Supplementary Information

Carbo-cyclohexadienes *vs carbo*-benzenes: structure and conjugative properties

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1. General remarks.

THF and diethyl ether were dried and distilled over sodium/benzophenone, pentane and dichloromethane over P_2O_5. All other reagents were used as commercially available. In particular, commercial solutions of *n*-BuLi were 2.5 M in hexane, solutions of ethylmagnesium bromide were 3 M in THF, solutions of tetrabutylammonium fluoride were 1 M in THF, solutions of HCl were 2 M in diethylether. Previously described procedures were used for the preparation of **2**,[1] **6**,[2] **9**,[2] **10d**,[3] **10g**,[4] **11d**,[3] **11g**.[4] All reactions were carried out under nitrogen or Argon using Schlenk and vacuum line techniques. Column chromatographies were carried out on silica gel (60 P, 70-200 mm). Silica gel thin–layer chromatography plates (60F254, 0.25 mm) were revealed by treatment with an ethanolic solution of phosphomolybdic acid (20 %). The following analytical instruments were used. ^1H, ^{13}C and ^{19}F NMR: Bruker DPX 300, Avance 300, Avance 400 or Avance 500 spectrometers (most of the NMR spectra were recorded in $CDCl_3$ solutions; NMR chemical shifts δ are in ppm, with positive values to high frequency relative to the tetramethylsilane reference for ^1H and ^{13}C nuclei, and to CCl_3F for ^{19}F nuclei; coupling constants *J* are in Hz). Mass spectrometry: Quadrupolar Nermag R10-10H spectrometer. UV: spectrometer Perkin-Elmer UV-Vis Win-Lab Lambda 35.

2. Voltammetric measurements.

Voltammetric measurements were carried out with a potentiostat Autolab PGSTAT100 controlled by GPES 4.09 software. Experiments were performed at room temperature in a home-made airtight three-electrode cell connected to a vacuum/argon line. The reference electrode consisted of a saturated calomel electrode (SCE) separated from the solution by a

bridge compartment. The counter electrode was a platinum wire of *ca* 1 cm^2 apparent surface. The working electrode was a Pt microdisk (0.5 mm diameter). The supporting electrolyte [*n*-Bu_4N][PF_6] was used as received (Fluka, 99% electrochemical grade) and simply degassed under argon. Dichloromethane was freshly distilled prior to use. The solutions used in the electrochemical studies were typically 10^{-3} M in *carbo*-cyclohexadiene and 0.1 M in supporting electrolyte. Before each measurement, the solutions were degassed by bubbling argon, and the working electrode was polished with a polishing machine (Presi P230). Typical instrumental parameters for recorded square-wave voltammograms were: SW frequency f = 20 Hz, SW amplitude Esw = 20 mV, and scan increment dE = 0.5 mV.

3. Crystal structure determination of 12b, 12c and 12d.

The X-ray data for compounds **12b**, **12c** and **12d** were collected at low temperature (105 K for **12b** and 193 K for **12c** and **12d**) on a GEMINI diffractometer (Oxford Diffraction) using the CuKα radiation (wavelength = 1.54180 Å) for **12b**, and on a Bruker-AXS APEX II Quazar diffractometer (**12c** and **12d**) using a 30 W air-cooled microfocus source with focusing multilayer optics MoKα radiation (wavelength = 0.71073 Å). Phi- and omega-scans were used. The structure of **12b** was solved by direct methods using SUPERFLIP,[5] and refined by full-matrix least-squares procedures using the programs of CRYSTALS.[6] Atomic scattering factors were taken from the International tables for X-ray Crystallography.[7] Absorption corrections were introduced using the program MULTISCAN.[8] The data of compounds **12c** and **12d** were integrated with SAINT,[9] and an empirical absorption correction with SADABS[10] was applied. The structures were solved by direct methods, using SHELXS-97 and refined using the least–squares method on F^2.[11] All non-hydrogen atoms of

12b, **12c** and **12d** were refined anisotropically and hydrogen atoms were refined using a riding model. CCDC- 1003439 (**12b**), CCDC-951896 (**12c**) and CCDC-951897 (**12d**) contain the supplementary crystallographic data. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif (or from the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or deposit@ccdc.cam.ac.uk).

Crystal data.

• **12b.** $C_{48}H_{30}F_6O_4$, M = 784.75, Monoclinic, space group *C2/c*, a = 13.07527(8) Å, b = 16.18867(10) Å, c = 19.01027(12) Å, β = 100.1002(6)°, V = 3961.57(4) Å^3, Z = 4, crystal 0.15 x 0.15 x 0.25 mm^3, 35692 reflections collected (3771 independent, R*int* = 0.0213), 262 parameters, R_1 [I>2σ(I)] = 0.049, *wR*2 [all data] = 0.065, largest diff. peak and hole: 0.61 and -0.21 e.Å^{-3}.

• **12c.** $C_{48}H_{24}F_{12}O_2$, $CHCl_3$, M = 980.04, Monoclinic, space group $P2_1/c$, a = 12.8182(16) Å, b = 35.815(5) Å, c = 9.7626(14) Å, β = 92.841(6)°, V = 4476.4(11) Å^3, Z = 4, crystal 0.20 x 0.20 x 0.04 mm^3, 59064 reflections collected (7551 independent, R*int* = 0.1994), 653 parameters, 102 restraints, R_1 [I>2σ(I)] = 0.0829, *wR*2 [all data] = 0.2414, largest diff. peak and hole: 0.573 and -0.326 e.Å^{-3}.

• **12d.** $C_{46}H_{26}F_6O_2$, CH_2Cl_2, M = 809.59, Triclinic, $P\bar{1}$, a = 12.5119(17) Å, b = 12.6648(17) Å, c = 14.9565(19) Å, α = 70.805(5)°, β = 66.621(5)°, γ = 67.665(5)°, V = 1967.5(5) Å^3, Z = 2, crystal 0.20 x 0.10 x 0.04 mm^3, 28934 reflections collected (7350 independent, R*int* = 0.0906), 669 parameters, 489 restraints, R_1 [I>2σ(I)] = 0.0672, *wR*2 [all data] = 0.1983, largest diff. peak and hole: 0.212 and -0.314 e.Å^{-3}.

4. Synthesis procedures and characterization of all new compounds

[3,6-dimethoxy-3,6-bis(trifluoromethyl)-8-[tris(propan-2-yl)silyl]octa-1,4,7-triyn-1-yl] tris (propan-2-yl)silane (3). To a solution of the triyne **2** (2.53 g, 4.34 mmol) in dry THF (40 mL) under stirring at -78 °C were added 3.82 mL (9.55 mmol) of *n*-BuLi. The stirring was maintained 15 min. at -78 °C and then 15 min. at room temperature, before cooling again at -78 °C and adding methyl triflate (1.08 mL, 9.55 mmol). The temperature was allowed to warm slowly up to room temperature and the resulting mixture was stirred overnight, before treatment with saturated aqueous NH_4Cl. The aqueous layer was extracted with diethylether and the combined organic layers were washed with brine, dried with $MgSO_4$ and evaporated under reduced pressure. The residue was purified by silicagel chromatography (pentane/DCM 9:1) to give **3** as a pale yellow oil in 89 % yield (2.35 g).

δ_H (CD_3-C(O)-CD_3) 1.11-1.14 (42 H, m, Si-C*H*-C*H*$_3$), 3.64 (6 H, s, O-C*H*$_3$). δ_F (CD_3-C(O)-CD_3) - 80.24, - 80.27 (2 s, C*F*$_3$). $\delta_{C\{H\}}$ (CD_3-C(O)-CD_3) 10.74 (s, *C*H-CH$_3$), 17.87 (s, CH-*C*H$_3$), 53.34 (s, O-*C*H$_3$), 70.77 (q, $^2J_{CF}$ 35 Hz, *C*-CF$_3$), 78.78 (s, C-*C*≡*C*-C), 92.72 (s, *C*≡C-Si), 95.38 (s, ≡*C*-Si), 121.50 (q, $^1J_{CF}$ 283 Hz, *C*F$_3$). MS (DCI/NH_3): *m/z* 628.3 (M - NH_4). HRMS (DCI/CH_4): *m/z* calcd for $C_{29}H_{45}OF_6Si_2$: 579.2913, found: 579.2928.

3,6-dimethoxy-3,6-bis(trifluoromethyl)octa-1,4,7-triyne (**1**). To a solution of the triyne **3** (1.80 g, 2.95 mmol) in THF (40 mL) under stirring at – 78 °C was added a TBAF solution (7.38 mL, 7.38 mmol). The stirring was maintained at the same temperature during 1.5 h before treatment with water. The organic layer was extracted with diethylether, the combined organic layers were washed with brine, dried over $MgSO_4$ and evaporated under reduced pressure. The residue was purified by silicagel chromatography (DCM/pentane 5:95) to give the bis-terminal triyne **1** as a quite volatile colorless oil which crystallized at low temperature

in 75 % yield (0.660 g).

δ_H ($CDCl_3$) 2.78 (2 H, s, ≡C-*H*), 3.61 (6 H, s, O-C*H*$_3$). δ_F ($CDCl_3$) -79.52, -79.53 (2s, C*F*$_3$). $\delta_{C\{H\}}$ ($CDCl_3$) 53.97 (s, O-*C*H$_3$), 70.54 (q, $^2J_{CF}$ 36 Hz, *C*-CF$_3$), 73.09, 78.43 (2s, C(CF$_3$)-*C*≡), 77.16 (s, ≡*C*-H), 121.07 (q, $^1J_{CF}$ 285 Hz, *C*F$_3$). MS (DCI/CH_4): *m/z* 299.0 (M+H). HRMS (DCI/CH_4): *m/z* calcd for $C_{12}H_9O_2F_6$: 299.0507, found: 299.0493.

4,7-dimethoxy-4,7-bis(trifluoromethyl)deca-2,5,8-triyne-1,10-diol (4a). To a solution of the triyne **1** (0.720 g, 2.41 mmol) in THF (20 mL) was added at -78 °C *n*-BuLi (2.2 mL, 5.50 mmol). The resulting mixture was stirred 10 min at – 78 °C and 1 h at room temperature. This solution was added to a suspension of *p*-formaldehyde (0.220 g, 7.3 mmol) in THF (5 mL) under stirring at – 78 °C. The reaction mixture was allowed to warm slowly up to room temperature and the stirring was maintained overnight before treatment with saturated aqueous NH_4Cl. The aqueous layer was extracted with diethylether, the combined organic layers were washed with brine, dried over $MgSO_4$ and evaporated under vacuum. The residue was purified by silicagel chromatography (acetone/pentane 2:8) to finally give the diol **4a** in 60 % yield (0.520 g) as a brown oil.

δ_H ($CDCl_3$, 400 MHz) 3.04 (2 H, br s, O*H*), 3.58 (6 H, s, O-C*H*$_3$), 4.40 (4 H, s, C*H*$_2$-OH). ($CDCl_3$, 282 MHz) δ_F -79.34 (s, C*F*$_3$). $\delta_{C\{H\}}$($CDCl_3$, 100 MHz) 50.44 (s, *C*H$_2$-OH), 53.90 (s, O-*C*H$_3$), 70.71 (q, $^2J_{CF}$ 36 Hz, *C*-CF$_3$), 74.78, 78.56 (2s, C(CF$_3$)-*C*≡), 87.20 (s, ≡*C*-CH$_2$OH), 121.18 (q, $^1J_{CF}$ 284 Hz, *C*F$_3$). MS (DCI/NH_3) 376.0 (M+NH_4). HRMS (DCI/CH_4): *m/z* calcd for $C_{14}H_{11}O_3F_6$ $[M\text{-}H_2O+H]^+$ 341.0612, found: 341.0610.

4,7-dimethoxy-1,10-bis(4-methoxyphenyl)-4,7-bis(trifluoromethyl)deca-2,5,8-triyne-1,10-

diol (4b). To a solution of the triyne **1** (0.63 g, 2.11 mmol) in dry THF (12 mL) under stirring at – 78 °C was added *n*-BuLi (1.90 mL, 4.75 mmol). The resulting mixture was stirred during 20 min. at – 78 °C and 50 min. at room temperature, before cooling again at – 78 °C and adding a solution of the *p*-anisaldehyde (0.64 mL, 5.27 mmol) in THF (2 mL). The reaction mixture was allowed to warm slowly up to room temperature and the stirring was maintained overnight. After treatment with saturated aqueous NH_4Cl and extractions of the aqueous layer with diethylether, the combined organic layers were washed with brine, dried over $MgSO_4$ and evaporated to dryness. The residue was purified by flash chromatography (diethylether/pentane 4:6 then 1:1) to finally give **4b** in 71 % yield (0.856 g) as pale yellow oil.

δ_H ($CDCl_3$, 300 MHz) 2.48 (2 H, br s, O*H*), 3.57 (6 H, s, O-CH_3), 3.80 (6 H, s, C_6H_4-OCH_3), 5.48 (2 H, s, C*H*-OH), 6.90 (4 H, d, $^3J_{HH}$ 8.6 Hz, *m*-C_6H_4-OCH_3), 7.42 (4 H, d, $^3J_{HH}$ 8.6 Hz, *o*-C_6H_5-OCH_3). δ_F ($CDCl_3$, 282 MHz) □79.21 (CF_3). $\delta_{C\{H\}}$($CDCl_3$, 63 MHz) 53.97 (C_6H_4-O*C*H_3), 55.24 (O-*C*H_3), 63.79 (*C*H-OH), 70.81 (q, $^2J_{CF}$ 36 Hz, *C*-CF_3), 75.66, 78.77, 89.07 (*C*≡*C*), 114.09 (*m*-*C*$_6H_4$-OCH_3), 121.21 (q, $^1J_{CF}$ 285 Hz, *C*F_3), 128.05 (*o*-*C*$_6H_4$-OCH_3), 131.35 (*i*-*C*$_6H_4$-OCH_3), 159.87 (*p*-*C*$_6H_4$-OCH_3). MS (DCI/CH_4) *m/z* 553.1 (M-OH). HRMS (DCI/CH_4): *m/z* calcd for $C_{28}H_{24}O_5F_6$ (M-OH): 553.1450, found: 553.1423. FT-IR: ν 3382 (OH), 2853-2959 (Csp2-H), 2237 (C≡C), 1611 (C=C), 1444-1463 (C=C), 1251 (C-OMe).

4,7-dimethoxy-1,10-diphenyl-4,7-bis(trifluoromethyl)deca-2,5,8-triyne-1,10-diol (4c). To a solution of the triyne **1** (0.200 g, 0.67 mmol) in THF (5 mL) under stirring at -78 °C was added *n*-BuLi (0.55 mL, 1.37 mmol). The resulting mixture was stirred 30 min at -78 °C, and 30 min at room temperature, before adding benzaldehyde (0.14 mL, 1.38 mmol) at -78 °C.

The temperature was allowed to warm slowly up to room temperature and the stirring was maintained overnight. After treatment with saturated aqueous NH_4Cl, the aqueous layer was extracted with diethylether and the combined organic layers were washed with brine, dried over $MgSO_4$ and evaporated under reduced pressure. The residue was purified by silicagel chromatography (pentane/acetone 9:1 to 6:4) to give the diol **4c** as a yellow oil in 93 % yield (0.32 g).

δ_H ($CDCl_3$, 400 MHz) 2.37 (2 H, br d, $^3J_{HH}$ 6.5 Hz, O*H*), 3.59 (6 H, s, O-CH_3), 5.57 (2 H, d, $^3J_{HH}$ 6.5 Hz, C*H*-OH), 7.36-7.44 (6 H, m, *m*-, *p*-C_6H_5), 7.54 (4 H, d, $^3J_{HH}$ 7.2 Hz, *o*-C_6H_5). δ_F ($CDCl_3$, 376 MHz) -79.2 (CF_3). $\delta_{C\{H\}}$ ($CDCl_3$, 100 MHz) 54.07 (O-*C*H_3), 64.38 (*C*H-OH), 70.88 (q, $^2J_{CF}$ 36 Hz, *C*-CF_3), 76.08, 78.79, 88.72 (*C*≡*C*), 121.22 (q, $^1J_{CF}$ 285 Hz, *C*F_3), 126.60, 126.63, 128.86 (*o*-, *m*-C_6H_5), 128.92 (*p*-C_6H_5), 139.03 (*i*-C_6H_5).

4,7-dimethoxy-4,7-bis(trifluoromethyl)deca-2,5,8-triynedial (5a). To a solution of the diol **4a** (0.300 g, 0.84 mmol) in 1,2-DCE (25 mL) was added IBX (1.17 g, 4.2 mmol). The resulting mixture was refluxed during 6 h and then cooled at 0 °C before filtration over celite. Evaporation of the filtrate gave the dialdehyde **5a** in 93 % yield (0.275 g) as a pale orange-brown oil.

δ_H ($CDCl_3$, 400 MHz) 3.64 (6 H, s, O-CH_3), 9.33 (2 H, s, C*H*O). δ_F ($CDCl_3$, 376 MHz) -78.46 (CF_3). $\delta_{C\{H\}}$ ($CDCl_3$, 100 MHz) 54.72 (s, OCH_3), 70.95 (q, $^2J_{CF}$ 36 Hz, *C*-CF_3), 78.31, 81.33, 84.40 (*C*≡*C*), 120.65 (q, $^1J_{CF}$ 286 Hz, *C*F_3), 174.66 (*C*HO). MS (DCI/CH_4) *m/z* 355.0 (M+H). HRMS (DCI/CH_4): *m/z* calcd for $C_{14}H_9O_4F_6$ (M+H): 355.0405, found: 355.0406.

4,7-dimethoxy-1,10-bis(4-methoxyphenyl)-4,7-bis(trifluoromethyl)deca-2,5,8-triyne-1,10-

dione (5b). To a solution of the diol **4b** (0.66 g, 1.16 mmol) in dry DCM (90 mL), was added MnO_2 (1.01 g, 11.6 mmol) at room temperature. The resulting mixture was stirred 3h and then filtered through celite. The filtrate was evaporated to dryness. The residue was purified by flash chromatography (diethylether/pentane 3:7 then 4:6) to finally give **5b** in 84 % yield (0.550 g) as a pale yellow oil.

δ_H ($CDCl_3$, 300 MHz) 3.72 (6 H, s, O-CH_3), 3.91 (6 H, s, C_6H_4-OCH_3), 6.99 (4 H, d, $^3J_{HH}$ 8.7 Hz, *m*-C_6H_4-OCH_3), 8.07 (4 H, d, $^3J_{HH}$ 8.7 Hz, *o*-C_6H_4-OCH_3). δ_F ($CDCl_3$, 282 MHz) □78.56 (CF_3). $\delta_{C\{H\}}$ ($CDCl_3$, 75 MHz) 54.72 (C_6H_4-OCH_3), 55.66 (O-CH_3), 71.04 (q, $^2J_{CF}$ 36 Hz, *C*-CF_3), 78.63, 79.14 (≡*C*-C-*C*≡), 84.25 (≡*C*-C=O), 114.26 (*m*-C_6H_4-OCH_3), 120.95 (q, $^1J_{CF}$ 286 Hz, *C*F_3), 129.28 (*o*-C_6H_4-OCH_3), 132.11 (*i*-C_6H_4-OCH_3), 165.23 (*p*-C_6H_4-OCH_3), 174.32 (*C*=O). MS (DCI/CH_4) *m/z* 567.1 (M+H). HRMS (DCI/CH_4) *m/z* calcd for $C_{28}H_{21}O_6F_6$ (M+H): 567.1242, found: 567.1218. IR: ν 2852-2923 (Csp2-H), 1649 (C=O), 1597 (C=C), 1259 (C-OMe).

4,7-dimethoxy-1,10-diphenyl-4,7-bis(trifluoromethyl)deca-2,5,8-triyne-1,10-dione (5c). To a solution of the diol **4c** (0.320 g, 0.63 mmol) in dichloroethane (40 mL) was added IBX (0.882 g, 3.15 mmol). The resulting mixture was stirred under reflux during 24 h and then cooled into an ice bath before filtration through celite. After evaporation of the solvent under reduced pressure, the residue was purified by silicagel chromatography (pentane/diethylether 8:2) to give the expected diketone **5c** as a pale solid in 94 % yield (0.30 g).

δ_H ($CDCl_3$, 400 MHz) 3.73 (6 H, s, O-CH_3), 7.52-7.56 (4 H, m, *m*-C_6H_5), 7.67-7.71 (2 H, m, *p*-C_6H_5), 8.11 (4 H, d, $^3J_{HH}$ 7.5 Hz, *o*-C_6H_5). δ_F ($CDCl_3$, 376 MHz) -78.48, -78.49 (CF_3). $\delta_{C\{H\}}$ ($CDCl_3$, 100 MHz) 54.81 (O-CH_3), 71.11 (q, $^2J_{CF}$ 36 Hz, *C*-CF_3), 78.63, 79.87, 84.03 (*C*≡*C*),

120.96 (q, $^{1}J_{CF}$ 286 Hz, *C*F_3), 128.99, 129.66 (*o*-, *m*-C_6H_5), 135.11 (*p*-C_6H_5), 135.87 (*i*-C_6H_5), 175.95 (*C*=O). MS (DCI/CH_4) *m/z* 507.1 (M+H). HRMS (DCI/CH_4) *m/z* calcd for $C_{26}H_{17}O_4F_6$ (M+H): 507.1031, found: 507.1017.

4,7,13,16-tetramethoxy-1,10-bis(4-methoxyphenyl)-4,7-diphenyl-13,16-bis (trifluoromethyl)cyclooctadeca-2,5,8,11,14,17-hexayne-1,10-diol (7b). To a solution of triyne **6** (0.139 g, 0.442 mmol) in THF (30 mL) was added *n*-BuLi (0.35 mL, 0.875 mmol) at – 78 °C. The mixture was stirred 20 min at – 78 °C and 30 min at room temperature (solution A). During that time, the triynedione **5b** (0.250 g, 0.442 mmol) was dissolved in THF (30 mL, solution B). The solutions A and B were simultaneously transferred into a 250 mL flask containing 90 mL THF under stirring at – 78 °C. The resulting mixture was allowed to warm slowly up to room temperature and stirring was continued for 15 h. After treatment with saturated aqueous NH_4Cl and extraction with Et_2O, the combined organic layers were washed with brine, dried over $MgSO_4$ and concentrated under reduced pressure. The residue was purified by flash chromatography (acetone/DCM/pentane 1:1:8 then 2:2:6) to finally give **7b** in 40 % yield (0.158 g) as pale brown oil.

δ_H ($CDCl_3$, 300 MHz) 3.39-3.69 (12 H, m, O-CH_3), 3.80-3.86 (6 H, m, C_6H_4-OCH_3), 6.82-7.00 (4 H, m, *m*-C_6H_4-OCH_3), 7.33-7.50 (6 H, m, *m*-, *p*-C_6H_5), 7.58-7.83 (8 H, m, *o*-C_6H_5, *o*-C_6H_4-OCH_3). δ_F ($CDCl_3$, 282 MHz) □79.13-(-78.91) (m, CF_3). $\delta_{C\{H\}}$ ($CDCl_3$, 75 MHz) 53.26-53.44, 53.82-54.21, 55.34-55.38 (m, O-*C*H_3), 64.49-64.71 (m, *C*-OCH_3), 70.77 (q, $^{2}J_{CF}$ 36.0 Hz, *C*-CF_3), 71.81-71.94 (m, *C*-OH), 74.61-74.82, 78.65-78.86, 82.95-83.30, 84.42-84.56, 85.90-86.32, 88.16-88.95 (m, *C*≡*C*), 114.01-114.05 (m, *m*-C_6H_4-OCH_3), 121.09 (q, $^{1}J_{CF}$ 285 Hz, *C*F_3), 126.27-126.50 (m, *m*-C_6H_5), 127.05-127.18 (m, *o*-C_6H_4-OCH_3), 128.44-128.55 (m,

o-C_6H_5), 129.28 (m, *o*-C_6H_4-OCH_3), 128.98-129.22 (m, *p*-C_6H_5), 132.01-132.24 (m, *i*-C_6H_4-OCH_3), 139.04-139.30 (m, *i*-C_6H_5), 160.16-160.28 (m, *p*-C_6H_4-OCH_3). MS (DCI/CH_4) *m/z* 881.2 (M+H). HRMS (DCI/CH_4) *m/z* calcd for $C_{50}H_{39}O_8F_6$ (M+H): 881.2549, found: 881.2552.

4,7,13,16-tetramethoxy-1,4,7,10-tetraphenyl-13,16-bis(trifluoromethyl)cyclooctadeca-2,5,8,11,14,17-hexayne-1,10-diol (7c). To a solution of the triyne **6** (0.182 g, 0.581 mmol) in THF (40 mL) at -78 °C was added *n*-BuLi (0.49 mL, 1.22 mmol) and the resulting solution was stirred during 50 min. Then, this mixture and a solution of the diketone **5c** (0.300 g, 0.593 mmol) in THF (40 mL) were simultaneously transferred into a flask containing 120 mL of THF under stirring at -78 °C. The mixture was allowed to warm slowly up to room temperature and the stirring was maintained overnight before treatment with saturated aqueous NH_4Cl. The aqueous layer was extracted with diethylether and the combined organic layers were washed with brine, dried over $MgSO_4$ and evaporated under reduced pressure. The residue was purified by silicagel chromatography (diethylether/pentane 2:8) to give the expected pericyclyne **7c** as a white solid in 18 % yield (0.086 g).

δ_H ($CDCl_3$, 400 MHz) 3.38-3.64 (12 H, m, O-CH_3), 7.35-7.47 (12 H, m, *m*-, *p*-C_6H_5), 7.66-7.82 (8 H, m, *o*-C_6H_5). δ_F ($CDCl_3$, 376 MHz) □79.15-(□78.90) (m, CF_3). $\delta_{C\{H\}}$ ($CDCl_3$, 100 MHz) 53.26-54.43, 53.82-54.22 (m, O-CH_3), 64.81-65.04 (m, *C*-OCH_3), 70.77 (q, $^2J_{CF}$ 36 Hz, *C*-CF_3), 71.87-71.97 (m, *C*-OH), 74.80-75.04, 78.67-78.89, 83.25-83.55, 84.34-84.57, 85.76-86.21, 88.10-88.62 (m, *C*≡*C*), 114.01-114.05 (m, *m*-C_6H_4-OCH_3), 121.07 (q, $^1J_{CF}$ 285 Hz, *C*F_3), 125.55-125.68, 126.25-126.43, 128.45-128.59, 128.75-128.82 (4 m, *o*-C_6H_5, *m*-C_6H_5), 129.12-129.22, 129.31-129.35 (2 m, *p*-C_6H_5), 139.74-140.05 (2 m, *i*-C_6H_5). MS (DCI/CH_4)

m/z 789.2 (M-OMe), 803.2 (M-OH), 820.2 (M). HRMS (DCI/CH_4) *m/z* calcd for $C_{48}H_{33}O_5F_6$ (M-OH): 803.2232, found: 803.2209.

4,7,13,16-tetramethoxy-4,7-diphenyl-13,16-bis(trifluoromethyl)-1,10-bis[4-(trifluoromethyl)phenyl]cyclooctadeca-2,5,8,11,14,17-hexayne-1,10-diol (7d). To a solution of the triyne **1** (0.199 g, 0.668 mmol) in THF (45 mL) was added *n*-BuLi (0.53 mL, 1.33 mmol) at – 78 °C. The mixture was stirred for 15 min at – 78 °C and 75 min at room temperature (solution A). During that time, the triynedione **10d** (0.440 g, 0.668 mmol) was dissolved in THF (45 mL, solution B). The solutions A and B were simultaneously transferred into a 500 mL flask containing 140 mL THF under stirring at – 78 °C. The resulting mixture was allowed to warm slowly up to room temperature and stirring was continued for 17 h. After treatment with saturated aqueous NH_4Cl and extraction of the aqueous layer with Et_2O, the combined organic layers were washed with brine, dried over $MgSO_4$ and concentrated under reduced pressure. The residue was purified by flash chromatography (acetone/pentane 5:95, 1:9 then 2:8) to finally give **7d** in 21 % yield (0.133 g) as a white solid.

δ_H ($CDCl_3$, 300 MHz) 3.39-3.61 (12 H, m, O-CH_3), 7.61 (6 H, br s, *m*-, *p*-C_6H_5), 7.61-7.93 (12 H, m, *o*-, *m*-C_6H_4-CF_3, *o*-C_6H_5). δ_F ($CDCl_3$, 282 MHz) □79.07-(□78.90) (m, CF_3), □62.75 (s, C_6H_4-CF_3). $\delta_{C\{H\}}$ ($CDCl_3$, 75 MHz) 53.27, 53.35, 53.42, 54.08, 54.17 (O-CH_3), 64.23, 64.21, 64.40, 64.43 (*C*-OCH_3), 70.74 (q, $^2J_{CF}$ 36 Hz, *C*-CF_3), 71.74, 71.82, 71.88 (*C*-OH), 75.34-75.52, 78.63-78.72, 84.02-84.56, 85.01-85.36, 87.24-87.95 (m, *C*≡*C*), 121.02 (q, $^1J_{CF}$ 285 Hz, CF_3), 123.74 (q, $^1J_{CF}$ 272 Hz, C_6H_4-CF_3), 125.77-126.27 (m, *o*-, *m*-C_6H_4-CF_3, *o*-C_6H_5), 128.66 (*m*-C_6H_5), 129.32, 129.38 (*p*-C_6H_5), 130.26-132.21 (m, *p*-C_6H_4-CF_3) 138.67-

138.99 (m, *i*-C_6H_5) 143.50-143.76 (*i*-C_6H_4-CF_3). MS (MALDI-TOF/DCTB) *m/z* 979.3 (M+Na). HRMS (MALDI-TOF/DCTB) *m/z* calcd for $C_{50}H_{39}O_8F_6$ (M+Na): 979,1905 found: 979.1969.

1,10-bis[4-(9H-carbazol-9-yl)phenyl]-4,7,13,16-tetramethoxy-13,16-diphenyl-4,7-bis(trifluoromethyl)cyclooctadeca-2,5,8,11,14,17-hexayne-1,10-diol (7e). To a solution of HMDS (0.21 mL, 1 mmol) in THF (15 mL) under stirring at -78 °C was added *n*-BuLi (0.39 mL, 0.98 mmol). The reaction mixture was stirred 30 min at -78 °C before adding a solution of the triyne **1** (0.049 g, 0.164 mmol) in THF (3 mL). The reaction mixture was stirred 30 min at -78 °C and was diluted with THF to 50 mL (volume of r.m.). Then, the reaction mixture and a solution of **10e** (0.140 g, 0.164 mmol) in THF (50 mL) were transferred simultaneously into a 500 mL flask containing THF (300 mL) under stirring at -78˚C. The temperature was allowed to increase slowly up to room temperature and the reaction mixture was stirred during 16 h before treatment with saturated aqueous NH_4Cl. The aqueous layer was extracted with diethylether and the combined organic layers were washed with brine, dried over $MgSO_4$ and evaporated under reduced pressure. It was not possible to purify this crude mixture.

HRMS-control: HRMS (MALDI-DCTB) *m/z* calcd for $C_{72}H_{48}N_2O_6F_6$: 1150.3417, found: 1150.3440.

1,10-bis[4-(1H-indol-1-yl)phenyl]-4,7,13,16-tetramethoxy-4,7-diphenyl-13,16-bis(trifluoromethyl)cyclooctadeca-2,5,8,11,14,17-hexayne-1,10-diol (7f). To a solution of HMDS (0.3 mL, 1.4 mmol) in THF (15 mL) under stirring at -78 °C was added *n*-BuLi (0.54 mL, 1.35 mmol). The reaction mixture was stirred 30 minutes at -78 °C before adding a

solution of the triyne **1** (0.067 g, 0.226 mmol) in THF (3 mL). The reaction mixture was stirred 30 minutes at -78 °C and was diluted with THF to 50 mL (volume of r.m.). Then, this solution and a solution of the diketone **10f** (0.170 g, 0.226 mmol) in THF (50 mL) were transferred simultaneously into a 500 mL flask containing THF (300 mL) under stirring at -78˚C. The temperature was allowed to increase slowly up to room temperature and the reaction mixture was stirred at room temperature for 16 h before treatment with saturated aqueous NH_4Cl. The aqueous layer was extracted with diethylether and the combined organic layers were washed with brine, dried over $MgSO_4$ and evaporated under reduced pressure. The residue was purified by silica gel chromatography (Acetone/Pentane 2:8) to give the [6]pericyclynediol **7f** as a light yellow solid in 38 % yield (90 mg).

mp 84 °C. δ_H ($CDCl_3$, 400 MHz) 3.43-3.73 (14 H, m, O-C*H*$_3$, O*H*), 6.72 (2 H, m, *H*3-indole), 7.24-7.97 (28 H, m, all the rest). δ_F ($CDCl_3$, 376 MHz) □79.37-(-78.86) (m, C*F*$_3$). $\delta_{C\{H\}}$ ($CDCl_3$, 100 MHz) 53.25-54.30 (m, O-*C*H$_3$), 64.51-64.63 (m, *C*-OCH$_3$), 70.70 (q, $^2J_{CF}$ 36 Hz, *C*-CF$_3$), 71.95 (*C*-OH), 75.20, 78.87, 83.65, 83.71, 84.53, 85.81 88.15, 88.39 (m, -*C*≡*C*-), 104.36 (m, *C*3-indole), 110.42 (m, *C*6-indole), 120.70 (*o*-C_6H_4-N), 121.11 (q, $^1J_{CF}$ 282 Hz, *C*F$_3$) 121.29 (*C*2-indole), 122.66 (*C*9-indole), 124.11-124.23 (m, *C*7-, *C*8-indole), 126.35-126.47 (m, *o*-C_6H_5), 127.09, 127.23, 127.64, 128.67, 129.37, 130.62 (m, *m*-, *p*-C_6H_5, *m*-C_6H_4-N), 129.53 (*C*4-indole), 135.72 (*C*5-indole), 137.79 (m, *i*-C_6H_4-N), 139.12 (m, *i*-C_6H_5), 140.68 (*p*-C_6H_4-N). MS (MALDI-TOF/DCTB) *m/z* 1050.3 (M). HRMS (MALDI-DCTB) *m/z* calcd for $C_{64}H_{44}N_2O_6F_6$: 1050.3104, found: 1050.3180.

4,7,13,16-tetramethoxy-4,7-diphenyl-13,16-bis(trifluoromethyl)-1,10-bis({2-[tris(propan-2-yl)silyl]ethynyl})cyclooctadeca-2,5,8,11,14,17-hexayne-1,10-diol (7g). To a solution of

the triyne **1** (0.235 g, 0.789 mmol) in THF (52 mL) was added *n*-BuLi (0.63 mL, 1.58 mmol) at – 78 °C. The mixture was stirred for 15 min at – 78 °C and 75 min at room temperature (solution A). During that time, diketone **10g** (0.577 g, 0.789 mmol) was dissolved in THF (52 mL, solution B). The solutions A and B were simultaneously transferred into a 500 mL flask containing 160 mL THF under stirring at – 78 °C. The resulting solution was allowed to warm slowly up to room temperature and stirring was continued for 17 h. After treatment with saturated aqueous NH_4Cl and extraction with diethylether, the combined organic layers were washed with brine, dried over $MgSO_4$ and concentrated under reduced pressure. The residue was purified by flash chromatography (acetone/pentane 5:95, 1:9 then 2:8) to finally give **7g** in 21 % yield (0.175 g) as a white solid.

δ_H ($CDCl_3$, 300 MHz) 1.10-1.14 (42 H, s, Si-C*H*-CH_3), 3.49-3.68 (12 H, m, O-CH_3), 7.39 (6 H, br s, *m*-, *p*-C$_6H_5$), 7.73-7.80 (4 H, m, *o*-C$_6H_5$). δ_F ($CDCl_3$, 282 MHz) -79.24-(-78.89) (m, CF_3). $\delta_{C\{H\}}$ ($CDCl_3$, 75 MHz) 11.01 (s, Si-*C*H-CH$_3$), 18.42 (s, Si-CH-*C*H$_3$), 53.21-53.56, 53.94-54.18 (m, O-*C*H$_3$), 70.71 (q, $^2J_{CF}$ 36 Hz, *C*-CF$_3$), 71.74-71.87, 72.25-72.55 (m, *C*-OH, *C*(OCH$_3$)(C$_6$H$_5$)), 78.36-78.67, 80.43-80.83, 83.18-83.59, 84.14-84.29, 85.51-86.22, 87.17-87.48, 101.53-101.91 (*C*≡*C*), 121.00 (q, $^1J_{CF}$ 283 Hz, *C*F$_3$), 126.47-126.56, 128.58 (*o*-, *m*-*C*$_6$H$_5$), 129.18-129.24 (m, *p*-*C*$_6$H$_5$), 138.97-139.22 (m, *i*-*C*$_6$H$_5$). MS (MALDI-TOF/DCTB) *m/z* 1051.5 (M+Na), 1067.5 (M+K). HRMS (MALDI-TOF/DCTB) *m/z* calcd for $C_{58}H_{66}O_6F_6NaSi_2$ (M+Na): 1051.4200 found: 1051.4163.

4,7,13,16-tetramethoxy-4,7-diphenyl-1,10-bis(2-phenylethynyl)-13,16-bis (trifluoromethyl)cyclooctadeca-2,5,8,11,14,17-hexayne-1,10-diol (7h). To a solution of HMDS (0.87 mL, 4.12 mmol), in THF (50 mL) at – 78 °C was added *n*-BuLi (1.6 mL, 4.0

mmol). The resulting mixture was stirred 30 min before adding a solution of the diketone **10h** (0.380 g, 0.67 mmol) in THF (10 mL) at the same temperature. After stirring 30 min at – 78 °C, the solution was diluted by addition of 40 mL of THF, to have a total volume of 100 mL. This reaction mixture, and a solution of the triyne **1** (0.200 g, 0.67 mmol) in THF (100 mL) were transferred simultaneously into a 1 L flask filled with 500 mL of THF under stirring at – 78 °C. The temperature was allowed to warm slowly up to room temperature and the stirring was maintained overnight before treatment with saturated aqueous NH_4Cl. After evaporation of about 500 mL of THF, the layers were separated, the aqueous one was extracted with diethylether, and the combined organic layers were washed with brine, dried over $MgSO_4$ and evaporated under reduced pressure. The residue was purified by silicagel chromatography (acetone/pentane 2:8) to give the expected [6]pericyclynediol **7h** as a pale brown solid in 13 % yield (0.080 g).

δ_H ($CDCl_3$, 400 MHz) 3.48-3.69 (14 H, m, O-CH_3, OH), 7.37-7.40 (12 H, m, *m*-, *p*-C_6H_5-C≡C, *m*-, *p*-C_6H_5-C-OCH_3), 7.49-7.52 (4 H, m, *o*-C_6H_5-C≡C), 7.75-7.82 (4 H, m, *o*-C_6H_5-C-OCH_3). δ_F ($CDCl_3$, 376 MHz) -79.06-(-78.78) (m, CF_3). $\delta_{C\{H\}}$ ($CDCl_3$, 75 MHz) 53.34-53.56, 54.07-54.41 (2 m, OCH$_3$), 70.85 (q, $^2J_{CF}$ 36 Hz, *C*-CF_3), 71.78-71.85, 72.24-72.33 (2 m, *C*(OCH_3)(C_6H_5), *C*-OH), 78.51-78.72, 80.60-80.85, 83.73-84.91, 86.17-86.34, 86.55-86.62 (m, *C*≡*C*), 121.00 (br s, *i*-C_6H_5-C≡C), 121.08 (q, $^1J_{CF}$ 285 Hz, CF_3), 126.49-126.59, 128.45, 128.59, 129.20-129.26, 129.52 (m, *o*-, *m*-, *p*-C_6H_5-C-OCH_3, *m*-, *p*-C_6H_5-C≡C), 132.02 (*o*-C_6H_5-C≡C), 138.83-139.27 (m, *i*-C_6H_5-C-OCH_3). MS (DCI/CH_4) *m/z* 837.2 (M-OMe), 851.2 (M-OH), 869.2 (M+H). HRMS (DCI/CH_4) *m/z* calcd for $C_{52}H_{35}O_6F_6$ (M+H): 869.2338, found: 869.2362.

1,10-bis[4-(9*H*-carbazol-9-yl)phenyl]-4,7-dimethoxy-4,7-diphenyldeca-2,5,8-triyne-1,10-dione (10e). To a solution of **11e** (0.150 g, 0.18 mmol) in DCM (50 mL) at 0˚C was added MnO_2 (0.450 g, 5.3 mmol). The reaction mixture was stirred 1 h at 0 ˚C and 3 h at room temperature. Then, the reaction mixture was filtered through celite, and the filtrate was evaporated under reduced pressure to give the diketone **10e** as a light solid in 91% yield (0.140 g).

mp 84 °C. δ_H ($CDCl_3$, 400 MHz) 3.73 (6 H, s, O-CH_3), 7.34 (4 H, d, $^3J_{HH}$ 7.8 Hz, *H*8-, *H*12-carbazole), 7.39-7.53 (14 H, m, *m*-, *p*-C_6H_5-C(OMe), *H*6-, *H*10-, *H*7-, *H*11-carbazole), 7.70 (4 H, d, $^3J_{HH}$ 8.2 Hz, *o*-C_6H_4-N), 7.89 (4 H, d, $^3J_{HH}$ 8.0 Hz, *o*-C_6H_5-C(OMe)), 8.15 (4 H, d, $^3J_{HH}$ 7.8 Hz, *H*9-, *H*13-carbazole), 7.70 (4 H, d, $^3J_{HH}$ 8.2 Hz, *o*-C_6H_4-N), 8.33 7.70 (4 H, d, $^3J_{HH}$ 8.2 Hz, *m*-C_6H_4-N). $\delta_{C\{H\}}$ ($CDCl_3$, 100 MHz) 54.09 (O*C*H_3), 72.23 (*C*-OCH_3), 83.58, 84.57, 89.68 (C-*C*≡*C*-C), 109.82 (*C*6-, *C*10-carbazole), 120.50 (*o*-C_6H_4-N), 120.91 (*C*8-, *C*12-carbazole), 124.05 (*C*3-, *C*4-carbazole), 126.31, 126.37, 126.53 (*C*9-, *C*13-, *C*7-, *C*11-carbazole, *o*-C_6H_5), 128.91 (*m*-C_6H_5), 129.66 (*p*-C_6H_5), 131.39 (*m*-C_6H_4-N), 134.61 (*p*-C_6H_4-N), 138.37, 139.95, 143.60 (*C*2-, *C*5-carbazole, *i*-C_6H_5, *i*-C_6H_4-N), 175.71 (*C*=O). MS (MALDI-TOF/DCTB) *m/z* 852.4 (M). HRMS (MALDI-TOF/DCTB) *m/z* calcd for $C_{60}H_{40}N_2O_4$: 852.2988, found: 852.2997.

1,10-bis[4-(1*H*-indol-1-yl)phenyl]-4,7-dimethoxy-4,7-diphenyldeca-2,5,8-triyne-1,10-dione (10f). To a solution of diol **11f** (0.210 g, 0.28 mmol) in DCM (50 mL) at 0 ˚C was added MnO_2 (0.720 g, 8.3 mmol). The resulting mixture was stirred 1 h at 0 ˚C and 3 h at room temperature before filtration through celite. The filtrate was evaporated under reduced pressure to give the diketone **10f** as a light solid in 91 % yield (180 mg).

mp 87 °C. δ_H ($CDCl_3$, 400 MHz) 3.74 (6 H, s, O-CH_3), 6.72 (2 H, s, *H*3-indole) 7.28-7.64 (20 H, m, *H*2-, *H*5-, *H*6-, *H*7-, *H*8-indole, *m*-, *p*-C_6H_5, *o*-C_6H_4-N), 7.73 (2 H, d, $^3J_{HH}$ 7.5 Hz, *H*9-indole), 7.91 (4 H, d, $^3J_{HH}$ 6.5 Hz, *o*-C_6H_5) 8.28 (4 H, d, $^3J_{HH}$ 8.2 Hz, *m*-C_6H_4-N). $\delta_{C\{H\}}$ ($CDCl_3$, 100 MHz) 54.09 (O-*C*H_3), 72.26 (*C*-OCH_3), 83.65, 84.60, 89.55 (-*C*≡*C*-), 105.72 (*C*3-indole), 110.73 (*C*6-indole), 121.37 (*o*-C_6H_4-N), 121.56, 123.13, 123,17, 124.31 (*C*2-, *C*7-, *C*8-, *C*9-indole), 126.57 (*o*-C_6H_5), 127.21 (*p*-C_6H_5), 128.93 (*m*-C_6H_5), 129.68 (*C*4-indole), 131.41 (*m*-C_6H_4-N), 133.78 (*i*-C_6H_4-N), 135.24 (*C*5-indole), 138.42 (*i*-C_6H_5), 145.08 (*p*-C_6H_4-N), 175.66 (*C*=O) . MS (MALDI-TOF/DCTB) *m/z* 752.2 (M). HRMS (MALDI-TOF/DCTB) *m/z* calcd for $C_{52}H_{36}N_2O_4$: 752.2675, found: 752.2744.

6,9-dimethoxy-1,6,9,14-tetraphenyltetradeca-1,4,7,10,13-pentayne-3,12-dione (10h). To a solution of **11h** (0.450 g, 0.78 mmol) in DCM (50 mL) was added MnO_2 (0.680 g, 7.82 mmol) at room temperature. The resulting mixture was stirred for 4 h and then filtered through celite and evaporated under reduced pressure to give the diketone **10h** in 96 % yield (0.430 g) as a light oil.

δ_H ($CDCl_3$, 300 MHz) 3.66 (6 H, s, O-CH_3), 7.40-7.55 (12 H, m, *m*-, *p*-C_6H_5-C≡C, *m*-, *p*-C_6H_5-C-OCH_3), 7.62 (4 H, d, $^3J_{HH}$ 6.9 Hz, *o*-C_6H_5-C≡C), 7.81 (4 H, d, $^3J_{HH}$ 6.3 Hz, *o*-C_6H_5-C-OCH_3). $\delta_{C\{H\}}$ ($CDCl_3$, 100 MHz) 54.00 (O-*C*H_3), 72.05 (*C*-OCH_3), 84.25, 85.47, 87.42, 89.18, 93.15 (*C*≡*C*), 119.08 (*i*-C_6H_5-C≡C), 126.54, 128.79 (*o*-, *m*-C_6H_5-C-OCH_3, *m*-C_6H_5-C≡C), 129.62, 131.59 (*p*-C_6H_5-C≡C, *p*-C_6H_5-C-OCH_3), 133.51 (*o*-C_6H_5-C≡C), 138.10 (*i*-C_6H_5-C-OCH_3), 159.84 (*C*=O). MS (DCI/NH_3) *m/z* 588.2 (M+NH_4). HRMS (DCI/CH_4) *m/z* calcd for $C_{39}H_{23}O_3$ (M-OMe): 539.1674, found: 539.1663.

1,10-bis[4-(9*H*-carbazol-9-yl)phenyl]-4,7-dimethoxy-4,7-diphenyldeca-2,5,8-triyne-1,10-diol (11e). To a solution of 9-(4-bromophenyl)-9Hcarbazole (0.124 g, 0.385 mmol) in THF (15 mL) under stirring at -78 °C was added *n*-BuLi (147 µl, 0.37 mmol). The reaction mixture was stirred during 1 h at -78 °C before adding a solution of the dialdehyde **9** (0.065 g, 0.175 mmol) in THF (3 mL). The temperature was allowed to increase slowly up to - 10 °C in 3 h before adding saturated aqueous NH_4Cl. The aqueous layer was extracted with diethylether and the combined organic layers were washed with brine, dried over $MgSO_4$ and evaporated under reduced pressure. The residue was purified by silica gel chromatography (EtOAc/Pentane 2:8) to give **11e** as a yellow solid in 32 % yield (0.150 g).

mp 73 °C. δ_H ($CDCl_3$, 400 MHz) 2.69 (2 H, br s, O*H*), 3.63 (6 H, s, O-C*H*$_3$), 5.72 (2 H, br s, C*H*-OH), 7.29-7.47 (18 H, m, *m-, p*-C$_6$*H*$_5$, *H*6-, *H*10-, *H*7-, *H*11-, *H*8-, *H*12-carbazole), 7.56 (4 H, d, $^3J_{HH}$ 7.9 Hz, *o*-C$_6$*H*$_4$-N), 7.78 (4 H, d, $^3J_{HH}$ 7.9 Hz, *m*-C$_6$*H*$_4$-N), 7.87 (4 H, d, $^3J_{HH}$ 7.5 Hz, *o*-C$_6$*H*$_5$), 8.18 (4 H, d, $^3J_{HH}$ 7.7 Hz, *H*9-, *H*13-carbazole). $\delta_{C\{H\}}$ ($CDCl_3$, 100 MHz) 53.50 (O-*C*H$_3$), 64.19 (*C*-OH), 72.1 (*C*-OCH$_3$), 84.46, 84.74, 86.70 (*C*≡*C*), 109.73 (*C*6-, *C*10-carbazole), 120.14 (*o*-*C*$_6$H$_4$-N), 120.38 (*C*8-, *C*12-carbazole), 123.49 (*C*3-, *C*4-carbazole), 126.03 (*C*9-, *C*13-carbazole), 126.6 (*o*-*C*$_6$H$_5$), 127.2 (*C*7-, *C*11-carbazole), 128.33 (*m*-*C*$_6$H$_4$-N), 128.63 (*m*-*C*$_6$H$_5$), 129.20 (*p*-*C*$_6$H$_5$), 137.99 (*C*2-, *C*5-carbazole) 138.95, 139.59, 140.71 (*i*-*C*$_6$H$_5$, *i*-, *p*-*C*$_6$H$_4$-N). MS (MALDI-TOF/DCTB) *m/z* 856.3 (M). HRMS (MALDI-TOF/DCTB) *m/z* calcd for $C_{60}H_{44}N_2O_4$: 856.3301, found: 856.3372.

1,10-bis[4-(1*H*-indol-1-yl)phenyl]-4,7-dimethoxy-4,7-diphenyldeca-2,5,8-triyne-1,10-diol (11f). To a solution of 1-(4-bromophenyl)-1H-indole (0.690 g, 2.53 mmol) in THF (15 mL) was added *n*-BuLi (0.92 mL, 2.3 mmol) at -78°C. The reaction mixture was stirred 1 h at -78

°C before adding a solution of the dialdehyde **9** (0.380 g, 1.03 mmol) in THF (3mL). The temperature was allowed to increase slowly up to -10 °C in 3 h before treatment with saturated aqueous NH_4Cl. The aqueous layer was extracted with diethylether and the combined organic layers were washed with brine, dried over $MgSO_4$ and evaporated under reduced pressure. The residue was purified by silica gel chromatography (EtOAc/Pentane 2:8) to give diol **11f** as yellow solid in 27 % yield (0.210 g).

mp 76 ˚C. δ_H ($CDCl_3$, 400 MHz) 2.70 (2 H, br s, O*H*), 3.60 (6 H, s, O-C*H*$_3$), 5.65 (2 H, s, C*H*-OH), 6.72 (2 H, s, *H*3-indole), 7.15-7.74 (24 H, m, *H*2-, *H*5-, *H*6-, *H*7-, *H*8-indole, *o*-, *m*-, *p*-C$_6$*H*$_5$, *o*-C$_6$*H*$_4$-N), 7.84 (4 H, d, $^3J_{HH}$ 7.3 Hz, *m*-C$_6$*H*$_4$-N). $\delta_{C\{H\}}$ ($CDCl_3$, 100 MHz) 53.47 (O-*C*H$_3$), 64.10 (*C*-OH), 72.08 (*C*-OCH$_3$), 84.29, 84.71, 86.74 (*C*≡*C*), 103.99 (*C*3-indole), 110.48 (*C*6-indole), 120.59 (*o*-*C*$_6$H$_4$-N), 121.26 (*C*2-indole), 122.56 (*C*9-indole), 124.31 (*C*7-, *C*8-indole), 126.59 (*o*-*C*$_6$H$_5$), 127.78, 128.10, 128.60 (*m*-, *p*-*C*$_6$H$_5$, *m*-*C*$_6$H$_4$-N), 129.17 (*C*4-indole), 135.72 (*C*5-indole), 137.73 (*i*-*C*$_6$H$_4$-N), 139.59, 139.98 (*i*-*C*$_6$H$_5$, *p*-*C*$_6$H$_4$-N). MS (MALDI-TOF/DCTB) *m/z* 756.3 (M). HRMS (MALDI-TOF/DCTB) *m/z* calcd for $C_{52}H_{40}N_2O_4Na$ (M+Na): 779.2886, found: 779.2939.

6,9-dimethoxy-1,6,9,14-tetraphenyltetradeca-1,4,7,10,13-pentayne-3,12-diol (11h). To a solution of phenylacetylene (0.30 mL, 2.73 mmol) in THF (5 mL) under stirring at – 78 °C was added *n*-BuLi (1.08 mL, 2.73 mmol). After 45 min, a solution of the dialdehyde **9** (0.40 g, 1.08 mmol) in THF (20 mL) was added at the same temperature. The resulting mixture was stirred at – 78 °C for 1.5 h before treatment with saturated aqueous NH_4Cl. The separated aqueous layer was extracted with diethylether, and the combined organic layers were washed with brine, dried over $MgSO_4$ and evaporated under reduced pressure. The residue was

purified by silicagel chromatography (acetone/DCM/Pentane 0.6:2.4:7 to 0.8:3.2:6) to finally give **11h** as a pale oil in 72 % yield (0.450 g).

δ_H ($CDCl_3$, 400 MHz) 3.43 (2 H, br s, O*H*), 3.59 (6 H, s, O-C*H*$_3$), 5.48 (2 H,d, $^3J_{HH}$ 8.0 Hz, C*H*-OH), 7.30-7.48 (16 H, m, *o*-, *m*-, *p*-C$_6$*H*$_5$-C≡C, *m*-, *p*-C$_6$*H*$_5$-C-OCH$_3$), 7.83 (4 H, d, $^3J_{HH}$ 8.0 Hz, *o*-C$_6$*H*$_5$-C-OCH$_3$). $\delta_{C\{H\}}$ ($CDCl_3$, 100 MHz) 52.63 (*C*H-OH), 53.47 (O-*C*H$_3$), 71.95 (*C*-OCH$_3$), 81.42, 84.55, 84.63, 84.77, 85.65 (*C*≡*C*), 121.88 (*i*-*C*$_6$H$_5$-C≡C), 126.67, 128.38, 128.56 (*o*-, *m*-*C*$_6$H$_5$-C-OCH$_3$, *m*-*C*$_6$H$_5$-C≡C), 128.95, 129.09 (*p*-*C*$_6$H$_5$-C-OCH$_3$, *p*-*C*$_6$H$_5$-C≡C), 131.90 (*o*-*C*$_6$H$_5$-C≡C), 139.47 (*i*-*C*$_6$H$_5$-C-OCH$_3$). MS (DCI/CH_4) *m/z* 557.2 (M-OH). HRMS (DCI/CH_4) *m/z* calcd for $C_{40}H_{29}O_3$ (M-OH): 557.2117, found: 557.2124.

13,16-dimethoxy-1,10-bis(4-methoxyphenyl)-4,7-diphenyl-13,16-bis(trifluoromethyl) cyclooctadeca-1,2,3,7,8,9-hexaen-5,11,14,17-tetrayne (12b). To a solution of **7b** (0.073 g, 0.083 mmol) under stirring at – 78 °C in dry diethylether (50 mL) was added $SnCl_2$ (0.157 g, 0.83 mmol) and then HCl·Et_2O (0.83 mL, 1.66 mmol). The temperature was allowed to increase slowly up to – 10 °C in 3 h, thus giving a dark green mixture. Then aqueous 1 M NaOH (2.0 mL) was added and the mixture was allowed to warm up to room temperature. The aqueous layer was extracted with diethylether and the organic layer was washed with brine, dried over $MgSO_4$ and evaporated under reduced pressure. The residue was purified by silica gel chromatography (DCM/pentane 1:9) to give **12b** as dark-red solid in 69 % yield (0.045 g). Few mg of one pure isomer could be isolated.

Characterization of one isolated diastereoisomer. mp (decomp) 187 °C. λ_{max} ($CHCl_3$) 604 (ε 62200 L.mol^{-1}.cm^{-1}), 442 (53200). δ_H ($CDCl_3$, 300 MHz) 3.71 (6 H, s, O-C*H*$_3$), 3.90 (6 H, s, C$_6$H$_4$-OC*H*$_3$), 7.00 (4 H, d, $^3J_{HH}$ 8.9 Hz, *m*-C$_6$*H*$_4$-OCH$_3$), 7.42-7.46 (2 H, m, *p*-C$_6$*H*$_5$), 7.51-

7.56 (4 H, m, *m*-C_6H_5), 7.73 (4 H, d, $^3J_{HH}$ 8.9 Hz, *o*-C_6H_4-OCH_3), 7.92 (4 H, d, $^3J_{HH}$ 7.2 Hz, *o*-C_6H_5). δ_F ($CDCl_3$, 282 MHz) –78.77 (CF_3), second diastereoisomer: -78.93 (CF_3). $\delta_{C\{H\}}$ ($CDCl_3$, 75 MHz) 54.33 (O-CH_3), 55.54 (C_6H_4-OCH_3), 71.38 (q, $^2J_{CF}$ 35 Hz, *C*-CF_3), 85.62, 86.91, 100.32, 102.53, 105.58 (*C*-*C*≡*C*-*C*), 114.59 (*m*-C_6H_4-OCH_3), 121.55 (q, $^1J_{CF}$ 285 Hz, CF_3), 127.46, 128.94, 128.97, 129.21, 129.47 (*m*-C_6H_4-OCH_3, *o*-, *m*-, *p*-C_6H_5), 130.89 (*i*-C_6H_4-OCH_3), 136.67 (*i*-C_6H_5), 145.43, 147.37 (C=*C*=*C*=C), 160.92 (*p*-C_6H_4-OCH_3). MS (MALDI-TOF/ DCTB) *m/z* 784.3 (M). HRMS (MALDI-TOF/DCTB) *m/z* calcd for $C_{48}H_{30}O_4F_6$: 784.2048, found: 784.2076.

13,16-dimethoxy-1,4,7,10-tetraphenyl-13,16-bis(trifluoromethyl)cyclooctadeca-1,2,3,7,8,9-hexaen-5,11,14,17-tetrayne (12c). To a solution of the pericyclynediol **7c** (0.085 g, 0.103 mmol) in DCM (40 mL) under stirring at -78 °C were added $SnCl_2$ (0.196 g, 1.03 mmol) and $HCl{\cdot}Et_2O$ (1.03 mL, 2.06 mmol). The resulting mixture was allowed to warm slowly up to -10 °C, and then it was removed from the cold bath and stirred during 10 min at room temperature before treatment with aqueous 1 M NaOH (2.06 mL). After filtration through celite, the organic layer was washed 3 times with brine, dried over $MgSO_4$ and evaporated under reduced pressure. The residue was purified by silicagel chromatography (pentane/diethylether 9:1) to give the expected *carbo*-cyclohexadiene **12c** as a dark violet solid in 20 % yield (0.015 g).

λ_{max} ($CHCl_3$) 574 (ε 56700 $L.mol^{-1}.cm^{-1}$), 420 (44200). δ_H ($CDCl_3$, 300 MHz) 3.70 (6 H, s, O-CH_3), 7.36-7.56 (12 H, m, *m*-, *p*-C_6H_5), 7.77 (4 H, d, $^3J_{HH}$ 7.5 Hz, *o*-C_6H_5), 7.92 (4 H, d, $^3J_{HH}$ 7.6 Hz, *o*-C_6H_5). δ_F ($CDCl_3$, 282 MHz) –78.70 (CF_3). $\delta_{C\{H\}}$ ($CDCl_3$, 75 MHz) 54.36 (O-CH_3), 71.40 (q, $^2J_{CF}$ 36 Hz, *C*-CF_3), 85.43, 87.41, 100.16, 103.13, 107.36 (*C*-*C*≡*C*-*C*), 121.56 (q, $^1J_{CF}$

285 Hz, *C*F_3), 127.53, 127.55 (*m*-*C*$_6$H$_5$), 129.00, 129.05 (*o*-*C*$_6$H$_5$), 129.52, 129.63 (*p*-*C*$_6$H$_5$), 136.28, 136.43 (*i*-*C*$_6$H$_5$), 147.94, 148.60 (C=*C*=*C*=C). MS (MALDI-TOF/ DCTB) *m/z* 724.2 (M). HRMS (MALDI-TOF/DCTB) *m/z* calcd for $C_{46}H_{26}O_2F_6$: 724.1837, found: 724.1903.

13,16-dimethoxy-4,7-diphenyl-13,16-bis(trifluoromethyl)-1,10-bis[4-(trifluoromethyl) phenyl]cyclooctadeca-1,2,3,7,8,9-hexaen-5,11,14,17-tetrayne (12d). To a solution of the **7d** (0.040 g, 0.042 mmol) under stirring at – 78 °C in dry DCM (20 mL) were added $SnCl_2$ (0.080 g, 0.42 mmol) and then $HCl{\cdot}OEt_2$ (0.42 mL, 0.84 mmol). The temperature was allowed to increase slowly up to – 5 °C, thus giving a red solution. Then aqueous 1 M NaOH (0.92 mL) was added and the mixture was allowed to warm up to room temperature. The aqueous layer was extracted with DCM and the combined organic layers were washed with brine, dried over $MgSO_4$ and evaporated under reduced pressure. The residue was purified by silica gel chromatography (DCM/pentane 1:9) to give **12d** as a blue solid in 39 % yield (0.014 g). Few mg of one pure isomer could be isolated.

Characterization of one isolated diastereoisomer. λ_{max} ($CHCl_3$) 572 (ε 78300 $L.mol^{-1}.cm^{-1}$), 420 (112900). δ_H ($CDCl_3$, 300 MHz) 3.70 (6 H, s, O-C*H*$_3$), 7.47-7.58 (6 H, m, *m*-, *p*- C$_6$H$_5$), 7.71 (4 H, d, $^3J_{HH}$ 8.1 Hz, *m*-C$_6$*H*$_4$-CF$_3$), 7.84-7.98 (8 H, m, *o*-C$_6$*H*$_5$, *o*-C$_6$*H*$_4$-CF$_3$). δ_F ($CDCl_3$, 282 MHz) –78.66 (C*F*$_3$), –62.73 (C$_6$H$_4$-C*F*$_3$); second diastereoisomer: -78.63 (C*F*$_3$), -62.73 (C$_6$H$_4$-C*F*$_3$). $\delta_{C\{H\}}$ ($CDCl_3$, 75 MHz) 54.42 (O-*C*H$_3$), 71.38 (q, $^2J_{CF}$ 36 Hz, *C*-CF$_3$), 84.82, 88.08, 100.53, 101.64, 109.33 (*C*-*C*≡*C*-*C*), 121.48 (q, $^1J_{CF}$ 285 Hz, *C*F$_3$-C(OCH$_3$)), 123.84 (q, $^1J_{CF}$ 272 Hz, C$_6$H$_4$-*C*F$_3$), 125.96 (q, $^3J_{CF}$ 3.7 Hz, *m*-*C*$_6$H$_4$-CF$_3$), 127.40, 127.77, 129.23 (*o*-, *m*-*C*$_6$H$_5$, *o*-*C*$_6$H$_4$-CF$_3$), 130.25 (*p*-*C*$_6$H$_5$), 130.87 (q, $^2J_{CF}$ 33 Hz, *p*-C$_6$*H*$_4$-CF$_3$), 136.11 (*i*-*C*$_6$H$_5$), 139.44 (*i*-C$_6$*H*$_4$-CF$_3$), 148.85, 149.25 (C=*C*=*C*=C). MS (MALDI-TOF/DCTB) *m/z* 860.3 (M).

HRMS (MALDI-TOF/DCTB) *m/z* calcd for $C_{48}H_{24}O_2F_{12}$: 860.1585, found: 860.1660.

9-(4-{10-[4-(9*H*-carbazol-9-yl)phenyl]-13,16-dimethoxy-4,7-diphenyl-13,16-bis (trifluoromethyl)cyclooctadeca-1,2,3,7,8,9-hexaen-5,11,14,17-tetrayn-1-yl}phenyl)-9H-carbazole (12e).

To a solution of **7e** (not purified) in dry DCM (20 mL) at -78 °C were added $SnCl_2$ (0.125 g, 0.70 mmol) and then $HCl{\cdot}Et_2O$ (0.7 mL, 0.14 mmol). The temperature of the reaction mixture was slowly increased up to -10 °C in 3 h. Then aqueous 1 M NaOH (1.6 mL) was added. The aqueous layer was extracted with DCM and the combined organic layers were washed with brine, dried over $MgSO_4$ and evaporated under reduced pressure. The residue was purified by silica gel chromatography (EtOAc/Pentane 5:95) to give **12e** (mixture of isomers) as a dark solid in 21% yield (for two steps) (0.022 g). 10 mg of one pure isomer could be separated.

Characterization of one isolated diastereoisomer: mp 92 °C. λ_{max} ($CHCl_3$) 243 (ε 21200 $L.mol^{-1}.cm^{-1}$), 417 (9500), 616 (9300). Fluo (λ_{ex} 243 nm), λ_{em} 427. δ_H ($CDCl_3$, 400 MHz) 3.78 (6 H, s, O-C*H*$_3$), 7.35 (4 H, m, *H*8-, *H*12-carbazole), 7.43-7.62 (14 H, m, *m*-, *p*-C$_6$*H*$_5$, *H*6-, *H*10-, *H*7-, *H*11-carbazole), 7.73 (4 H, d, $^3J_{HH}$ 8.3 Hz, *o*-C$_6$*H*$_4$-N), 7.96-8.07 (8 H, m, *o*-C$_6$*H*$_5$, *m*-C$_6$*H*$_4$-N), 8.19 (4 H, d, $^3J_{HH}$ 7.5 Hz, *H*9-, *H*13-carbazole). δ_F ($CDCl_3$, 376 MHz) –78.60 (C*F*$_3$), second diastereoisomer: -78.72 (C*F*$_3$). $\delta_{C\{H\}}$ ($CDCl_3$, 100 MHz) 54.09 (O-*C*H$_3$), 73.33 (*C*-OCH$_3$), 81.88, 85.91, 87.75 (*C*≡*C*), 109.89 (*C*6-, *C*10-carbazole), 120.47, 120.51 (*o*-*C*$_6$H$_4$-N, *C*8-, *C*12-carbazole), 123.79 (*C*3-, *C*4-carbazole), 125.53, 126.19, 127.14, 127.71, 128.77, 129.19, 129.87, 130.89 (*C*9-, *C*13-, *C*7-, *C*11-carbazole, *o*-, *m*-, *p*-*C*$_6$H$_5$, *m*-*C*$_6$H$_4$-N, *C*F$_3$), 135.16, 136.45, 138.86, 140.43 (*p*-*C*$_6$H$_4$-N, *C*2-, *C*5-carbazole, *i*-*C*$_6$H$_5$, *i*-*C*$_6$H$_4$-N), 145.98, 147.63 (=*C*=*C*=). The very small quadruplet of *C*-CF$_3$ carbons was not observed. MS

(MALDI-TOF/DCTB) *m/z* 1054.3 (M). HRMS (MALDI-TOF/DCTB) *m/z* calcd for $C_{70}H_{40}N_2O_4F_6$: 1054.2994, found: 1054.3030.

1-(4-{10-[4-(1H-indol-1-yl)phenyl]-13,16-dimethoxy-4,7-diphenyl-13,16-bis (trifluoromethyl)cyclooctadeca-1,2,3,7,8,9-hexaen-5,11,14,17-tetrayn-1-yl}phenyl)-1H-indole (12f). To a solution of **7f** (0.090 g, 0.085 mmol) in dry DCM (20 mL) at -78 °C were added $SnCl_2$ (0.163 g, 0.85 mmol) and then $HCl{\cdot}Et_2O$ (0.85 mL, 1.7 mmol). The temperature of the reaction mixture was slowly increased up to -10 °C in 3 h. Then aqueous 1 M NaOH (2.0 mL) was added. The aqueous layer was extracted with DCM and the combined organic layers were washed with brine, dried over $MgSO_4$ and evaporated under reduced pressure. The residue was purified by silica gel chromatography (EtOAc/Pentane 5:95) to give the *carbo*-cylohexadiene **12f** (mixture of isomers) as a dark solid in 44 % yield (0.036 g). 12 mg of one pure isomer could be separated.

Characterization of one isolated diastereoisomer: mp 148 °C. λ_{max} ($CHCl_3$) 243 (ε 9900 $L.mol^{-1}.cm^{-1}$), 412 (6200), 615 (7700). Fluo (λ_{ex} 348 nm), λ_{em} 485; (λ_{ex} 243 nm), λ_{em} 326, 341, 485. δ_H ($CDCl_3$, 400 MHz) 3.76 (6 H, s, O-C*H*$_3$), 6.77 (2 H, br s, *H*3-indole), 7.20-7.35 (4 H, m, *H*7-, *H*8-indole), 7.41 (2 H, s, *H*2-indole), 7.37-7.79 (14 H, m, *m*-, *p*-C_6H_5, *m*-C_6H_4-N, *H*6-, *H*9-indole), 7.87-8.02 (8 H, m, *o*-C_6H_4-N, *o*-C_6H_5). δ_F ($CDCl_3$, 376 MHz) -78.66 (C*F*$_3$), second diastereoisomer: -78.83 (C*F*$_3$). $\delta_{C\{H\}}$ ($CDCl_3$, 100 MHz) 54.44 (O-*C*H$_3$), 85.26, 87.58, 100.71, 102.07, 107.46 (*C*-*C*≡*C*-*C*), 104.81 (*C*3-indole), 110.67 (*C*6-indole), 120.92 (*o*-C_6H_4-N), 121.39 (*C*2-indole), 122.82 (*C*9-indole), 124.08 (*C*7-, *C*8-indole), 127.44, 127.65, 128.63, 128.83, 129.14, 130.90 (*m*-, *p*-C_6H_5, *m*-C_6H_4-N), 129.75 (*C*4-indole), 134.26 (*i*-C_6H_4-N), 135.52 (*C*5-indole), 136.45 (*i*-C_6H_5), 140.68 (*p*-C_6H_4-N), 147.10, 147.92 (=*C*=*C*=). The small

quadruplet of *C*-CF_3 carbons could not be detected. MS (MALDI-TOF/DCTB) *m/z* 954.2 (M). HRMS (MALDI-TOF/DCTB) *m/z* calcd for $C_{62}H_{36}N_2O_2F_6$: 954.2681, found: 954.2658.

{2-[13,16-dimethoxy-4,7-diphenyl-13,16-bis(trifluoromethyl)-10-{2-[tris(propan-2-yl)silyl]ethynyl}cyclooctadeca-1,2,3,7,8,9-hexaen-5,11,14,17-tetrayn-1-yl]ethynyl} tris(propan-2-yl)silane (12g). To a solution of **7g** (0.066 g, 0.064 mmol) under stirring at – 78 °C in dry DCM (20 mL) were added $SnCl_2$ (0.122 g, 0.65 mmol) and then $HCl{\cdot}OEt_2$ (0.62 mL, 1.28 mmol). The temperature was allowed to increase slowly up to – 20 °C, thus giving a brown solution. Then aqueous 1 M NaOH (1.40 mL) was added and the mixture was allowed to warm up to room temperature. The aqueous layer was extracted with DCM and the combined organic layers were washed with brine, dried over $MgSO_4$ and evaporated under reduced pressure. The residue was purified by silica gel chromatography (DCM/pentane 5:95) to give a pure diastereoisomer of **12g** as a dark-purple solid in 2 % yield (2 mg).

δ_H ($CDCl_3$, 300 MHz) 1.15 (42 H, br s, Si-C*H*-CH_3), 3.62 (6 H, s, OCH_3), 7.43-7.45 (6 H, br d, *m*-, *p*-C_6H_5), 7.82-7.85 (4 H, br d, *o*-C_6H_5). δ_F ($CDCl_3$, 282 MHz) -78.75 (CF_3). MS (MALDI-TOF/DCTB) *m/z* 932.4 (M). HRMS (MALDI-TOF/DCTB) *m/z* calcd for $C_{56}H_{58}O_2F_6Si_2$: 932.3880, found: 932.3913.

13,16-dimethoxy-4,7-diphenyl-1,10-bis(2-phenylethynyl)-13,16-bis(trifluoromethyl) cyclooctadeca-1,2,3,7,8,9-hexaen-5,11,14,17-tetrayne (12h). To a solution of the [6]pericyclynediol **7h** (0.050 g, 0.06 mmol) in DCM (20 mL) under stirring at – 78 °C were added $SnCl_2$ (0.115 g, 0.60 mmol) and $HCl{\cdot}Et_2O$ (0.6 mL, 1.2 mmol). The resulting mixture was stirred 1 h at – 78 °C and then 1 h at room temperature before treatment with 1 M NaOH

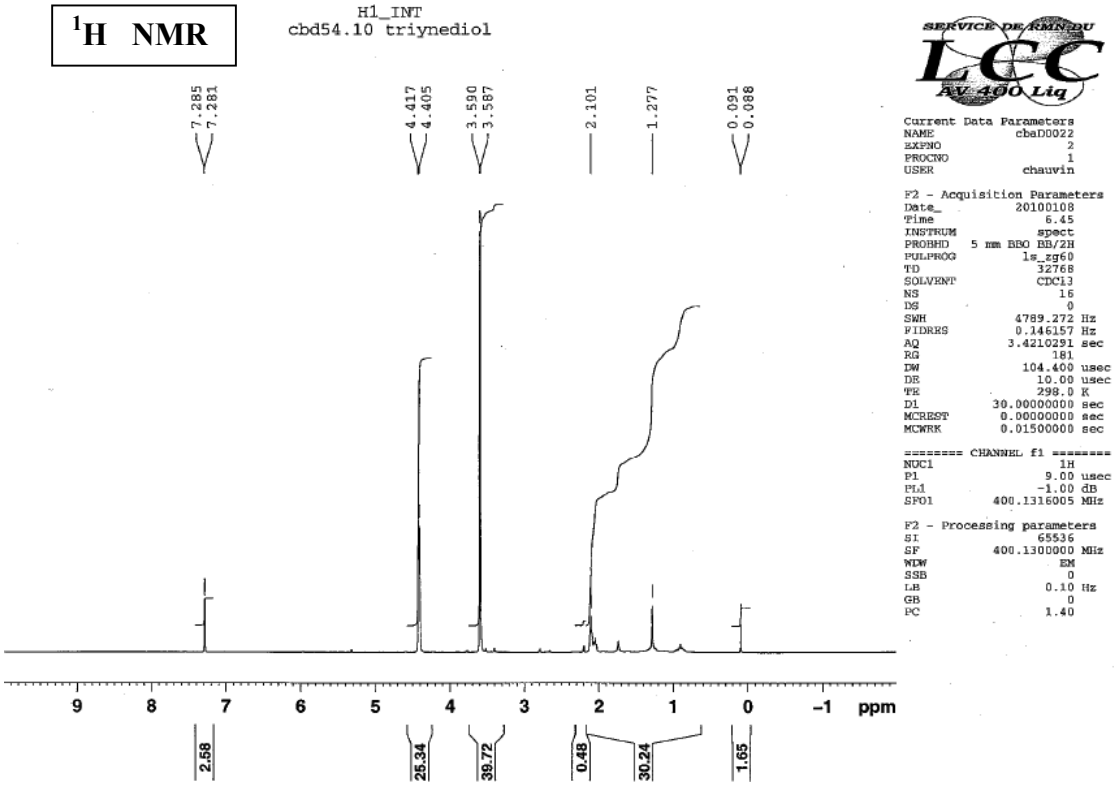
(1.2 mL). The separated organic layer was washed with brine, dried over $MgSO_4$ and concentrated to a 5 mL volume without going to dryness. This solution was directly deposited on a silicagel chromatography column (DCM/Pentane 2:8 to 5:5). The blue-green fraction corresponding to the two diastereoisomers (two spots on TLC) of the *carbo*-cyclohexadiene **12h** was concentrated under vacuum without going to dryness (product instable in the solid state). The NMR tube was prepared by addition of $CDCl_3$ in the DCM/pentane solution of **12h** followed by selective evaporation of the non-deuterated solvents under reduced pressure. The instability of the product did not allow determining a yield for this step.

Mixture of diastereoisomers. λ_{max} ($CHCl_3$, 300 MHz) 435, 594. δ_H ($CDCl_3$) 3.69, 3.72 (6 H, 2 s, O-CH_3), 7.28-7.59 (16 H, m, *o*-, *m*-, *p*-C_6H_5-C≡C, *m*-, *p*-C_6H_5-C≤), 7.90-7.92 (4 H, m, *o*-C_6H_5-C≤). δ_F ($CDCl_3$, 282 MHz) -78.78-(-78.57) (2 s, CF_3). $\delta_{C\{H\}}$ ($CDCl_3$, 75 MHz) 54.34, 54.42 (2 s, O-CH$_3$), 71.27 (q, $^2J_{CF}$ 35 Hz, C-CF$_3$), 83.24-83.34, 84.13, 85.44, 85.74, 88.50-88.55, 98.42, 101.22-101.26, 108.87 (*C*-*C*≡*C*-*C*, *C*≡*C*-C_6H_5), 121.36 (q, $^1J_{CF}$ 284 Hz, CF$_3$), 122.02 (*i*-C_6H_5-C≡C), 127.73, 128.58, 129.06 (*o*-, *m*-C_6H_5≤, *m*-C_6H_5-C≡C), 129.62, 130.25 (*p*-C_6H_5≤, *p*-C_6H_5-C≡C), 131.94 (*o*-C_6H_5-C≡C), 135.56 (*i*-C_6H_5≤), 143.56, 153.36 (=*C*=*C*=). MS (MALDI-TOF/DCTB) *m/z* 772.2 (M). HRMS (MALDI-TOF/DCTB) *m/z* calcd for $C_{50}H_{26}O_2F_6$: 772.1837, found: 772.1794.

5. Copies of ^{1}H, ^{13}C and ^{19}F NMR spectra and MS analyses

4a. 4,7-dimethoxy-4,7-bis(trifluoromethyl)deca-2,5,8-triyne-1,10-diol.

^{1}H NMR

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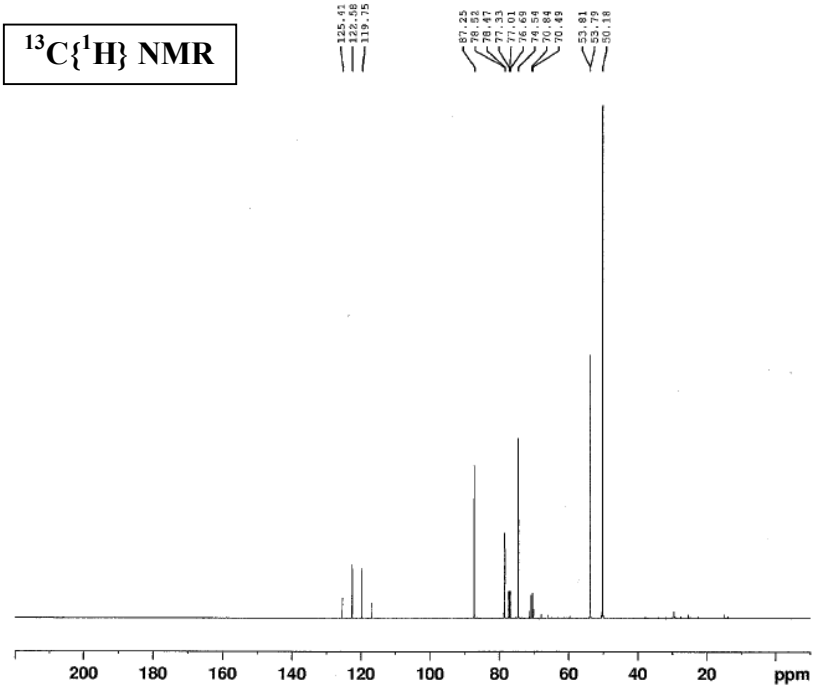
Current Data Parameters
NAME                  cbaD0022
EXPNO                        2
PROCNO                       1
USER                   chauvin

F2 - Acquisition Parameters
Date_                 20100108
Time                      6.45
INSTRUM                  spect
PROBHD    5 mm BBO BB/2H
PULPROG                ls_zg60
TD                        32768
SOLVENT                 CDCl3
NS                           16
DS                            0
SWH                  4789.272 Hz
FIDRES               0.146157 Hz
AQ                   3.42102912 sec
RG                          181
DW                    104.400 usec
DE                     10.00 usec
TE                     298.0 K
D1                    30.00000000 sec
MCREST                0.00000000 sec
MCWRK                 0.01500000 sec

======== CHANNEL f1 ========
NUC1                         1H
P1                         9.00 usec
PL1                       -1.00 dB
SFO1                400.1316005 MHz

F2 - Processing parameters
SI                          65536
SF                  400.1300000 MHz
WDW                            EM
SSB                             0
LB                         0.10 Hz
GB                              0
PC                           1.40
```

$^{13}C\{^{1}H\}$ NMR

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Current Data Parameters
NAME                  ariG0040
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20110725
Time                      8.02
INSTRUM                  spect
PROBHD    5 mm PABBO BB-
PULPROG               ls_zgdc60
TD                        65536
SOLVENT                 CDCl3
NS                         1024
DS                            4
SWH                 23148.148 Hz
FIDRES               0.353213 Hz
AQ                   1.4156276 sec
RG                         2050
DW                    21.600 usec
DE                     6.50 usec
TE                     298.0 K
D1                    1.00000000 sec
D11                   0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        13C
P1                         9.70 usec
PLW1                70.00000000 W
SFO1                100.6308781 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                         1H
PCPD2                     90.00 usec
PLW2                17.00000000 W
PLW12                0.34386000 W
SFO2                400.1616006 MHz

F2 - Processing parameters
SI                          131072
SF                  100.6203130 MHz
WDW                            EM
SSB                   0
LB                         1.00 Hz
GB                    0
PC                           1.50
```
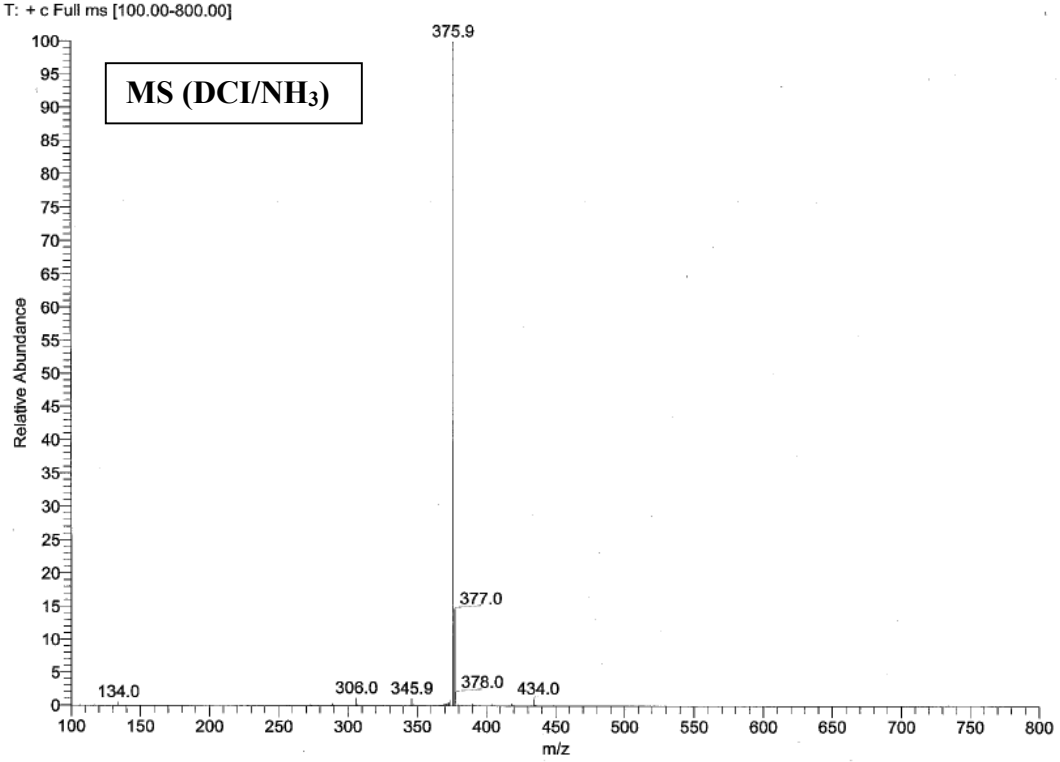
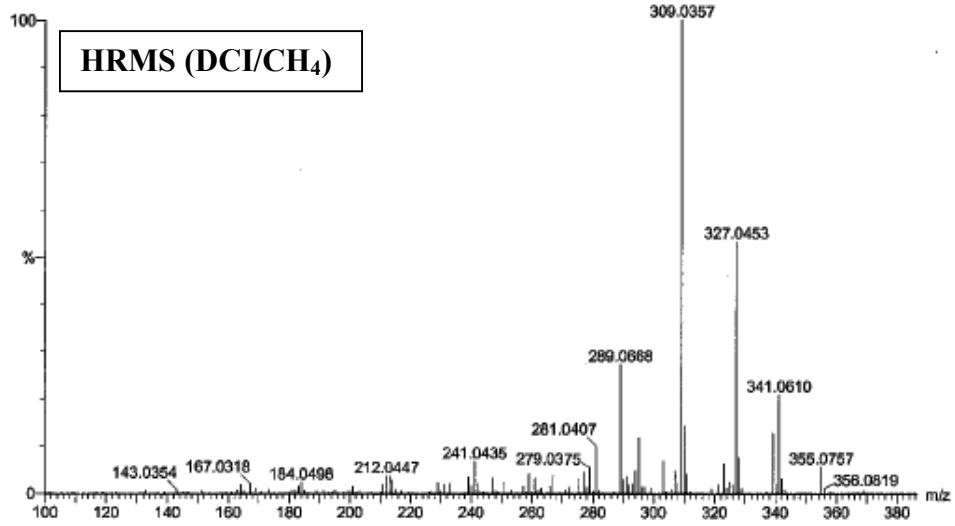

CBd5401 #6-11 RT: 0.15-0.26 AV: 6 NL: 8.12E7
T: + c Full ms [100.00-800.00]



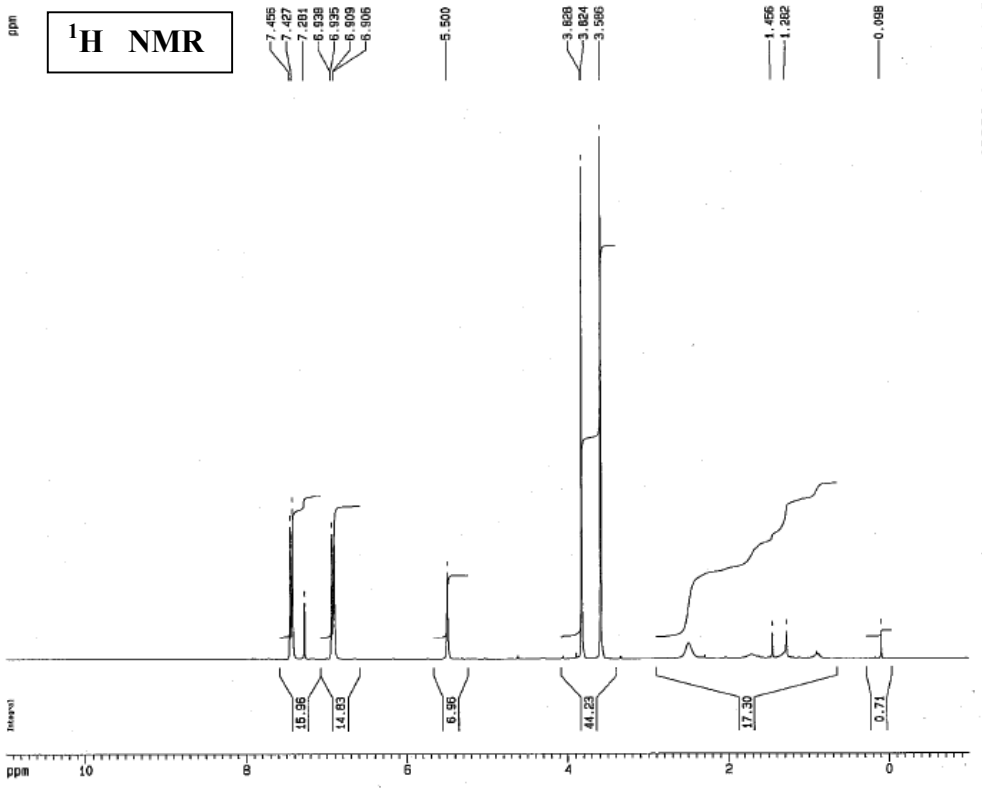
DCI-CH4
20110722-AR227B1 3 (0.083) Cm (3:6-29x5.000)
GCT Premier CAB109
22-Jul-2011 15:09:00
TOF MS CI+
3.61e+004



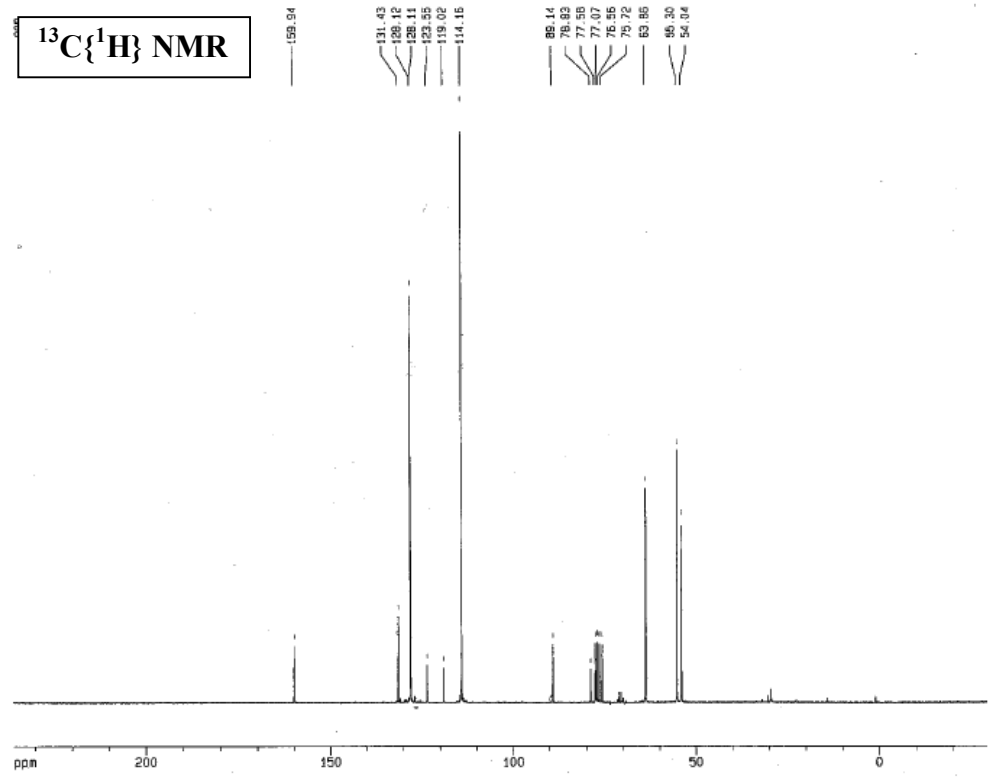
Minimum:	5.00				-1.5		
Maximum:	100.00		5.0	5.0	100.0		
Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
341.0610	20.79	341.0612	-0.2	-0.6	6.5	40.0	F3 C14 H11 O3
		341.0601	0.9	2.6	10.5	68.0	F6 C17 H10 O2
		341.0625	-1.5	-4.4	13.5	98.4	F5 C19 H11 O4
		341.0614	-0.4	-1.2	17.5	186.1	F2 C22 H10 O3
		341.0603	0.7	2.1	21.5	297.8	F C25 H9 O2

4b. 4,7-dimethoxy-1,10-bis(4-methoxyphenyl)-4,7-bis(trifluoromethyl)deca-2,5,8-triyne-1,10-diol

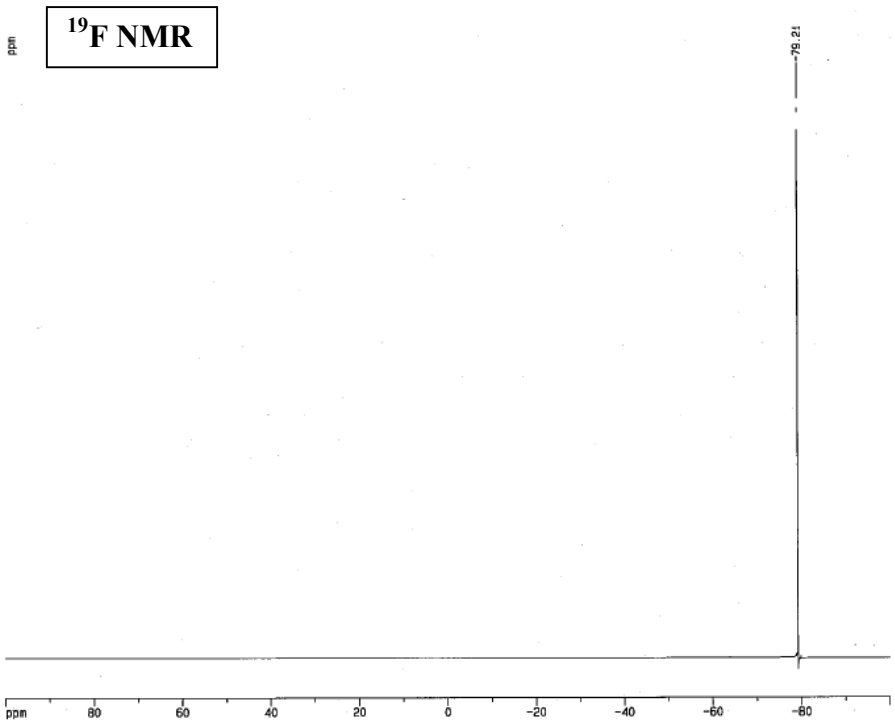
^{1}H NMR



^{13}C{^{1}H} NMR



^{19}F NMR



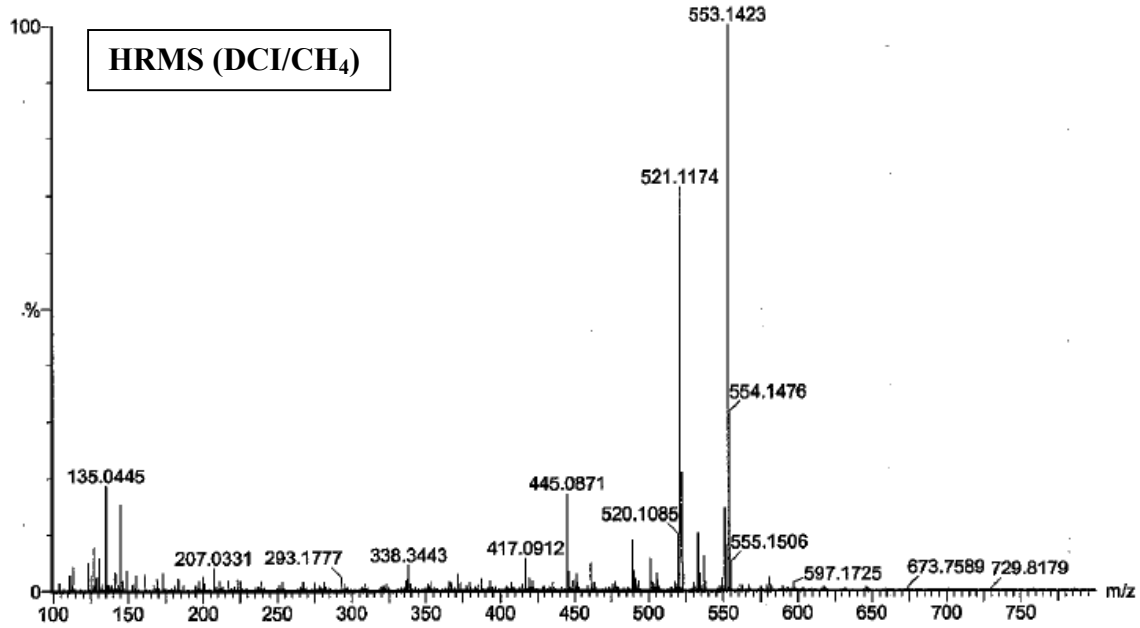

```
**********
* L.C.C. du C.N.R.S *
* Service de R.M.N. *
*      DPX 300      *
**********

Current Data Parameters
NAME                ve180168
EXPNO                      2
PROCNO                     1
USER                chauvin

F2 - Acquisition Parameters
Date_            20101206
Time                 13.09
INSTRUM              dpx300
PROBHD     5 mm BBF ATM
PULPROG            1s_zg60
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      4
SWH              56497.176 Hz
FIDRES           0.862078 Hz
AQ               0.5800436 sec
RG                  1290.2
DW                 8.850 usec
DE                10.00 usec
TE                   298 K
D1               1.00000000 sec

========== CHANNEL f1 ==========
NUC1                  19F
P1                 10.50 usec
PL1                -4.00 dB
SFO1         282.4043552 MHz

F2 - Processing parameters
SI                  65536
SF           282.4043950 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40

1D NMR plot parameters
CX                  22.00 cm
F1P               100.030 ppm
F1               28248.80 Hz
F2P              -100.028 ppm
F2               -28248.38 Hz
PPMCM             9.09353 ppm/cm
HZCM          2568.05347 Hz/cm
```

DCI/CH4 Pos
20101215-AR82 23 (0.750) Cm (14:23-46:51x5.000)

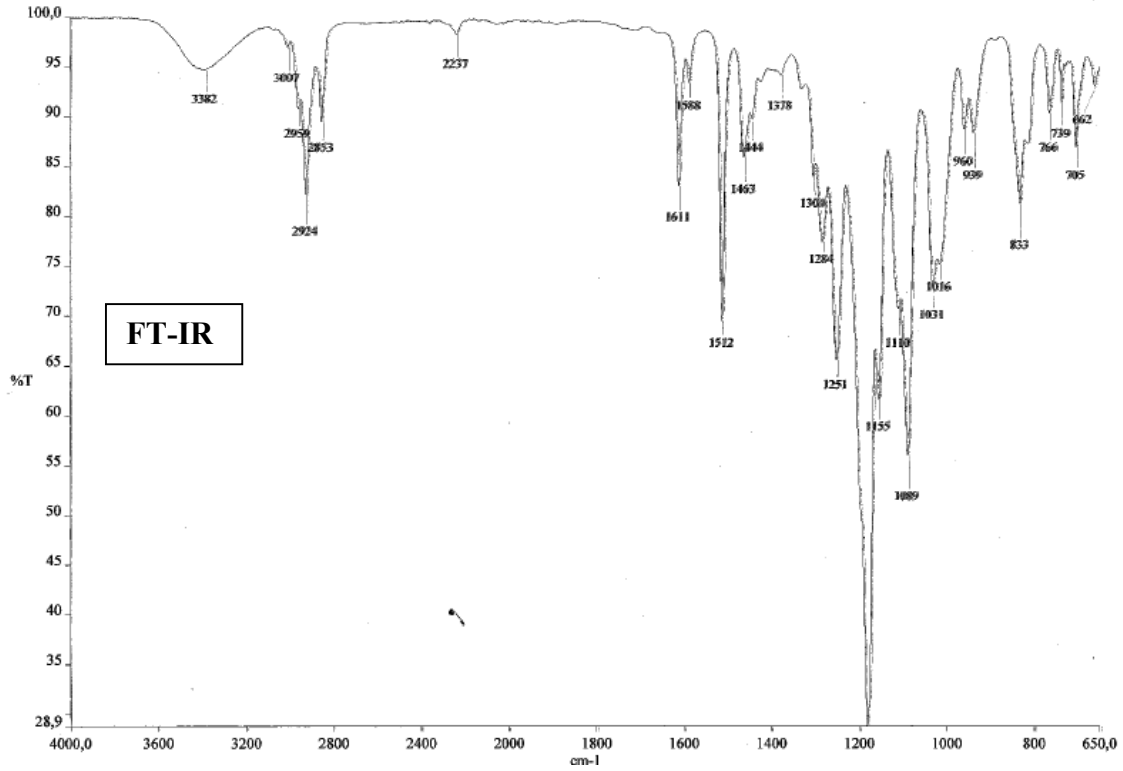
GCT Premier CAB109

15-Dec-2010 15:51:40
TOF MS CI+
1.83e+005

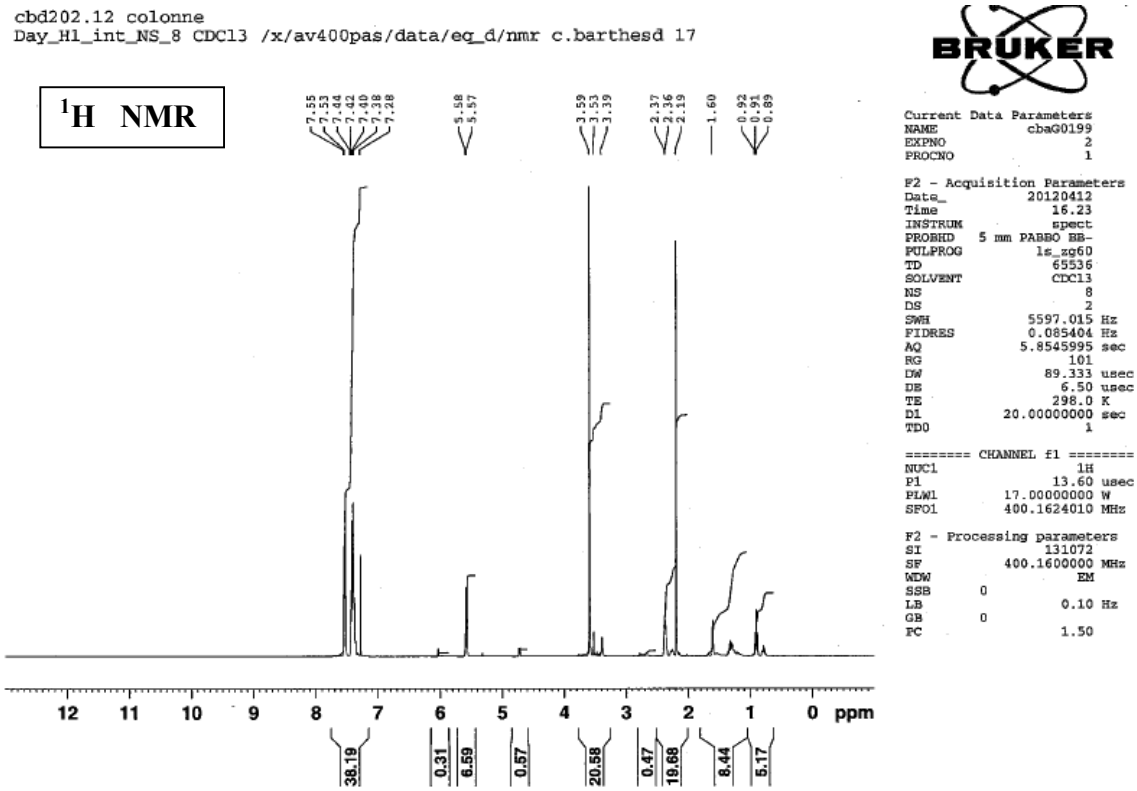
HRMS (DCI/CH_4)



Minimum:				-1.5					
Maximum:		10.0	5.0	50.0					
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula			
553.1423	553.1450	-2.7	-4.9	14.5	164.4	C28	H23	O5	F6
	553.1438	-1.5	-2.7	18.5	705.4	C31	H22	O4	F5
	553.1427	-0.4	-0.7	22.5	1845.1	C34	H21	O3	F4
	553.1415	0.8	1.4	26.5	3525.3	C37	H20	O2	F3
	553.1440	-1.7	-3.1	29.5	5593.9	C39	H21	O4	
	553.1404	1.9	3.4	30.5	5678.4	C40	H19	O	F2

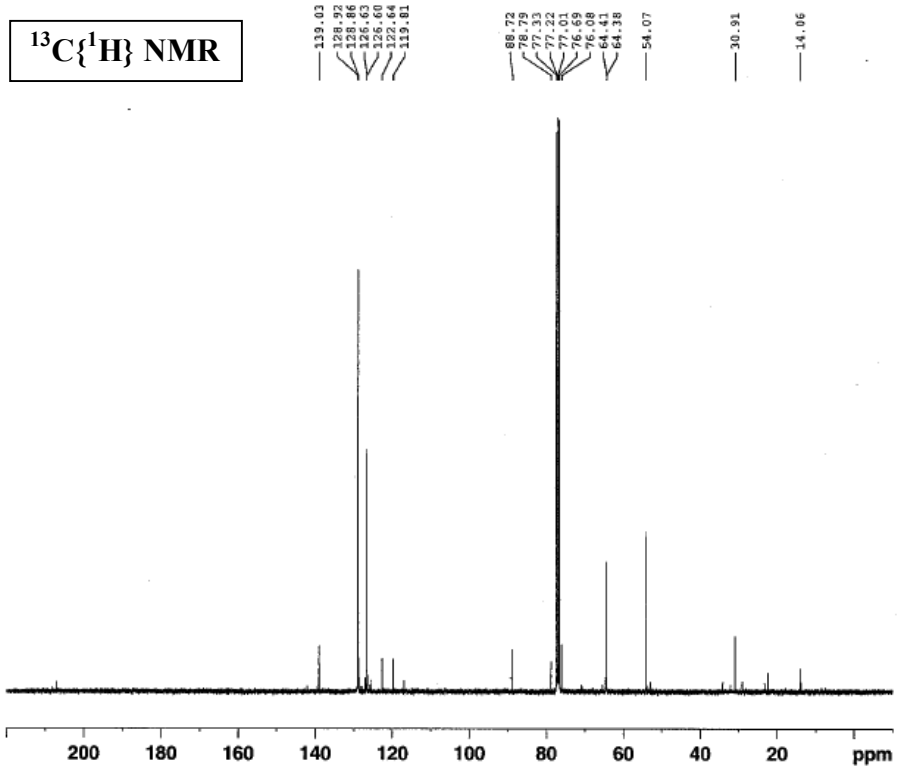


4c. 4,7-dimethoxy-1,10-diphenyl-4,7-bis(trifluoromethyl)deca-2,5,8-triyne-1,10-diol



cbd202.12
Night_C13_DECOUPLE_H1_NS_5000 CDCl3 /x/av400pas/data/eq_d/nmr c.barthesd 17

$^{13}C\{^{1}H\}$ NMR



Current Data Parameters
NAME cbaG0201
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120413
Time 1.21
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG 1s_zgdc60
TD 65536
SOLVENT CDCl3
NS 5000
DS 4
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 2050
DW 21.600 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

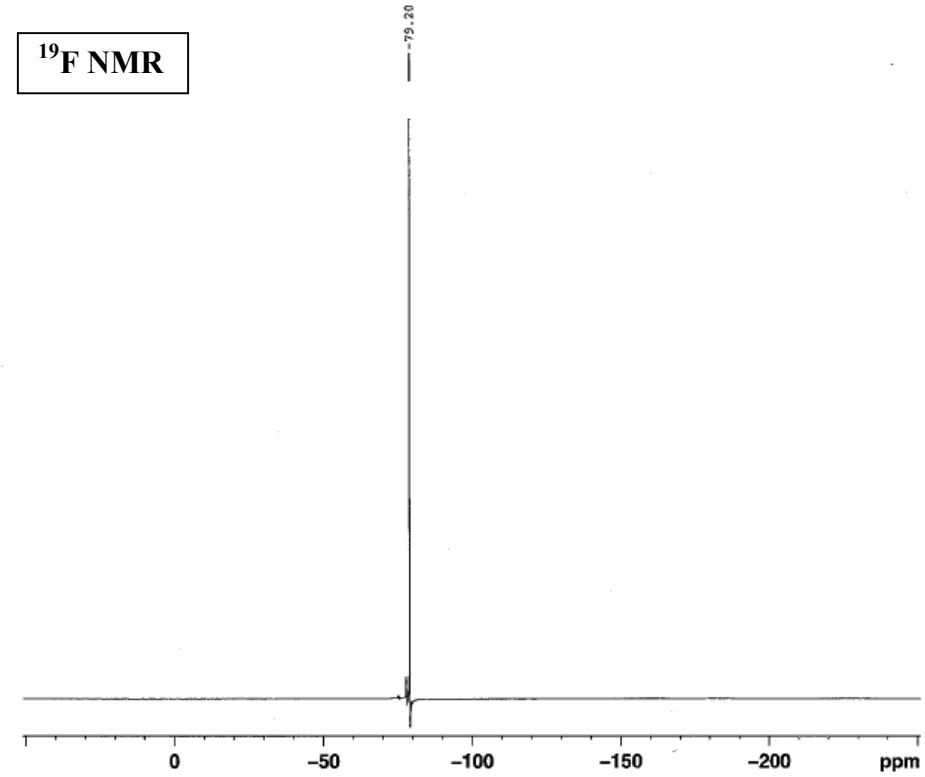
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PLW1 70.00000000 W
SFO1 100.6308781 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 17.00000000 W
PLW12 0.38819000 W
SFO2 400.1616006 MHz

F2 - Processing parameters
SI 131072
SF 100.6203130 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.50

cbd202.12 colonne
Day_F19_NS_40 CDCl3 /x/av400pas/data/eq_d/nmr c.barthesd 17

^{19}F NMR



Current Data Parameters
NAME cbaG0199
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120412
Time 16.17
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG 1s_zg60
TD 131072
SOLVENT CDCl3
NS 40
DS 4
SWH 113636.367 Hz
FIDRES 0.866977 Hz
AQ 0.5767668 sec
RG 645
DW 4.400 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1

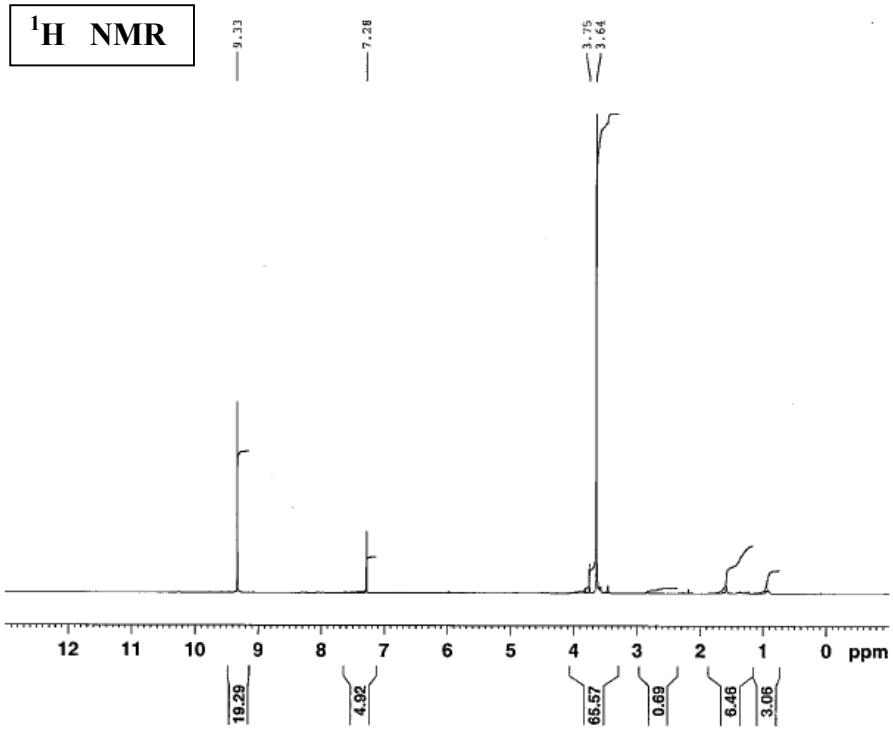
======== CHANNEL f1 ========
NUC1 19F
P1 11.65 usec
PLW1 25.00000000 W
SFO1 376.4889413 MHz

F2 - Processing parameters
SI 65536
SF 376.5265940 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

5a. 4,7-dimethoxy-4,7-bis(trifluoromethyl)deca-2,5,8-triynedial

cbd168.12
Night_H1_int_NS_8 CDCl3 /x/av400pas/data/eq_d/nmr c.barthesd 60

^{1}H NMR

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Current Data Parameters
NAME                  cbaG0123
EXPNO                        2
PROCNO                       1

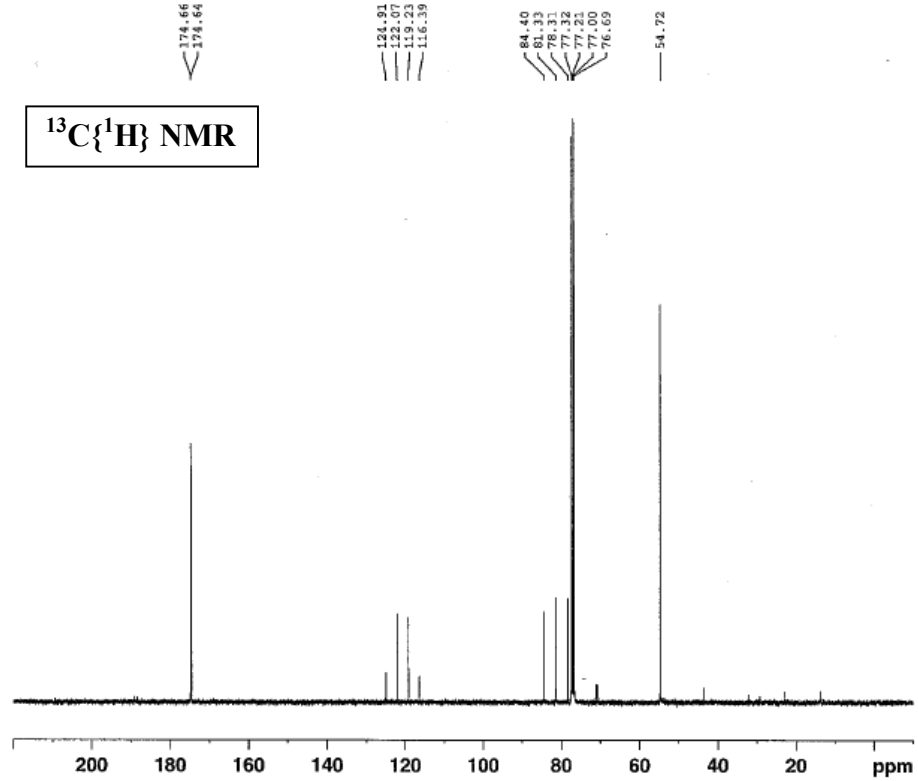
F2 - Acquisition Parameters
Date_              20120228
Time                   19.26
INSTRUM                spect
PROBHD    5 mm PABBO BB-
PULPROG             ls_zg60
TD                     65536
SOLVENT                CDCl3
NS                         8
DS                         2
SWH                5597.015 Hz
FIDRES             0.085404 Hz
AQ                 5.8545995 sec
RG                       101
DW                    89.333 usec
DE                     6.50 usec
TE                    298.0 K
D1                20.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     13.60 usec
PLW1              17.00000000 W
SFO1              400.1624010 MHz

F2 - Processing parameters
SI                    131072
SF                400.1600000 MHz
WDW                       EM
SSB          0
LB                     0.10 Hz
GB           0
PC                     1.50
```

cbd168.12
Night_C13_DECOUPLE_H1_NS_5000 CDCl3 /x/av400pas/data/eq_d/nmr c.barthesd 60

^{13}C{^{1}H} NMR




```
Current Data Parameters
NAME                  cbaG0123
EXPNO                        3
PROCNO                       1

F2 - Acquisition Parameters
Date_              20120228
Time                   22.55
INSTRUM                spect
PROBHD    5 mm PABBO BB-
PULPROG           ls_zgdc60
TD                     65536
SOLVENT                CDCl3
NS                      5000
DS                         4
SWH               23148.148 Hz
FIDRES             0.353213 Hz
AQ                 1.4156276 sec
RG                      2050
DW                    21.600 usec
DE                     6.50 usec
TE                    298.0 K
D1                1.00000000 sec
D11               0.03000000 sec
TD0                        1

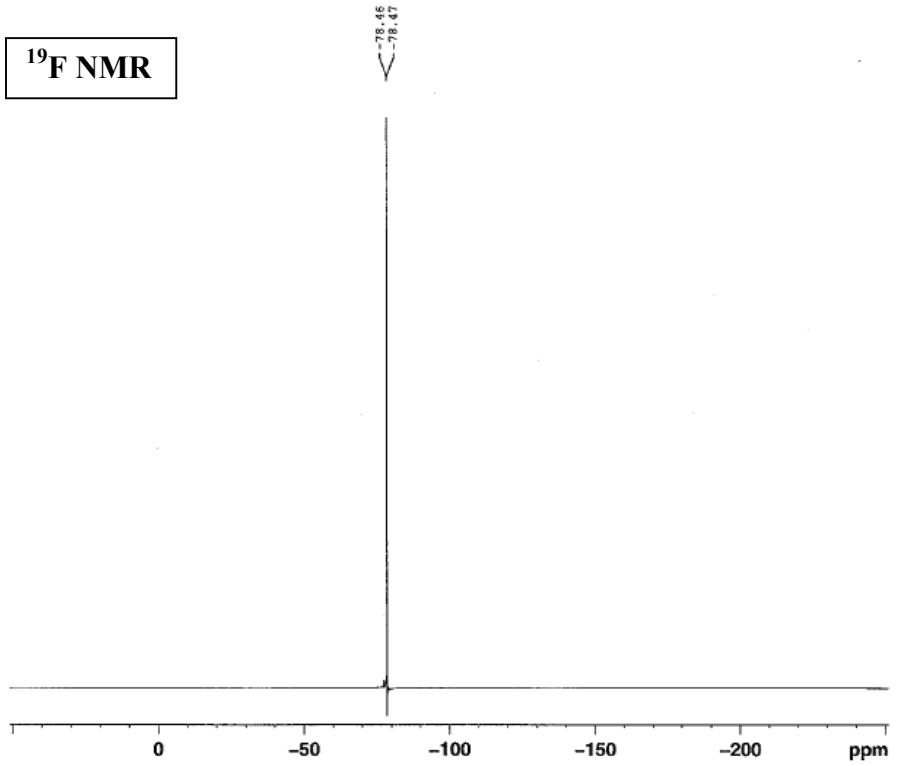
======== CHANNEL f1 ========
NUC1                      13C
P1                     9.00 usec
PLW1              70.00000000 W
SFO1              100.6308781 MHz

======== CHANNEL f2 ========
CPDPRG2               waltz16
NUC2                      1H
PCPD2                 90.00 usec
PLW2              17.00000000 W
PLW12              0.38819000 W
SFO2              400.1616006 MHz

F2 - Processing parameters
SI                    131072
SF                100.6203130 MHz
WDW                       EM
SSB          0
LB                     1.00 Hz
GB           0
PC                     1.50
```

cbd168.12
Night_F19_NS_40 CDCl3 /x/av400pas/data/eq_d/nmr c.barthesd 60

^{19}F NMR

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Current Data Parameters
NAME                  cbaG0123
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20120228
Time                     19.20
INSTRUM                  spect
PROBHD    5 mm PABBO BB-
PULPROG                ls_zg60
TD                       131072
SOLVENT                  CDCl3
NS                          40
DS                           4
SWH                  113636.367 Hz
FIDRES                 0.866977 Hz
AQ                   0.5767668 sec
RG                         645
DW                       4.400 usec
DE                       6.50 usec
TE                       298.0 K
D1                   1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       19F
P1                       11.65 usec
PLW1                25.00000000 W
SFO1                376.4889413 MHz

F2 - Processing parameters
SI                       65536
SF                  376.5265940 MHz
WDW                         EM
SSB          0
LB                        0.30 Hz
GB           0
PC                        1.00
```
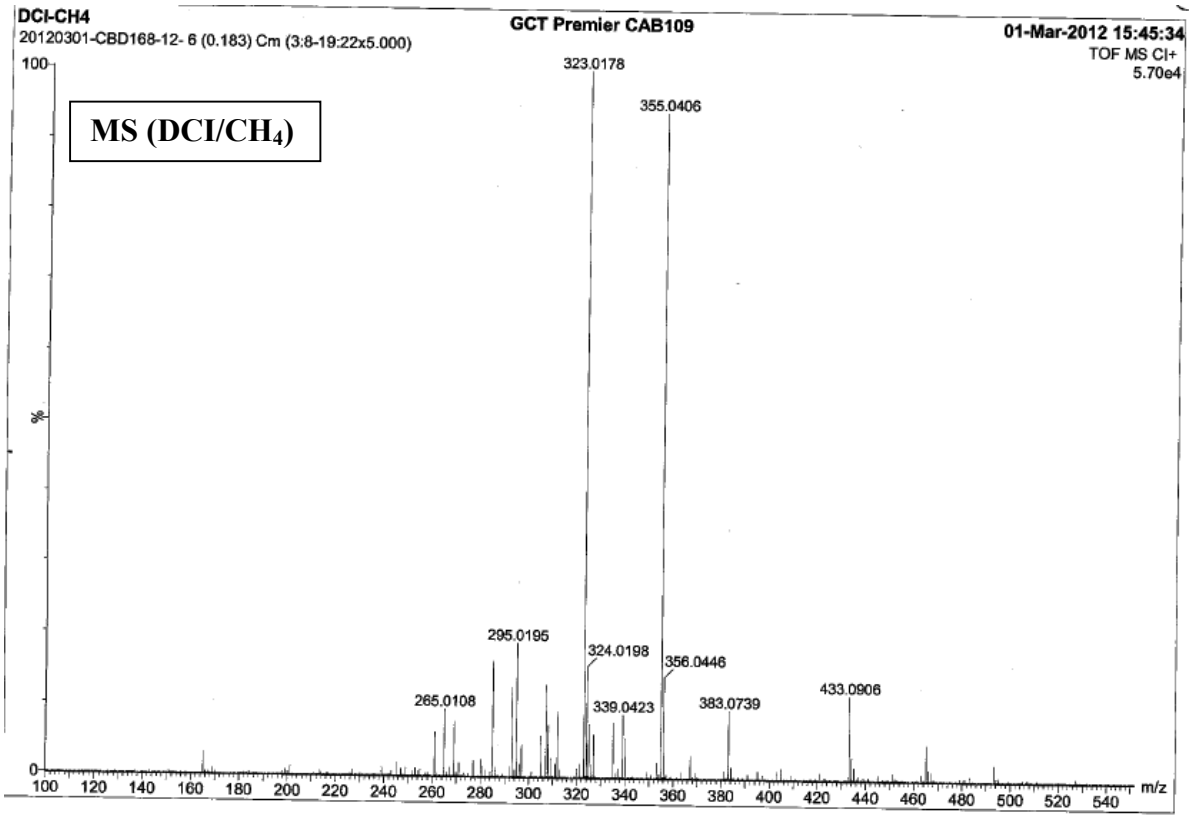



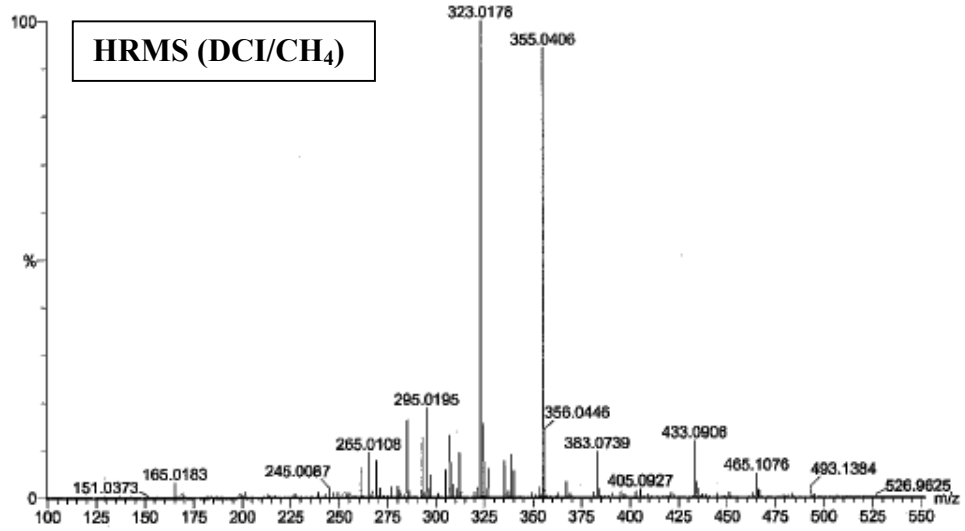
MS (DCI/CH_4)

DCI-CH4
20120301-CBD168-12- 6 (0.163) Cm (3:8-19:22x5.000)

GCT Premier CAB109

01-Mar-2012 15:45:34
TOF MS CI+
5.70e+004

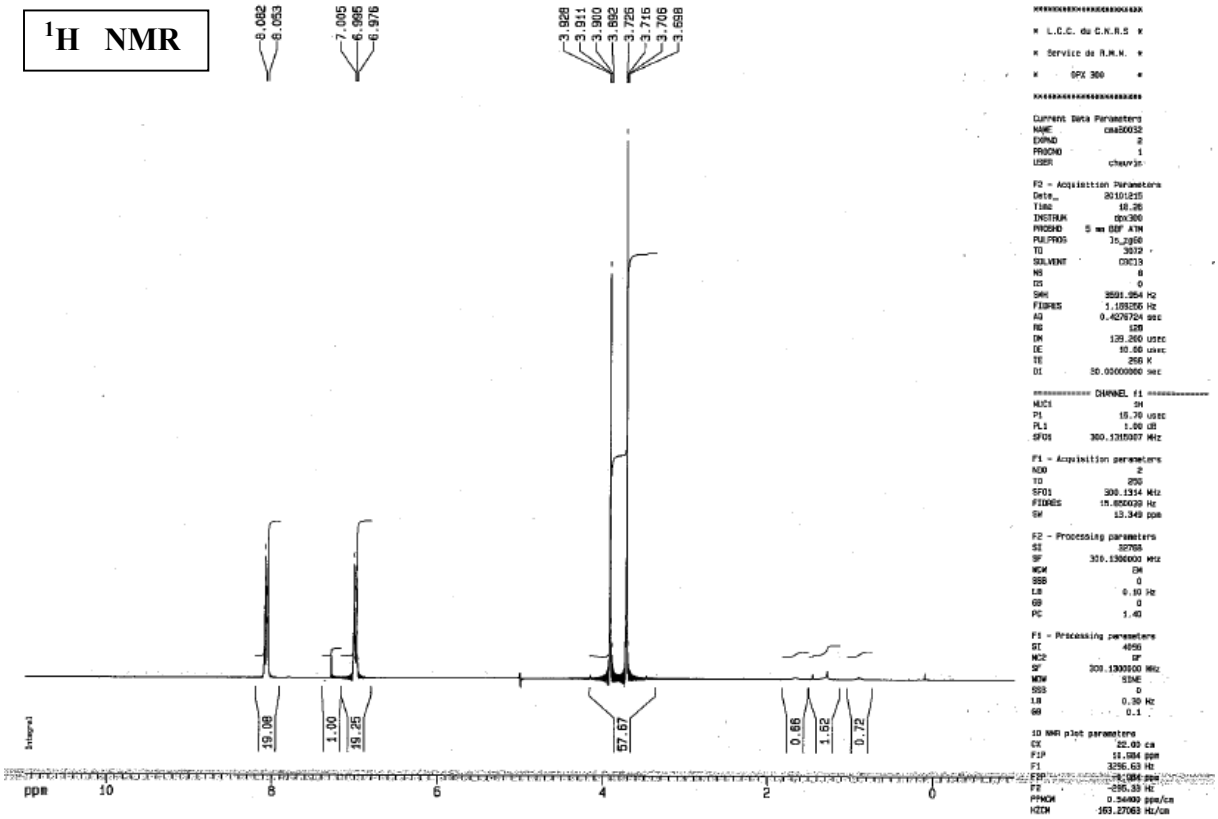
HRMS (DCI/CH_4)



Minimum:				-1.5					
Maximum:		1.3	5.0	50.0					
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula			
355.0406	355.0405	0.1	0.3	7.5	1172.6	C14	H9	O4	F6
	355.0394	1.2	3.4	11.5	1837.6	C17	H8	O3	F5
	355.0418	-1.2	-3.4	14.5	3570.7	C19	H9	O5	F2
	355.0407	-0.1	-0.3	18.5	4934.7	C22	H8	O4	F
	355.0395	1.1	3.1	22.5	6561.5	C25	H7	O3	

5b. 4,7-dimethoxy-1,10-bis(4-methoxyphenyl)-4,7-bis(trifluoromethyl)deca-2,5,8-triyne-1,10-dione

^{1}H NMR



Current Data Parameters
NAME cma50032
EXPNO 2
PROCNO 1
USER chauvin

F2 - Acquisition Parameters
Date_ 20101215
Time 18.26
INSTRUM dpx300
PROBHD 5 mm BBF ATM
PULPROG zg30
TD 32672
SOLVENT CDCl3
NS 8
DS 0
SWH 3591.954 Hz
FIDRES 1.100256 Hz
AQ 0.4276724 sec
RG 128
DW 139.200 usec
DE 10.00 usec
TE 298 K
D1 50.00000000 sec

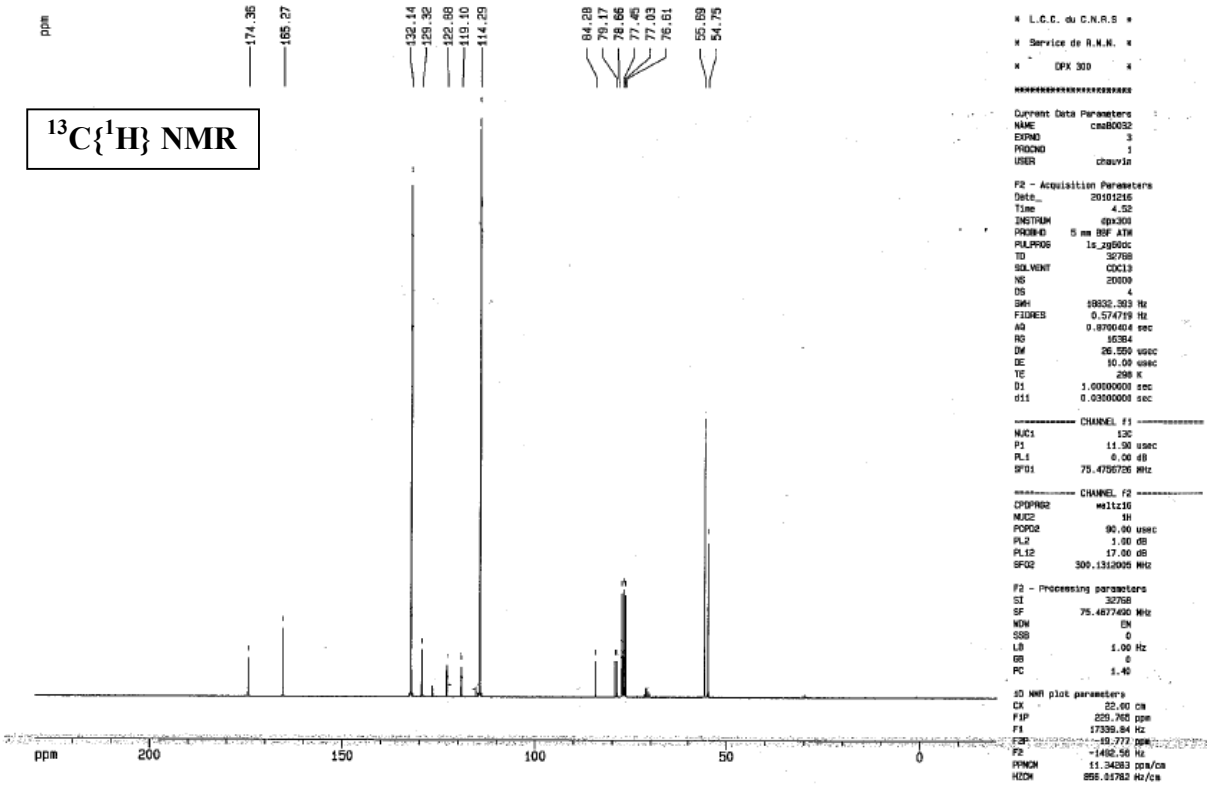
========== CHANNEL f1 ==========
NUC1 1H
P1 15.70 usec
PL1 1.00 dB
SFO1 300.1315007 MHz

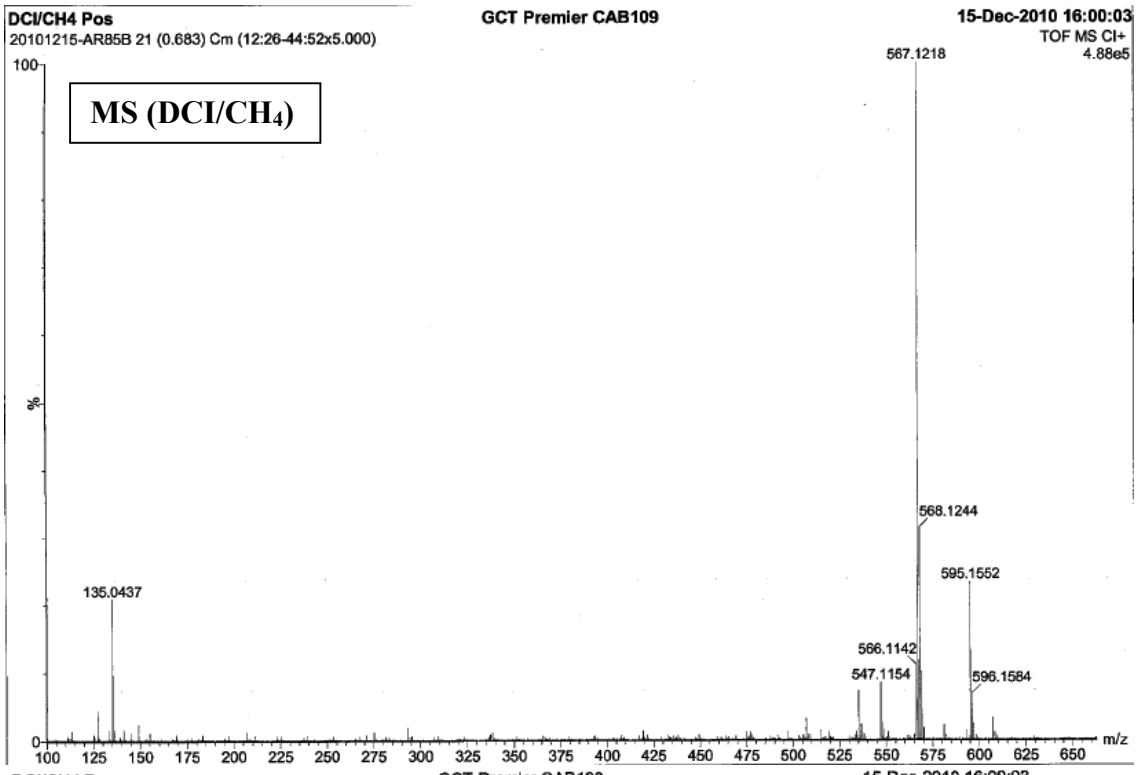
F1 - Acquisition parameters
ND0 2
TD 256
SFO1 300.1314 MHz
FIDRES 15.650039 Hz
SW 13.349 ppm

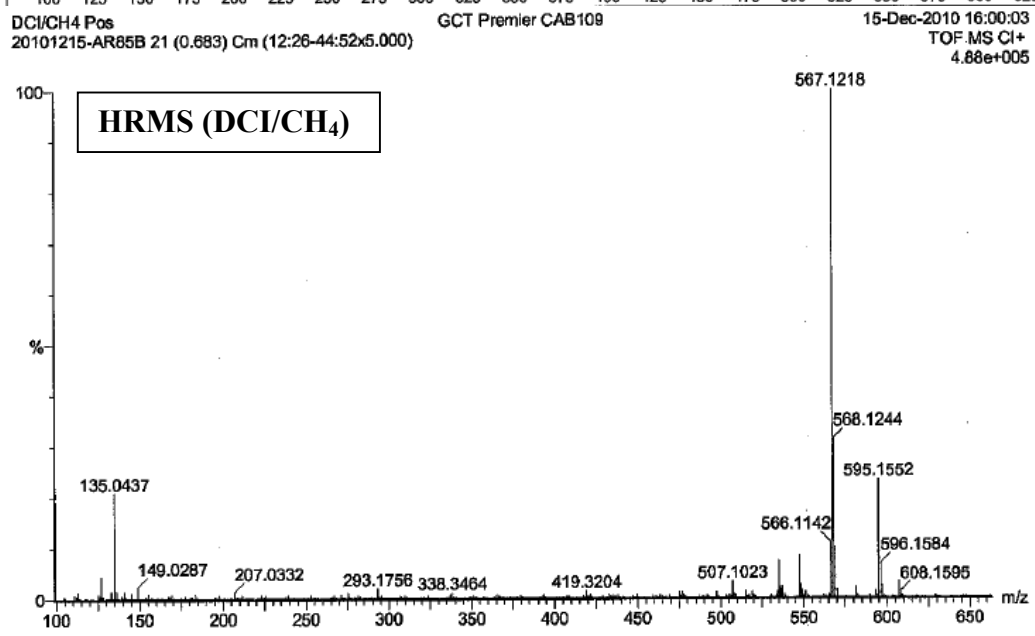
F2 - Processing parameters
SI 32768
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 4096
MC2 QF
SF 300.1300000 MHz
WDW SINE
SSB 0
LB 0.30 Hz
GB 0.1

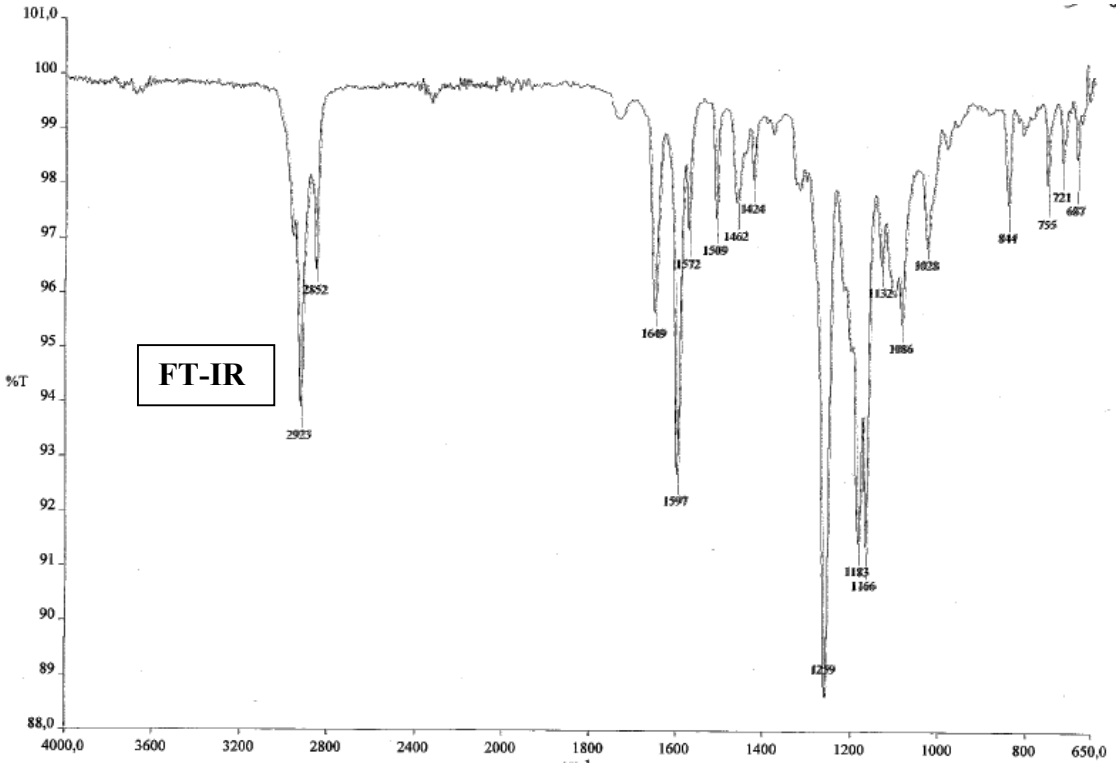
1D NMR plot parameters
CX 22.00 cm
F1P 10.084 ppm
F1 3256.63 Hz
F2P -0.084 ppm
F2 -295.33 Hz
PPMCM 0.54400 ppm/cm
HZCM 163.27063 Hz/cm


$^{13}C\{^{1}H\}$ NMR
ppm
174.36
165.27
132.14
129.32
122.88
119.10
114.29
84.28
79.17
78.66
77.45
77.03
76.61
55.69
54.75
ppm 200 150 100 50 0
* L.C.C. du C.N.R.S *
* Service de R.M.N. *
* DPX 300 *
Current Data Parameters
NAME cma80032
EXPNO 3
PROCNO 1
USER chauvin
F2 - Acquisition Parameters
Date_ 20101216
Time 4.52
INSTRUM dpx300
PROBHD 5 mm BBF ATM
PULPROG ls_zg60c
TD 32768
SOLVENT CDCl3
NS 20000
DS 4
SWH 18832.393 Hz
FIDRES 0.574719 Hz
AQ 0.8700404 sec
RG 16384
DW 26.550 usec
DE 10.00 usec
TE 298 K
D1 1.00000000 sec
d11 0.03000000 sec
CHANNEL f1
NUC1 13C
P1 11.90 usec
PL1 0.00 dB
SFO1 75.4756726 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 1.00 dB
PL12 17.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 22.60 cm
F1P 229.765 ppm
F1 17339.84 Hz
F2P -19.772 ppm
F2 -1492.56 Hz
PPMCM 11.34383 ppm/cm
HZCM 856.01782 Hz/cm
^{19}F NMR
ppm
-78.55
ppm 80 60 40 20 0 -20 -40 -60 -80
* L.C.C. du C.N.R.S *
* Service de R.M.N. *
* DPX 300 *
Current Data Parameters
NAME cma80032
EXPNO 1
PROCNO 1
USER chauvin
F2 - Acquisition Parameters
Date_ 20101215
Time 18.19
INSTRUM dpx300
PROBHD 5 mm BBF ATM
PULPROG ls_zg60
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 56497.176 Hz
FIDRES 0.862078 Hz
AQ 0.5800436 sec
RG 574.7
DW 8.850 usec
DE 10.00 usec
TE 298 K
D1 1.00000000 sec
CHANNEL f1
NUC1 19F
P1 10.50 usec
PL1 -4.00 dB
SFO1 282.4043552 MHz
F2 - Processing parameters
SI 65536
SF 282.4043550 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 22.00 cm
F1P 100.030 ppm
F1 28248.80 Hz
F2P -100.028 ppm
F2 -28248.38 Hz
PPMCM 9.09353 ppm/cm
HZCM 2568.05347 Hz/cm

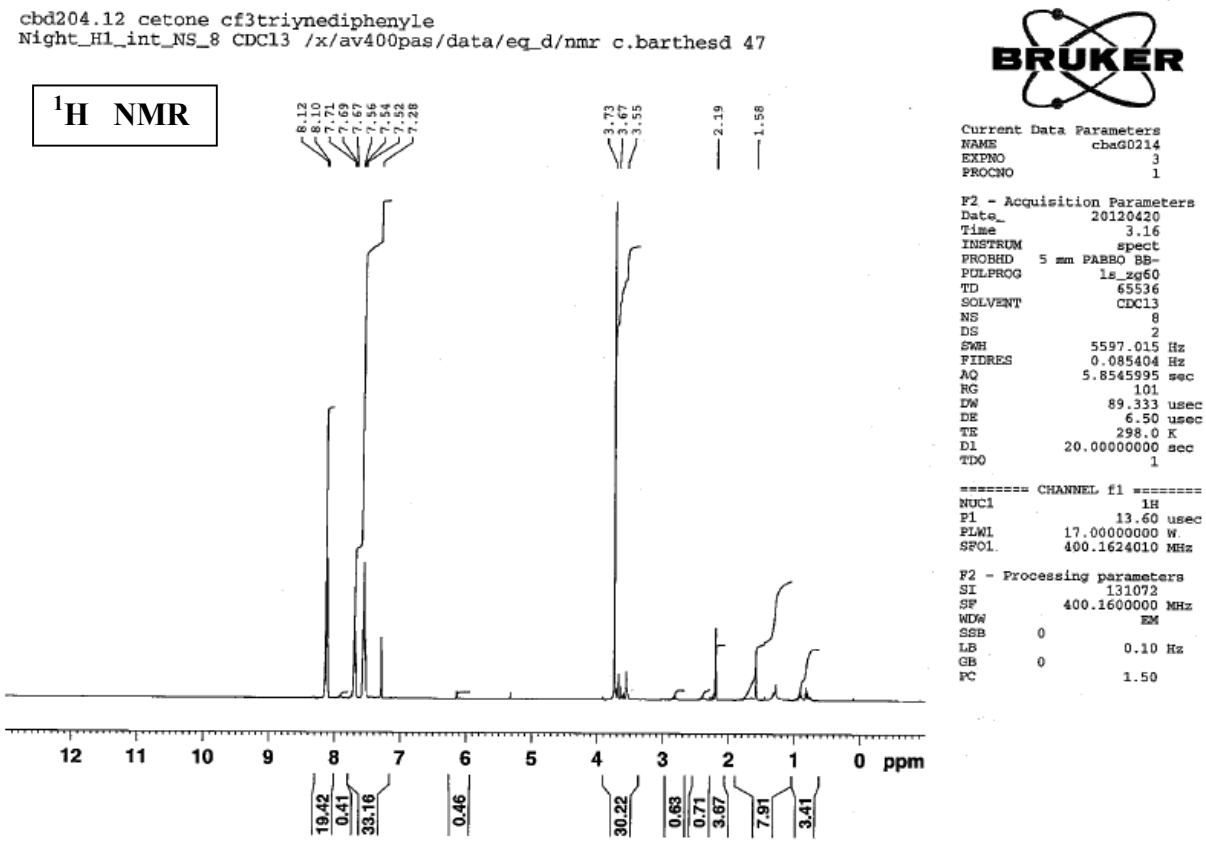




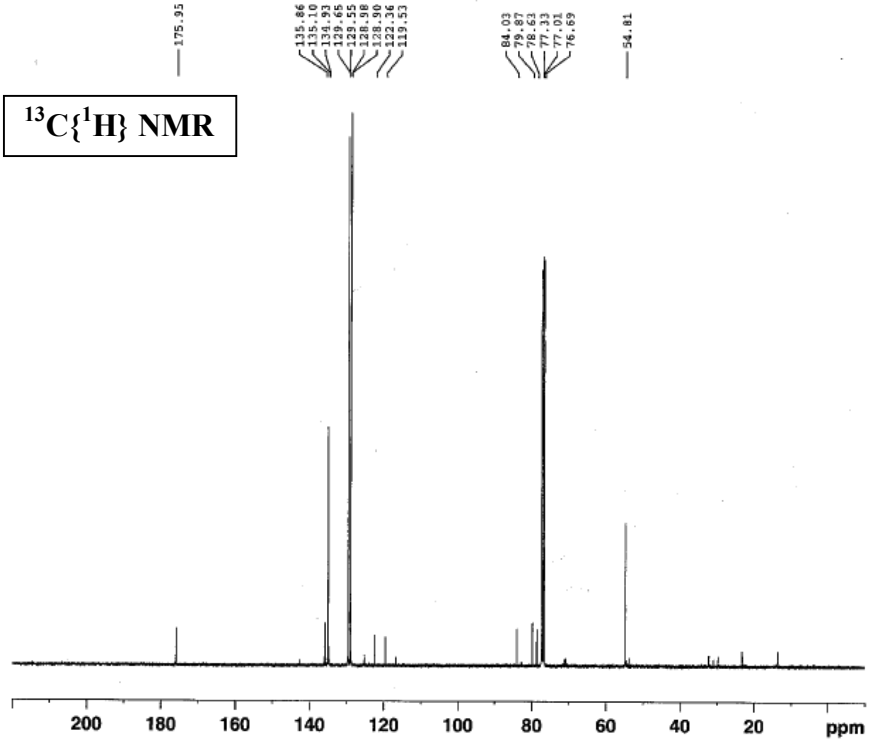
Minimum:				-1.5					
Maximum:		10.0	5.0	50.0					
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula			
567.1218	567.1231	-1.3	-2.3	19.5	3350.8	C31	H20	O5	F5
	567.1242	-2.4	-4.2	15.5	3822.4	C28	H21	O6	F6
	567.1219	-0.1	-0.2	23.5	4145.5	C34	H19	O4	F4
	567.1244	-2.6	-4.6	26.5	5016.9	C36	H20	O6	F
	567.1208	1.0	1.8	27.5	6082.6	C37	H18	O3	F3
	567.1232	-1.4	-2.5	30.5	7803.9	C39	H19	O5	
	567.1197	2.1	3.7	31.5	9007.5	C40	H17	O2	F2



5c. 4,7-dimethoxy-1,10-diphenyl-4,7-bis(trifluoromethyl)deca-2,5,8-triyne-1,10-dione



cbd204.12 cetone cf3triynediphenyle
Night_C13_DECOUPLE_H1_NS_5000 CDCl3 /x/av400pas/data/eq_d/nmr c.barthesd 47



$^{13}C\{^{1}H\}$ NMR

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Current Data Parameters
NAME                  cbaG0214
EXPNO                        2
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20120420
Time                      3.10
INSTRUM                  spect
PROBHD     5 mm PABBO BB-
PULPROG               ls_zgdc60
TD                        65536
SOLVENT                   CDCl3
NS                         5000
DS                            4
SWH                   23148.148 Hz
FIDRES                0.353213 Hz
AQ                    1.4156276 sec
RG                         2050
DW                       21.600 usec
DE                        6.50 usec
TE                       298.0 K
D1                    1.00000000 sec
D11                   0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                        13C
P1                         9.00 usec
PLW1                70.00000000 W
SFO1               100.6308781 MHz

======== CHANNEL f2 ========
CPDPRG2                  waltz16
NUC2                         1H
PCPD2                     90.00 usec
PLW2                17.00000000 W
PLW12                0.38819000 W
SFO2               400.1616006 MHz

F2 - Processing parameters
SI                        131072
SF                 100.6203130 MHz
WDW                           EM
SSB                0
LB                         1.00 Hz
GB                 0
PC                         1.50
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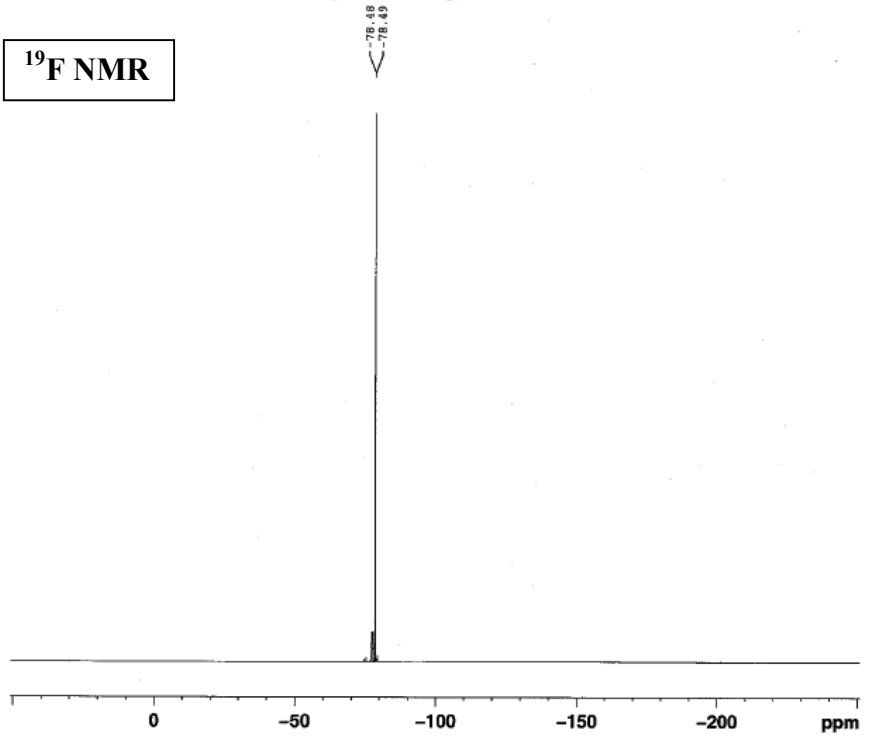
cbd204.12 cetone cf3triynediphenyle
Night_F19_NS_40 CDCl3 /x/av400pas/data/eq_d/nmr c.barthesd 47



^{19}F NMR

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Current Data Parameters
NAME                  cbaG0214
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20120419
Time                     23.41
INSTRUM                  spect
PROBHD     5 mm PABBO BB-
PULPROG                ls_zg60
TD                        131072
SOLVENT                   CDCl3
NS                           40
DS                            4
SWH                  113636.367 Hz
FIDRES                0.866977 Hz
AQ                    0.5767668 sec
RG                          724
DW                        4.400 usec
DE                        6.50 usec
TE                       298.0 K
D1                    1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                        19F
P1                        11.65 usec
PLW1                25.00000000 W
SFO1               376.4889413 MHz

F2 - Processing parameters
SI                         65536
SF                 376.5265940 MHz
WDW                           EM
SSB                0
LB                         0.30 Hz
GB                 0
PC                         1.00
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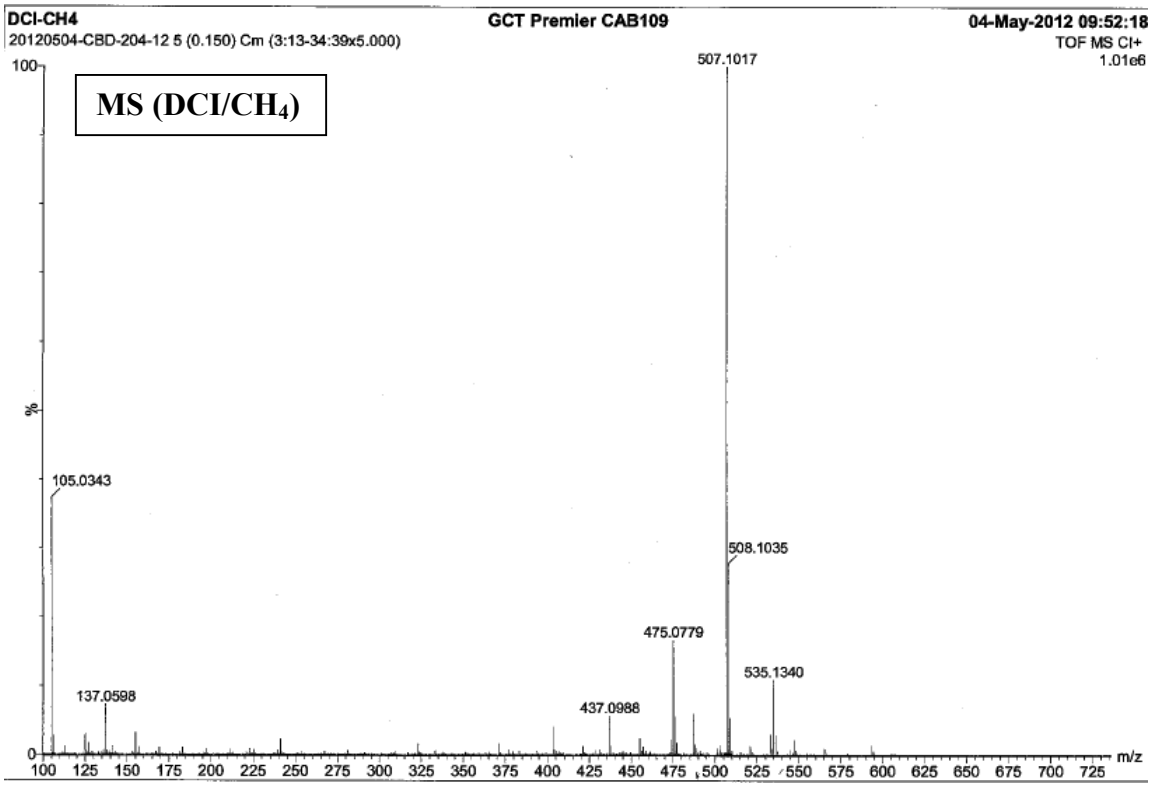
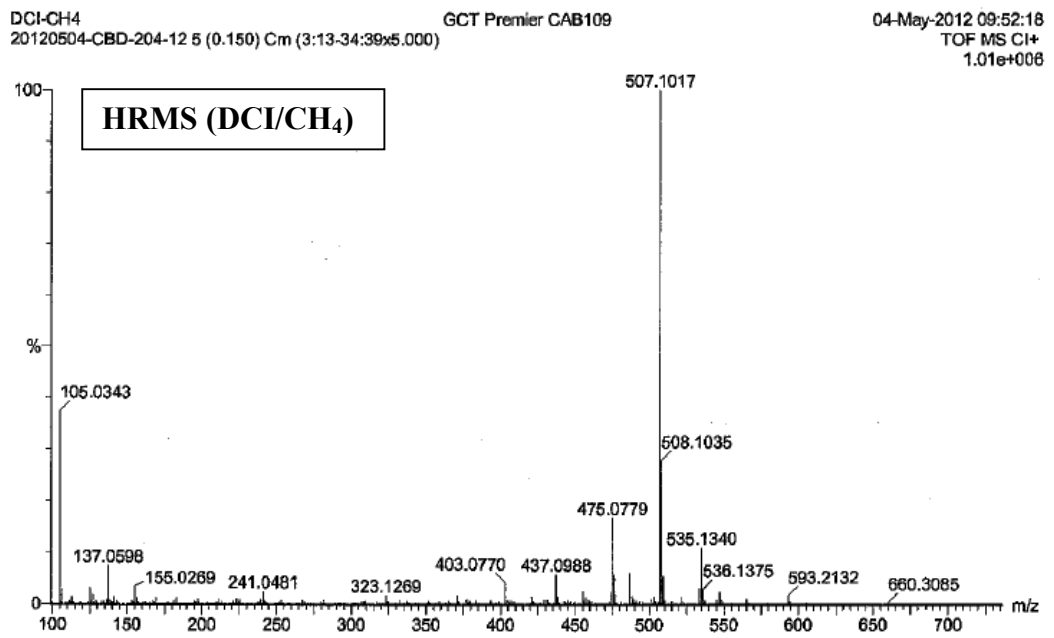
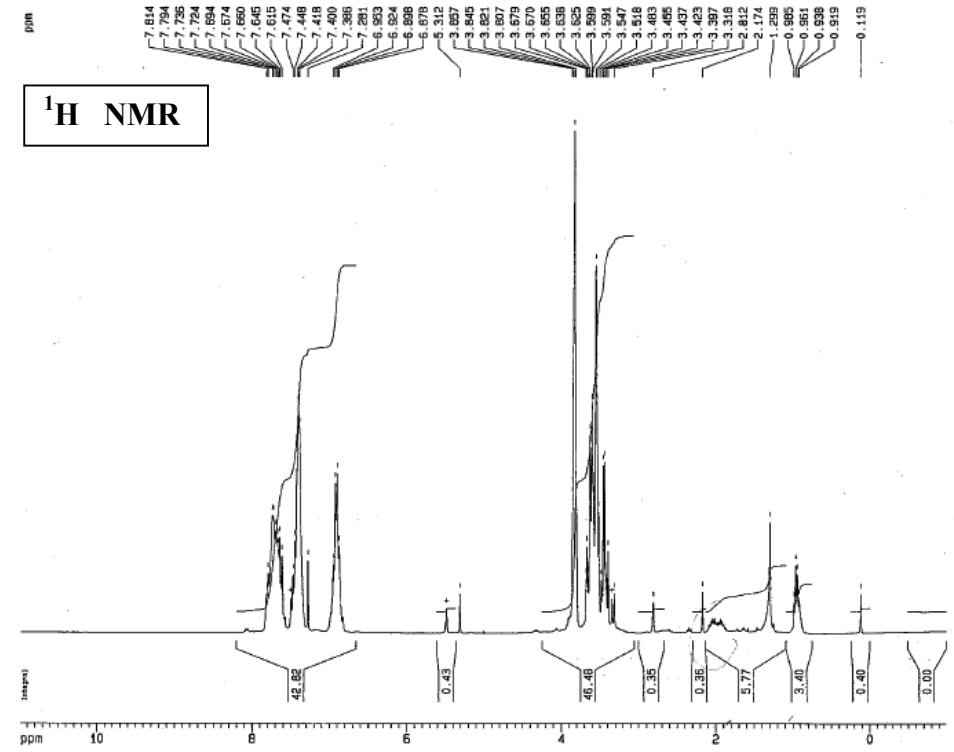




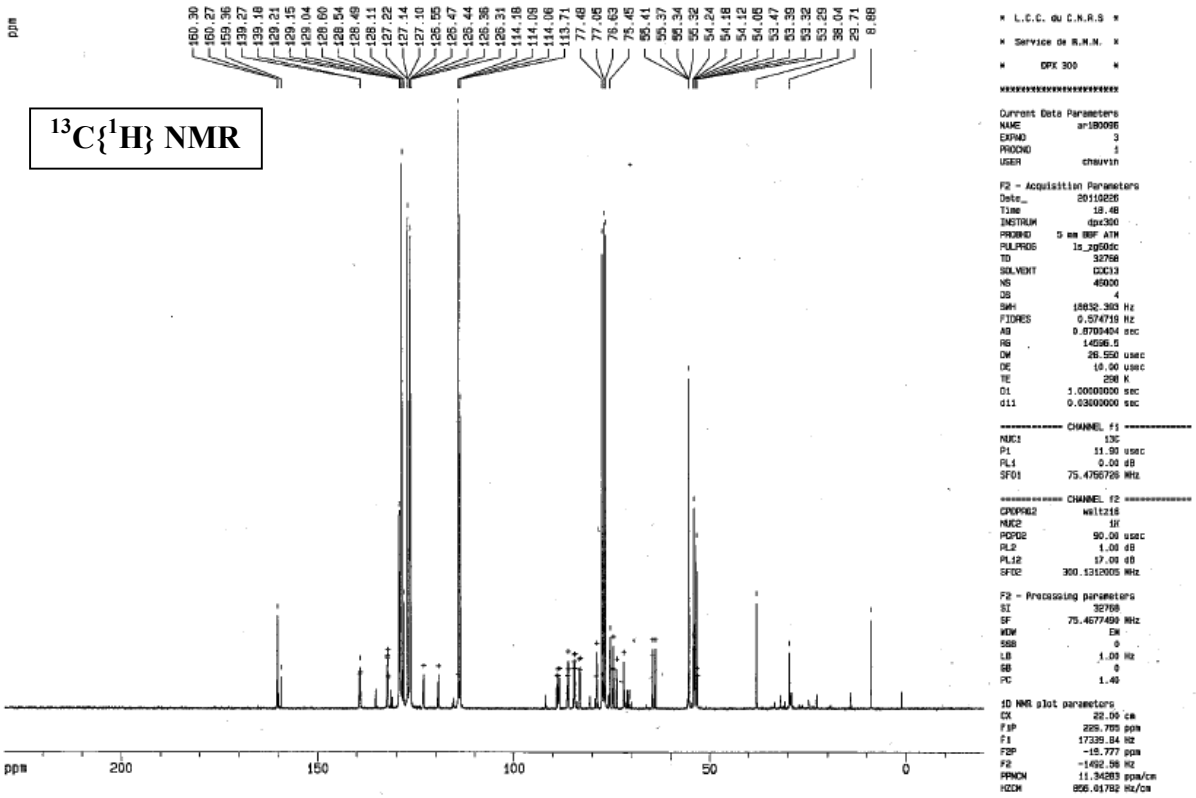
Minimum:				-1.5		
Maximum:		1.3	5.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
507.1017	507.1031	-1.4	-2.8	15.5	595.8	C26 H17 O4 F6
	507.0995	2.2	4.3	16.5	1550.5	C27 H15 O F8
	507.1007	1.0	2.0	12.5	1844.7	C24 H16 O2 F9
	507.1020	-0.3	-0.6	19.5	3266.5	C29 H16 O3 F5
	507.1018	-0.1	-0.2	8.5	5799.3	C21 H17 O3 F10
	507.1008	0.9	1.8	23.5	9095.6	C32 H15 O2 F4
	507.1033	-1.6	-3.2	26.5	16305.2	C34 H16 O4 F
	507.0997	2.0	3.9	27.5	17704.9	C35 H14 O F3

7b. **4,7,13,16-tetramethoxy-1,10-bis(4-methoxyphenyl)-4,7-diphenyl-13,16-bis (trifluoromethyl)cyclooctadeca-2,5,8,11,14,17-hexayne-1,10-diol**

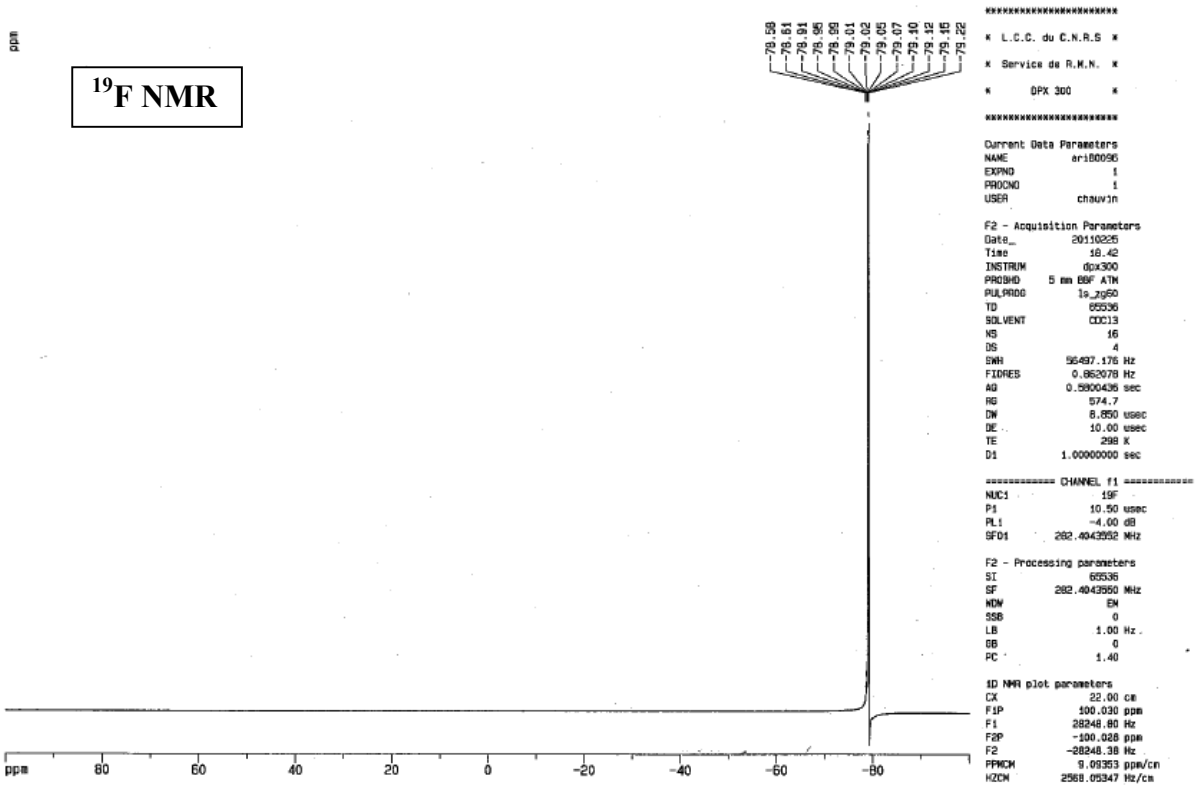
^{1}H NMR



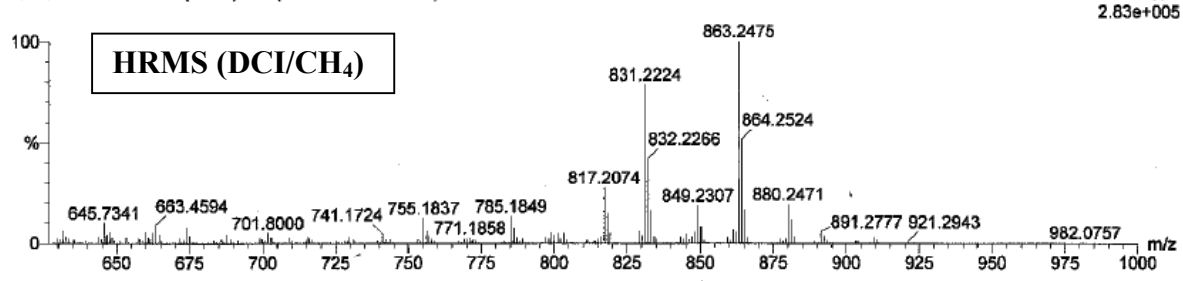
^{13}C{^{1}H} NMR



^{19}F NMR



HRMS (DCI/CH_4)

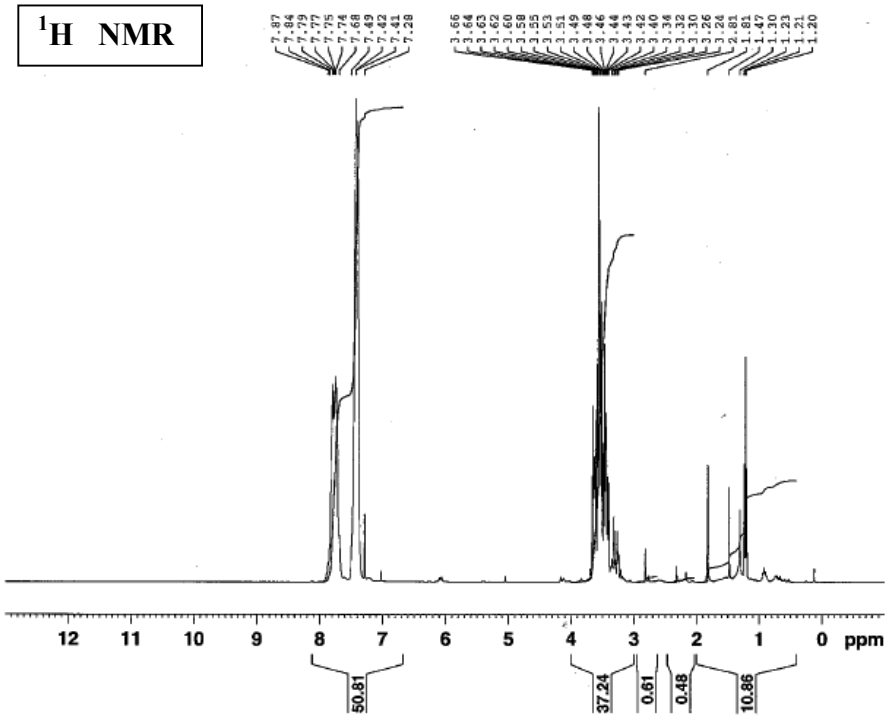


Minimum:				-1.5					
Maximum:		50.0	10.0	50.0					
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula			
881.2552	881.2549	0.3	0.3	28.5	7720.1	C50	H39	O8	F6 — MH+
	881.2538	1.4	1.6	32.5	8489.5	C53	H38	O7	F5
	881.2574	-2.2	-2.5	31.5	8508.4	C52	H40	O10	F3
	881.2490	6.2	7.0	37.5	9226.5	C57	H35	O3	F6
	881.2526	2.6	3.0	36.5	9232.8	C56	H37	O6	F4
	881.2562	-1.0	-1.1	35.5	9244.2	C55	H39	O9	F2
	881.2515	3.7	4.2	40.5	9937.1	C59	H36	O5	F3
	881.2551	0.1	0.1	39.5	9942.0	C58	H38	O8	F
	881.2479	7.3	8.3	41.5	9946.1	C60	H34	O2	F5
	881.2539	1.3	1.5	43.5	10600.8	C61	H37	O7	
	881.2503	4.9	5.6	44.5	10610.4	C62	H35	O4	F2
	881.2468	8.4	9.5	45.5	10624.8	C63	H33	O	F4
	881.2492	6.0	6.8	48.5	11243.1	C65	H34	O3	F

7c. 4,7,13,16-tetramethoxy-1,4,7,10-tetraphenyl-13,16-bis(trifluoromethyl)cyclooctadeca-2,5,8,11,14,17-hexayne-1,10-diol

cbd208.12.1.1.caract
Night_H1_int_NS_8 CDCl3 /x/av400pas/data/eq_d/nmr c.barthesd 44

^{1}H NMR



Current Data Parameters
NAME cbaG0221
EXPNO 3
PROCNO 1

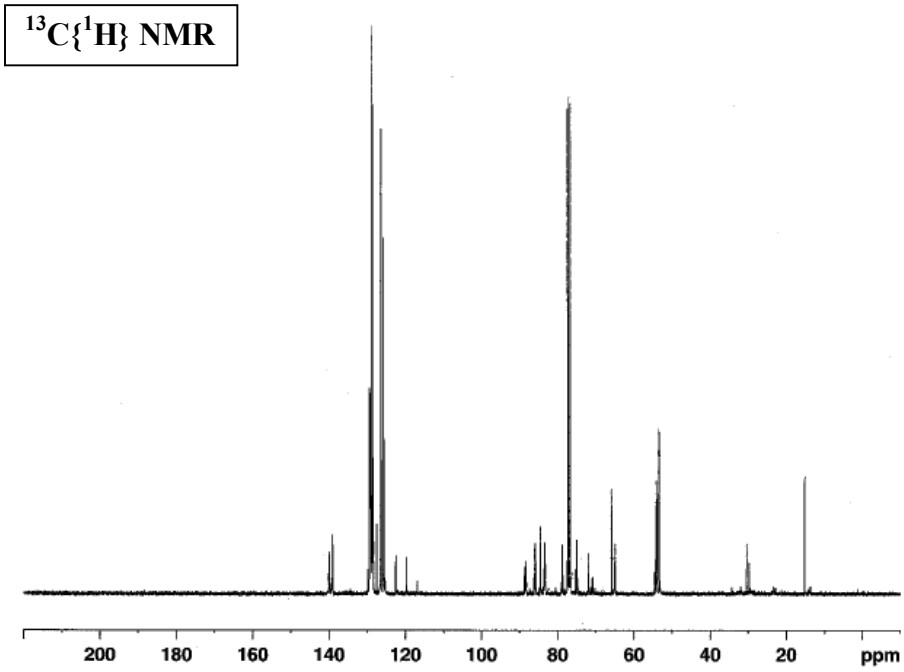
F2 - Acquisition Parameters
Date_ 20120426
Time 19.15
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG ls_zg60
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 5597.015 Hz
FIDRES 0.085404 Hz
AQ 5.8545995 sec
RG 36
DW 89.333 usec
DE 6.50 usec
TE 298.0 K
D1 20.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.60 usec
PLW1 17.00000000 W
SFO1 400.1624010 MHz

F2 - Processing parameters
SI 131072
SF 400.1600000 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.50

cbd208.12.1.1.caract
Night_C13_DECOUPLE_H1_NS_5000 CDCl3 /x/av400pas/data/eq_d/nmr c.barthesd 44

^{13}C{^{1}H} NMR



Current Data Parameters
NAME cbaG0221
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120426
Time 22.43
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG ls_zgdc60
TD 65536
SOLVENT CDCl3
NS 5000
DS 4
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 2050
DW 21.600 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

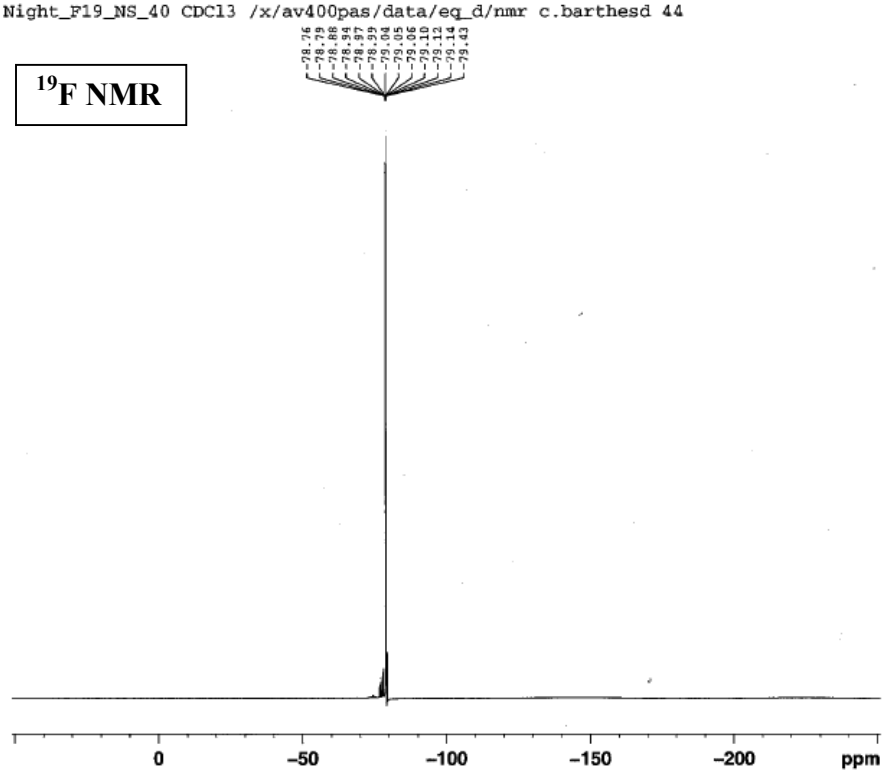
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PLW1 70.00000000 W
SFO1 100.6308781 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 17.00000000 W
PLW12 0.38819000 W
SFO2 400.1616006 MHz

F2 - Processing parameters
SI 131072
SF 100.6203130 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.50

cbd208.12.1.1.caract
Night_F19_NS_40 CDCl3 /x/av400pas/data/eq_d/nmr c.barthesd 44

^{19}F NMR



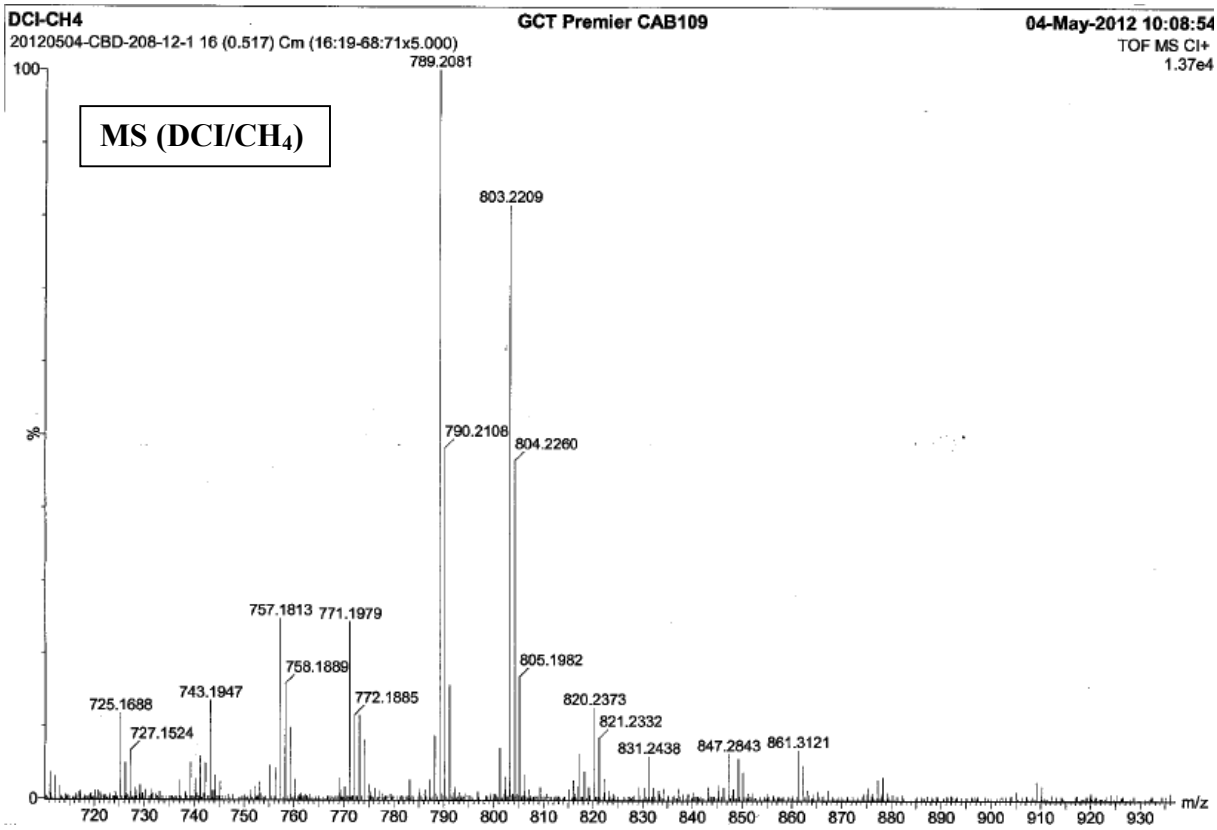
Current Data Parameters
NAME cbaG0221
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120426
Time 22.45
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG ls_zg60
TD 131072
SOLVENT CDCl3
NS 40
DS 4
SWH 113636.367 Hz
FIDRES 0.866977 Hz
AQ 0.5767668 sec
RG 812
DW 4.400 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1

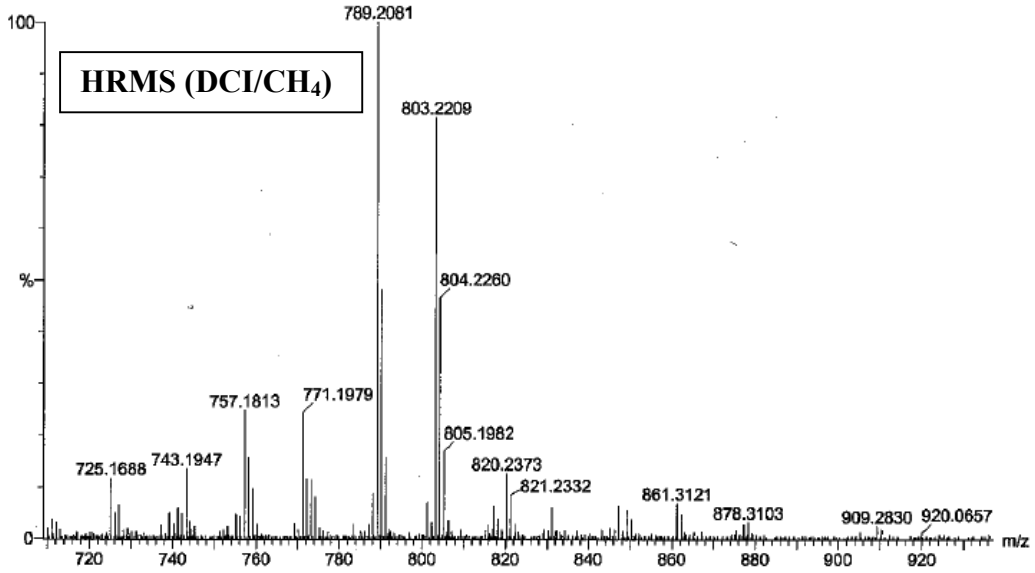
======== CHANNEL f1 ========
NUC1 19F
P1 11.65 usec
PLW1 25.00000000 W
SFO1 376.4889413 MHz

F2 - Processing parameters
SI 65536
SF 376.5265940 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

MS (DCI/CH_4)

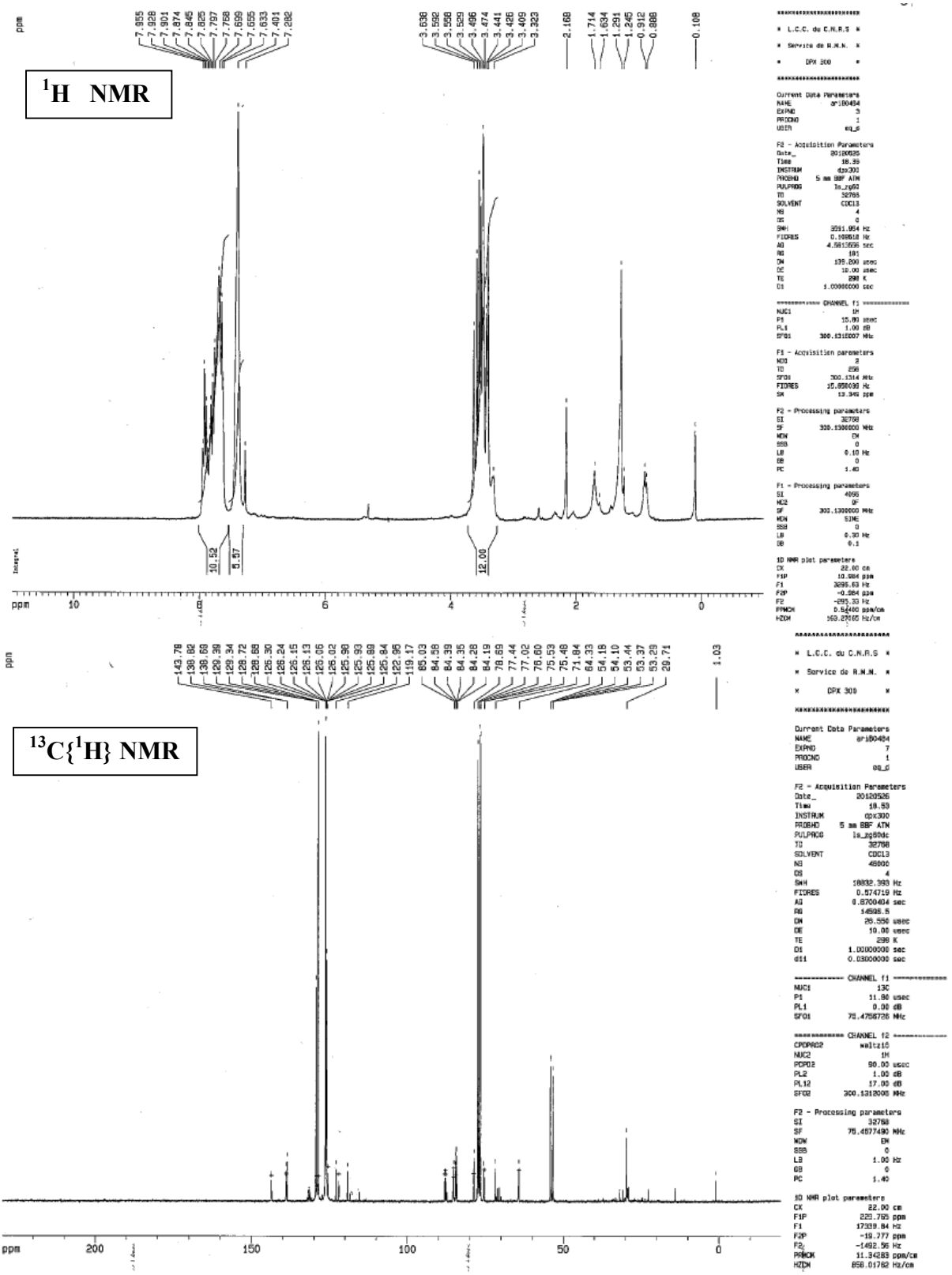


DCI-CH4 GCT Premier CAB109 04-May-2012 10:08:54
20120504-CBD-208-12-1 16 (0.517) Cm (16:19-68:71x5.000) TOF MS CI+
1.37e+004

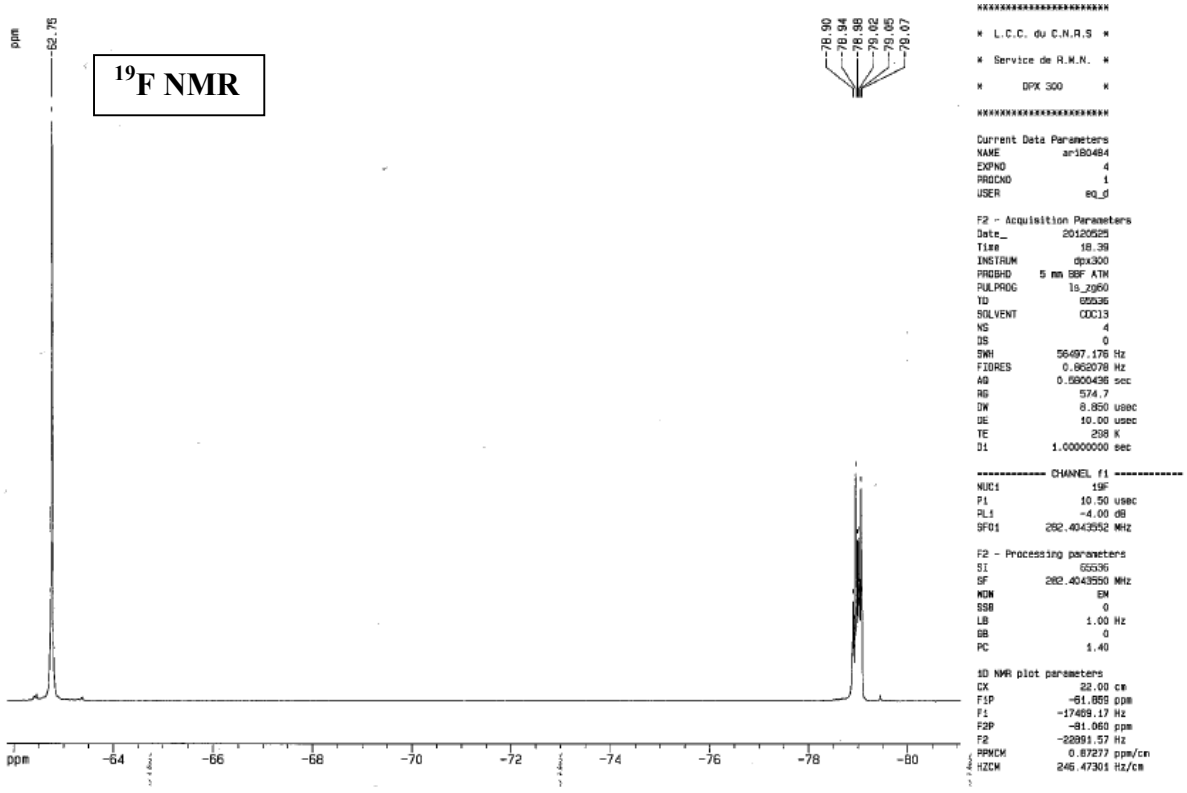


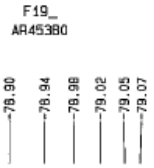
Minimum:				-1.5					
Maximum:		1.3	5.0	50.0					
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula			
803.2209	803.2209	0.0	0.0	37.5	29.4	C54	H31	O3	F4
	803.2234	-2.5	-3.1	40.5	32.6	C56	H32	O5	F
	803.2173	3.6	4.5	38.5	34.8	C55	H29	F6	
	803.2198	1.1	1.4	41.5	38.2	C57	H30	O2	F3
	803.2221	-1.2	-1.5	33.5	41.1	C51	H32	O4	F5
	803.2186	2.3	2.9	45.5	66.2	C60	H29	O	F2
	803.2232	-2.3	-2.9	29.5	75.0	C48	H33	O5	F6
	803.2175	3.4	4.2	49.5	111.6	C63	H28	F	
	803.2245	-3.6	-4.5	36.5	2875.4	C53	H33	O6	F2

7d. **4,7,13,16-tetramethoxy-4,7-diphenyl-13,16-bis(trifluoromethyl)-1,10-bis[4-(trifluoromethyl)phenyl]cyclooctadeca-2,5,8,11,14,17-hexayne-1,10-diol**

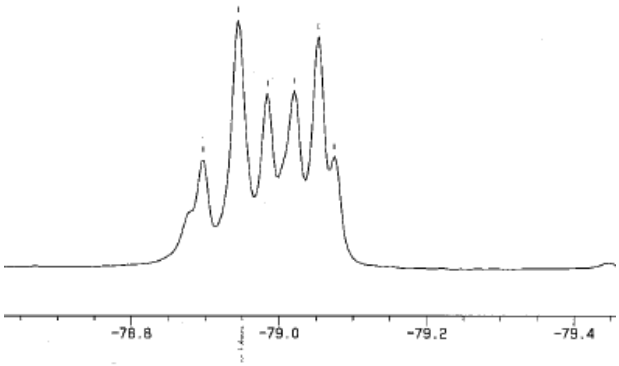


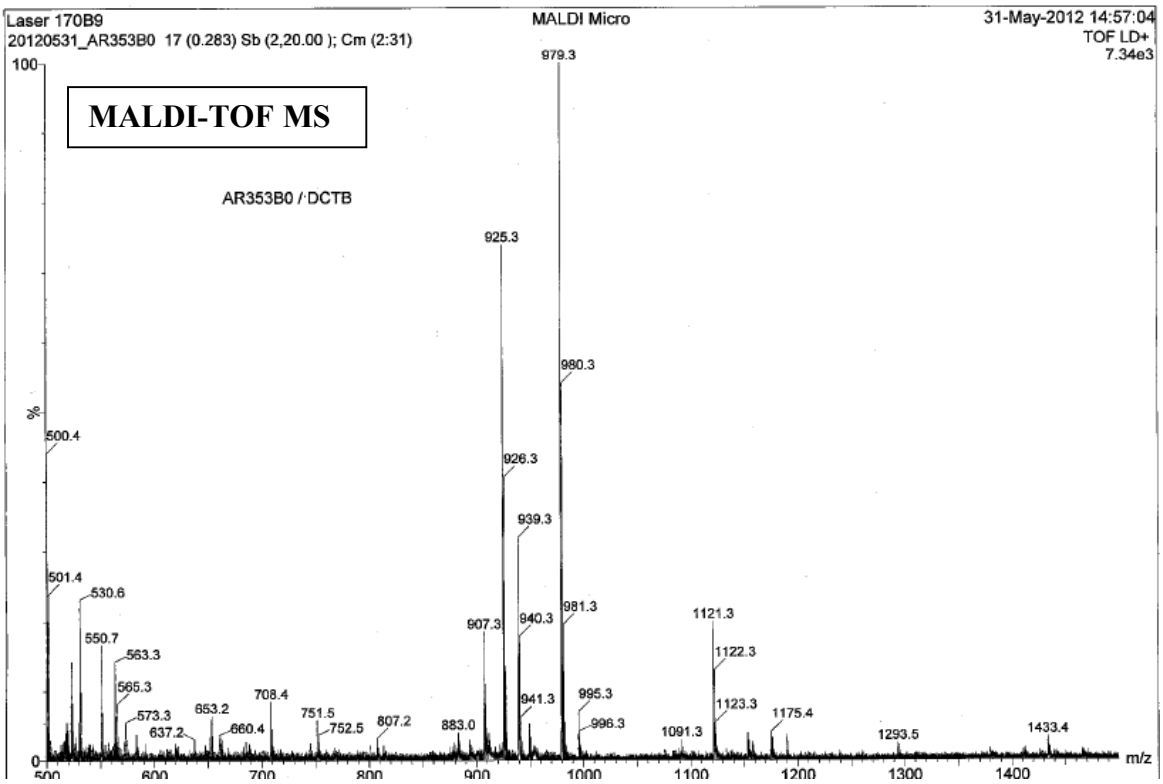
^{19}F NMR

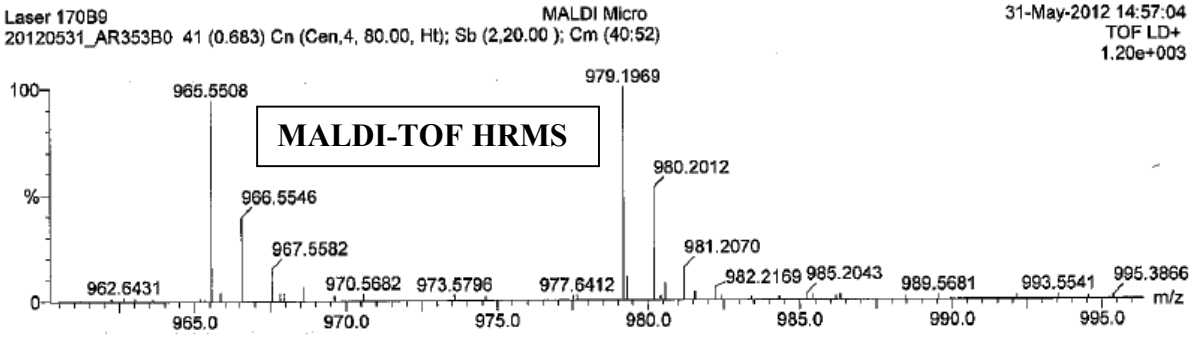




^{19}F NMR

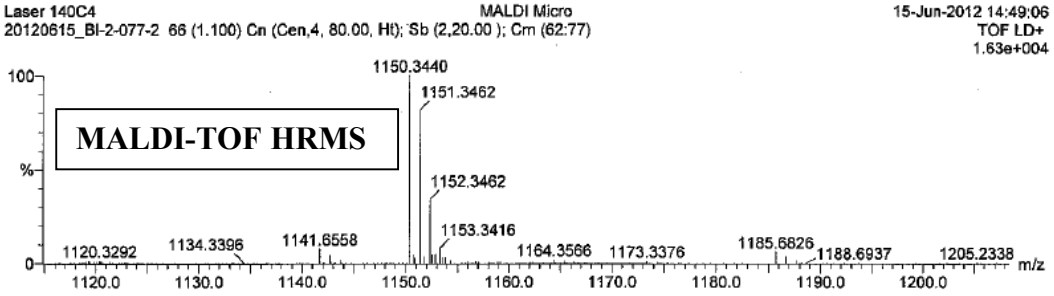






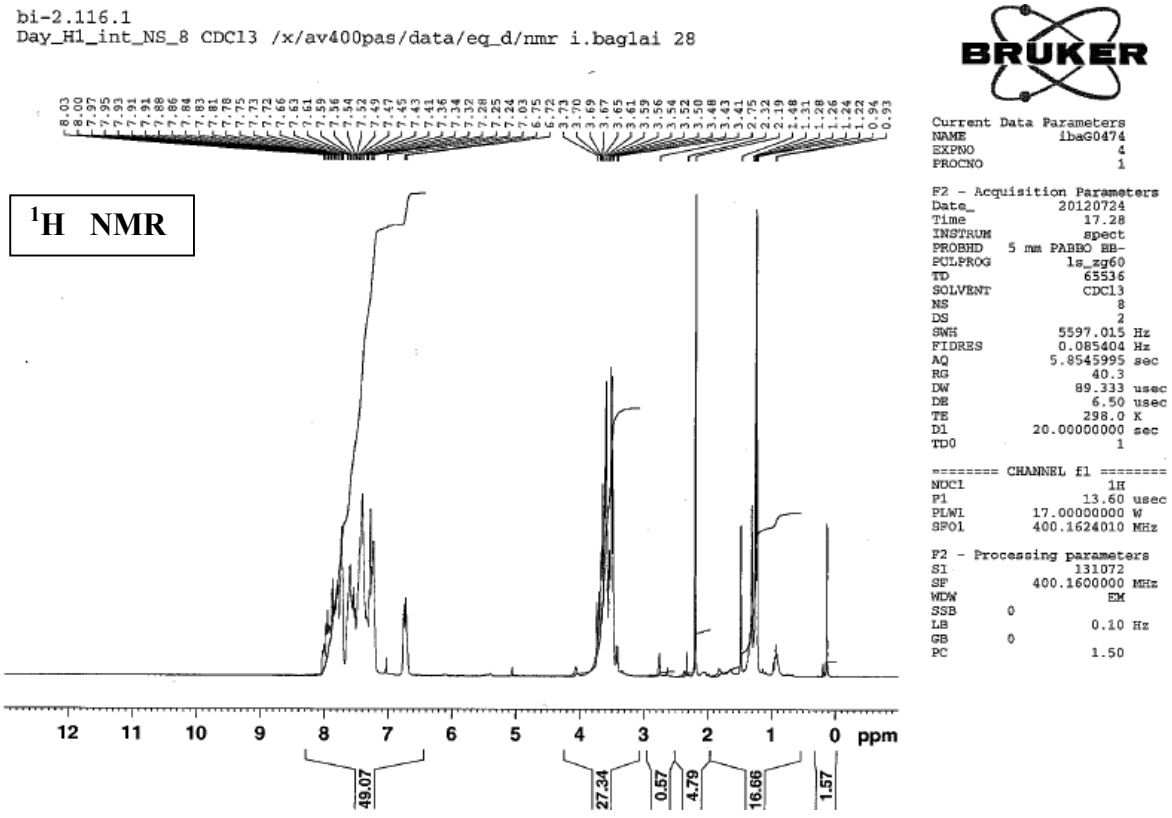
Minimum:				-1.5		
Maximum:		5.0	10.0	70.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
979.1969	979.1916	5.3	5.4	24.5	0.2	C47 H33 O7 F13 23Na
	979.1905	6.4	6.5	28.5	1.9	C50 H32 O6 F12 23Na ←
	979.1894	7.5	7.7	32.5	6.1	C53 H31 O5 F11 23Na
	979.2058	-8.9	-9.1	32.5	7.2	C54 H32 O3 F12 23Na
	979.1918	5.1	5.2	35.5	11.3	C55 H32 O7 F8 23Na
	979.1882	8.7	8.9	36.5	12.4	C56 H30 O4 F10 23Na
	979.2046	-7.7	-7.9	36.5	13.8	C57 H31 O2 F11 23Na
	979.1907	6.2	6.3	39.5	19.4	C58 H31 O6 F7 23Na
	979.1895	7.4	7.6	43.5	29.4	C61 H30 O5 F6 23Na
	979.2059	-9.0	-9.2	43.5	31.7	C62 H31 O3 F7 23Na
	979.2048	-7.9	-8.1	47.5	43.3	C65 H30 O2 F6 23Na

7e. 1,10-bis[4-(9H-carbazol-9-yl)phenyl]-4,7,13,16-tetramethoxy-13,16-diphenyl-4,7-bis(trifluoromethyl)cyclooctadeca-2,5,8,11,14,17-hexayne-1,10-diol



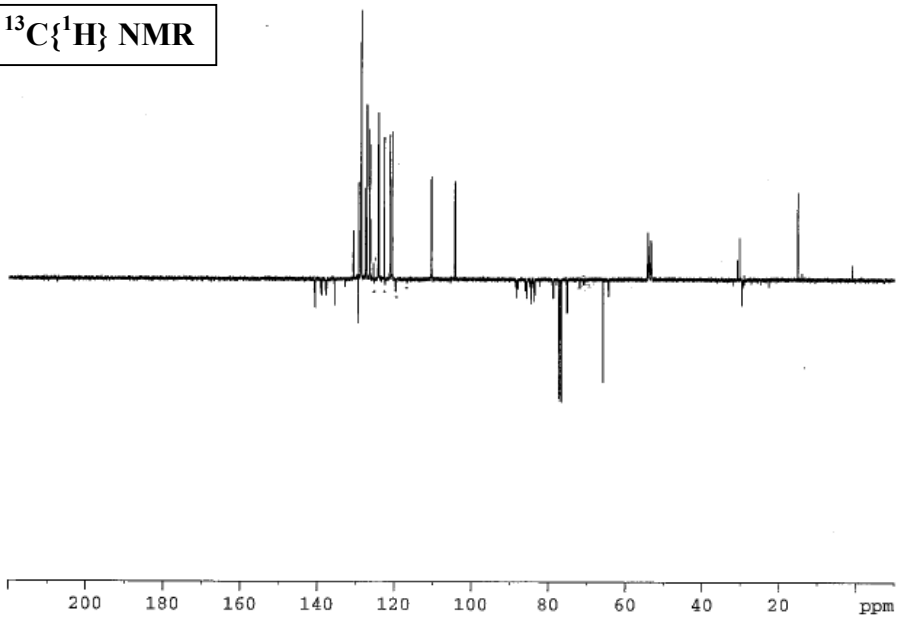
Minimum:				-1.5		
Maximum:		5.0	5.0	55.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
1150.3440	1150.3417	2.3	2.0	47.0	0.3	C72 H48 N2 O6 F6 ←
	1150.3455	-1.5	-1.3	47.5	0.8	C72 H47 N3 O4 F7
	1150.3468	-2.8	-2.4	47.0	4.1	C74 H49 O5 F7
	1150.3480	-4.0	-3.5	43.0	5.8	C71 H50 O6 F8
	1150.3479	-3.9	-3.4	50.5	12.0	C74 H48 N3 O6 F4
	1150.3443	-0.3	-0.3	51.5	13.6	C75 H46 N3 O3 F6
	1150.3466	-2.6	-2.3	43.5	13.7	C69 H48 N3 O5 F8
	1150.3405	3.5	3.0	51.0	14.6	C75 H47 N2 O5 F5
	1150.3407	3.3	2.9	52.5	18.1	C76 H44 N3 F8
	1150.3457	-1.7	-1.5	51.0	27.4	C77 H48 O4 F6
	1150.3495	-5.5	-4.8	51.5	29.3	C77 H47 N O2 F7
	1150.3421	1.9	1.7	52.0	32.3	C78 H46 O F8
	1150.3468	-2.8	-2.4	54.5	45.0	C77 H47 N3 O5 F3
	1150.3394	4.6	4.0	55.0	52.6	C78 H46 N2 O4 F4
	1150.3481	-4.1	-3.6	54.0	68.4	C79 H49 O6 F3
	1150.3445	-0.5	-0.4	55.0	74.0	C80 H47 O3 F5

7f. 1,10-bis[4-(1H-indol-1-yl)phenyl]-4,7,13,16-tetramethoxy-4,7-diphenyl-13,16-bis(trifluoromethyl)cyclooctadeca-2,5,8,11,14,17-hexayne-1,10-diol



bi-2.116.1
Night_C13_JMOD_H1_NS_3000 CDCl3 /x/av400pas/data/eq_d/nmr i.baglai 28

$^{13}C\{^{1}H\}$ NMR



Current Data Parameters
NAME ibaG0474
EXPNO 5
PROCNO 1

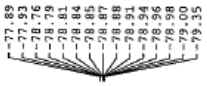
F2 - Acquisition Parameters
Date_ 20120725
Time 8.24
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG 1s_jmod
TD 65536
SOLVENT CDCl3
NS 3000
DS 4
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 2050
DW 21.600 usec
DE 6.50 usec
TE 298.0 K
CNST2 145.0000000
CNST11 1.0000000
D1 1.00000000 sec
D20 0.00689655 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
P2 18.00 usec
PLW1 70.00000000 W
SFO1 100.6308781 MHz

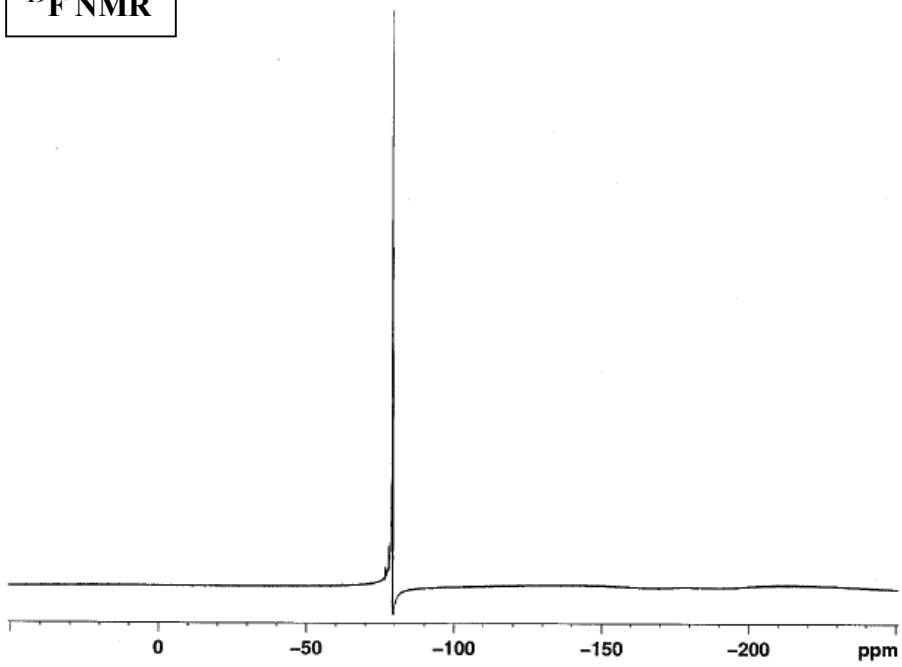
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 17.00000000 W
PLW12 0.38819000 W
SFO2 400.1616006 MHz

F2 - Processing parameters
SI 131072
SF 100.6203130 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.50

bi-2.116.1
Day_F19_NS_40 CDCl3 /x/av400pas/data/eq_d/nmr i.baglai 28



^{19}F NMR

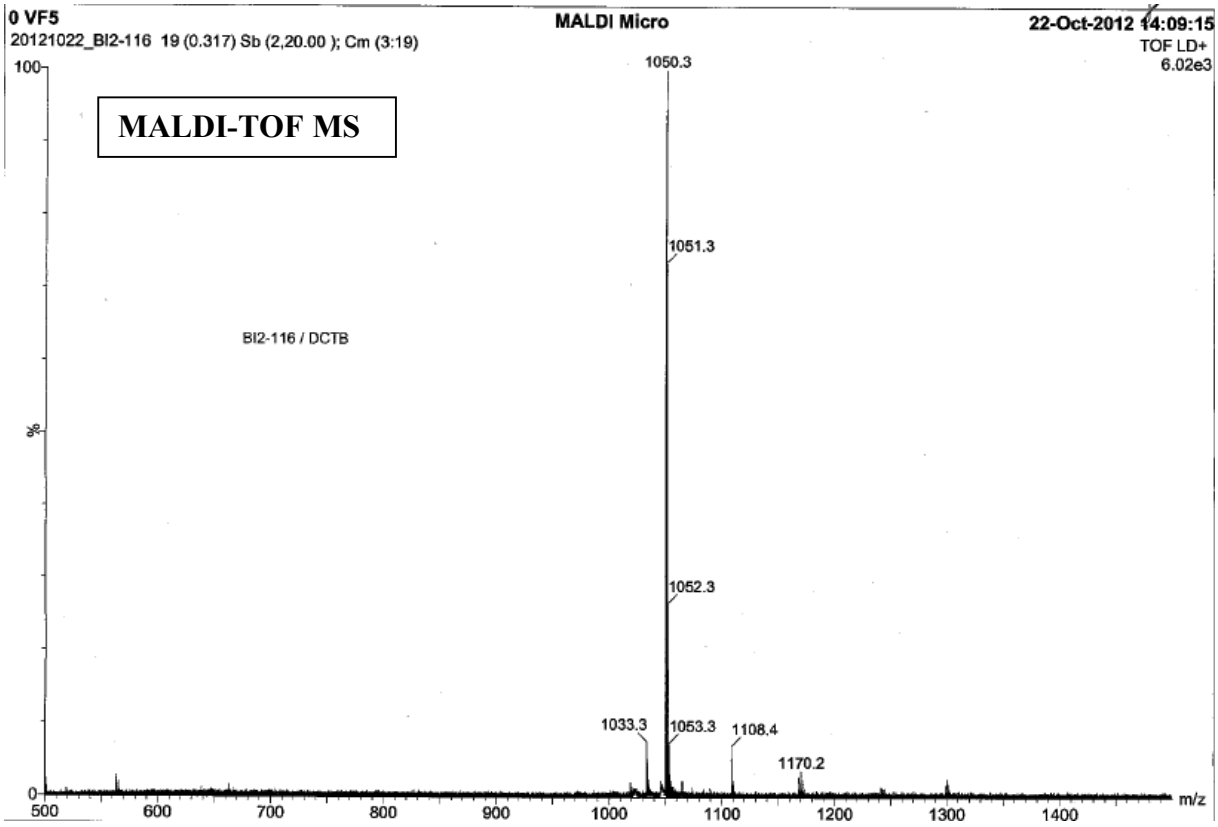


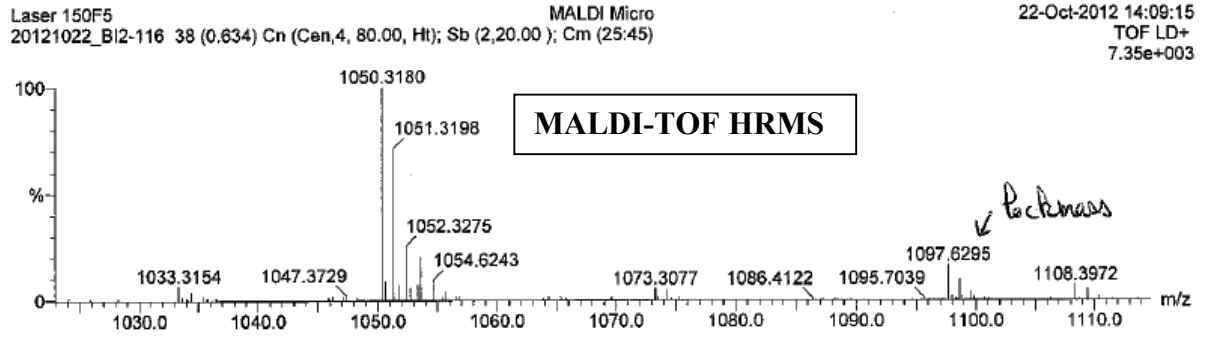
Current Data Parameters
NAME ibaG0474
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120724
Time 17.21
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG 1s_zg60
TD 131072
SOLVENT CDCl3
NS 40
DS 4
SWH 113636.367 Hz
FIDRES 0.866977 Hz
AQ 0.5767658 sec
RG 724
DW 4.400 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 19F
P1 11.65 usec
PLW1 25.00000000 W
SFO1 376.4889413 MHz

F2 - Processing parameters
SI 65536
SF 376.5265940 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

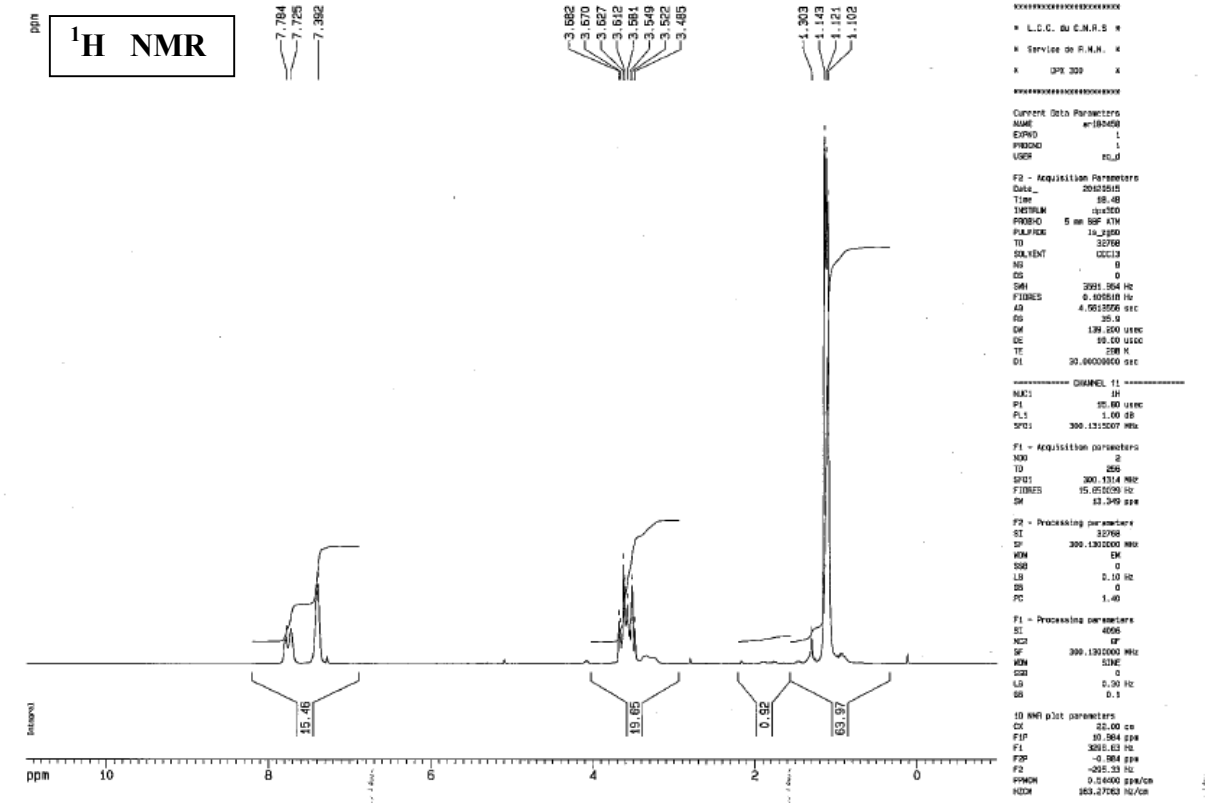




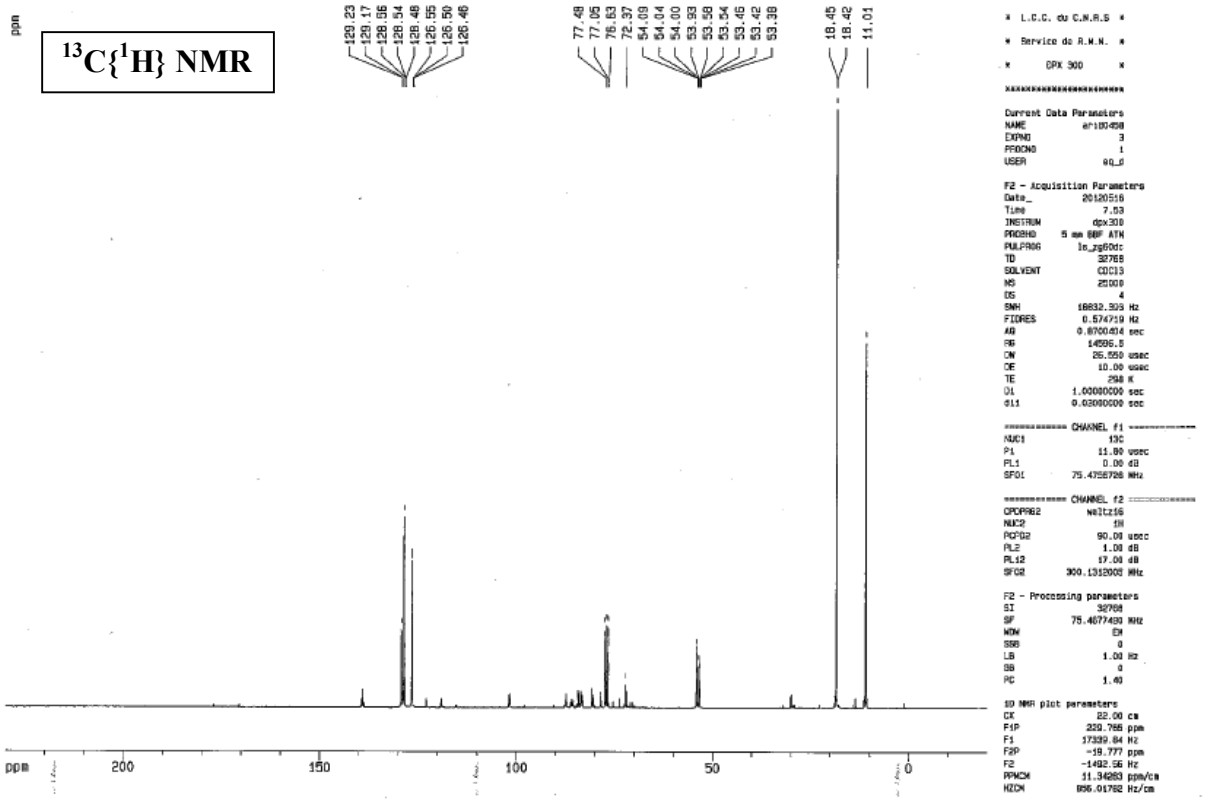
Minimum:				-1.5		
Maximum:		5.0	8.0	45.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
1050.3180	1050.3153	2.7	2.6	37.5	0.9	C61 H44 N3 O5 F8
	1050.3167	1.3	1.2	37.0	1.0	C63 H46 O6 F8
	1050.3241	-6.1	-5.8	36.5	2.2	C62 H47 N O7 F7
	1050.3115	6.5	6.2	37.0	2.3	C61 H45 N2 O7 F7
	1050.3178	0.2	0.2	40.5	3.8	C63 H45 N3 O7 F5
	1050.3142	3.8	3.6	41.5	4.9	C64 H43 N3 O4 F7
	1050.3104	7.6	7.2	41.0	7.2	C64 H44 N2 O6 F6
	1050.3229	-4.9	-4.7	40.5	9.3	C65 H46 N O6 F6
	1050.3193	-1.3	-1.2	41.5	9.7	C66 H44 N O3 F8
	1050.3155	2.5	2.4	41.0	10.8	C66 H45 O5 F7
	1050.3166	1.4	1.3	44.5	18.7	C66 H44 N3 O6 F4
	1050.3218	-3.8	-3.6	44.5	28.6	C68 H45 N O5 F5
	1050.3256	-7.6	-7.2	45.0	29.0	C68 H44 N2 O3 F6
	1050.3180	0.0	0.0	44.0	29.3	C68 H46 O7 F4
	1050.3144	3.6	3.4	45.0	32.7	C69 H44 O4 F6

7g. **4,7,13,16-tetramethoxy-4,7-diphenyl-13,16-bis(trifluoromethyl)-1,10-bis({2-[tris(propan-2-yl)silyl]ethynyl})cyclooctadeca-2,5,8,11,14,17-hexayne-1,10-diol**

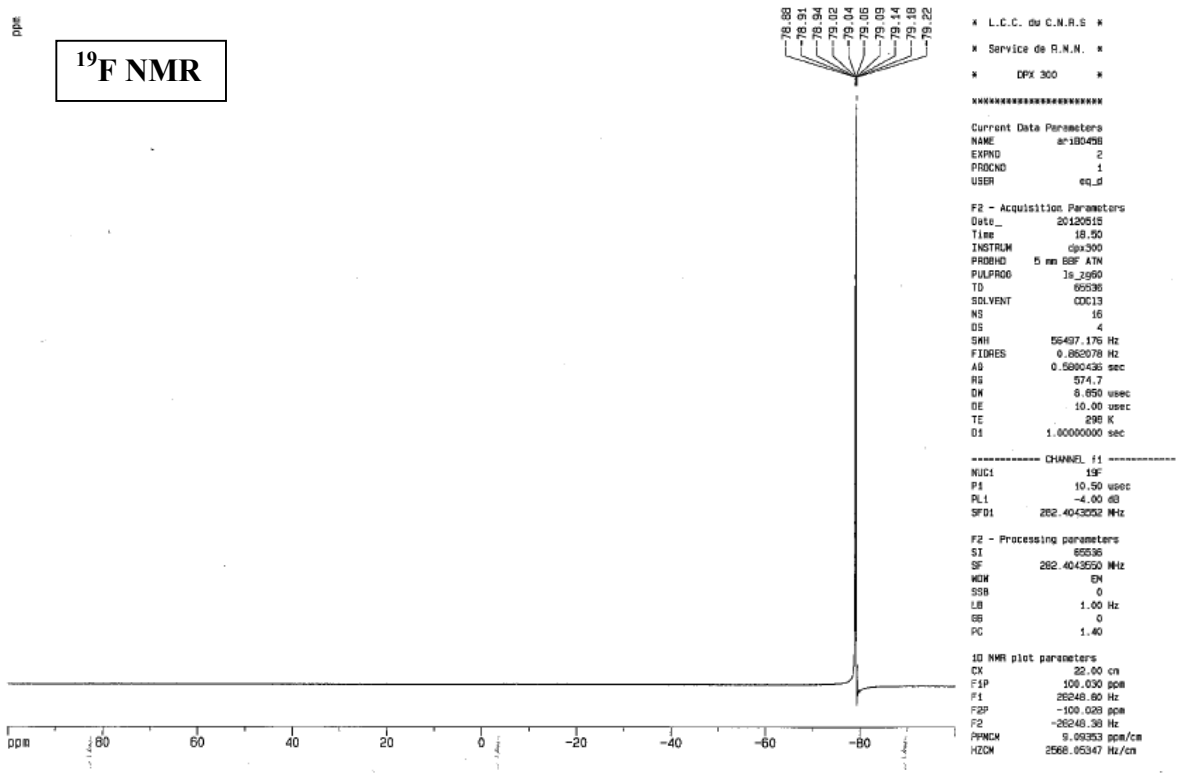
^{1}H NMR



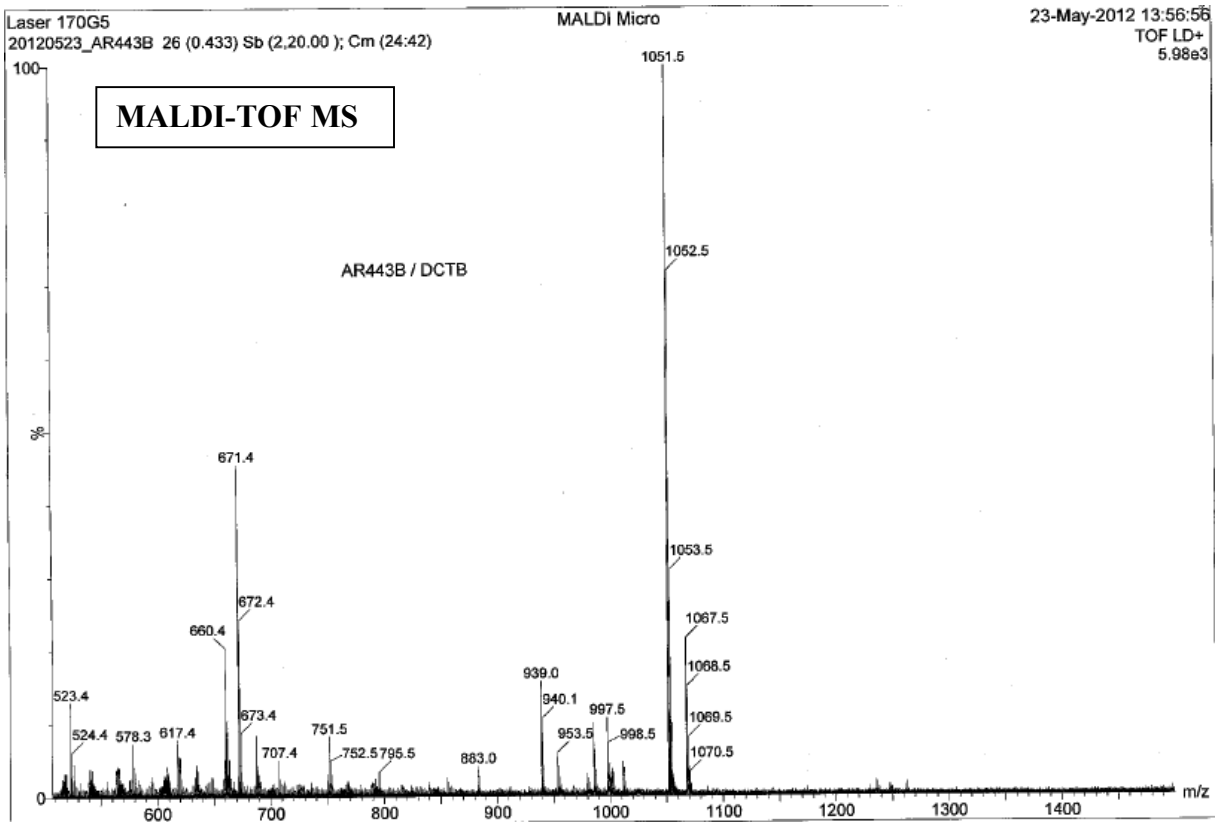
^{13}C{^{1}H} NMR

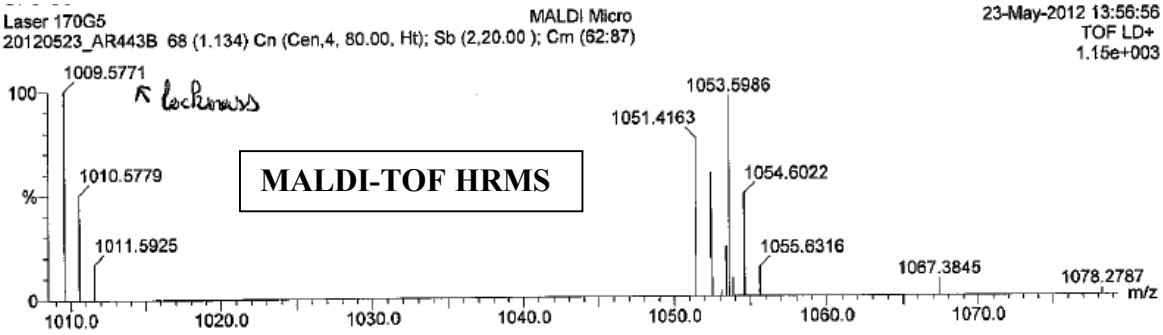


^{19}F NMR



MALDI-TOF MS

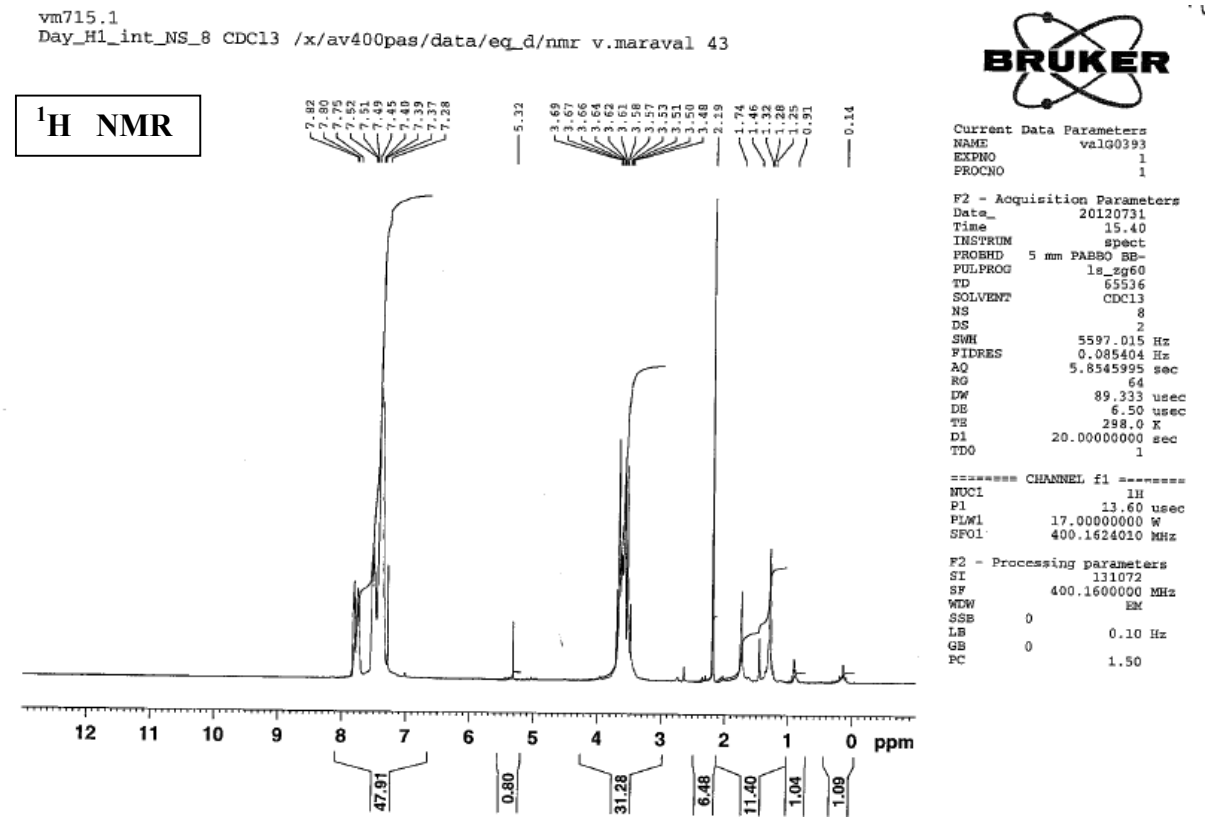




MALDI-TOF HRMS

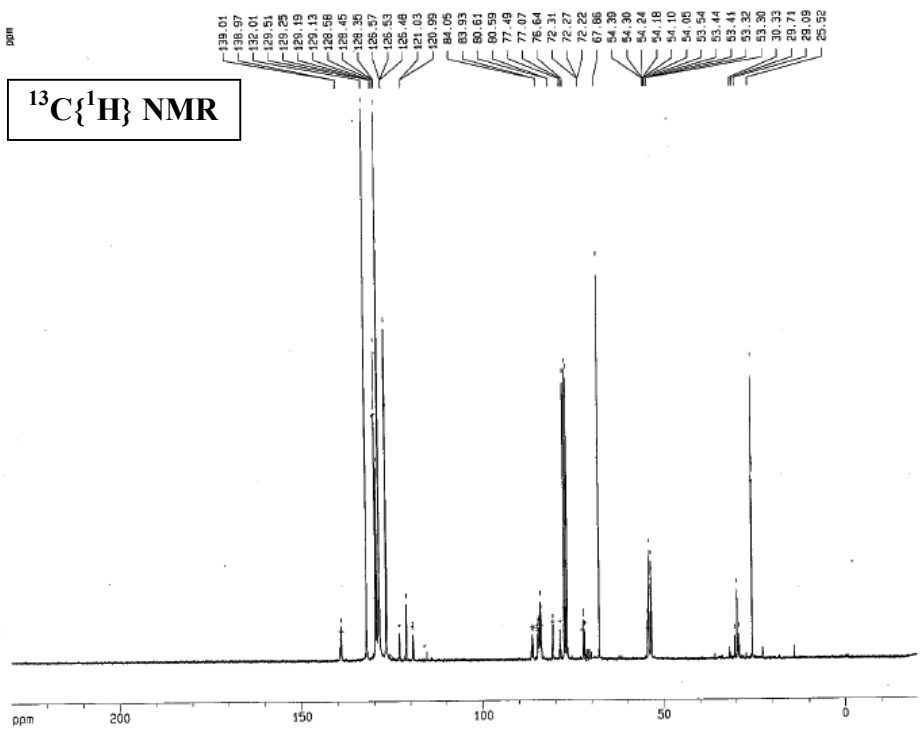
Minimum:				-1.5							
Maximum:		5.0	5.0	70.0							
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula					
1051.4163	1051.4180	-1.7	-1.6	25.5	1.3	C59	H63	O6	F7	23Na	Si
	1051.4211	-4.8	-4.6	20.5	2.9	C55	H67	O7	F7	23Na	Si2
	1051.4200	-3.7	-3.5	24.5	3.7	C58	H66	O6	F6	23Na	Si2 ←
	1051.4184	-2.1	-2.0	24.5	13.2	C58	H67	O3	F7	23Na	Si3
	1051.4208	-4.5	-4.3	27.5	16.5	C60	H66	O3	F7	Si3	
	1051.4208	-4.5	-4.3	27.5	17.7	C60	H68	O5	F4	23Na	Si3

7h. 4,7,13,16-tetramethoxy-4,7-diphenyl-1,10-bis(2-phenylethynyl)-13,16-bis (trifluoromethyl)cyclooctadeca-2,5,8,11,14,17-hexayne-1,10-diol



^{1}H NMR

$^{13}C\{^{1}H\}$ NMR

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**********
* L.C.C. du C.N.R.S *
* Service de R.M.N. *
*      DPX 300      *
**********

Current Data Parameters
NAME            val80391
EXPNO                  2
PROCNO                 1
USER                 ed_d

F2 - Acquisition Parameters
Date_           20120802
Time                7.53
INSTRUM           spx300
PROBHD   5 mm BBF ATM
PULPROG       ls_zg60dc
TD                 32768
SOLVENT            CDCl3
NS                 26500
DS                     4
SWH           10832.393 Hz
FIDRES         0.574719 Hz
AQ             0.6700404 sec
RG                 16384
DW                26.550 usec
DE                10.00 usec
TE                  298 K
D1            1.00000000 sec
d11           0.03000000 sec

========== CHANNEL f1 ==========
NUC1                13C
P1                11.80 usec
PL1                0.00 dB
SFO1         75.4756726 MHz

========== CHANNEL f2 ==========
CPDPRG2          waltz16
NUC2                1H
PCPD2             90.00 usec
PL2                1.00 dB
PL12              17.00 dB
SFO2         300.1312005 MHz

F2 - Processing parameters
SI                 32768
SF          75.4677490 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 22.00 cm
F1P              229.765 ppm
F1              17339.64 Hz
F2P              -19.777 ppm
F2              -1492.55 Hz
PPMCM           11.34283 ppm/cm
HZCM            856.01762 Hz/cm
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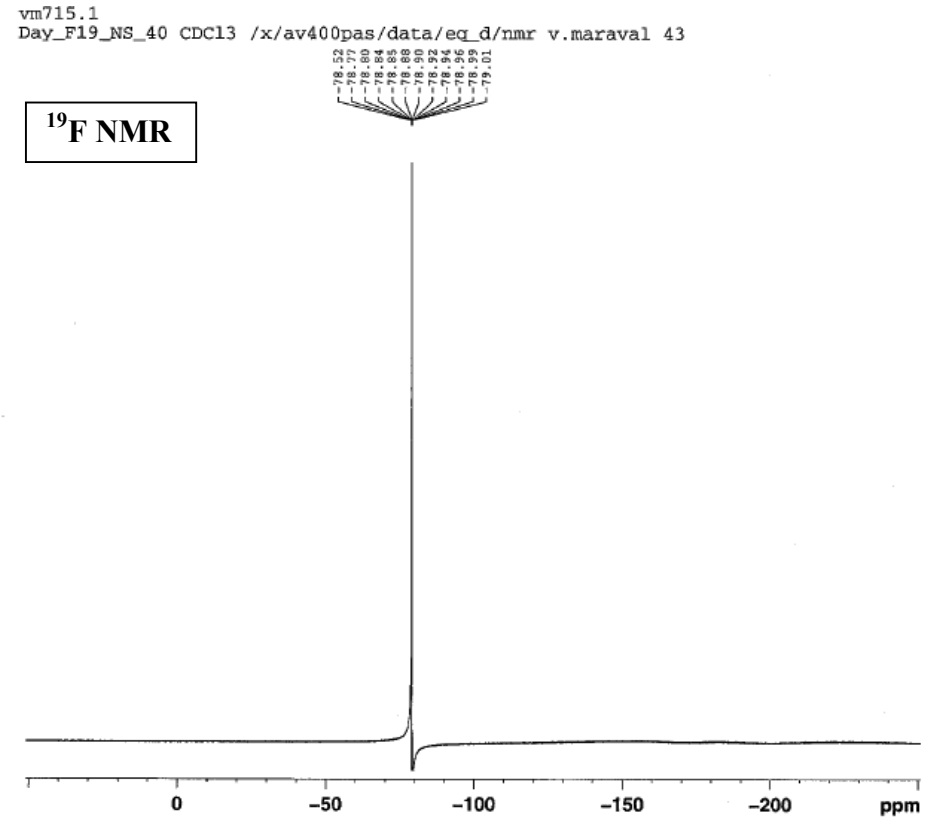
^{19}F NMR

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Current Data Parameters
NAME                 valG0393
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_               20120731
Time                   15.42
INSTRUM                spect
PROBHD   5 mm PABBO BB-
PULPROG             ls_zg60
TD                    131072
SOLVENT                CDCl3
NS                        40
DS                         4
SWH               113636.367 Hz
FIDRES              0.866977 Hz
AQ                  0.5767668 sec
RG                        724
DW                      4.400 usec
DE                      6.50 usec
TE                    298.0 K
D1                1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                      19F
P1                     11.65 usec
PLW1             25.00000000 W
SFO1            376.4889413 MHz

F2 - Processing parameters
SI                        65536
SF               376.5265940 MHz
WDW                          EM
SSB                 0
LB                        0.30 Hz
GB                  0
PC                        1.00
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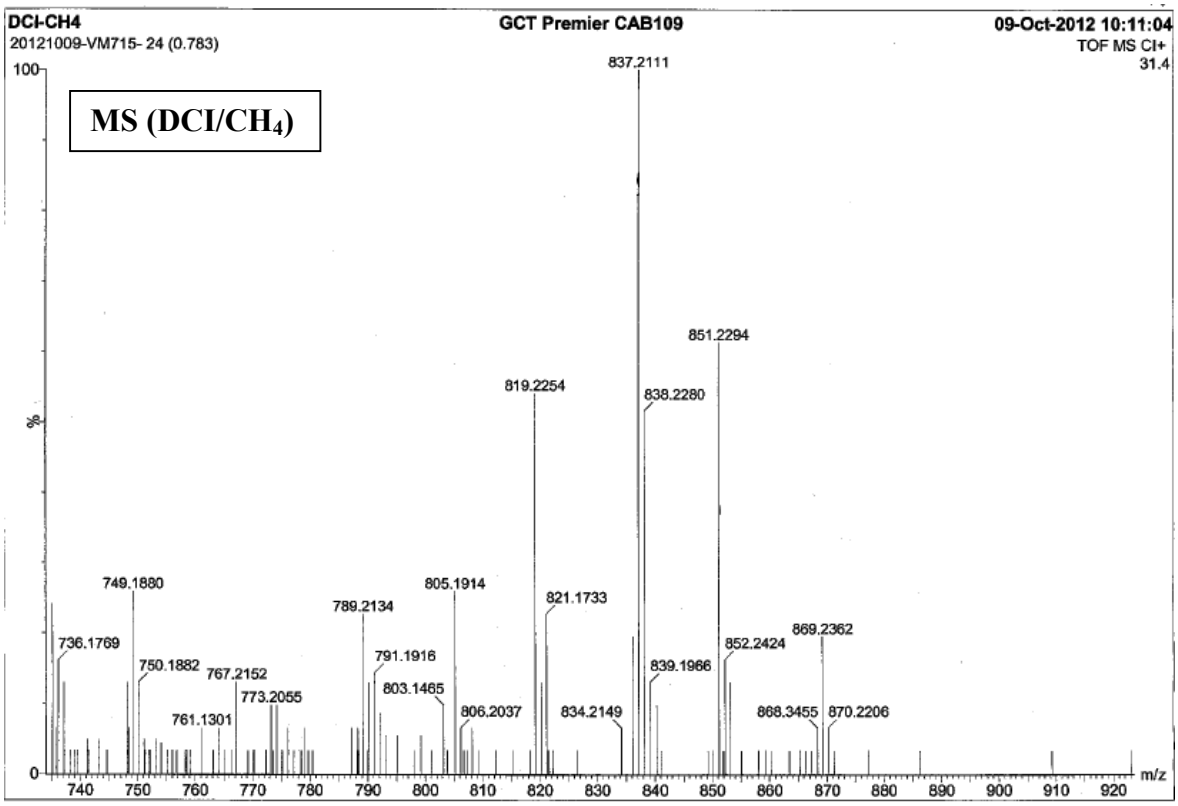
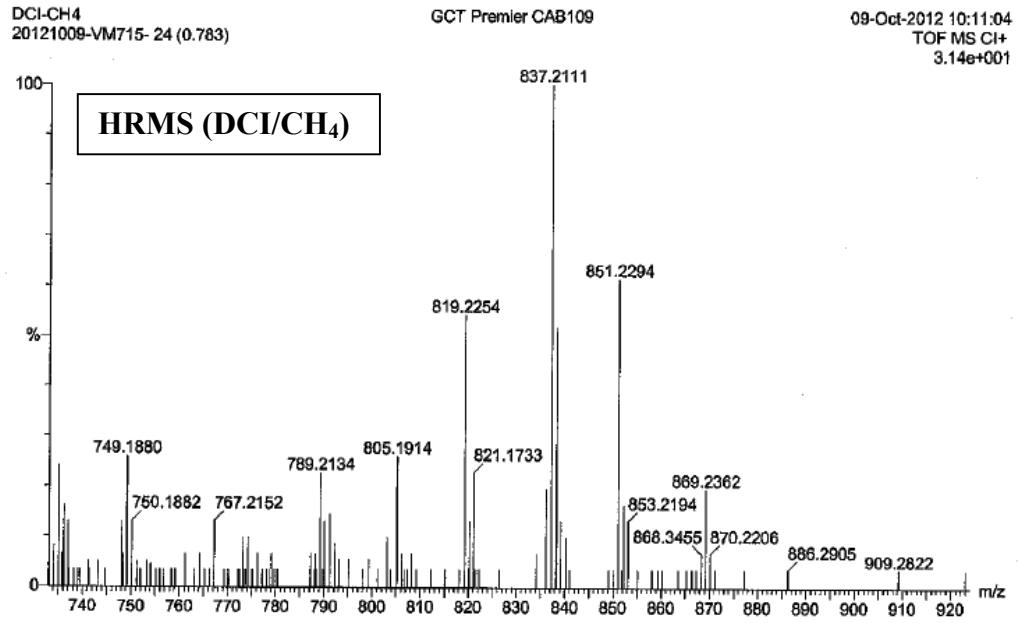
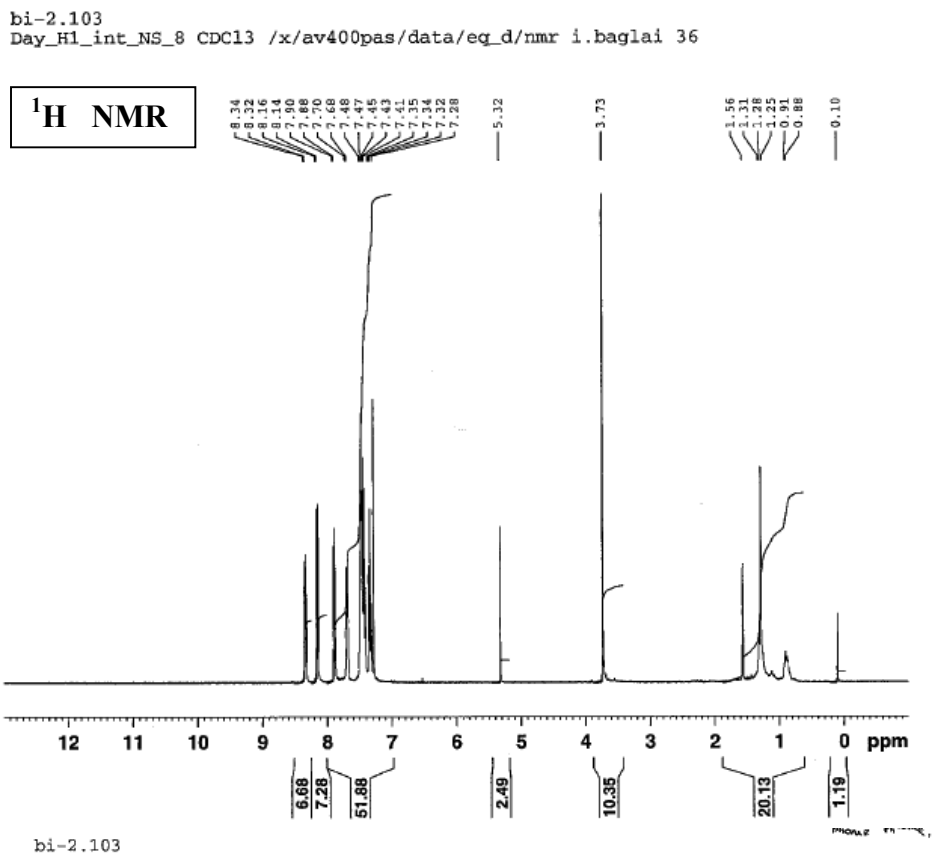




Minimum:				-1.5		
Maximum:		1.3	6.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
869.2362	869.2385	-2.3	-2.6	27.5	0.2	C48 H38 O10 F5
	869.2374	-1.2	-1.4	31.5	0.3	C51 H37 O9 F4
	869.2338	2.4	2.8	32.5	0.3	C52 H35 O6 F6 → MH+
	869.2362	0.0	0.0	35.5	0.3	C54 H36 O8 F3
	869.2326	3.6	4.1	36.5	0.4	C55 H34 O5 F5
	869.2387	-2.5	-2.9	38.5	0.4	C56 H37 O10
	869.2351	1.1	1.3	39.5	0.4	C57 H35 O7 F2
	869.2315	4.7	5.4	40.5	0.4	C58 H33 O4 F4
	869.2339	2.3	2.6	43.5	0.5	C60 H34 O6 F
	869.2328	3.4	3.9	47.5	0.5	C63 H33 O5

10e. **1,10-bis[4-(9*H*-carbazol-9-yl)phenyl]-4,7-dimethoxy-4,7-diphenyldeca-2,5,8-triyne-1,10-dione**



BRUKER

```
Current Data Parameters
NAME                 ibaG0434
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_               20120705
Time                   14.57
INSTRUM                spect
PROBHD     5 mm PABBO BB-
PULPROG             ls_zg60
TD                    65536
SOLVENT               CDCl3
NS                        8
DS                        2
SWH                5597.015 Hz
FIDRES             0.085404 Hz
AQ                 5.8545995 sec
RG                      114
DW                   89.333 usec
DE                     6.50 usec
TE                   298.0 K
D1                20.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                      13.60 usec
PLW1               17.00000000 W
SFO1              400.1624010 MHz

F2 - Processing parameters
SI                     131072
SF                400.1600000 MHz
WDW                        EM
SSB                 0
LB                      0.10 Hz
GB                  0
PC                      1.50
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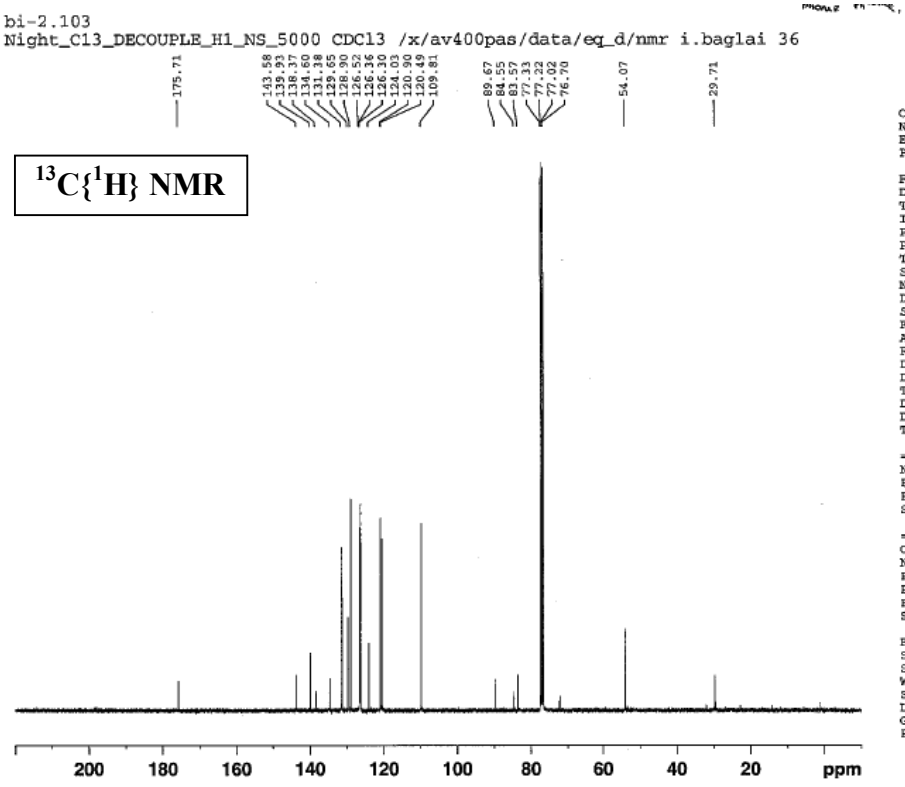

BRUKER

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Current Data Parameters
NAME                 ibaG0434
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_               20120707
Time                   19.40
INSTRUM                spect
PROBHD     5 mm PABBO BB-
PULPROG           ls_zgdc60
TD                    65536
SOLVENT               CDCl3
NS                     5000
DS                        4
SWH               23148.148 Hz
FIDRES             0.353213 Hz
AQ                 1.4156276 sec
RG                     2050
DW                   21.600 usec
DE                     6.50 usec
TE                   298.0 K
D1                 1.00000000 sec
D11                0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                      13C
P1                       9.00 usec
PLW1               70.00000000 W
SFO1              100.6308781 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                   90.00 usec
PLW2               17.00000000 W
PLW12               0.38819000 W
SFO2              400.1616006 MHz

F2 - Processing parameters
SI                     131072
SF                100.6203130 MHz
WDW                        EM
SSB                 0
LB                      1.00 Hz
GB                  0
PC                      1.50
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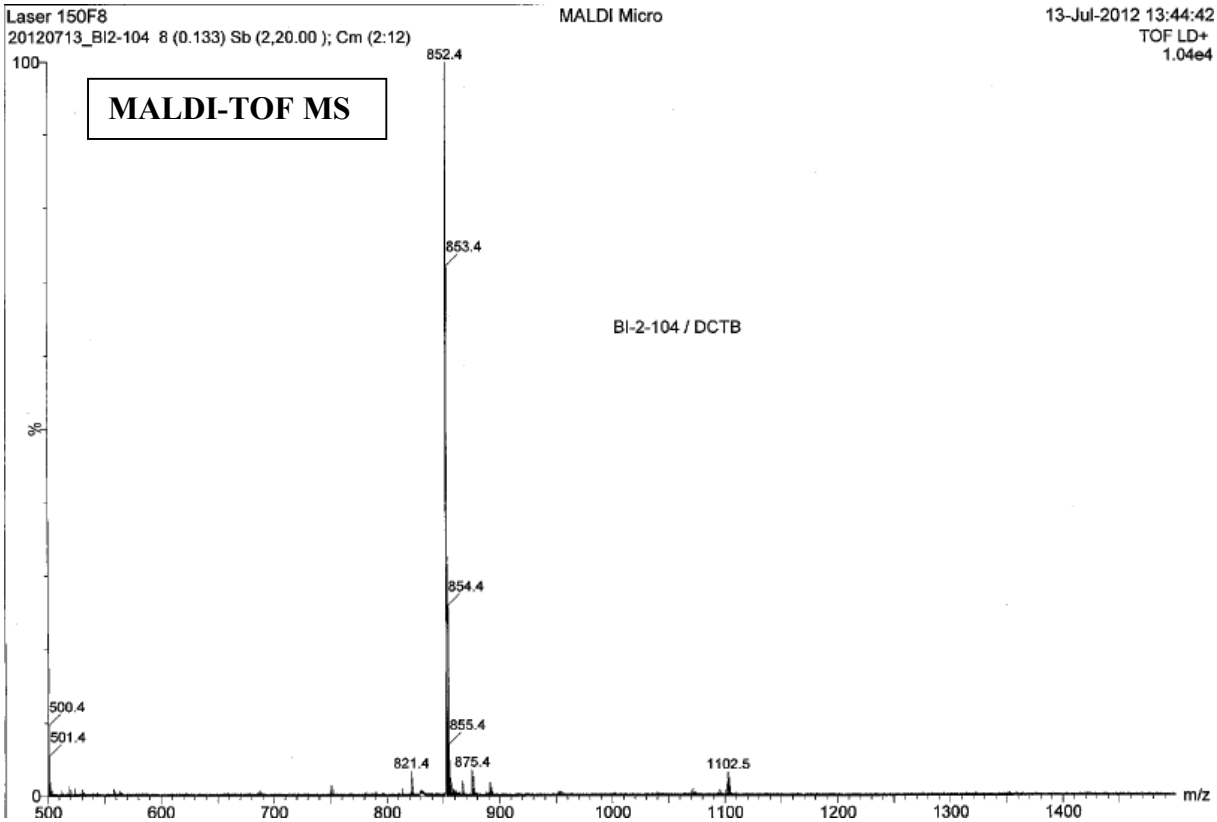
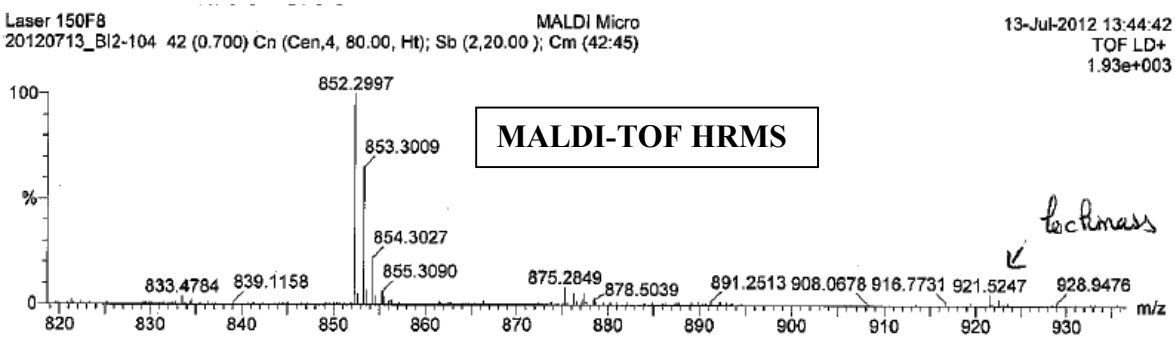
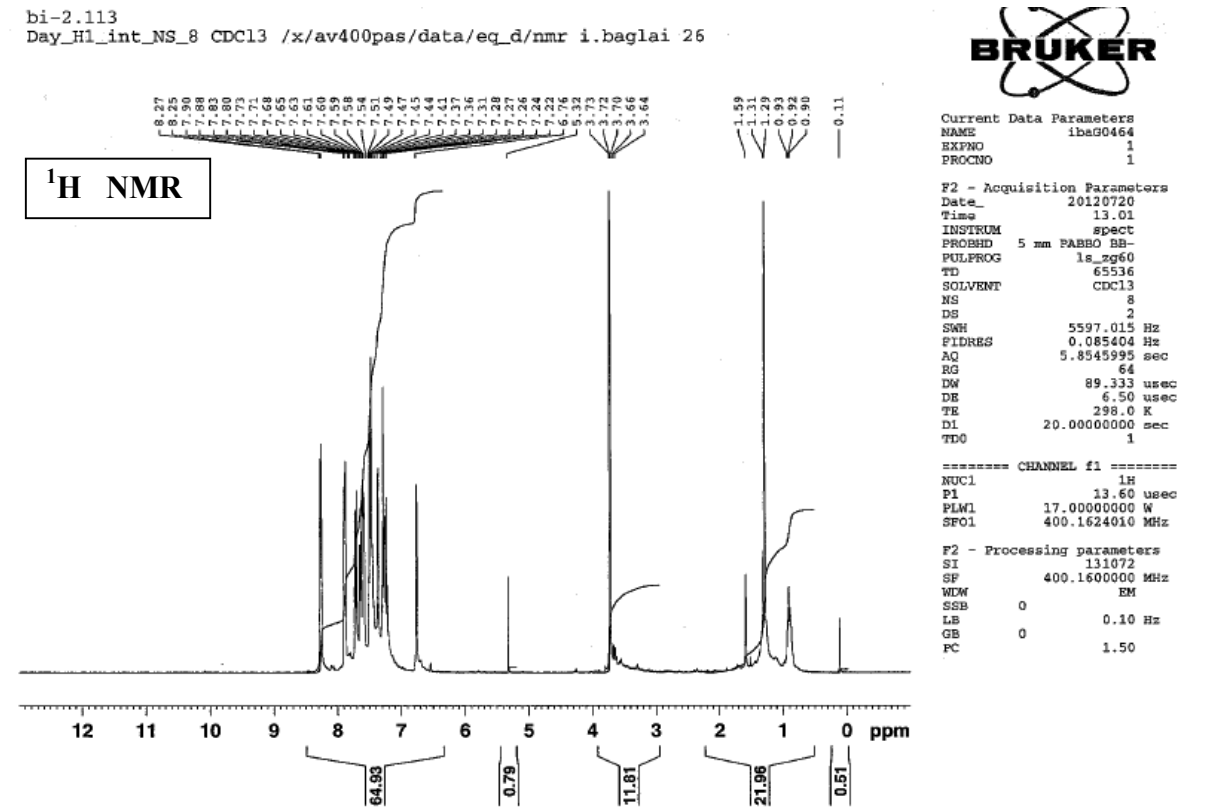
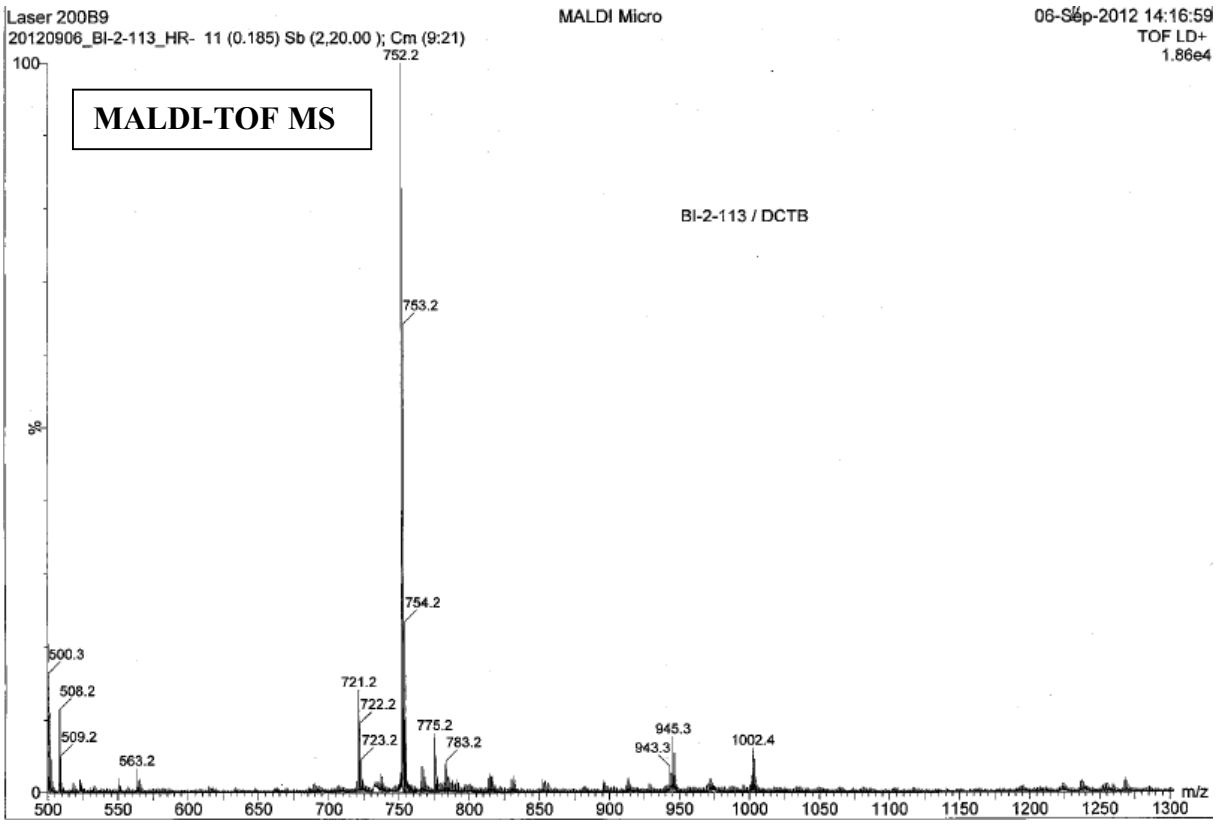




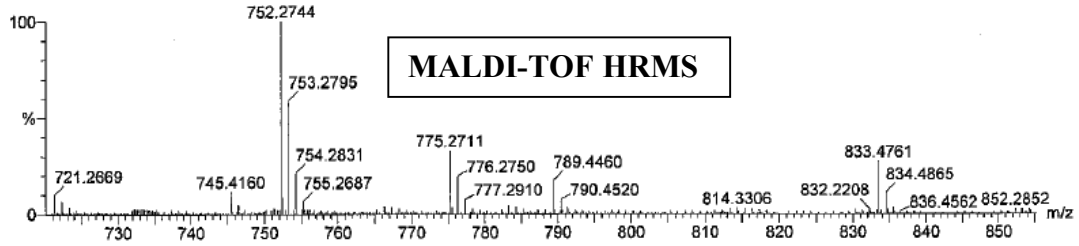
Minimum:				-1.5		
Maximum:		5.0	10.0	55.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
852.2997	852.3074	-7.7	-9.0	37.5	1.2	C56 H42 N3 O6
	852.2988	0.9	1.1	42.0	1.4	C60 H40 N2 O4 ←
	852.3015	-1.8	-2.1	46.5	6.2	C63 H38 N3 O
	852.3028	-3.1	-3.6	46.0	9.4	C65 H40 O2

10f. **1,10-bis[4-(1*H*-indol-1-yl)phenyl]-4,7-dimethoxy-4,7-diphenyldeca-2,5,8-triyne-1,10-dione**





Laser 20089 MALDI Micro 06-Sep-2012 14:16:59
20120906_BI-2-113_HR- 40 (0.668) Cn (Cen,4, 80.00, Ht); Sb (2,20.00); Cm (32:44) TOF LD+
8.98e+003



Minimum:				-1.5					
Maximum:		5.0	10.0	55.0					
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula			
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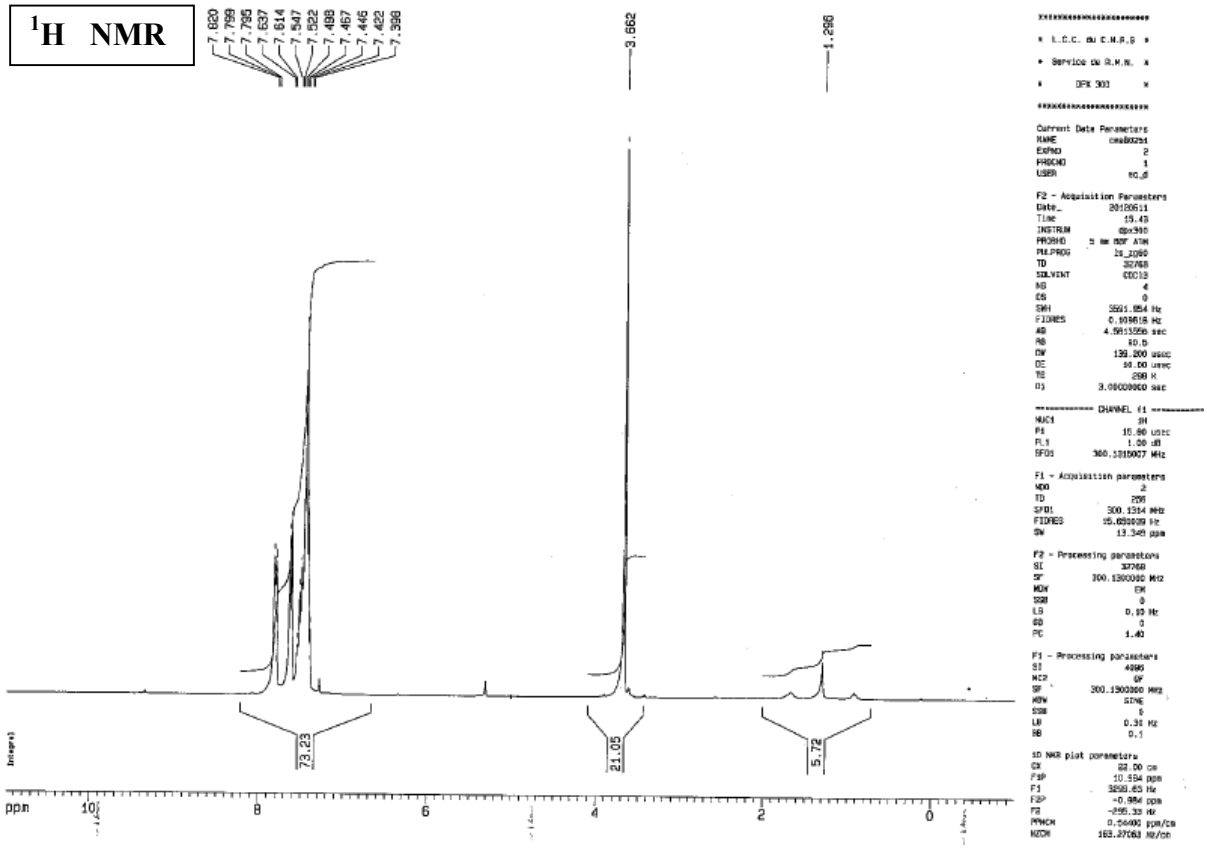
	752.2715	2.9	3.9	40.0	16.6	C57	H36	O2	
	752.2702	4.2	5.6	40.5	17.1	C55	H34	N3	O
	752.2814	-7.0	-9.3	40.5	19.6	C54	H34	N5	
	752.2688	5.6	7.4	41.0	21.2	C53	H32	N6	
	752.2675	6.9	9.2	36.0	22.4	C52	H36	N2	O4
	752.2787	-4.3	-5.7	36.0	23.5	C51	H36	N4	O3
	752.2774	-3.0	-4.0	31.0	29.4	C50	H40	O7	
	752.2774	-3.0	-4.0	36.5	34.7	C49	H34	N7	O2
	752.2761	-1.7	-2.3	31.5	44.6	C48	H38	N3	O6
	752.2747	-0.3	-0.4	32.0	64.0	C46	H36	N6	O5
	752.2720	2.4	3.2	27.5	112.3	C43	H38	N5	O8

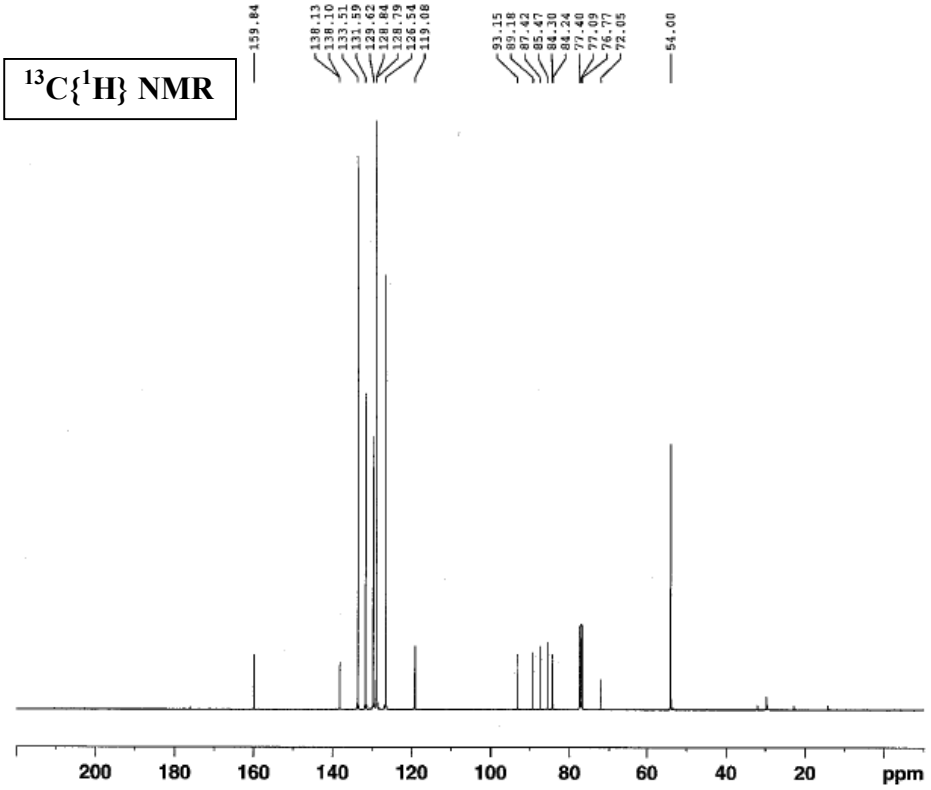
10h. 6,9-dimethoxy-1,6,9,14-tetraphenyltetradeca-1,4,7,10,13-pentayne-3,12-dione

^{1}H NMR



vm694
Night_C13_DECOUPLE_H1_NS_5000 CDCl3 /x/av400pas/data/eq_d/nmr v.maraval 23

$^{13}C\{^{1}H\}$ NMR

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Current Data Parameters
NAME                  valG0340
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20120611
Time                     22.31
INSTRUM                  spect
PROBHD    5 mm PABBO BB-
PULPROG              ls_zgdc60
TD                       65536
SOLVENT                  CDCl3
NS                        5000
DS                           4
SWH                  23148.148 Hz
FIDRES                0.353213 Hz
AQ                    1.4156276 sec
RG                         2050
DW                       21.600 use
DE                        6.50 use
TE                      298.0 K
D1                 1.00000000 sec
D11                0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        13C
P1                         9.00 use
PLW1               70.00000000 W
SFO1              100.6308781 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                     90.00 use
PLW2               17.00000000 W
PLW12               0.38819000 W
SFO2              400.1616006 MHz

F2 - Processing parameters
SI                       131072
SF                100.6203130 MHz
WDW                          EM
SSB                 0
LB                        1.00 Hz
GB                  0
PC                        1.50
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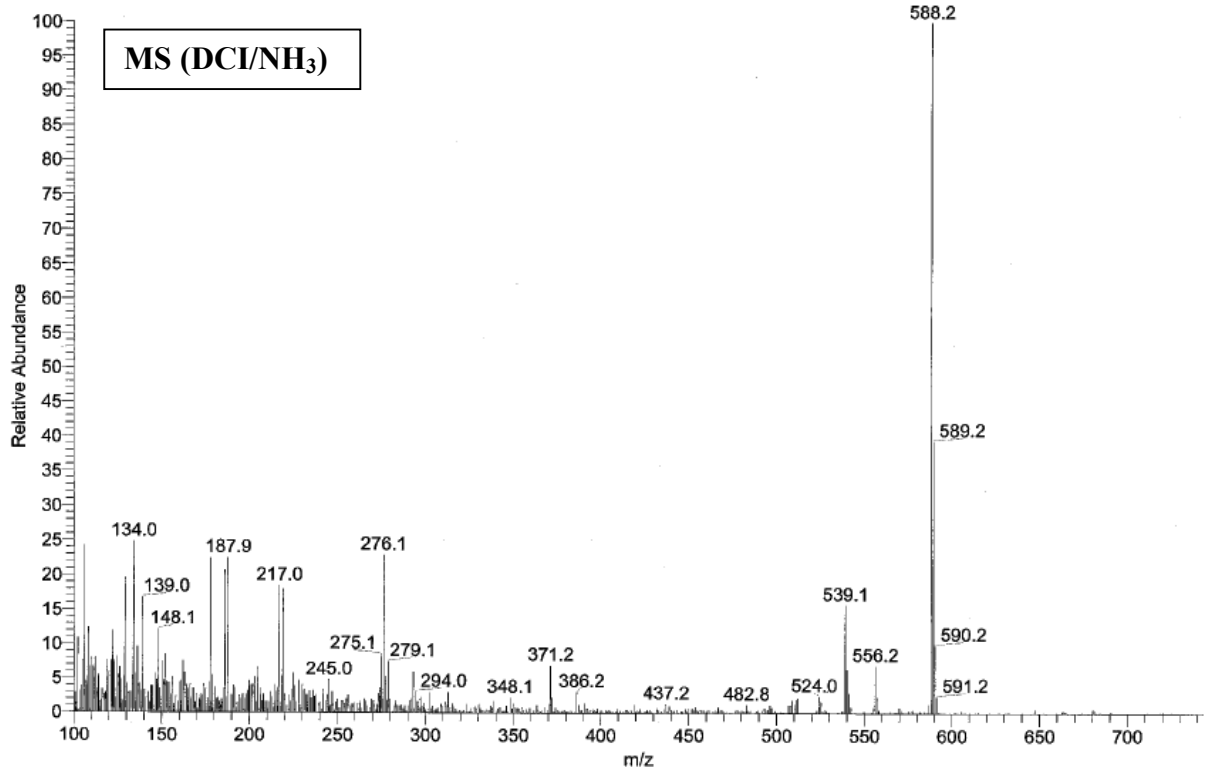
VM694 #13-14 RT: 0.30-0.32 AV: 2 NL: 1.16E5
T: + c Full ms [100.00-800.00]

MS (DCI/NH_3)

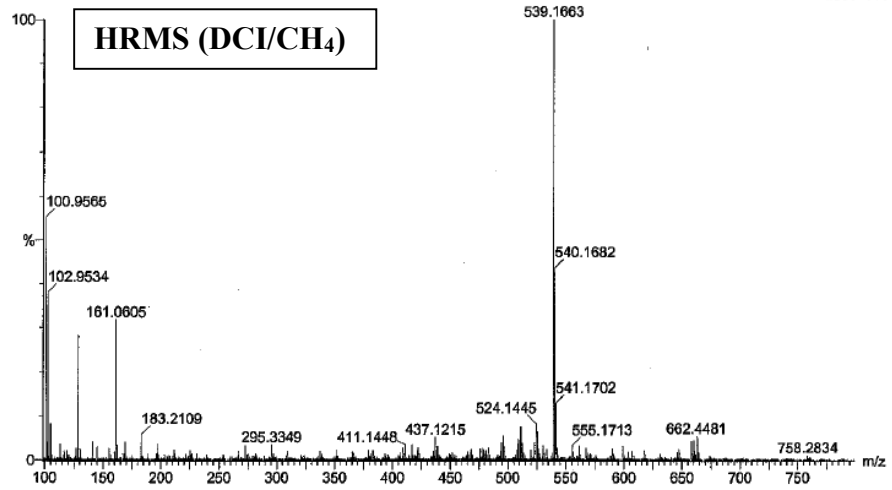


DCI-CH4
20120614-VM694 14 (0.450) Cm (14:15-36x5.000)

GCT Premier CAB109

14-Jun-2012 14:42:02
TOF MS CI+
2.58e+003

HRMS (DCI/CH_4)

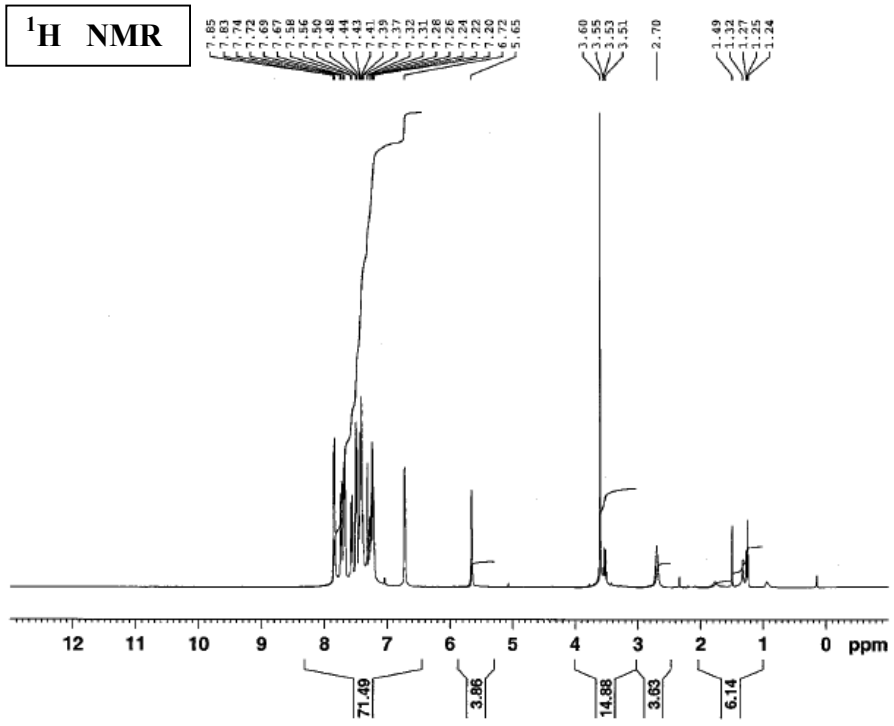


Minimum:				-1.5					
Maximum:		1.3	5.0	50.0					
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula			
539.1663	539.1674	-1.1	-2.0	33.0	7.0	C42	H21	N	
	539.1647	1.6	3.0	28.5	7.3	C39	H23	O3	— $MH^+ - CH_3OH$
	539.1679	-1.6	-3.0	20.5	51.9	C28	H23	N6	O6
	539.1666	-0.3	-0.6	15.5	63.5	C27	H27	N2	O10
	539.1666	-0.3	-0.6	21.0	64.7	C26	H21	N9	O5
	539.1652	1.1	2.0	16.0	77.7	C25	H25	N5	O9
	539.1639	2.4	4.5	16.5	93.7	C23	H23	N8	O8

11f. 1,10-bis[4-(1*H*-indol-1-yl)phenyl]-4,7-dimethoxy-4,7-diphenyldeca-2,5,8-triyne-1,10-diol

bi-2.110.
Night_H1_int_NS_8 CDCl3 /x/av400pas/data/eq_d/nmr i.baglai 22

^{1}H NMR



BRUKER

Current Data Parameters
NAME ibaG0458
EXPNO 2
PROCNO 1

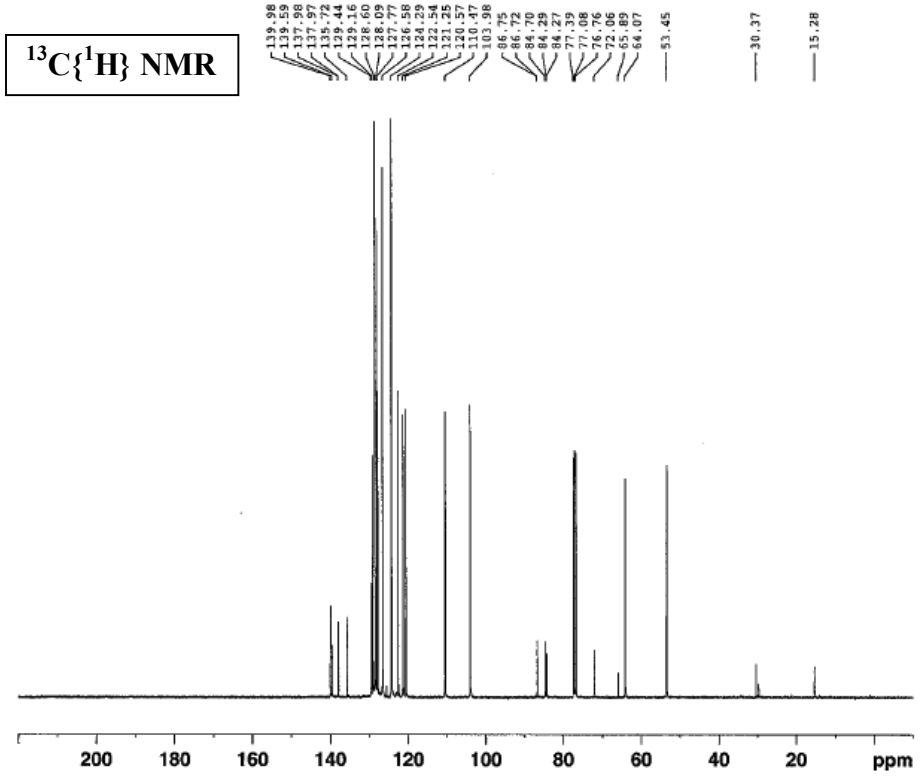
F2 - Acquisition Parameters
Date_ 20120719
Time 4.10
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG 1s_zg60
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 5597.015 Hz
FIDRES 0.085404 Hz
AQ 5.8545995 sec
RG 40.3
DW 89.333 usec
DE 6.50 usec
TE 298.0 K
D1 20.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.60 usec
PLW1 17.00000000 W
SFO1 400.1624010 MHz

F2 - Processing parameters
SI 131072
SF 400.1600000 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.50

bi-2.110.
Night_C13_DECOUPLE_H1_NS_5000 CDCl3 /x/av400pas/data/eq_d/nmr i.baglai 22

$^{13}C\{^{1}H\}$ NMR



Current Data Parameters
NAME ibaG0458
EXPNO 1
PROCNO 1

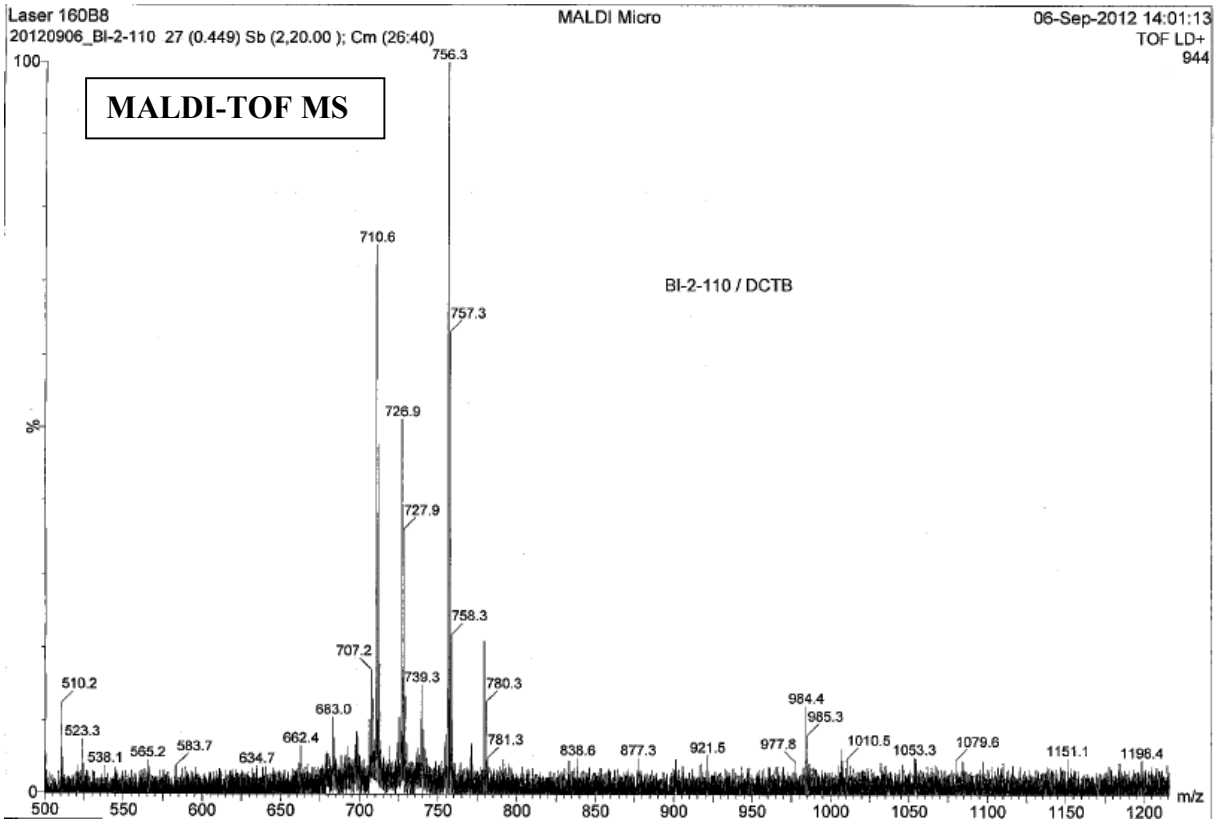
F2 - Acquisition Parameters
Date_ 20120719
Time 4.04
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG 1s_zgdc60
TD 65536
SOLVENT CDCl3
NS 5000
DS 4
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 2050
DW 21.600 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PLW1 70.00000000 W
SFO1 100.6308781 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 17.00000000 W
PLW12 0.38819000 W
SFO2 400.1616006 MHz

F2 - Processing parameters
SI 131072
SF 100.6203130 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.50

MALDI-TOF MS

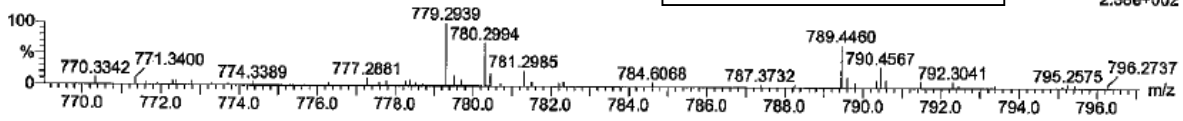


0 V
20120906_BI-2-110_HR- 22 (0.368) Cn (Cen,4, 80.00, Ht); Sb (2,20.00); Cm (22:24)

MALDI Micro

MALDI-TOF HRMS

06-Sep-2012 14:23:17
TOF LD+
2.38e+002

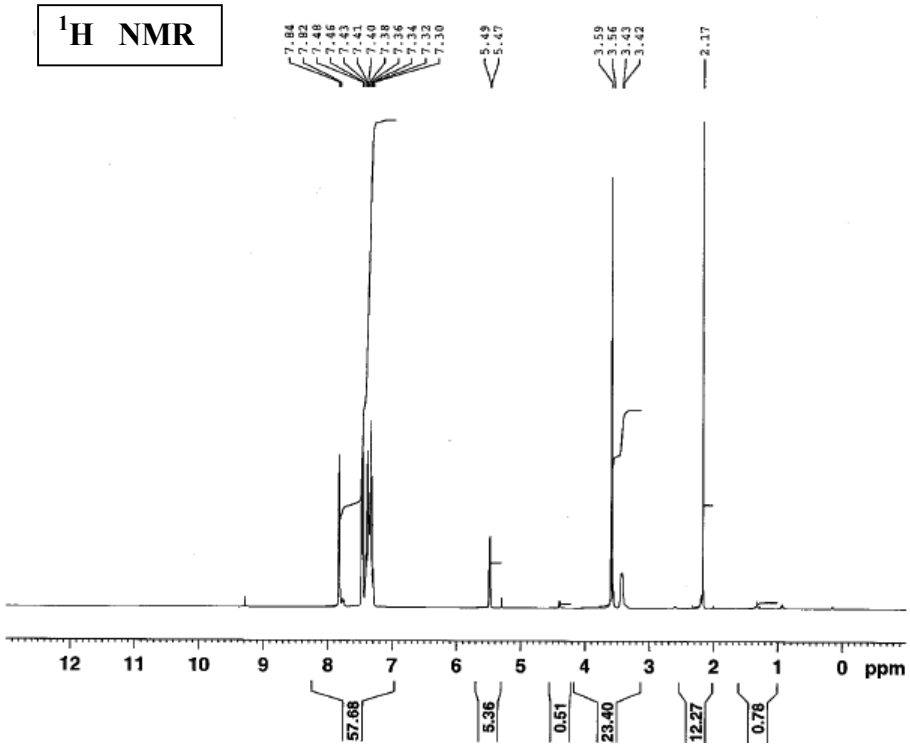


Minimum:				-1.5							
Maximum:		3.0	10.0	50.0							
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula				
779.2939	779.2950	-1.1	-1.4	40.5	37.7	1.3	C59	H39	O2		
	779.2937	0.2	0.3	41.0	37.8	1.5	C57	H37	N3	O	
	779.2926	1.3	1.7	37.5	38.1	1.7	C57	H40	O2	23Na	
	779.2913	2.6	3.3	38.0	38.6	2.3	C55	H38	N3	O	23Na
	779.2910	2.9	3.7	36.5	38.8	2.5	C54	H39	N2	O4	
	779.2896	4.3	5.5	37.0	39.6	3.3	C52	H37	N5	O3	
	779.2971	-3.2	-4.1	29.0	40.0	3.7	C48	H42	N3	O6	23Na
	779.2998	-5.9	-7.6	33.5	40.2	3.9	C51	H40	N4	O3	23Na
	779.2886	5.3	6.8	33.5	40.3	3.9	C52	H40	N2	O4	23Na
	779.2995	-5.6	-7.2	32.0	40.3	3.9	C50	H41	N3	O6	
	779.3012	-7.3	-9.4	33.0	40.5	4.1	C53	H42	N	O4	23Na
	779.2872	6.7	8.6	34.0	41.2	4.9	C50	H38	N5	O3	23Na
	779.2870	6.9	8.9	32.5	41.5	5.2	C49	H39	N4	O6	

11h. 6,9-dimethoxy-1,6,9,14-tetraphenyltetradeca-1,4,7,10,13-pentayne-3,12-diol

vm693.3.2
Day_H1_int_NS_8 CDCl3 /x/av400pas/data/eq_d/nmr v.maraval 1

^{1}H NMR



BRUKER

Current Data Parameters
NAME valG0339
EXPNO 1
PROCNO 1

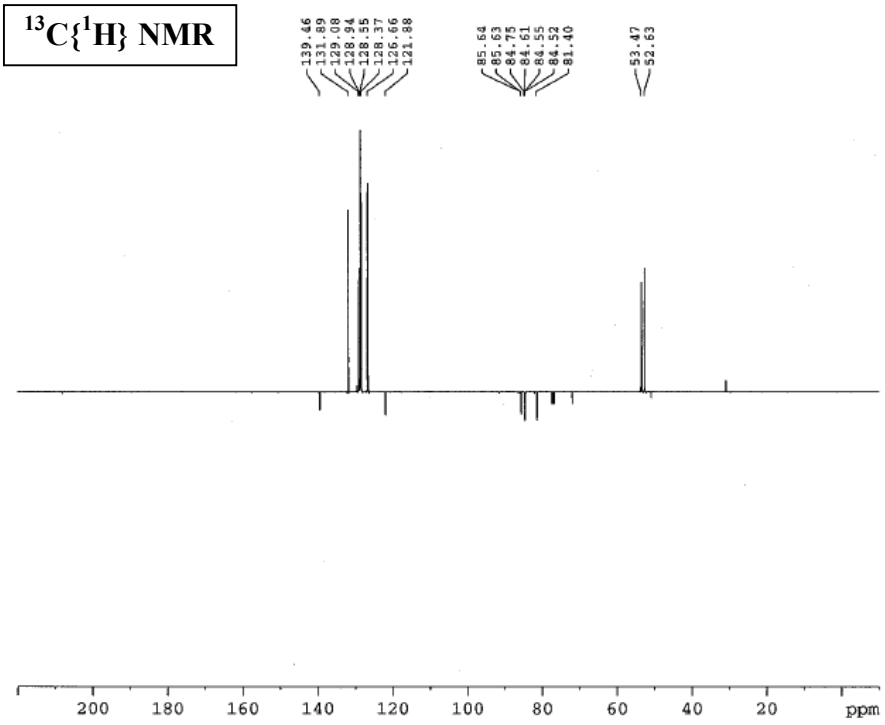
F2 - Acquisition Parameters
Date_ 20120608
Time 17.10
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG 1s_zg60
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 5597.015 Hz
FIDRES 0.085404 Hz
AQ 5.8545995 sec
RG 14.2
DW 89.333 usec
DE 6.50 usec
TE 298.0 K
D1 20.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.60 usec
PLW1 17.00000000 W
SFO1 400.1624010 MHz

F2 - Processing parameters
SI 131072
SF 400.1600000 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.50

vm693.3.2
Night_C13_JMOD_H1_NS_3000 CDCl3 /x/av400pas/data/eq_d/nmr v.maraval 1

$^{13}C\{^{1}H\}$ NMR



Current Data Parameters
NAME valG0339
EXPNO 3
PROCNO 1

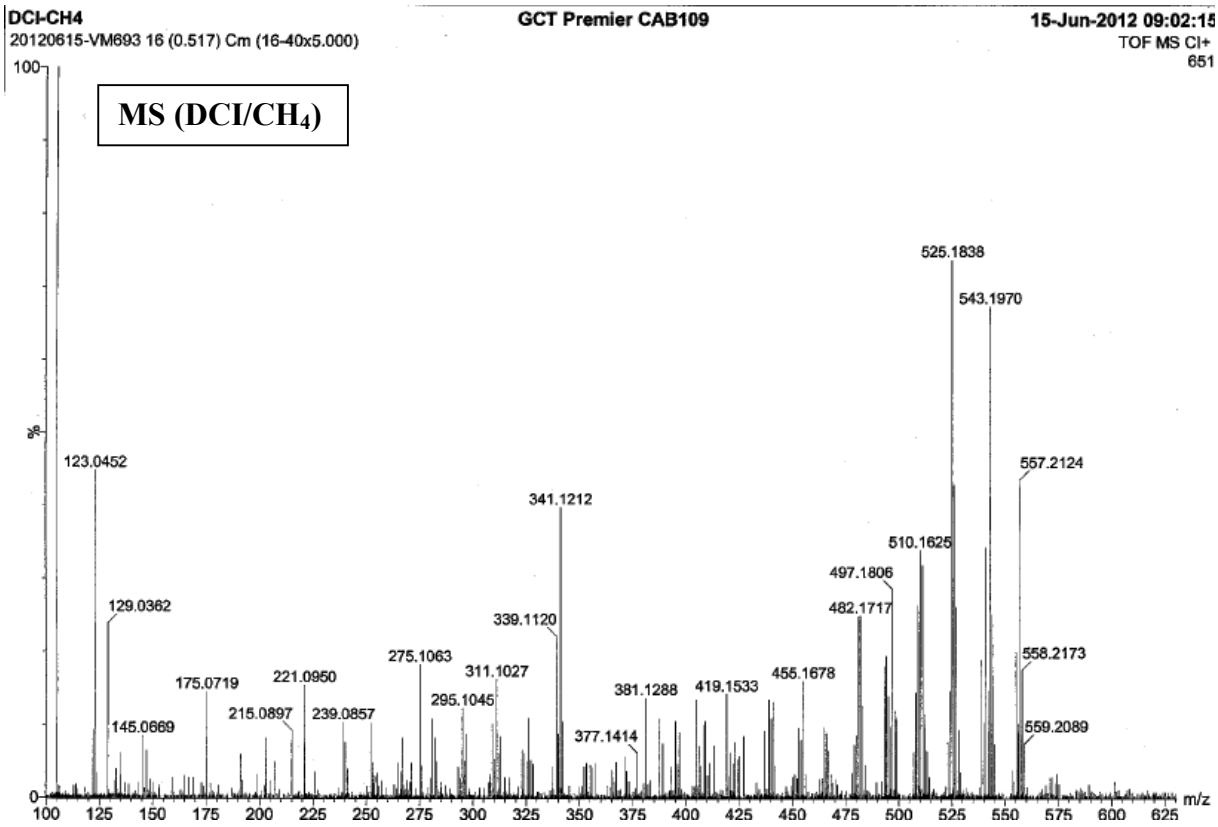
F2 - Acquisition Parameters
Date_ 20120609
Time 15.22
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG ls_jmod
TD 65536
SOLVENT CDCl3
NS 3000
DS 4
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 2050
DW 21.600 usec
DE 6.50 usec
TE 298.0 K
CNST2 145.0000000
CNST11 1.0000000
D1 1.00000000 sec
D20 0.00689655 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
P2 18.00 usec
PLW1 70.00000000 W
SFO1 100.6308781 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 17.00000000 W
PLW12 0.38819000 W
SFO2 400.1616006 MHz

F2 - Processing parameters
SI 131072
SF 100.6203130 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.50

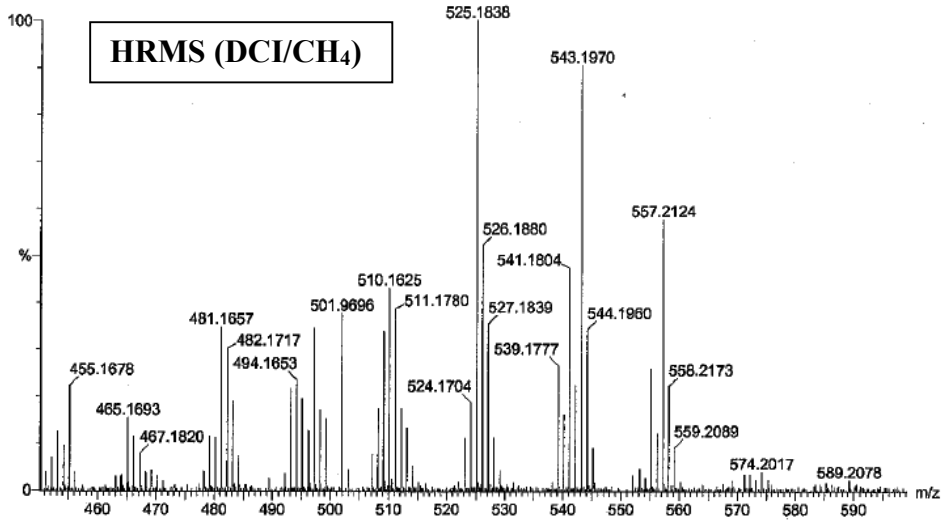
MS (DCI/CH_4)



DCI-CH4
20120615-VM693 16 (0.517)

GCT Premier CAB109

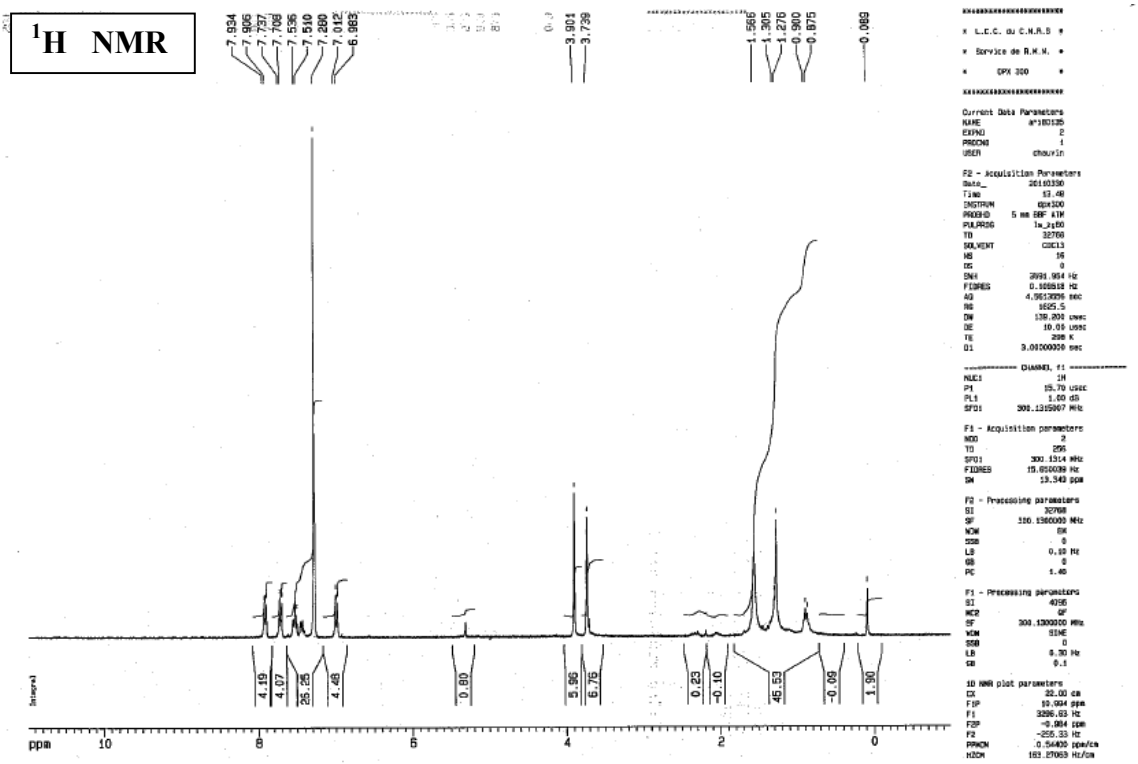
15-Jun-2012 09:02:15
TOF MS CI+
5.36e+002



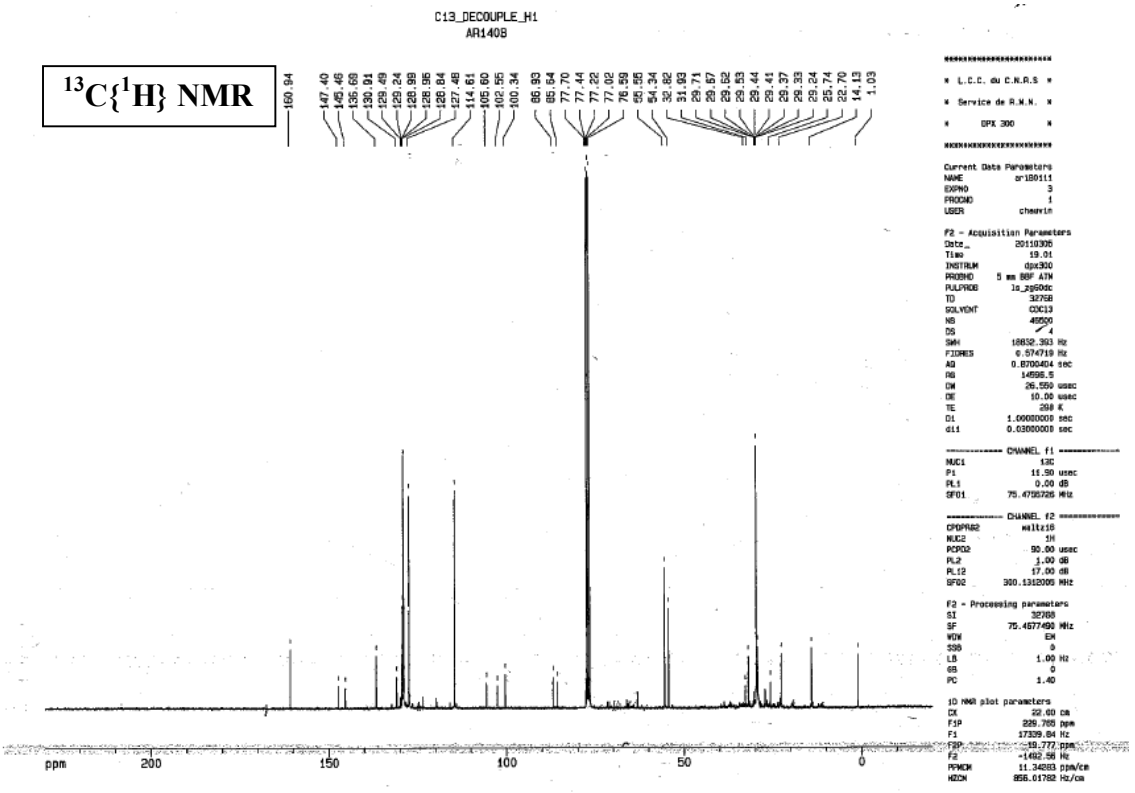
Minimum:				-1.5					
Maximum:		1.3	5.0	50.0					
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula			
557.2124	557.2117	0.7	1.3	26.5	4.2	C40	H29	O3	← M−H2O+H+
	557.2103	2.1	3.8	27.0	4.5	C38	H27	N3	O2
	557.2144	-2.0	-3.6	31.0	5.0	C43	H27	N	
	557.2149	-2.5	-4.5	18.5	6.8	C29	H29	N6	O6
	557.2135	-1.1	-2.0	13.5	7.2	C28	H33	N2	O10
	557.2135	-1.1	-2.0	19.0	8.0	C27	H27	N9	O5
	557.2122	0.2	0.4	14.0	8.6	C26	H31	N5	O9
	557.2108	1.6	2.9	14.5	10.2	C24	H29	N8	O8

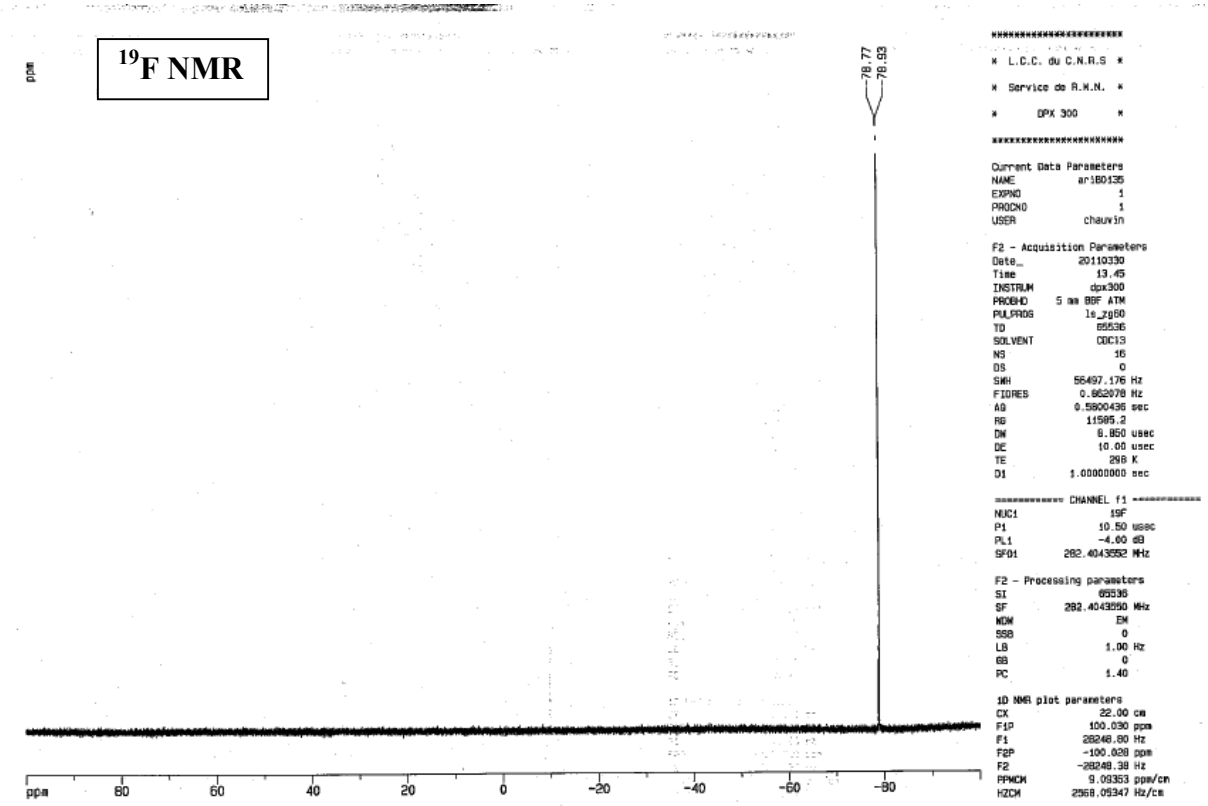
12b. 13,16-dimethoxy-1,10-bis(4-methoxyphenyl)-4,7-diphenyl-13,16-bis(trifluoromethyl) cyclooctadeca-1,2,3,7,8,9-hexaen-5,11,14,17-tetrayne

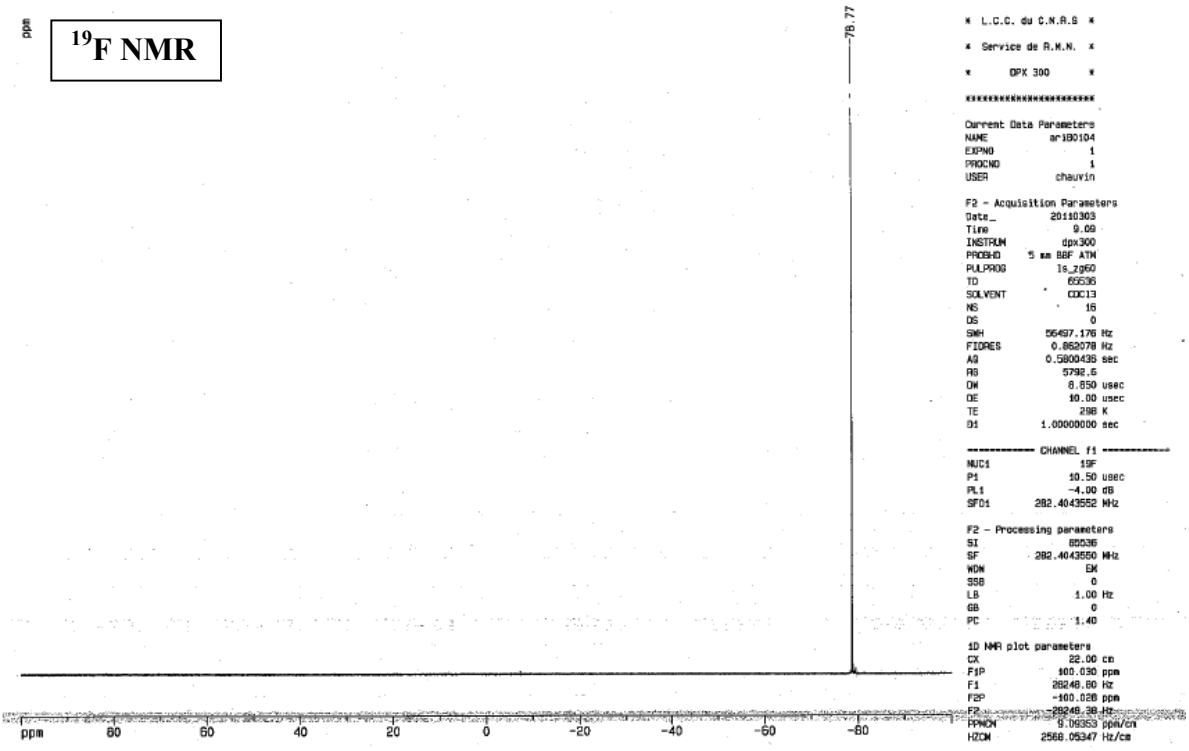
^{1}H NMR

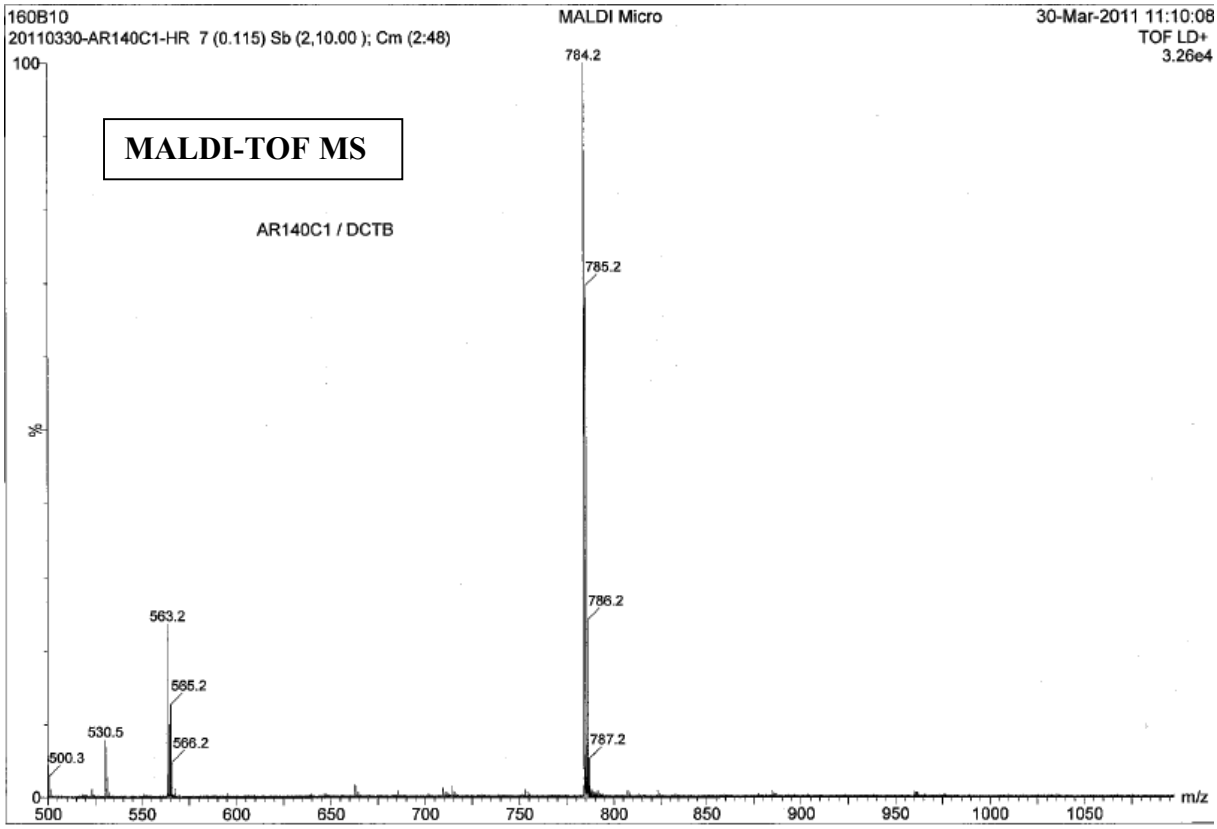


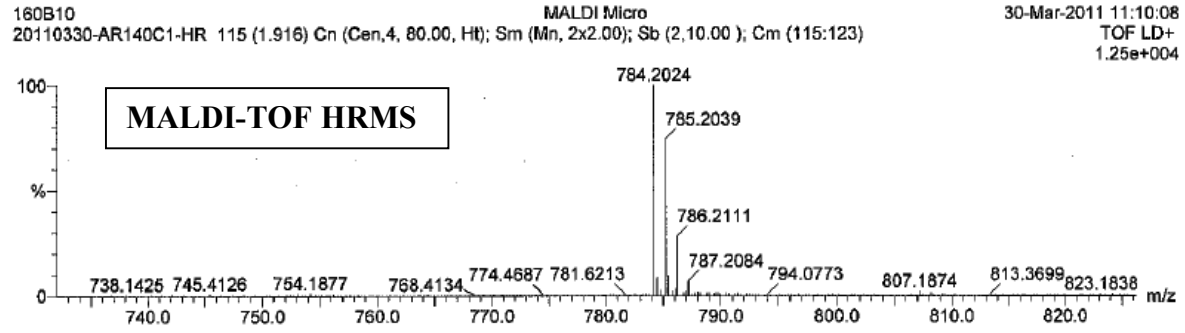
^{13}C{^{1}H} NMR




19F NMR
ppm
78.77
78.93
80
60
40
20
0
-20
-40
-60
-80
* L.C.C. du C.N.R.S *
* Service de R.M.N. *
* DPX 300 *
Current Data Parameters
NAME ar1B0135
EXPNO 1
PROCNO 1
USER chauvin
F2 - Acquisition Parameters
Date_ 20110330
Time 13.45
INSTRUM dpx300
PROBHD 5 mm BBF ATM
PULPROG 1s_zg60
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 56497.176 Hz
FIDRES 0.862078 Hz
AQ 0.5800436 sec
RG 11585.2
DW 8.850 usec
DE 10.00 usec
TE 298 K
D1 1.00000000 sec
CHANNEL f1
NUC1 19F
P1 10.50 usec
PL1 -4.00 dB
SF01 282.4043552 MHz
F2 - Processing parameters
SI 65536
SF 282.4043550 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 22.00 cm
F1P 100.030 ppm
F1 28248.80 Hz
F2P -100.028 ppm
F2 -28248.38 Hz
PPMCM 9.09353 ppm/cm
HZCM 2568.05347 Hz/cm


19F NMR
ppm
78.77
80
60
40
20
0
-20
-40
-60
-80
* L.C.C. du C.N.R.S *
* Service de R.M.N. *
* DPX 300 *
Current Data Parameters
NAME ar1B0104
EXPNO 1
PROCNO 1
USER chauvin
F2 - Acquisition Parameters
Date_ 20110303
Time 9.09
INSTRUM dpx300
PROBHD 5 mm BBF ATM
PULPROG 1s_zg60
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 56497.176 Hz
FIDRES 0.862078 Hz
AQ 0.5800436 sec
RG 5792.6
DW 8.850 usec
DE 10.00 usec
TE 298 K
D1 1.00000000 sec
CHANNEL f1
NUC1 19F
P1 10.50 usec
PL1 -4.00 dB
SF01 282.4043552 MHz
F2 - Processing parameters
SI 65536
SF 282.4043550 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 22.00 cm
F1P 100.030 ppm
F1 28248.80 Hz
F2P -100.028 ppm
F2 -28248.38 Hz
PPMCM 9.09353 ppm/cm
HZCM 2568.05347 Hz/cm

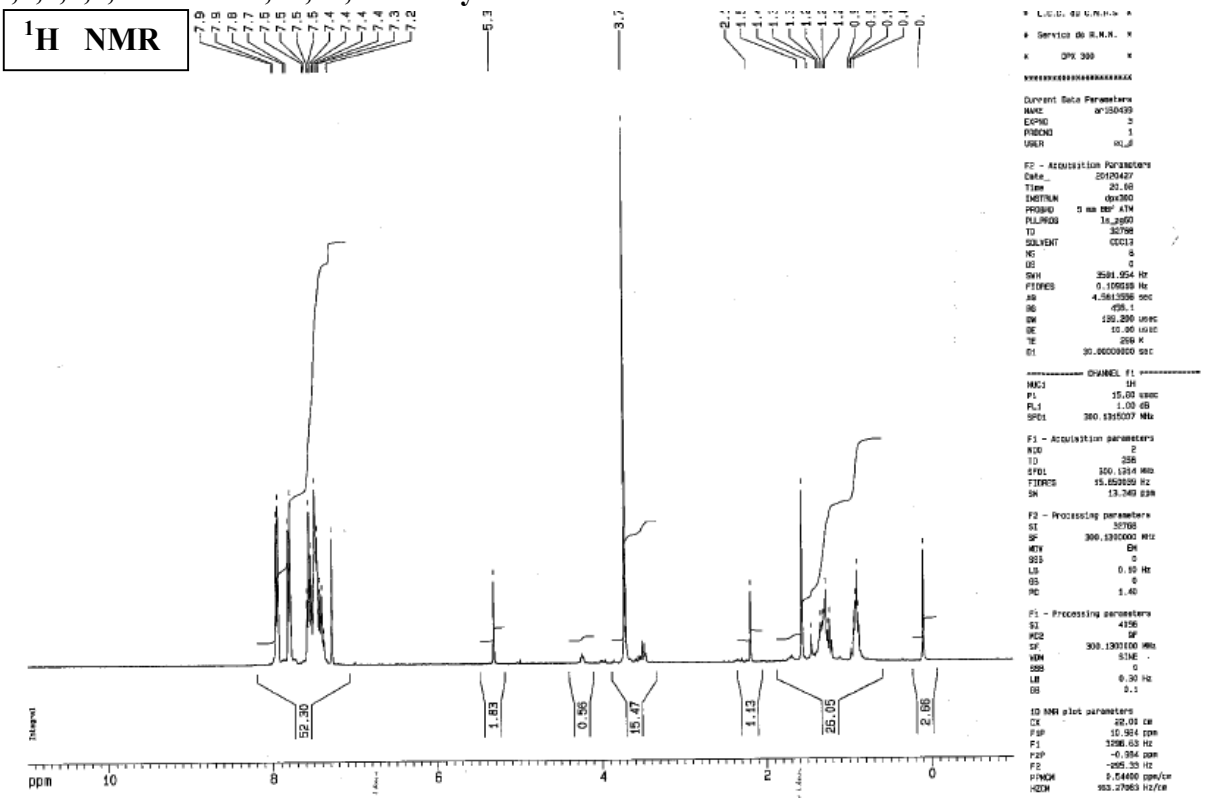




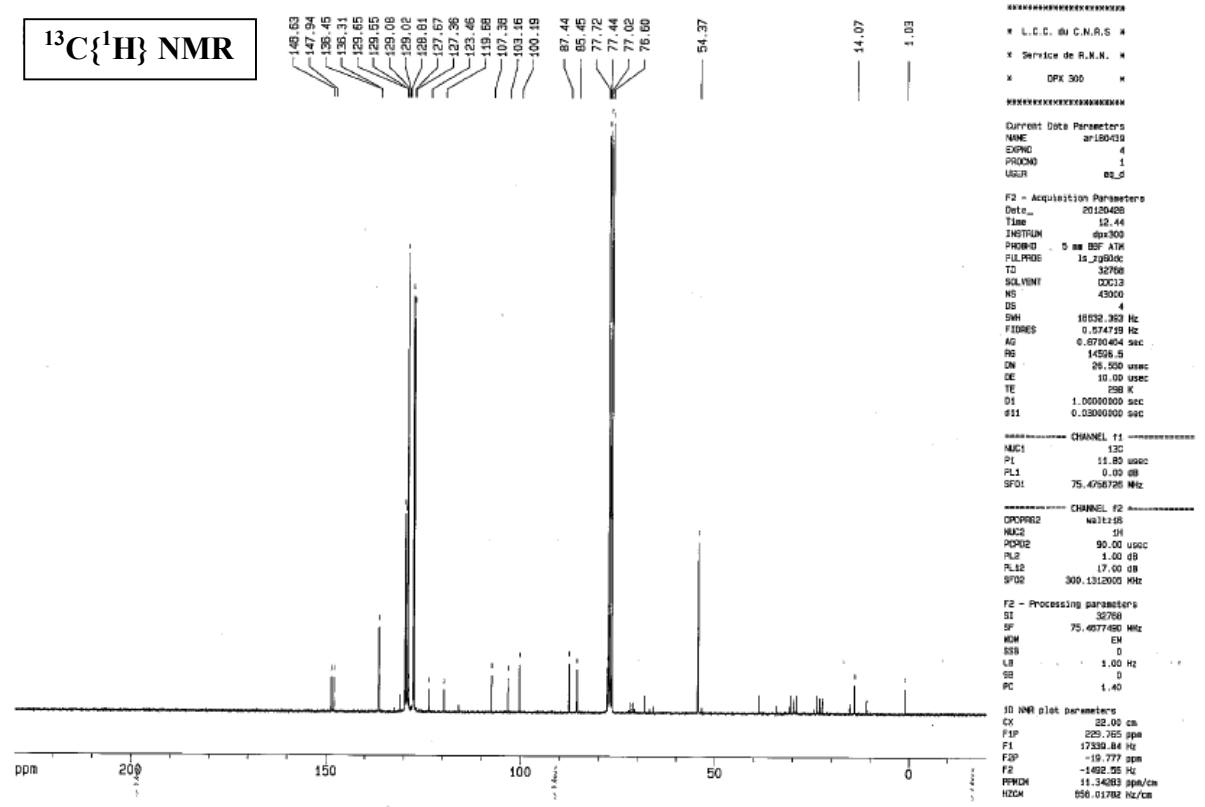
Minimum:				-1.5		
Maximum:		30.0	5.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
784.2024	784.2025	-0.1	-0.1	39.0	250.1	C54 H28 O2 F4
	784.2014	1.0	1.3	43.0	152.1	C57 H27 O F3
	784.2037	-1.3	-1.7	35.0	374.9	C51 H29 O3 F5
→	784.2048	-2.4	-3.1	31.0	525.8	C48 H30 O4 F6
	784.2050	-2.6	-3.3	42.0	162.8	C56 H29 O4 F
	784.2060	-3.6	-4.6	27.0	703.6	C45 H31 O5 F7
	784.2061	-3.7	-4.7	38.0	264.7	C53 H30 O5 F2

12c. **13,16-dimethoxy-1,4,7,10-tetraphenyl-13,16-bis(trifluoromethyl)cyclooctadeca-1,2,3,7,8,9-hexaen-5,11,14,17-tetrayne**

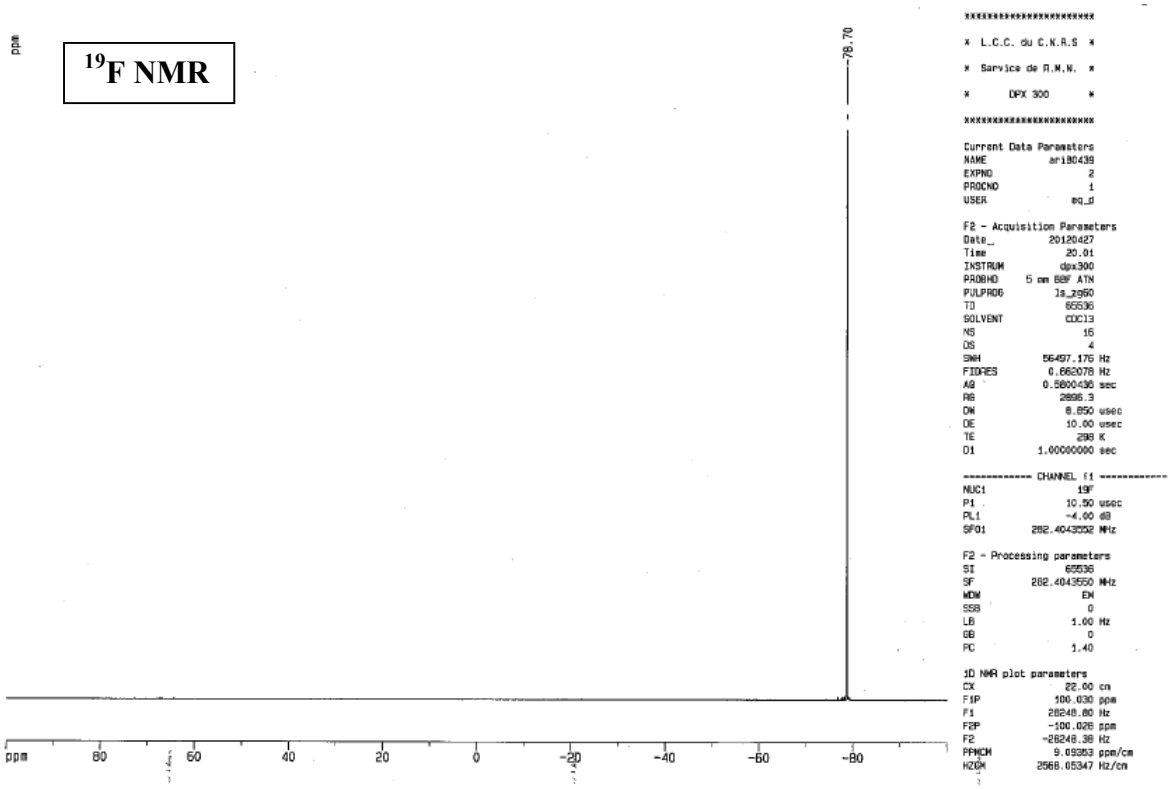
^{1}H NMR



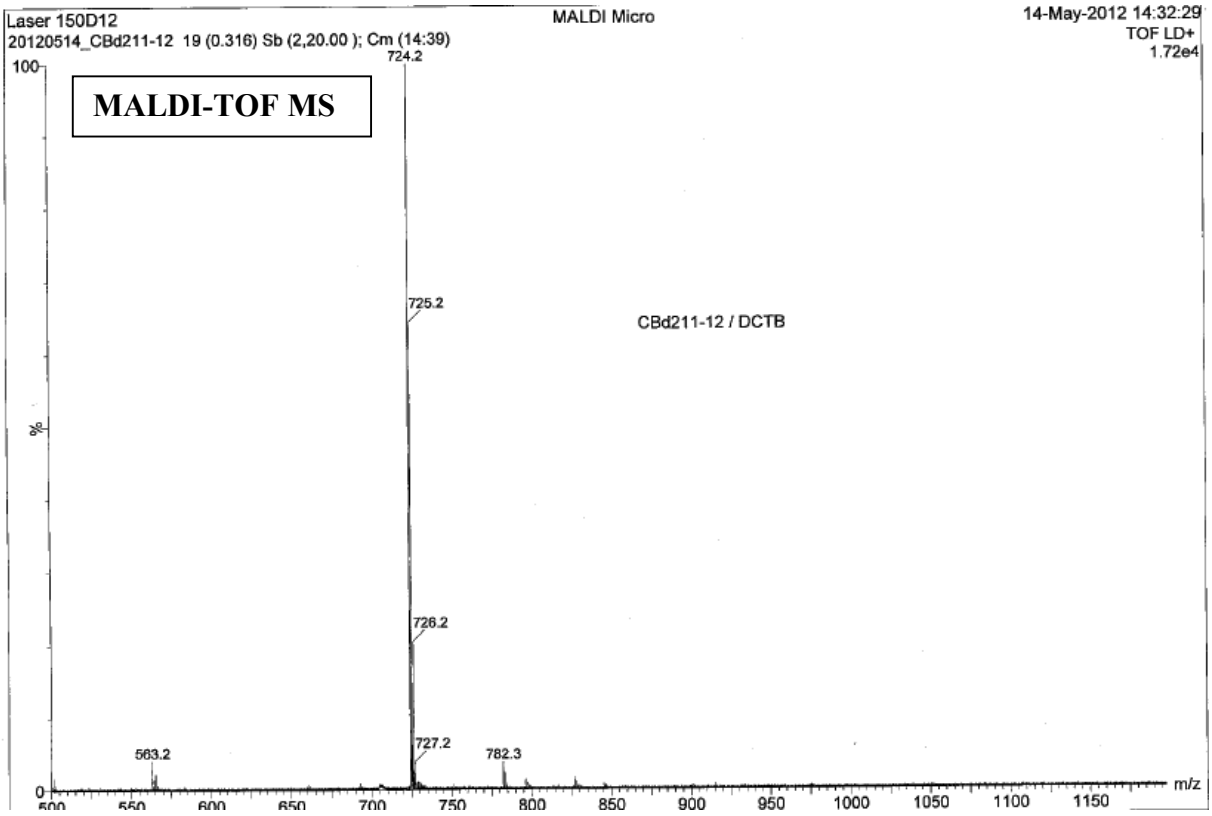
^{13}C{^{1}H} NMR

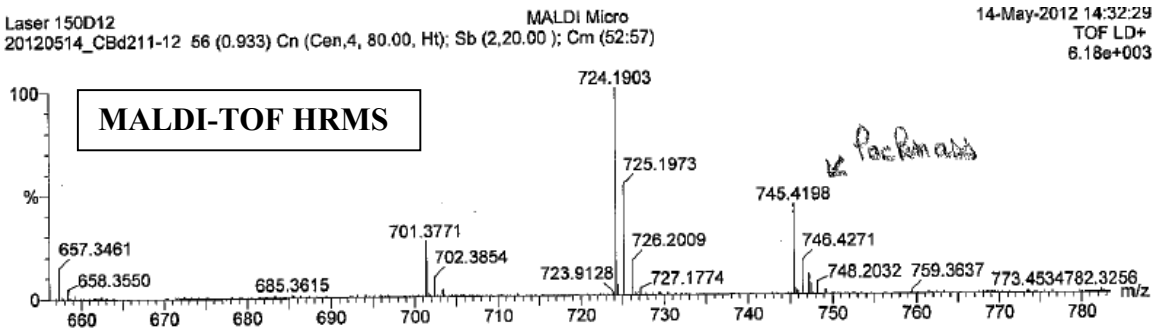


^{19}F NMR



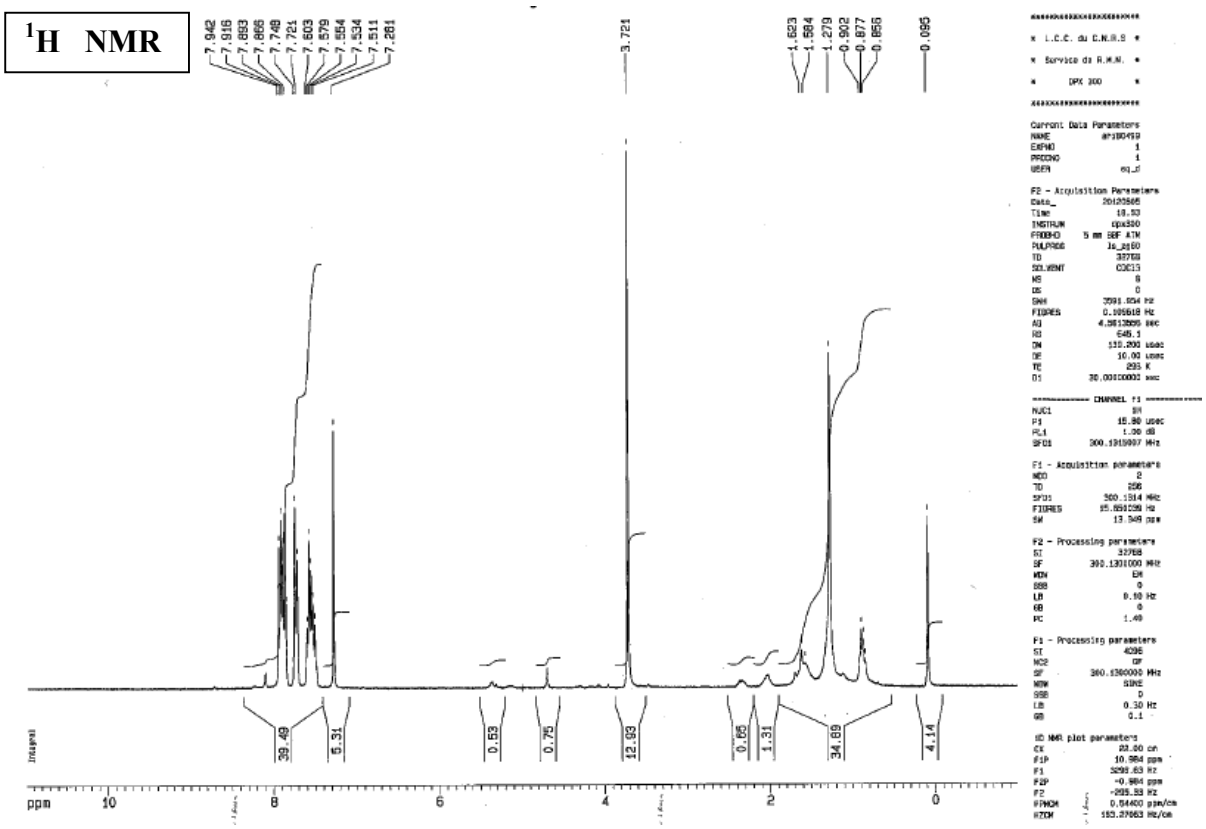
MALDI-TOF MS



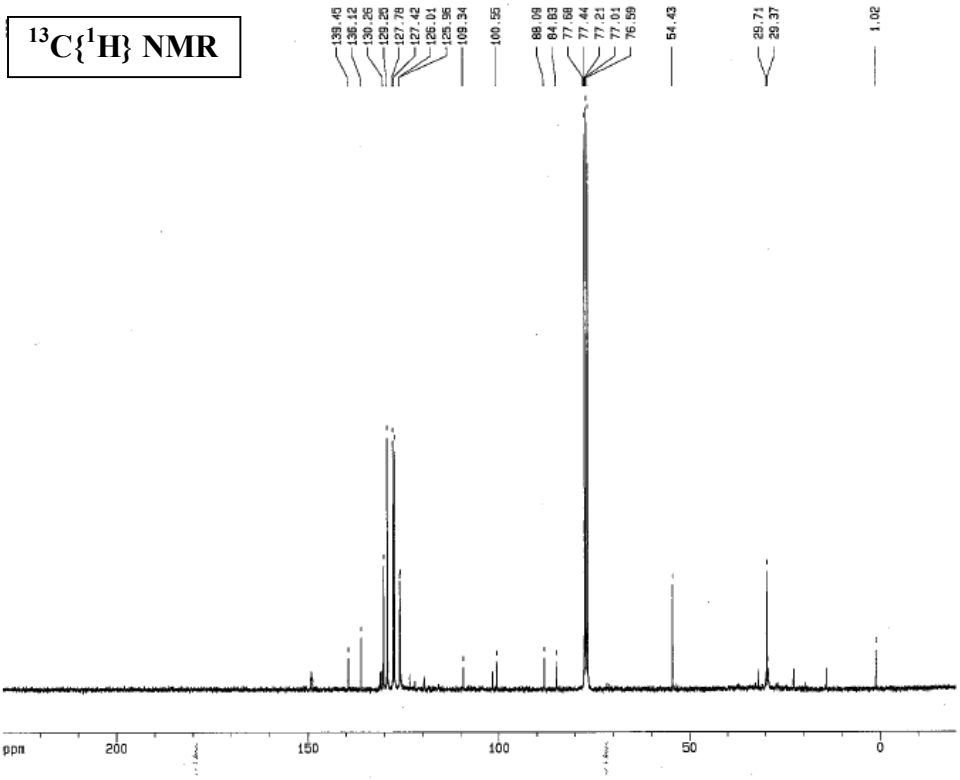


Minimum:			-1.5			
Maximum:		5.0	10.0	70.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
724.1903	724.1861	4.2	5.8	34.0	6.4	C48 H27 O4 F3
	724.1850	5.3	7.3	38.0	8.4	C51 H26 O3 F2
	724.1873	3.0	4.1	30.0	17.6	C45 H28 O5 F4
	724.1837	6.6	9.1	31.0	20.4	C46 H26 O2 F6 ←
	724.1839	6.4	8.8	42.0	22.8	C54 H25 O2 F
	724.1848	5.5	7.6	27.0	43.6	C43 H27 O3 F7
	724.1860	4.3	5.9	23.0	81.1	C40 H28 O4 F8
	724.1871	3.2	4.4	19.0	133.6	C37 H29 O5 F9

12d. **13,16-dimethoxy-4,7-diphenyl-13,16-bis(trifluoromethyl)-1,10-bis[4-(trifluoromethyl) phenyl]cyclooctadeca-1,2,3,7,8,9-hexaen-5,11,14,17-tetrayne**



$^{13}C\{^{1}H\}$ NMR




```
**********
* L.C.C. du C.N.R.S *
* Service de R.M.N. *
*      DPX 300      *
**********

Current Data Parameters
NAME            ari80510
EXPNO                  5
PROCNO                 1
USER               eq_d

F2 - Acquisition Parameters
Date_          20120510
Time               8.57
INSTRUM          dpx300
PROBHD   5 mm BBF ATM
PULPROG       ls_zg60dc
TD                32768
SOLVENT           CDCl3
NS                27515
DS                    4
SWH           18832.393 Hz
FIDRES        0.574719 Hz
AQ            0.8701404 sec
RG                14596.5
DW                26.550 usec
DE                10.00 usec
TE                 298 K
D1            1.00000000 sec
d11           0.03000000 sec

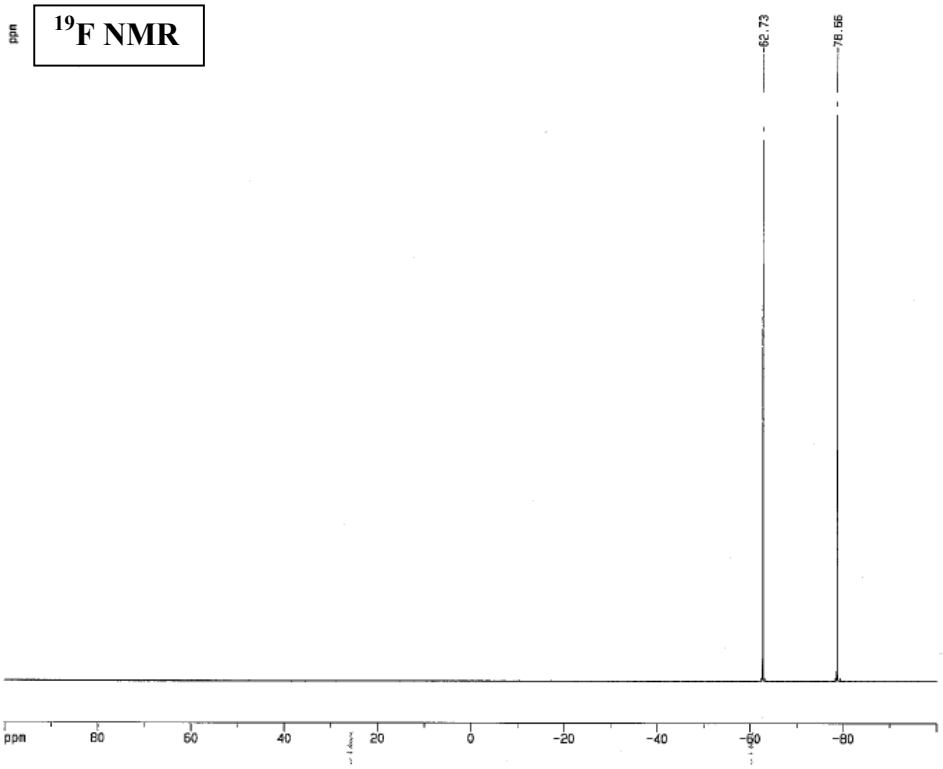
========== CHANNEL f1 ==========
NUC1                13C
P1                11.80 usec
PL1               0.00 dB
SFO1         75.4755728 MHz

========== CHANNEL f2 ==========
CPDPRG2          waltz16
NUC2                1H
PCPD2             90.00 usec
PL2               1.00 dB
PL12              17.00 dB
SFO2         300.1312005 MHz

F2 - Processing parameters
SI                32768
SF           75.4677490 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                22.00 cm
F1P              229.765 ppm
F1               17339.84 Hz
F2P              -19.777 ppm
F2               -1492.56 Hz
PPMCM            11.34283 ppm/cm
HZCM             856.01762 Hz/cm
```

^{19}F NMR

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**********
* L.C.C. du C.N.R.S *
* Service de R.M.N. *
*      DPX 300      *
**********

Current Data Parameters
NAME            ari80499
EXPNO                  2
PROCNO                 1
USER               eq_d

F2 - Acquisition Parameters
Date_          20120505
Time              19.55
INSTRUM          dpx300
PROBHD   5 mm BBF ATM
PULPROG          ls_zg60
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    4
SWH           56497.176 Hz
FIDRES        0.862078 Hz
AQ            0.5800436 sec
RG                2896.3
DW                8.850 usec
DE                10.00 usec
TE                 298 K
D1            1.00000000 sec

========== CHANNEL f1 ==========
NUC1                19F
P1                10.50 usec
PL1               -4.00 dB
SFO1         282.4043552 MHz

F2 - Processing parameters
SI                65536
SF           282.4043550 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                22.00 cm
F1P              100.030 ppm
F1               28248.80 Hz
F2P              -100.028 ppm
F2               -28248.38 Hz
PPMCM            9.09353 ppm/cm
HZCM             2568.05347 Hz/cm
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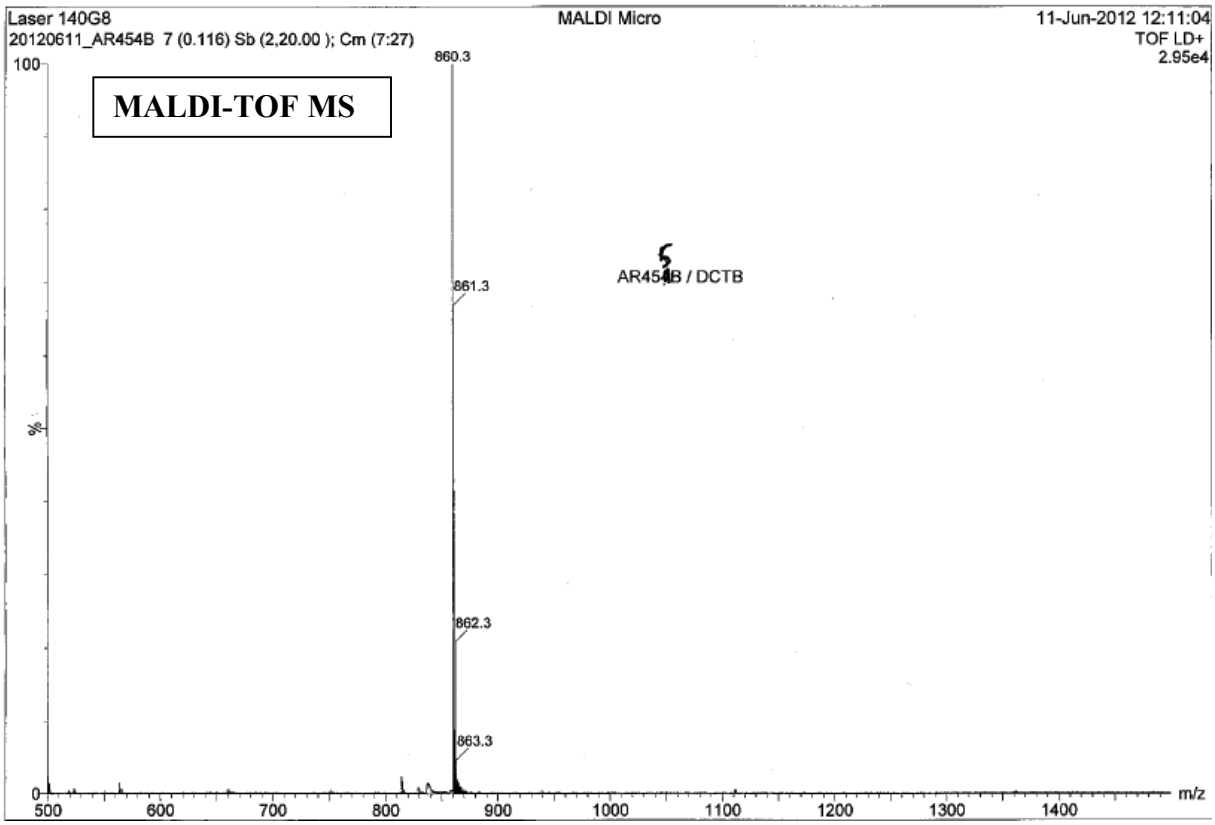
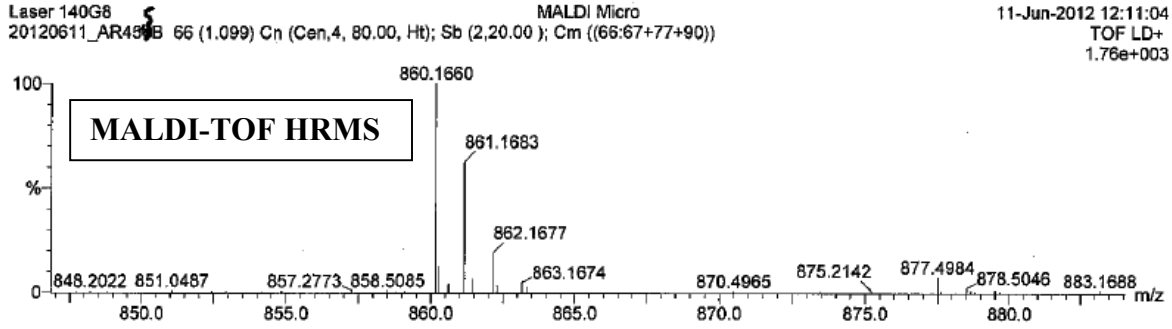


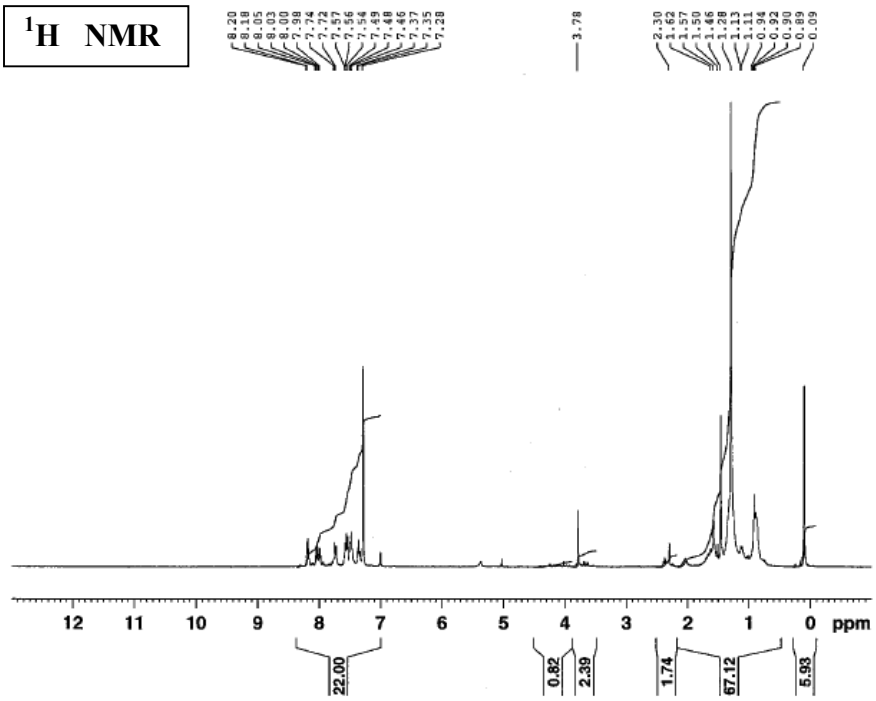


Minimum:				-1.5		
Maximum:		5.0	10.0	90.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
860.1660	860.1586	7.4	8.6	42.0	0.7	C56 H23 O2 F7
	860.1598	6.2	7.2	38.0	1.4	C53 H24 O3 F8
	860.1575	8.5	9.9	46.0	3.7	C59 H22 O F6
	860.1609	5.1	5.9	34.0	5.8	C50 H25 O4 F9
	860.1585	7.5	8.7	31.0	13.3	C48 H24 O2 F12
	860.1596	6.4	7.4	27.0	25.2	C45 H25 O3 F13

12e. **9-(4-{10-[4-(9*H*-carbazol-9-yl)phenyl]-13,16-dimethoxy-4,7-diphenyl-13,16-bis(trifluoromethyl)cyclooctadeca-1,2,3,7,8,9-hexaen-5,11,14,17-tetrayn-1-yl}phenyl)-9H-carbazole**

bi-2.109.1
Night_H1_int_NS_8 CDCl3 /x/av400pas/data/eq_d/nmr i.baglai 1

^{1}H NMR



BRUKER

```
Current Data Parameters
NAME                  ibaG0451
EXPNO                        3
PROCNO                       1

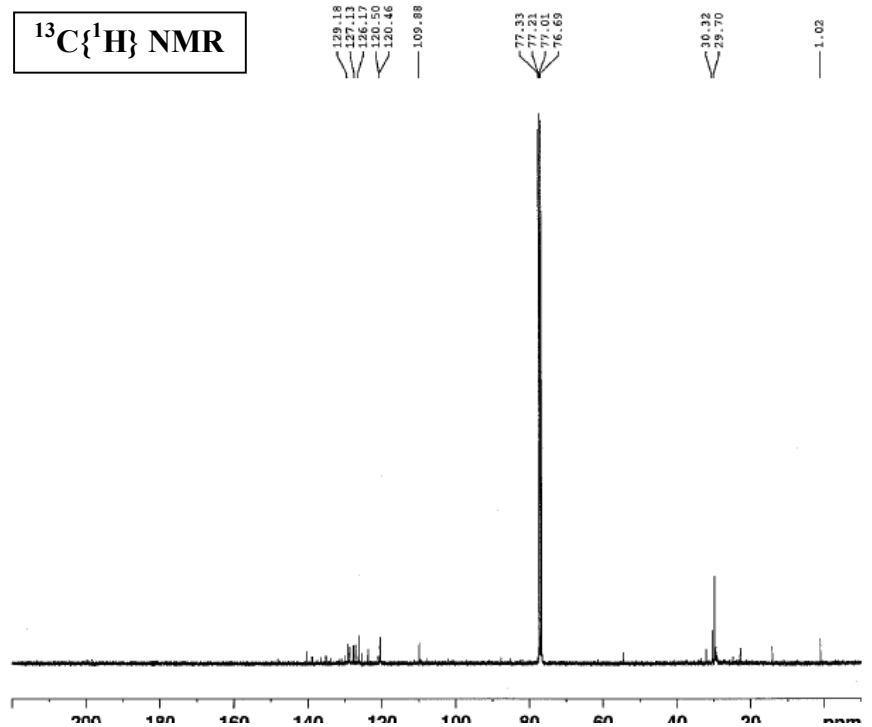
F2 - Acquisition Parameters
Date_                 20120717
Time                      2.35
INSTRUM                  spect
PROBHD     5 mm PABBO BB-
PULPROG               1s_zg60
TD                       65536
SOLVENT                  CDCl3
NS                           8
DS                           2
SWH                5597.015 Hz
FIDRES             0.085404 Hz
AQ                 5.8545995 sec
RG                         114
DW                    89.333 usec
DE                     6.50 usec
TE                     298.0 K
D1                20.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        1H
P1                     13.60 usec
PLW1              17.00000000 W
SFO1             400.1624010 MHz

F2 - Processing parameters
SI                       131072
SF                400.1600000 MHz
WDW                          EM
SSB          0
LB                     0.10 Hz
GB           0
PC                     1.50
```

bi-2.109.1
Night_C13_DECOUPLE_H1_NS_5000 CDCl3 /x/av400pas/data/eq_d/nmr i.baglai 1

^{13}C{^{1}H} NMR



BRUKER

```
Current Data Parameters
NAME                  ibaG0451
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20120717
Time                      6.03
INSTRUM                  spect
PROBHD     5 mm PABBO BB-
PULPROG             1s_zgdc60
TD                       65536
SOLVENT                  CDCl3
NS                        5000
DS                           4
SWH               23148.148 Hz
FIDRES             0.353213 Hz
AQ                 1.4156276 sec
RG                        2050
DW                    21.600 usec
DE                     6.50 usec
TE                     298.0 K
D1                 1.00000000 sec
D11                0.03000000 sec
TD0                          1

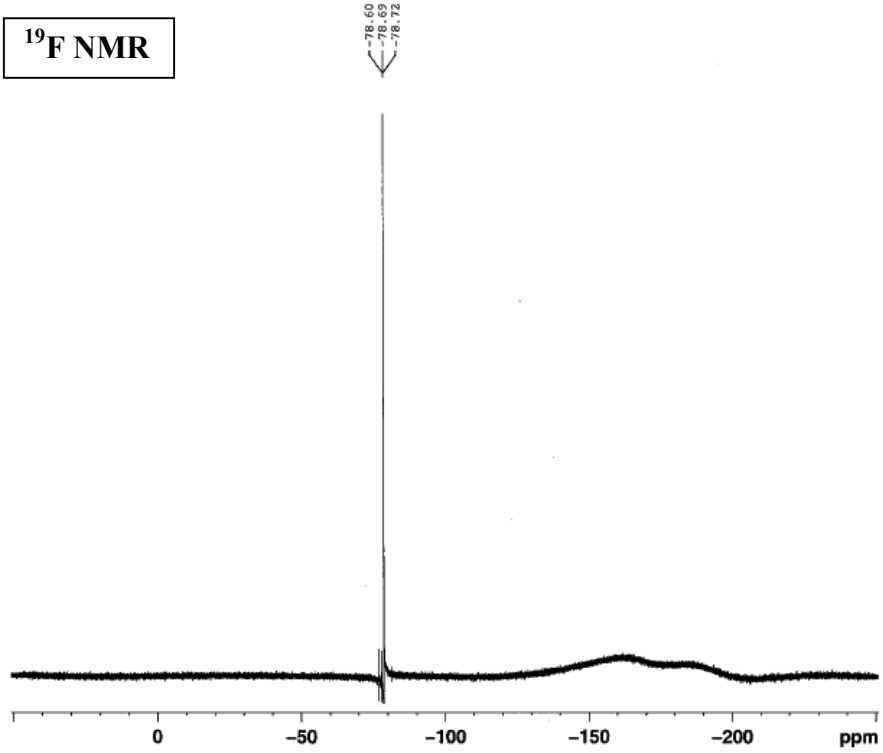
======== CHANNEL f1 ========
NUC1                       13C
P1                      9.00 usec
PLW1              70.00000000 W
SFO1             100.6308781 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                        1H
PCPD2                  90.00 usec
PLW2              17.00000000 W
PLW12              0.38819000 W
SFO2             400.1616006 MHz

F2 - Processing parameters
SI                       131072
SF                100.6203130 MHz
WDW                          EM
SSB          0
LB                     1.00 Hz
GB           0
PC                     1.50
```

bi-2.109.1
Night_F19_NS_40 CDCl3 /x/av400pas/data/eq_d/nmr i.baglai 1

^{19}F NMR




```
Current Data Parameters
NAME                  ibaG0451
EXPNO                        2
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20120717
Time                      6.08
INSTRUM                  spect
PROBHD    5 mm PABBO BB-
PULPROG                1s_zg60
TD                       131072
SOLVENT                  CDCl3
NS                          40
DS                           4
SWH                 113636.367 Hz
FIDRES                0.866977 Hz
AQ                    0.5767668 sec
RG                         406
DW                       4.400 usec
DE                       6.50 usec
TE                       298.0 K
D1                    1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       19F
P1                       11.65 usec
PLW1                25.00000000 W
SFO1                376.4889413 MHz

F2 - Processing parameters
SI                       65536
SF                  376.5265940 MHz
WDW                         EM
SSB                  0
LB                       0.30 Hz
GB                   0
PC                       1.00
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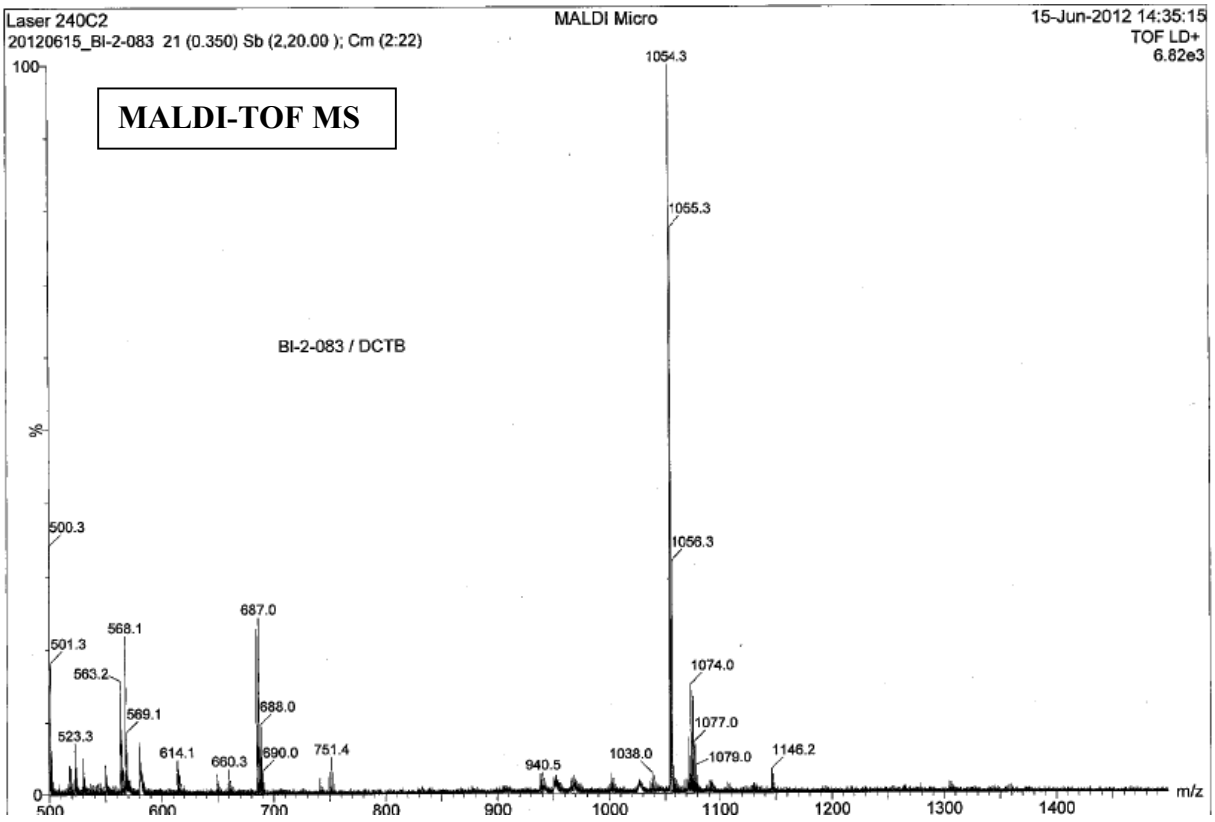
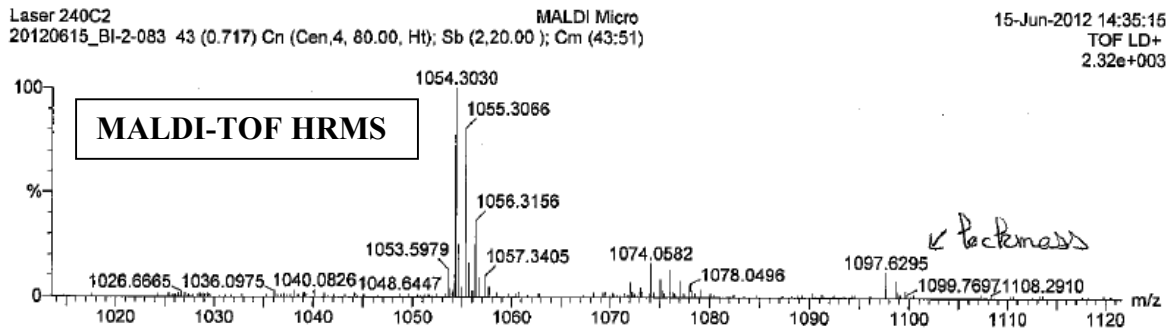
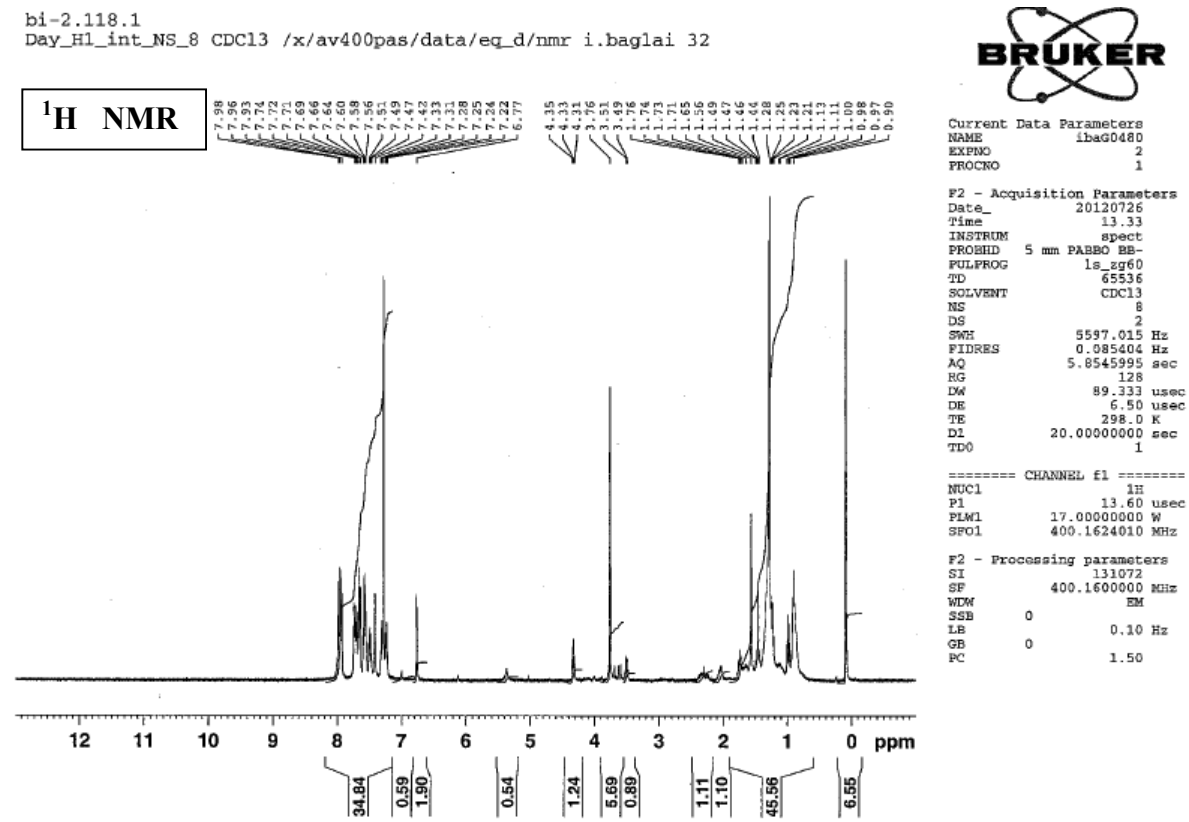




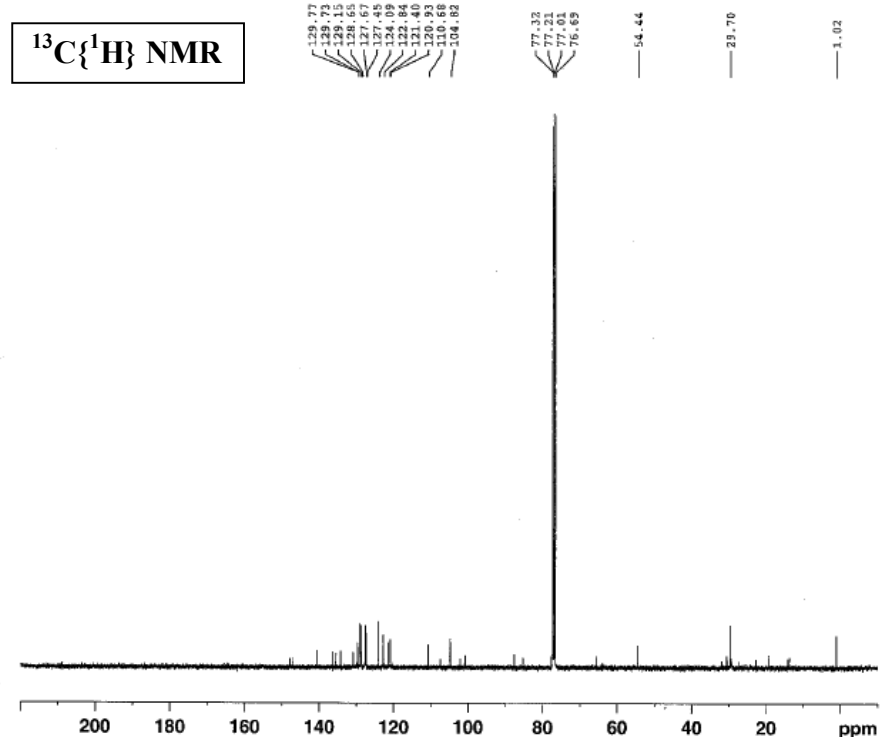
Minimum:				-1.5		
Maximum:		5.0	5.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
1054.3030	1054.3046	-1.6	-1.5	49.0	4.4	C72 H41 O F7
	1054.3082	-5.2	-4.9	48.0	4.6	C71 H43 O4 F5
	1054.2994	3.6	3.4	49.0	6.5	C70 H40 N2 O2 F6
	1054.3032	-0.2	-0.2	49.5	6.8	C70 H39 N3 F7
	1054.3068	-3.8	-3.6	48.5	7.1	C69 H41 N3 O3 F5
	1054.3057	-2.7	-2.6	45.0	10.2	C69 H42 O2 F8
	1054.3005	2.5	2.4	45.0	13.8	C67 H41 N2 O3 F7
	1054.3044	-1.4	-1.3	45.5	14.3	C67 H40 N3 O F8
	1054.3080	-5.0	-4.7	44.5	15.2	C66 H42 N3 O4 F6
	1054.3017	1.3	1.2	41.0	25.0	C64 H42 N2 O4 F8

12f. **1-(4-{10-[4-(1H-indol-1-yl)phenyl]-13,16-dimethoxy-4,7-diphenyl-13,16-bis (trifluoromethyl)cyclooctadeca-1,2,3,7,8,9-hexaen-5,11,14,17-tetrayn-1-yl}phenyl)-1H-indole**



bi-2.118.1
Night_C13_DECOUPLE_H1_NS_5000 CDCl3 /x/av400pas/data/eq_d/nmr i.baglai 32

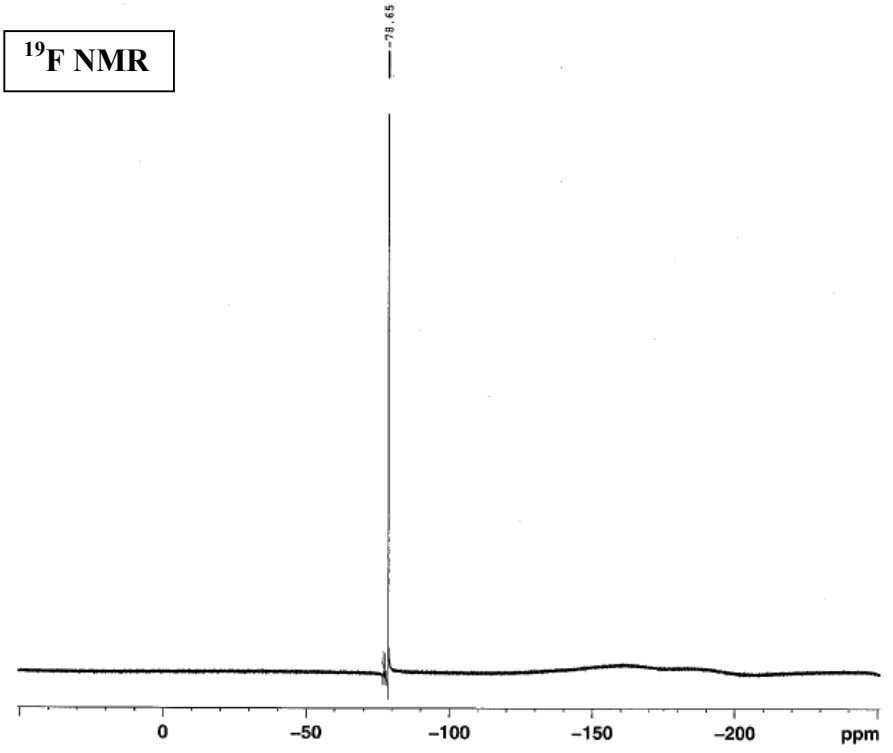
$^{13}C\{^{1}H\}$ NMR



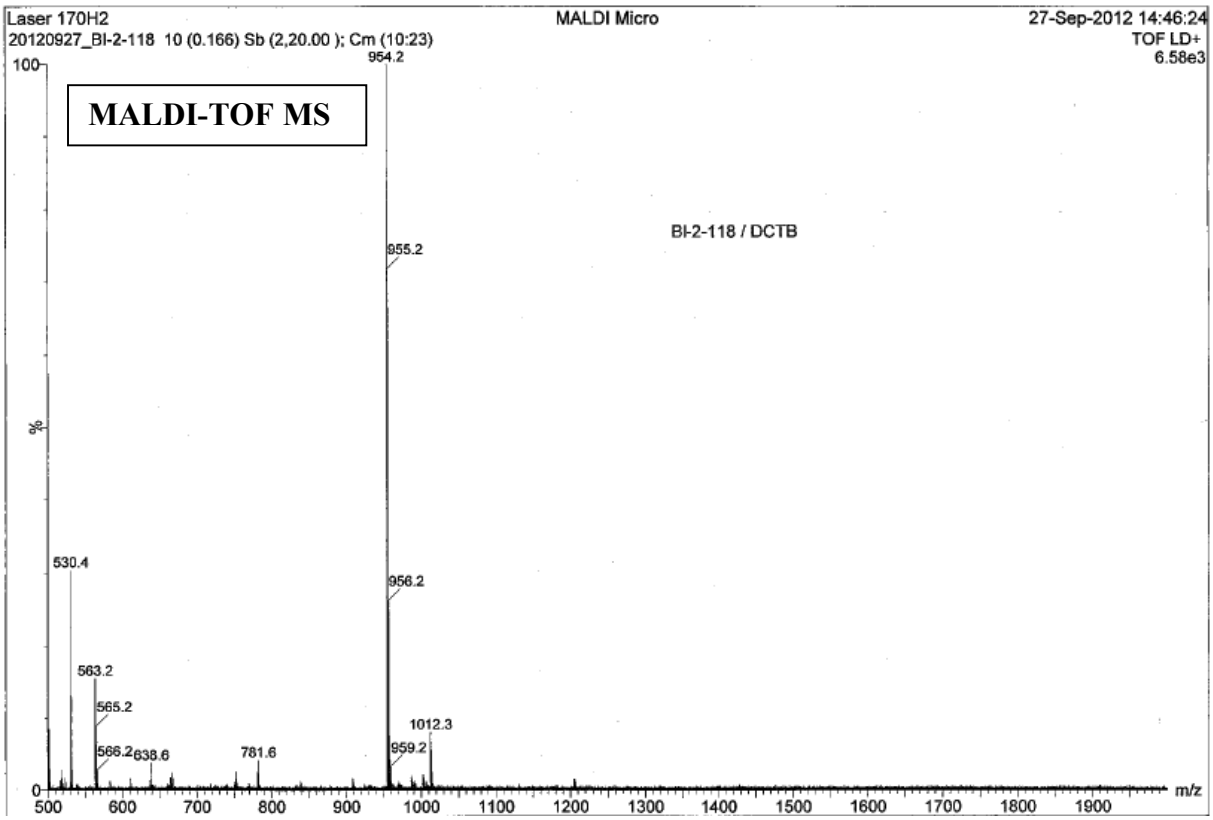

```
Current Data Parameters
NAME                  ibaG0480
EXPNO                        3
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20120727
Time                      2.02
INSTRUM                  spect
PROBHD    5 mm PABBO BB-
PULPROG              ls_zgdc60
TD                       65536
SOLVENT                  CDCl3
NS                        5000
DS                           4
SWH                 23148.148 Hz
FIDRES               0.353213 Hz
AQ                   1.4156276 sec
RG                        2050
DW                      21.600 usec
DE                       6.50 usec
TE                     298.0 K
D1                   1.00000000 sec
D11                  0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       13C
P1                        9.00 usec
PLW1                70.00000000 W
SFO1               100.6308781 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                       1H
PCPD2                    90.00 usec
PLW2                17.00000000 W
PLW12                0.38819000 W
SFO2               400.1616006 MHz

F2 - Processing parameters
SI                       131072
SF                 100.6203130 MHz
WDW                          EM
SSB          0
LB                        1.00 Hz
GB           0
PC                        1.50
```

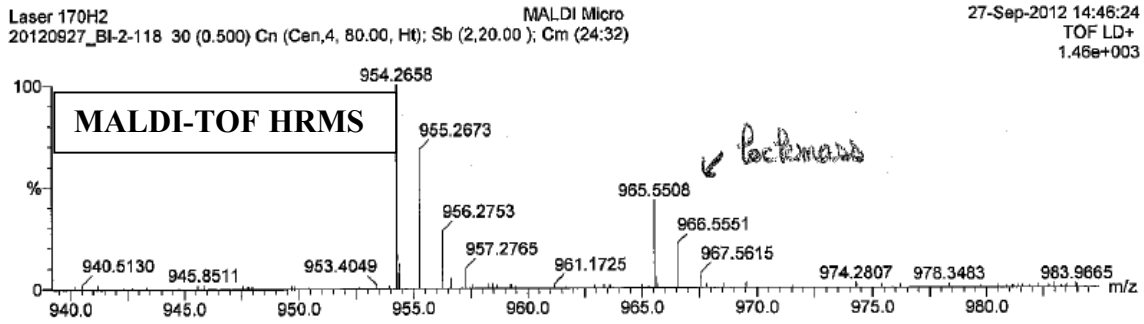
bi-2.118.1
Day_F19_NS_40 CDCl3 /x/av400pas/data/eq_d/nmr i.baglai 32

^{19}F NMR



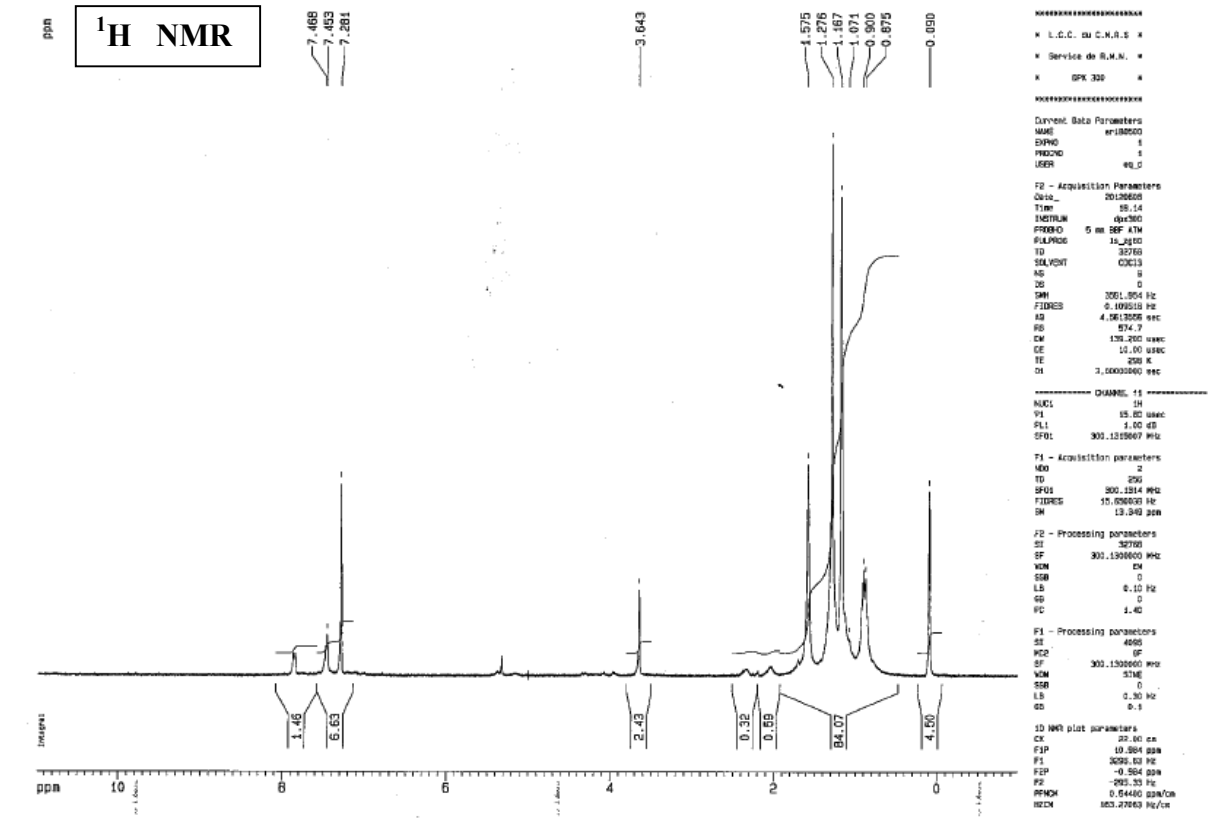

```
Current Data Parameters
NAME                  ibaG0480
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20120726
Time                     13.26
INSTRUM                  spect
PROBHD    5 mm PABBO BB-
PULPROG               ls_zg60
TD                       131072
SOLVENT                  CDCl3
NS                           40
DS                           4
SWH                 113636.367 Hz
FIDRES               0.866977 Hz
AQ                   0.5767668 sec
RG                         645
DW                      4.400 usec
DE                       6.50 usec
TE                     298.0 K
D1                   1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       19F
P1                       11.65 usec
PLW1                25.00000000 W
SFO1               376.4889413 MHz

F2 - Processing parameters
SI                        65536
SF                 376.5265940 MHz
WDW                          EM
SSB          0
LB                        0.30 Hz
GB           0
PC                        1.00
```

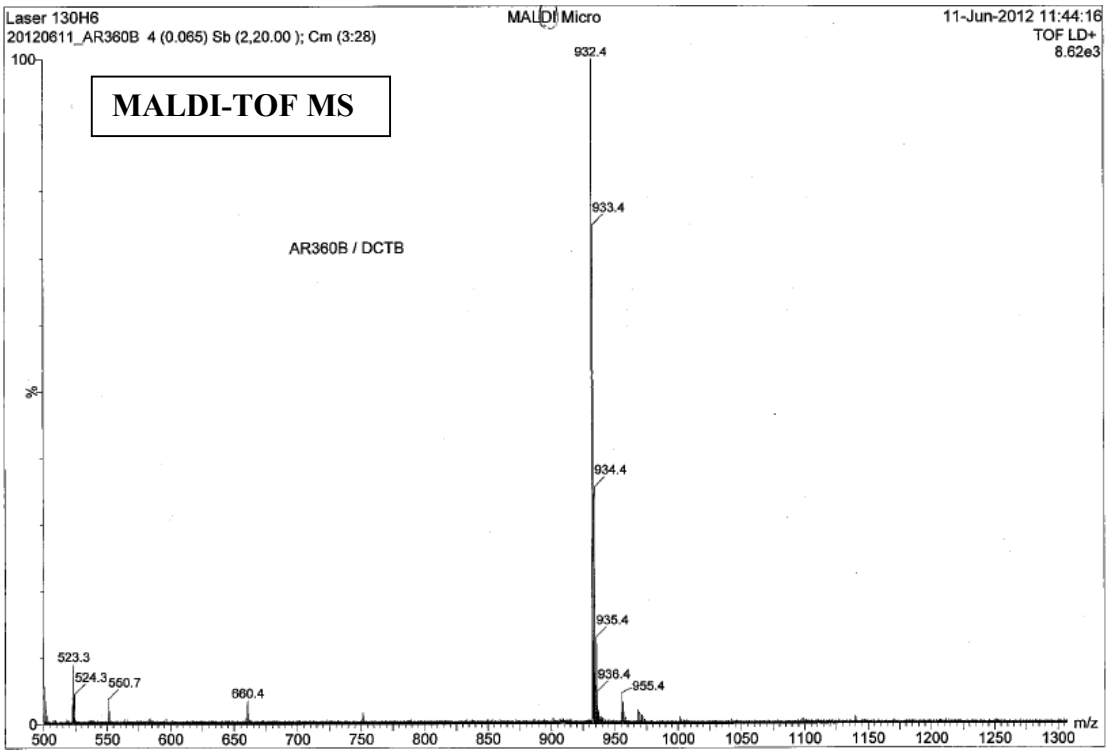


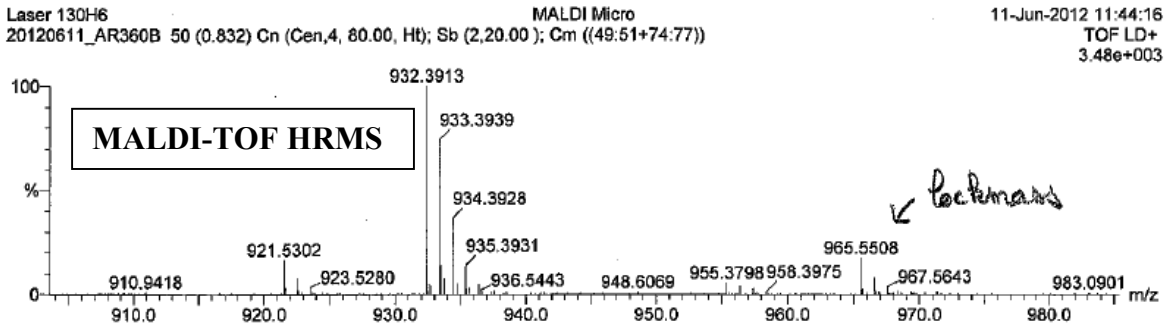


Minimum:				-1.5		
Maximum:		5.0	5.0	55.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
954.2658	954.2631	2.7	2.8	46.5	2.5	C65 H36 N O3 F4
	954.2670	-1.2	-1.3	47.0	2.9	C65 H35 N2 O F5
	954.2681	-2.3	-2.4	43.0	3.5	C62 H36 N2 O2 F6
	954.2620	3.8	4.0	50.5	4.4	C68 H35 N O2 F3
	954.2658	0.0	0.0	51.0	4.6	C68 H34 N2 F4
	954.2618	4.0	4.2	39.5	6.1	C60 H36 N O2 F8
	954.2692	-3.4	-3.6	39.0	6.6	C59 H37 N2 O3 F7

12g. **{2-[13,16-dimethoxy-4,7-diphenyl-13,16-bis(trifluoromethyl)-10-{2-[tris(propan-2-yl)silyl]ethynyl}cyclooctadeca-1,2,3,7,8,9-hexaen-5,11,14,17-tetrayn-1-yl]ethynyl} tris(propan-2-yl)silane**

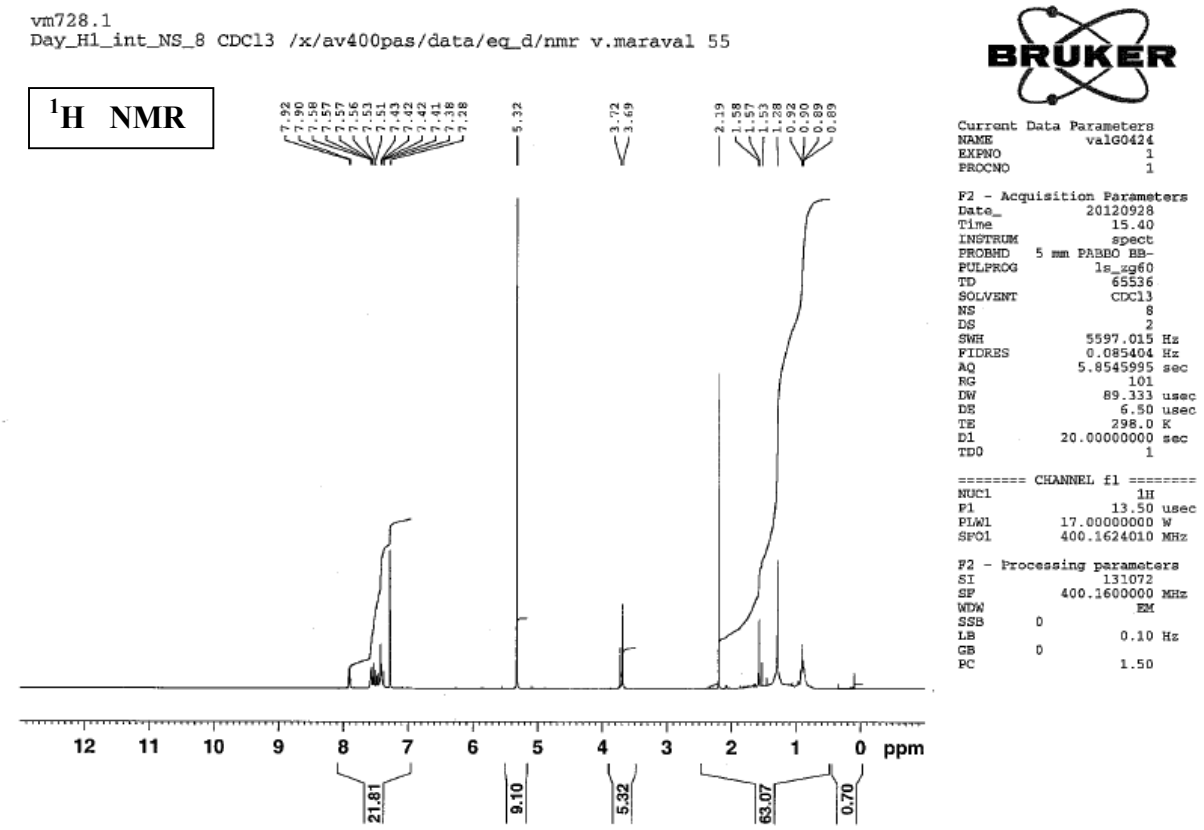






Minimum:				-1.5		
Maximum:		5.0	5.0	90.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
932.3913	932.3904	0.9	1.0	30.0	0.8	C58 H59 O4 F3 Si2
	932.3868	4.5	4.8	31.0	1.7	C59 H57 O F5 Si2
	932.3880	3.3	3.5	27.0	3.4	C56 H58 O2 F6 Si2
	932.3891	2.2	2.4	23.0	10.1	C53 H59 O3 F7 Si2
	932.3873	4.0	4.3	35.0	10.4	C62 H55 O3 F3 Si
	932.3884	2.9	3.1	31.0	19.1	C59 H56 O4 F4 Si
	932.3902	1.1	1.2	19.0	22.3	C50 H60 O4 F8 Si2
	932.3871	4.2	4.5	24.0	51.9	C54 H56 O3 F8 Si

12h. 13,16-dimethoxy-4,7-diphenyl-1,10-bis(2-phenylethynyl)-13,16-bis(trifluoromethyl) cyclooctadeca-1,2,3,7,8,9-hexaen-5,11,14,17-tetrayne



Current Data Parameters
NAME valG0424
EXPNO 1
PROCNO 1

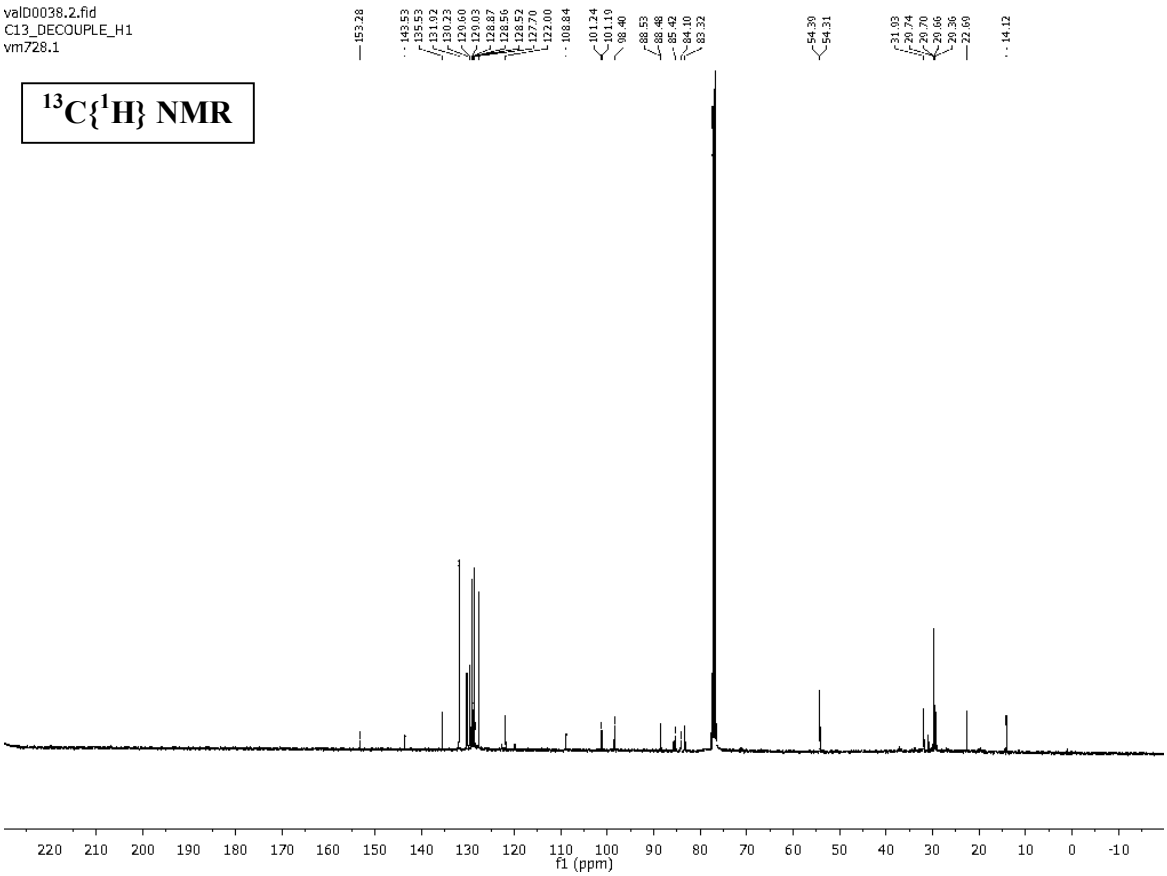
F2 - Acquisition Parameters
Date_ 20120928
Time 15.40
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG ls_zg60
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 5597.015 Hz
FIDRES 0.085404 Hz
AQ 5.8545995 sec
RG 101
DW 89.333 usec
DE 6.50 usec
TE 298.0 K
D1 20.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.50 usec
PLW1 17.00000000 W
SFO1 400.1624010 MHz

F2 - Processing parameters
SI 131072
SF 400.1600000 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.50

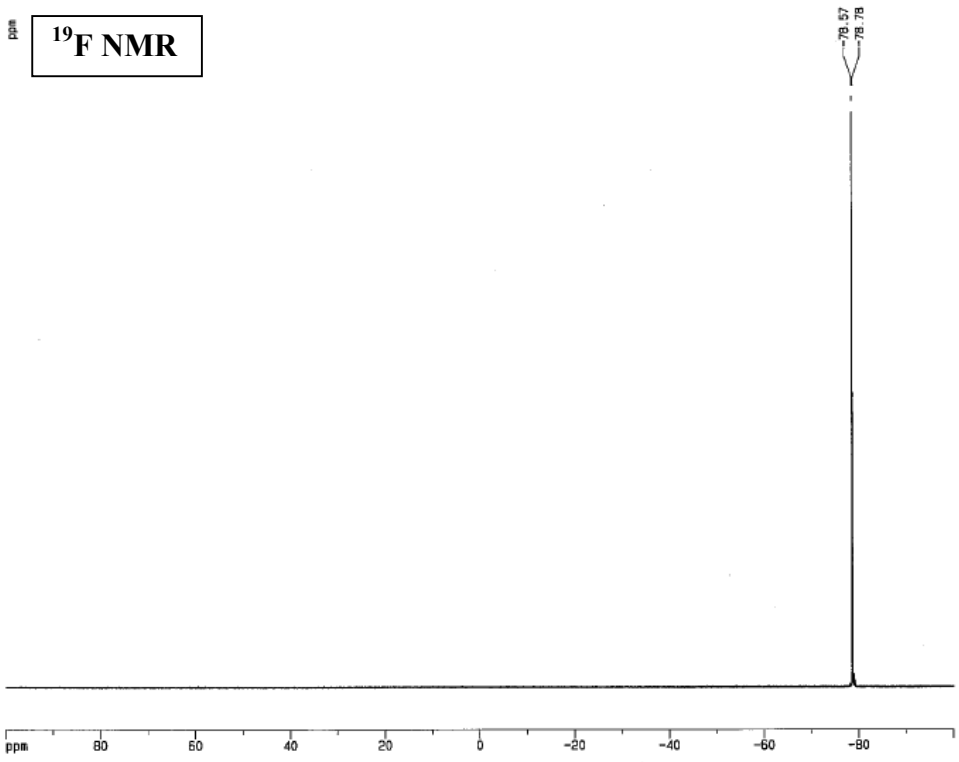
valD0038.2.fid
C13_DECOUPLE_H1
vm728.1

$^{13}C\{^{1}H\}$ NMR



F19_
vm728.1

^{19}F NMR

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* L.C.C. du C.N.R.S  *
* Service de R.M.N.  *
*      DPX 300       *
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Current Data Parameters
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EXPNO                    2
PROCNO                   1
USER                eq_d

F2 - Acquisition Parameters
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TD                  65536
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NS                     16
DS                      4
SWH              56497.176 Hz
FIDRES           0.862078 Hz
AQ               0.5800436 sec
RG                 5792.6
DW                 8.850 usec
DE                 10.00 usec
TE                  298 K
D1               1.00000000 sec

============ CHANNEL f1 ============
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P1                 10.60 usec
PL1                -4.00 dB
SF01          282.4043552 MHz

F2 - Processing parameters
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SF            282.4043550 MHz
WDW                    EM
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GB                      0
PC                   1.40

1D NMR plot parameters
CX                  22.00 cm
F1P               100.030 ppm
F1                28248.80 Hz
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F2               -28248.38 Hz
PPMCM             9.09353 ppm/cm
HZCM           2568.05347 Hz/cm
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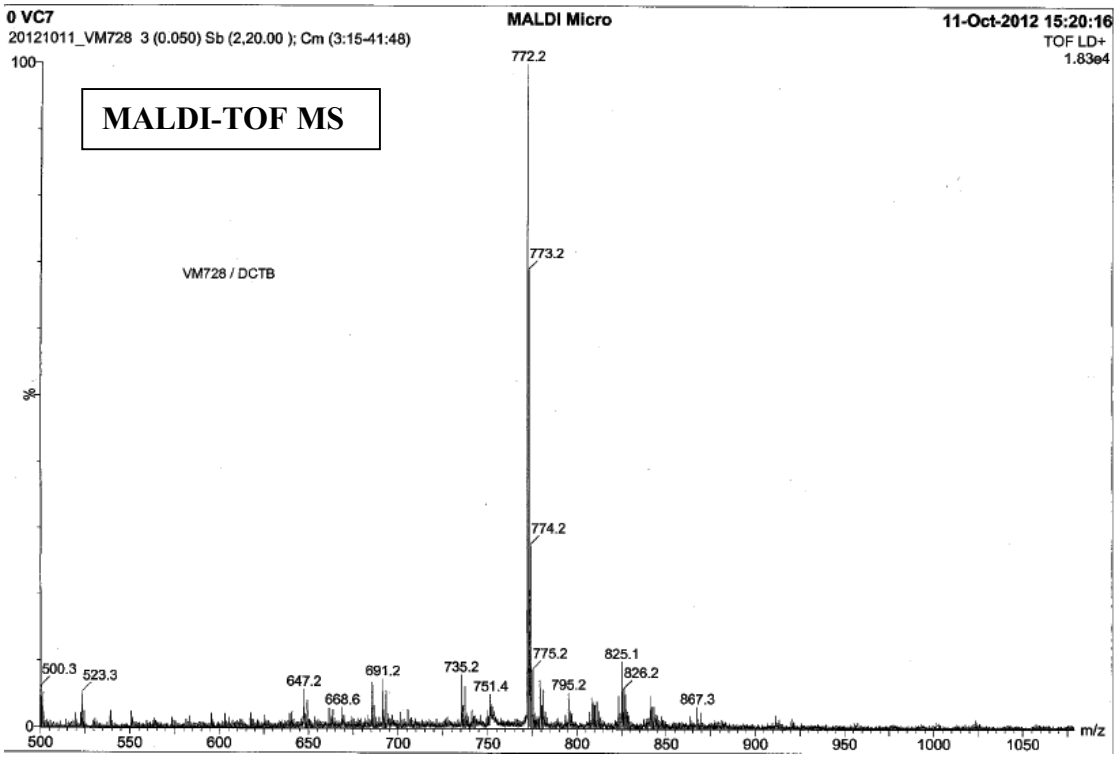
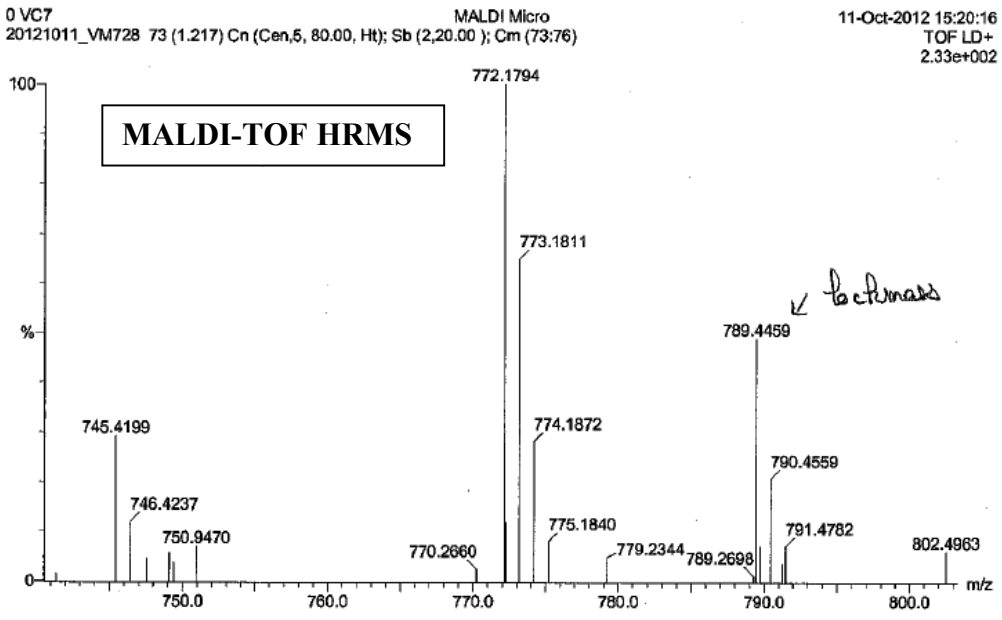


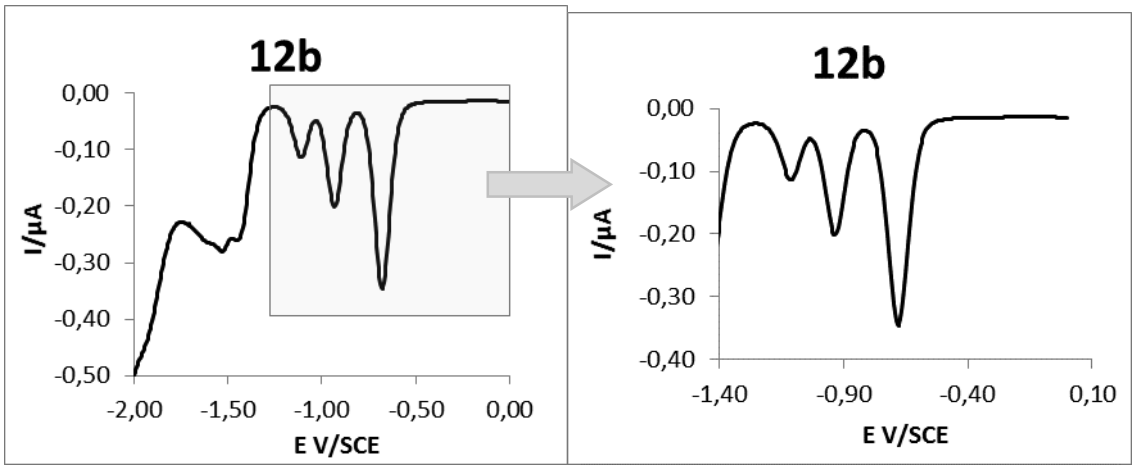


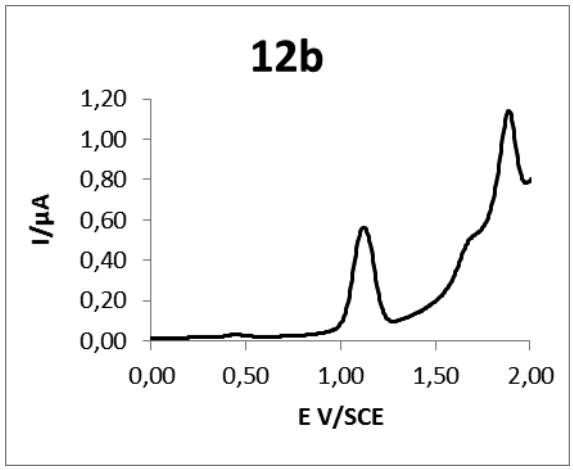
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
Minimum:				-1.5			
Maximum:		3.0	10.0	60.0			
772.1794	772.1814	-2.0	-2.6	43.0	15.8	0.5	C56 H24 F4
	772.1826	-3.2	-4.1	39.0	16.7	1.5	C53 H25 O F5
	772.1837	-4.3	-5.6	35.0	17.8	2.6	C50 H26 O2 F6
	772.1861	-6.7	-8.7	38.0	18.3	3.0	C52 H27 O4 F3
	772.1848	-5.4	-7.0	31.0	19.0	3.7	C47 H27 O3 F7
	772.1860	-6.6	-8.5	27.0	20.1	4.8	C44 H28 O4 F8

6. Voltammograms

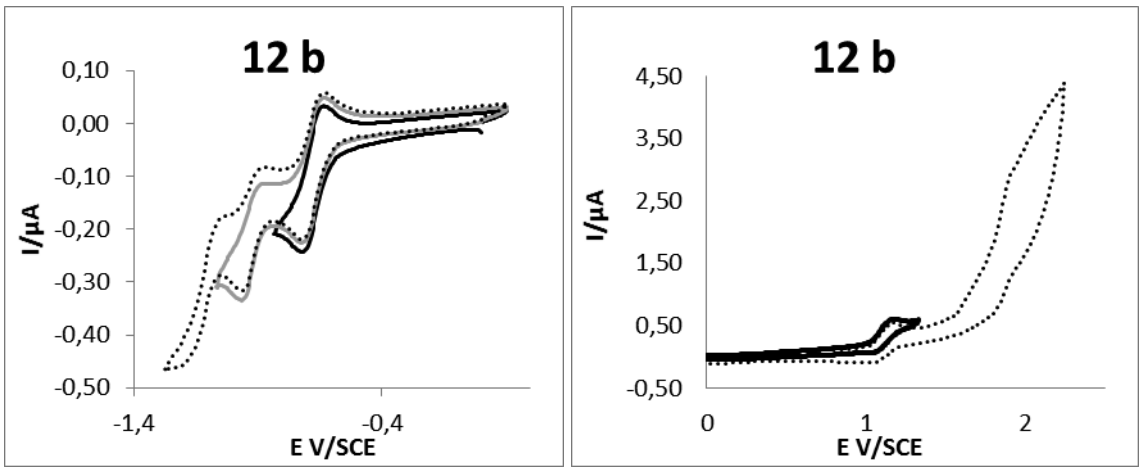
12b. 13,16-dimethoxy-1,10-bis(4-methoxyphenyl)-4,7-diphenyl-13,16-bis(trifluoromethyl) cyclooctadeca-1,2,3,7,8,9-hexaen-5,11,14,17-tetrayne

SW voltammograms for reduction and oxidation of **12b** in CH_2Cl_2 (containing 0.1 $mol.L^{-1}$ $(nBu_4N)PF_6$) on Pt microdisk (r = 0.25 mm).

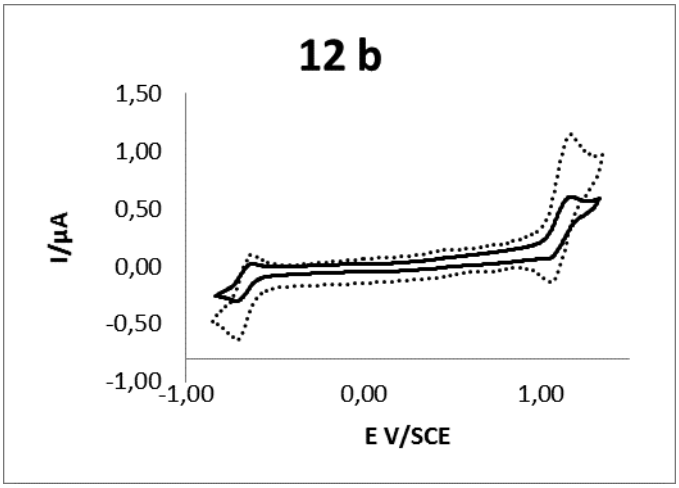




Cyclic voltammograms for reduction and oxidation of **12b** in CH_2Cl_2 (containing 0.1 $mol.L^{-1}$ $(nBu_4N)PF_6$) on Pt microdisk (r = 0.25 mm), scan rate is 0.1 V/s.

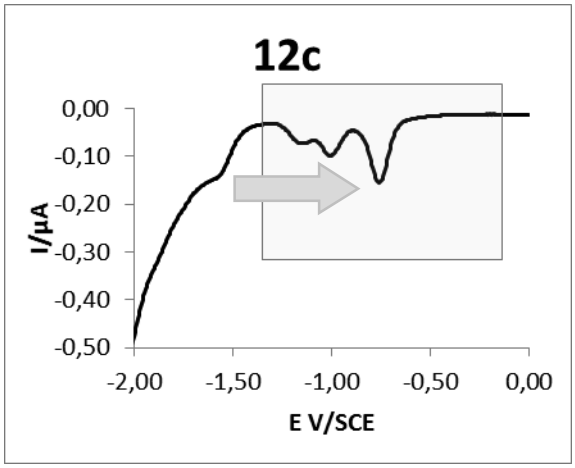


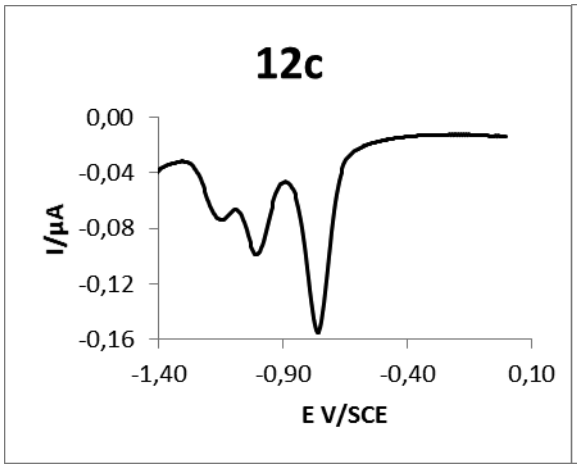
Influence of the scan rate: Cyclic voltammograms for first reduction and first oxidation of **12b** in CH_2Cl_2 (containing 0.1 $mol.L^{-1}$ $(nBu_4N)PF_6$) on Pt microdisk (r = 0.25 mm), scan rate is 0.1 V/s (solid line) and 0.5 V/s (dotted line).

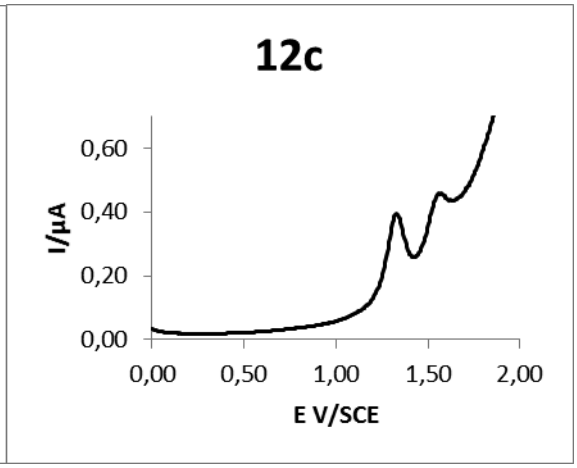


12c. **13,16-dimethoxy-1,4,7,10-tetraphenyl-13,16-bis(trifluoromethyl)cyclooctadeca-1,2,3,7,8,9-hexaen-5,11,14,17-tetrayne**

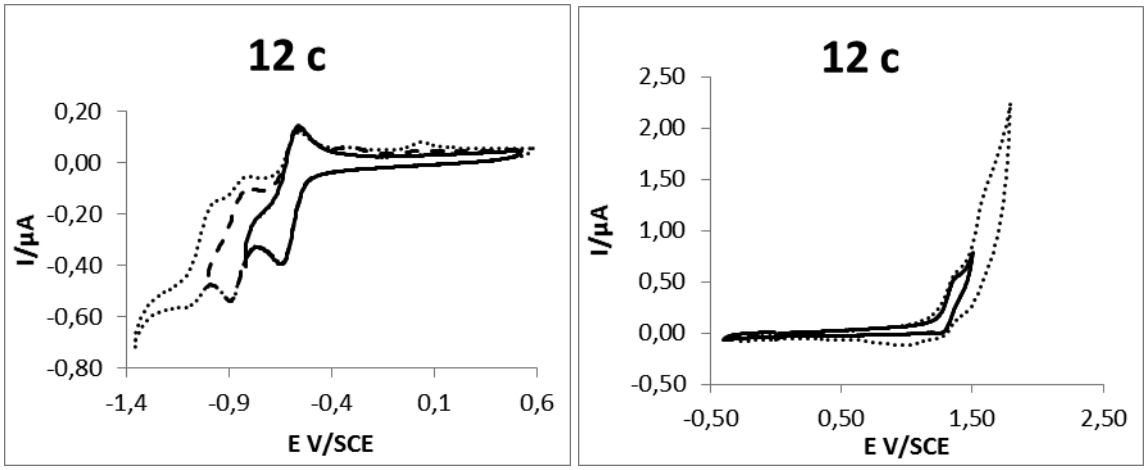
SW voltammograms for reduction and oxidation of **12c** in CH_2Cl_2 (containing 0.1 $mol.L^{-1}$ $(nBu_4N)PF_6$) on Pt microdisk (r = 0.25 mm).





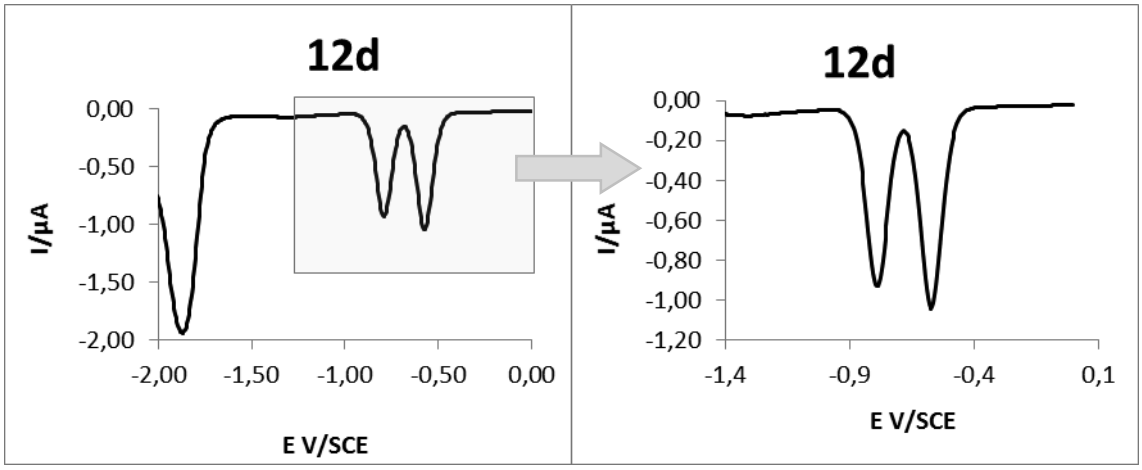


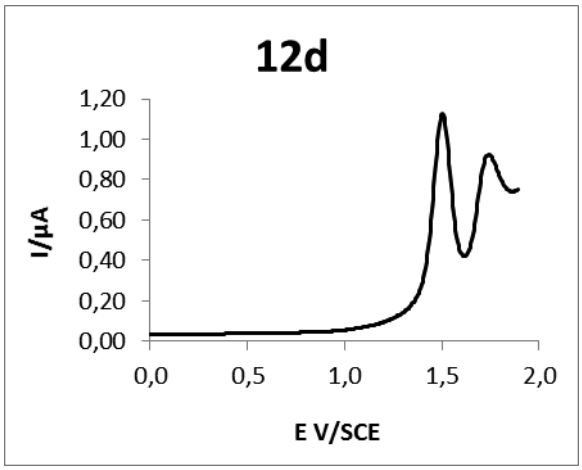
Cyclic voltammograms for reduction and oxidation of **12c** in CH_2Cl_2 (containing 0.1 mol.L^{-1} $(nBu_4N)PF_6$) on Pt microdisk (r = 0.25 mm), scan rate is 0.2 V/s



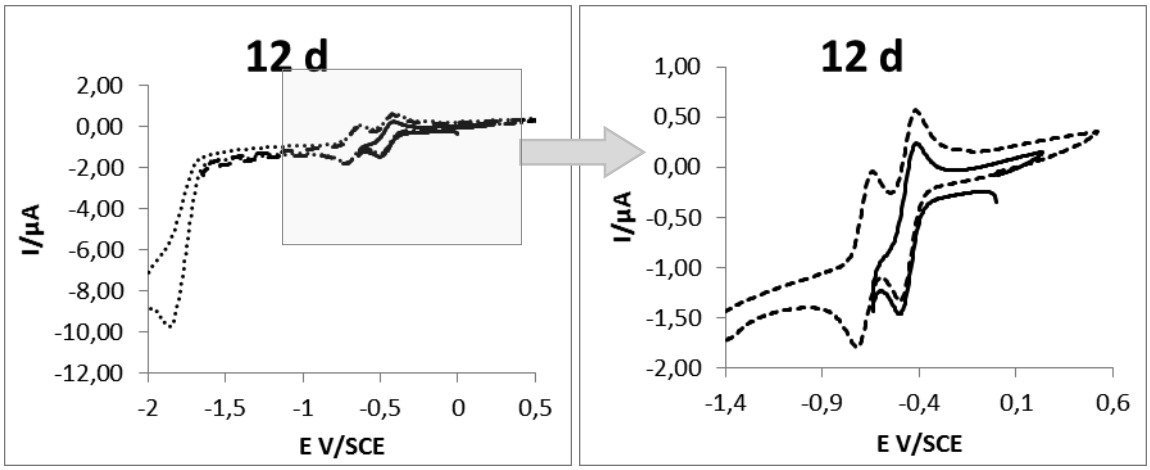
12d. 13,16-dimethoxy-4,7-diphenyl-13,16-bis(trifluoromethyl)-1,10-bis[4-(trifluoromethyl) phenyl]cyclooctadeca-1,2,3,7,8,9-hexaen-5,11,14,17-tetrayne

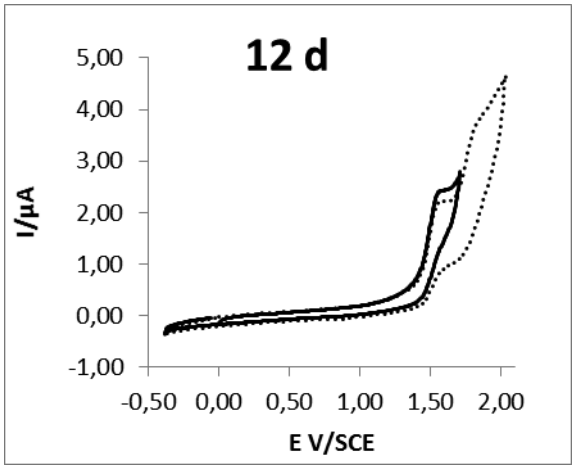
SW voltammograms for reduction and oxidation of **12d** in CH_2Cl_2 (containing 0.1 mol.L^{-1} $(nBu_4N)PF_6$) on Pt microdisk (r = 0.25 mm).





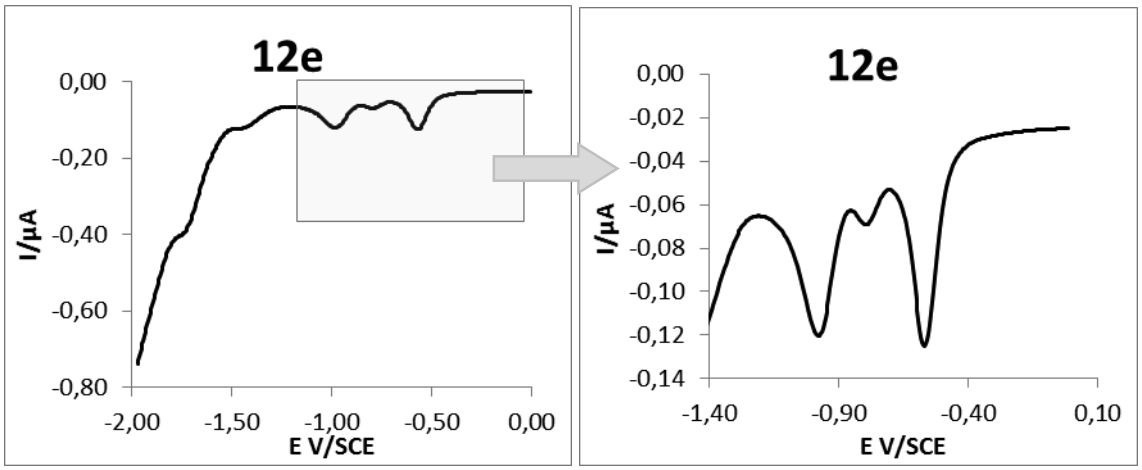
Cyclic voltammograms for reduction and oxidation of **12d** in CH_2Cl_2 (containing 0.1 $mol.L^{-1}$ $(nBu_4N)PF_6$) on Pt microdisk (r = 0.25 mm), scan rate is 0.2 V/s

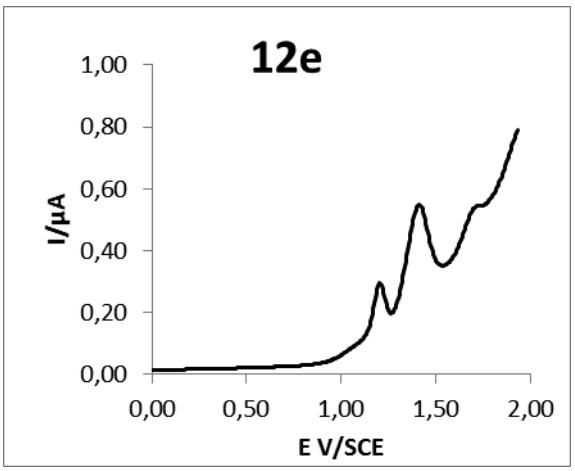




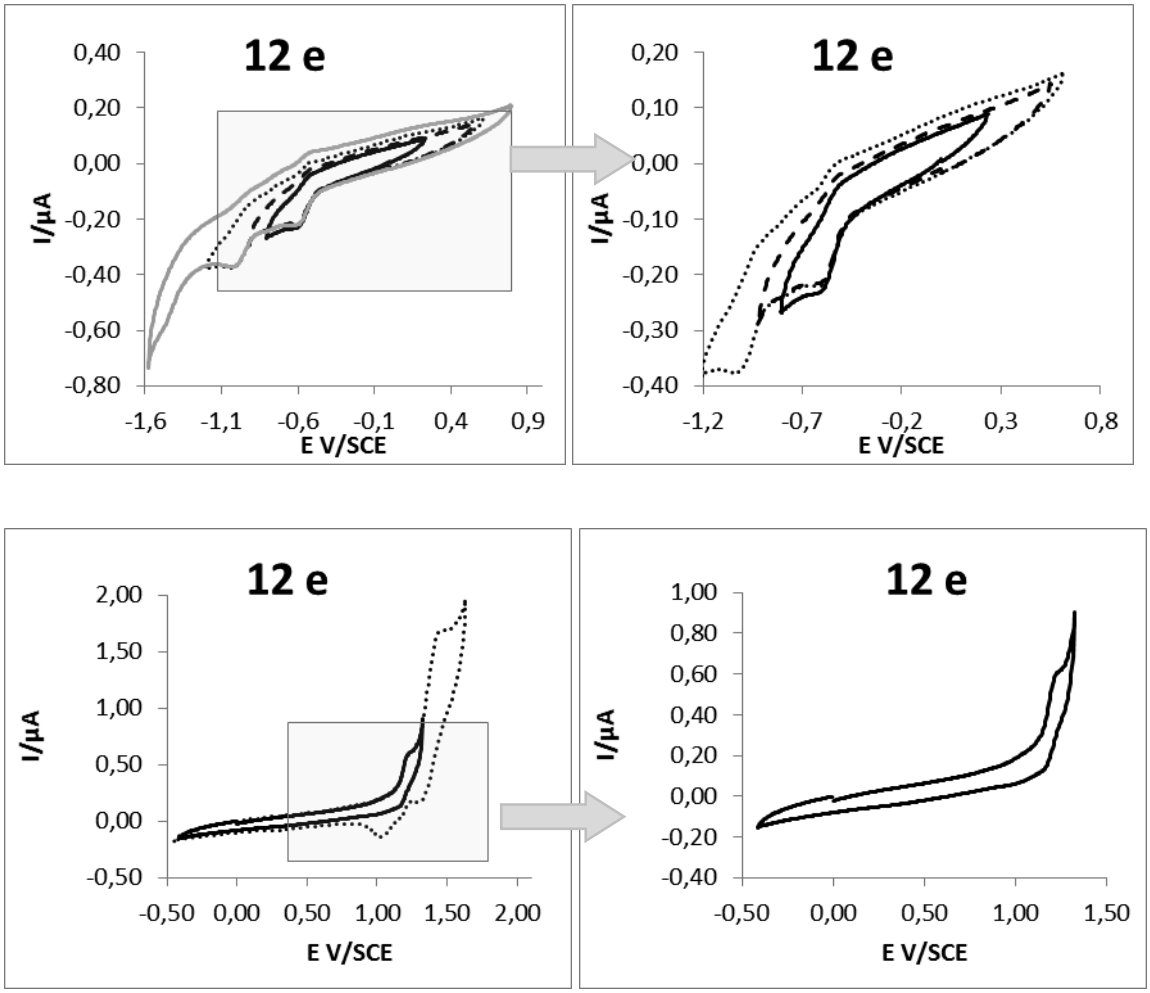
12e. **9-(4-{10-[4-(9*H*-carbazol-9-yl)phenyl]-13,16-dimethoxy-4,7-diphenyl-13,16-bis(trifluoromethyl)cyclooctadeca-1,2,3,7,8,9-hexaen-5,11,14,17-tetrayn-1-yl}phenyl)-9H-carbazole**

SW voltammograms for reduction and oxidation of **12e** in CH_2Cl_2 (containing 0.1 mol.L^{-1} $(nBu_4N)PF_6$) on Pt microdisk (r = 0.25 mm).



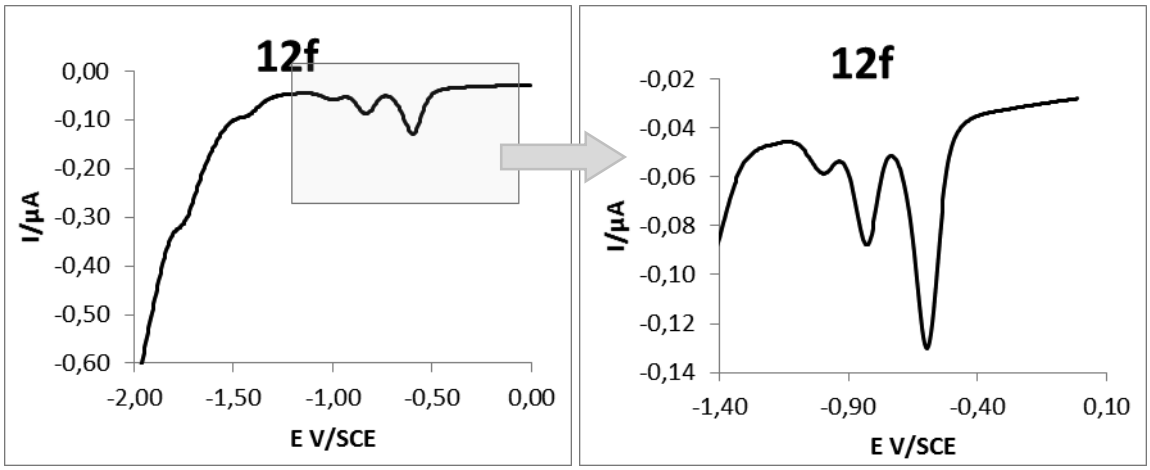


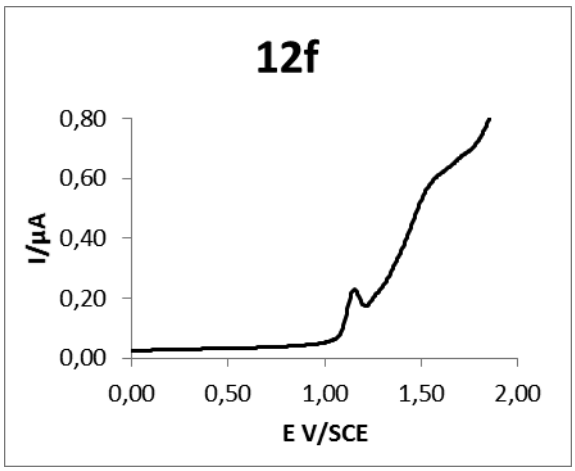
Cyclic voltammograms for reduction and oxidation of **12e** in CH_2Cl_2 (containing 0.1 mol.L^{-1} $(nBu_4N)PF_6$) on Pt microdisk (r = 0.25 mm), scan rate is 0.2 V/s.



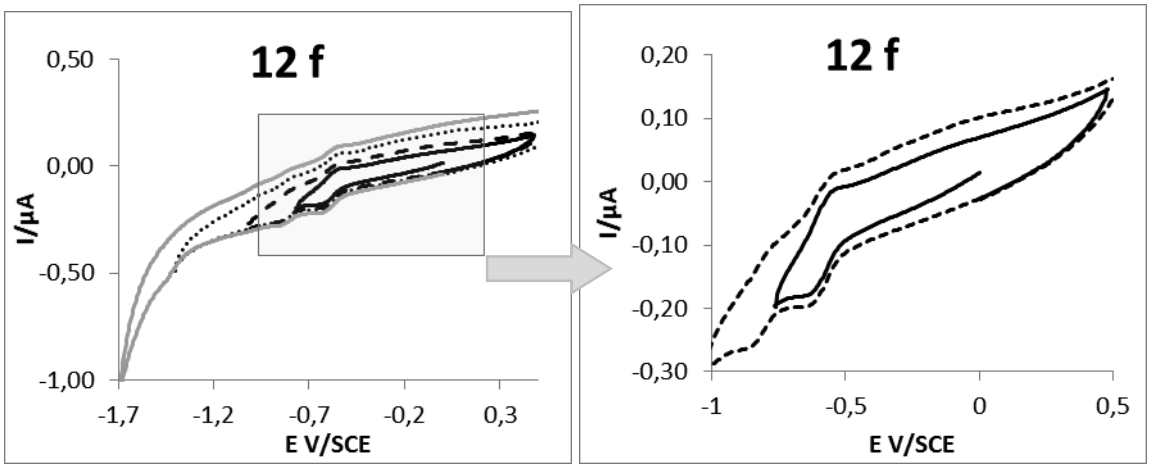
12f. **1-(4-{10-[4-(1H-indol-1-yl)phenyl]-13,16-dimethoxy-4,7-diphenyl-13,16-bis (trifluoromethyl)cyclooctadeca-1,2,3,7,8,9-hexaen-5,11,14,17-tetrayn-1-yl}phenyl)-1H-indole**

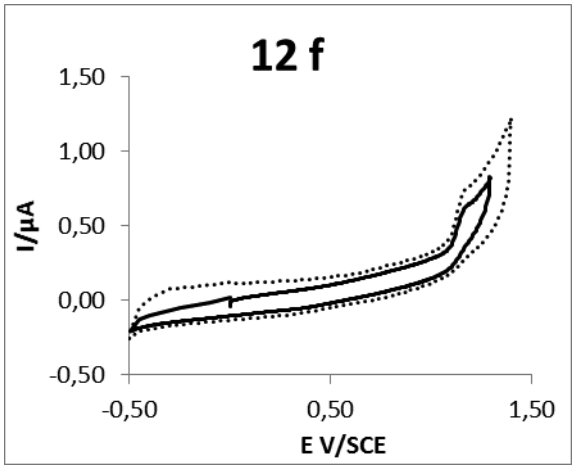
SW voltammograms for reduction and oxidation of **12f** in CH_2Cl_2 (containing 0.1 mol.L^{-1} (nBu_4N)PF_6) on Pt microdisk (r = 0.25 mm).



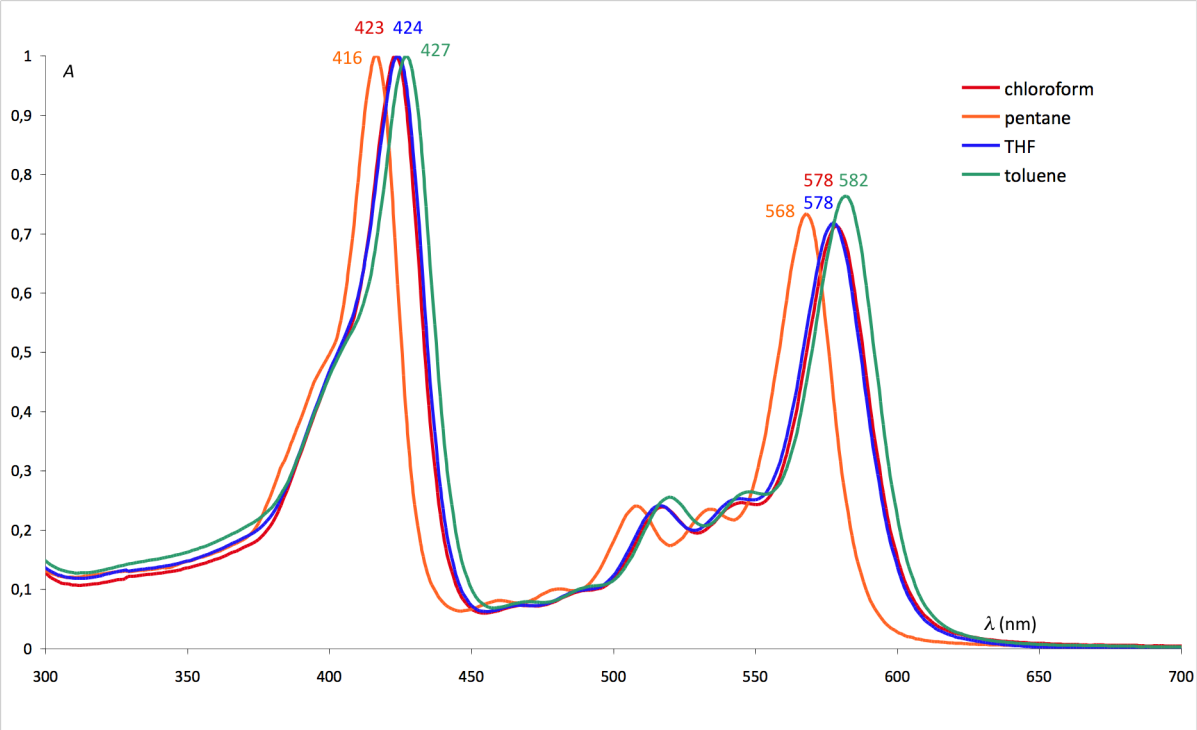


Cyclic voltammograms for reduction and oxidation of **12f** in CH_2Cl_2 (containing 0.1 mol.L^{-1} $(nBu_4N)PF_6$) on Pt microdisk (r = 0.25 mm), scan rate is 0.2 V/s





7. UV-vis absorption spectra and solvatochromism of 12d.



8. References.

1. V. Maraval, L. Leroyer, A. Harano, C. Barthes, A. Saquet, C. Duhayon, T. Shinmyuzo, R. Chauvin, *Chem. Eur. J. 2011*, **17**, 5086.

2. a) L. Maurette, C. Tedeschi, E. Sermot, M. Soleilhavoup, F. Hussain, B. Donnadieu, R. Chauvin, *Tetrahedron 2004*, **60**, 10077; b) L. Leroyer, C. Zou, V. Maraval, R. Chauvin, *CR Chimie 2009*, **12**, 412.

3. A. Rives, V. Maraval, N. Saffon-Merceron, R. Chauvin, *Chem.□Eur. J. 2014*, **20**, 483.

4. A. Rives, I. Baglai, V. Malytskiy, V. Maraval, N. Saffon-Merceron, Z. V. Voitenko, R. Chauvin, *Chem. Commun. 2012*, 8763.

5 . Palatinus L., Chapuis G. *J. Appl. Cryst. 2007*, **40**, 786.

6. Betteridge, P. W., Carruthers, J. R., Cooper, R. I., Prout, K.; Watkin, D. J. *J. Appl. Cryst. 2003*, **36**, 1487.

7. International Tables for X-ray Crystallography, Vol. IV, Kynoch Press, Birmingham,

England, *1974*.

8. R. H. Blessing, *Acta Cryst*. *1995*, **a51**, 33.

9. SAINT-NT Bruker AXS: Madison Wisconsin, *2000*.

10. SADABS (*2006* and *2008*), Program for data correction, Bruker-AXS.

11. SHELXL-97, Program for Crystal Structure Refinement, G. M. Sheldrick, University of Göttingen, *Acta Crystallogr., Sect. A 2008*, **64**, 112-122.