Supplementary Material (ESI) for Chemical Communications

Ferrocenium Salts Mediate *Para-tert*-butylcalixarene Synthesis

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General protocols

All reactions requiring anhydrous conditions were conducted in flame-dried glass apparatus under an atmosphere of argon. Water refers to distilled water. Magnesium sulphate dried GPR was obtained from VWR BDH (catalog number 291186R) and all commercially available chemicals, reagents and salts were used as supplied.

Melting points were recorded using open capillary tubes on a Gallenkamp melting point apparatus and are uncorrected. Infrared spectra were recorded on a Perkin-Elmer Paragon 1000FT infrared spectrometer either as a thin film or neat sample. ^{1}H- and ^{13}C-NMR spectra were recorded in Fourier transform mode at the field strength specified either on a Bruker AC-400 or a Varian Gemini 300 spectrometer and unless otherwise stated deuterated chloroform was used as solvent. The ^{1}H-spectra were recorded in ppm and are referenced to the residual $CHCl_3$ signal located at δ 7.26. ^{13}C-NMR spectra were recorded in ppm and referenced to the residual $CHCl_3$ signal found at δ 77.00. Multiplicities in the NMR spectra are described as: s = singlet, d = doublet, t = triplet, q= quartet, m = multiplet, br = broad; coupling constants are reported in Hz. Low resolution mass spectra were run on a either a Micromass Quattro II, Finnigan MAT95 or MAT 900 spectrometer. Ion mass/charge (m/z) ratios are reported as values in atomic mass units. Microwave syntheses were performed on a Personal Chemistry Emrys Creator. Thin layer chromatography was performed on Merck aluminium plates coated with 0.2 mm silica gel-60 F_{254}. Flash column chromatography was performed on silica gel (Kieselgel 60).

General procedure for the synthesis of $\mathbf{2_{[4]}}$ - $\mathbf{2_{[9]}}$

Para-tert-butylphenol (450 mg, 3 mmol), *s*-trioxane (270 mg, 3 mmol), ferrocenium tetrafluoroborate (818 mg, 3 mmol) were added to dichloromethane (4 mL) in a Biotage microwave vial of 2.0-5.0 mL capacity. This heterogeneous mixture was heated (after sealing with aluminum crimp cap with a PTFE lined rubber seal) in the microwave synthesiser at 120 °C for 30 min. After which, HPLC analysis of the reaction mixture indicated complete consumption of the starting material (**1**) and the formation of mixture of *para-tert*-butyl calix[n]arenes ($\mathbf{2_{[4]} - 2_{[9]}}$) in ~88 - 90 % yield (Figure 3).

The reaction mixture was diluted with dichloromethane (10 mL) and filtered through a pad of celite to remove insoluble solids. The residue was washed with dichloromethane (5 mL). The

combined organic extracts were washed with brine (5 mL). The organic layer was dried using anhydrous magnesium sulphate, filtered and the solvent removed under reduced pressure affording a white solid.

The individual calix[4-9]arenes were isolated (in 85 - 99% HPLC purity, Figure 7) by gradient flash chromatography using cyclohexane–toluene-ethanol (90:10:00 to 60:48:2 in 120 min using 2 mL/min flow rate) solvent system (monitored by HPLC). The isolated yields of individual calix[4-9]arenes were: calix[4]arene (48 mg, 9.8 %), calix[5]arene (15 mg, 3.1 %), calix[6]arene (66 mg, 13.6 %), calix[7]arene (93 mg, 19.5 %), calix[8]arene (68 mg, 12.4%) and calix[9]arene (23 mg, 4.2 %).

HPLC Procedure:

HPLC analysis was performed on a Perkin-Elmer LC 200 instrument equipped with a diode-array detector and Varian Pursuit RP C18 column (4.6 mm i.d., 250 mm) packed with 5 μm reverse-phase silica. The analysis procedure followed was similar to that reported by Gutsche *et al*.[1] The HPLC separation of calix[4-9]arenes was achieved using a mixture of two eluents (at 280 nm), A and B at a flow rate of 2 mL/min. Eluent A was acetonitrile with 1% acetic acid and eluent B was a mixture of dichloromethane and *tert*-butyl methyl ether (12:9 ratio) with 1% acetic acid. An isocratic run using a 80/20 mixture of A and B for 15 min was found suitable for separation of calix[4-12]arenes. (Note: A significant shift in retention times was observed as a function of the variable room temperature. Higher retention times were noticed in cold conditions.)

LC-MS Procedure:

LC-MS studies were performed using Shimadzu LCMS-2010EV single quadrupole mass-spectrometer. LC was performed on Shimadzu prominence HPLC system using Pathfinder PS silica (3.5 μm, 2.1 x 50 mm) column and mobile phase used was acetonitrile (80%) and 5 mmol ammonium acetate in water (20%) at a flow rate of 0.2 mL/min. Mass spectrometry was[1] performed using ESI in negative ion mode detection.

[1] D. R. Stewart, C. D. Gutsche, *Journal of the American Chemical Society* **1999**, *121*, 4136.

5,11,17,23-Tetrakis(1,1-dimethylethyl)-25,26,27,28-tetrahydroxycalix[4]arene ($2_{[4]}$)

m.p. 335-340°C (reported[2] 342-344°C); FTIR (neat) 3152, 2953, 2864, 1479, 1199 cm^{-1}; ^{1}H ^{2}NMR (400 MHz, $CDCl_3$, 20°C, TMS) δ=10.33 (br s, 4H, ArO*H*), 7.03 (s, 8H, Ar*H*), 4.22 (d, *J*(H,H)=14.0 Hz, 4H, ArC*H*HAr), 3.46 (d, *J*(H,H)=14.0 Hz, 4H, ArCH*H*Ar), 1.23 (s, 36H, 4 × C(CH_3)$_3$), MS (APCI) *m/z* calc'd for ($C_{44}H_{56}O_4$) required 648.9 found 649.3.

5,11,17,23,29-Pentakis(1,1-dimethylethyl)-35,36,37,38,39-pentahydroxycalix[5]arene ($2_{[5]}$)

m.p. 285-288°C; FTIR 3262, 2957, 2868, 1484, 1201 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$–(d_5)Pyridine (10%), 20°C, TMS) δ=8.64 (br s, 5H, ArO*H*), 7.15 (s, 10H, Ar*H*), 3.75 (br s, 10H, ArCH_2Ar), 1.18 (s, 45H, 5 × C(CH_3)$_3$); ^{13}C NMR (100 MHz, $CDCl_3$, 20°C, TMS) δ=147.8, 144.2, 128.0, 125.9 (Ar), 34.1 (*C*(CH$_3$)$_3$), 31.7 (Ar*C*H$_2$Ar), 31.6 (C(*C*H$_3$)$_3$), MS (APCI) *m/z* calc'd for ($C_{55}H_{70}O_5$) required 811.1 found 811.2.

5,11,17,23,29,35-Hexakis(1,1-dimethylethyl)-37,38,39,40,41,42-hexahydroxycalix[6]arene ($2_{[6]}$)

m.p. >350°C (reported[2] 372-374°C); FTIR (thin film) 3134, 2957, 2866, 1482, 1200 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$–(d_5)Pyridine (10%), 20°C, TMS) δ=10.44 (br s, 6H, ArO*H*), 7.10 (s, 12H, Ar*H*), 3.82 (br s, 12H, ArCH_2Ar), 1.20 (s, 54H, 6 × C(CH_3)$_3$); ^{13}C NMR (100 MHz, $CDCl_3$, 20°C, TMS) δ=147.5, 144.5, 127.2, 126.4 (Ar), 34.2 (*C*(CH$_3$)$_3$), 33.5 (Ar*C*H$_2$Ar), 31.7 (C(*C*H$_3$)$_3$); MS (APCI) *m/z* calc'd for ($C_{66}H_{84}O_6$) required 973.4 found 973.4.

5,11,17,23,29,35,41-Heptakis(1,1-dimethylethyl)-43,44,45,46,47,48,49-heptahydroxy calix[7]arene ($2_{[7]}$)

m.p. 235-238°C; FTIR 3143, 2956, 2883, 1483, 1200 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$–(d_5)Pyridine (10%), 20°C, TMS) δ=7.15 (s, 14H, Ar*H*), 3.88 (br s, 14H, ArCH_2Ar), 1.24 (s, 63H, 7 × C(CH_3)$_3$); ^{13}C NMR (100 MHz, $CDCl_3$, 20°C, TMS) δ=147.5, 144.6, 127.6, 126.3

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[2] A. Arduini, A. Casnati, in *Macrocycle Synthesis A Practical Approach,*(Eds.: D. Parker), Oxford University Press, Oxford, **1996**, pp145-173.

(Ar), 34.2 ($C(CH_3)_3$), 33.1 (ArCH_2Ar), 31.7 ($C(CH_3)_3$); MS (APCI) *m/z* calc'd for ($C_{77}H_{98}O_7$) required 1135.5 found 1135.5.

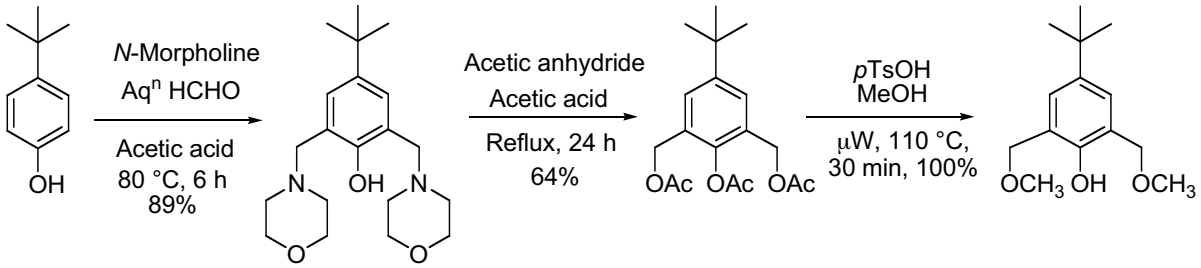
5,11,17,23,29,35,41,47-Octakis(1,1-dimethylethyl)-49,50,51,52,53,54,55,56-octahydroxycalix[8]arene ($2_{[8]}$)

m.p. >360°C (reported[2] 418-420°C); FTIR 3191, 2953, 2348, 1485, 1201; ^{1}H NMR (400 MHz, $CDCl_3$, 20°C, TMS) δ=9.62 (br s, 8H, ArO*H*), 7.17 (s, 16H, Ar*H*), 4.35 (d, *J*(H,H)= 12.8 Hz, 8H, ArC*H*HAr), 3.48 (d, *J*(H,H)=12.8 Hz, 8H, ArCH*H*Ar), 1.24 (s, 72H, 8 × $C(CH_3)_3$); ^{13}C NMR (100 MHz, $CDCl_3$, 20°C, TMS) δ=146.8, 144.9, and 128.9 (Ar), 125.7 (ArH), 34.2 ($C(CH_3)_3$), 32.5 (ArCH_2Ar), 31.7 ($C(CH_3)_3$); MS (MALDI) *m/z* calc'd for ($C_{88}H_{112}O_8Na$) required 1320.8 found 1320.8.

5,11,17,23,29,35,41,47,53-Nonakis(1,1-dimethylethyl)-55,56,57,58,59,60,61,62,63-nonahydroxycalix[9]arene[1] ($2_{[9]}$)

m.p. 302–304°C; FTIR 3194, 2955, 2360, 1484, 1202; ^{1}H NMR (400 MHz, $CDCl_3$–(d_5)Pyridine (10%), 20°C, TMS) δ=7.09 (d, 18H, *J*(H,H)=3.3 Hz, Ar*H*), 3.84 (br s, 18H, ArCH_2Ar), 1.17 (s, 81H, 9 × $C(CH_3)_3$); ^{13}C NMR (100 MHz, $CDCl_3$, 20°C, TMS) δ=147.2, 144.5, and 128.0 (Ar), 125.9 (ArH), 34.2 ($C(CH_3)_3$), 32.6 (ArCH_2Ar), 31.5 ($C(CH_3)_3$); MS (MALDI) *m/z* calc'd for ($C_{99}H_{126}O_9Na$) required 1482.9 found 1483.0.

Synthesis of 2,6-*bis*(methoxymethyl)-*para*-*tert*-butylphenol (4):



2,6-*Bis*(morpholinomethyl)-*para*-*tert*-butyl phenol:

A solution of *para*-*tert*-butylphenol (7.5 g, 50mmol) in glacial acetic acid (20 mL) was cooled to 10 °C. *N*-Morpholine (9.6 mL, 110 mmol) and aqueous formaldehyde (11.0 mL, 38% solution, 110 mmol) were sequentially added under an atmosphere of argon. The flask was flushed with argon and closed with a glass stopper. The mixture was heated at 80 °C for 6 h. The solvent was removed under reduced pressure. The residue was treated with saturated sodium bicarbonate solution (50 mL) and extracted with dichloromethane (3 × 50 mL). The combined organic extracts were washed with saturated sodium bicarbonate solution (50 mL),

dried using anhydrous magnesium sulphate and filtered. Evaporation of the solvent *in vacuo* and subsequent purification *via* column chromatography using dichloromethane / methanol (95 : 05) solvent system afforded the product as white waxy solid (18.2 g, 89.2%). m.p. 108-110°C; FTIR (thin film) 3418, 2925, 2859, 1641, 1493, 1452, 1116, 1050, 824 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$, 20°C, TMS) δ=11.83 (s, 1H, AcO*H*), 7.01 (s, 2H, ArH), 3.66-3.64 (m, 12H), 2.56 (s, 8H, 4 × CH_2), 1.92 (s, 3H, C*H*$_3$COOH), 1.36 (s, 9H, C(CH_3)$_3$) ppm; ^{13}C NMR (100 MHz, $CDCl_3$, 20°C, TMS) δ=175.7 (CH_3*C*OOH), 154.1, 141.7, 127.1, 120.2, 66.5, 59.1, 52.7, 34.1, 31.6, 22.5 (*C*H_3COOH) ppm; MS (ES) *m/z* calc'd for ($C_{22}H_{36}N_2O_5$) required 408.2 found 408.2 $[M + CH_3COOH]^+$.

O-Acetyl-2,6-*bis*(acetoxymethyl)-*para*-*tert*-butylphenol:

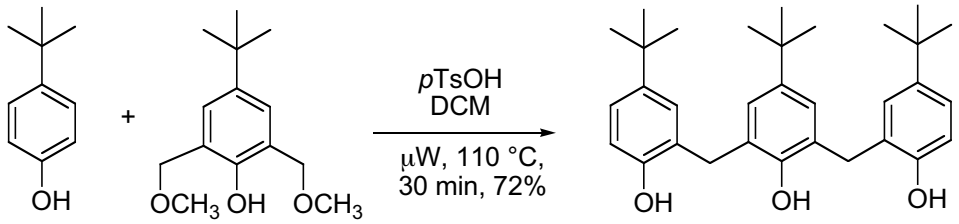
A mixture of 2,6-*bis*(morpholinomethyl)-*para*-*tert*-butylphenol (17.5 g, 42.9 mmol), acetic anhydride (50 mL) and glacial acetic acid (5 mL) was heated at reflux for 24 h under an atmosphere of nitrogen. The solvent was removed by vacuum distillation. The resultant oil was dissolved in dichloromethane (200 mL). The organic solution was washed successively with saturated sodium bicarbonate solution (3 × 75 mL) and water (75 mL). The organic layer was dried over anhydrous magnesium sulphate, filtered and the solvent was removed *in vacuo*. Purification *via* column chromatography using *n*-hexane / ethyl acetate as the eluent (80 : 20) afforded the triacetate as a clear oil (9.2 g, 63.8%). FTIR (thin film) 2962, 2867, 1765, 1741, 1606, 1484, 1367, 1225, 1177, 1026 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$, 20°C, TMS) δ=7.40 (s, 2H, ArH), 5.02 (s, 4H, 2 × CH_2O), 2.31 (s, 3H, ArOCOCH_3), 2.06 (s, 6H, 2 × COCH_3), 1.31 (s, 9H, C(CH_3)$_3$) ppm; ^{13}C NMR (100 MHz, $CDCl_3$, 20°C, TMS) δ=170.7, 169.5, 149.5, 145.9, 128.4, 128.3, 61.9, 34.7, 31.5, 21.0, 20.7 ppm; MS (ES) *m/z* calc'd for ($C_{18}H_{24}O_6Na$) required 359.2 found 359.2 $[M + Na]^+$.

2,6-*Bis*(methoxymethyl)-*para*-*tert*-butylphenol:

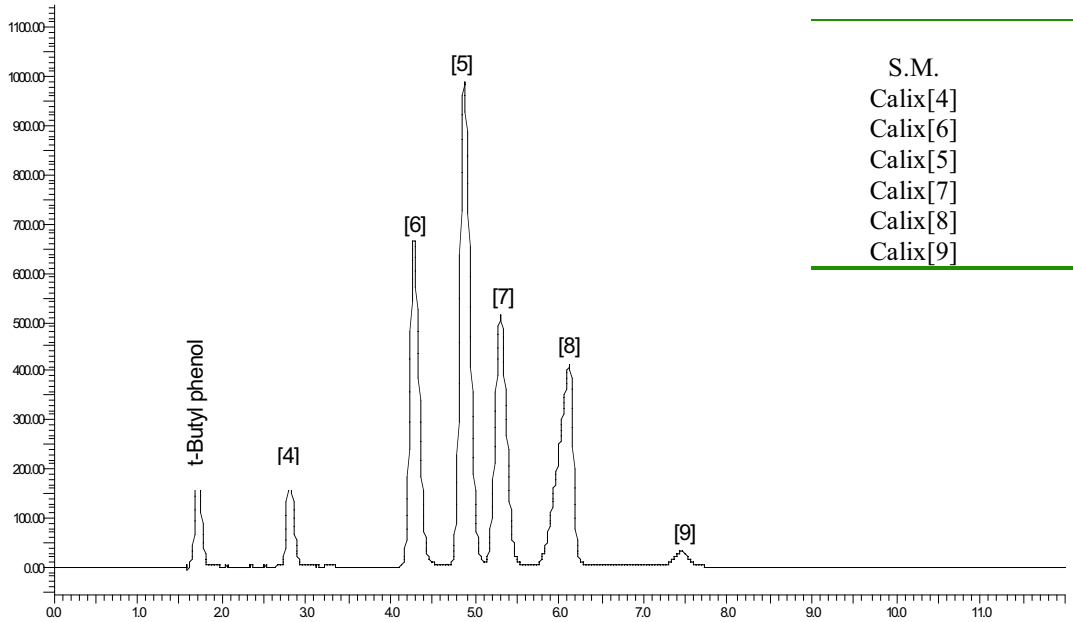
A solution of *O*-acetyl-2,6-*bis*(acetoxymethyl)-*para*-*tert*-butylphenol (1.0 g, 2.98 mmol) and *p*TsOH (40 mg, 0.15 mmol) in dry methanol (5.5 mL) was heated under microwave irradiation at 120 °C for 30 min in a sealed reaction tube. The solvent was removed *in vacuo* and residue re-dissolved in dichloromethane (15 mL). The organic layer was successively washed with 10% $NaHCO_3$ and brine. The organic solution was dried over anhydrous sodium sulphate. Evaporation of the solvent afforded **4** as clear oil in quantitative yield (710 mg, 100%). FTIR (thin film) 3425, 2929, 2865, 1112, 1050, 824 cm^{-1}; ^{1}H NMR (400 MHz,

$CDCl_3$, 20°C, TMS) δ=7.62 (br, 1H, O*H*), 7.12 (s, 2H, ArH), 4.58 (s, 4H, 2 × CH_2), 3.44 (s, 6H, 2 × OCH_3), 1.27 (s, 9H, $C(CH_3)_3$) ppm; ^{13}C NMR (75 MHz, $CDCl_3$, 20°C, TMS) δ=151.7, 141.9, 125.1, 122.5, 71.8, 57.9, 33.5, 31.0 ppm; MS (ES) *m/z* calc'd for ($C_{14}H_{22}O_3Na$) required 261.3 found 261.3 $[M + Na]^+$.

Synthesis of 2,6-[*bis*(5-*tert*-butylsalicyl)]-*para*-*tert*-butylphenol (3):

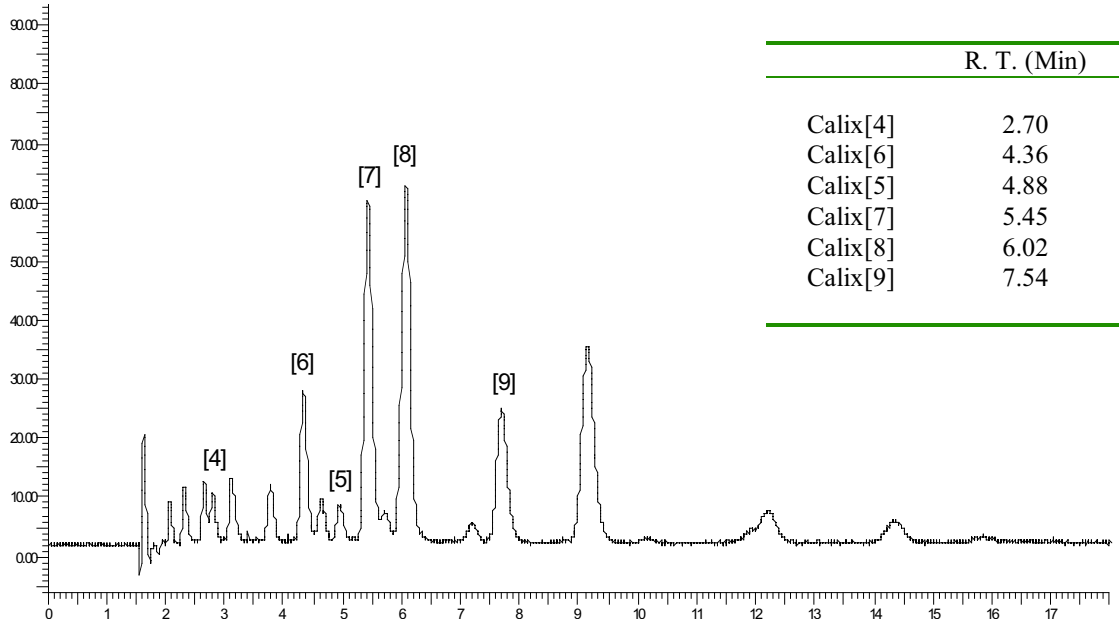


A solution of 2,6-*bis*(methoxymethyl)-*para*-*tert*-butylphenol (330 mg, 1.38 mmol) and *para*-*tert*-butylphenol (590 mg, 3.92 mmol) in dry dichloromethane (4 mL) containing *p*TsOH (28 mg, 0.1 mmol) was heated under microwave irradiation at 110 °C for 30 min in a sealed reaction tube. The organic layer was washed with 10% $NaHCO_3$, brine, dried over anhydrous magnesium sulphate and solvent evaporated under reduced pressure. Column chromatography using *n*-hexane-DCM (1:1) afforded the trimer **3** (475 mg, 72.5 %); FTIR (thin film) 3338, 2926, 2860, 1607, 1494, 1452, 1050 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$, 20°C, TMS) δ=7.28 (d, *J*(H,H)=2.4 Hz, 2H, ArH), 7.18 (d, *J*(H,H)=1.1 Hz, 2H, ArH), 7.06 (dd, *J*(H,H)=8.4, 2.4 Hz, 2H, ArH), 6.73 (d, *J*(H,H)=8.4 Hz, 2H, ArH), 3.90 (s, 4H, 2 × CH_2), 1.28 (s, 9H, $C(CH_3)_3$), 1.25 (s, 18H, 2 × $C(CH_3)_3$) ppm; ^{13}C NMR (100 MHz, $CDCl_3$, 20°C, TMS) δ=149.8, 147.5, 144.5, 144.5, 127.8, 127.5, 127.0, 126.2, 125.0, 115.9, 34.3, 34.2, 32.3, 31.8, 31.7 ppm; MS (ES) *m/z* calc'd for ($C_{32}H_{42}O_3Na$) required 497.3 found 497.4 $[M + Na]^+$.



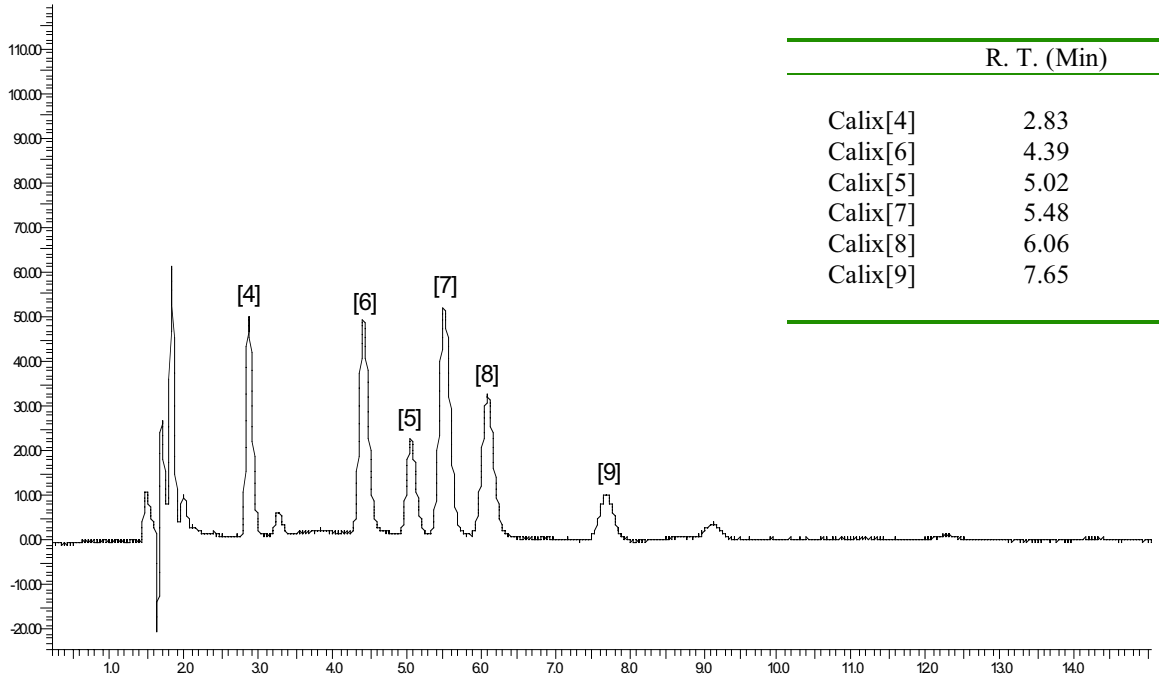
	R. T. (Min)
S.M.	1.73
Calix[4]	2.81
Calix[6]	4.31
Calix[5]	4.79
Calix[7]	5.35
Calix[8]	6.10
Calix[9]	7.48

Figure 1: HPLC chromatogram of the standard mixture of *para-tert*-butylcalix[4-9]arenes and *para-tert*-butylphenol



	R. T. (Min)	Comp. (%)
Calix[4]	2.70	2.5
Calix[6]	4.36	11.2
Calix[5]	4.88	2.3
Calix[7]	5.45	16.8
Calix[8]	6.02	19.5
Calix[9]	7.54	9.2

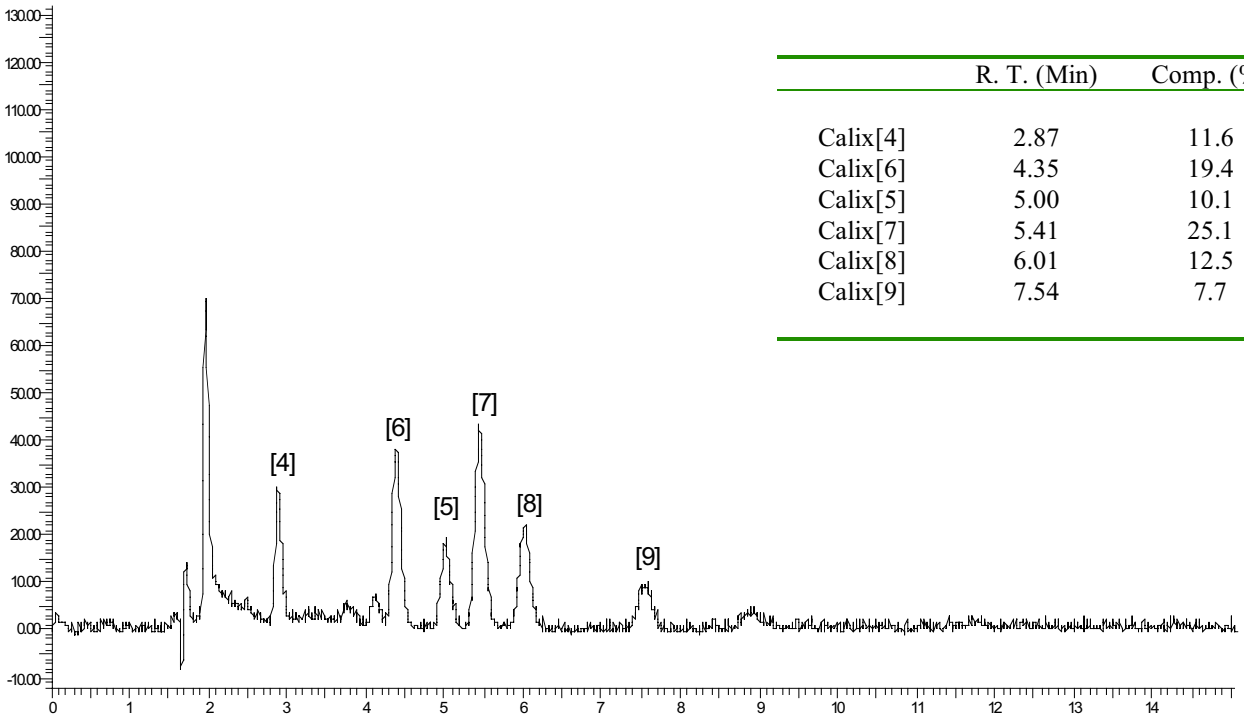
Figure 2: HPLC chromatogram of the reaction mixture after 7 hours reflux using the procedure given in reference 1.



	R. T. (Min)	Comp. (%)
Calix[4]	2.83	14.2
Calix[6]	4.39	18.9
Calix[5]	5.02	8.9
Calix[7]	5.48	22.6
Calix[8]	6.06	18.5
Calix[9]	7.65	5.1

Figure 3: HPLC chromatogram of the reaction mixture for the synthesis of $\mathbf{2}_{[4]} - \mathbf{2}_{[9]}$ using **1** and $[Fc]^+BF_4^-$

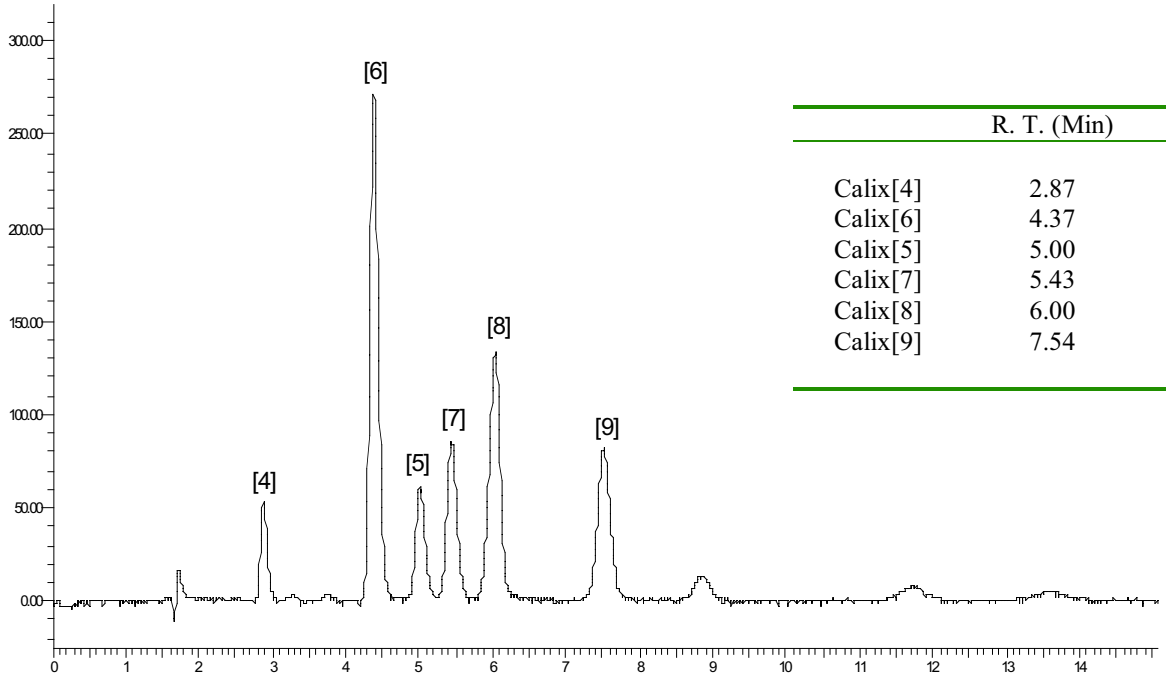
(A solution of **1** (450 mg, 3 mmol), $[Fc]^+BF_4^-$ (818 mg, 3 mmol) and *s*-trioxane (270 mg, 3 mmol) in CH_2Cl_2 (4 mL) was placed in a sealed reaction tube and irradiated with microwaves at 120 °C for 30 min.)



	R. T. (Min)	Comp. (%)
Calix[4]	2.87	11.6
Calix[6]	4.35	19.4
Calix[5]	5.00	10.1
Calix[7]	5.41	25.1
Calix[8]	6.01	12.5
Calix[9]	7.54	7.7

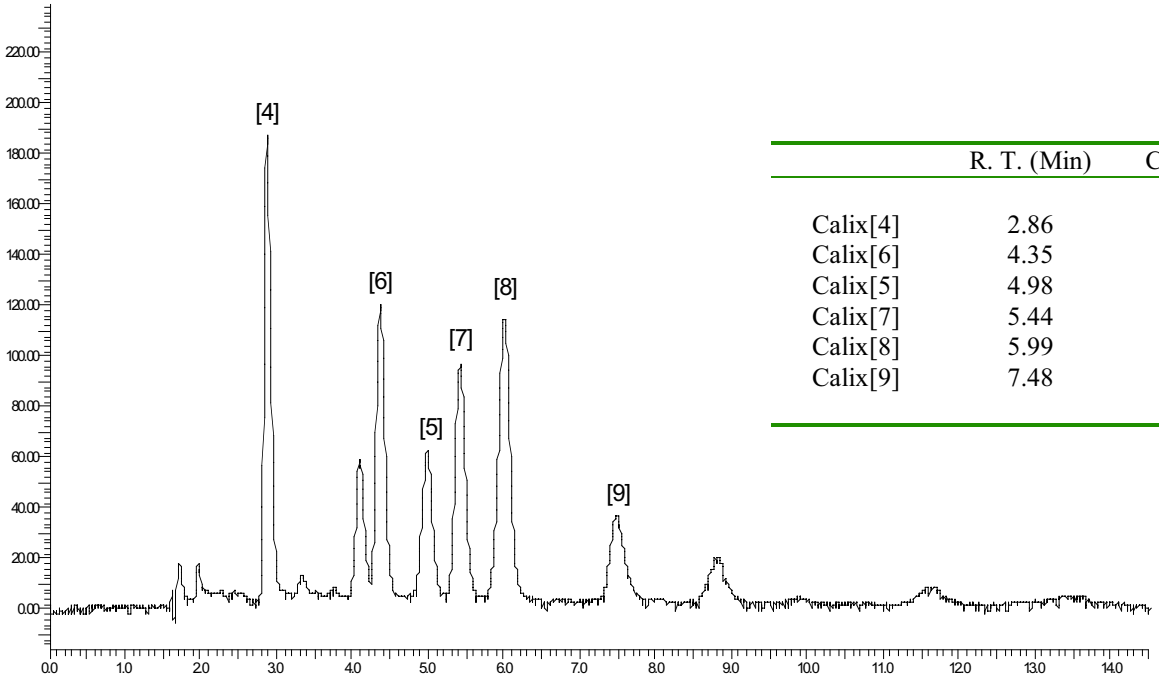
Figure 4: HPLC chromatogram of the reaction mixture for the synthesis of $\mathbf{2}_{[4]} - \mathbf{2}_{[9]}$ using **1** and $[Fc]^+PF_6^-$

(A solution of **1** (150 mg, 1 mmol), $[Fc]^+PF_6^-$ (330 mg, 1 mmol) and *s*-trioxane (90 mg, 1 mmol) in CH_2Cl_2 (4 mL) was placed in a sealed reaction tube and irradiated with microwaves at 120 °C for 60 min.)



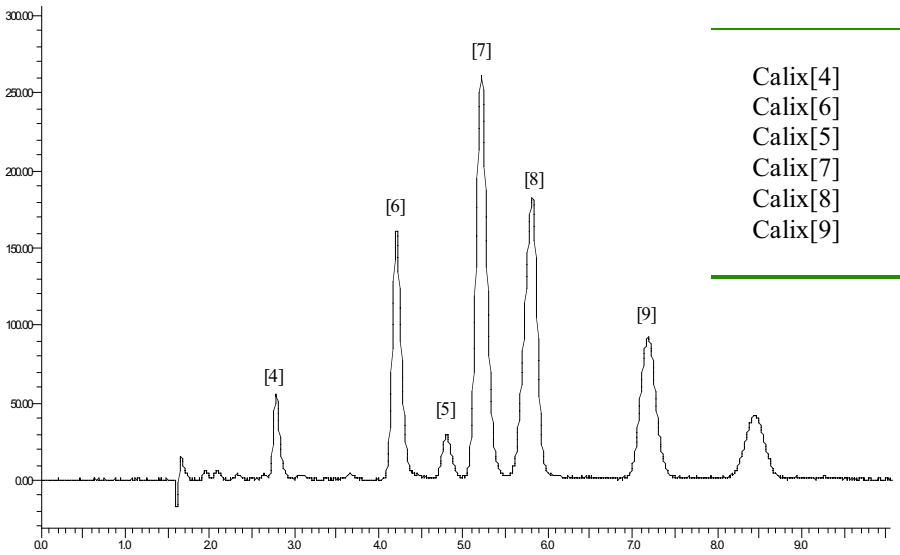
	R. T. (Min)	Comp. (%)
Calix[4]	2.87	4.9
Calix[6]	4.37	33.5
Calix[5]	5.00	8.1
Calix[7]	5.43	11.8
Calix[8]	6.00	20.1
Calix[9]	7.54	14.7

Figure 5: HPLC chromatogram of the reaction mixture for the synthesis of $\mathbf{2}_{[4]} - \mathbf{2}_{[9]}$ using **3** and $[Fc]^{+}BF_4^{-}$
(A solution of **3** (475 mg, 1 mmol), $[Fc]^{+}BF_4^{-}$ (270 mg, 1mmol) and s-trioxane (100 mg, 1.1 mmol) in CH_2Cl_2 (4 mL) was placed in a sealed reaction tube and irradiated with microwaves at 120 °C for 60 min.)



	R. T. (Min)	Comp. (%)
Calix[4]	2.86	18.8
Calix[6]	4.35	13.5
Calix[5]	4.98	8.9
Calix[7]	5.44	14.9
Calix[8]	5.99	19.2
Calix[9]	7.48	7.1

Figure 6: HPLC chromatogram of the reaction mixture for the synthesis of $\mathbf{2}_{[4]} - \mathbf{2}_{[9]}$ using **3, 4** and $[Fc]^{+}BF_4^{-}$
(A solution of **3** (475 mg, 1 mmol), **4** (240 mg, 1 mmol) and $[Fc]^{+}BF_4^{-}$ (270 mg, 1 mmol) in CH_2Cl_2 (4 mL) was placed in a sealed reaction tube and irradiated with microwaves at 120 °C for 60 min.)



	R. T. (Min)	Comp. (%)
Calix[4]	2.86	3.7
Calix[6]	4.35	14.8
Calix[5]	4.98	2.8
Calix[7]	5.44	30.0
Calix[8]	5.99	24.4
Calix[9]	7.48	13.9

Figure 7: HPLC chromatogram of the reaction mixture for the synthesis of $\mathbf{2}_{[4]}$ – $\mathbf{2}_{[9]}$ using **1**, trioxane and silver triflate

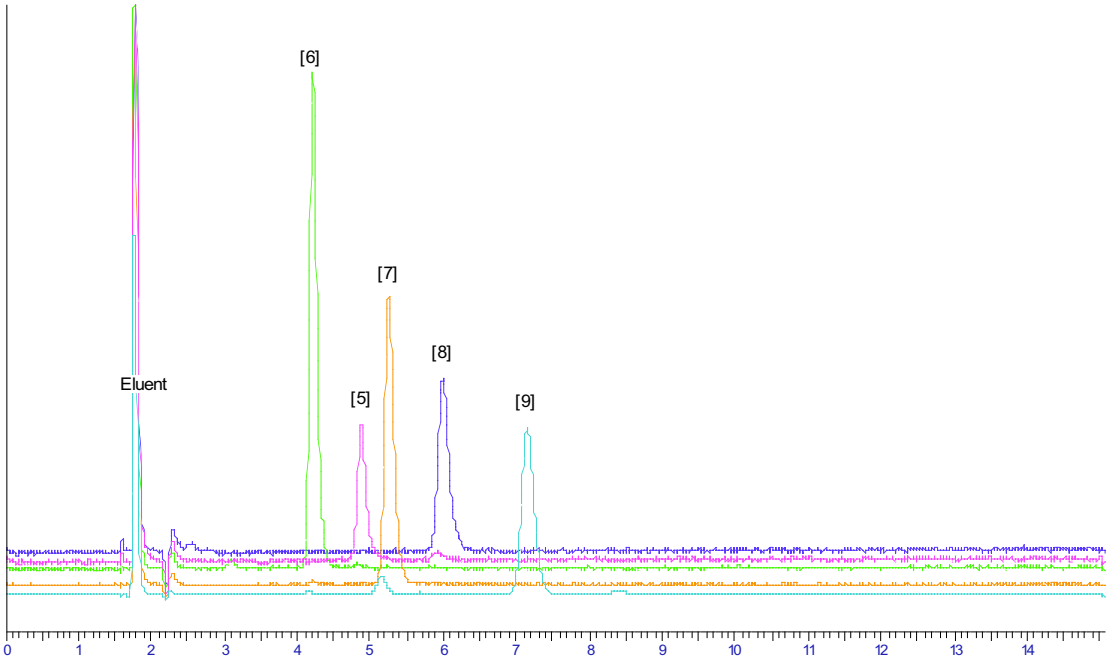
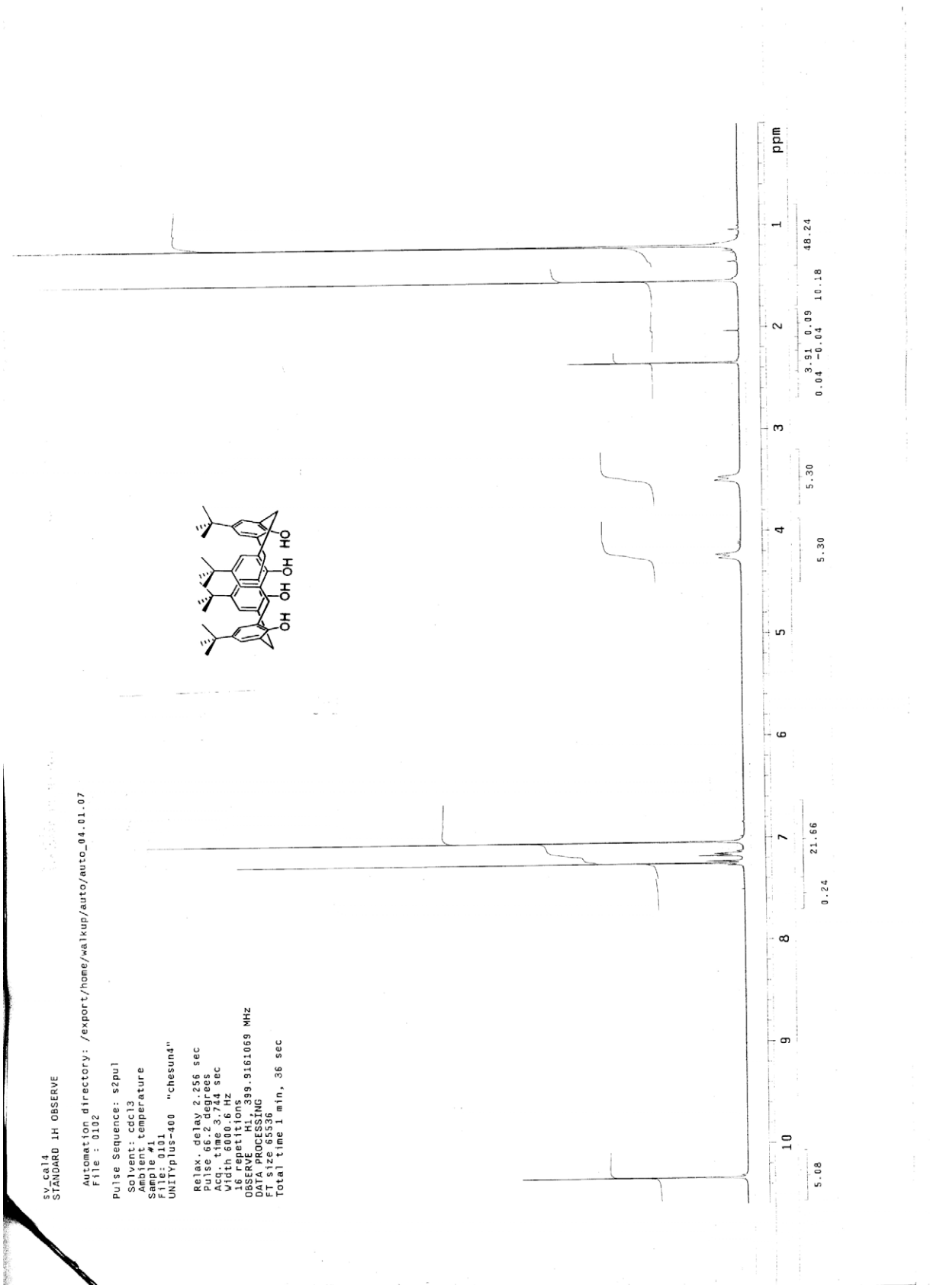


Figure 8: Overlay view of HPLC chromatograms of *para-tert*-butyl calix[5-9]arenes isolated by flash column chromatography from the reaction mixture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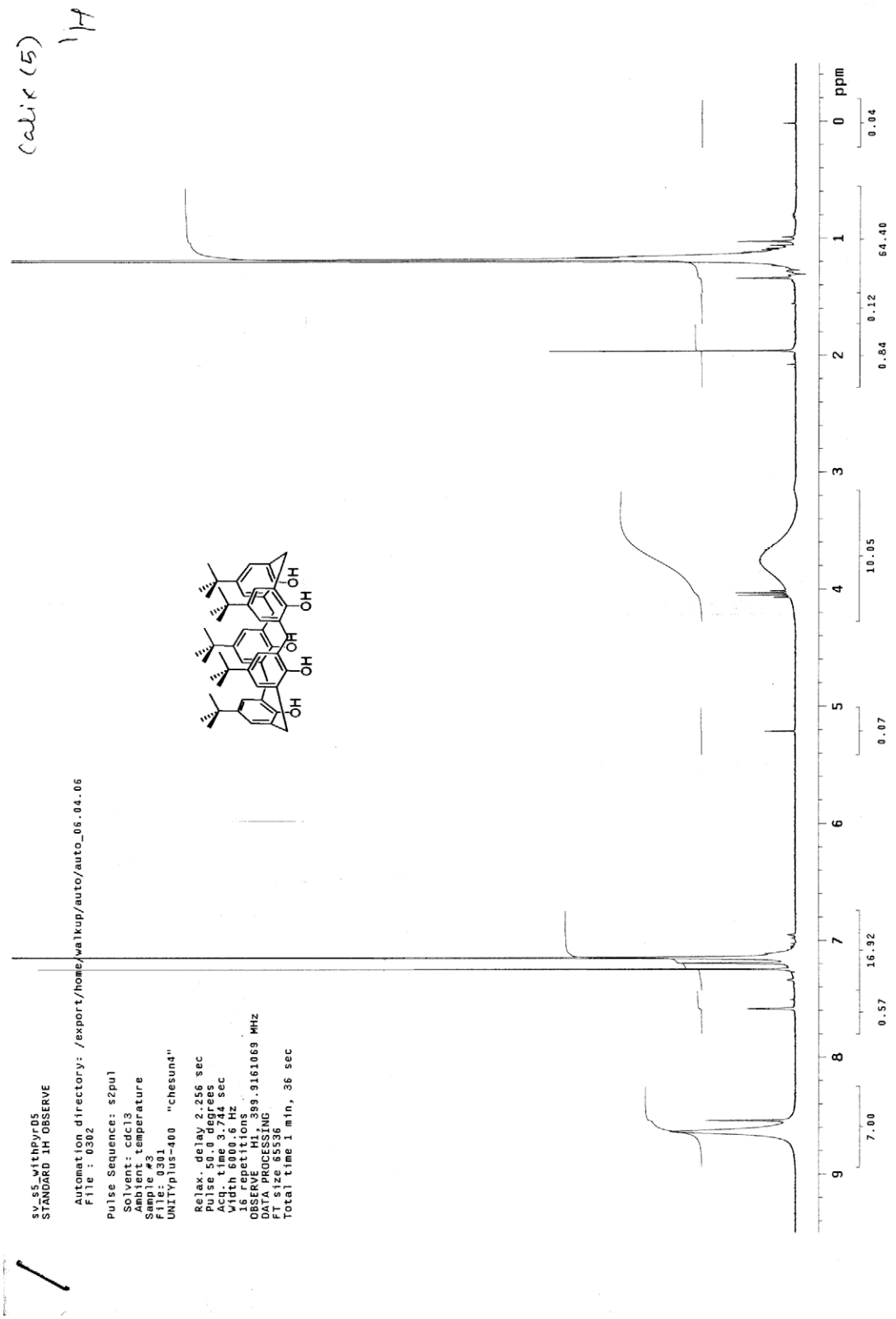
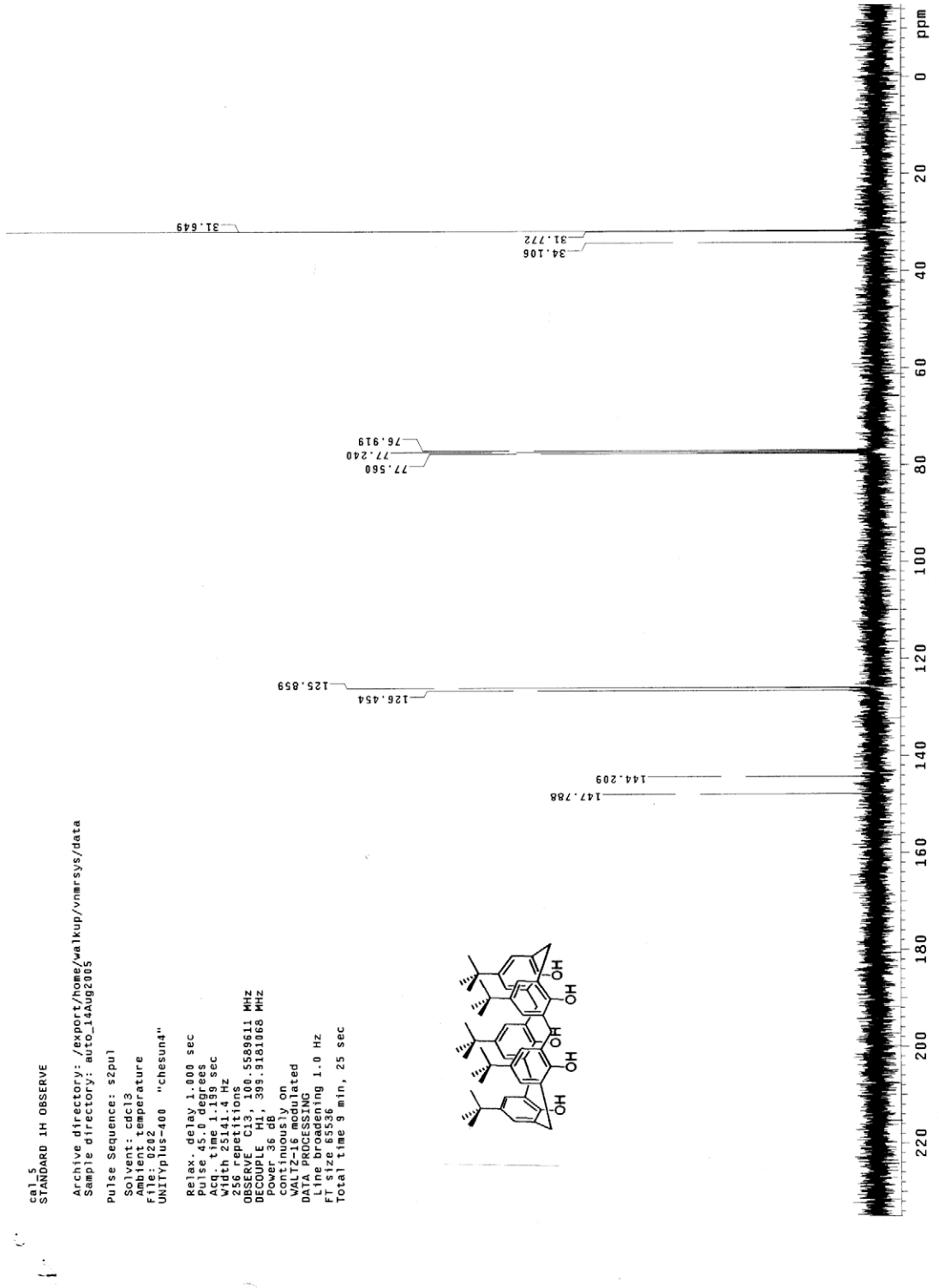
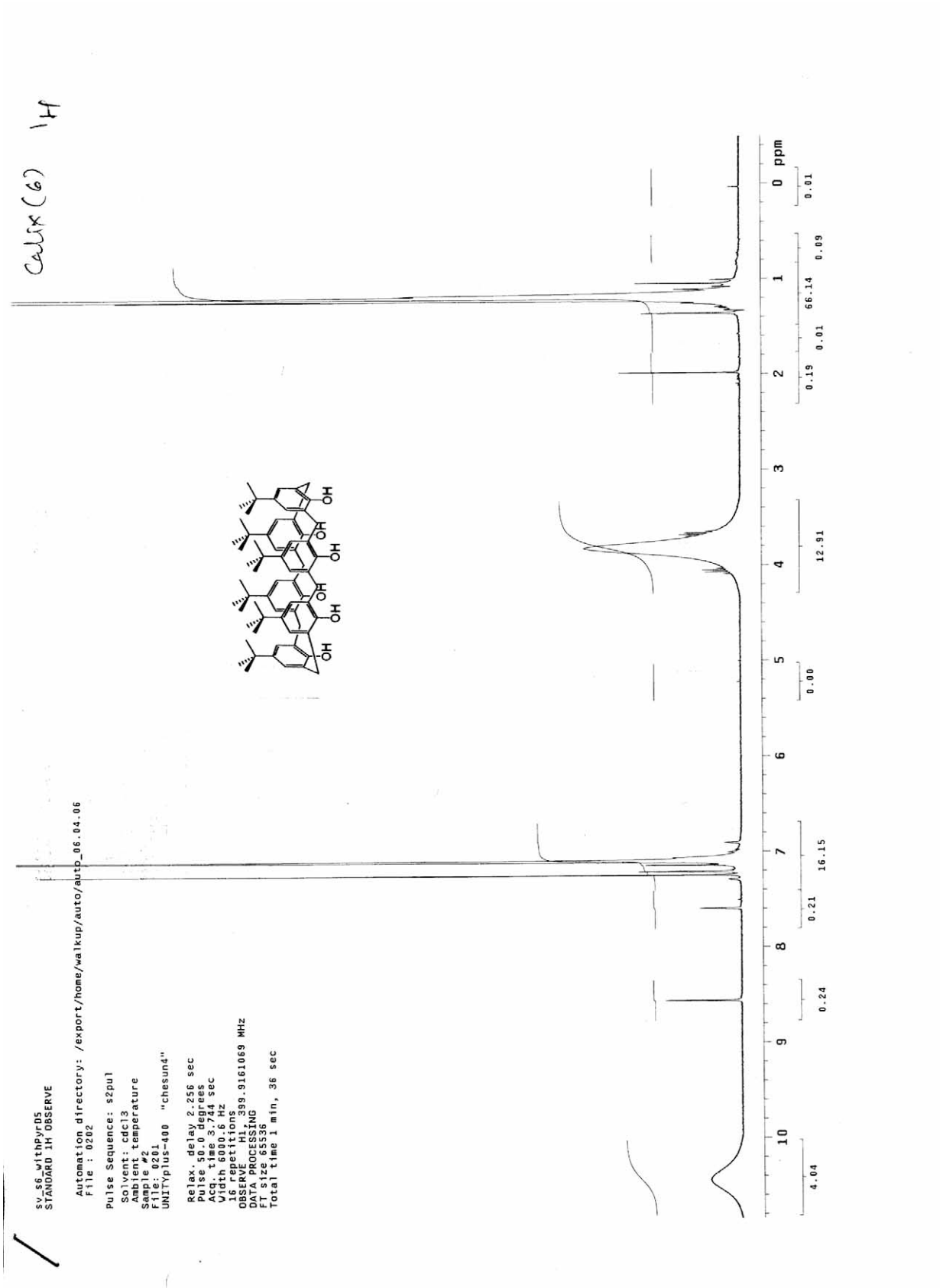
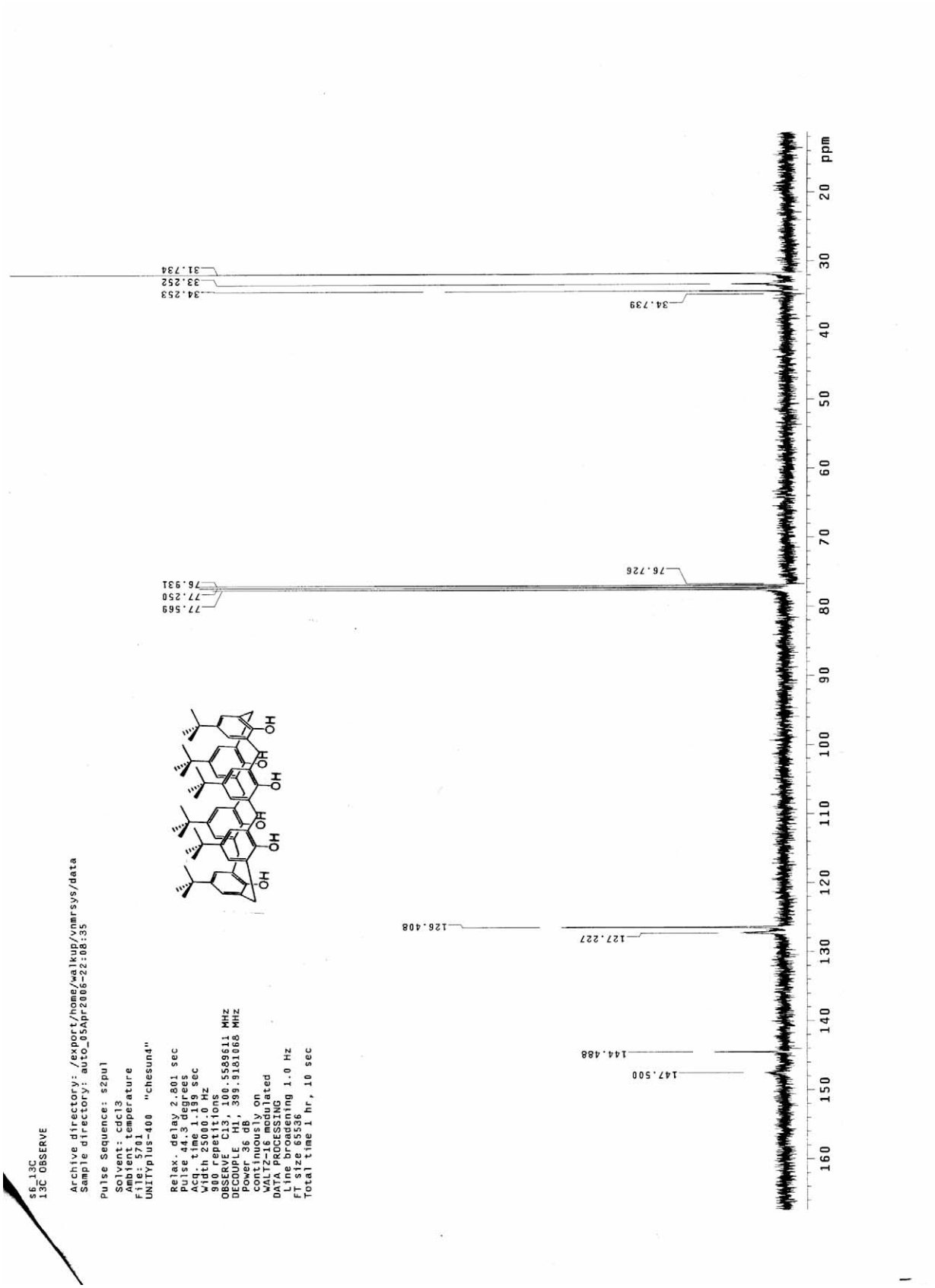
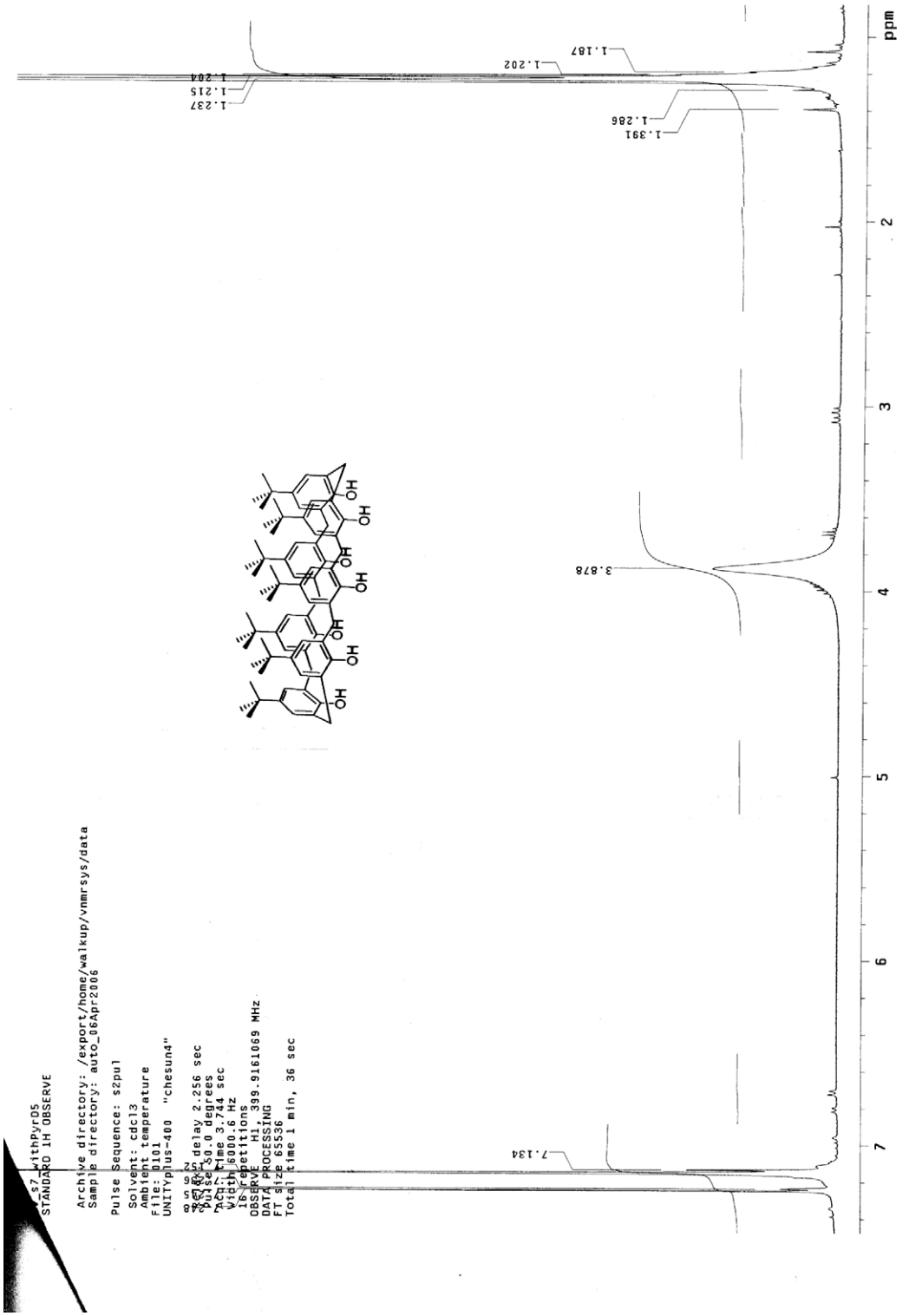
STANDARD 1H OBSERVE
Automation directory: /export/home/walkup/auto/auto_04.01.07
File : 0102
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Sample #1
File 101
UNITYplus-400 "chesun4"
Relax. delay 2.256 sec
Pulse 66.2 degrees
Acq. time 3.744 sec
Width 6000.6 Hz
16 repetitions
OBSERVE H1, 399.9161069 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 36 sec
OH
OH
OH
HO
ppm
5.08
0.24
21.66
5.30
5.30
3.91
0.04
0.09
-0.04
10.18
48.24

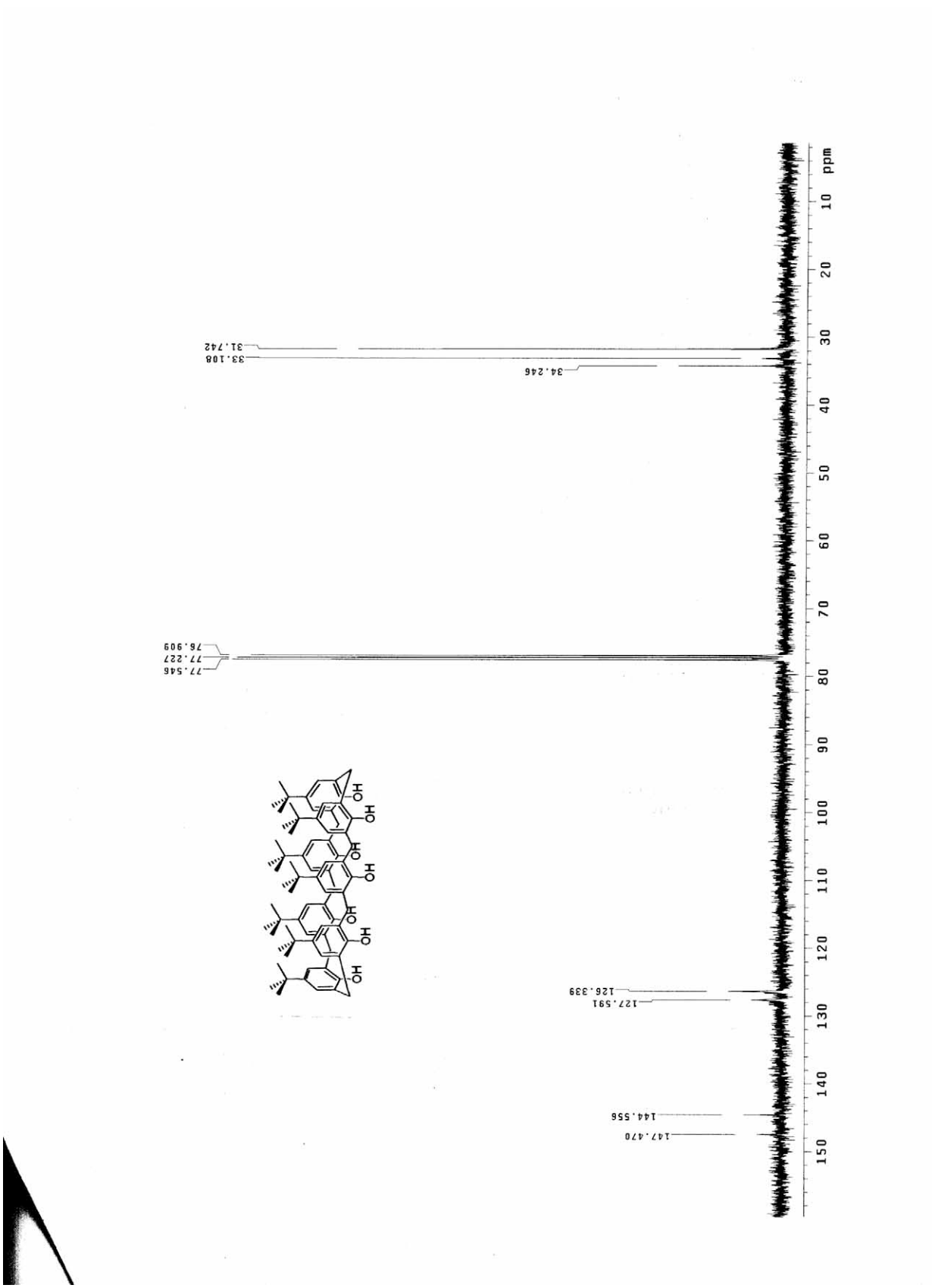

(calix (5)
1H
sv_65_withPyrDS
STANDARD 1H OBSERVE
Automation directory: /export/home/walkup/auto/auto_06.04.06
File : 0302
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Sample #3
File: 0301
UNITYplus-400 "chesun4"
Relax. delay 2.256 sec
Pulse 50.0 degrees
Acq. time 3.744 sec
Width 6000.6 Hz
16 repetitions
OBSERVE H1, 399.9161069 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 36 sec
OH
OH
OH
OH
OH
ppm
7.00
0.57
16.92
0.07
10.05
0.84
0.12
64.40
0.04


cal_5
STANDARD 1H OBSERVE
Archive directory: /export/home/walkup/vnmrsys/data
Sample directory: auto_14Aug2005
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
File: 0202
UNITYplus-400 "chesun4"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.199 sec
Width 25141.4 Hz
256 repetitions
OBSERVE C13, 100.5589611 MHz
DECOUPLE H1, 399.9181068 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 9 min, 25 sec
31.649
31.772
34.106
76.919
77.240
77.560
125.859
126.454
144.209
147.788
HO
HO
HO
HO
HO
ppm
0
20
40
60
80
100
120
140
160
180
200
220

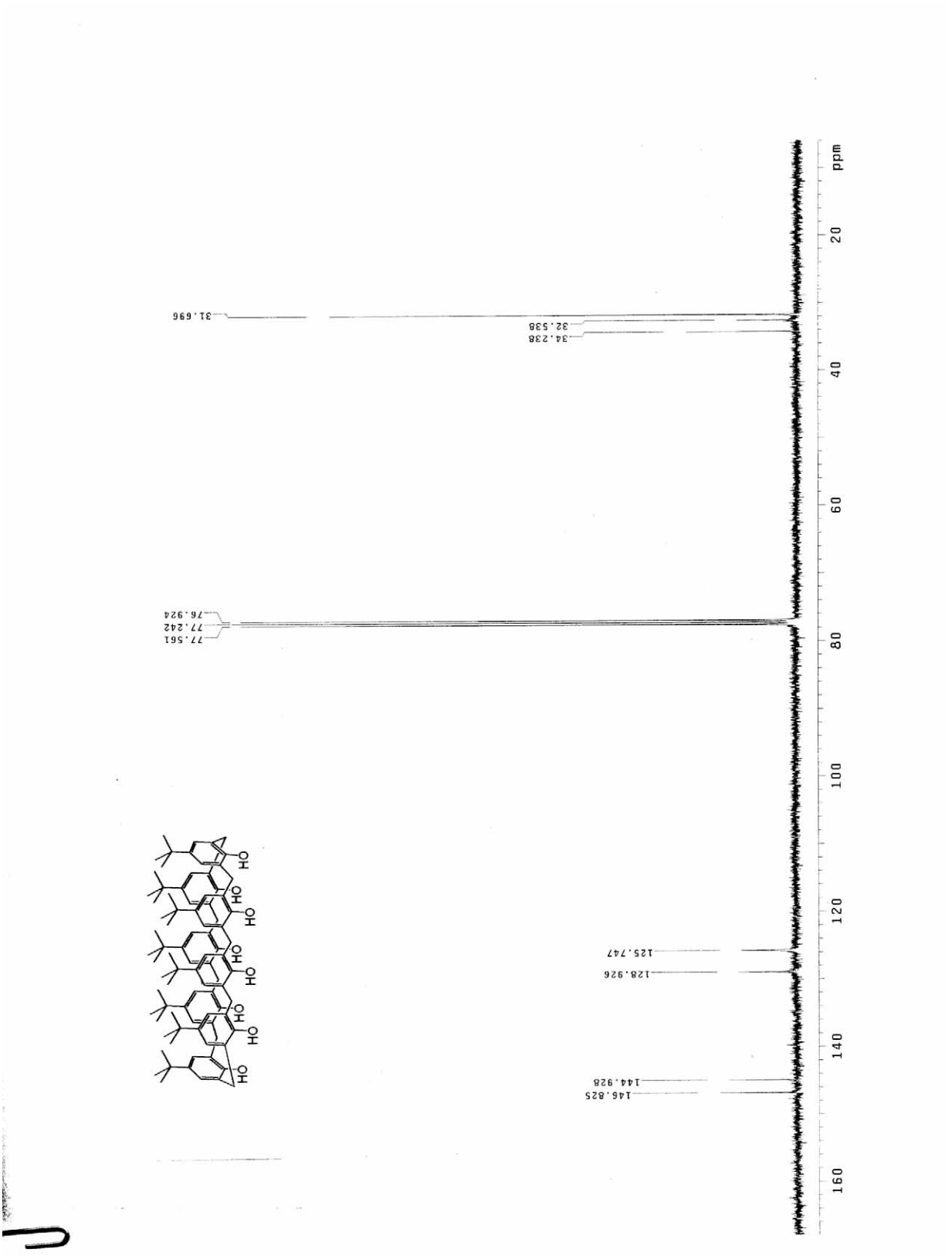

Calix (6) 1H
sv_s6_withPyrDS
STANDARD 1H OBSERVE
Automation directory: /export/home/walkup/auto/auto_06.04.06
File : 0202
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Sample #2
File: 0201
UNITYplus-400 "chesun4"
Relax. delay 2.256 sec
Pulse 50.0 degrees
Acq. time 3.744 sec
Width 6000.6 Hz
16 repetitions
OBSERVE H1, 399.9161069 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 36 sec
OH
10 9 8 7 6 5 4 3 2 1 0 ppm
4.04 0.24 0.21 16.15 0.00 12.91 0.19 0.01 66.14 0.09 0.01

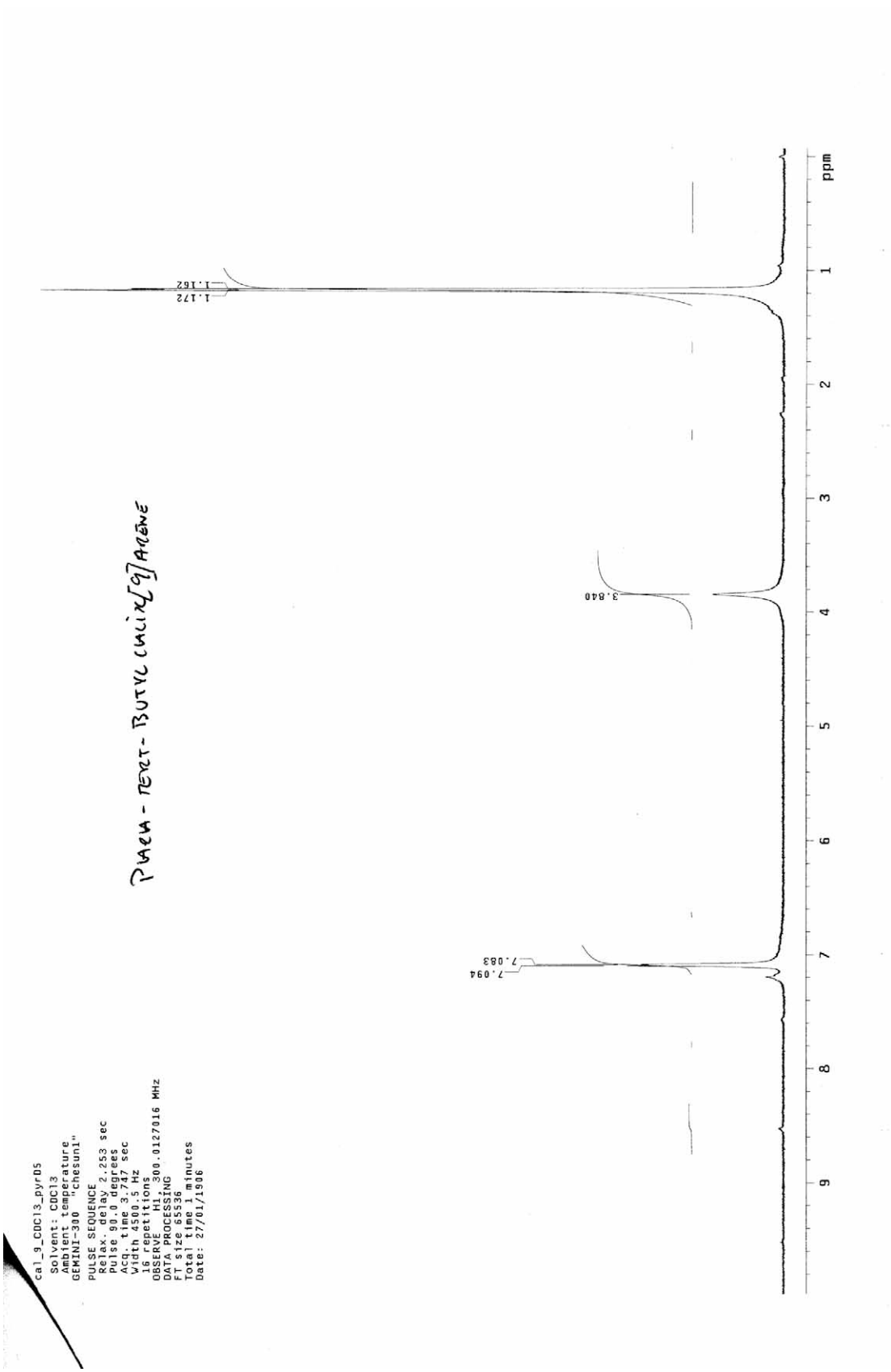

31.734
33.252
34.253
34.739
76.726
76.931
77.250
77.569
126.408
127.227
144.488
147.500
ppm
HO
OH

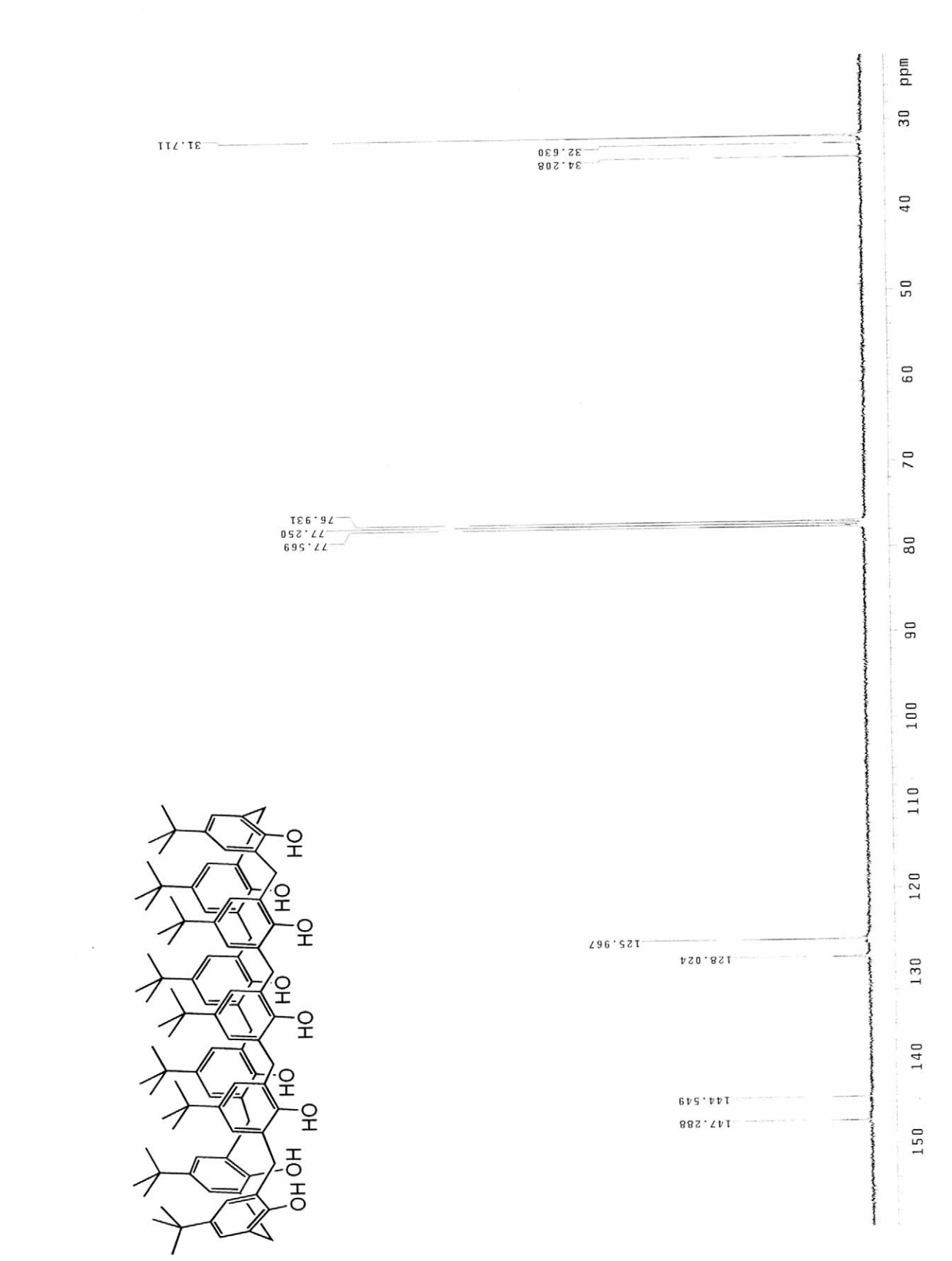

1.187
1.202
1.204
1.215
1.237
1.286
1.391
3.878
7.134
ppm
1
2
3
4
5
6
7
HO
HO
HO
HO
HO
HO
Archive directory: /export/home/walkup/vnmrsys/data
Sample directory: auto_06Apr2006
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
File: 0101
UNITYplus-400 "chesun4"
observe H1 399.9161069 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 36 sec


ppm
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
31.742
33.108
34.246
76.909
77.227
77.546
126.339
127.591
144.556
147.470
OH
OH
OH
OH
OH
OH


cal_8
STANDARD 1H OBSERVE
Archive directory: /export/home/walkup/vnmrsys/data
Sample directory: auto_14Aug2005
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
File: 550...
UNITYplus-400 "chesun4"
Relax. delay 2.256 sec
Pulse 50.0 degrees
Acq. time 3.744 sec
Width 6000.6 Hz
16 repetitions
OBSERVE H1, 399.9161069 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 36 sec
HO
1.243
1.550
3.474
3.472
3.506
4.341
4.373
7.169
7.249
9.623
0 ppm


31.696
32.538
34.238
76.924
77.242
77.561
125.747
128.926
144.928
146.825
ppm
20
40
60
80
100
120
140
160
HO


cal_9_CDCl3_pyr05
Solvent: CDCl3
Ambient temperature
GEMINI-300 "chesun1"
PULSE SEQUENCE
Relax. delay 2.253 sec
Pulse 90.0 degrees
Acq. time 3.747 sec
Width 4500.5 Hz
16 repetitions
OBSERVE H1, 300.0127016 MHz
DATA PROCESSING
FT size 65536
Total time 1 minutes
Date: 27/01/1906
PYRENE - TERT - BUTYL CHLORIDE
1.162
1.172
3.840
7.083
7.094
ppm


31.711
32.630
34.208
76.931
77.250
77.569
125.967
128.024
144.549
147.288
ppm
30
40
50
60
70
80
90
100
110
120
130
140
150
HO
OH