Supporting Information for

Use of Residual Dipolar Couplings in Conformational Analysis of Meta-Substituted Calix[4]arenes

Lukáš Vrzal,[a] Karolína Flídrová,[b] Tomáš Tobrman,[b] Hana Dvořáková,[a] and Pavel Lhoták*[b]

[a] Laboratory of NMR Spectroscopy, Institute of Chemical Technology Prague (ICTP), 166 28 Prague 8, Czech Republic

[b]Department of Organic Chemistry, ICTP, Prague 6, Czech Republic

E-mail: lhotakp@vscht.cz

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EXPERIMENTAL PROCEDURES AND CHARACTERIZATIONS

GENERAL EXPERIMENTAL PROCEDURES

All chemicals were purchased from commercial sources and used without further purification. Solvents were dried and distilled using conventional methods. Melting points were measured on Heiztisch Mikroskop – Polytherm A (Wagner & Munz, Germany). NMR spectra were performed on Varian Gemini 300 (^{1}H: 300 MHz, ^{13}C: 75 MHz) and on Bruker Advance DRX 500 (^{1}H: 500 MHz, ^{13}C: 125 MHz) spectrometers. Deuterated solvents used are indicated in each case. Chemical shifts (δ) are expressed in ppm and are referred to the residual peak of the solvent or TMS as an internal standard; coupling constants (J) are in Hz. The mass analyses were performed using ESI technique on Q–TOF (Micromass) spectrometer. Elemental analyses were done on Perkin–Elmer 240, Elementar vario EL (Elementar, Germany) or Mitsubishi TOX–100 instruments. All samples were dried in the desiccator over P_2O_5 under vacuum (1 Torr) at 80 °C for 8 hours. The IR spectra were measured on an FT–IR spectrometer Nicolet 740 or Bruker IFS66 spectrometers equipped with a heatable Golden Gate Diamante ATR–Unit (SPECAC) in KBr. 100 Scans for one spectrum were co–added at a spectral resolution of 4 cm^{-1}. The courses of the reactions were monitored by TLC using TLC aluminum sheets with Silica gel 60 F_{254} (Merck). The column chromatography was performed using Silica gel 60 (Merck).

General remark: All organomercury derivatives are considered potentially hazardous and require special consideration!

4, 17-BIS(CHLORO-MERCURIO)-25,26,27,28-TETRAPROPOXYCALIX[4]ARENE (2) AND 4, 18-BIS(CHLOROMERCURIO)-25,26,27,28-TETRAPROPOXYCALIX[4]ARENE (3)

They were prepared on a 0.5 g scale using procedure currently published by our group: Flídrová K., Böhm S., Dvořáková H., Eigner V., Lhoták P.: *Organic Letters*, **2014**, *16*, 138-141.

GENERAL PROCEDURE FOR THE NITROSATION OF CHLOROMERCURIO CALIXARENES

A corresponding chloromercurio calixarene **2** or **3** was dissolved in chloroform and cooled down in an ice-bath. Isoamyl nitrite (1.5 eq. per chloromercurio group), concentrated hydrochloric acid (3 eq.) and acetic acid (6 eq.) were added and the reaction mixture was stirred at 0 °C for 3 hours. Saturated solution of $NaHCO_3$ was poured into the reaction mixture and the two-phase system was vigorously stirred for 20 minutes. The organic layer was separated, washed with solution of $NaHCO_3$, then twice with water, and dried over $MgSO_4$. The crude reaction mixture was separated using short column of silica gel (CH_2Cl_2:hexane 1:1, v/v) to remove inorganic by-products. Yields were essentially quantitative.

4,17-DINITROSO-25,26,27,28-TETRAPROPOXYCALIX[4]ARENE (4)

Compound **4** was obtained according to General procedure in 96% yield (0.14 g, yellow-green glass) using: 0.23 g of calixarene **2**, 0.15 ml of isoamyl nitrite, 0.30 ml of concentrated HCl, 0.70 ml of glacial AcOH and 25 ml of $CHCl_3$. M.p. glass-like compound. ^{1}H-NMR (300 MHz, $CDCl_3$, 25 °C) δ 7.53 (br s, 1H, ArH), 7.43 (br s, 1H, ArH), 6.81 (d,1H, J=8.5 Hz, ArH), 6.49 (d, 2H, J=4.9 Hz, ArH), 6.33 (d, 2H, J=3.7 Hz, ArH), 6.26-6.30 (m, 2H, ArH), 5.70 (d, 1H, J=8.5 Hz, ArH), 5.59 (d, 1H, J=13.5 Hz, $ArCH_2Ar$), 4.77 (d, 1H, J=13.3 Hz, $ArCH_2Ar$), 4.50-4.62 (m, 3H, $ArCH_2Ar$), 4.10-4.24 (m, 4H, OCH_2), 3.72-

3.96 (m, 4H, OCH_2), 3.37 (d, 2H, *J*=13.8 Hz, $ArCH_2Ar$), 3.21 (d, 1H, *J*=13.5 Hz, $ArCH_2Ar$), 1.87-2.13 (m, 8H, OCH_2CH_2), 0.96-1.13 (m, 12 H, CH_3) ppm.

^{13}C-NMR (75 MHz, $CDCl_3$, 25 °C) δ 166.0, 165.1, 163.9, 159.2, 156.1, 155.9, 146.5, 141.8, 137.5, 137.2, 134.0, 133.3, 132.8, 132.7, 129.3, 128.5, 128.3, 128.1, 127.8, 122.9, 122.8, 100.2, 77.5, 76.8, 31.6, 31.3, 31.2, 24.2, 23.7, 23.6, 23.5, 23.4, 10.8, 10.3 ppm.

HRMS (TOF MS ESI$^+$): [M+H]+ calculated: 651.34286 found: 651.34260 (20%); $[M+Na]^+$ calculated: 673.32481 found: 673.32451 (100%); $[M+K]^+$ calculated: 689.29875 found: 689.29825 (20%).

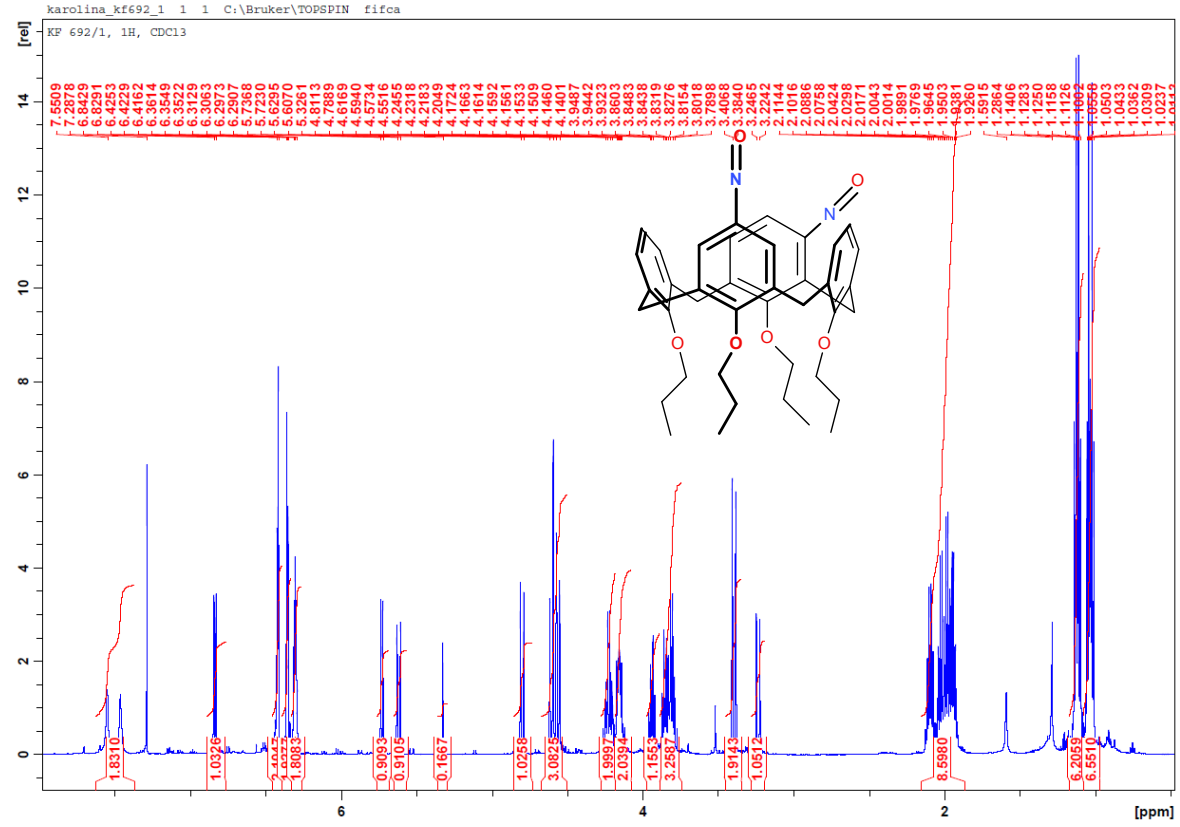
4,18-DINITROSO-25,26,27,28-TETRAPROPOXYCALIX[4]ARENE (5)

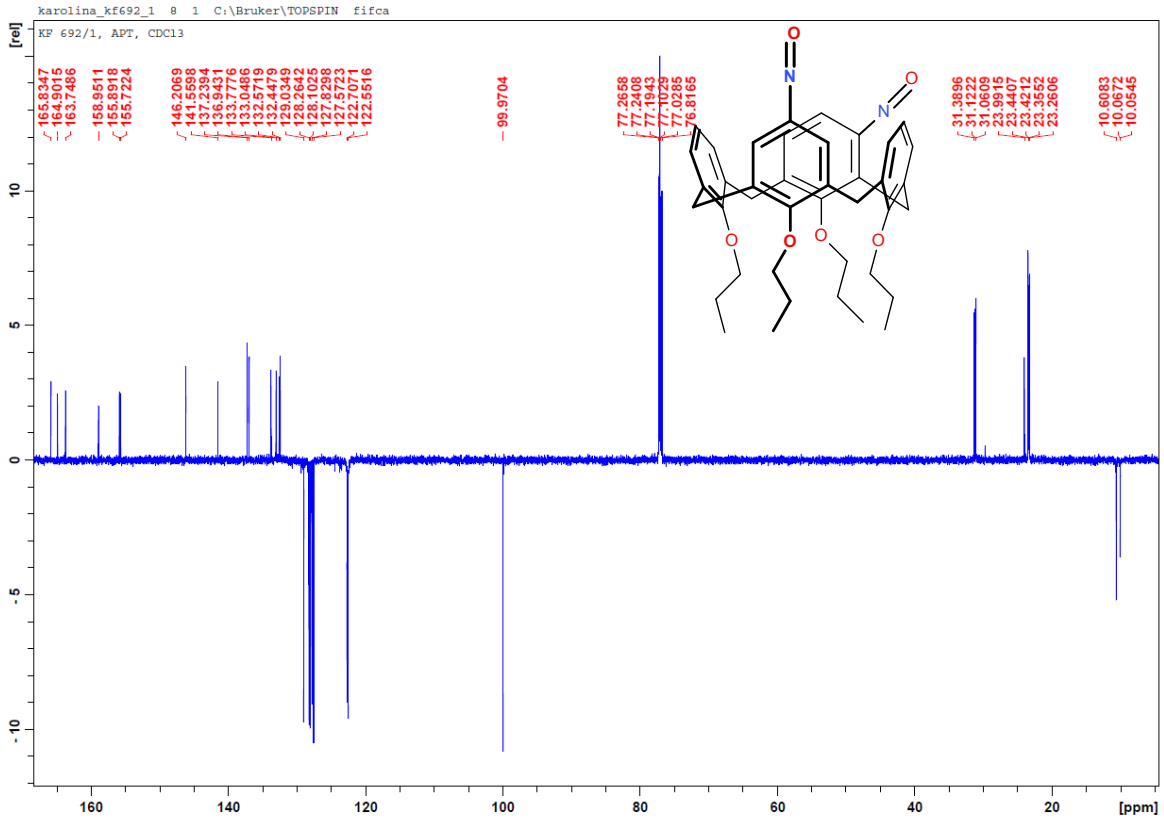
Compound **5** was obtained according to General procedure in 95% yield (0.11 g, yellow-green glass) using: 0.19 g of calixarene **3**, 0.10 ml of isoamyl nitrite, 0.20 ml of concentrated HCl, 0.65 ml of concentrated AcOH and 25 ml of $CHCl_3$. M.p. 78-82 °C. ^{1}H-NMR (300 MHz, $CDCl_3$, 25 °C) δ 6.98 (d, 2H, *J*=8.5 Hz, ArH), 6.23 (t, 2H, *J*=7.6 Hz, ArH), 6.10-6.19 (m, 4H, ArH), 5.94 (d, 2H, *J*=8.2 Hz, ArH), 5.72 (d, 2H, *J*=13.5 HZ, $ArCH_2Ar$), 4.80 (d, 2H, *J*=13.5 Hz, $ArCH_2Ar$), 4.59 (d, 2H, *J*=13.2 Hz, $ArCH_2Ar$), 4.15-4.33 (m, 4H, OCH_2), 3.72-3.92 (m, 4H, OCH_2), 3.26 (d, 2H, *J*=13.4 Hz, $ArCH_2Ar$), 2.07-2.22 (m, 4H, OCH_2CH_2), 1.88-2.02 (m, 4H, OCH_2CH_2), 1.17 (t, 6H, *J*=7.3 Hz, CH_3), 1.02 (t, 6H, *J*=7.6 Hz, CH_3) ppm.

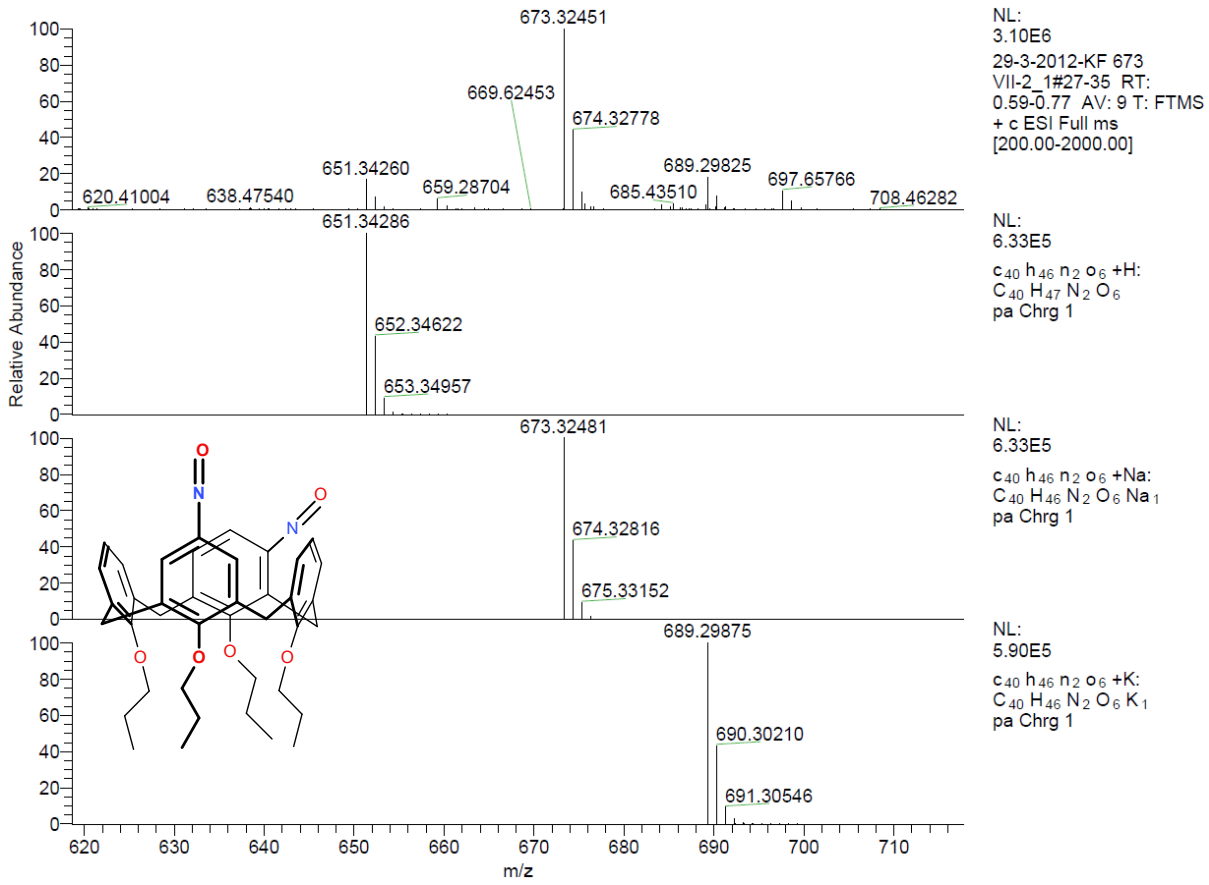
^{13}C-NMR (75 MHz, $CDCl_3$, 25 °C) δ 166.6, 159.7, 155.9, 147.4, 142.5, 133.5, 132.0, 129.0, 127.9, 127.6, 122.8, 100.2, 77.5, 77.3, 31.7, 23.8, 23.5, 23.3, 11.0, 10.2 ppm.

HRMS (TOF MS ESI$^+$): [M+H]+ calculated: 651.34286 found: 651.34290 (10%); $[M+Na]^+$ calculated: 673.32481 found: 673.32483 (100%); $[M+K]^+$ calculated: 689.29875 found: 689.29816 (20%). IR ν=1584 cm^{-1}.

CHARACTERIZATIONS OF COMPOUND 4



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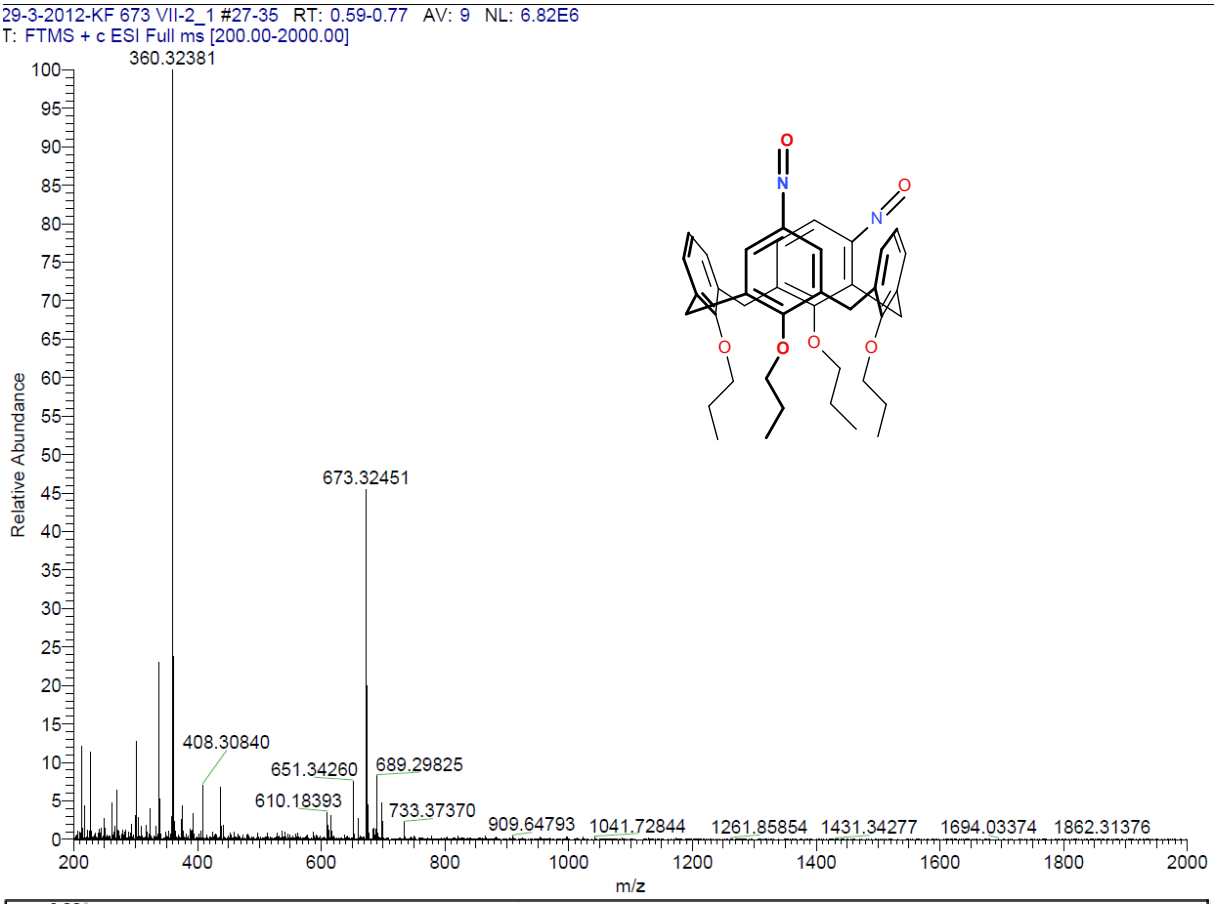
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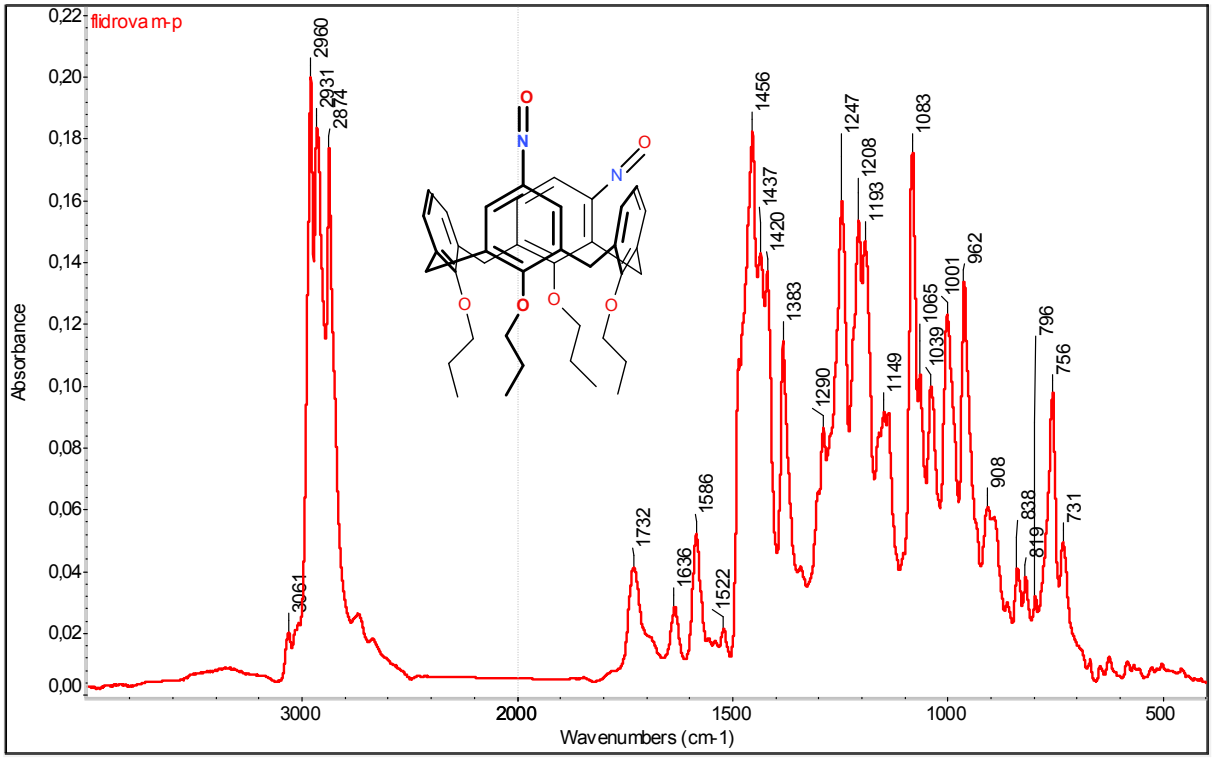
Relative Abundance
m/z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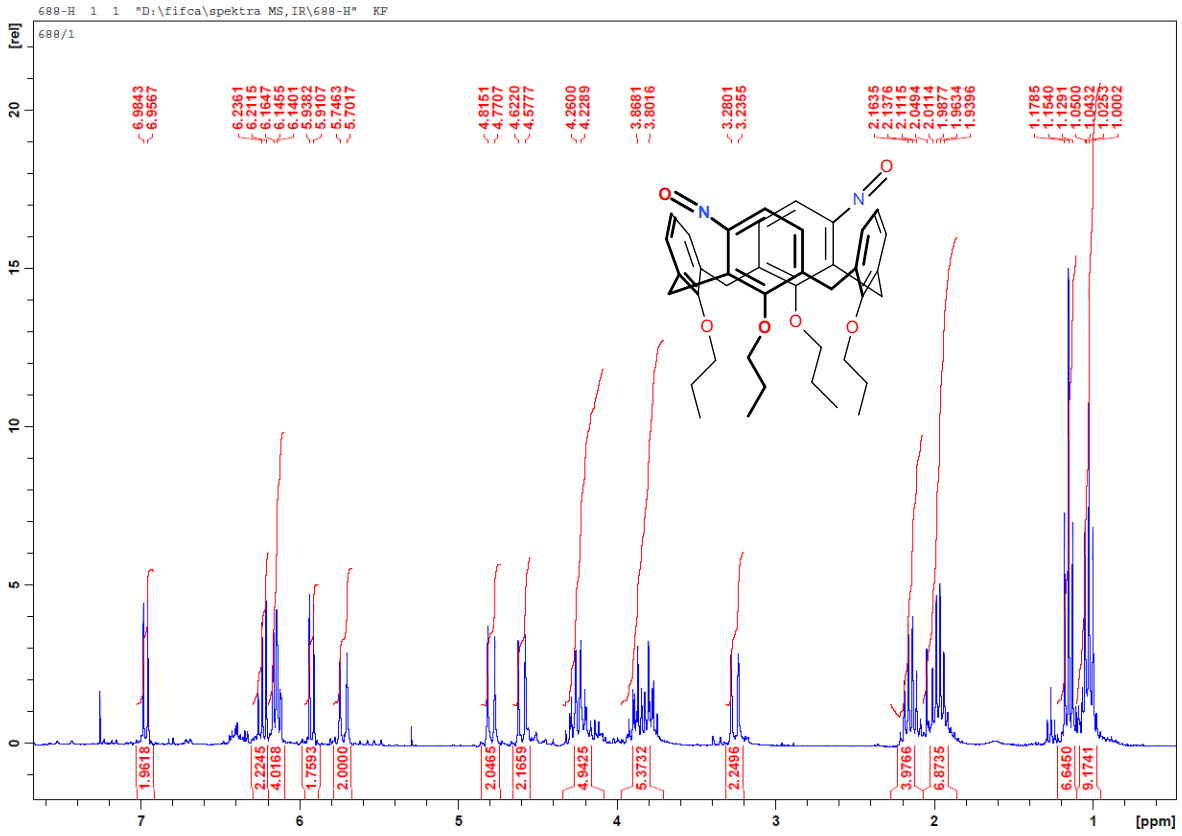
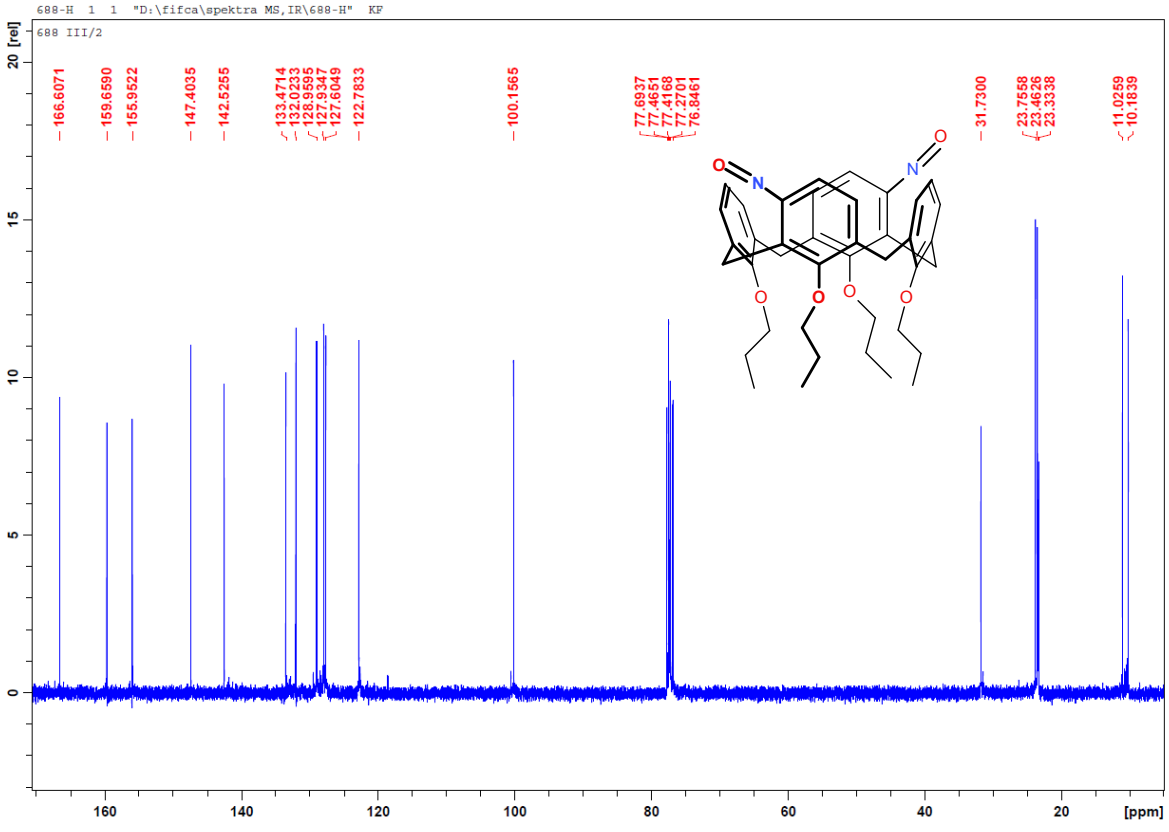
1208
1193
1149
1083
1065
1039
1001
962
908
838
819
796
756
731
Absorbance
Wavenumbers (cm-1)

CHARACTERIZATIONS OF COMPOUND 5





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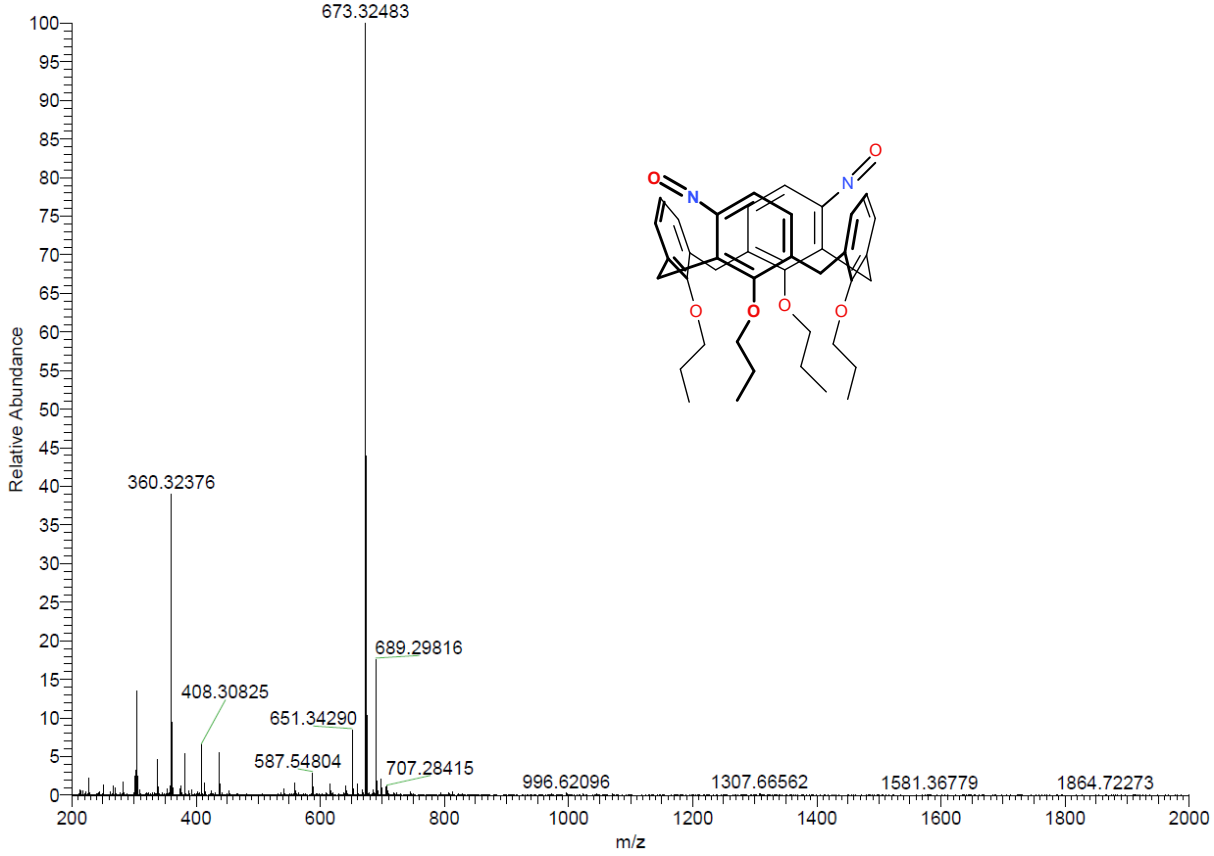

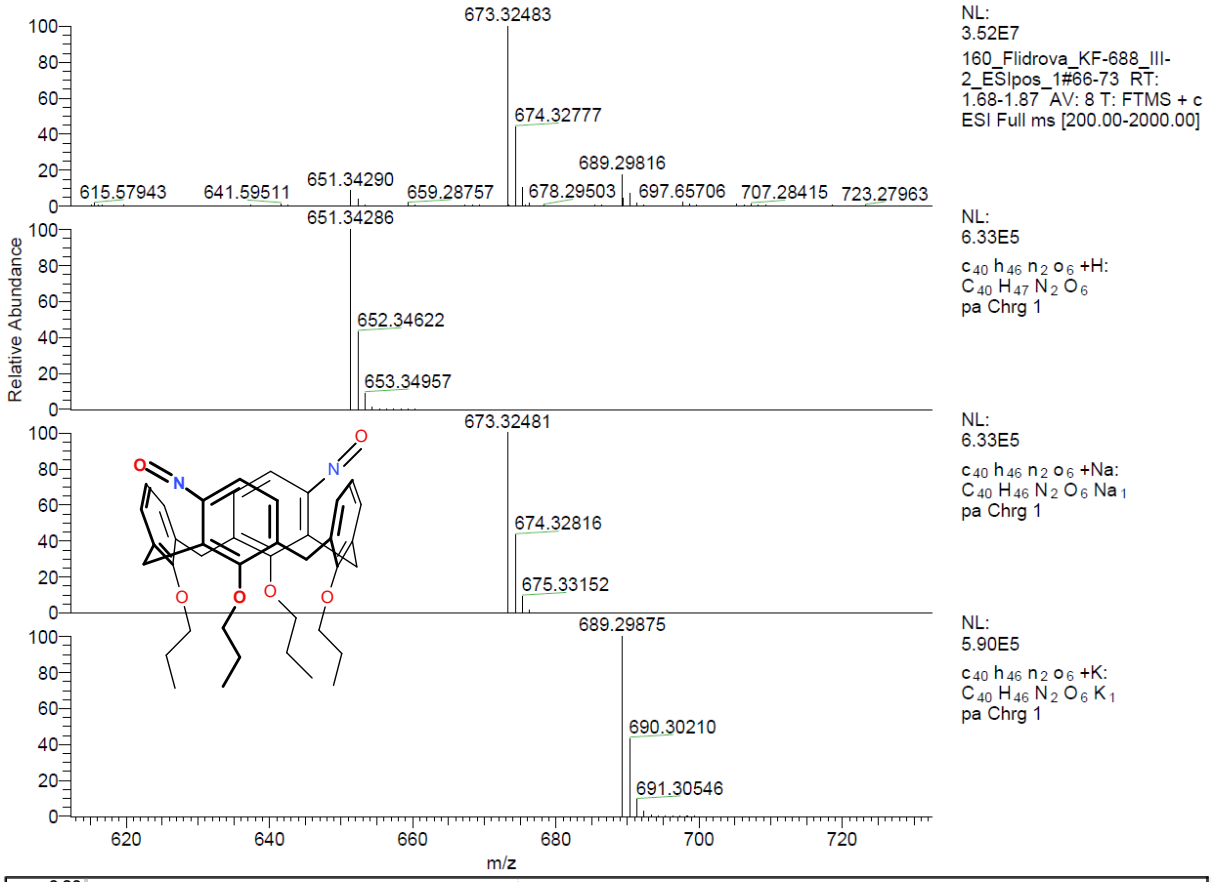
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m/z
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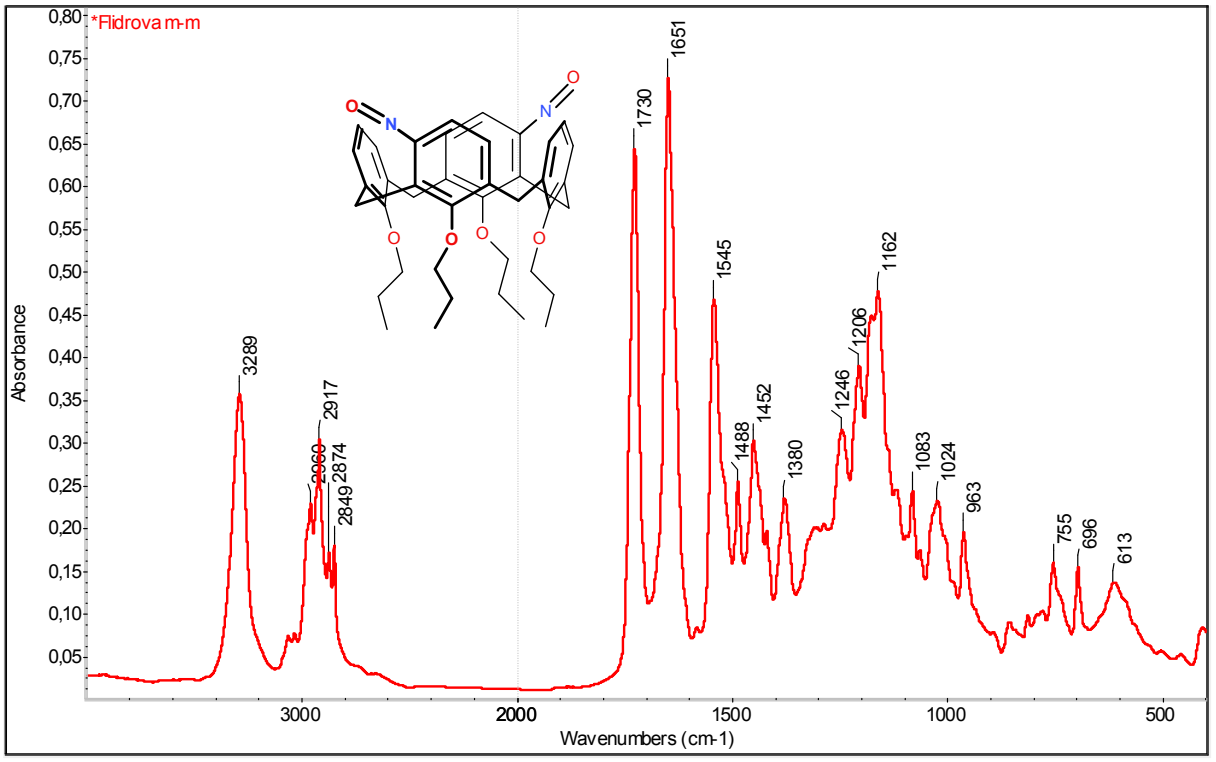
640
660
680
700
720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1452
1380
1246
1206
1162
1083
1024
963
755
696
613
3000
2000
1500
1000
500

NMR SECTION

USED CHEMICALS

Chloroform-d_1 ($CDCl_3$) – degree of deuteration min. 99.8 % and dimethylsulfoxide-d_6 (DMSO) – degree of deuteration min. 99.8 % were purchased from Merck, Germany. Phenylalanine derived polyacetylene alignment medium (medium A) was prepared by our group from commercially available compounds (Sigma-Aldrich). High molecular weight poly(γ-benzyl-D-glutamate (medium B) was prepared and kindly provided by research group of C. M. Thiele. Poly(γ-ethyl-L-glutamate) – M>100 000 g/mol (medium C) was purchased from Sigma-Aldrich.

MEASUREMENTS CONDITIONS

All NMR data were acquired on Bruker Avance^{III} 600 MHz (proton frequency) NMR spectrometer equipped with triple resonance cryo-probe. Scalar coupling constants ($^1J_{C\text{-}H}$) and total splitting ($^1T_{C\text{-}H}$) were measured using heteronuclear (^{1}H-^{13}C) CLIP-HSQC pulse sequence without proton decoupling in direct (F2) domain. Nine heteronuclear one bond RDCs were sufficient to obtain structural information, so it was unnecessary to measure any other type of RDCs.

1D | ^{1}H NMR SPECTRA

π/2 pulse for ^{1}H nuclei was approximately 9 μs. Spectral width: 7 kHz, size of fid: 32k data points, relaxation period: 10 s, number of scans: 8.

1D | ^{2}H NMR SPECTRA

π/2 pulse for ^{2}H nuclei was approximately 68 μs. Spectral width: 4 kHz, size of fid: 16k data points, relaxation period: 10 s, number of scans: 8.

1D | ^{13}C-APT NMR SPECTRA

^{13}C NMR spectra with proton decoupling - *waltz16* (decoupling pulse 100 μs, power level 23.6 dB). π/2 pulse for ^{13}C nuclei was approximately 12 μs. Spectral width: 31.5 kHz, size of fid: 64k data points, polarization transfer: 7 ms, relaxation period: 2.0 s, number of scans: 20 000.

1D | ^{13}C (DE)COUPLED NMR SPECTRA (Z-RESTORED)

Modified version of classical 1D ^{13}C NMR experiment with straight baseline free of distortions. Spectral width: 19.5 kHz, size of fid: 64k data points, relaxation period: 1 s, number of scans: 4k - 16k.

For pulse sequence details see ref.: Y. Xia, S. Moran, E. P. Nikonowiczb, X. Gaoa, *Magn. Res. Chem.*, **2007**, *46*, 432.

2D | ^{1}H-^{1}H COSY SPECTRA

Spectral width: 3.8 kHz in both domains, size of fid: 1024 (F2) a 256 (F1) data points, relaxation period: 1.0 s, number of scans: 12.

2D | ^{1}H-^{13}C HMQC SPECTRA

Spectral width: 4.0 kHz (F2) a 21.4 kHz (F1), size of fid: 2048 (F2) a 256 (F1) data points, polarization transfer: 3.5 ms, relaxation period: 1.0 s, number of scans: 16.

2D | CLIP ^{1}H-^{13}C HSQC SPECTRA

2D ^{1}H-^{13}C CLIP HSQC is a modification of classical F2-coupled HSQC experiment providing clear spectra without antiphase artefacts and other spectral distortions. Residual dipolar coupling constants were measured from splitting in the direct domain (F2). Spectral width: 4.7 kHz (F2) a 19.6 kHz (F1), size of fid: 8k (F2) a 256 (F1) data points, polarization transfer: 3,125 ms, relaxation period: 0.1 s, number of scans: 16.

For pulse sequence details see ref.: A. Enthart, J. C. Freudenberger, J. Furrer, H. Kessler, B. Luy, *J. Magn. Reson.*, **2008**, *192*, 314.

2D | ^{1}H-^{13}C HMBC SPECTRA

Spectral width: 4,0 kHz (F2) a 27,7 kHz (F1), size of fid: 2k (F2) a 256 (F1) data points, polarization transfer: 70 ms, relaxation period: 1,0 s, number of scans: 64.

1D | ^{1}H DPFGSE – NOE SPECTRA

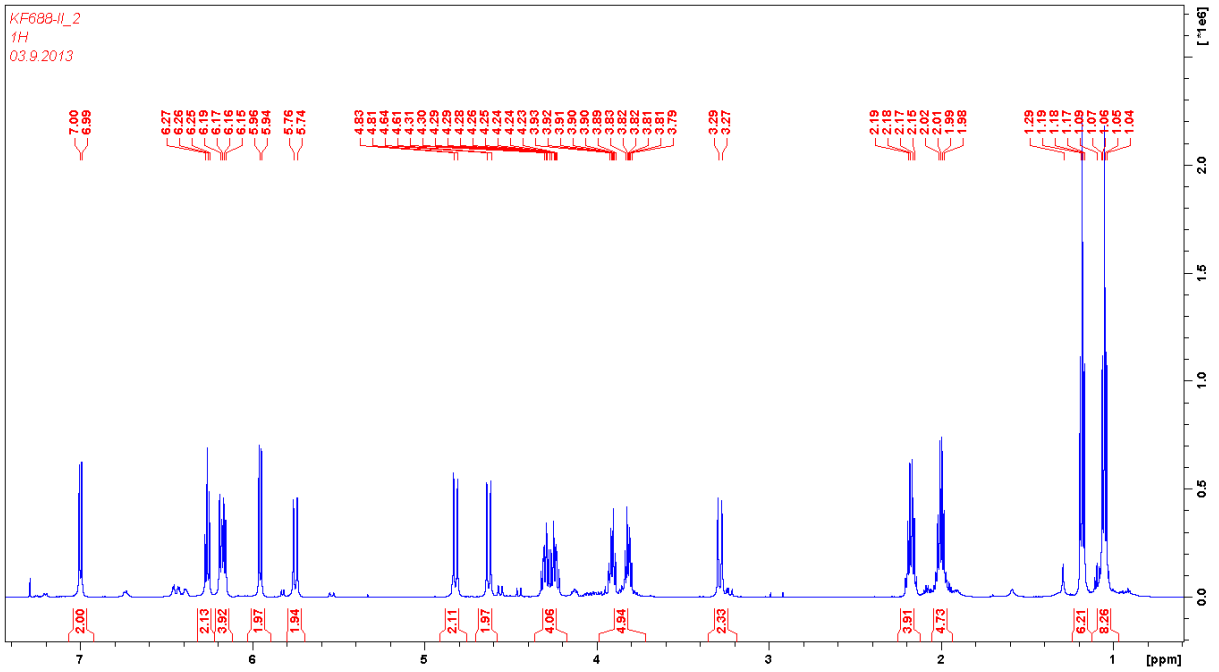
Nuclear Overhauser effect (NOE) based experiment with selective inversion – DPFGSE sequence. Selective inversion was performed via 80ms *q3-gaussian cascade.* Spectral width: 12.0 kHz, size of fid: 32k data points, mixing time: 0.2-1.6 s, relaxation period: 2.0 s, number of scans: 128.

DYNAMIC ^{1}H NMR MEASUREMENTS

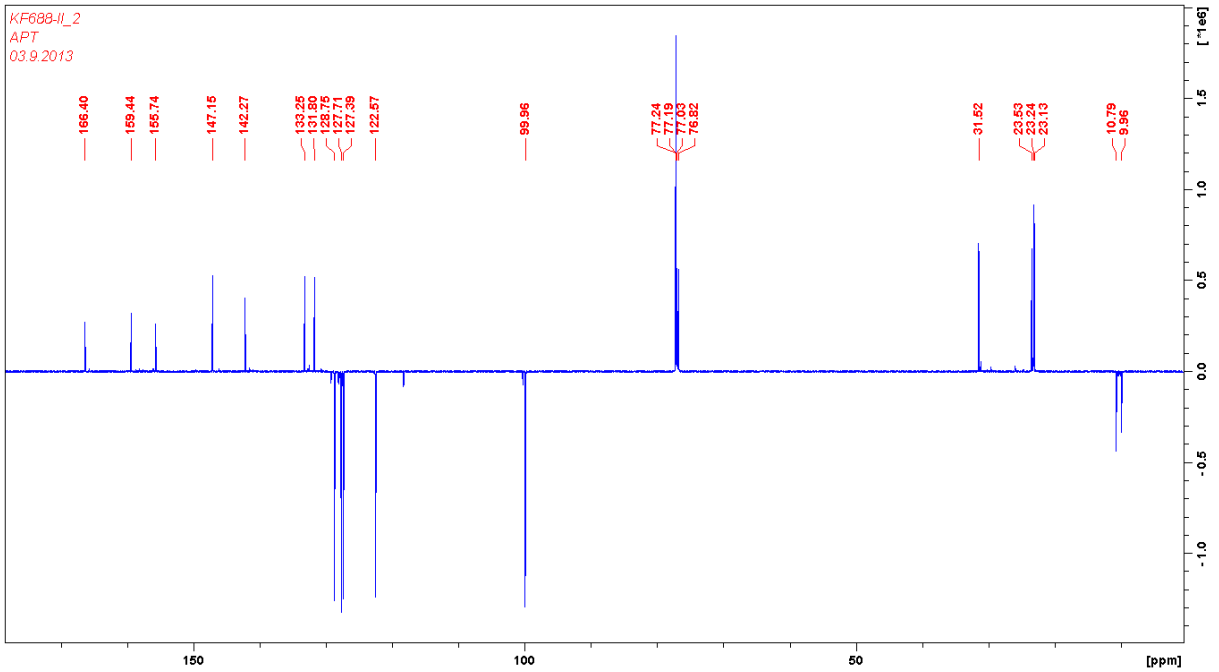
Dynamic NMR spectra were acquired on Bruker Avance^{III} 500 MHz (proton frequency) NMR spectrometer equipped with BBOF probe in temperature range 193-298K.

NMR CHARACTERIZATION

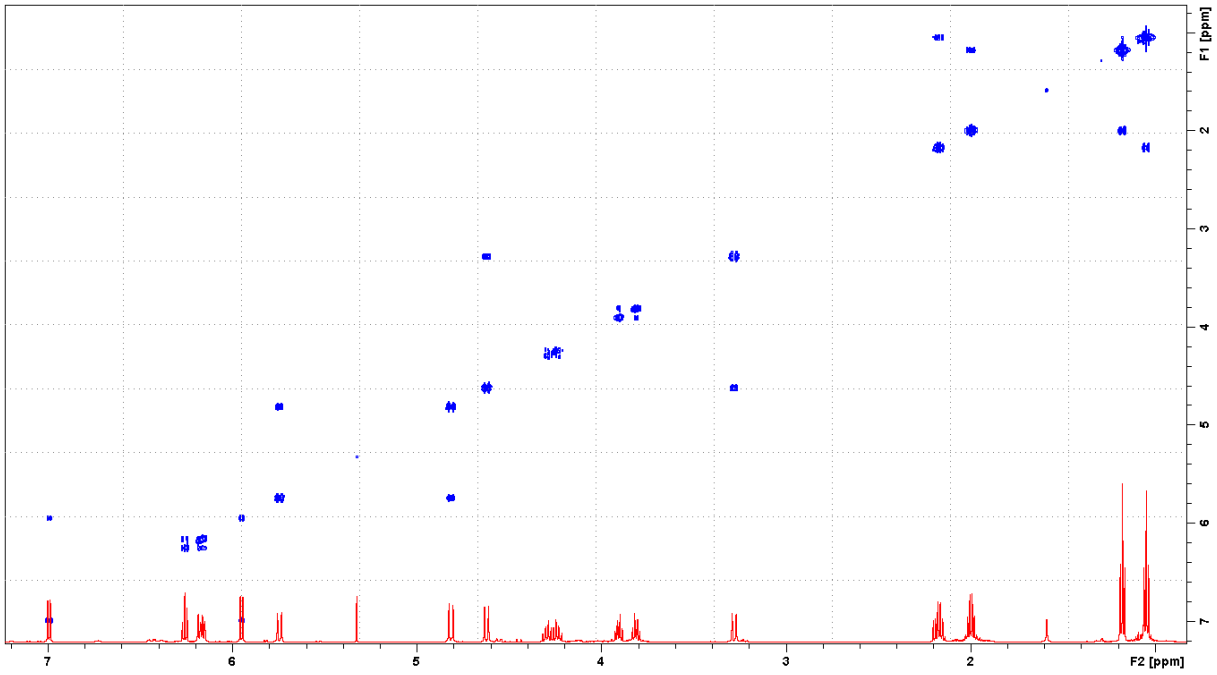
^{1}H NMR SPECTRUM OF 5 (600 MHZ, $CDCl_3$)



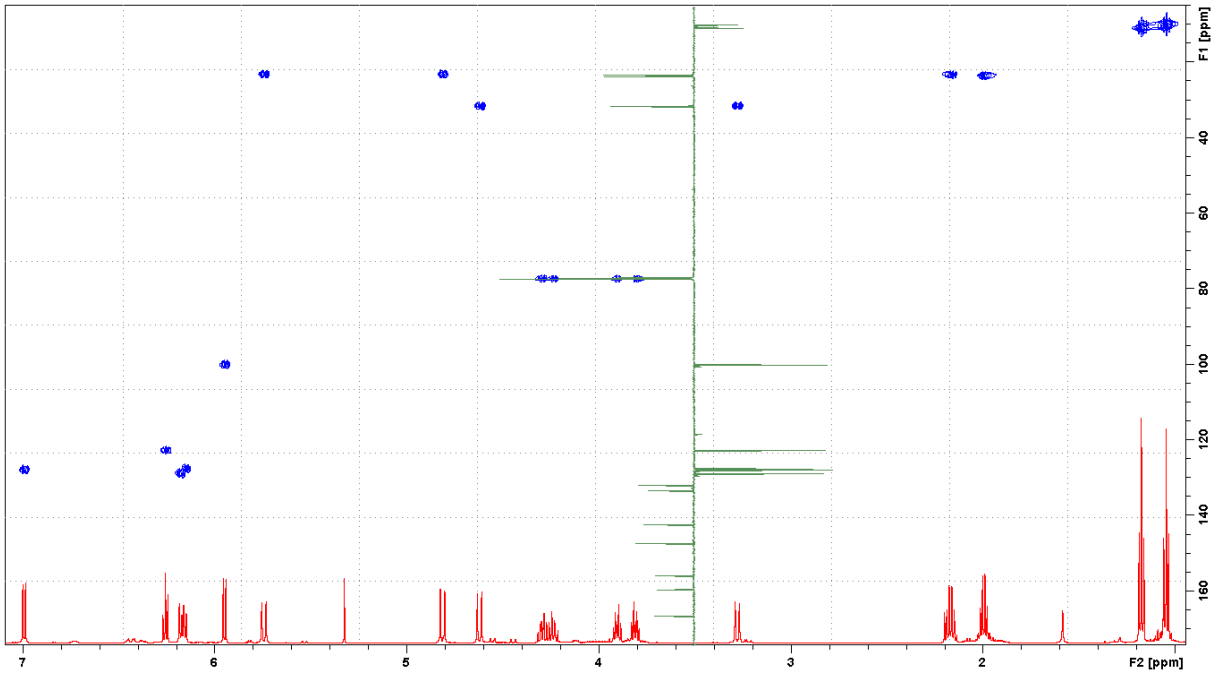
^{13}C NMR SPECTRUM OF 5 (600 MHZ, $CDCl_3$)



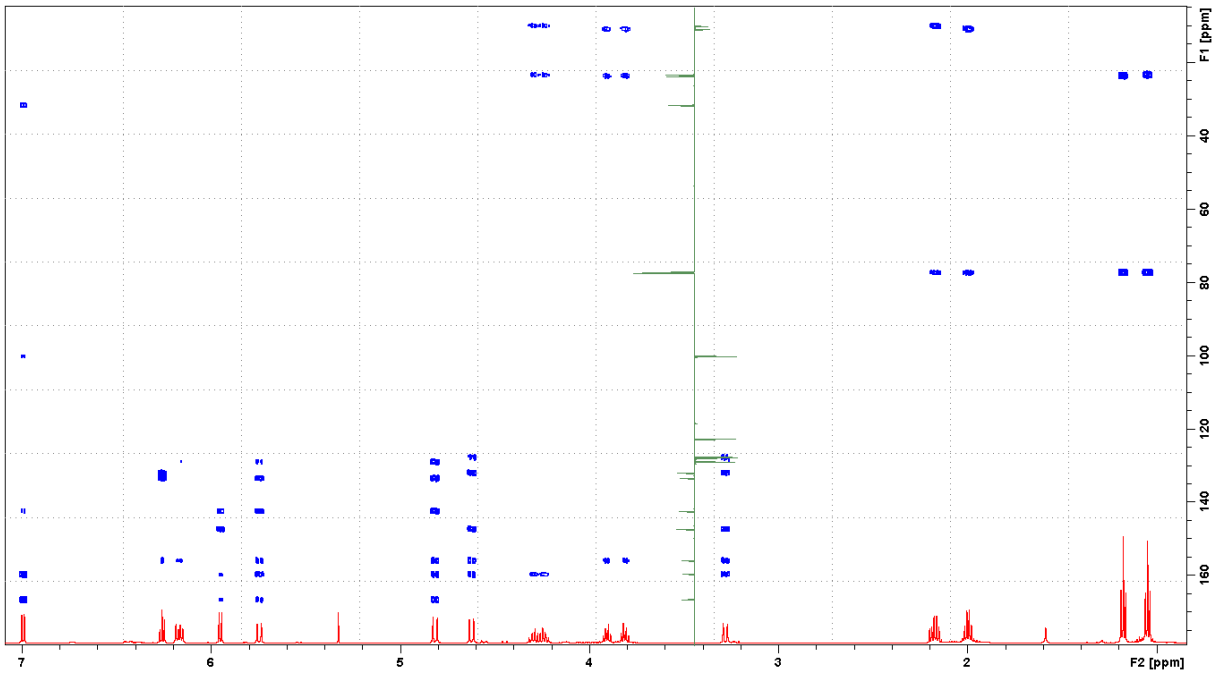
^{1}H-^{1}H COSY SPECTRUM OF 5 (600 MHZ, $CDCl_3$)



^{1}H-^{13}C HMQC SPECTRUM OF 5 (600 MHZ, $CDCl_3$)



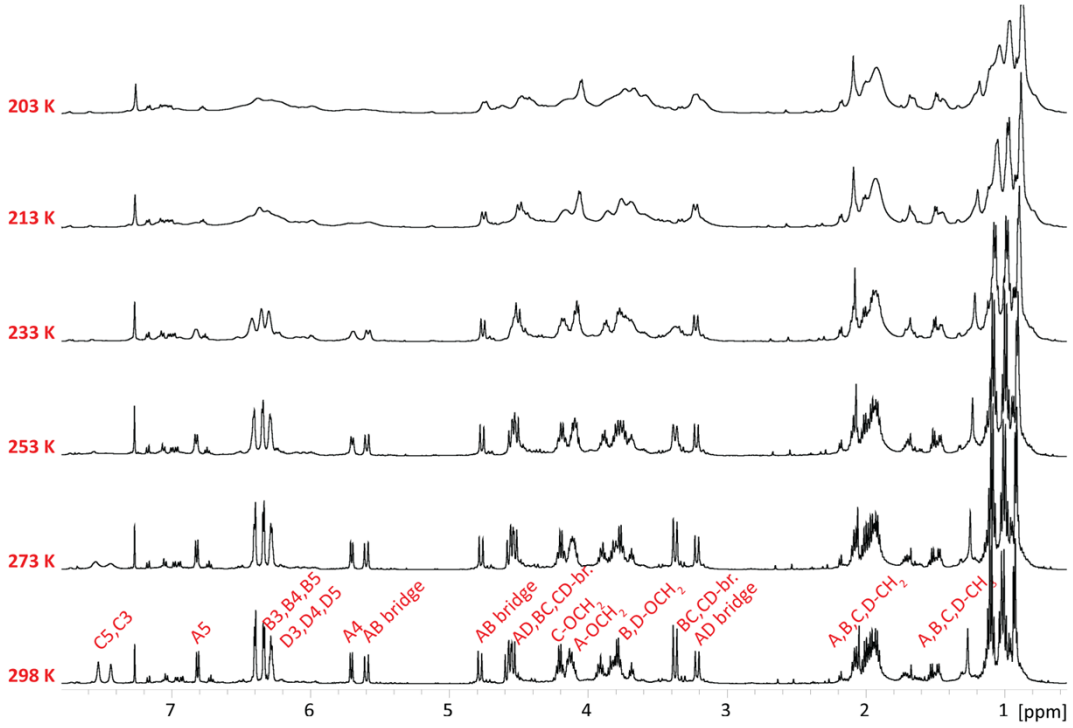
^{1}H-^{13}C HMBC SPECTRUM OF 5 (600 MHZ, $CDCl_3$)



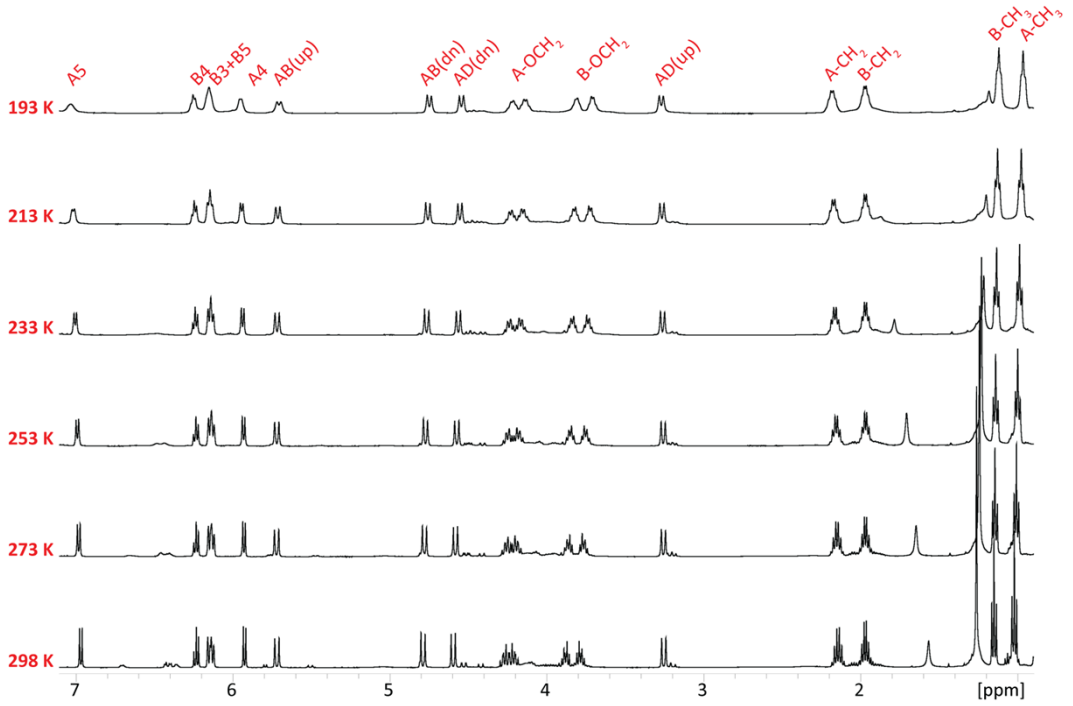
1D ^{1}H-DPFGSE NOE EXPERIMENTS OF 5 (600 MHZ, $CDCl_3$)

DYNAMIC NMR MEASUREMENTS

TEMPERATURE DEPENDENT ^{1}H NMR SPECTRA OF 4 (500 MHZ, $CDCl_3$)



TEMPERATURE DEPENDENT ^{1}H NMR SPECTRA OF 5 (500 MHZ, $CDCl_3$)



RDC SECTION

GPC ANALYSIS OF POLYACETYLENE ALIGNMENT MEDIUM

Waters chromatograph, 2xPL Mixed C columns, 5 μm, 0.8 ml/min $CHCl_3$, 35°C, PS calibration.

Analysis results were out of calibration ➔ $M_w \sim 10^6$ g/mol (approximately).

ANISOTROPIC SAMPLE PREPARATION

Alignment medium was prepared due to high viscosity of liquid crystalline solution in the NMR tube. Weighted amount of alignment medium (polyacetylene or polyglutamate) and calixarene derivative was placed into the NMR tube and added deuterated chloroform. To obtain well resolved spectra it is necessary to homogenise the sample. After a few hours NMR tube was centrifuged upside down to mix up the viscous content. Then the capillary with DMSO-d_6 as the external standard was added. Homogeneity of the anisotropic sample was monitored via ^{2}H NMR spectra measurement (quadrupolar splitting of $CDCl_3$). Mixing procedure was repeated till the lines in ^{2}H NMR spectrum was narrow and no signal of residual non-aligned solvent was observed.

Tab. 1: Preparation of anisotropic solutions.

Alignment medium	**A**	**B**	**C**
m_{medium} [mg]	190.5	80.4	71.1
$m_{calixarene}$ [mg]	26.5	20.7	25.2
m_{CDCl3} [mg]	881.0	900.0	870.0
w_{medium} [%]	17.3	8.0	7.4
ν_Q [Hz]	~60	~265	~239

RDC ANALYSIS

We utilized 9 independent values of one bond heteronuclear (^{1}H-^{13}C) coupling constants. RDC constant were elucidated from the line splitting in ^{13}C NMR spectrum without proton decoupling as the contribution to the scalar coupling. In case of signal overlaps CLIP HSQC spectra were used. Total line splitting in anisotropic solution ($^1T_{C\text{-}H}$) is expressed by the following equation: $^1T_{C\text{-}H} = 2^1D_{C\text{-}H} + {}^1J_{C\text{-}H}$ (Figure 1), where $^1D_{C\text{-}H}$ is residual dipolar coupling constant and $^1J_{C\text{-}H}$ represents scalar coupling constant elucidated from ^{13}C NMR spectrum without proton decoupling of isotropic solution (compound in an organic solvent).

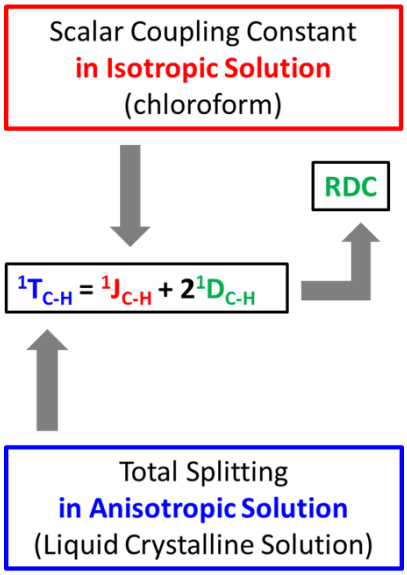


Figure 1: RDC computation scheme.

The measurement of RDCs has two steps (Figure 2) – NMR spectra acquisition of isotropic solution (compound in an organic solvent) and of anisotropic solution (liquid crystalline solution). Residual dipolar coupling constants were calculated by subtraction of scalar coupling constant in isotropic solution from the total splitting in liquid crystalline solution.

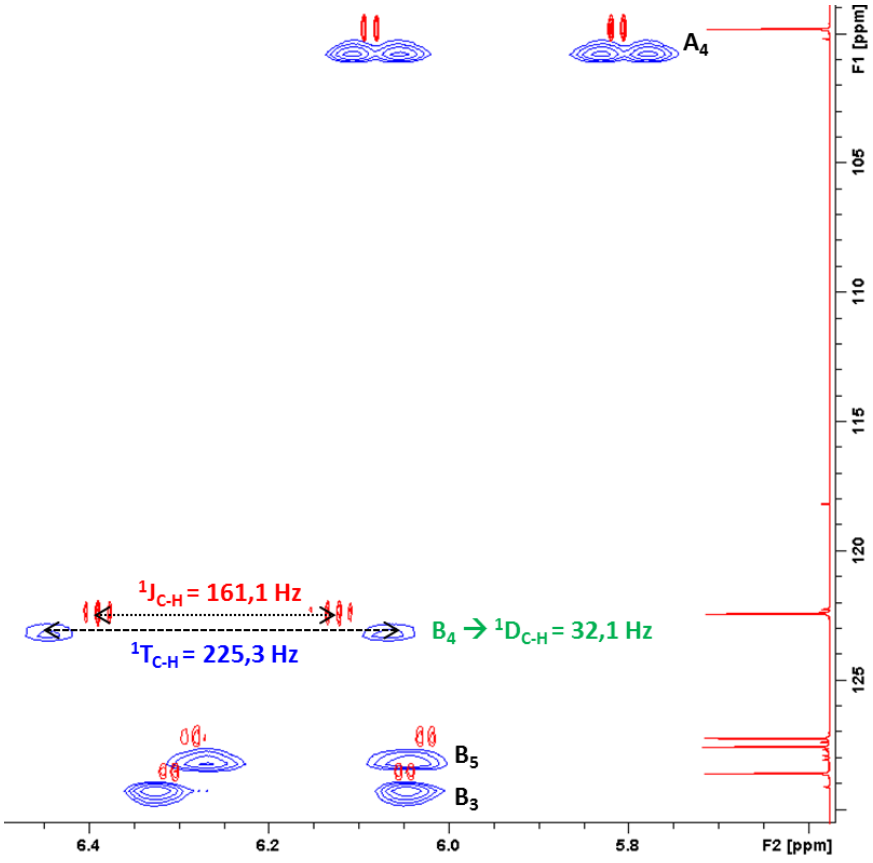


Figure 2: ^{1}H-^{13}C CLIP HSQC spectrum of isotropic solution (red) and anisotropic sample (blue).

STATISTICAL FORMULAS

$$x_{\mathrm{rms}} = \sqrt{\frac{1}{n}\left({x_1}^2 + {x_2}^2 + \cdots + {x_n}^2\right)}$$

$$r_{xy} = \frac{\sum_{i=1}^{n}(x_i - \bar{x})(y_i - \bar{y})}{(n-1)s_x s_y} = \frac{\sum_{i=1}^{n}(x_i - \bar{x})(y_i - \bar{y})}{\sqrt{\sum_{i=1}^{n}(x_i - \bar{x})^2 \sum_{i=1}^{n}(y_i - \bar{y})^2}},$$

$$r_{xy} = \frac{\sum x_i y_i - n\bar{x}\bar{y}}{(n-1)s_x s_y} = \frac{n\sum x_i y_i - \sum x_i \sum y_i}{\sqrt{n\sum x_i^2 - (\sum x_i)^2}\ \sqrt{n\sum y_i^2 - (\sum y_i)^2}}.$$

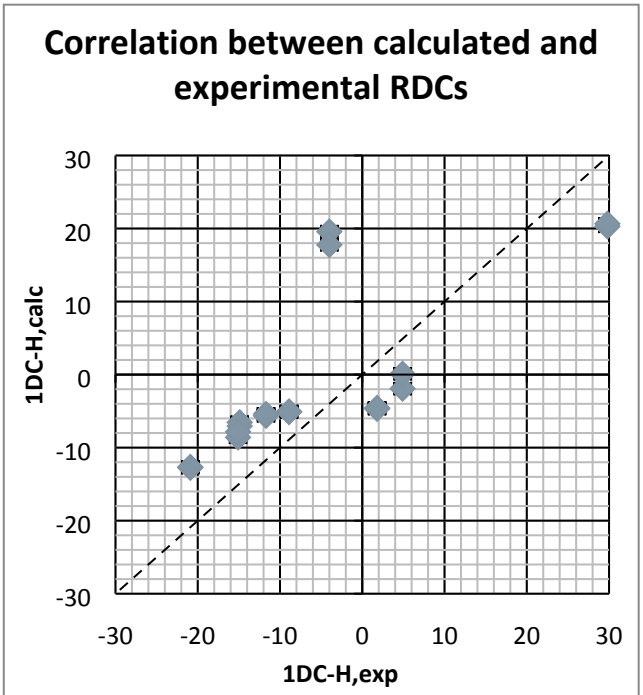
5*(I)* @ POLYACETYLENE WITH PHENYLALANINE SIDE CHAIN (MEDIUM A)

EVALUATION OF RDCs

	$^1J_{C\text{-}H}$ [Hz]	$^1T_{C\text{-}H}$ [Hz]	RDC [Hz]	$^1D_{C\text{-}H}$ [Hz]
CH-A4	164.6	168.1	3.5	1.8
CH-A5	160.9	131.2	-29.7	-14.9
CH-B3	157.0	149.0	-8.0	-4.0
CH-B4	161.1	130.9	-30.2	-15.1
CH-B5	157.6	167.4	9.8	4.9
CH_2-AB (up)	131.8	191.4	59.6	29.8
CH_2-AB (dn)	132.0	114.2	-17.8	-8.9
CH_2-AD (up)	128.6	86.9	-41.7	-20.9
CH_2-AD (dn)	132.1	108.8	-23.3	-11.7

FITTING RESULTS

$^1D_{C\text{-}H}$	$^1D_{C\text{-}H,exp}$	$^1D_{C\text{-}H,calc}$
A4	1.8	-4.7
C4	1.8	-4.6
A5	-14.9	-7.1
C3	-14.9	-6.5
B3	-4.0	19.6
D5	-4.0	17.8
B4	-15.1	-8.6
D4	-15.1	-7.8
B5	4.9	-1.9
D3	4.9	0.1
CH_2-AB (up)	29.8	20.3
CH_2-AB (up)	29.8	20.7
CH_2-AB (dn)	-8.9	-5.1
CH_2-AB (dn)	-8.9	-5.1
CH_2-AD (up)	-20.8	-12.7
CH_2-AD (up)	-20.9	-12.7
CH_2-AD (dn)	-11.7	-5.6
CH_2-AD (dn)	-11.7	-5.4



n =	18
RMS =	10.061
R(xy) =	0.763
R^2 =	58.2%

5*(II)* @ POLYACETYLENE WITH PHENYLALANINE SIDE CHAIN (MEDIUM A)

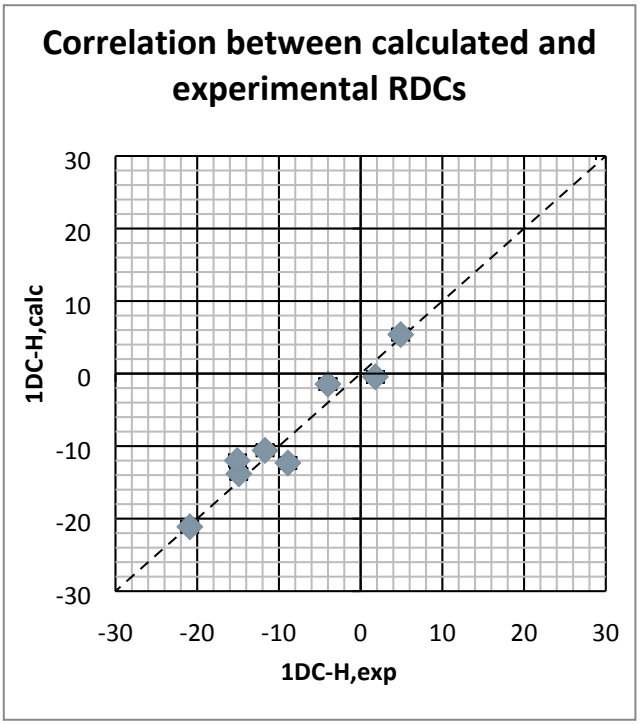
EVALUATION OF RDCs

Subtraction of scalar coupling constants in isotropic solution (chloroform) from total splitting in liquid crystalline solution (phenylalanine polyacetylene).

	$^1J_{C\text{-}H}$ [Hz]	$^1T_{C\text{-}H}$ [Hz]	RDC [Hz]	$^1D_{C\text{-}H}$ [Hz]
CH-A4	164.6	168.1	3.5	1.8
CH-A5	160.9	131.2	-29.7	-14.9
CH-B3	157.0	149.0	-8.0	-4.0
CH-B4	161.1	130.9	-30.2	-15.1
CH-B5	157.6	167.4	9.8	4.9
CH_2-AB (up)	131.8	191.4	59.6	29.8
CH_2-AB (dn)	132.0	114.2	-17.8	-8.9
CH_2-AD (up)	128.6	86.9	-41.7	-20.9
CH_2-AD (dn)	132.1	108.8	-23.3	-11.7

FITTING RESULTS

$^1D_{C-H}$	$^1D_{C-H.exp}$	$^1D_{C-H.calc}$
A4	1.8	-0.5
C4	1.8	-0.4
A5	-14.9	-13.8
C3	-14.9	-13.8
B3	-4.0	-1.5
D5	-4.0	-1.5
B4	-15.1	-12.0
D4	-15.1	-12.0
B5	4.9	5.4
D3	4.9	5.4
CH_2-AB (up)	29.8	30.4
CH_2-AB (up)	29.8	30.4
CH_2-AB (dn)	-8.9	-12.3
CH_2-AB (dn)	-8.9	-12.3
CH_2-AD (up)	-20.9	-21.1
CH_2-AD (up)	-20.9	-21.1
CH_2-AD (dn)	-11.7	-10.6
CH_2-AD (dn)	-11.7	-10.6



n =	18
RMS =	1.998
R(xy) =	0.991
R^2 =	98.1%

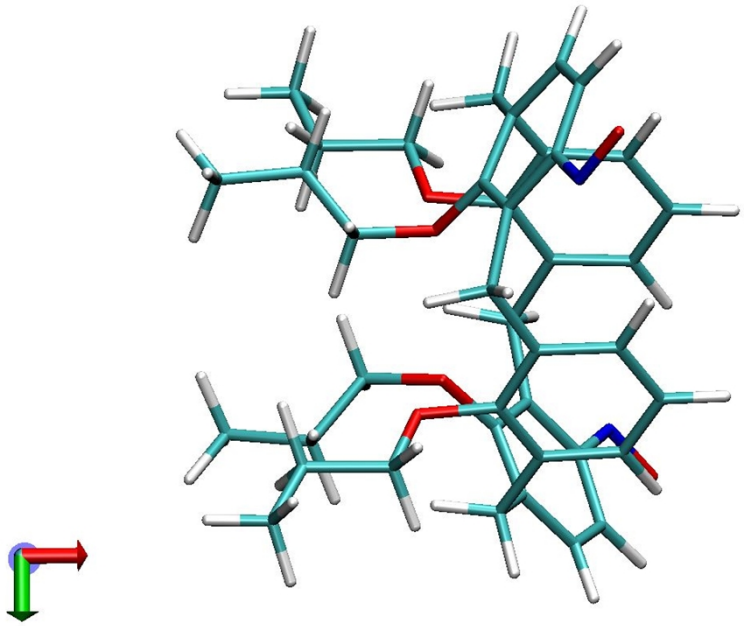
PALES output file (fitting procedure results)

```
REMARK Molecular Alignment Simulation.

REMARK Simulation parameters.

DATA PALES_MODE DC

DATA TENSOR_MODE SVD (Order Matrix Method)


REMARK Order matrix.

DATA SAUPE_MATRIX     S(zz)       S(xx-yy)      S(xy)      S(xz)      S(yz)
DATA SAUPE        -2.5607e-004  1.0917e-003 -3.2036e-004 -2.9514e-008  6.8403e-008

DATA IRREDUCIBLE_REP    A0          A1R          A1I         A2R         A2I
DATA IRREDUCIBLE    -4.0596e-004  3.8203e-008  8.8542e-008  7.0653e-004  4.1468e-
004
DATA IRREDUCIBLE GENERAL_MAGNITUDE   1.2276e-003


REMARK Mapping of coordinates.

DATA MAPPING_COOR   Szz_d(x)  Szz_d(y)  Syy_d(x)  Syy_d(y)  Sxx_d(x)  Sxx_d(y)
DATA MAPPING       -0.26538  -0.00005   1.30542  -0.00023   0.00027   1.57056
DATA MAPPING INV    2.87622   0.00005  -1.83617   0.00023  -0.00048  -1.57056


REMARK Eigensystem & Euler angles for clockwise rotation about z. y'. z''.

DATA EIGENVALUES (Sxx_d.Syy_d.Szz_d)   -2.5607e-004 -5.0486e-004  7.6093e-004
DATA EIGENVECTORS           (x_coor      y_coor      z_coor)
DATA EIGENVECTORS X_AXIS  1.0547e-004  2.1404e-004  1.0000e+000
DATA EIGENVECTORS Y_AXIS  2.6227e-001  9.6499e-001 -2.3421e-004
DATA EIGENVECTORS Z_AXIS  9.6499e-001 -2.6227e-001 -4.5644e-005

DATA Q_EULER_SOLUTIONS    ALPHA     BETA    GAMMA
DATA Q_EULER_ANGLES  1   180.01    90.00   195.20
DATA Q_EULER_ANGLES  2     0.01    90.00   195.20
DATA Q_EULER_ANGLES  3   359.99    90.00    15.20
DATA Q_EULER_ANGLES  4   179.99    90.00    15.20


REMARK Euler angles (psi/theta/phi) for rotation about x. y. z.

DATA EULER_SOLUTIONS 6
DATA EULER_ANGLES   -1.#J  -74.80   -1.#J
DATA EULER_ANGLES   -1.#J  -74.80   -1.#J
DATA EULER_ANGLES   -1.#J  -74.80   -1.#J
DATA EULER_ANGLES   -1.#J  -74.80   -1.#J
DATA EULER_ANGLES   90.00  254.80  -89.97
DATA EULER_ANGLES   90.00  254.80  -89.97

DATA Da  3.804661e-004
DATA Dr  8.292899e-005


DATA Aa  7.609322e-004
DATA Ar  1.658580e-004


DATA Da_HN       8.212436e+000
DATA Rhombicity  2.179669e-001
```

```
REMARK Dipolar couplings.

DATA N                      18
DATA RMS                    1.998
DATA Chi2                   71.859
DATA CORR R                 0.991
DATA Q SAUPE                0.086
DATA REGRESSION OFFSET      0.280 +/- 0.512 [Hz]
DATA REGRESSION SLOPE       0.988 +/- 0.034 [Hz]
DATA REGRESSION BAX SLOPE 0.997 +/- 0.024 [Hz]

VARS    RESID_I RESNAME_I ATOMNAME_I RESID_J RESNAME_J ATOMNAME_J DI D_OBS D D_DIFF
DD W
FORMAT  %4d %4s %4s %4d %4s %4s %9.2f %9.3f %9.3f %9.3f %.2f %.2f

    1  CAL  C40    1  CAL  H41  47292.49    1.8000   -0.4503    2.2503  1.0000 1.00
    1  CAL  C13    1  CAL  H22  47292.49    1.8000   -0.4495    2.2495  1.0000 1.00
    1  CAL  C54    1  CAL  H51  47052.89  -14.9000  -13.8186   -1.0814  1.0000 1.00
    1  CAL  C19    1  CAL   H7  47052.89  -14.9000  -13.8145   -1.0855  1.0000 1.00
    1  CAL  C31    1  CAL  H26  46972.77   -4.0000   -1.4570   -2.5430  1.0000 1.00
    1  CAL  C45    1  CAL  H30  46972.77   -4.0000   -1.4559   -2.5441  1.0000 1.00
    1  CAL  C39    1  CAL  H55  47181.87  -15.1000  -11.9874   -3.1126  1.0000 1.00
    1  CAL  C46    1  CAL  H43  47187.93  -15.1000  -11.9885   -3.1115  1.0000 1.00
    1  CAL  C16    1  CAL   H6  46923.96    4.9000    5.3806   -0.4806  1.0000 1.00
    1  CAL  C35    1  CAL  H61  46923.96    4.9000    5.3804   -0.4804  1.0000 1.00
    1  CAL   C4    1  CAL  H25  46417.77   29.8000   30.3701   -0.5701  1.0000 1.00
    1  CAL   C2    1  CAL  H56  46417.77   29.8000   30.3673   -0.5673  1.0000 1.00
    1  CAL   C4    1  CAL   H3  46609.84   -8.9000  -12.3230    3.4230  1.0000 1.00
    1  CAL   C2    1  CAL   H1  46609.84   -8.9000  -12.3212    3.4212  1.0000 1.00
    1  CAL  C37    1  CAL  H24  45715.64  -20.9000  -21.1225    0.2225  1.0000 1.00
    1  CAL  C10    1  CAL  H32  45715.64  -20.9000  -21.1198    0.2198  1.0000 1.00
    1  CAL  C37    1  CAL   H8  46602.07  -11.7000  -10.5931   -1.1069  1.0000 1.00
    1  CAL  C10    1  CAL  H14  46602.07  -11.7000  -10.5935   -1.1065  1.0000 1.00
```

ROTATED STRUCTURE

B_0 (external magnetic field induction vector) passes through the plane of the paper.



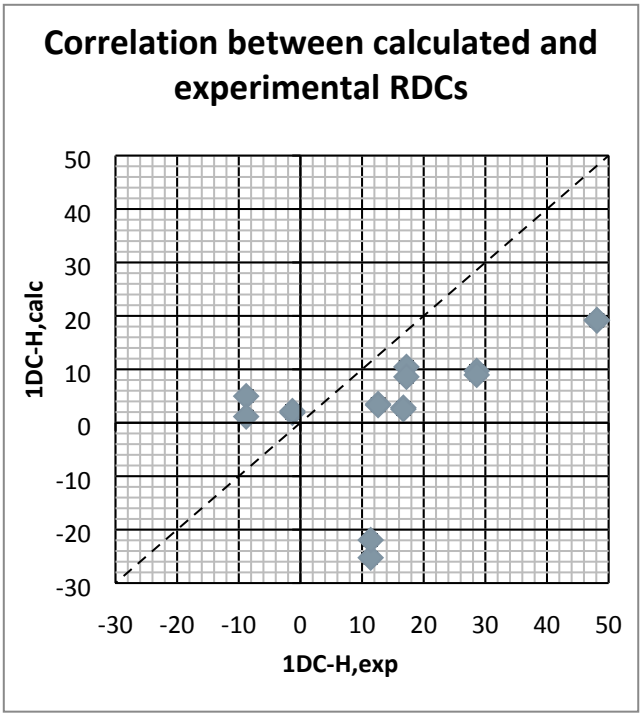
5(*l*) @ POLY(γ-BENZYL-D-GLUTAMATE) WITH HIGH MOLECULAR WEIGHT (MEDIUM B)

EVALUATION OF RDCs

	$^1J_{C\text{-}H}$ [Hz]	$^1T_{C\text{-}H}$ [Hz]	RDC [Hz]	$^1D_{C\text{-}H}$ [Hz]
CH-A4	164.6	161.9	-2.7	-1.3
CH-A5	160.9	218.0	57.1	28.6
CH-B3	157.0	179.7	22.7	11.4
CH-B4	161.1	195.5	34.4	17.2
CH-B5	157.6	139.9	-17.7	-8.8
CH_2-AB (up)	131.8	n/a	n/a	n/a
CH_2-AB (dn)	132.0	165.3	33.3	16.7
CH_2-AD (up)	128.6	224.8	96.2	48.1
CH_2-AD (dn)	132.1	157.2	25.1	12.6

FITTING RESULTS

$^1D_{C\text{-}H}$	$^1D_{C\text{-}H.exp}$	$^1D_{C\text{-}H.calc}$
A4	-1.3	1.9
C4	-1.3	2.2
A5	28.6	9.7
C3	28.6	9.0
B3	11.4	-25.3
D5	11.4	-21.9
B4	17.2	10.5
D4	17.2	8.6
B5	-8.8	5.0
D3	-8.8	1.2
CH_2-AB (up)	n/a	n/a
CH_2-AB (up)	n/a	n/a
CH_2-AB (dn)	16.7	2.8
CH_2-AB (dn)	16.7	2.5
CH_2-AD (up)	48.1	19.1
CH_2-AD (up)	48.1	19.2
CH_2-AD (dn)	12.6	3.5
CH_2-AD (dn)	12.6	3.3



n =	16
RMS =	19.144
R(xy) =	0.494
R^2 =	24.4%

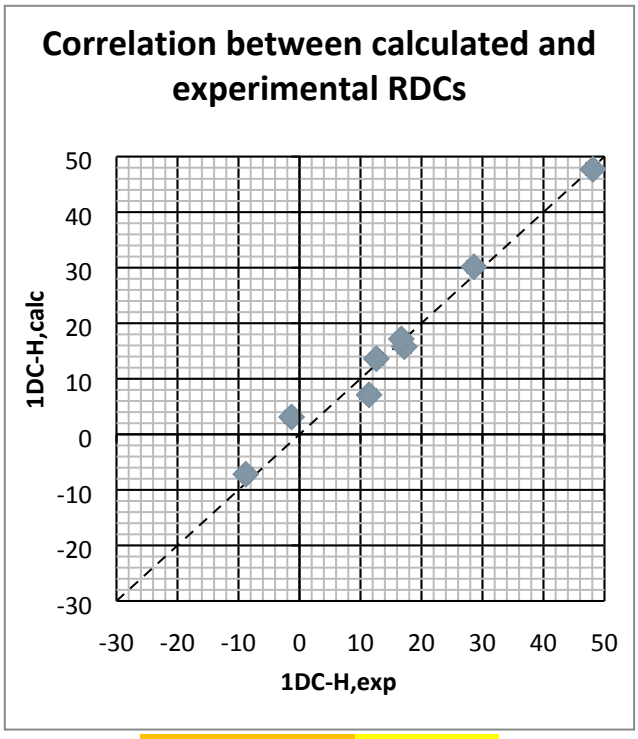
5(*II*) @ POLY(γ-BENZYL-D-GLUTAMATE) WITH HIGH MOLECULAR WEIGHT (MEDIUM B)

EVALUATION OF RDCs

	$^1J_{C\text{-}H}$ [Hz]	$^1T_{C\text{-}H}$ [Hz]	RDC [Hz]	$^1D_{C\text{-}H}$ [Hz]
CH-A4	164.6	161.9	-2.7	-1.3
CH-A5	160.9	218.0	57.1	28.6
CH-B3	157.0	179.7	22.7	11.4
CH-B4	161.1	195.5	34.4	17.2
CH-B5	157.6	139.9	-17.7	-8.8
CH_2-AB (up)	131.8	n/a	n/a	n/a
CH_2-AB (dn)	132.0	165.3	33.3	16.7
CH_2-AD (up)	128.6	224.8	96.2	48.1
CH_2-AD (dn)	132.1	157.2	25.1	12.6

FITTING RESULTS

$^1D_{C\text{-}H}$	$^1D_{C\text{-}H.exp}$	$^1D_{C\text{-}H.calc}$
A4	-1.3	3.1
C4	-1.3	3.1
A5	28.6	30.2
C3	28.6	30.2
B3	11.4	7.1
D5	11.4	7.1
B4	17.2	15.8
D4	17.2	15.8
B5	-8.8	-7.2
D3	-8.8	-7.2
CH_2-AB (up)	n/a	n/a
CH_2-AB (up)	n/a	n/a
CH_2-AB (dn)	16.7	17.2
CH_2-AB (dn)	16.7	17.2
CH_2-AD (up)	48.1	47.7
CH_2-AD (up)	48.1	47.7
CH_2-AD (dn)	12.6	13.6
CH_2-AD (dn)	12.6	13.6



n =	16
RMS =	2.410
R(xy) =	0.990
R^2 =	97.9%

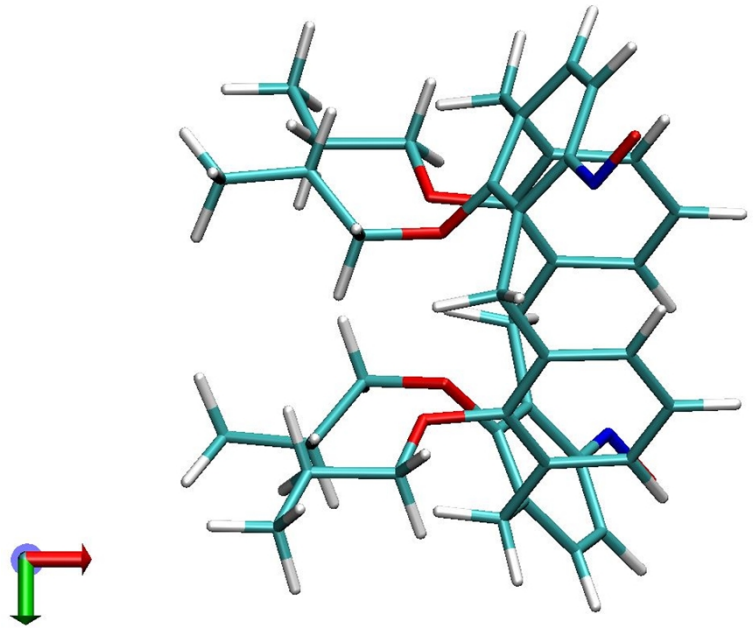
PALES output file (fitting procedure results)

```
REMARK Molecular Alignment Simulation.

REMARK Simulation parameters.

DATA PALES_MODE DC

DATA TENSOR_MODE SVD (Order Matrix Method)


REMARK Order matrix.

DATA SAUPE_MATRIX     S(zz)       S(xx-yy)      S(xy)      S(xz)      S(yz)
DATA SAUPE         3.3770e-004 -2.2367e-003  7.5176e-004  2.3915e-008 -4.3174e-008

DATA IRREDUCIBLE_REP    A0          A1R          A1I         A2R         A2I
DATA IRREDUCIBLE     5.3536e-004 -3.0956e-008 -5.5885e-008 -1.4476e-003 -9.7310e-
004
DATA IRREDUCIBLE GENERAL_MAGNITUDE   2.5242e-003


REMARK Mapping of coordinates.

DATA MAPPING_COOR   Szz_d(x)  Szz_d(y)  Syy_d(x)  Syy_d(y)  Sxx_d(x)  Sxx_d(y)
DATA MAPPING       -0.29591  -0.00002   1.27488  -0.00004   0.00004   1.57075
DATA MAPPING INV    2.84568   0.00002  -1.86671   0.00004  -0.00010  -1.57075


REMARK Eigensystem & Euler angles for clockwise rotation about z. y'. z''.

DATA EIGENVALUES (Sxx_d.Syy_d.Szz_d)    3.3770e-004  1.1787e-003 -1.5164e-003
DATA EIGENVECTORS           (x_coor      y_coor      z_coor)
DATA EIGENVECTORS X_AXIS  3.0199e-005  3.3461e-005  1.0000e+000
DATA EIGENVECTORS Y_AXIS  2.9161e-001  9.5654e-001 -4.0813e-005
DATA EIGENVECTORS Z_AXIS  9.5654e-001 -2.9161e-001 -1.9129e-005

DATA Q_EULER_SOLUTIONS    ALPHA     BETA    GAMMA
DATA Q_EULER_ANGLES  1   180.00    90.00   196.95
DATA Q_EULER_ANGLES  2     0.00    90.00   196.95
DATA Q_EULER_ANGLES  3   360.00    90.00    16.95
DATA Q_EULER_ANGLES  4   180.00    90.00    16.95


REMARK Euler angles (psi/theta/phi) for rotation about x. y. z.

DATA EULER_SOLUTIONS 8
DATA EULER_ANGLES   -1.#J  -73.05   -1.#J
DATA EULER_ANGLES   -1.#J  -73.05   -1.#J
DATA EULER_ANGLES   -1.#J  -73.05   -1.#J
DATA EULER_ANGLES   -1.#J  -73.05   -1.#J
DATA EULER_ANGLES   -1.#J  253.05   -1.#J
DATA EULER_ANGLES   -1.#J  253.05   -1.#J
DATA EULER_ANGLES   -1.#J  253.05   -1.#J
DATA EULER_ANGLES   -1.#J  253.05   -1.#J

DATA Da -7.581946e-004
DATA Dr -2.803313e-004


DATA Aa -1.516389e-003
DATA Ar -5.606627e-004


DATA Da_HN      -1.636578e+001
DATA Rhombicity  3.697353e-001
```

```
REMARK Dipolar couplings.

DATA N                     16
DATA RMS                   2.410
DATA Chi2                  92.925
DATA CORR R                0.990
DATA Q SAUPE               0.051
DATA REGRESSION OFFSET     1.049 +/- 0.839 [Hz]
DATA REGRESSION SLOPE      0.957 +/- 0.037 [Hz]
DATA REGRESSION BAX SLOPE 0.967 +/- 0.026 [Hz]

VARS    RESID_I RESNAME_I ATOMNAME_I RESID_J RESNAME_J ATOMNAME_J DI D_OBS D D_DIFF
DD W
FORMAT  %4d %4s %4s %4d %4s %4s %9.2f %9.3f %9.3f %9.3f %.2f %.2f

    1  CAL  C40    1  CAL  H41  47292.49   -1.3000    3.1173   -4.4173  1.0000 1.00
    1  CAL  C13    1  CAL  H22  47292.49   -1.3000    3.1173   -4.4173  1.0000 1.00
    1  CAL  C54    1  CAL  H51  47052.89   28.6000   30.1798   -1.5798  1.0000 1.00
    1  CAL  C19    1  CAL   H7  47052.89   28.6000   30.1771   -1.5771  1.0000 1.00
    1  CAL  C31    1  CAL  H26  46972.77   11.4000    7.1091    4.2909  1.0000 1.00
    1  CAL  C45    1  CAL  H30  46972.77   11.4000    7.1088    4.2912  1.0000 1.00
    1  CAL  C39    1  CAL  H55  47181.87   17.2000   15.7704    1.4296  1.0000 1.00
    1  CAL  C46    1  CAL  H43  47187.93   17.2000   15.7718    1.4282  1.0000 1.00
    1  CAL  C16    1  CAL   H6  46923.96   -8.8000   -7.1848   -1.6152  1.0000 1.00
    1  CAL  C35    1  CAL  H61  46923.96   -8.8000   -7.1851   -1.6149  1.0000 1.00
    1  CAL   C4    1  CAL   H3  46609.84   16.7000   17.1711   -0.4711  1.0000 1.00
    1  CAL   C2    1  CAL   H1  46609.84   16.7000   17.1701   -0.4701  1.0000 1.00
    1  CAL  C37    1  CAL  H24  45715.64   48.1000   47.7110    0.3890  1.0000 1.00
    1  CAL  C10    1  CAL  H32  45715.64   48.1000   47.7093    0.3907  1.0000 1.00
    1  CAL  C37    1  CAL   H8  46602.07   12.6000   13.6107   -1.0107  1.0000 1.00
    1  CAL  C10    1  CAL  H14  46602.07   12.6000   13.6112   -1.0112  1.0000 1.00
```

ROTATED STRUCTURE

B_0 (external magnetic field induction vector) passes through the plane of the paper.



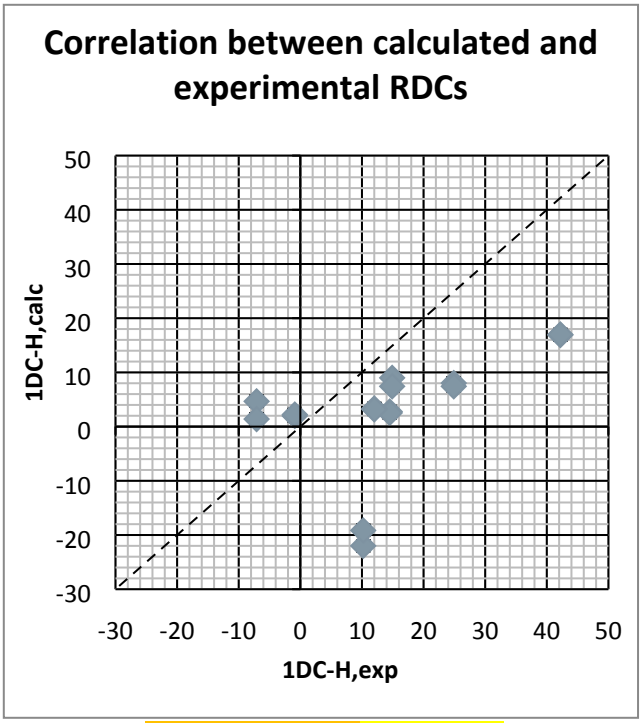
5(*I*) @ POLY(γ-BENZYL-L-GLUTAMATE), COMMERCIALLY AVAILABLE (MEDIUM C)

EVALUATION OF RDCs

	$^1J_{C\text{-}H}$ [Hz]	$^1T_{C\text{-}H}$ [Hz]	RDC [Hz]	$^1D_{C\text{-}H}$ [Hz]
CH-A4	164.6	162,8	-1,8	-0,9
CH-A5	160.9	210,7	49,8	24,9
CH-B3	157.0	177,4	20,4	10,2
CH-B4	161.1	190,8	29,7	14,9
CH-B5	157.6	143,3	-14,3	-7,1
CH_2-AB (up)	131.8	n/a	n/a	n/a
CH_2-AB (dn)	132.0	161,0	29,0	14,5
CH_2-AD (up)	128.6	213,0	84,4	42,2
CH_2-AD (dn)	132.1	156,0	23,9	12,0

FITTING RESULTS

$^1D_{C\text{-}H}$	$^1D_{C\text{-}H.exp}$	$^1D_{C\text{-}H.calc}$
A4	-0,9	2,0
C4	-0,9	2,2
A5	24,9	8,0
C3	24,9	7,4
B3	10,2	-22,0
D5	10,2	-19,2
B4	14,9	9,0
D4	14,9	7,4
B5	-7,1	4,7
D3	-7,1	1,4
CH_2-AB (up)	n/a	n/a
CH_2-AB (up)	n/a	n/a
CH_2-AB (dn)	14,5	2,8
CH_2-AB (dn)	14,5	2,5
CH_2-AD (up)	42,2	16,9
CH_2-AD (up)	42,2	17,0
CH_2-AD (dn)	12,0	3,3
CH_2-AD (dn)	12,0	3,2



n =	16
RMS =	16,801
R(xy) =	0,485
R^2 =	23,6%

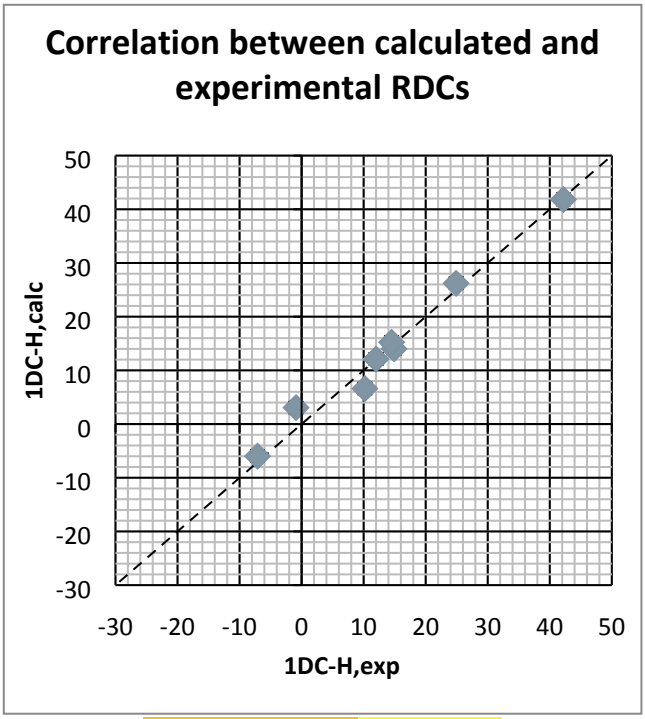
5(*II*) @ POLY(γ-BENZYL-L-GLUTAMATE), COMMERCIALLY AVAILABLE (MEDIUM C)

EVALUATION OF RDCs

	$^1J_{C\text{-}H}$ [Hz]	$^1T_{C\text{-}H}$ [Hz]	RDC [Hz]	$^1D_{C\text{-}H}$ [Hz]
CH-A4	164.6	162,8	-1,8	-0,9
CH-A5	160.9	210,7	49,8	24,9
CH-B3	157.0	177,4	20,4	10,2
CH-B4	161.1	190,8	29,7	14,9
CH-B5	157.6	143,3	-14,3	-7,1
CH_2-AB (up)	131.8	n/a	n/a	n/a
CH_2-AB (dn)	132.0	161,0	29,0	14,5
CH_2-AD (up)	128.6	213,0	84,4	42,2
CH_2-AD (dn)	132.1	156,0	23,9	12,0

FITTING RESULTS

$^1D_{C\text{-}H}$	$^1D_{C\text{-}H.exp}$	$^1D_{C\text{-}H.calc}$
A4	-0,9	3,1
C4	-0,9	3,1
A5	24,9	26,2
C3	24,9	26,2
B3	10,2	6,6
D5	10,2	6,6
B4	14,9	14,0
D4	14,9	14,0
B5	-7,1	-6,0
D3	-7,1	-6,0
CH_2-AB (up)	14,5	15,2
CH_2-AB (up)	14,5	15,2
CH_2-AB (dn)	n/a	n/a
CH_2-AB (dn)	n/a	n/a
CH_2-AD (up)	42,2	41,8
CH_2-AD (up)	42,2	41,8
CH_2-AD (dn)	12,0	12,1
CH_2-AD (dn)	12,0	12,1



n =	16
RMS =	2,034
R(xy) =	0,990
R^2 =	98,0%

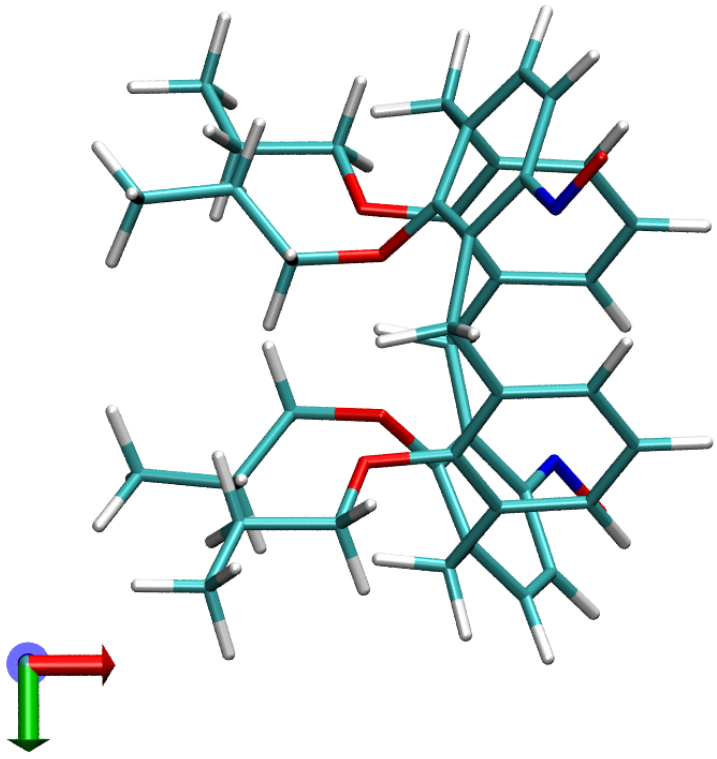
PALES output file (fitting procedure results)

```
REMARK Molecular Alignment Simulation.

REMARK Simulation parameters.

DATA PALES_MODE DC

DATA TENSOR_MODE SVD (Order Matrix Method)


REMARK Order matrix.

DATA SAUPE_MATRIX     S(zz)       S(xx-yy)      S(xy)      S(xz)      S(yz)
DATA SAUPE         2.9984e-004 -1.9588e-003  6.7079e-004  1.5864e-008 -2.8279e-008

DATA IRREDUCIBLE_REP    A0          A1R          A1I         A2R         A2I
DATA IRREDUCIBLE     4.7535e-004 -2.0534e-008 -3.6604e-008 -1.2677e-003 -8.6828e-
004
DATA IRREDUCIBLE GENERAL_MAGNITUDE   2.2244e-003


REMARK Mapping of coordinates.

DATA MAPPING_COOR   Szz_d(x)  Szz_d(y)  Syy_d(x)  Syy_d(y)  Sxx_d(x)  Sxx_d(y)
DATA MAPPING       -0.30026  -0.00001   1.27054  -0.00003   0.00003   1.57076
DATA MAPPING INV    2.84133   0.00001  -1.87106   0.00003  -0.00008  -1.57076


REMARK Eigensystem & Euler angles for clockwise rotation about z, y', z''.

DATA EIGENVALUES (Sxx_d,Syy_d,Szz_d)    2.9984e-004  1.0372e-003 -1.3370e-003
DATA EIGENVECTORS          (x_coor      y_coor      z_coor)
DATA EIGENVECTORS X_AXIS  2.2679e-005  2.4670e-005  1.0000e+000
DATA EIGENVECTORS Y_AXIS  2.9577e-001  9.5526e-001 -3.0274e-005
DATA EIGENVECTORS Z_AXIS  9.5526e-001 -2.9577e-001 -1.4368e-005

DATA Q_EULER_SOLUTIONS    ALPHA     BETA    GAMMA
DATA Q_EULER_ANGLES  1   180.00    90.00   197.20
DATA Q_EULER_ANGLES  2     0.00    90.00   197.20
DATA Q_EULER_ANGLES  3   360.00    90.00    17.20
DATA Q_EULER_ANGLES  4   180.00    90.00    17.20


REMARK Euler angles (psi/theta/phi) for rotation about x, y, z.

DATA EULER_SOLUTIONS 8
DATA EULER_ANGLES   -1.#J  -72.80   -1.#J
DATA EULER_ANGLES   -1.#J  -72.80   -1.#J
DATA EULER_ANGLES   -1.#J  -72.80   -1.#J
DATA EULER_ANGLES   -1.#J  -72.80   -1.#J
DATA EULER_ANGLES   -1.#J  252.80   -1.#J
DATA EULER_ANGLES   -1.#J  252.80   -1.#J
DATA EULER_ANGLES   -1.#J  252.80   -1.#J
DATA EULER_ANGLES   -1.#J  252.80   -1.#J

DATA Da -6.685039e-004
DATA Dr -2.457743e-004


DATA Aa -1.337008e-003
DATA Ar -4.915485e-004


DATA Da_HN      -1.442979e+001
DATA Rhombicity  3.676482e-001
```

```
REMARK Dipolar couplings.

DATA N                     16
DATA RMS                   2.034
DATA Chi2                  66.200
DATA CORR R                0.990
DATA Q SAUPE               0.048
DATA REGRESSION OFFSET     0.864 +/- 0.720 [Hz]
DATA REGRESSION SLOPE      0.959 +/- 0.036 [Hz]
DATA REGRESSION BAX SLOPE 0.969 +/- 0.025 [Hz]

VARS    RESID_I RESNAME_I ATOMNAME_I RESID_J RESNAME_J ATOMNAME_J DI D_OBS D D_DIFF
DD W
FORMAT  %4d %4s %4s %4d %4s %4s %9.2f %9.3f %9.3f %9.3f %.2f %.2f

    1  CAL  C40    1  CAL  H41  47292.49   -0.9000    3.0634   -3.9634  1.0000 1.00
    1  CAL  C13    1  CAL  H22  47292.49   -0.9000    3.0634   -3.9634  1.0000 1.00
    1  CAL  C54    1  CAL  H51  47052.89   24.9000   26.2056   -1.3056  1.0000 1.00
    1  CAL  C19    1  CAL   H7  47052.89   24.9000   26.2039   -1.3039  1.0000 1.00
    1  CAL  C31    1  CAL  H26  46972.77   10.2000    6.6042    3.5958  1.0000 1.00
    1  CAL  C45    1  CAL  H30  46972.77   10.2000    6.6040    3.5960  1.0000 1.00
    1  CAL  C39    1  CAL  H55  47181.87   14.9000   14.0014    0.8986  1.0000 1.00
    1  CAL  C46    1  CAL  H43  47187.93   14.9000   14.0029    0.8971  1.0000 1.00
    1  CAL  C16    1  CAL   H6  46923.96   -7.1000   -5.9785   -1.1215  1.0000 1.00
    1  CAL  C35    1  CAL  H61  46923.96   -7.1000   -5.9787   -1.1213  1.0000 1.00
    1  CAL   C4    1  CAL   H3  46609.84   14.5000   15.2347   -0.7347  1.0000 1.00
    1  CAL   C2    1  CAL   H1  46609.84   14.5000   15.2340   -0.7340  1.0000 1.00
    1  CAL  C37    1  CAL  H24  45715.64   42.2000   41.8218    0.3782  1.0000 1.00
    1  CAL  C10    1  CAL  H32  45715.64   42.2000   41.8207    0.3793  1.0000 1.00
    1  CAL  C37    1  CAL   H8  46602.07   12.0000   12.1106   -0.1106  1.0000 1.00
    1  CAL  C10    1  CAL  H14  46602.07   12.0000   12.1109   -0.1109  1.0000
```

1.00Rotated structure

B_0 (external magnetic field induction vector) passes through the plane of the paper.



Tab. 2: Comparison of RDC values of structure 5 in various alignment media.

	$^1J_{CH}$ ($CDCl_3$)	$^1D_{CH}$ (A)	$^1D_{CH}$ (B)	$^1D_{CH}$ (C)
CH-A4	164,6	1,8	-1,3	-0,9
CH-A5	160,9	-14,9	28,6	24,9
CH-B3	157,0	-4,0	11,4	10,2
CH-B4	161,1	-15,1	17,2	14,9
CH-B5	157,6	4,9	-8,8	-7,1
CH_2-AB (up)	131,8	29,8	n/a	n/a
CH_2-AB (dn)	132,0	-8,9	16,7	14,5
CH_2-AD (up)	128,6	-20,9	48,1	42,2
CH_2-AD (dn)	132,1	-11,7	12,6	12,0

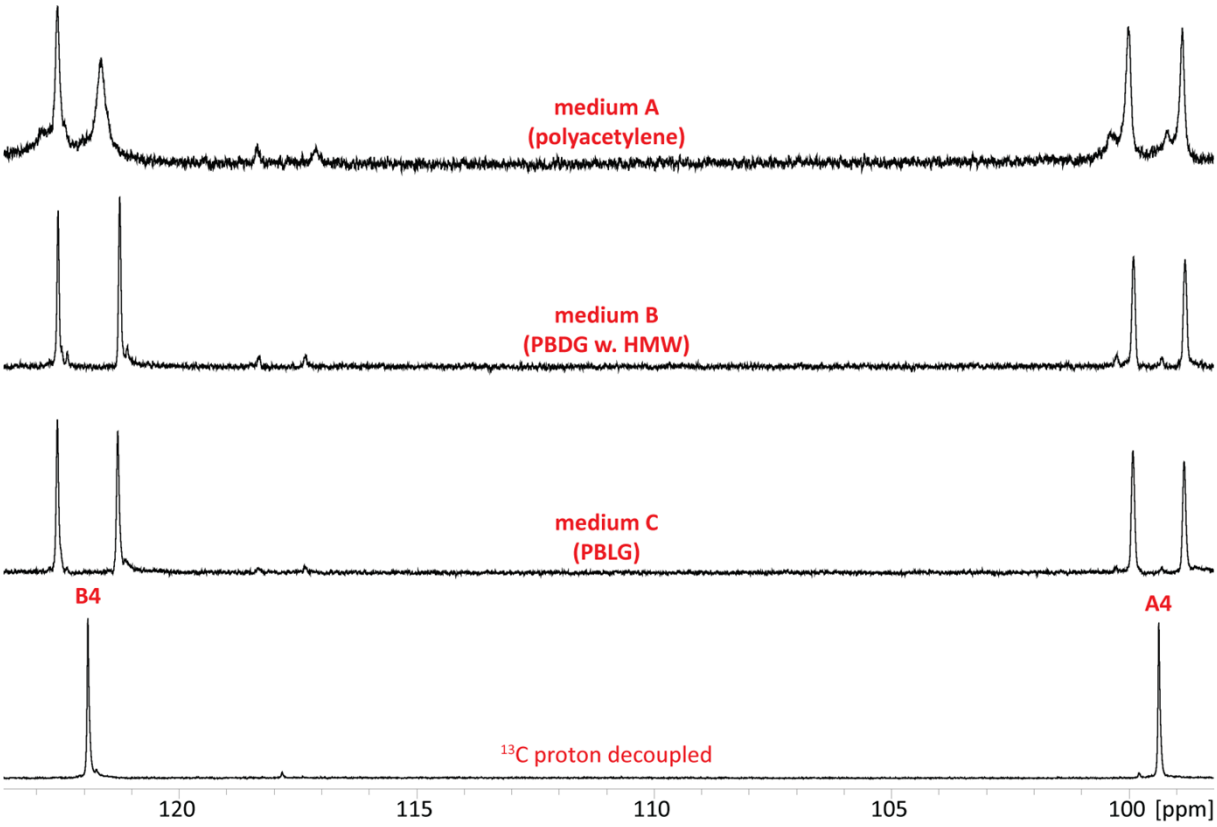
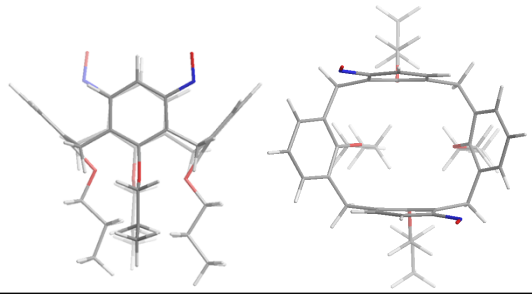
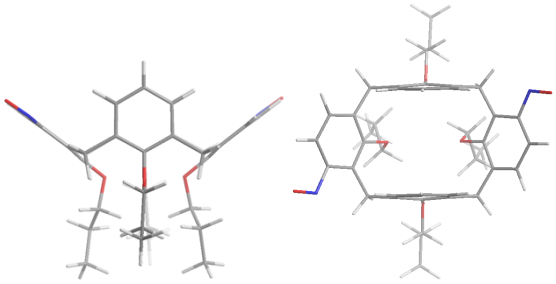
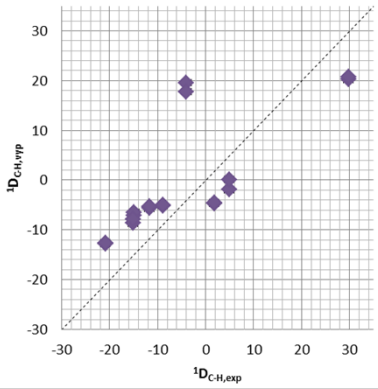
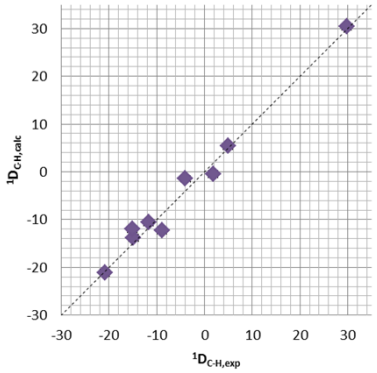
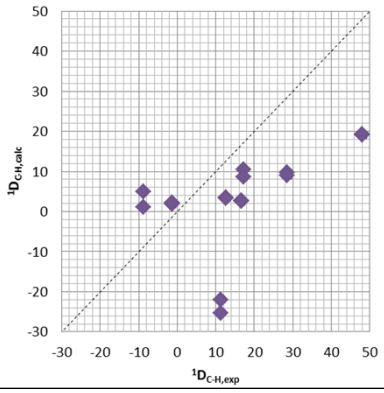
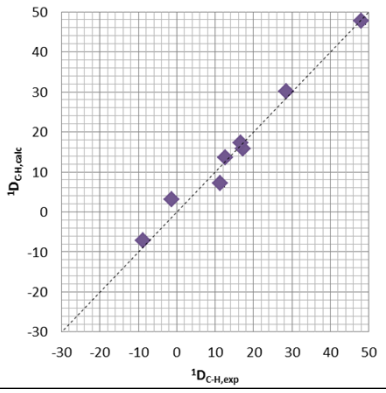
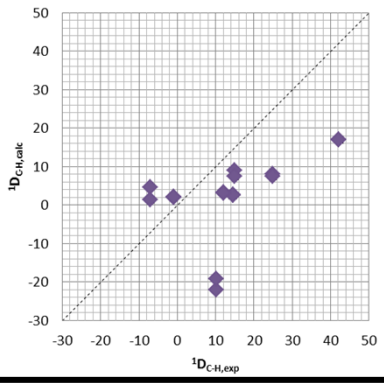
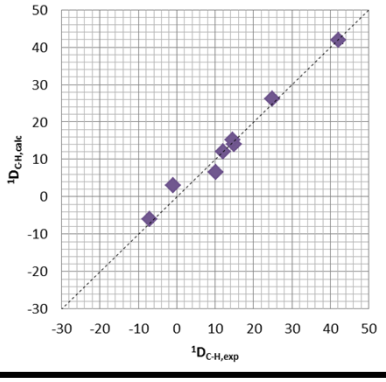
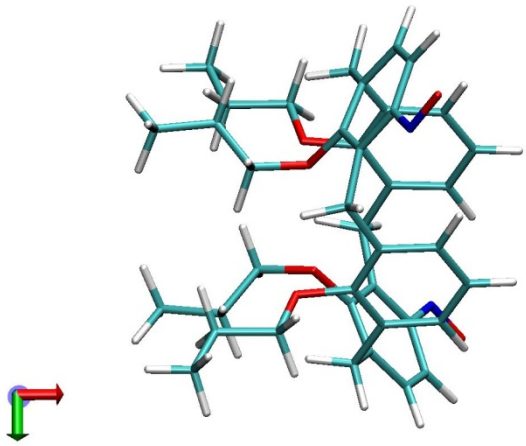
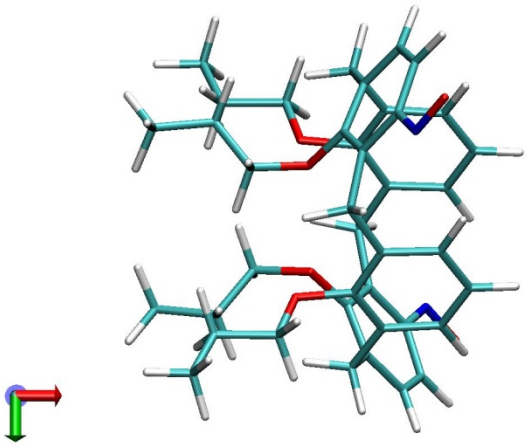
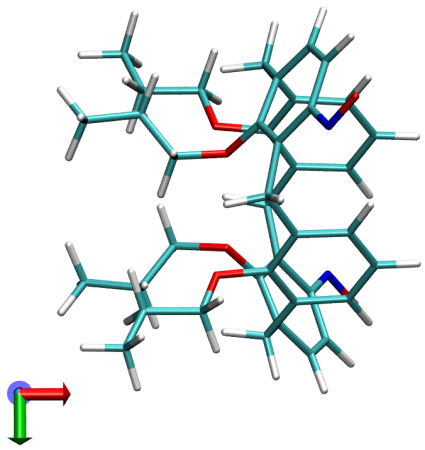


Fig. 1: Comparison of selected region of ^{13}C Z-restored proton coupled spectra of 5 in various alignment media.

Tab. 3: Comparison of fitting quality of structures 5 (*I*) and 5(*II*) in various alignment media.

	5 (*I*) – wrong conformer	5 (*II*) – right conformer
input structures		
medium A		
medium B		
medium C		

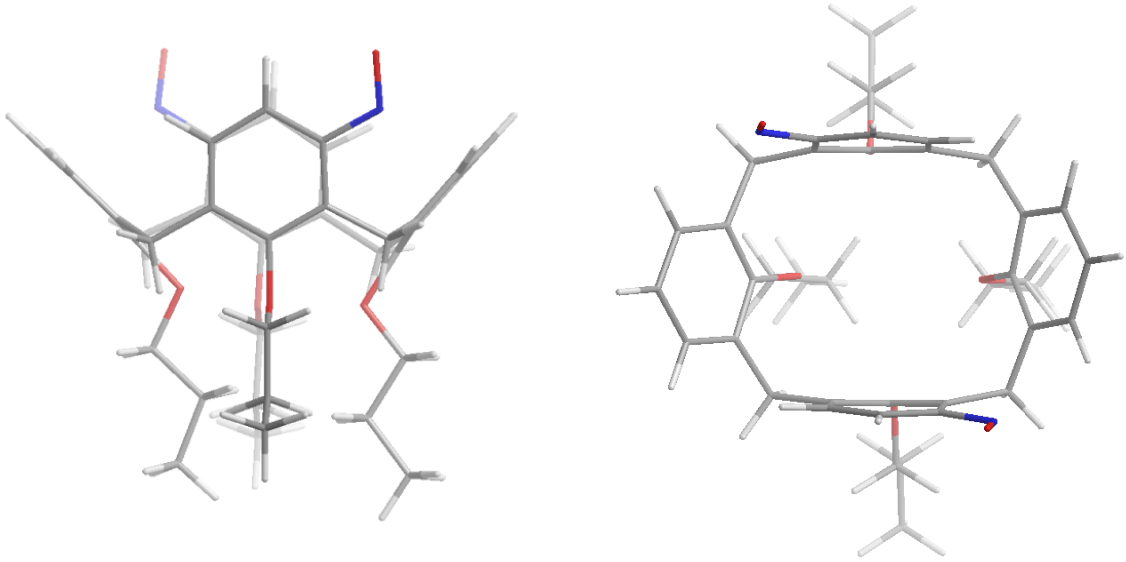
Tab. 4: Comparison of molecular orientations within the magnetic field.

Alignment medium	Orientation of 5 within the magnetic field
A PhePAC	
B HMW-PBDG	
C PBLG	

DFT OPTIMIZATION OF 5(*I*) AND 5(*II*) CONFORMERS

***Ab initio* geometry optimizations were performer by program Gaussian03**
(M. J. Frisch, H. B. Schlegel, G. E. Scuseria, M. A. Robb, J. R. Cheeseman, J. A. Montgomery, Jr., T. Vreven, K. N. Kudin, J. C. Burant, J. M. Millam, S. S. Iyengar, J. Tomasi, V. Barone, B. Mennucci, M. Cossi, G. Scalmani, N. Rega, G. A. Petersson, H. Nakatsuji, M. Hada, M. Ehara, K. Toyota, R. Fukuda, J. Hasegawa, M. Ishida, T. Nakajima, Y. Honda, O. Kitao, H. Nakai, M. Klene, X. Li, J. E. Knox, H. P. Hratchian, J. B. Cross, V. Bakken, C. Adamo, J. Jaramillo, R. Gomperts, R. E. Stratmann, O. Yazyev, A. J. Austin, R. Cammi, C. Pomelli, J. W. Ochterski, P. Y. Ayala, K. Morokuma, G. A. Voth, P. Salvador, J. J. Dannenberg, V. G. Zakrzewski, S. Dapprich, A. D. Daniels, M. C. Strain, O. Farkas, D. K. Malick, A. D. Rabuck, K. Raghavachari, J. B. Foresman, J. V. Ortiz, Q. Cui, A. G. Baboul, S. Clifford, J. Cioslowski, B. B. Stefanov, G. Liu, A. Liashenko, P. Piskorz, I. Komaromi, R. L. Martin, D. J. Fox, T. Keith, M. A. Al-Laham, C. Y. Peng, A. Nanayakkara, M. Challacombe, P. M. W. Gill, B. Johnson, W. Chen, M. W. Wong, C. Gonzalez, and J. A. Pople, Gaussian, Inc., Wallingford CT, **2004**.)

FINAL GEOMETRY of 5(*I*)
DFT method: RB3LYP/6-31G*



REMARK This PDB file was created by CS Chem3D.

ATOM	1	O1	1	1.159	1.488	1.366	O
ATOM	2	O2	1	-0.879	0.316	-1.816	O
ATOM	3	H3	1	2.294	-4.698	-0.033	H
ATOM	4	O4	1	-2.418	1.652	0.543	O
ATOM	5	H5	1	-3.275	-3.089	2.645	H
ATOM	6	H6	1	-2.770	-4.074	-3.962	H
ATOM	7	C7	1	-2.550	0.372	1.052	C
ATOM	8	O8	1	2.551	0.372	-1.392	O
ATOM	9	C9	1	-3.493	2.522	0.935	C
ATOM	10	H10	1	1.861	-1.589	5.546	H
ATOM	11	C11	1	-2.371	-3.181	-3.489	C
ATOM	12	C12	1	-3.201	-2.379	-2.707	C
ATOM	13	C13	1	-2.712	-1.212	-2.105	C
ATOM	14	C14	1	-3.223	-0.606	0.293	C
ATOM	15	C15	1	-3.488	-1.843	0.917	C
ATOM	16	C16	1	-3.030	-2.126	2.210	C
ATOM	17	C17	1	-2.270	-1.188	2.881	C
ATOM	18	C18	1	-2.010	0.076	2.320	C
ATOM	19	C19	1	2.272	-3.649	-0.305	C
ATOM	20	C20	1	1.644	-3.197	-1.449	C

```
ATOM     21  C21         1       1.698  -1.841  -1.818                       C
ATOM     22  C22         1       0.993  -1.390  -3.093                       C
ATOM     23  C23         1      -3.251   3.903   0.343                       C
ATOM     24  H24         1      -3.546   2.575   2.033                       H
ATOM     25  H25         1      -4.449   2.109   0.580                       H
ATOM     26  C26         1       2.896  -2.728   0.547                       C
ATOM     27  C27         1      -1.370  -0.867  -2.330                       C
ATOM     28  C28         1       2.632  -1.001   3.624                       C
ATOM     29  C29         1      -4.360   4.893   0.716                       C
ATOM     30  H30         1      -3.176   3.812  -0.748                       H
ATOM     31  H31         1      -2.280   4.273   0.695                       H
ATOM     32  H32         1      -4.165   5.879   0.281                       H
ATOM     33  H33         1      -4.437   5.019   1.802                       H
ATOM     34  H34         1      -5.337   4.556   0.350                       H
ATOM     35  C35         1       2.383  -0.939  -0.980                       C
ATOM     36  C36         1       2.915  -1.346   0.261                       C
ATOM     37  C37         1       3.412  -0.331   1.290                       C
ATOM     38  C38         1       2.440  -0.234   2.468                       C
ATOM     39  C39         1      -3.587  -0.377  -1.171                       C
ATOM     40  C40         1      -1.022  -2.856  -3.638                       C
ATOM     41  C41         1      -0.496  -1.702  -3.048                       C
ATOM     42  C42         1       1.682  -1.011   4.644                       C
ATOM     43  C43         1      -1.118   1.068   3.068                       C
ATOM     44  C44         1       1.282   0.559   2.387                       C
ATOM     45  C45         1       0.484  -0.316   4.483                       C
ATOM     46  C46         1       0.250   0.456   3.340                       C
ATOM     47  C47         1       3.769   0.567  -2.131                       C
ATOM     48  C48         1       3.864   2.024  -2.560                       C
ATOM     49  H49         1       3.777  -0.095  -3.010                       H
ATOM     50  H50         1       4.629   0.290  -1.504                       H
ATOM     51  H51         1       3.829   2.659  -1.666                       H
ATOM     52  H52         1       2.981   2.276  -3.160                       H
ATOM     53  C53         1       5.142   2.306  -3.358                       C
ATOM     54  H54         1       5.190   3.358  -3.658                       H
ATOM     55  H55         1       5.187   1.698  -4.269                       H
ATOM     56  H56         1       6.040   2.088  -2.768                       H
ATOM     57  H57         1      -4.234  -2.665  -2.542                       H
ATOM     58  H58         1      -1.863  -1.426   3.858                       H
ATOM     59  H59         1       1.108  -3.895  -2.086                       H
ATOM     60  H60         1       1.437  -1.911  -3.951                       H
ATOM     61  H61         1       1.164  -0.322  -3.232                       H
ATOM     62  H62         1       3.529  -1.605   3.712                       H
ATOM     63  H63         1       4.394  -0.633   1.664                       H
ATOM     64  H64         1       3.509   0.637   0.797                       H
ATOM     65  H65         1      -4.632  -0.651  -1.338                       H
ATOM     66  H66         1      -3.475   0.686  -1.390                       H
ATOM     67  H67         1      -0.369  -3.504  -4.220                       H
ATOM     68  H68         1      -1.022   1.974   2.470                       H
ATOM     69  H69         1      -1.593   1.335   4.021                       H
ATOM     70  H70         1      -0.279  -0.368   5.256                       H
ATOM     71  C71         1      -1.018   1.413  -2.733                       C
ATOM     72  C72         1      -0.320   2.624  -2.143                       C
ATOM     73  C73         1      -0.413   3.852  -3.054                       C
ATOM     74  C74         1       1.742   2.743   1.759                       C
ATOM     75  C75         1       1.539   3.781   0.668                       C
ATOM     76  C76         1       2.176   5.125   1.036                       C
ATOM     77  H77         1      -2.085   1.617  -2.910                       H
ATOM     78  H78         1      -0.576   1.139  -3.704                       H
ATOM     79  H79         1      -0.768   2.826  -1.167                       H
ATOM     80  H80         1       0.724   2.353  -1.957                       H
ATOM     81  H81         1       0.102   4.710  -2.608                       H
ATOM     82  H82         1      -1.455   4.148  -3.229                       H
ATOM     83  H83         1       0.045   3.665  -4.034                       H
ATOM     84  H84         1       2.815   2.602   1.960                       H
ATOM     85  H85         1       1.281   3.080   2.701                       H
ATOM     86  H86         1       0.465   3.905   0.492                       H
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ATOM     87  H87         1       1.968   3.398  -0.264                       H
ATOM     88  H88         1       2.016   5.864   0.243                       H
ATOM     89  H89         1       3.258   5.029   1.186                       H
ATOM     90  H90         1       1.749   5.534   1.960                       H
ATOM     91  N91         1      -4.307  -2.765   0.186                       N
ATOM     92  O92         1      -4.512  -3.839   0.742                       O
ATOM     93  N93         1       3.613  -3.153   1.713                       N
ATOM     94  O94         1       3.575  -4.356   1.945                       O
CONECT    1   44   74
CONECT    2   27   71
CONECT    3   19
CONECT    4    7    9
CONECT    5   16
CONECT    6   11
CONECT    7    4   14   18
CONECT    8   35   47
CONECT    9    4   23   24   25
CONECT   10   42
CONECT   11    6   12   40
CONECT   12   11   13   57
CONECT   13   12   27   39
CONECT   14    7   15   39
CONECT   15   14   16   91
CONECT   16    5   15   17
CONECT   17   16   18   58
CONECT   18    7   17   43
CONECT   19    3   20   26
CONECT   20   19   21   59
CONECT   21   20   22   35
CONECT   22   21   41   60   61
CONECT   23    9   29   30   31
CONECT   24    9
CONECT   25    9
CONECT   26   19   36   93
CONECT   27    2   13   41
CONECT   28   38   42   62
CONECT   29   23   32   33   34
CONECT   30   23
CONECT   31   23
CONECT   32   29
CONECT   33   29
CONECT   34   29
CONECT   35    8   21   36
CONECT   36   26   35   37
CONECT   37   36   38   63   64
CONECT   38   28   37   44
CONECT   39   13   14   65   66
CONECT   40   11   41   67
CONECT   41   22   27   40
CONECT   42   10   28   45
CONECT   43   18   46   68   69
CONECT   44    1   38   46
CONECT   45   42   46   70
CONECT   46   43   44   45
CONECT   47    8   48   49   50
CONECT   48   47   51   52   53
CONECT   49   47
CONECT   50   47
CONECT   51   48
CONECT   52   48
CONECT   53   48   54   55   56
CONECT   54   53
CONECT   55   53
CONECT   56   53
CONECT   57   12
CONECT   58   17
```

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CONECT   59   20
CONECT   60   22
CONECT   61   22
CONECT   62   28
CONECT   63   37
CONECT   64   37
CONECT   65   39
CONECT   66   39
CONECT   67   40
CONECT   68   43
CONECT   69   43
CONECT   70   45
CONECT   71    2   72   77   78
CONECT   72   71   73   79   80
CONECT   73   72   81   82   83
CONECT   74    1   75   84   85
CONECT   75   74   76   86   87
CONECT   76   75   88   89   90
CONECT   77   71
CONECT   78   71
CONECT   79   72
CONECT   80   72
CONECT   81   73
CONECT   82   73
CONECT   83   73
CONECT   84   74
CONECT   85   74
CONECT   86   75
CONECT   87   75
CONECT   88   76
CONECT   89   76
CONECT   90   76
CONECT   91   15   92
CONECT   92   91
CONECT   93   26   94
CONECT   94   93
END
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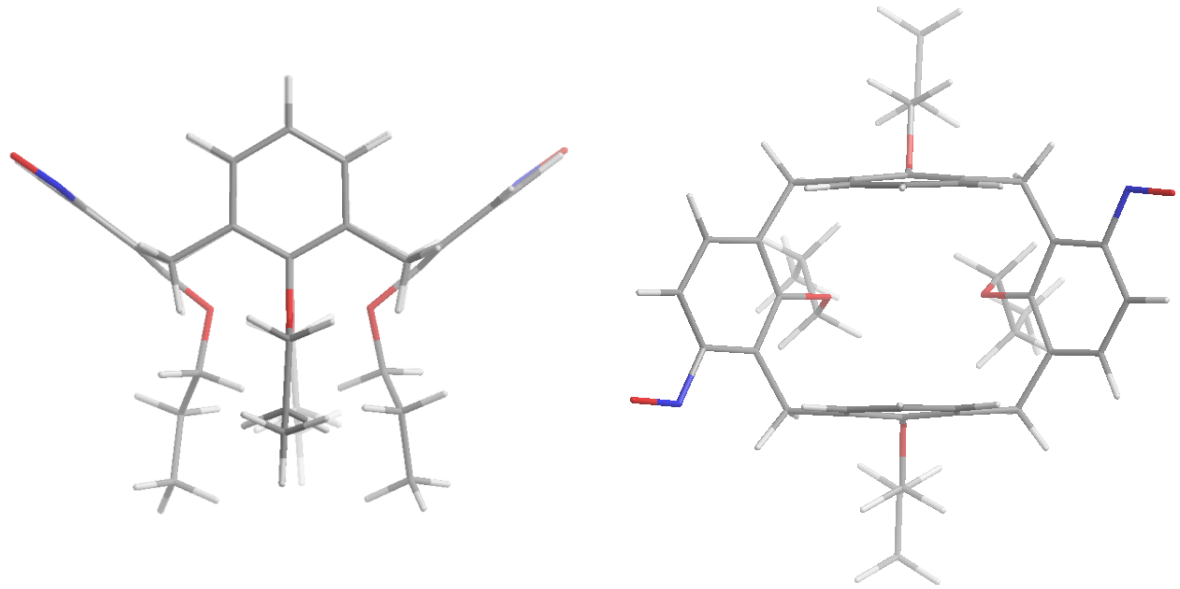
FINAL GEOMETRY of 5(*II*)
DFT method: RB3LYP/6-31G*



REMARK This PDB file was created by CS Chem3D.

ATOM	1	H1	1	-3.446	1.080	0.688	H
ATOM	2	C2	1	-3.536	0.885	-0.381	C
ATOM	3	H3	1	3.446	-1.080	0.688	H
ATOM	4	C4	1	3.536	-0.885	-0.381	C
ATOM	5	C5	1	1.927	0.668	2.050	C
ATOM	6	H6	1	-0.616	-3.137	-3.021	H
ATOM	7	H7	1	-2.987	-4.302	-1.855	H
ATOM	8	H8	1	0.682	3.373	0.739	H
ATOM	9	C9	1	2.225	-3.627	1.344	C
ATOM	10	C10	1	-0.895	-3.428	-0.329	C
ATOM	11	H11	1	3.221	-3.620	0.877	H
ATOM	12	C12	1	-3.481	-0.612	-0.663	C
ATOM	13	C13	1	-4.261	-2.578	-1.913	C
ATOM	14	H14	1	-0.682	-3.373	0.739	H
ATOM	15	H15	1	-3.221	3.620	0.877	H
ATOM	16	C16	1	0.243	-2.735	-2.488	C
ATOM	17	H17	1	-1.699	4.522	0.978	H
ATOM	18	C18	1	-4.415	-1.241	-1.515	C
ATOM	19	C19	1	-3.142	-3.283	-1.510	C
ATOM	20	C20	1	2.415	1.396	-0.179	C
ATOM	21	C21	1	2.188	2.702	-0.654	C
ATOM	22	H22	1	-5.016	-3.011	-2.561	H
ATOM	23	H23	1	2.999	0.435	2.080	H
ATOM	24	H24	1	1.012	4.487	-0.592	H
ATOM	25	H25	1	4.505	-1.264	-0.712	H
ATOM	26	H26	1	3.118	-1.025	-3.058	H
ATOM	27	C27	1	2.410	-1.607	-1.114	C
ATOM	28	C28	1	0.283	-2.826	-1.092	C
ATOM	29	C29	1	1.399	-2.297	-0.421	C
ATOM	30	H30	1	-3.118	1.025	-3.058	H
ATOM	31	C31	1	2.335	-1.544	-2.510	C
ATOM	32	H32	1	-1.012	-4.487	-0.592	H
ATOM	33	C33	1	3.481	0.612	-0.663	C
ATOM	34	C34	1	-2.415	-1.396	-0.179	C
ATOM	35	C35	1	-0.243	2.735	-2.488	C
ATOM	36	C36	1	-0.283	2.826	-1.092	C
ATOM	37	C37	1	0.895	3.428	-0.329	C

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ATOM     38  C38         1      -2.188  -2.702  -0.654                       C
ATOM     39  C39         1       1.273  -2.125  -3.198                       C
ATOM     40  C40         1       4.261   2.578  -1.913                       C
ATOM     41  H41         1       5.016   3.011  -2.561                       H
ATOM     42  C42         1      -1.399   2.297  -0.421                       C
ATOM     43  H43         1      -1.233   2.074  -4.282                       H
ATOM     44  O44         1       1.501  -2.449   0.956                       O
ATOM     45  C45         1      -2.335   1.544  -2.510                       C
ATOM     46  C46         1      -1.273   2.124  -3.198                       C
ATOM     47  O47         1      -1.501   2.449   0.956                       O
ATOM     48  O48         1      -1.501  -0.849   0.686                       O
ATOM     49  O49         1       1.501   0.849   0.686                       O
ATOM     50  C50         1       4.415   1.241  -1.515                       C
ATOM     51  H51         1       2.987   4.302  -1.855                       H
ATOM     52  C52         1      -2.410   1.607  -1.114                       C
ATOM     53  C53         1      -2.225   3.628   1.344                       C
ATOM     54  C54         1       3.142   3.283  -1.510                       C
ATOM     55  H55         1       1.233  -2.074  -4.282                       H
ATOM     56  H56         1      -4.505   1.264  -0.712                       H
ATOM     57  H57         1       1.698  -4.522   0.978                       H
ATOM     58  H58         1       1.382  -0.210   2.403                       H
ATOM     59  O59         1      -6.331  -1.022  -2.676                       O
ATOM     60  N60         1      -5.512  -0.440  -1.974                       N
ATOM     61  H61         1       0.616   3.137  -3.021                       H
ATOM     62  N62         1       5.512   0.440  -1.974                       N
ATOM     63  O63         1       6.331   1.022  -2.676                       O
ATOM     64  C64         1      -1.927  -0.668   2.050                       C
ATOM     65  H65         1      -1.382   0.210   2.403                       H
ATOM     66  H66         1      -2.999  -0.435   2.080                       H
ATOM     67  C67         1       2.346  -3.658   2.861                       C
ATOM     68  H68         1       1.340  -3.655   3.299                       H
ATOM     69  H69         1       2.837  -2.734   3.195                       H
ATOM     70  C70         1       3.129  -4.879   3.354                       C
ATOM     71  H71         1       3.201  -4.882   4.447                       H
ATOM     72  H72         1       2.644  -5.815   3.051                       H
ATOM     73  H73         1       4.150  -4.889   2.953                       H
ATOM     74  C74         1      -2.346   3.658   2.861                       C
ATOM     75  H75         1      -1.340   3.655   3.299                       H
ATOM     76  H76         1      -2.837   2.735   3.195                       H
ATOM     77  C77         1      -3.129   4.880   3.354                       C
ATOM     78  H78         1      -3.201   4.883   4.447                       H
ATOM     79  H79         1      -2.644   5.815   3.051                       H
ATOM     80  H80         1      -4.149   4.889   2.953                       H
ATOM     81  C81         1      -1.638  -1.885   2.926                       C
ATOM     82  H82         1      -2.201  -2.748   2.547                       H
ATOM     83  H83         1      -0.574  -2.131   2.836                       H
ATOM     84  C84         1      -2.008  -1.629   4.392                       C
ATOM     85  H85         1      -3.075  -1.399   4.502                       H
ATOM     86  H86         1      -1.794  -2.506   5.011                       H
ATOM     87  H87         1      -1.442  -0.785   4.805                       H
ATOM     88  C88         1       1.637   1.885   2.926                       C
ATOM     89  H89         1       2.201   2.748   2.547                       H
ATOM     90  H90         1       0.574   2.131   2.836                       H
ATOM     91  C91         1       2.008   1.629   4.391                       C
ATOM     92  H92         1       3.075   1.399   4.502                       H
ATOM     93  H93         1       1.794   2.506   5.011                       H
ATOM     94  H94         1       1.442   0.785   4.805                       H
CONECT    1    2
CONECT    2    1   12   52   56
CONECT    3    4
CONECT    4    3   25   27   33
CONECT    5   23   49   58   88
CONECT    6   16
CONECT    7   19
CONECT    8   37
CONECT    9   11   44   57   67
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CONECT   10   14   28   32   38
CONECT   11    9
CONECT   12    2   18   34
CONECT   13   18   19   22
CONECT   14   10
CONECT   15   53
CONECT   16    6   28   39
CONECT   17   53
CONECT   18   12   13   60
CONECT   19    7   13   38
CONECT   20   21   33   49
CONECT   21   20   37   54
CONECT   22   13
CONECT   23    5
CONECT   24   37
CONECT   25    4
CONECT   26   31
CONECT   27    4   29   31
CONECT   28   10   16   29
CONECT   29   27   28   44
CONECT   30   45
CONECT   31   26   27   39
CONECT   32   10
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CONECT   34   12   38   48
CONECT   35   36   46   61
CONECT   36   35   37   42
CONECT   37    8   21   24   36
CONECT   38   10   19   34
CONECT   39   16   31   55
CONECT   40   41   50   54
CONECT   41   40
CONECT   42   36   47   52
CONECT   43   46
CONECT   44    9   29
CONECT   45   30   46   52
CONECT   46   35   43   45
CONECT   47   42   53
CONECT   48   34   64
CONECT   49    5   20
CONECT   50   33   40   62
CONECT   51   54
CONECT   52    2   42   45
CONECT   53   15   17   47   74
CONECT   54   21   40   51
CONECT   55   39
CONECT   56    2
CONECT   57    9
CONECT   58    5
CONECT   59   60
CONECT   60   18   59
CONECT   61   35
CONECT   62   50   63
CONECT   63   62
CONECT   64   48   65   66   81
CONECT   65   64
CONECT   66   64
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CONECT   69   67
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CONECT   71   70
CONECT   72   70
CONECT   73   70
CONECT   74   53   75   76   77
CONECT   75   74
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CONECT   76   74
CONECT   77   74   78   79   80
CONECT   78   77
CONECT   79   77
CONECT   80   77
CONECT   81   64   82   83   84
CONECT   82   81
CONECT   83   81
CONECT   84   81   85   86   87
CONECT   85   84
CONECT   86   84
CONECT   87   84
CONECT   88    5   89   90   91
CONECT   89   88
CONECT   90   88
CONECT   91   88   92   93   94
CONECT   92   91
CONECT   93   91
CONECT   94   91
END
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