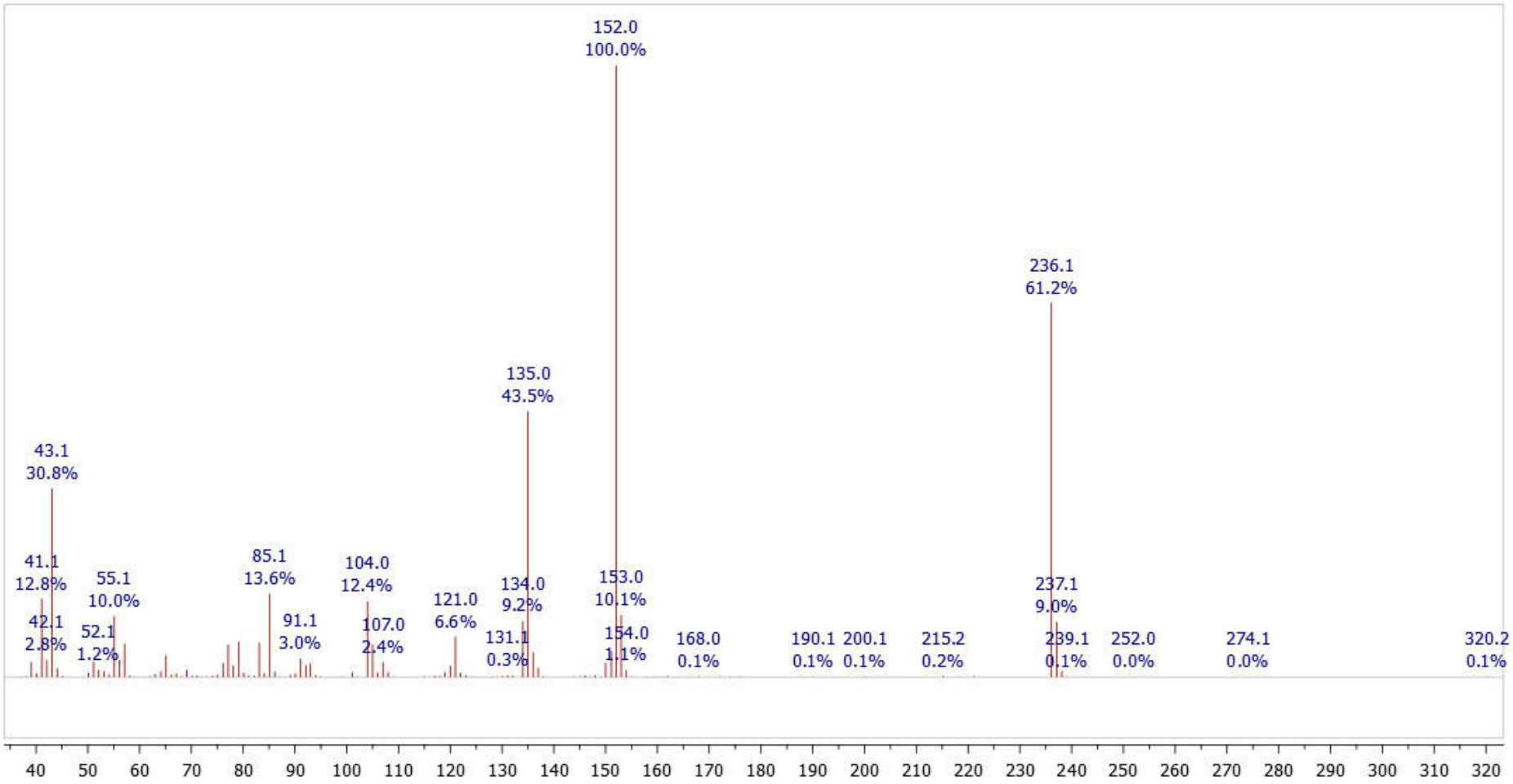
^{1}H-NMR (400 MHz, $CDCl_3$) spectrum of 1-(bis(hexyloxy)methyl)-3-nitrobenzene and the corresponding expansions with signal assignment



^{1}H NMR spectra obtained in a series of ^{1}H selective homodecoupling experiments with signal assignment



^{13}C-NMR (100.6 MHz, $CDCl_3$) spectrum of 1-(bis(hexyloxy)methyl)-3-nitrobenzene with signal assignment



EI-MS spectrum of 1-(bis(hexyloxy)methyl)-3-nitrobenzene