

## Use of Isomeric, Aromatic Dialdehydes in Synthesis of Photoactive, Positional Isomers of Higher Analogs of *o*-Bromo(hetero)acenaldehydes

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### Experimental Section

**General:** The  $^1\text{H}$  NMR (200 MHz) and  $^{13}\text{C}$  NMR (50 MHz) spectra were recorded using a Bruker AV 200 spectrometer. The mass spectra of pure compounds were obtained using a Finigan Mat 95 spectrometer. Melting points were determined using Boetius apparatus. UV-visible absorption spectra were obtained with a Specord S600 diode array spectrophotometer in 5-cm cuvettes. Room-temperature emission spectra were acquired with a Perkin Elmer LS50 luminescence spectrometer. Quantum yields were calculated using quinine sulfate dihydrate in 0.1 *N* HClO<sub>4</sub> as the standard reference material ( $\Phi=0.59$ ). Column chromatography was done using Merck silica gel (F<sub>254</sub> 60, 270-400 mesh). Organic solvents were purified by standard procedures. Aldehydes: 2,5-dibromobenzene-1,4-dicarbaldehyde and 2,4-dibromobenzene-1,5-dicarbaldehyde have been prepared according to the following procedure: M. C. Bonifacio, C. R. Robertson, J. Y. Jung, B. T. King, *J. Org. Chem.*, 2005, **70** 8522.

The stability of **6a** towards photooxidation was investigated by monitoring the absorbance as a function of time (0-100 minute range) decay of 10<sup>-6</sup> M chlorobenzene solution in a quartz cuvette at room temperature, under ambient atmosphere and exposed under 9W UV/Vis lamp with broad band light with maximum at 370 nm (Fig.1.).

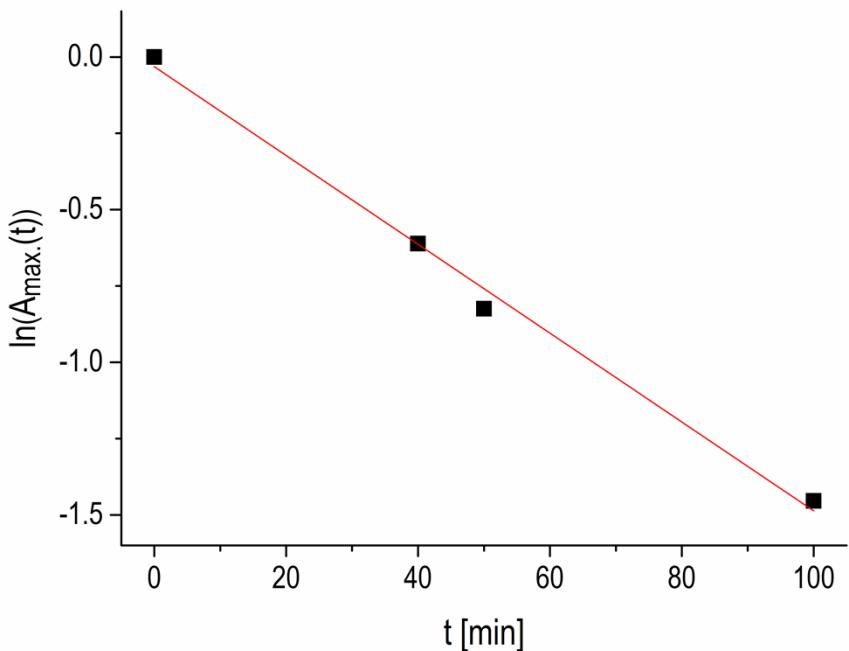
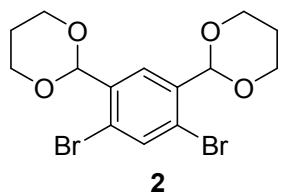
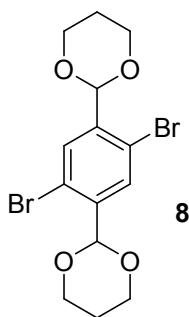


Fig. 1. Intensity decrease of the absorption band of 6a in chlorobenzene under UV/vis light exposure at room temperature under ambient atmosphere.

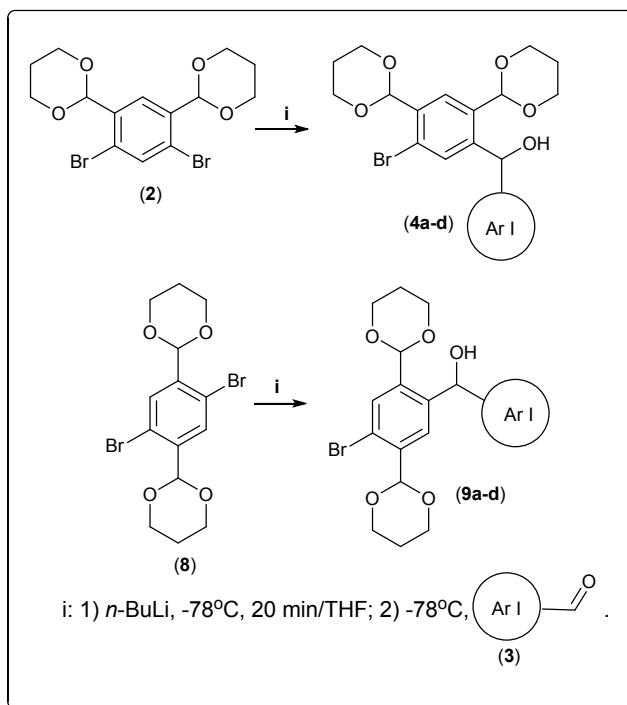
**General procedure for synthesis of protected dibromo dialdehydes 2 and 8:** To a solution of 2,5-dibromobenzene-1,4-dicarbaldehyde (**1**) or 2,4-dibromobenzene-1,5-dicarbaldehyde (**7**) (10 g, 34 mmol) in toluene (50 mL) were added *Amberlite IR 120* (as a catalyst) and 1,3-propandiol (4.9 mL, 68 mmol). The resulting mixture was refluxed for 6 h using a Dean-Stark trap for the azeotropic removal of water. After evaporation of the solvent, the residue was diluted with chloroform (100 mL), filtered off and organic layer was washed with water (50 mL), brine (50 mL) and again with water (3x50 mL). The organic layer was dried ( $\text{MgSO}_4$ ) and then evaporated under vacuum to give corresponding diacetals (**2**) or (**8**) as crystals. All new compounds prepared by the above procedure were characterized spectroscopically as shown below.



Yield: 58 %; white crystals, mp 166-167°C;  $^1\text{H}$  NMR (200MHz,  $\text{C}_6\text{D}_6$ , 25°C, TMS,  $\delta$ ): 0.57 (d,  $\text{H}_{\text{eq}}^{2,2'}$ , 2H), 1.65-1.82 (m,  $\text{H}_{\text{ax}}^{2,2'}$ , 2H), 3.37-3.49 (m,  $\text{H}_{\text{eq}}^{4,4',6,6'}$ , 4H), 3.75-3.83 (m,  $\text{H}_{\text{ax}}^{4,4',6,6'}$ , 4H), 5.61 (s, OCHO, 2H), 7.59 (s, Ar-H, 1H), 8.60 (s, Ar-H, 1H);  $^{13}\text{C}$  NMR (50MHz,  $\text{C}_6\text{D}_6$ , 25°C, TMS,  $\delta$ ): 26.31 (s,  $\text{OCH}_2\text{CH}_2\text{CH}_2\text{O}$ ), 67.88 (s,  $\text{OCH}_2\text{CH}_2\text{CH}_2\text{O}$ ), 101.22 (s, OCHO), 124.01 (s,  $\text{C}_{\text{Ar}}\text{-H}$ ), 129.57 (s,  $\text{C}_{\text{Ar}}\text{-H}$ ), 136.49 (s,  $\text{C}_{\text{Ar}}\text{-Br}$ ), 138.71 (s,  $\text{C}_{\text{Ar}}\text{-CHOC}_3\text{H}_6\text{O}$ ); MS (EI, 70eV): m/z (%): 408.9 (17.2) [M], 87.1 (100) [- $\text{C}_4\text{H}_7\text{O}_2$ ]; HR MS (EI, 70 eV): *m/z* calcd for  $\text{C}_{14}\text{H}_{16}\text{Br}_2\text{O}_4$ : 405.94155; found: 405.94202.

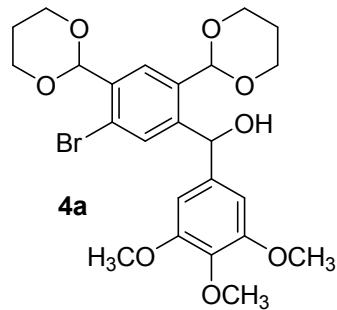


Yield: 78 %; white crystals, mp 230°C; <sup>1</sup>H NMR (200MHz, C<sub>6</sub>D<sub>6</sub>, 25 °C, TMS, δ): 0.57 (d, H<sub>eq</sub><sup>2,2'</sup>, 2H), 1.67-1.82 (m, H<sub>ax</sub><sup>2,2'</sup>, 2H), 3.35-3.47 (m, H<sub>eq</sub><sup>4,4',6,6'</sup>, 4H), 3.74-3.82 (m, H<sub>ax</sub><sup>4,4',6,6'</sup>, 4H), 5.56 (s, OCHO, 2H), 8.24 (s, Ar-H, 1H); <sup>13</sup>C NMR (50MHz, C<sub>6</sub>D<sub>6</sub>, 25 °C, TMS, δ): 26.33 (s, OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 67.88 (s, OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 100.91 (s, OCHO), 122.40 (s, C<sub>Ar</sub>-H), 133.62 (s, C<sub>Ar</sub>-Br), 140.95 (s, C<sub>Ar</sub>-CHOC<sub>3</sub>H<sub>6</sub>O); MS (EI, 70eV): m/z (%) 408.9 (100) [M], 87.1 (12) [-C<sub>4</sub>H<sub>7</sub>O<sub>2</sub>]; HR MS (EI, 70 eV): *m/z* calcd for C<sub>14</sub>H<sub>16</sub>Br<sub>2</sub>O<sub>4</sub>: 405.94155; found: 405.94488.

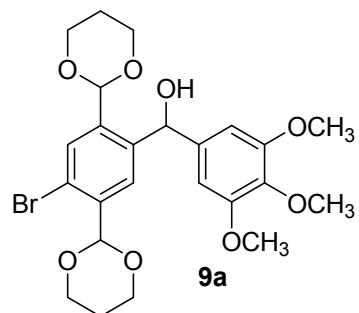


**General procedure for synthesis of diarylmethanols 4a-d and 9a-d:** The 1,5-dibromo-2,4-bis(1,3-dioxan-2-yl)-benzene (**2**) or 1,4-dibromo-2,5-bis(1,3-dioxan-2-yl)-benzene (**8**) (1 mmol) was dissolved in dry THF (50 mL), cooled to -78 °C and then *n*-BuLi in heptane (2.6 M, 1.2 mmol) was added. The resulting mixture was stirred for 40 min under argon atmosphere. The corresponding (hetero)aromatic aldehydes (**3**) (1.1 mmol) in dry THF (3 mL), was added at -78 °C and stirring was continued for 1.5 h from -78°C to room temperature. Then saturated aqueous solution of NH<sub>4</sub>Cl was added and the organic layer was evaporated. The residue was diluted with ethyl acetate (50 mL) and washed with water (3 x 20 mL). The organic layer was dried (MgSO<sub>4</sub>) and then filtrated. The solvent was removed in vacuum and the residue purified by column chromatography (mixtures *n*-hexane/acetone) to give the corresponding

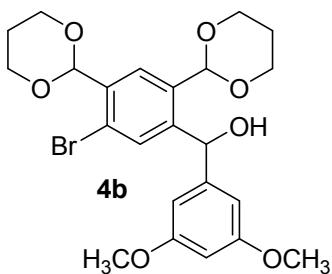
diarylmethanols (**4a-d**) and (**9a-d**) as solids. All new compounds prepared by the above procedure were characterized spectroscopically as shown below.



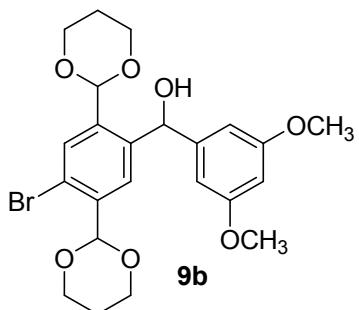
Yield: 31 %; yellow foam, mp 80-82°C; <sup>1</sup>H NMR (200MHz, C<sub>6</sub>D<sub>6</sub>, 25 °C, TMS, δ): 0.58 (d, H<sub>eq</sub><sup>5,5'</sup>, 2H), 1.65-1.83 (m, H<sub>ax</sub><sup>5,5'</sup>, 2H), 3.26-3.49 (m, H<sub>ax</sub><sup>4,4',6,6'</sup>, 4H), 3.38 (s, *m*-ArOCH<sub>3</sub>, 6H), 3.69-3.86 (m, H<sub>eq</sub><sup>4,4',6,6'</sup>, 4H), 3.85 (s, *p*-ArOCH<sub>3</sub>, 3H), 5.21 (s, OCHO, 1H), 5.52 (s, OCHO, 1H), 6.53 (s, CHOH, 1H), 6.97 (s, *o*-Ar(OCH<sub>3</sub>)<sub>3</sub>, 2H), 7.82 (s, Ar-H, 1H), 7.84 (s, Ar-H, 1H); <sup>13</sup>C NMR (50MHz, C<sub>6</sub>D<sub>6</sub>, 25 °C, TMS, δ): 26.30 (s, 2x OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 56.40 (s, 2x *m*-Ar(OCH<sub>3</sub>)<sub>2</sub>), 61.18 (s, *p*-ArOCH<sub>3</sub>), 67.98 (s, 2x OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 73.00 (s, CHOH), 101.95 (s, 2x OCHO), 105.16 (s, 2x *o*-C<sub>Ar</sub>-H), 126.40 (s, C<sub>Ar</sub>-H), 132.36 (s, C<sub>Ar</sub>-H), 137.65 (s, C<sub>Ar</sub>-CHOCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 138.12 (s, C<sub>Ar</sub>-CHOCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 138.96 (s, 2x C<sub>Ar</sub>-CHOH), 139.61 (s, *p*-C<sub>Ar</sub>-OCH<sub>3</sub>), 141.49 (s, C<sub>Ar</sub>-Br), 143.06 (s, *ipso*-C<sub>Ar</sub>Ar(OCH<sub>3</sub>)<sub>3</sub>), 154.65 (s, 2x *m*-C<sub>Ar</sub>-OCH<sub>3</sub>); MS (EI, 70eV): m/z (%) 524 (6.75) [M], 450 (57.49) [M, (-OC<sub>3</sub>H<sub>6</sub>O)], 370 (100) [M, (-HOC<sub>3</sub>H<sub>6</sub>OH, -OC<sub>3</sub>H<sub>6</sub>O)]; HR MS (EI, 70 eV): *m/z* calcd for C<sub>24</sub>H<sub>29</sub>BrO<sub>8</sub>: 524.10253 ; found: 524.10186.



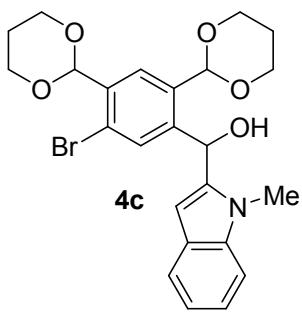
Yield: 60 %; yellow oil, <sup>1</sup>H NMR (200MHz, C<sub>6</sub>D<sub>6</sub>, 25 °C, TMS, δ): 0.55 (d, H<sub>eq</sub><sup>5,5'</sup>, 2H), 1.66-1.85 (m, H<sub>ax</sub><sup>5,5'</sup>, 2H), 3.24-3.66 (m, H<sub>ax</sub><sup>4,4',6,6'</sup>, 4H), 3.34 (s, *m*-ArOCH<sub>3</sub>, 6H), 3.72-3.88 (m, H<sub>eq</sub><sup>4,4',6,6'</sup>, 4H), 3.84 (s, *p*-ArOCH<sub>3</sub>, 3H), 5.39 (s, OCHO, 1H), 5.76 (s, OCHO, 1H), 6.35 (s, CHOH, 1H), 6.86 (s, *o*-Ar(OCH<sub>3</sub>)<sub>3</sub>, 2H), 7.79 (s, Ar-H, 1H), 8.51 (s, Ar-H, 1H); <sup>13</sup>C NMR (50MHz, C<sub>6</sub>D<sub>6</sub>, 25 °C, TMS, δ): 26.19 (s, OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 26.49 (s, OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 54.06 (s, 2x *m*-Ar(OCH<sub>3</sub>)<sub>2</sub>), 58.87 (s, *p*-ArOCH<sub>3</sub>), 67.96 (s, 2x OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 72.40 (s, CHOH), 101.69 (s, 2x OCHO), 105.25 (s, 2x *o*-C<sub>Ar</sub>-H), 124.46 (s, C<sub>Ar</sub>-H), 133.98 (s, C<sub>Ar</sub>-H), 136.86 (s, C<sub>Ar</sub>-CHOCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 138.94 (s, 2x C<sub>Ar</sub>-CHOH), 139.22 (s, *p*-C<sub>Ar</sub>-OCH<sub>3</sub>), 138.94 (s, C<sub>Ar</sub>-Br), 146.22 (s, *ipso*-C<sub>Ar</sub>Ar(OCH<sub>3</sub>)<sub>3</sub>), 154.85 (s, 2x *m*-C<sub>Ar</sub>-OCH<sub>3</sub>); MS (CI, isobutane): m/z (%) 525 (30) [M+1], 451 (100) [M+1 (-OC<sub>3</sub>H<sub>6</sub>O)]; HR MS (EI, 70 eV): *m/z* calcd for C<sub>24</sub>H<sub>29</sub>BrO<sub>8</sub>: 524.10253; found: 524.10296.



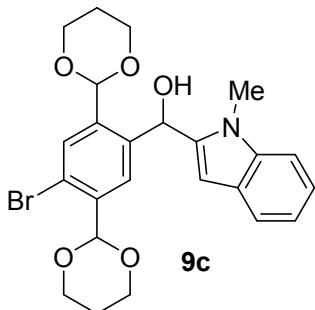
Yield: 76 %; yellow foam, mp 68-70 °C; <sup>1</sup>H NMR (200MHz, CDCl<sub>3</sub>, 25 °C, TMS, δ): 1.42 (d, H<sub>eq</sub><sup>5,5'</sup>, 2H), 2.10-2.23 (m, H<sub>ax</sub><sup>5,5'</sup>, 2H), 3.71 (s, OH, 1H), 3.73 (s, *m*-ArOCH<sub>3</sub>, 6H), 3.81-4.02 (m, H<sub>ax</sub><sup>4,4',6,6'</sup>, 4H), 4.18-4.23 (m, H<sub>eq</sub><sup>4,4',6,6'</sup>, 4H), 5.46 (s, OCHO, 1H), 5.68 (s, OCHO, 1H), 6.17 (s, CHOH, 1H), 6.35 (s, *p*-Ar-H, 1H), 6.51 (s, *o*-Ar-H, 2H), 7.33 (s, Ar-H, 1H), 7.83 (s, Ar-H, 1H); <sup>13</sup>C NMR (50MHz, CDCl<sub>3</sub>, 25 °C, TMS, δ): 25.58 (s, 2x OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 34.02 (s, 2x OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 55.56 [s, 2x *m*-Ar(OCH<sub>3</sub>)<sub>2</sub>], 62.07 (s, 2x OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 67.51 (s, 2x OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 84.44 (s, CHOH), 100.47 (s, OCHO), 100.54 (s, OCHO), 104.87 (s, C<sub>Ar</sub>-H), 106.04 (s, C<sub>Ar</sub>-H), 122.86 (s, C<sub>Ar</sub>-H), 126.34 (s, C<sub>Ar</sub>-H), 136.84 (s, C<sub>Ar</sub>), 137.18 (s, C<sub>Ar</sub>), 137.57 (s, C<sub>Ar</sub>), 142.03 (s, C<sub>Ar</sub>-H), 143.27 (s, C<sub>Ar</sub>), 144.88 (s, C<sub>Ar</sub>), 145.38 (s, C<sub>Ar</sub>); MS (EI, 70eV): m/z (%) 494.9 (10) [M]; 420.0 (100) [M, (-O(CH<sub>2</sub>)<sub>3</sub>O-)], 87.0 (31) [M, ((-CHO<sub>2</sub>(CH<sub>2</sub>)<sub>3</sub>)]; HR MS (EI, 70 eV): *m/z* calcd for C<sub>23</sub>H<sub>27</sub>BrO<sub>7</sub>: 494.09235; found: 494.09299.



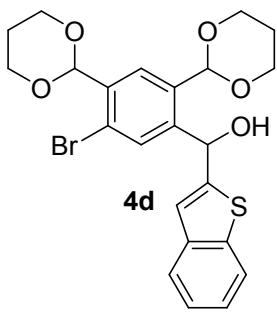
Yield: 63 %; yellow foam, mp 61-63 °C; <sup>1</sup>H NMR (200MHz, CDCl<sub>3</sub>, 25 °C, TMS, δ): 1.34 (d, H<sub>eq</sub><sup>5,5'</sup>, 2H), 2.01-2.13 (m, H<sub>ax</sub><sup>5,5'</sup>, 2H), 3.66 (s, *m*-ArOCH<sub>3</sub>, 6H), 3.80-4.00 (m, H<sub>ax</sub><sup>4,4',6,6'</sup>, 4H), 4.06-4.15 (m, H<sub>eq</sub><sup>4,4',6,6'</sup>, 4H), 5.31 (s, OCHO, 1H), 5.61 (s, OCHO, 1H), 9.92 (s, CHOH, 1H), 6.35 (s, *p*-Ar-H, 1H), 6.29 (s, *o*-Ar-H, 2H), 7.66 (s, Ar-H, 1H), 7.75 (s, Ar-H, 1H); <sup>13</sup>C NMR (50MHz, CDCl<sub>3</sub>, 25 °C, TMS, δ): 25.36 (s, 2x OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 25.54 (s, 2x OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 55.35 [s, 2x *m*-Ar(OCH<sub>3</sub>)<sub>2</sub>], 61.61 (s, 2x OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 67.31 (s, 2x OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 67.44 (s, 2x OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 72.91 (s, CHOH), 98.66 (s, OCHO), 99.32 (s, OCHO), 100.54 (s, C<sub>Ar</sub>-H), 104.38 (s, C<sub>Ar</sub>-H), 121.65 (s, C<sub>Ar</sub>-H), 128.97 (s, C<sub>Ar</sub>-H), 131.07 (s, C<sub>Ar</sub>), 137.79 (s, C<sub>Ar</sub>), 138.00 (s, C<sub>Ar</sub>), 140.93 (s, C<sub>Ar</sub>-H), 143.63 (s, C<sub>Ar</sub>), 145.17 (s, C<sub>Ar</sub>), 150.98 (s, C<sub>Ar</sub>); MS (EI, 70eV): m/z (%) 494,9 (2.76) [M]; 420,0 (43.60) [M, -(-O(CH<sub>2</sub>)<sub>3</sub>O-)], 87 (100) [M, ((-CHO<sub>2</sub>(CH<sub>2</sub>)<sub>3</sub>)]; HR MS (EI, 70 eV): *m/z* calcd for C<sub>23</sub>H<sub>27</sub>BrO<sub>7</sub>: 494.09235; found: 490.09260.



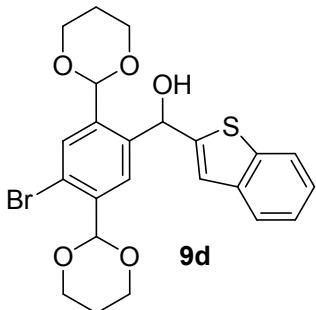
Yield: 71 %; yellow foam, mp 125 °C; <sup>1</sup>H NMR (200MHz, CDCl<sub>3</sub>, 25 °C, TMS, δ): 1.25-1.44 (m, H<sub>eq</sub><sup>5,5'</sup>, 2H), 2.14-2.27 (m, H<sub>ax</sub><sup>5,5'</sup>, 2H), 3.49 (s, CH<sub>3</sub>, 1H), 3.84-4.19 (m, H<sub>ax</sub><sup>4,4',6,6'</sup>, 4H), 4.22-4.24 (m, H<sub>eq</sub><sup>4,4',6,6'</sup>, 4H), 5.56 (s, OCHO, 1H), 5.75 (s, OCHO, 1H), 6.46 (s, CHOH, 1H), 6.52 (s, Ar-H, 1H), 7.22 (d, Ar-H, 1H), 7.29 (t, Ar-H, 2H), 7.46 (s, Ar-H, 1H), 7.61 (d, Ar-H, 1H), 7.92 (s, Ar-H, 1H); <sup>13</sup>C NMR (50MHz, C<sub>6</sub>D<sub>6</sub>, 25 °C, TMS, δ): 25.31 (s, OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 26.41 (s, OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 29.87 (s, CH<sub>3</sub>), 65.48 (s, CHOH), 67.11 (s, OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 67.15 (s, OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 67.30 (s, OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 100.33 (s, OCHO), 100.79 (s, OCHO), 101.24 (s, C<sub>Ar</sub>-H), 108.79 (s, C<sub>Ar</sub>-H), 119.13 (s, C<sub>Ar</sub>-H), 120.99 (s, C<sub>Ar</sub>-H), 121.21 (s, C<sub>Ar</sub>-H), 127.23 (s, C<sub>Ar</sub>-H), 131.91 (s, C<sub>Ar</sub>-H), 121.66 (s, C<sub>Ar</sub>), 135.08 (s, C<sub>Ar</sub>), 136.92 (s, C<sub>Ar</sub>), 137.75 (s, C<sub>Ar</sub>), 139.88 (s, C<sub>Ar</sub>), 142.38 (s, C<sub>Ar</sub>), 155.86 (s, C<sub>Ar</sub>); MS (EI, 70eV): m/z (%) 489 (24) [M+1], 412 (20) [M+1 (HOC<sub>3</sub>H<sub>6</sub>OH)]; HR MS (EI, 70 eV): m/z calcd for C<sub>24</sub>H<sub>26</sub>BrNO<sub>5</sub>: 487.10253; found: 487.09829.



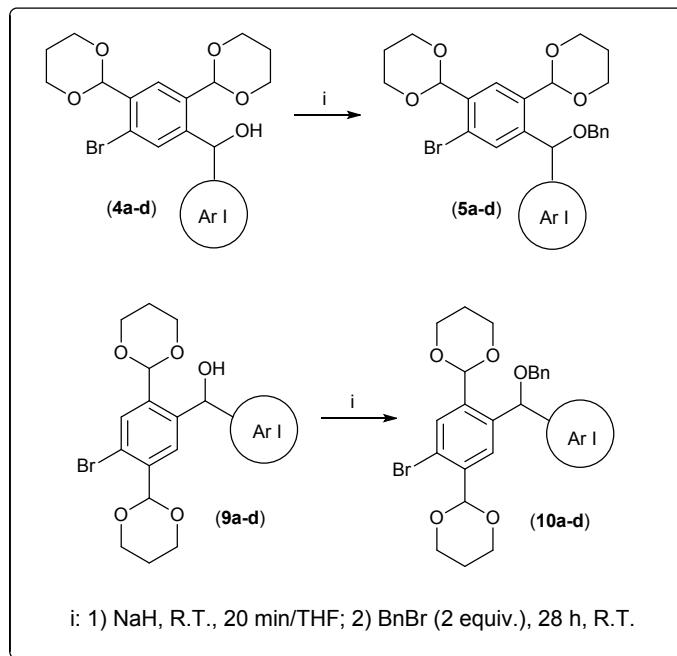
Yield: 60 %; yellow oil, <sup>1</sup>H NMR (200MHz, C<sub>6</sub>D<sub>6</sub>, 25 °C, TMS, δ): 1.24-1.44 (m, H<sub>eq</sub><sup>5,5'</sup>, 2H), 2.16-2.29 (m, H<sub>ax</sub><sup>5,5'</sup>, 2H), 3.49 (s, CH<sub>3</sub>, 1H), 3.85-4.20 (m, H<sub>ax</sub><sup>4,4',6,6'</sup>, 4H), 4.20-4.23 (m, H<sub>eq</sub><sup>4,4',6,6'</sup>, 4H), 5.57 (s, OCHO, 1H), 5.70 (s, OCHO, 1H), 6.47 (s, CHOH, 1H), 6.55 (s, Ar-H, 1H), 7.26 (d, Ar-H, 1H), 7.28 (t, Ar-H, 2H), 7.48 (s, Ar-H, 1H), 7.92 (d, Ar-H, 1H), 7.97 (s, Ar-H, 1H); <sup>13</sup>C NMR (50MHz, C<sub>6</sub>D<sub>6</sub>, 25 °C, TMS, δ): 25.33 (s, OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 26.43 (s, OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 29.88 (s, CH<sub>3</sub>), 65.40 (s, CHOH), 67.10 (s, OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 67.22 (s, OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 67.29 (s, OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 100.35 (s, OCHO), 100.80 (s, OCHO), 101.26 (s, C<sub>Ar</sub>-H), 108.81 (s, C<sub>Ar</sub>-H), 119.10 (s, C<sub>Ar</sub>-H), 120.99 (s, C<sub>Ar</sub>-H), 122.20 (s, C<sub>Ar</sub>-H), 127.20 (s, C<sub>Ar</sub>-H), 131.90 (s, C<sub>Ar</sub>-H), 121.60 (s, C<sub>Ar</sub>), 134.04 (s, C<sub>Ar</sub>), 137.92 (s, C<sub>Ar</sub>), 135.75 (s, C<sub>Ar</sub>), 138.81 (s, C<sub>Ar</sub>-Br), 143.37 (s, C<sub>Ar</sub>), 156.86 (s, C<sub>Ar</sub>); MS (EI, 70 eV): m/z (%) 489 (30) [M+1], 412 (15) [M+1 (HOC<sub>3</sub>H<sub>6</sub>OH)]; HR MS (EI, 70 eV): m/z calcd for C<sub>24</sub>H<sub>26</sub>BrNO<sub>5</sub>: 487.46780.10253; found: 487.100085.



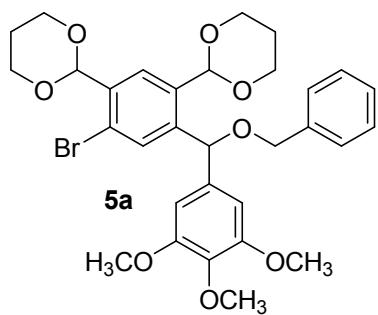
Yield: 63 %; white solid, mp 195-197 °C; <sup>1</sup>H NMR (200MHz, CDCl<sub>3</sub>, 25 °C, TMS, δ): 1.37-1.49 (m, H<sub>eq</sub><sup>5,5'</sup>, 2H), 2.13-2.37 (m, H<sub>ax</sub><sup>5,5'</sup>, 2H), 3.81-4.01 (m, H<sub>ax</sub><sup>4,4',6,6'</sup>, 4H), 4.21-4.32 (m, H<sub>eq</sub><sup>4,4',6,6'</sup>, 4H), 5.55 (s, OCHO, 1H), 5.79 (s, OCHO, 1H), 6.51 (s, CHOH, 1H), 7.08 (s, Ar-H, 1H), 7.28-7.39 (m, Ar-H, 2H), 7.65 (s, Ar-H, 1H), 7.68-7.73 (m, Ar-H, 1H), 7.81-7.86 (m, Ar-H, 1H), 7.93 (s, Ar-H, 1H); <sup>13</sup>C NMR (50MHz, CDCl<sub>3</sub>, 25 °C, TMS, δ): 25.36 (s, OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 25.59 (s, OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 67.49 (s, 2x OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 69.37 (s, CHOH), 100.49 (s, OCHO), 100.72 (s, OCHO), 121.06 (s, C<sub>Ar</sub>-H), 122.27 (s, C<sub>Ar</sub>-H), 123.15 (s, C<sub>Ar</sub>-H), 123.46 (s, C<sub>Ar</sub>-H), 123.92 (s, C<sub>Ar</sub>-H), 124.14 (s, C<sub>Ar</sub>-H), 127.28 (s, C<sub>Ar</sub>-H), 132.82 (s, C<sub>Ar</sub>), 135.24 (s, C<sub>Ar</sub>), 137.23 (s, C<sub>Ar</sub>), 139.64 (s, C<sub>Ar</sub>), 139.68 (s, C<sub>Ar</sub>), 143.17 (s, C<sub>Ar</sub>), 147.31 (s, C<sub>Ar</sub>); MS (EI, 70eV): m/z (%) 490 (15) [M]; 416 (100) [M, (-OC<sub>3</sub>H<sub>6</sub>O)], 414 (88) [M, (-HOC<sub>3</sub>H<sub>6</sub>OH)]; HR MS (EI, 70 eV): *m/z* calcd for C<sub>23</sub>H<sub>23</sub>BrO<sub>5</sub>S: 490.04318, found: 490.04278.



Yield: 62 %; colorless oil, <sup>1</sup>H NMR (200MHz, CDCl<sub>3</sub>, 25 °C, TMS, δ): 1.39-1.50 (m, H<sub>eq</sub><sup>5,5'</sup>, 2H), 2.12-2.35 (m, H<sub>ax</sub><sup>5,5'</sup>, 2H), 3.80-4.00 (m, H<sub>ax</sub><sup>4,4',6,6'</sup>, 4H), 4.20-4.30 (m, H<sub>eq</sub><sup>4,4',6,6'</sup>, 4H), 5.58 (s, OCHO, 1H), 5.70 (s, OCHO, 1H), 6.53 (s, CHOH, 1H), 7.01 (s, Ar-H, 1H), 7.29-7.49 (m, Ar-H, 2H), 7.60 (s, Ar-H, 1H), 7.68-7.77 (m, Ar-H, 1H), 7.87-7.89 (m, Ar-H, 1H), 7.92 (s, Ar-H, 1H); <sup>13</sup>C NMR (50MHz, CDCl<sub>3</sub>, 25 °C, TMS, δ): 24.37 (s, OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 26.50 (s, OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 66.50 (s, 2x OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 69.40 (s, CHOH), 100.50 (s, OCHO), 100.79 (s, OCHO), 121.16 (s, C<sub>Ar</sub>-H), 122.17 (s, C<sub>Ar</sub>-H), 123.25 (s, C<sub>Ar</sub>-H), 123.46 (s, C<sub>Ar</sub>-H), 123.99 (s, C<sub>Ar</sub>-H), 124.14 (s, C<sub>Ar</sub>-H), 128.29 (s, C<sub>Ar</sub>-H), 132.91 (s, C<sub>Ar</sub>), 135.34 (s, C<sub>Ar</sub>), 137.42 (s, C<sub>Ar</sub>), 139.68 (s, C<sub>Ar</sub>), 140.08 (s, C<sub>Ar</sub>), 144.07 (s, C<sub>Ar</sub>), 146.33 (s, C<sub>Ar</sub>); MS (EI, 70eV): m/z (%) 490 (20) [M]; 416 (100) [M, (-OC<sub>3</sub>H<sub>6</sub>O)], 414 (60) [M, (-HOC<sub>3</sub>H<sub>6</sub>OH)]; HR MS (EI, 70 eV): *m/z* calcd for C<sub>23</sub>H<sub>23</sub>BrO<sub>5</sub>S: 490.05318; found: 490.04393.

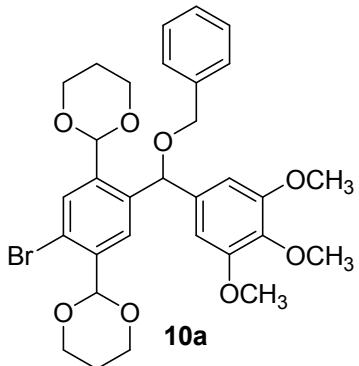


**General procedure for synthesis of *O*-benzyl protected diarylmethanols **5a-d** and **10a-d**:** To a suspension of NaH (1.3 mmol, 60% in mineral oil) in dry THF (3 mL), a solution of the corresponding diarylomethanol (**4a-d**) or (**9a-d**) (1 mmol) in THF (5 mL) was added at room temperature. The resulting mixture was stirred for 30 min then benzyl bromide (1.5 mmol) was added. Stirring was continued for 28 h at the same temperature. After evaporation of the solvent, the residue was diluted with ethyl acetate and washed with water (3x20 mL). The organic layer was dried ( $\text{MgSO}_4$ ) and then filtered. The solvent was removed to give the crude product which was purified by column chromatography (hexane/acetone = 2:1). All new compounds (**5a-d**) and (**10a-d**) prepared by the above procedure were characterized spectroscopically as shown below.

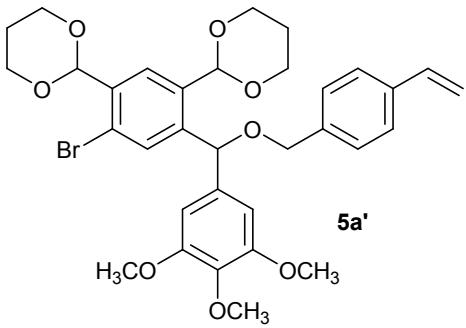


Yield: 30 %; yellow oil,  $^1\text{H}$  NMR (200MHz,  $\text{C}_6\text{D}_6$ , 25 °C, TMS,  $\delta$ ): 0.59 (d,  $\text{H}_{\text{eq}}^{5,5'}$ , 2H), 1.67-1.80 (m,  $\text{H}_{\text{ax}}^{5,5'}$ , 2H), 3.23-3.50 (m,  $\text{H}_{\text{ax}}^{4,4',6,6'}$ , 4H), 3.39 (s, *m*-ArOCH<sub>3</sub>, 6H), 3.64-3.86 (m,  $\text{H}_{\text{eq}}^{4,4',6,6'}$ , 4H), 3.81 (s, *p*-ArOCH<sub>3</sub>, 3H), 4.47 (d, CH<sub>A</sub>H<sub>B</sub>Ph, 1H<sub>A</sub>,  $^2\text{J}_{\text{HH}} = 0.06$  Hz), 4.57 (d, CH<sub>A</sub>H<sub>B</sub>Ph, 1H<sub>B</sub>,  $^2\text{J}_{\text{HH}} = 0.06$  Hz), 5.50 (s, OCHO, 1H), 5.74 (s, OCHO, 1H), 6.03 (s, CHOBn, 1H), 6.90 (s, *o*-Ar(OCH<sub>3</sub>)<sub>3</sub>, 2H), 7.33-7.40 (m, Ph, 5H), 8.30 (s, Ar-H, 1H), 8.43 (s, Ar-H, 1H);  $^{13}\text{C}$  NMR (50MHz,  $\text{C}_6\text{D}_6$ , 25 °C, TMS,  $\delta$ ): 26.35 (s, 2x OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 56.41 (s, 2x *m*-Ar(OCH<sub>3</sub>)<sub>2</sub>), 61.10 (s, *p*-ArOCH<sub>3</sub>), 67.83 (s, 2x OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 71.47 (s, OCH<sub>2</sub>Ph), 78.93 (s, CHOBn), 99.81 (s, OCHO), 101.68 (s, OCHO), 106.18 (s, 2x *o*-C<sub>Ar</sub>-H), 122.88 (s,

$C_{Ar}-H$ ), 127.42 (s, *m*-Ph), 129.18 (s, *p*-Ph), 129.82 (s, *o*-Ph), 132.51 (s,  $C_{Ar}-H$ ), 138.24 (s, 2x  $C_{Ar}-CHOCH_2CH_2CH_2O$ ), 139.36 (s,  $C_{Ar}-CHOBn$ ), 139.93 (s, *p*- $C_{Ar}-OCH_3$ ), 140.15 (s,  $C_{Ar}-Br$ ), 140.42 (s, *ipso*- $C_{Ar}Ar(OCH_3)_3$ ), 154.73 (s, 2x *m*- $C_{Ar}-OCH_3$ ); MS (EI, 70eV): m/z (%) 614 (9.88) [M], 525 (100) [M, (-OC<sub>3</sub>H<sub>6</sub>O, -CH<sub>3</sub>)], 91 (78.78) [M, (PhCH<sub>2</sub>O)]; HR MS (EI, 70 eV): *m/z* calcd for C<sub>31</sub>H<sub>35</sub>BrO<sub>8</sub>: 614.15074; found: 614.15014.

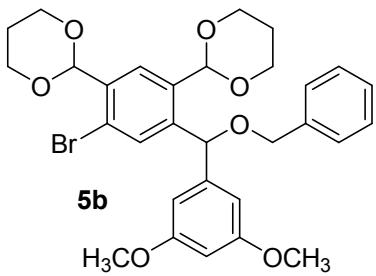


Yield: 30 %; yellow oil, <sup>1</sup>H NMR (200MHz, C<sub>6</sub>D<sub>6</sub>, 25°C, TMS,  $\delta$ ): 0.55 (d, H<sub>eq</sub><sup>5,5'</sup>, 2H), 1.52-1.85 (m, H<sub>ax</sub><sup>5,5'</sup>, 2H), 3.27-3.63 (m, H<sub>ax</sub><sup>4,4',6,6'</sup>, 4H), 3.37 (s, *m*-ArOCH<sub>3</sub>, 6H), 3.69-3.92 (m, H<sub>eq</sub><sup>4,4',6,6'</sup>, 4H), 3.83 (s, *p*-ArOCH<sub>3</sub>, 3H), 4.47 (d, CH<sub>A</sub>H<sub>B</sub>Ph, 1H<sub>A</sub>, <sup>2</sup>J<sub>HH</sub> = 0.06 Hz), 4.54 (d, CH<sub>A</sub>H<sub>B</sub>Ph, 1H<sub>B</sub>, <sup>2</sup>J<sub>HH</sub> = 0.06 Hz), 5.49 (s, OCHO, 1H), 5.78 (s, OCHO, 1H), 6.12 (s, CHOBn, 1H), 6.91 (s, *o*-Ar(OCH<sub>3</sub>)<sub>3</sub>, 2H), 6.98-7.02 (m, Ph, 5H), 7.12 (s, Ar-H, 1H), 7.30 (s, Ar-H, 1H); <sup>13</sup>C NMR (50MHz, C<sub>6</sub>D<sub>6</sub>, 25°C, TMS,  $\delta$ ): 26.35 (s, OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 26.35 (s, OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 56.42 (s, 2x *m*-Ar(OCH<sub>3</sub>)<sub>2</sub>), 61.12 (s, *p*-ArOCH<sub>3</sub>), 67.84 (s, 2x OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 71.44 (s, OCH<sub>2</sub>Ph), 78.06 (s, CHOBn), 100.88 (s, OCHO), 101.72 (s, OCHO), 105.88 (s, 2x *o*-C<sub>Ar</sub>-H), 124.45 (s, C<sub>Ar</sub>-H), 127.86 (s, *m*-Ph), 128.32 (s, *p*-Ph), 129.89 (s, *o*-Ph), 133.38 (s, C<sub>Ar</sub>-H), 137.74 (s, 2x C<sub>Ar</sub>-CHOCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 138.21 (s, C<sub>Ar</sub>-CHOBn), 138.67 (s, *p*-C<sub>Ar</sub>-OCH<sub>3</sub>), 139.86 (s, C<sub>Ar</sub>-Br), 143.58 (s, *ipso*-C<sub>Ar</sub>Ar(OCH<sub>3</sub>)<sub>3</sub>), 154.84 (s, 2x *m*-C<sub>Ar</sub>-OCH<sub>3</sub>); MS (EI, 70eV): m/z (%) 614 (5.89) [M], 449 (73.93) [M, (-OC<sub>3</sub>H<sub>6</sub>O, PhCH<sub>2</sub>O)], 91 (100) [M, (PhCH<sub>2</sub>O)]; HR MS (EI, 70 eV): *m/z* calcd for C<sub>31</sub>H<sub>35</sub>BrO<sub>8</sub>: 614.15074; found: 614.15001.

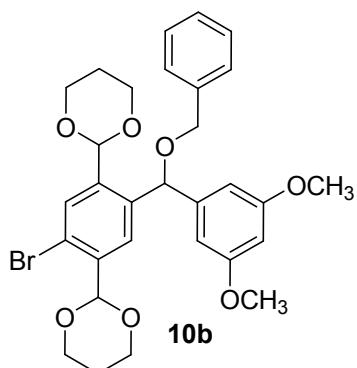


Yield: 96 %; yellow foam, mp 48-50 °C; <sup>1</sup>H NMR (200MHz, CDCl<sub>3</sub>, 25 °C, TMS,  $\delta$ ): 0.83 (d, H<sub>eq</sub><sup>5,5'</sup>, 2H), 1.71-2.50 (m, H<sub>ax</sub><sup>5,5'</sup>, 2H), 3.75 (s, *m*-ArOCH<sub>3</sub>, 6H), 3.80 (s, *p*-ArOCH<sub>3</sub>, 3H), 3.91-4.14 (m, H<sub>ax</sub><sup>4,4',6,6'</sup>, 4H), 4.49-3.52 (m, H<sub>eq</sub><sup>4,4',6,6'</sup>, 4H), 4.25-4.72 (dd, CH<sub>A</sub>H<sub>B</sub>Ph, 2H<sub>A</sub>, <sup>2</sup>J<sub>HH</sub>=0.06 Hz), 5.17 (d-CH=CH<sub>2</sub>, 1H,), 5.40 (s, OCHO, 1H), 5.70 (d, -CH=CH<sub>2</sub>, 1H); 5.94 (s, OCHO, 1H), 6.20 (s, CHOPh, 1H), 6.53-6.67 (q, -

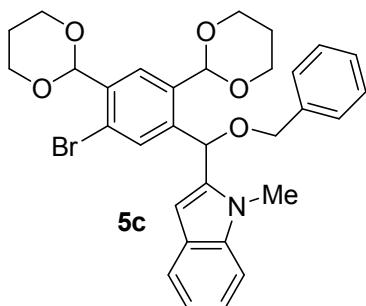
CH=CH<sub>2</sub>, 1H); 6.68 [s, *o*-Ar(OCH<sub>3</sub>)<sub>3</sub>, 2H], 7.29-7.34 (dd, Ph, 4H), 7.45 (s, Ar-H, 1H), 7.90 (s, Ar-H, 1H); <sup>13</sup>C NMR (50MHz, CDCl<sub>3</sub>, 25 °C, TMS, δ): 25.49 (s, 2x OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 25.67 (s, 2x OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 55.86 (s, 2x *m*-Ar(OCH<sub>3</sub>)<sub>2</sub>), 60.65 (s, *p*-ArOCH<sub>3</sub>), 67.11 (s, 2x OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 67.17 (s, 2x OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 70.12 (s, OCH<sub>2</sub>Ph), 75.93 (s, CHOBn), 99.76 (s, OCHO), 99.96 (s, OCHO), 103.53 (s, 2x *o*-C<sub>Ar</sub>-H), 113.69 (s, -CH=CH<sub>2</sub>), 122.96 (s, C<sub>Ar</sub>-H), 126.03 (s, *m*-Ph), 126.37 (s, C<sub>Ar</sub>-H), 126.68 (s, *o*-Ph), 132.23 (s, C<sub>Ar</sub>-H), 135.90 (s, C<sub>Ar</sub>-CHOBn), 136.45 (s, *p*-C<sub>Ar</sub>-OCH<sub>3</sub>), 136.53 (s, 2x C<sub>Ar</sub>-CHOCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 136.72 (CHOCH<sub>2</sub>-C<sub>Ar</sub>), 136.82 (s, 2x C<sub>Ar</sub>-CHOCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 136.82 (s, CH=CH<sub>2</sub>), 137.08 (s, C-CH=CH<sub>2</sub>), 137.84 (s, C<sub>Ar</sub>-Br), 142.08 (s, *ipso*-C<sub>Ar</sub>Ar(OCH<sub>3</sub>)<sub>3</sub>), 154.90 (s, 2x *m*-C<sub>Ar</sub>-OCH<sub>3</sub>); MS (EI, 70eV): m/z (%) 642.2 (2) [M]; 445.2 (100) [M, (-CH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>CH=CH<sub>2</sub>, -Br)], 117.2 (70) [CH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>CH=CH<sub>2</sub>]; HR MS (EI, 70 eV): *m/z* calcd for C<sub>33</sub>H<sub>37</sub>BrO<sub>8</sub>: 640.16698; found: 640.16714.



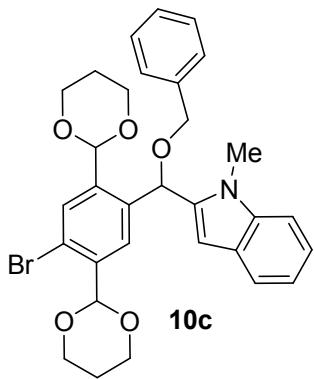
Yield: 46 %; yellow foam, mp 60-62 °C; <sup>1</sup>H NMR (200MHz, CDCl<sub>3</sub>, 25 °C, TMS, δ): 1.26-1.49 (d, H<sub>eq</sub><sup>5,5'</sup>, 2H), 2.03-2.26 (m, H<sub>ax</sub><sup>5,5'</sup>, 2H), 3.71 (s, 2 x *m*-ArOCH<sub>3</sub>, 6H), 3.93-4.05 (m, H<sub>ax</sub><sup>4,4',6,6'</sup>, 4H), 4.09-4.22 (m, H<sub>eq</sub><sup>4,4',6,6'</sup>, 4H), 4.49 (d, CH<sub>A</sub>H<sub>B</sub>Ph, 1H<sub>A</sub>, <sup>2</sup>J<sub>HH</sub> = 0,06 Hz), 4.51 (d, CH<sub>A</sub>H<sub>B</sub>Ph, 1H<sub>B</sub>, <sup>2</sup>J<sub>HH</sub> = 0,06 Hz), 5,39 (s, OCHO, 1H), 5.76 (s, OCHO, 1H), 5.90 (s, CHOBn, 1H), 6.32 (s, *o*-Ar(OCH<sub>3</sub>)<sub>3</sub>, 1H), 6.55 [s, *o*-Ar(OCH<sub>3</sub>)<sub>3</sub>, 2H], 7.30-7.33 (m, Ph, 5H), 7.64 (s, Ar-H, 1H), 7.86 (s, Ar-H, 1H); <sup>13</sup>C NMR (50MHz, CDCl<sub>3</sub>, 25 °C, TMS, δ): 25.63 (s, 2x OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 55.26 [s, 2x *m*-Ar(OCH<sub>3</sub>)<sub>2</sub>], 67.17 (s, 2x OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 67.24 (s, 2x OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 67.51 (s, 2x OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 70.43 (s, OCH<sub>2</sub>Ph), 76.16 (s, CHOBn), 99.16 (s, OCHO), 99.90 (s, OCHO), 100.64 (s, C<sub>Ar</sub>-H), 105.09 (s, C<sub>Ar</sub>-H), 123.05 (s, C<sub>Ar</sub>), 126.56 (s, C<sub>Ar</sub>-H), 126.90 (s, C<sub>Ar</sub>), 127.46 (s, C<sub>Ar</sub>-H), 127.83 (s, C<sub>Ar</sub>-H), 128.28 (s, C<sub>Ar</sub>-H), 132.31 (s, C<sub>Ar</sub>-H), 132.31 (s, C<sub>Ar</sub>), 136.02 (s, C<sub>Ar</sub>), 136.41 (s, C<sub>Ar</sub>), 142.12 (s, C<sub>Ar</sub>), 143.90 (s, C<sub>Ar</sub>); MS (EI, 70eV): m/z (%) 585,1 (1) [M]; 487,1 (56) [M, (-OCH<sub>2</sub>Ph)], 419 (100) [M, (-OCH<sub>2</sub>Ph, 2 x -OCH<sub>3</sub>)], 91 (86) [CH<sub>2</sub>Ph]; HR MS (EI, 70 eV): *m/z* calcd for C<sub>30</sub>H<sub>33</sub>BrO<sub>7</sub>: 585.13980; found: 585.13987.



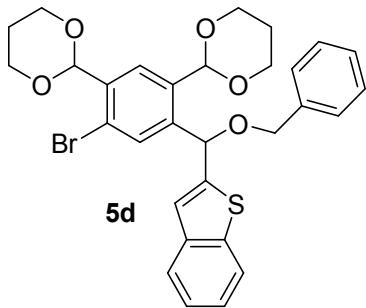
Yield: 69 %; yellow oil,  $^1\text{H}$  NMR (200MHz,  $\text{CDCl}_3$ , 25 °C, TMS,  $\delta$ ): 1.24-1.42 (d,  $\text{H}_{\text{eq}}^{5,5'}$ , 2H), 1.96-2.29 (m,  $\text{H}_{\text{ax}}^{5,5'}$ , 2H), 3.71 (s, 2 x *m*-ArOCH<sub>3</sub>, 6H), 3.90-4.26 (m,  $\text{H}_{\text{ax}}^{4,4',6,6'}$ , 8H), 4.41 (d,  $\text{CH}_A\text{H}_B\text{Ph}$ , 1H<sub>A</sub>,  $^2\text{J}_{\text{HH}}$  = 0,06 Hz), 4.54 (d,  $\text{CH}_A\text{H}_B\text{Ph}$ , 1H<sub>B</sub>,  $^2\text{J}_{\text{HH}}$  = 0,06 Hz), 5.42 (s, OCHO, 1H), 5.67 (s, OCHO, 1H), 5.74 (s, CHOBn, 1H), 6.32 (s, *o*-Ar(OCH<sub>3</sub>)<sub>3</sub>, 1H), 6.52 (s, *o*-Ar(OCH<sub>3</sub>)<sub>3</sub>, 2H), 7.30-7.33 (m, Ph, 5H), 7.81 (s, Ar-H, 1H), 7.83 (s, Ar-H, 1H);  $^{13}\text{C}$  NMR (50MHz,  $\text{CDCl}_3$ , 25 °C, TMS,  $\delta$ ): 24.33 (s, 2x OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 54.00 [s, 2x *m*-Ar(OCH<sub>3</sub>)<sub>2</sub>], 65.97 (s, 2x OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 66.01 (s, 2x OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 66.18 (s, 2x OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 66.21 (s, 2x OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 69.29 (s, OCH<sub>2</sub>Ph), 75.17 (s, CHOBn), 97.20 (s, OCHO), 99.34 (s, OCHO), 104.13 (s, C<sub>Ar</sub>-H), 120.45 (s, C<sub>Ar</sub>-H), 126.24 (s, C<sub>Ar</sub>-H), 126.81 (s, C<sub>Ar</sub>-H), 126.93 (s, C<sub>Ar</sub>), 127.01 (s, C<sub>Ar</sub>-H), 129.55 (s, C<sub>Ar</sub>-H), 136.83 (s, C<sub>Ar</sub>), 137.02 (s, C<sub>Ar</sub>), 137.16 (s, C<sub>Ar</sub>), 137.37 (s, C<sub>Ar</sub>), 142.50 (s, C<sub>Ar</sub>), 159.34 (s, C<sub>Ar</sub>); MS (EI, 70eV): m/z (%) 585,1 (1) [M]; 487,1 (56) [M, (-OCH<sub>2</sub>Ph)], 419 (100) [M, (-OCH<sub>2</sub>Ph, 2 x -OCH<sub>3</sub>)], 91 (86) [CH<sub>2</sub>Ph]; HR MS (EI, 70 eV): *m/z* calcd for C<sub>30</sub>H<sub>33</sub>BrO<sub>7</sub>: 585.13987; found: 585.13985.



Yield: 78 %; yellow oil,  $^1\text{H}$  NMR (200MHz,  $\text{CDCl}_3$ , 25 °C, TMS,  $\delta$ ): 1.21-1.40 (m,  $\text{H}_{\text{eq}}^{5,5'}$ , 2H), 1.90-2.40 (m,  $\text{H}_{\text{ax}}^{5,5'}$ , 2H), 3.45 (s, CH<sub>3</sub>, 1H), 3.65-3.80 (m,  $\text{H}_{\text{ax}}^{4,4',6,6'}$ , 2H), 3.97-4.22 (m,  $\text{H}_{\text{eq}}^{4,4',6,6'}$ , 4H), 4.25-4.53 (m,  $\text{H}_{\text{ax}}^{4,4',6,6'}$ , 2H), 4.55-4.75 (d,  $\text{CH}_A\text{H}_B\text{Ph}$ , 1H<sub>A</sub>,  $^2\text{J}_{\text{HH}}$ =0.06 Hz), 4.61-4.69 (d,  $\text{CH}_A\text{H}_B\text{Ph}$ , 1H<sub>B</sub>,  $^2\text{J}_{\text{HH}}$ =0.06 Hz), 5.54 (s, OCHO, 1H), 5.84 (s, OCHO, 1H), 6.33 (s, CHOBn, 1H), 7.02 (s, Ar-H, 1H), 7.29-7.43 (m, Ph, Ar-H, 7H), 7.70 (t, Ar-H, 1H), 7.80 (t, Ar-H, 1H), 7.97 (s, Ar-H, 1H), 8.02 (s, Ar-H, 1H);  $^{13}\text{C}$  NMR (50MHz,  $\text{CDCl}_3$ , 25 °C, TMS,  $\delta$ ): 26.03 (s, OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 27.13 (s, OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 30.08 (s, CH<sub>3</sub>), 66.04 (s, CHOBn), 67.12 (s, OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 67.36 (s, OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 67.41 (s, OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 72.41 (s, OCH<sub>2</sub>Ph), 100.36 (s, OCHO), 100.81 (s, OCHO), 102.29 (s, C<sub>Ar</sub>-H), 109.82 (s, C<sub>Ar</sub>-H), 120.15 (s, C<sub>Ar</sub>-H), 121.93 (s, C<sub>Ar</sub>-H), 122.83 (s, C<sub>Ar</sub>-H), 127.50 (s, C<sub>Ar</sub>-H), 127.95 (s, *m*-Ph), 128.47 (s, *p*-Ph), 129.90 (s, *o*-Ph), 131.69 (s, C<sub>Ar</sub>), 131.94 (s, C<sub>Ar</sub>-H), 135.37 (s, C<sub>Ar</sub>), 136.65 (s, C<sub>Ar</sub>), 137.23 (s, C<sub>Ar</sub>), 138.19 (s, C<sub>Ar</sub>), 138.29 (s, C<sub>Ar</sub>), 142.27 (s, C<sub>Ar</sub>), 157.06 (s, C<sub>Ar</sub>); MS (EI, 70eV): m/z (%) 578 (7) [M+1], 413 (70) [M, (-OC<sub>3</sub>H<sub>6</sub>O, PhCH<sub>2</sub>O)], 91 (100) [M, (PhCH<sub>2</sub>)].

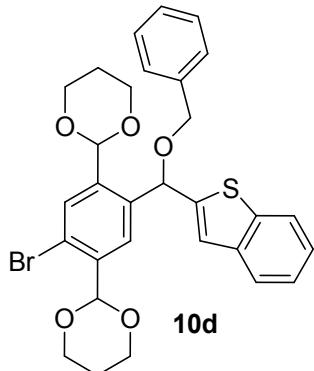


Yield: 30 %; yellow oil,  $^1\text{H}$  NMR (200MHz,  $\text{CDCl}_3$ , 25 °C, TMS,  $\delta$ ): 1.25-1.43 (m,  $\text{H}_{\text{eq}}^{5,5'}$ , 2H), 1.91-2.44 (m,  $\text{H}_{\text{ax}}^{5,5'}$ , 2H), 3.45 (s,  $\text{CH}_3$ , 1H), 3.64-3.83 (m,  $\text{H}_{\text{ax}}^{4,4',6,6'}$ , 2H), 3.99-4.23 (m,  $\text{H}_{\text{eq}}^{4,4',6,6'}$ , 4H), 4.26-4.51 (m,  $\text{H}_{\text{ax}}^{4, 4', 6, 6'}$ , 2H), 4.54-4.76 (d,  $\text{CH}_\text{A}\text{H}_\text{B}\text{Ph}$ , 1 $\text{H}_\text{A}$ ,  $^2\text{J}_{\text{HH}}=0.06$  Hz), 4.62-4.70 (d,  $\text{CH}_\text{A}\text{H}_\text{B}\text{Ph}$ , 1 $\text{H}_\text{B}$ ,  $^2\text{J}_{\text{HH}}=0.06$  Hz), 5.55 (s, OCHO, 1H), 5.83 (s, OCHO, 1H), 6.35 (s, CHOBn, 1H), 7.00 (s, Ar-H, 1H), 7.30-7.40 (m, Ph, Ar-H, 7H), 7.71 (t, Ar-H, 1H), 7.81 (t, Ar-H, 1H), 7.99 (s, Ar-H, 1H), 8.01 (s, Ar-H, 1H);  $^{13}\text{C}$  NMR (50MHz,  $\text{CDCl}_3$ , 25 °C, TMS,  $\delta$ ): 26.83 (s,  $\text{OCH}_2\text{CH}_2\text{CH}_2\text{O}$ ), 27.16 (s,  $\text{OCH}_2\text{CH}_2\text{CH}_2\text{O}$ ), 30.08 (s,  $\text{CH}_3$ ), 66.00 (s, CHOBn), 67.02 (s,  $\text{OCH}_2\text{CH}_2\text{CH}_2\text{O}$ ), 67.34 (s,  $\text{OCH}_2\text{CH}_2\text{CH}_2\text{O}$ ), 67.51 (s,  $\text{OCH}_2\text{CH}_2\text{CH}_2\text{O}$ ), 72.40 (s,  $\text{OCH}_2\text{Ph}$ ), 100.25 (s, OCHO), 100.65 (s, OCHO), 101.13 (s,  $\text{C}_{\text{Ar}}\text{-H}$ ), 106.54 (s,  $\text{C}_{\text{Ar}}\text{-H}$ ), 118.19 (s,  $\text{C}_{\text{Ar}}\text{-H}$ ), 121.34 (s,  $\text{C}_{\text{Ar}}\text{-H}$ ), 123.24 (s,  $\text{C}_{\text{Ar}}\text{-H}$ ), 126.21 (s,  $\text{C}_{\text{Ar}}\text{-H}$ ), 128.16 (s, *m*-Ph), 128.42 (s, *p*-Ph), 130.90 (s, *o*-Ph), 132.91 (s,  $\text{C}_{\text{Ar}}\text{-H}$ ), 134.60 (s,  $\text{C}_{\text{Ar}}$ ), 134.71 (s,  $\text{C}_{\text{Ar}}$ ), 135.22 (s,  $\text{C}_{\text{Ar}}$ ), 135.75 (s,  $\text{C}_{\text{Ar}}$ ), 138.91 (s,  $\text{C}_{\text{Ar}}$ ), 139.09 (s,  $\text{C}_{\text{Ar}}$ ), 142.69 (s,  $\text{C}_{\text{Ar}}$ ), 157.16 (s,  $\text{C}_{\text{Ar}}$ ); MS (EI, 70eV): m/z (%) 578 (10) [ $\text{M}+1$ ], 413 (70) [M, (-OC<sub>3</sub>H<sub>6</sub>O, PhCH<sub>2</sub>O)], 61 (100) [M, (PhCH<sub>2</sub>)].

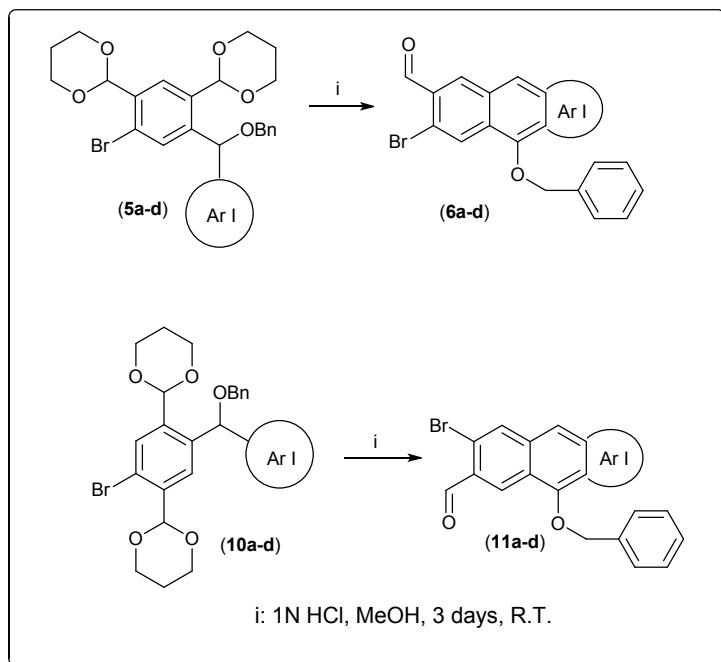


Yield: 40 %; yellow foam, mp 62-64 °C;  $^1\text{H}$  NMR (200MHz,  $\text{CDCl}_3$ , 25 °C, TMS,  $\delta$ ): 1.25-1.47 (m,  $\text{H}_{\text{eq}}^{5,5'}$ , 2H), 1.96-2.39 (m,  $\text{H}_{\text{ax}}^{5,5'}$ , 2H), 3.68-3.81 (m,  $\text{H}_{\text{ax}}^{4,4',6,6'}$ , 2H), 3.98-4.20 (m,  $\text{H}_{\text{eq}}^{4,4',6,6'}$ , 4H), 4.26-4.52 (m,  $\text{H}_{\text{ax}}^{4,4',6,6'}$ , 2H), 4.56-4.76 (d,  $\text{CH}_\text{A}\text{H}_\text{B}\text{Ph}$ , 1 $\text{H}_\text{A}$ ,  $^2\text{J}_{\text{HH}}=0.05$  Hz), 4.62-4.70 (d,  $\text{CH}_\text{A}\text{H}_\text{B}\text{Ph}$ , 1 $\text{H}_\text{B}$ ,  $^2\text{J}_{\text{HH}}=0.05$  Hz), 5.52 (s, OCHO, 1H), 5.81 (s, OCHO, 1H), 6.36 (s, CHOBn, 1H), 7.01 (s, Ar-H, 1H), 7.30-7.44 (m, Ph, Ar-H, 7H), 7.69 (t, Ar-H, 1H), 7.82 (t, Ar-H, 1H), 7.98 (s, Ar-H, 1H), 8.00 (s, Ar-H, 1H);  $^{13}\text{C}$  NMR (50MHz,  $\text{CDCl}_3$ , 25 °C, TMS,  $\delta$ ): 25.28 (s,  $\text{OCH}_2\text{CH}_2\text{CH}_2\text{O}$ ), 25.47 (s,  $\text{OCH}_2\text{CH}_2\text{CH}_2\text{O}$ ), 67.00 (s,  $\text{OCH}_2\text{CH}_2\text{CH}_2\text{O}$ ), 67.05 (s,  $\text{OCH}_2\text{CH}_2\text{CH}_2\text{O}$ ), 67.32 (s,  $\text{OCH}_2\text{CH}_2\text{CH}_2\text{O}$ ), 70.43 (s,  $\text{OCH}_2\text{Ph}$ ), 73.40 (s, CHOBn), 100.07 (s, OCHO), 100.40 (s, OCHO), 121.75 (s,  $\text{C}_{\text{Ar}}\text{-H}$ ), 122.01 (s,  $\text{C}_{\text{Ar}}\text{-H}$ ), 123.04 (s,  $\text{C}_{\text{Ar}}\text{-H}$ ), 123.39 (s,  $\text{C}_{\text{Ar}}\text{-H}$ ), 123.87 (s,  $\text{C}_{\text{Ar}}\text{-H}$ ), 126.76 (s,  $\text{C}_{\text{Ar}}\text{-H}$ ), 127.45 (s, *m*-Ph), 127.61 (s, *p*-Ph), 128.16 (s,

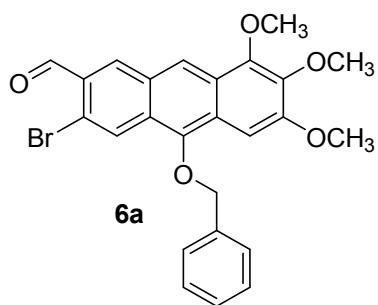
*o*-Ph), 128.57 (s, C<sub>Ar</sub>-H), 132.06 (s, C<sub>Ar</sub>), 136.00 (s, C<sub>Ar</sub>), 137.04(s, C<sub>Ar</sub>), 137.91 (s, C<sub>Ar</sub>), 139.32 (s, C<sub>Ar</sub>), 139.70 (s, C<sub>Ar</sub>), 141.23 (s, C<sub>Ar</sub>), 146.51(s, C<sub>Ar</sub>); MS (EI, 70eV): m/z (%) 614 (8) [M+2], 431 (100) [M, (-OC<sub>3</sub>H<sub>6</sub>O-)], 91 (76) [(PhCH<sub>2</sub>O)]; HR MS (EI, 70 eV): *m/z* calcd for C<sub>30</sub>H<sub>29</sub>BrO<sub>5</sub>S: 580.09244, found: 580.09218.



Yield: 38 %; yellow oil, <sup>1</sup>H NMR (200MHz, CDCl<sub>3</sub>, 25 °C, TMS, δ): 1.22-1.52 (m, H<sub>eq</sub><sup>5,5'</sup>, 2H), 1.93-2.39 (m, H<sub>ax</sub><sup>5,5'</sup>, 2H), 3.67-3.86 (m, H<sub>ax</sub><sup>4,4',6,6'</sup>, 2H), 3.99-4.22 (m, H<sub>eq</sub><sup>4,4',6,6'</sup>, 4H), 4.27-4.52 (m, H<sub>ax</sub><sup>4,4',6,6'</sup>, 2H), 4.55-4.75 (d, CH<sub>A</sub>H<sub>B</sub>Ph, 1H<sub>A</sub>, <sup>2</sup>J<sub>HH</sub>=0.05 Hz), 4.64-4.72 (d, CH<sub>A</sub>H<sub>B</sub>Ph, 1H<sub>B</sub>, <sup>2</sup>J<sub>HH</sub>= 0.05 Hz), 5.53 (s, OCHO, 1H), 5.86 (s, OCHO, 1H), 6.37 (s, CHOBn, 1H), 7.09 (s, Ar-H, 1H), 7.31-7.45 (m, Ph, Ar-H, 7H), 7.70 (t, Ar-H, 1H), 7.84 (t, Ar-H, 1H), 7.99 (s, Ar-H, 1H), 8.02 (s, Ar-H, 1H); <sup>13</sup>C NMR (50MHz, CDCl<sub>3</sub>, 25 °C, TMS, δ): 25.31 (s, OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 25.51 (s, OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 67.07 (s, OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 67.09 (s, OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 67.41 (s, OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O), 70.53 (s, OCH<sub>2</sub>Ph), 73.40 (s, CHOBn), 100.11(s, OCHO), 100.45 (s, OCHO), 121.78 (s, C<sub>Ar</sub>-H), 122.58 (s, C<sub>Ar</sub>-H), 123.34 (s, C<sub>Ar</sub>-H), 123.43 (s, C<sub>Ar</sub>-H), 123.99 (s, C<sub>Ar</sub>-H), 126.70 (s, C<sub>Ar</sub>-H), 127.60 (s, *m*-Ph), 127.69 (s, *p*-Ph), 128.21(s, *o*-Ph), 128.69 (s, C<sub>Ar</sub>-H), 132.13 (s, C<sub>Ar</sub>), 136.59 (s, C<sub>Ar</sub>), 136.89 (s, C<sub>Ar</sub>), 137.97 (s, C<sub>Ar</sub>), 139.34 (s, C<sub>Ar</sub>), 139.79 (s, C<sub>Ar</sub>), 142.22 (s, C<sub>Ar</sub>), 146.58(s, C<sub>Ar</sub>); MS (EI, 70eV): m/z (%) 614 (5) [M+2], 431 (100) [M, (-OC<sub>3</sub>H<sub>6</sub>O-)], 91 (66) [(PhCH<sub>2</sub>O)]; HR MS (EI, 70 eV): *m/z* calcd for C<sub>30</sub>H<sub>29</sub>BrO<sub>5</sub>S: 580.09244; found: 580.09249.

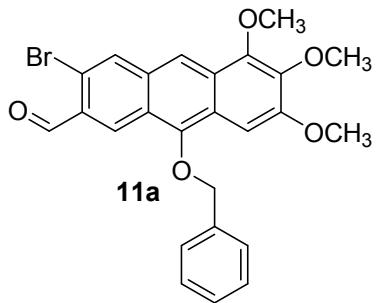


**General procedure for synthesis of *o*-bromoacenaldehydes **6a-d** and **11a-d**:** The *O*-protected diarylmethanol (**5a-d**) and (**10a-d**) (0.244 g, 0563 mmol) was dissolved in methanol (30 mL) and then an aqueous solution of 1 *N* HCl (17 mL) was added. The mixture was stirred at ambient temperature for 3 days. The reaction mixture was extracted with ethyl acetate (50 mL) and the organic layer was washed with water (20 mL), NaHCO<sub>3</sub> aq. (20 mL) and again with water (20 mL) and then dried (MgSO<sub>4</sub>). The solvent was removed and the products (**6a-d**, **11a-d**) were purified with column chromatography (petroleum ether/acetone 10:1). All new compounds prepared by the above procedure were characterized spectroscopically as shown below.

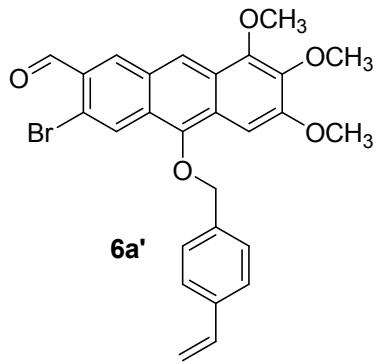


Yield: 99 %; orange solid, mp 155 °C; <sup>1</sup>H NMR (200MHz, C<sub>6</sub>D<sub>6</sub>, 25 °C, TMS, δ): 3.31 (s, OCH<sub>3</sub>, 3H); 3.80 (s, OCH<sub>3</sub>, 3H); 3.89 (s, OCH<sub>3</sub>, 3H), 4.93 (s, OCH<sub>2</sub>Ph, 2H); 7.06-7.30 (m, C<sub>6</sub>H<sub>5</sub>, 5H); 7.91 (s, Ar, 1H); 8.32 (s, Ar, 1H); 9.09 (s, Ar, 1H) 10.50 (s, CHO, 1H); <sup>13</sup>C NMR (50MHz, C<sub>6</sub>D<sub>6</sub>, 25 °C, TMS, δ): 55.86 (s, OCH<sub>3</sub>), 61.58 (s, OCH<sub>3</sub>), 61.81 (s, OCH<sub>3</sub>); 79.10 (s, OCH<sub>2</sub>Ph), 97.18 (s, C<sub>Ar</sub>H); 107.47 (s, C<sub>Ar</sub>H); 116.55 (s, C<sub>Ar</sub>H); 119.25 (s, C<sub>Ar</sub>H), 123.70 (s, C<sub>Ar</sub>-OMe), 124.51 (s, C<sub>Ar</sub>-OMe ), 125.83 (s, C<sub>Ar</sub>-OMe), 128.21 (s, 2x *o*-Ph), 129.57 (s, *p*-Ph), 129.96 (s, 2x *m*-Ph), 130.76 (s, *ipso*-C<sub>6</sub>H<sub>5</sub>), 133.71 (s), 134.02 (s), 135.32 (s), 139.06 (s), 143.67 (s), 153.88 (s), 155.09 (s, C-OCH<sub>2</sub>Ph), 191.32 (s, CHO), MS (EI,

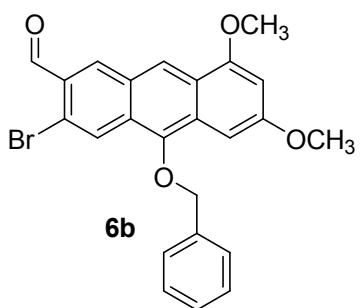
70eV): m/z (%) 480 (46) (M), 390 (100) (M(-Ph)); HR MS (EI, 70 eV): *m/z* calcd for C<sub>25</sub>H<sub>21</sub>BrO<sub>5</sub> calculated: 480.05636; found: 480.05838.



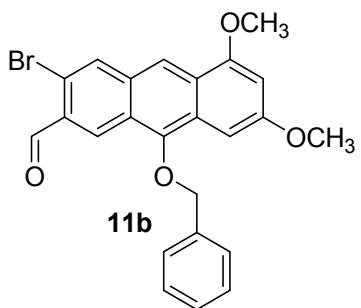
Yield: 79 %; orange solid mp 165 °C; <sup>1</sup>H NMR (200MHz, C<sub>6</sub>D<sub>6</sub>, 25 °C, TMS, δ): 3.31 (s, OCH<sub>3</sub>, 3H), 3.77 (s, OCH<sub>3</sub>, 3H), 3.88 (s, OCH<sub>3</sub>, 3H), 4.85 (s, OCH<sub>2</sub>Ph, 2H), 7.06-7.34 (m, C<sub>6</sub>H<sub>5</sub>, 5H), 8.50 (s, Ar, 1H), 8.55 (s, Ar, 1H), 8.59 (s, Ar), 10.50 (s, CHO, 1H); <sup>13</sup>C NMR (50MHz, C<sub>6</sub>D<sub>6</sub>, 25 °C, TMS, δ): 55.81 (s, OCH<sub>3</sub>), 61.55 (s, OCH<sub>3</sub>), 61.88 (s, OCH<sub>3</sub>), 78.10 (s, OCH<sub>2</sub>Ph), 96.76 (s, C<sub>Ar</sub>-H), 105.63 (s, C<sub>Ar</sub>-H), 120.12 (s, C<sub>Ar</sub>-H), 121.24 (s, C<sub>Ar</sub>-H), 125.15 (s, C<sub>Ar</sub>-OMe), 125.63 (s, C<sub>Ar</sub>-OMe), 125.93 (s, C<sub>Ar</sub>-OMe), 127.62 (s, *p*-Ph), 127.75 (s, 2x *o*-Ph), 129.57 (s, 2x *m*-Ph), 130.61 (s, *ipso*-Ph), 124.71 (s), 138.44 (s), 142.53 (s), 149.02 (s), 149.53 (s), 155.15 (s), 156.28 (s, C-OCH<sub>2</sub>Ph), 191.22 (s, CHO), MS (EI, 70eV): m/z (%) 480 (2.58) [M]; 391 (100) [M(-Ph)]; HR MS (EI, 70 eV): *m/z* calcd for C<sub>25</sub>H<sub>21</sub>BrO<sub>5</sub>: 480.05707; found: 480.05792.



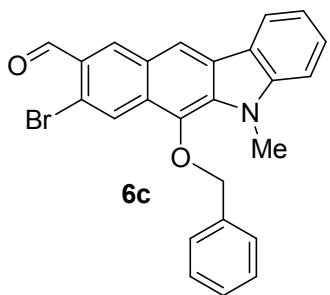
Yield: 99 %; orange solid, mp 142-145 °C; <sup>1</sup>H NMR (200MHz, CDCl<sub>3</sub>, 25 °C, TMS, δ): 3.86 (s, OCH<sub>3</sub>, 3H), 3.99 (s, OCH<sub>3</sub>, 3H), 4.11 (s, OCH<sub>3</sub>, 3H), 5.10 (s, OCH<sub>2</sub>Ph, 2H), 5.30 (d, -CH=CH<sub>2</sub>, 1H), 5.77 (d, -CH=CH<sub>2</sub>, 1H), 6.66-6.68 (q, -CH=CH<sub>2</sub>, 1H), 7.09 (s, Ar, 1H), 7.45 (s, Ar, 4H), 8.31 (s, Ar, 1H), 8.47 (s, Ar, 1H), 8.51 (s, Ar, 1H), 10.39 (s, CHO, 1H); <sup>13</sup>C NMR (50MHz, CDCl<sub>3</sub>, 25 °C, TMS, δ): 55.57 (s, OCH<sub>3</sub>), 61.27 (s, OCH<sub>3</sub>), 61.53 (s, OCH<sub>3</sub>), 95.55 (s, OCH<sub>2</sub>Ph), 114.58 (s, -CH=CH<sub>2</sub>), 118.88 (s, C<sub>Ar</sub>H), 120.17 (s, C<sub>Ar</sub>H), 125.29 (s, C<sub>Ar</sub>H), 126.29 (s, C ), 126.58 (s, C<sub>Ar</sub>H), 126.65 (s, 2x *o*-Ph); 128.18 (s, C); 128.43 (s, 2x *m*-Ph); 129.17 (s, C<sub>Ar</sub>-OMe); 134.28 (s, C<sub>Ar</sub>-OMe); 136.26 (s, C<sub>Ar</sub>-OMe), 136.45 (s, -CH=CH<sub>2</sub>), 137.74 (s, C-CH=CH<sub>2</sub>), 137.92 (s, *ipso*-Ph), 141.22 (s, C), 145.59 (s, C), 147.56 (s, C-CHO), 148.59 (s, C-Br), 154.95 (s, C-OCH<sub>2</sub>Ph), 191.91 (s, CHO); MS (EI, 70eV): m/z (%) 508.0 (1.88) [M], 391.0 (100) [M, (-CH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>CH=CH<sub>2</sub>)]; HR MS (EI, 70 eV): *m/z* calcd for C<sub>27</sub>H<sub>23</sub>BrO<sub>5</sub>: 506.07167; found: 506.07350.



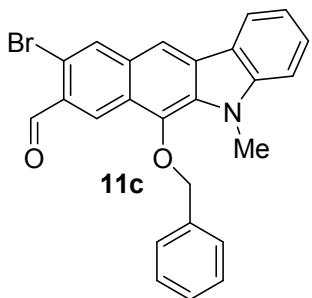
Yield: 89 %; orange solid, mp 130-132 °C;  $^1\text{H}$  NMR (200MHz,  $\text{CDCl}_3$ , 25 °C, TMS,  $\delta$ ): 3.82 (s, OCH<sub>3</sub>, 3H), 4.01 (s, OCH<sub>3</sub>, 3H), 5.16 (s, OCH<sub>2</sub>Ph, 2H), 6.44 (s, Ar, 1H), 6.93 (s, Ar, 1H), 7.30-7.41 (m, C<sub>6</sub>H<sub>5</sub>, 5H), 8.62 (s, Ar, 1H), 8.57 (s, Ar, 1H), 8.37 (s, Ar, 1H), 10.44 (s, CHO, 1H);  $^{13}\text{C}$  NMR (50MHz,  $\text{CDCl}_3$ , 25 °C, TMS,  $\delta$ ): 53.81 (s, OCH<sub>3</sub>), 55.23 (s, OCH<sub>3</sub>), 90.99 (s, OCH<sub>2</sub>Ph), 97.87 (s, C<sub>Ar</sub>-H), 98.33 (s, C<sub>Ar</sub>-H), 102.77 (s, C<sub>Ar</sub>-H), 118.33 (s, C<sub>Ar</sub>-H), 119.56 (s, C<sub>Ar</sub>-H), 123.89 (s, C<sub>Ar</sub>), 125.84 (s, C<sub>Ar</sub>), 127.57 (s, C<sub>Ar</sub>-H), 128.14 (s, C<sub>Ar</sub>-H), 128.33 (s, C<sub>Ar</sub>-H), 131.58 (s, C<sub>Ar</sub>), 134.57 (s, C<sub>Ar</sub>), 137.07 (s, C<sub>Ar</sub>), 148.08 (s, C<sub>Ar</sub>), 156.99 (s, C<sub>Ar</sub>), 157.26 (s, C<sub>Ar</sub>), 158.41 (s, C<sub>Ar</sub>), 159.83 (s, C<sub>Ar</sub>), 191.80 (s, CHO); MS (EI, 70eV): m/z (%) 452,1 (5) [M]; 361 (100) [M, (-CH<sub>2</sub>Ph)]; HR MS (EI, 70 eV): *m/z* calcd for C<sub>24</sub>H<sub>19</sub>BrO<sub>4</sub>: 450.04780; found: 450.04778.



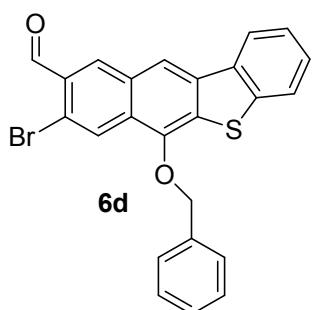
Yield: 90 %; orange solid, mp 136-138 °C;  $^1\text{H}$  NMR (200MHz,  $\text{CDCl}_3$ , 25 °C, TMS,  $\delta$ ): 3.84 (s, OCH<sub>3</sub>, 3H), 4.09 (s, OCH<sub>3</sub>, 3H), 5.14 (s, OCH<sub>2</sub>Ph, 2H), 6.55 (s, Ar, 1H), 6.99 (s, Ar, 1H), 7.31-7.48 (m, C<sub>6</sub>H<sub>5</sub>, 5H), 8.68 (s, Ar, 1H), 8.60 (s, Ar, 1H), 8.40 (s, Ar, 1H), 10.49 (s, CHO, 1H);  $^{13}\text{C}$  NMR (50MHz,  $\text{CDCl}_3$ , 25 °C, TMS,  $\delta$ ): 53.90 (s, OCH<sub>3</sub>), 56.29 (s, OCH<sub>3</sub>), 91.90 (s, OCH<sub>2</sub>Ph), 98.80 (s, C<sub>Ar</sub>-H), 98.20 (s, C<sub>Ar</sub>-H), 103.70 (s, C<sub>Ar</sub>-H), 119.20 (s, C<sub>Ar</sub>-H), 120.55 (s, C<sub>Ar</sub>-H), 124.90 (s, C<sub>Ar</sub>), 126.87 (s, C<sub>Ar</sub>), 127.17 (s, C<sub>Ar</sub>-H), 129.70 (s, C<sub>Ar</sub>-H), 129.38 (s, C<sub>Ar</sub>-H), 132.58 (s, C<sub>Ar</sub>), 135.55 (s, C<sub>Ar</sub>), 137.15 (s, C<sub>Ar</sub>), 149.99 (s, C<sub>Ar</sub>), 157.99 (s, C<sub>Ar</sub>), 158.26 (s, C<sub>Ar</sub>), 159.49 (s, C<sub>Ar</sub>), 160.10 (s, C<sub>Ar</sub>), 192.12 (s, CHO); MS (EI, 70eV): m/z (%) 452,1 (3) [M]; 361 (100) [M, (-CH<sub>2</sub>Ph)]; HR MS (EI, 70 eV): *m/z* calcd for C<sub>24</sub>H<sub>19</sub>BrO<sub>4</sub>: 450.04778; found: 450.047802.



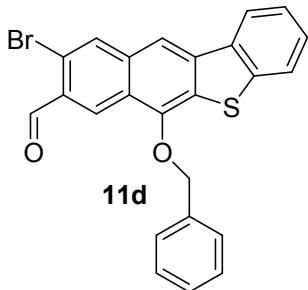
Yield: 89 %; yellow solid, mp 169-170 °C; <sup>1</sup>H NMR (200MHz, CDCl<sub>3</sub>, 25 °C, TMS, δ): 4.11 (s, CH<sub>3</sub>, 3H), 5.17 (s, OCH<sub>2</sub>Ph, 2H), 7.06-7.30 (m, C<sub>6</sub>H<sub>5</sub>, Ar-H, 8H), 8.42 (d, Ar-H, 1H), 8.46 (d, Ar-H, 2H), 8.65 (s, Ar, 1H), 10.48 (s, CHO, 1H); <sup>13</sup>C NMR (50MHz, CDCl<sub>3</sub>, 25 °C, TMS, δ): 31.47 (s, CH<sub>3</sub>), 79.10 (s, OCH<sub>2</sub>Ph), 108.75 (s, C<sub>Ar</sub>H), 117.71 (s, C<sub>Ar</sub>H), 119.50 (s, C<sub>Ar</sub>H), 120.23 (s, C<sub>Ar</sub>H), 121.11 (s, C<sub>Ar</sub>H), 122.52 (s, C<sub>Ar</sub>H), 125.35 (s, C<sub>Ar</sub>H), 126.35 (s, C<sub>Ar</sub>), 127.67 (s, C<sub>Ar</sub>), 127.94 (s, C<sub>Ar</sub>), 128.32 (s, C<sub>Ar</sub>), 128.36 (s, C<sub>Ar</sub>), 128.61 (s, 2x *o*-Ph), 128.70 (s, *p*-Ph), 128.84 (s, 2x *m*-Ph), 129.88 (s, C<sub>Ar</sub>-Br), 133.23 (s, *ipso*-C<sub>6</sub>H<sub>5</sub>), 136.09 (s, C<sub>Ar</sub>), 136.35 (s, C<sub>Ar</sub>), 144.30 (s, C-OCH<sub>2</sub>Ph), 191.91 (s, CHO); MS (EI, 70eV): m/z (%) 443 (20) [M]; 353 (100) [M(-CH<sub>2</sub>Ph)]. HR MS (EI, 70 eV): *m/z* calcd for C<sub>25</sub>H<sub>18</sub>BrNO<sub>2</sub>: 444.34689; found: 444.34805.



Yield: 79 %; light yellow solid, mp 172-174 °C; <sup>1</sup>H NMR (200MHz, CDCl<sub>3</sub>, 25 °C, TMS, δ): 4.10 (s, CH<sub>3</sub>, 3H), 5.20 (s, OCH<sub>2</sub>Ph, 2H), 7.04-7.30 (m, C<sub>6</sub>H<sub>5</sub>, Ar-H, 8H), 8.44 (d, Ar, 1H), 8.46 (d, Ar, 2H), 8.67 (s, Ar, 1H), 10.50 (s, CHO, 1H); <sup>13</sup>C NMR (50MHz, CDCl<sub>3</sub>, 25 °C, TMS, δ): 31.45 (s, CH<sub>3</sub>), 80.10 (s, OCH<sub>2</sub>Ph), 109.15 (s, C<sub>Ar</sub>H), 116.81 (s, C<sub>Ar</sub>H), 118.90 (s, C<sub>Ar</sub>H), 120.23 (s, C<sub>Ar</sub>H), 121.15 (s, C<sub>Ar</sub>H), 123.02 (s, C<sub>Ar</sub>H), 126.35 (s, C<sub>Ar</sub>H), 127.30 (s, C<sub>Ar</sub>), 127.68 (s, C<sub>Ar</sub>), 127.96 (s, C<sub>Ar</sub>), 127.28 (s, C<sub>Ar</sub>), 127.33 (s, C<sub>Ar</sub>), 128.61 (s, 2x *o*-Ph), 128.70 (s, *p*-Ph), 129.84 (s, 2x *m*-Ph), 129.81 (s, C<sub>Ar</sub>-Br), 137.28 (s, *ipso*-C<sub>6</sub>H<sub>5</sub>), 136.07 (s, C<sub>Ar</sub>), 138.38 (s, C<sub>Ar</sub>), 145.31 (s, C-OCH<sub>2</sub>Ph), 192.92 (s, CHO); MS (EI, 70eV): m/z (%) 443 (25) [M]; 353 (100) [M(PhCH<sub>2</sub>)]. HR MS (EI, 70 eV): *m/z* calcd for C<sub>25</sub>H<sub>18</sub>BrNO<sub>2</sub>: 444.34689; found: 444.34834.



Yield: 15 %; yellow solid, mp 182-184 °C,  $^1\text{H}$  NMR (200MHz,  $\text{CDCl}_3$ , 25 °C, TMS,  $\delta$ ): 5.37 (s,  $\text{OCH}_2\text{Ph}$ , 2H), 7.49 (d, Ar, 2H), 7.49-7.70 (m,  $\text{C}_6\text{H}_5$ , 5H), 7.89 (m, Ar, 1H), 8.23 (m, Ar, 1H), 8.45 (s, Ar, 1H), 8.49 (s, Ar, 1H), 8.70 (s, Ar, 1H), 10.60 (s, CHO, 1H);  $^{13}\text{C}$  NMR (50MHz,  $\text{CDCl}_3$ , 25 °C, TMS,  $\delta$ ): 75.34 (s,  $\text{OCH}_2\text{Ph}$ ), 118.11 (s,  $\text{C}_{\text{Ar}}\text{H}$ ), 120.21 (s,  $\text{C}_{\text{Ar}}\text{H}$ ), 122.44 (s,  $\text{C}_{\text{Ar}}\text{H}$ ), 123.04 (s,  $\text{C}_{\text{Ar}}\text{H}$ ), 125.20 (s,  $\text{C}_{\text{Ar}}\text{H}$ ), 126.26 (s,  $\text{C}_{\text{Ar}}\text{H}$ ), 128.50 (s, 2x *o*-Ph), 128.74 (s, *p*-Ph), 129.88 (s, 2x *m*-Ph), 130.76 (s,  $\text{C}_{\text{Ar}}$ ), 133.71 (s,  $\text{C}_{\text{Ar}}$ ), 134.02 (s,  $\text{C}_{\text{Ar}}$ ), 135.32 (s,  $\text{C}_{\text{Ar}}$ ), 139.06 (s,  $\text{C}_{\text{Ar}}$ ), 143.67 (s,  $\text{C}_{\text{Ar}}$ ), 146.45 (s,  $\text{C}_{\text{Ar}}$ ), 149.55 (s,  $\text{C}_{\text{Ar}}$ ) 153.88 (s,  $\text{C}_{\text{Ar}}$ ), 155.09 (s,  $\text{C}_{\text{Ar}}$ ), 191.89 (s, CHO), MS (EI, 70eV): m/z (%) 382 (33) [M]; 305.9 (100) [M(-Ph)]; HR MS (EI, 70 eV): *m/z* calcd for  $\text{C}_{24}\text{H}_{15}\text{BrO}_2\text{S}$ : 445.99720; found: 445.99805.



Yield: 18 %; yellow solid, mp 193-195 °C;  $^1\text{H}$  NMR (200MHz,  $\text{CDCl}_3$ , 25 °C, TMS,  $\delta$ ): 5.35 (s,  $\text{OCH}_2\text{Ph}$ , 2H), 7.44 (d, Ar, 2H), 7.50-7.78 (m,  $\text{C}_6\text{H}_5$ , 5H), 7.88 (m, Ar, 1H), 8.25 (m, Ar, 1H), 8.42 (s, Ar, 1H), 8.51 (s, Ar, 1H), 8.65 (s, Ar, 1H), 10.52 (s, CHO, 1H);  $^{13}\text{C}$  NMR (50MHz,  $\text{CDCl}_3$ , 25 °C, TMS,  $\delta$ ): 75.34 (s,  $\text{OCH}_2\text{Ph}$ ), 118.11 (s,  $\text{C}_{\text{Ar}}\text{H}$ ), 120.21 (s,  $\text{C}_{\text{Ar}}\text{H}$ ), 122.44 (s,  $\text{C}_{\text{Ar}}\text{H}$ ), 123.04 (s,  $\text{C}_{\text{Ar}}\text{H}$ ), 125.20 (s,  $\text{C}_{\text{Ar}}\text{H}$ ), 126.26 (s,  $\text{C}_{\text{Ar}}\text{H}$ ), 128.50 (s, 2x *o*-Ph), 128.74 (s, *p*-Ph), 129.88 (s, 2x *m*-Ph), 130.76 (s,  $\text{C}_{\text{Ar}}$ ), 133.71 (s,  $\text{C}_{\text{Ar}}$ ), 134.02 (s,  $\text{C}_{\text{Ar}}$ ), 135.32 (s,  $\text{C}_{\text{Ar}}$ ), 139.06 (s,  $\text{C}_{\text{Ar}}$ ), 140, 33 (s,  $\text{C}_{\text{Ar}}$ ), 142,11 (s,  $\text{C}_{\text{Ar}}$ ) 143.67 (s,  $\text{C}_{\text{Ar}}$ ), 153.88 (s,  $\text{C}_{\text{Ar}}$ ), 155.09 (s,  $\text{C}_{\text{Ar}}$ ), 191.89 (s, CHO); MS (EI, 70eV): m/z (%) 382 (26) [M]; 305.9 (100) [M, (-Ph)] HR MS (EI, 70 eV): *m/z* calcd for  $\text{C}_{24}\text{H}_{15}\text{BrO}_2\text{S}$ : 445.99720; found: 445.99650.

\*\*\* NMR LAB \*\*\*

\*\* AVANCE DRX500 \*\*

Tel: 042 6803 307

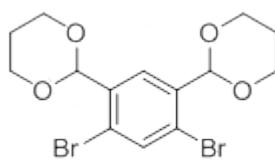
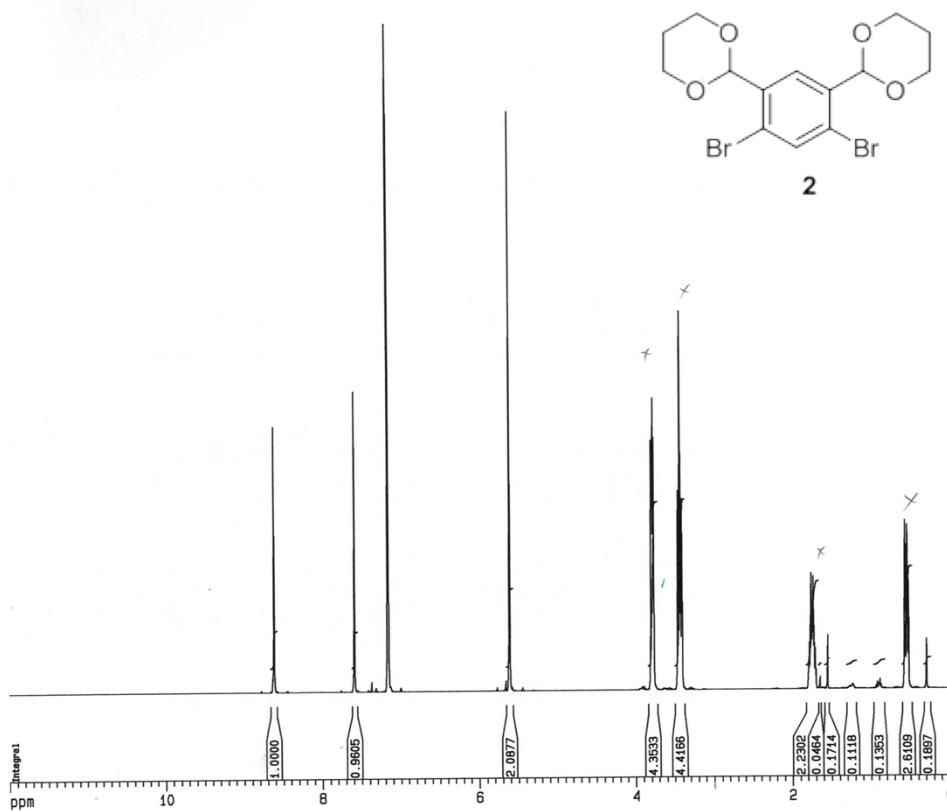
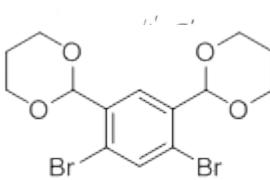
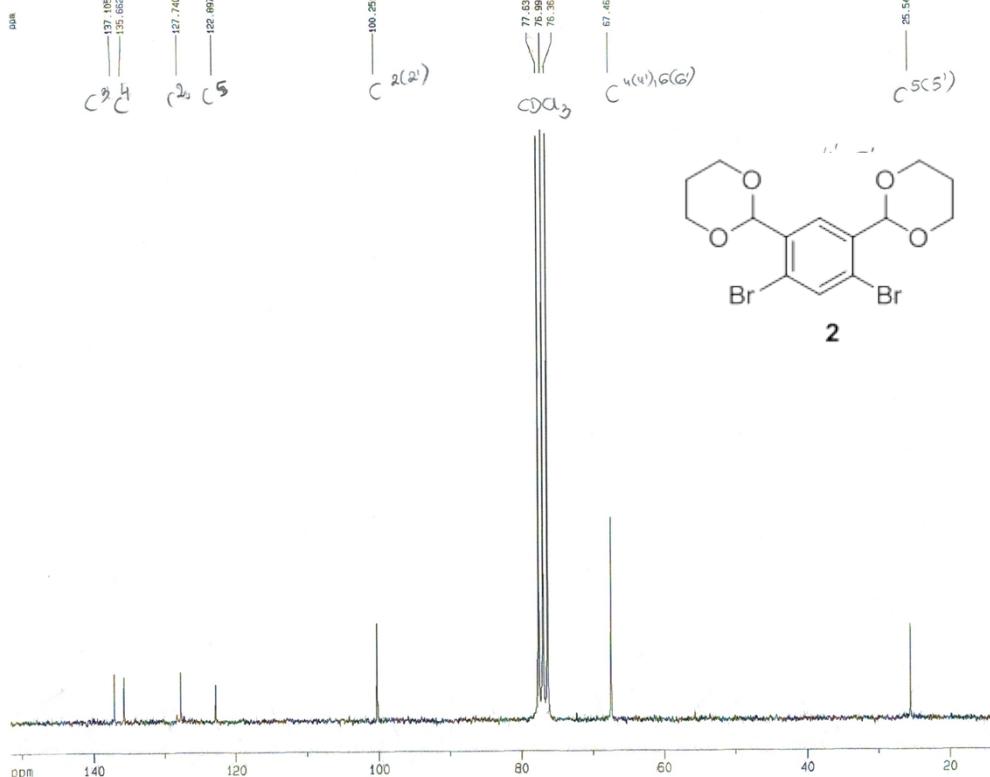
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**2****2**

\*\*\* NMR LAB \*\*\*

\*\* AVANCE AV 200 \*\*

Tel: 0426803 226

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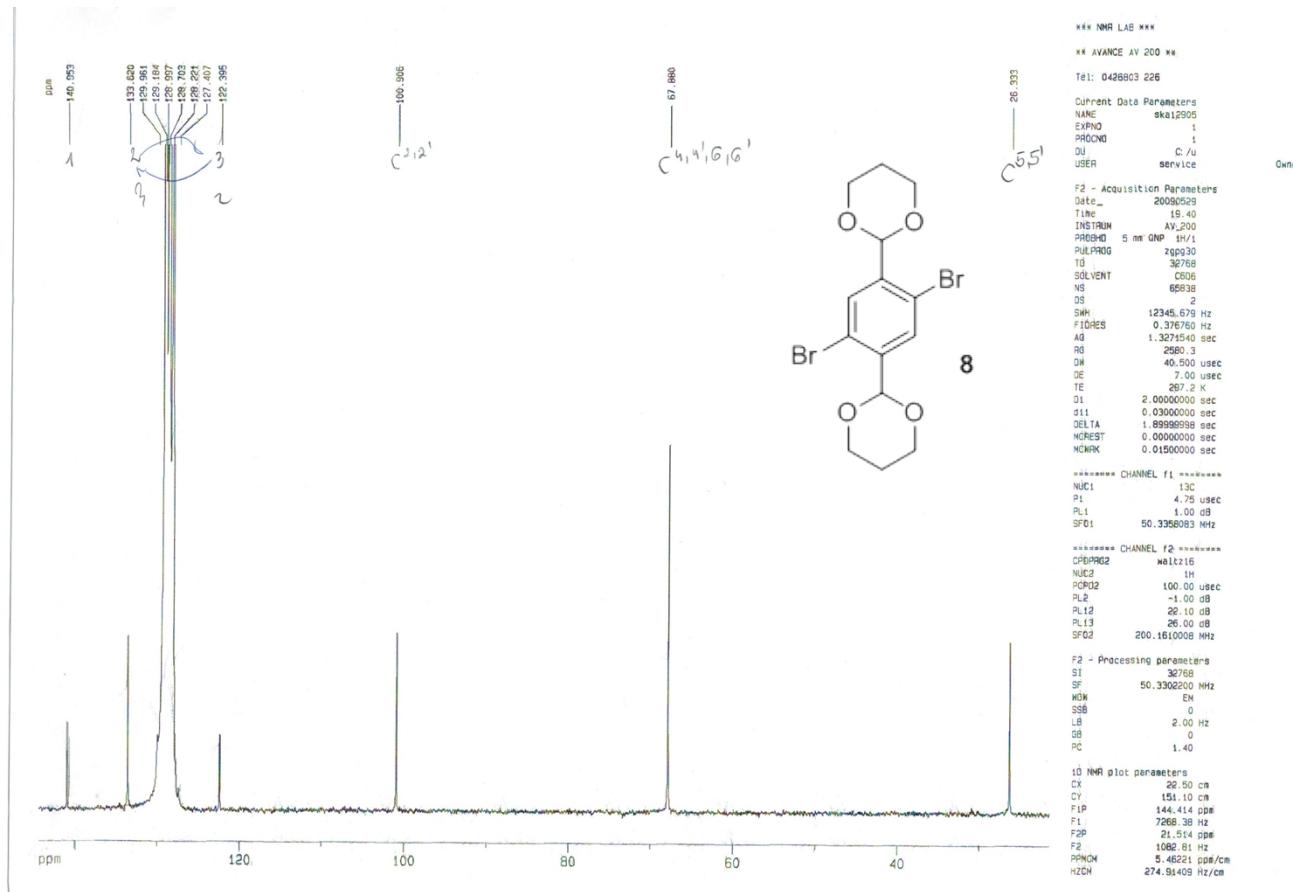
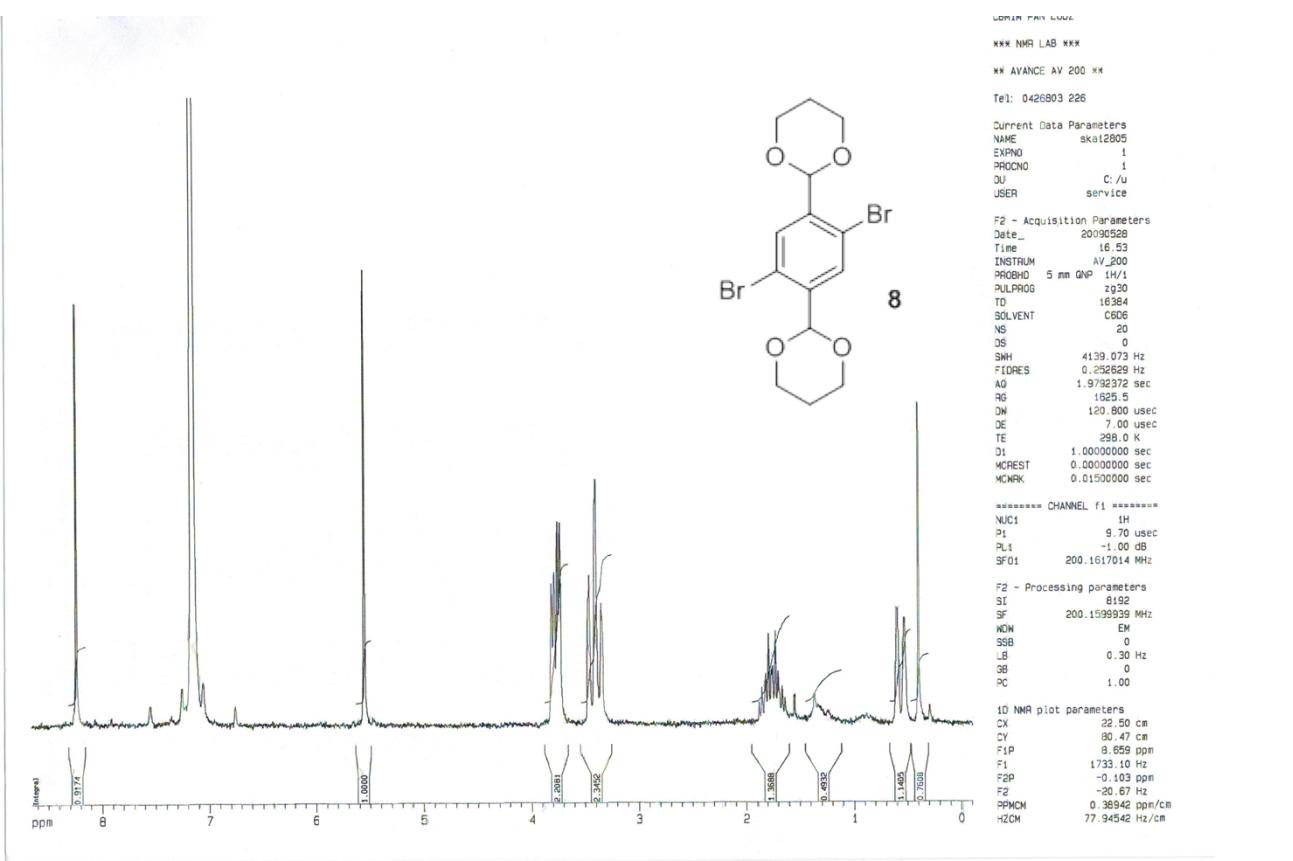
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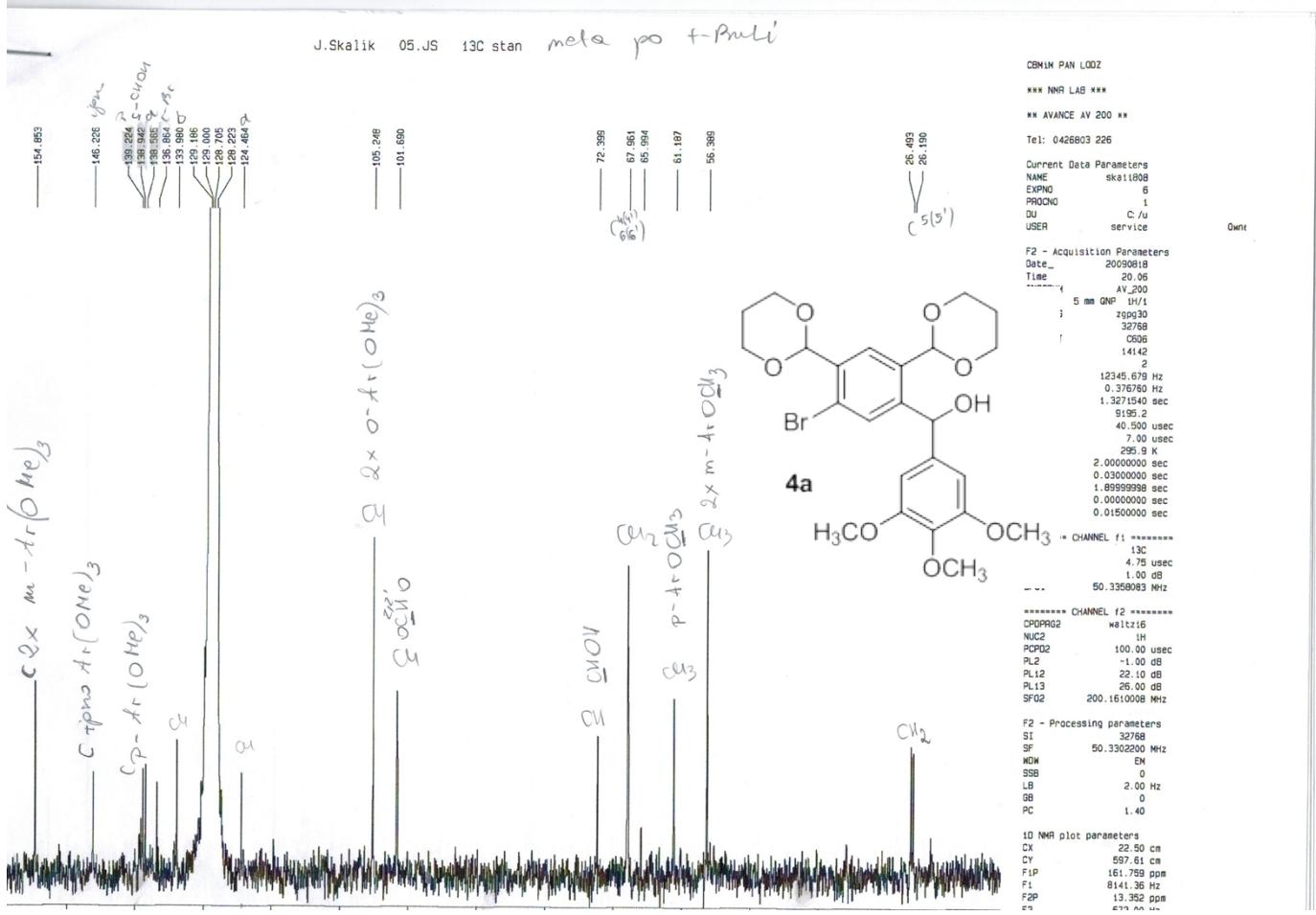
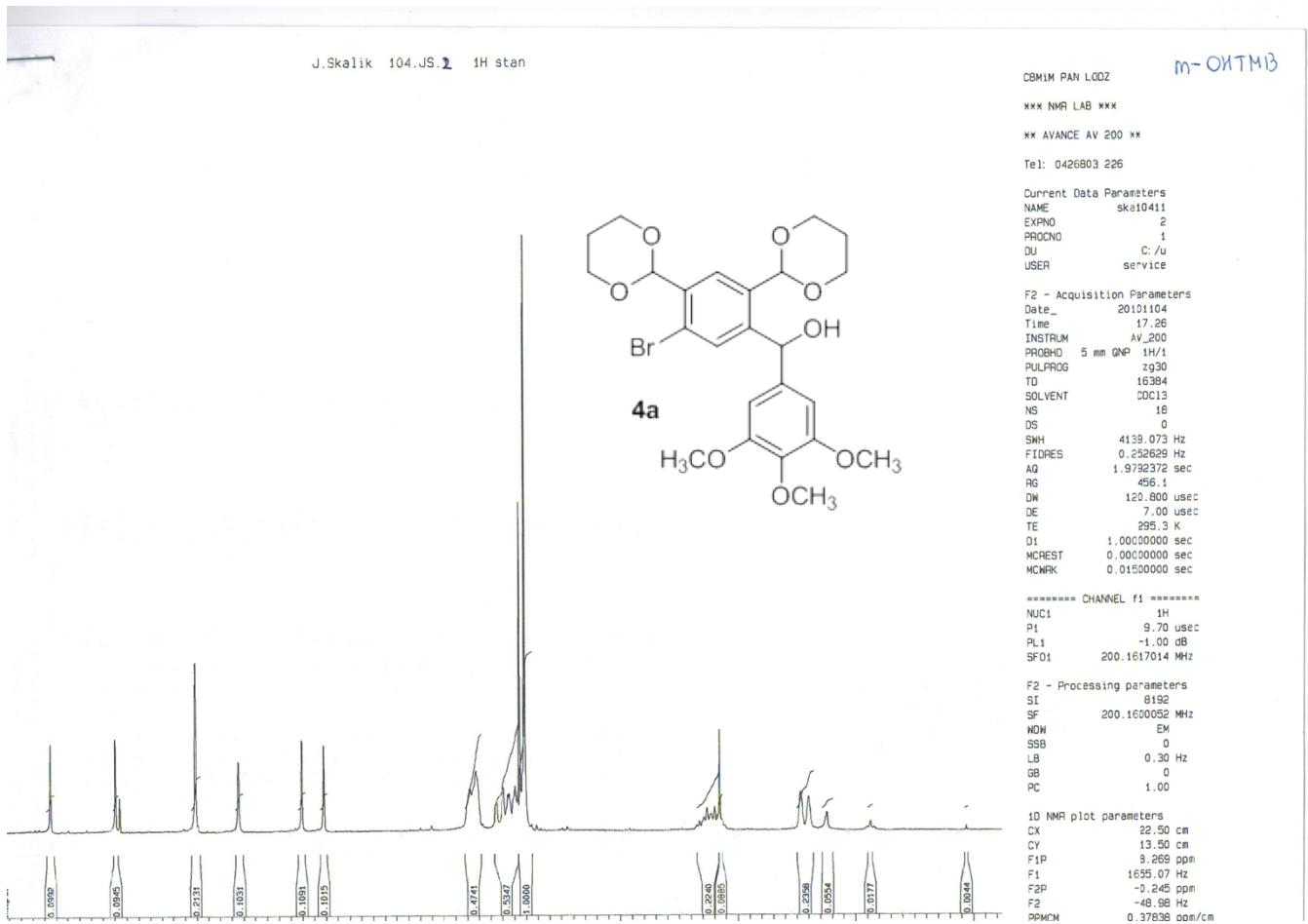
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 SF01 50.335083 MHz

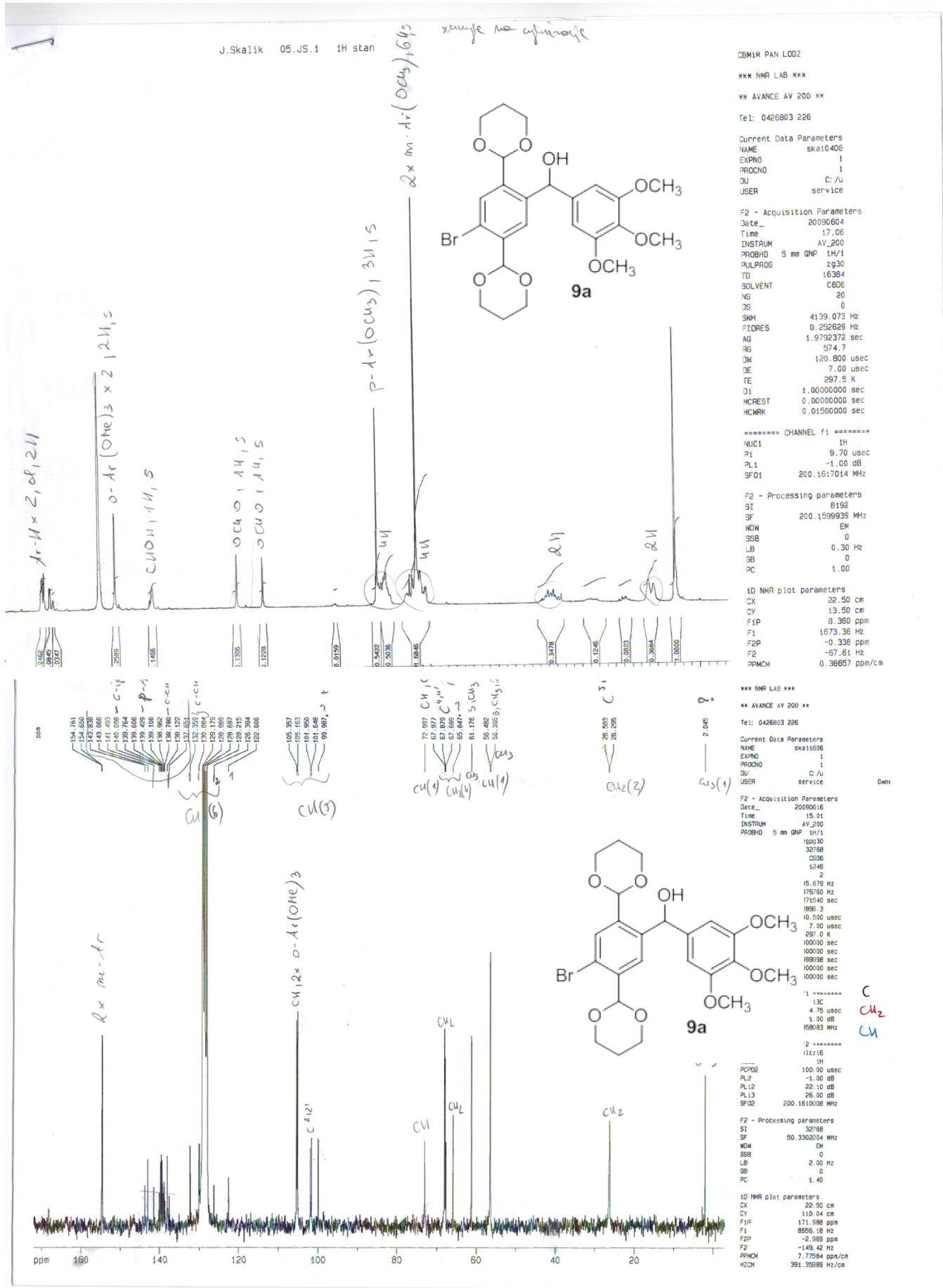
\*\*\*\*\* CHANNEL f2 \*\*\*\*\*  
 CPDPRG2 waltz16  
 NUC2 1H  
 PCPD2 100.00 usec  
 PL2 -1.00 dB  
 PL12 -22.10 dB  
 PL13 -26.00 dB  
 SF02 200.1610008 MHz

F2 - Processing parameters  
 SI 32768  
 SF 50.3302736 MHz  
 DW EM  
 SSB 0  
 LB 2.00 Hz  
 GB 0  
 PC 1.40

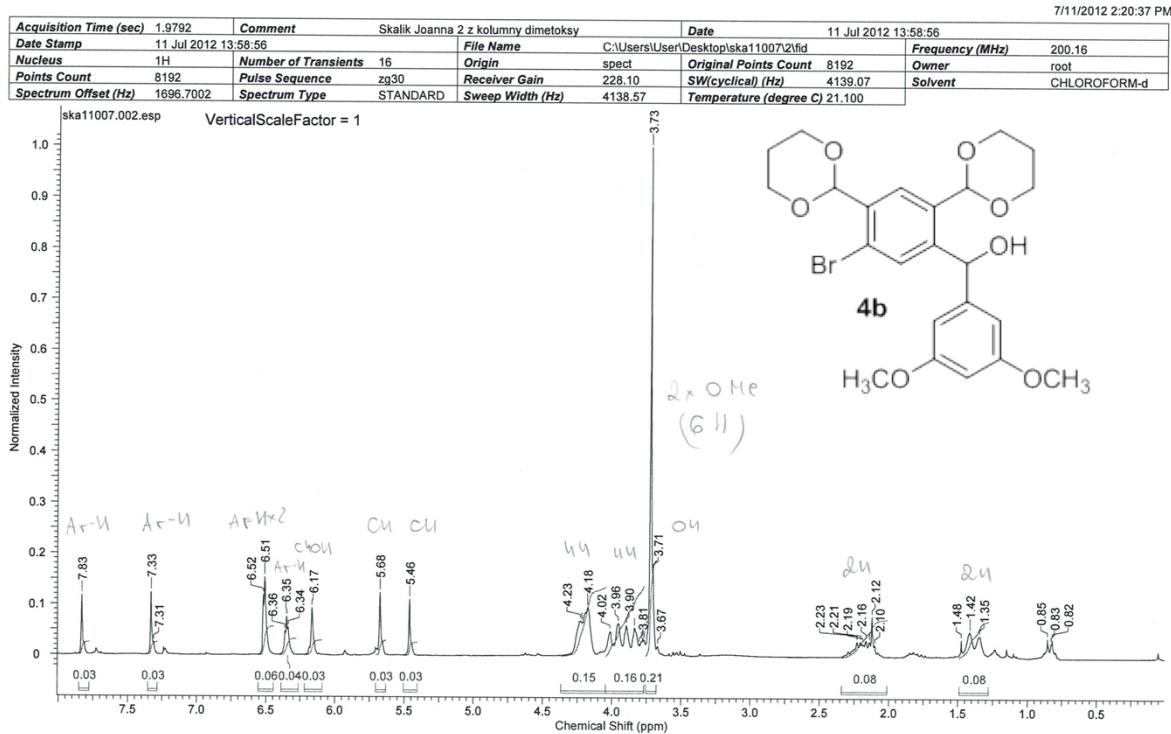
1D NMR plot parameters  
 CX 22.50 cm  
 CY 13.50 cm  
 F1P 151.582 ppm  
 F1 7629.15 Hz  
 F2P 151.582 ppm  
 F2 889.54 Hz  
 PPMCM 6.12894 ppm/cm  
 HZCM 398.47137 Hz/cm



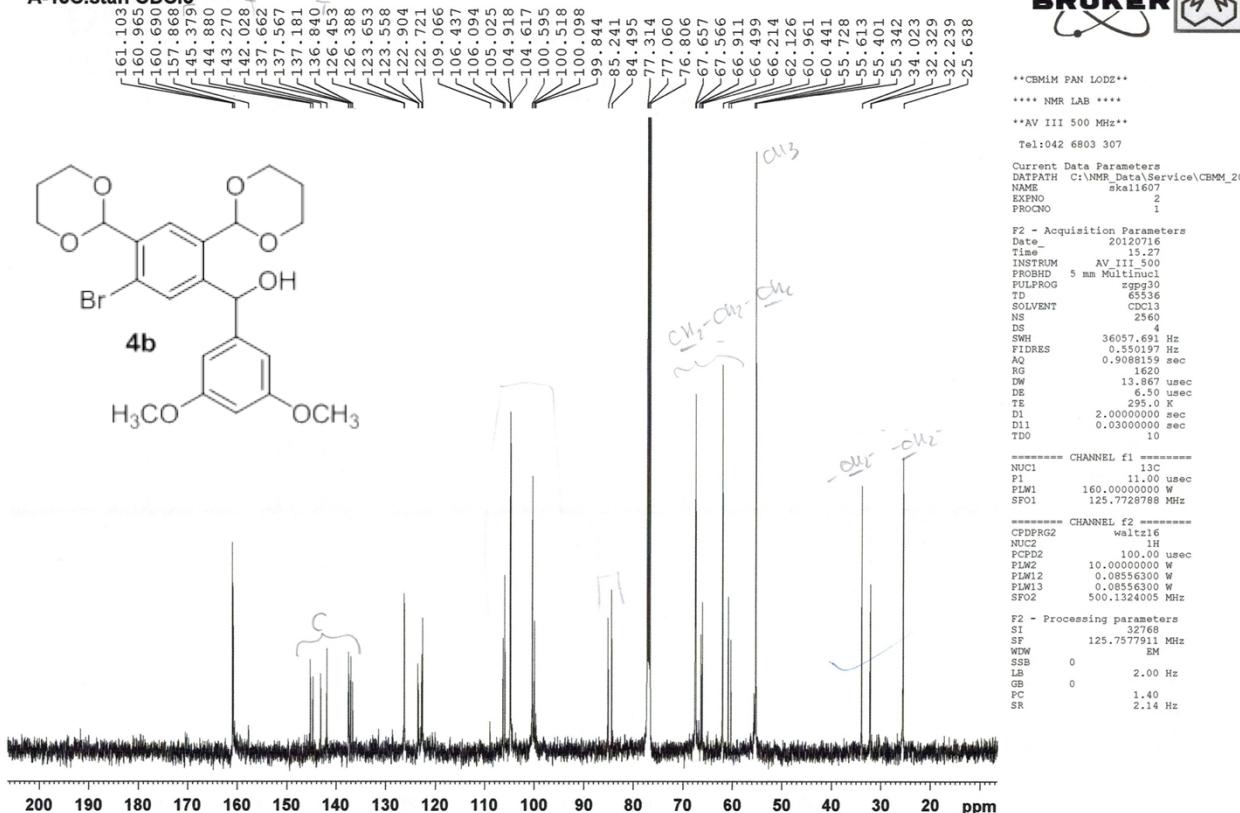




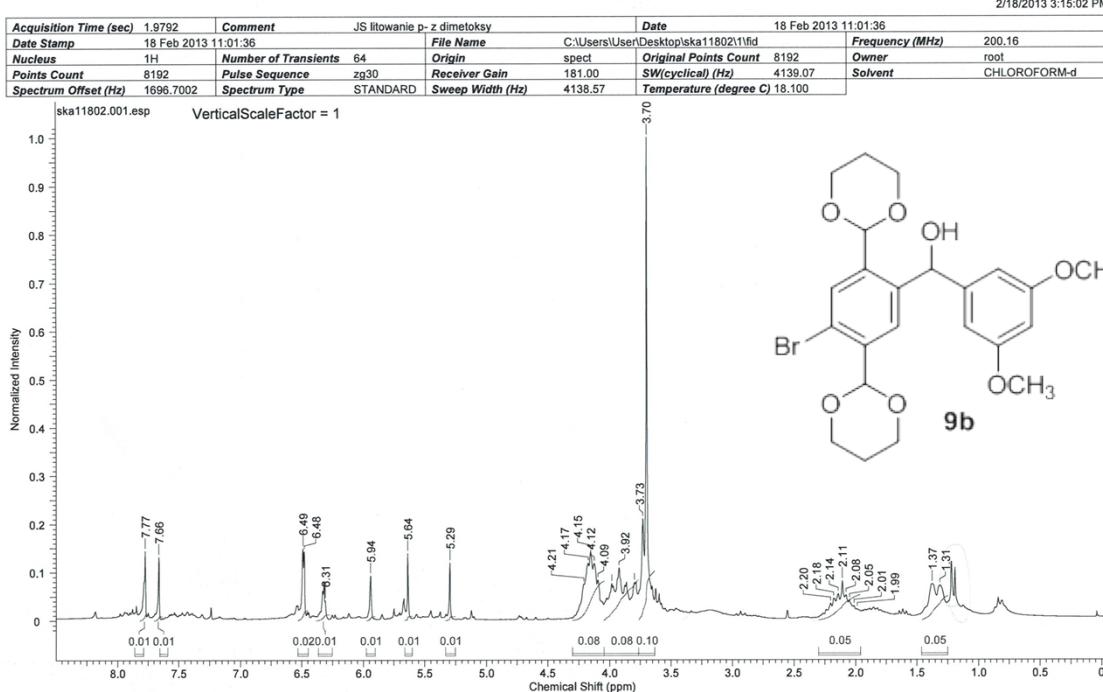
This report was created by ACD/NMR Processor Academic Edition. For more information go to [www.acdlabs.com/nmrproc/](http://www.acdlabs.com/nmrproc/)  
**186.JS**



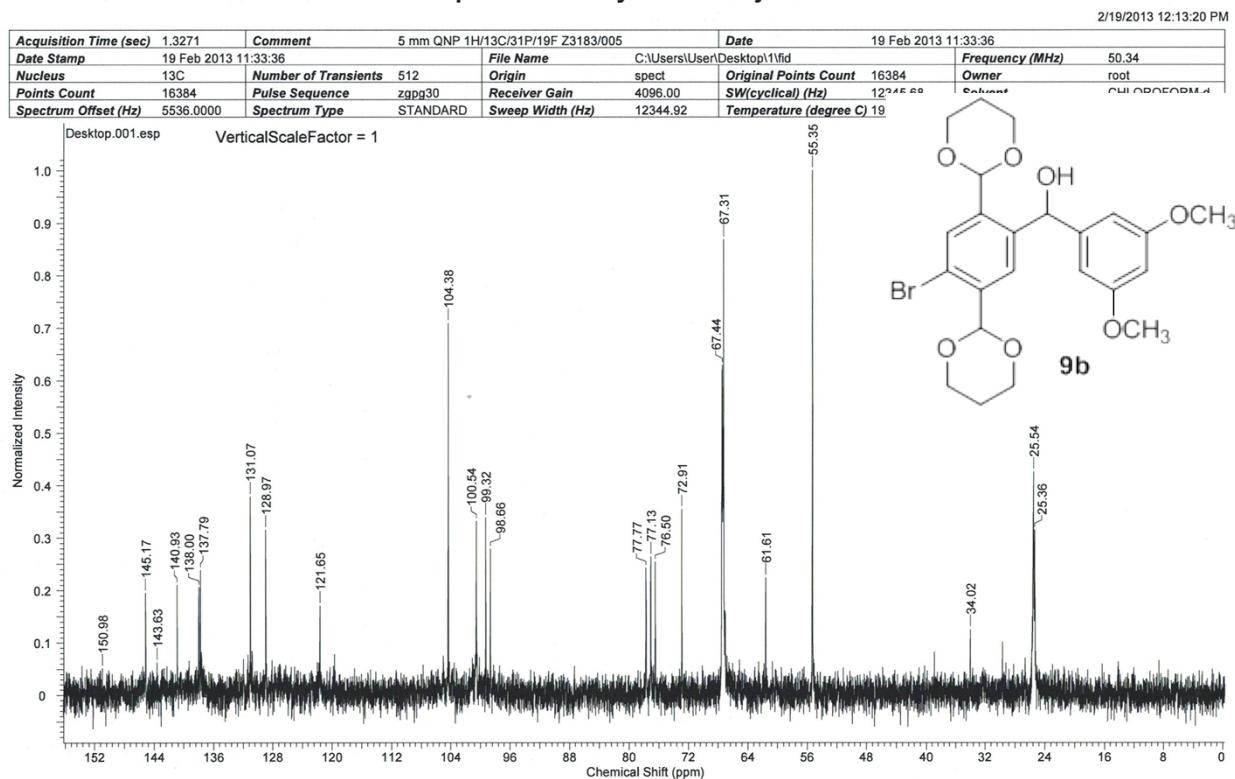
j. skalik =186.JS=  
A-13C.stan CDCl<sub>3</sub>



This report was created by ACD/NMR Processor Academic Edition. For more information go to [www.acdlabs.com/nmrproc/](http://www.acdlabs.com/nmrproc/)  
**alkohol p- z dimetoksybenzaldehydem**



This report was created by ACD/NMR Processor Academic Edition. For more information go to [www.acdlabs.com/nmrproc/](http://www.acdlabs.com/nmrproc/)  
**alkohol p- z dimetoksybenzaldehydem 13C**

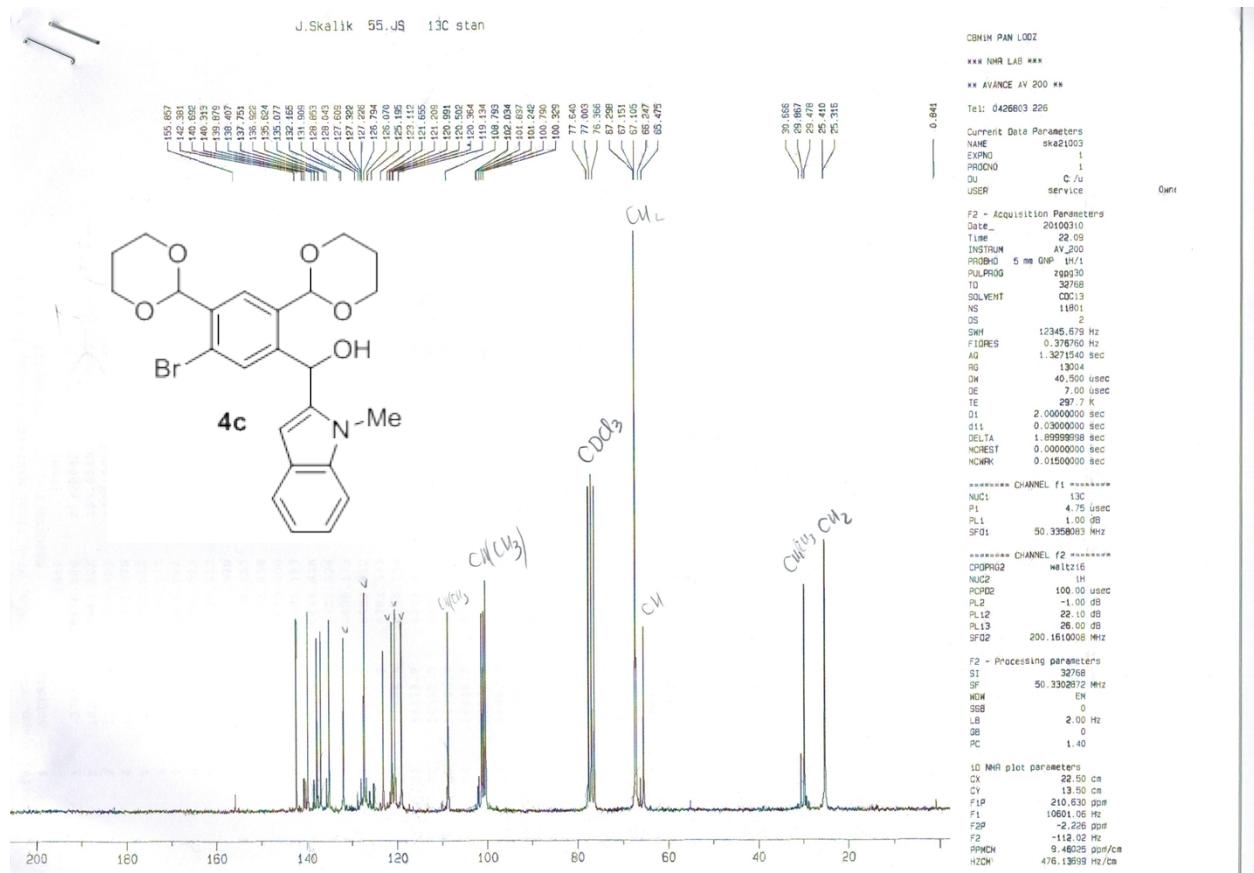
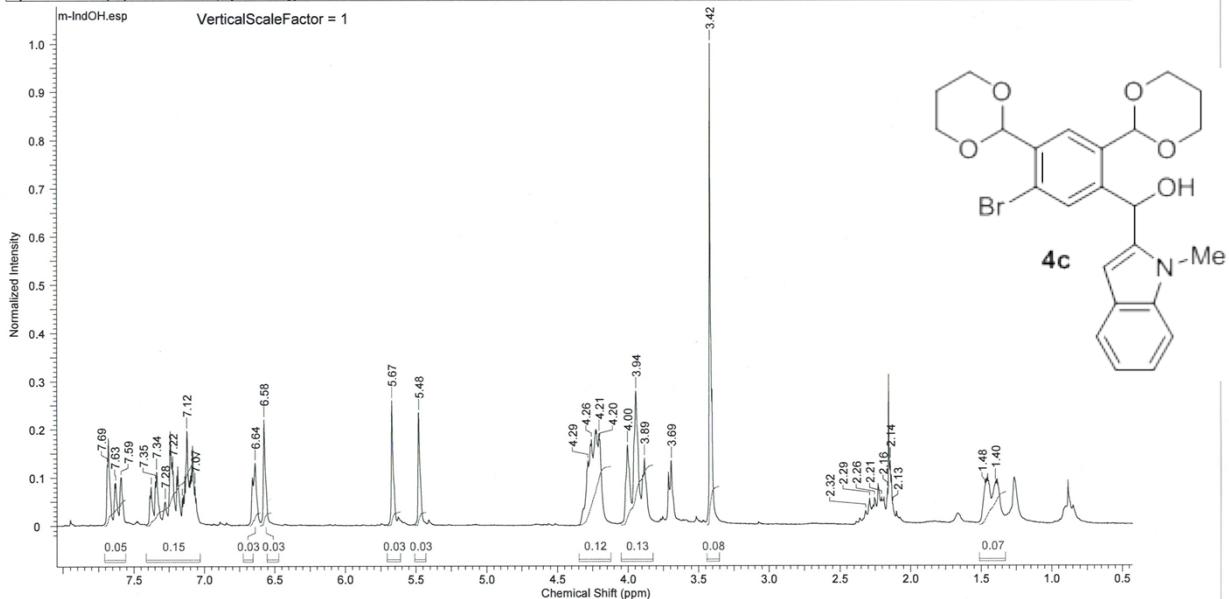


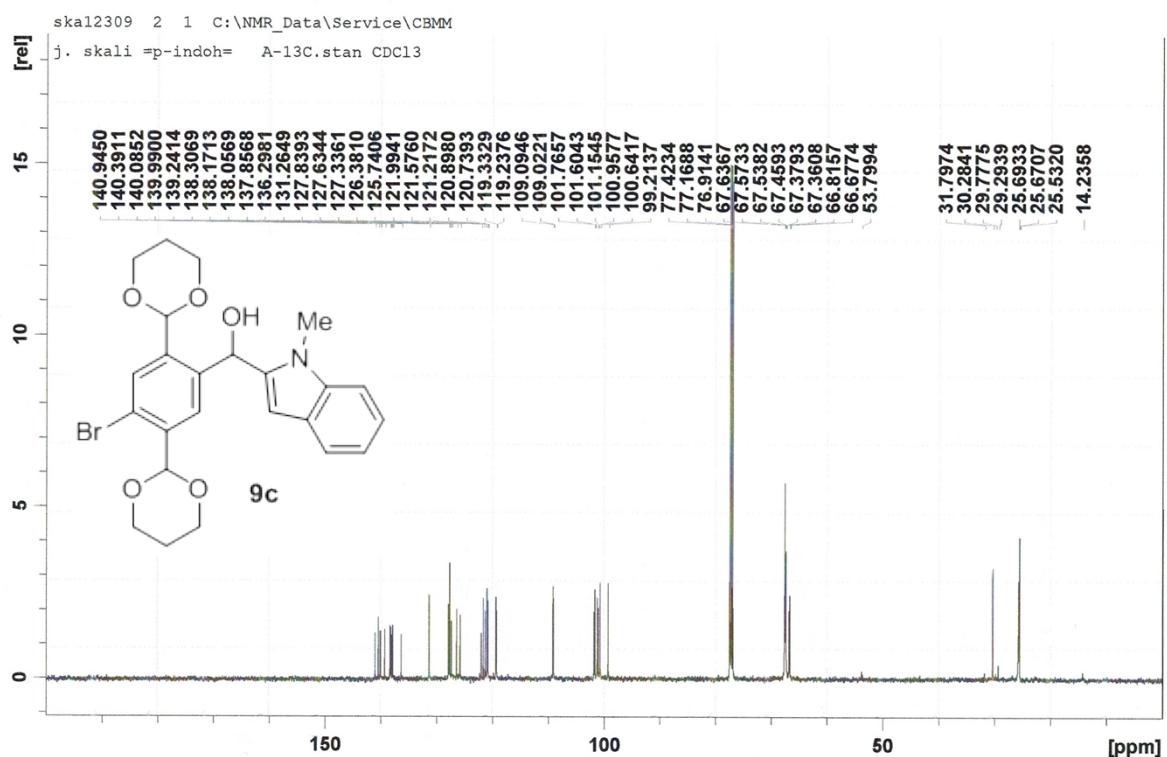
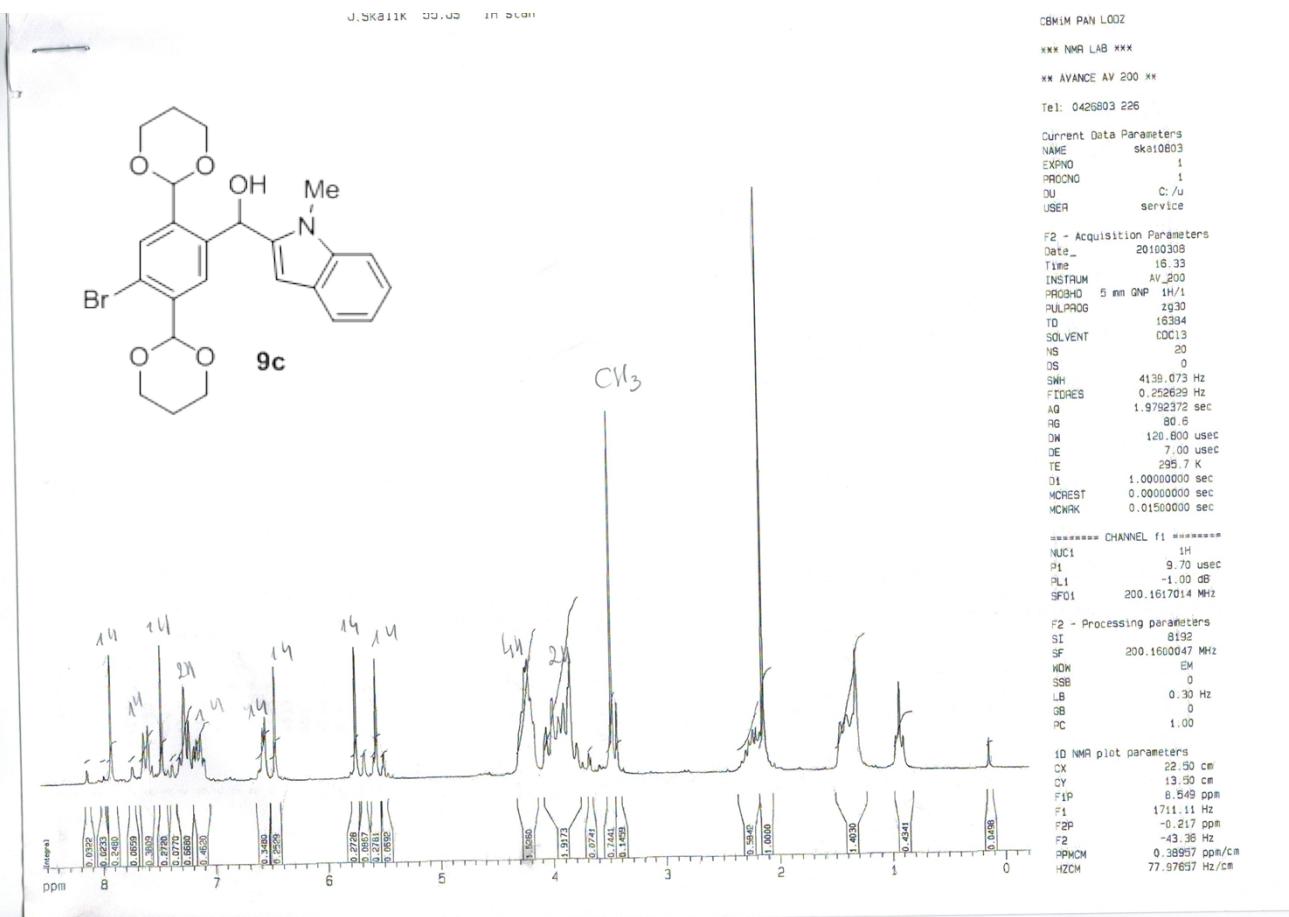
This report was created by ACD/NMR Processor Academic Edition. For more information go to [www.acdlabs.com/nmrproc/](http://www.acdlabs.com/nmrproc/)

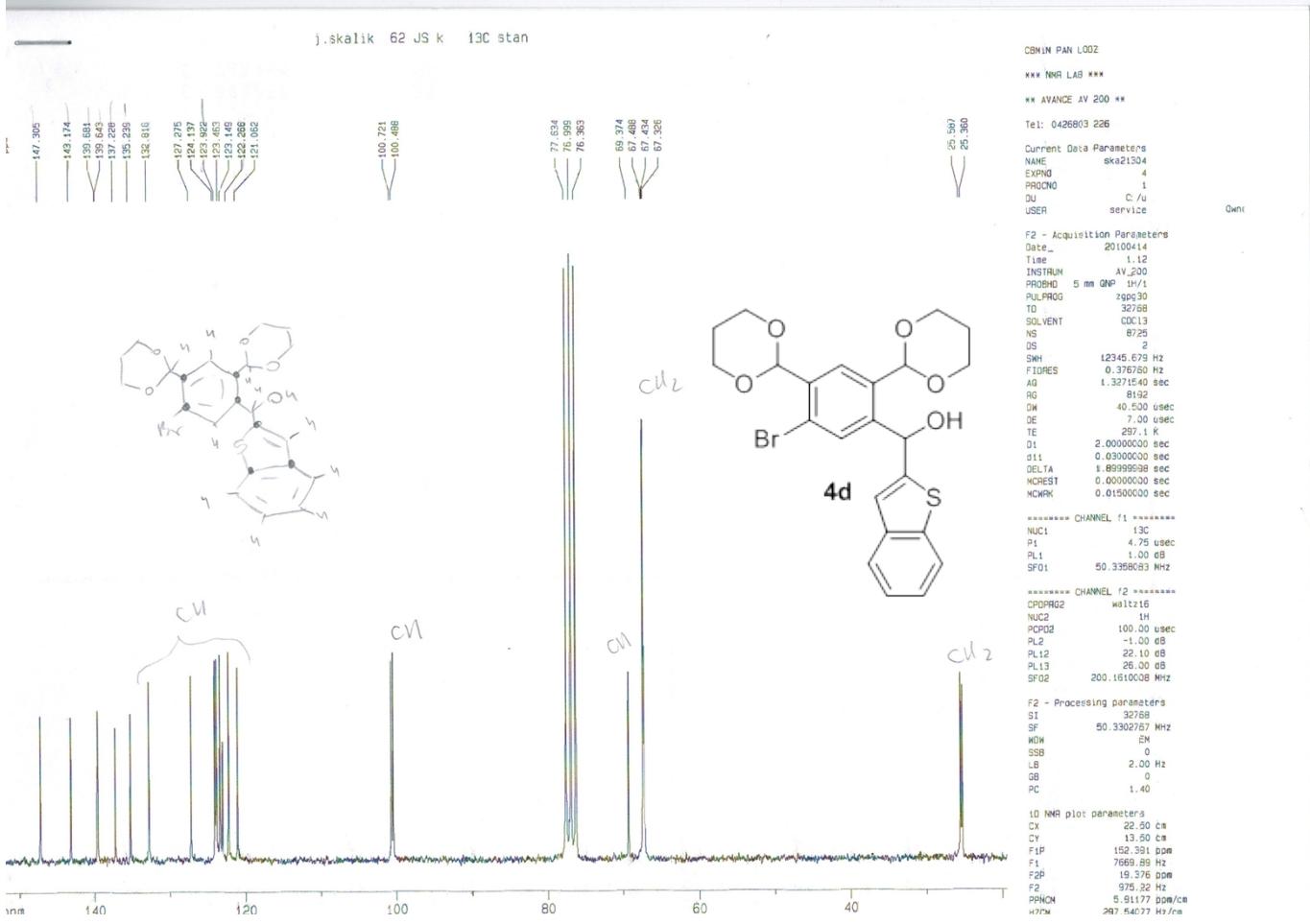
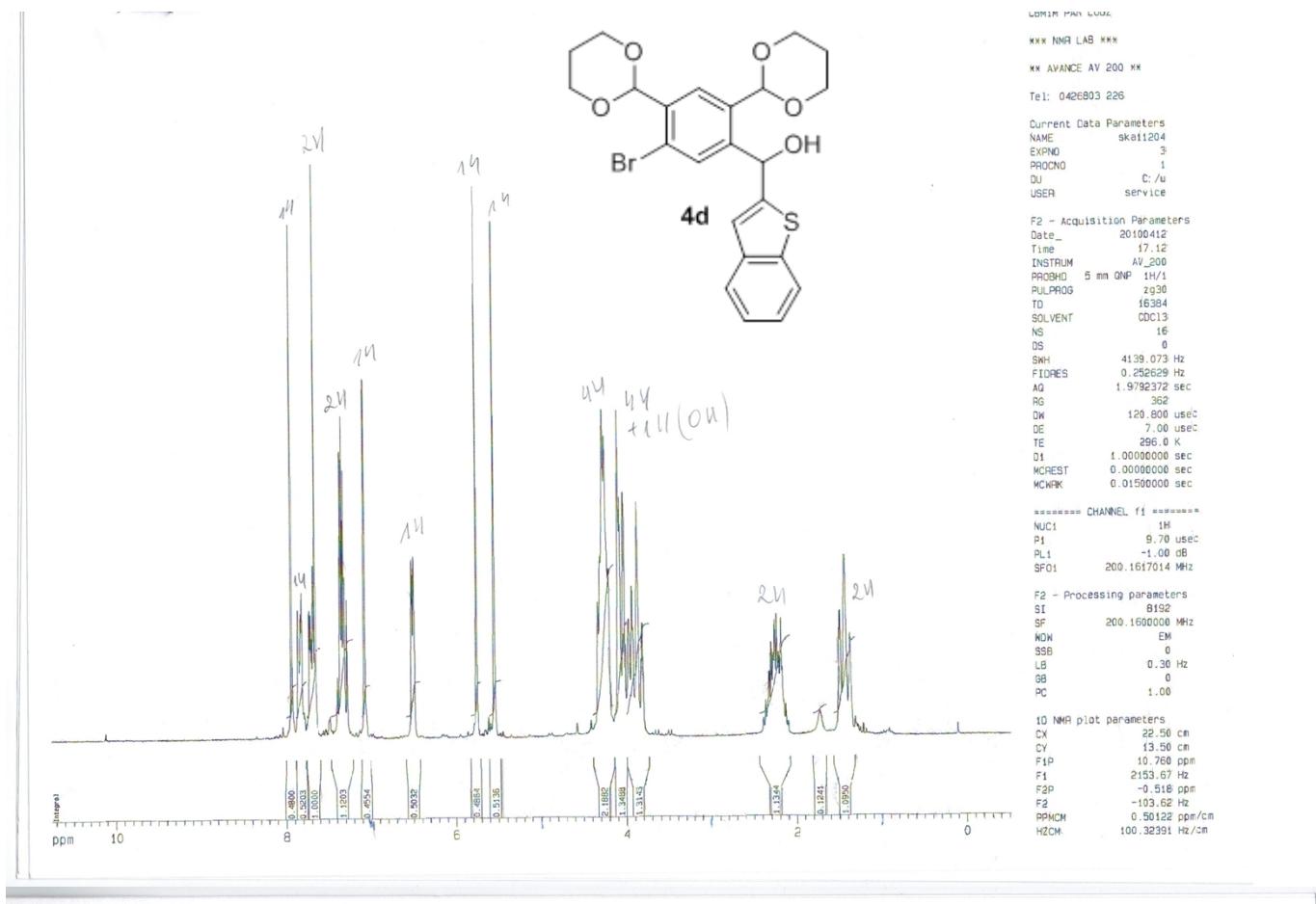
## m-IndOH

10/1/2013 9:07:51 AM  
Skalik J. m-Indoh

<u>Acquisition Time (sec)</u>	1.9792	<u>Comment</u>	Skalik J. m-Indoh	<u>Date</u>	30 Sep 2013 15:52:00	<u>Frequency (MHz)</u>	200.16
<u>Date Stamp</u>	30 Sep 2013 15:52:00	<u>File Name</u>	C:\Users\user\Desktop\m-IndOH\fid	<u>Original Points Count</u>	8192	<u>Owner</u>	root
<u>Nucleus</u>	1H	<u>Number of Transients</u>	32	<u>Origin</u>	spect	<u>Solvent</u>	CHLOROFORM-d
<u>Points Count</u>	8192	<u>Pulse Sequence</u>	zg30	<u>Receiver Gain</u>	406.40	<u>Sweep (cyclical) (Hz)</u>	4139.07
<u>Spectrum Offset (Hz)</u>	1696.7002	<u>Spectrum Type</u>	STANDARD	<u>Sweep Width (Hz)</u>	4138.57	<u>Temperature (degree C)</u>	19.400



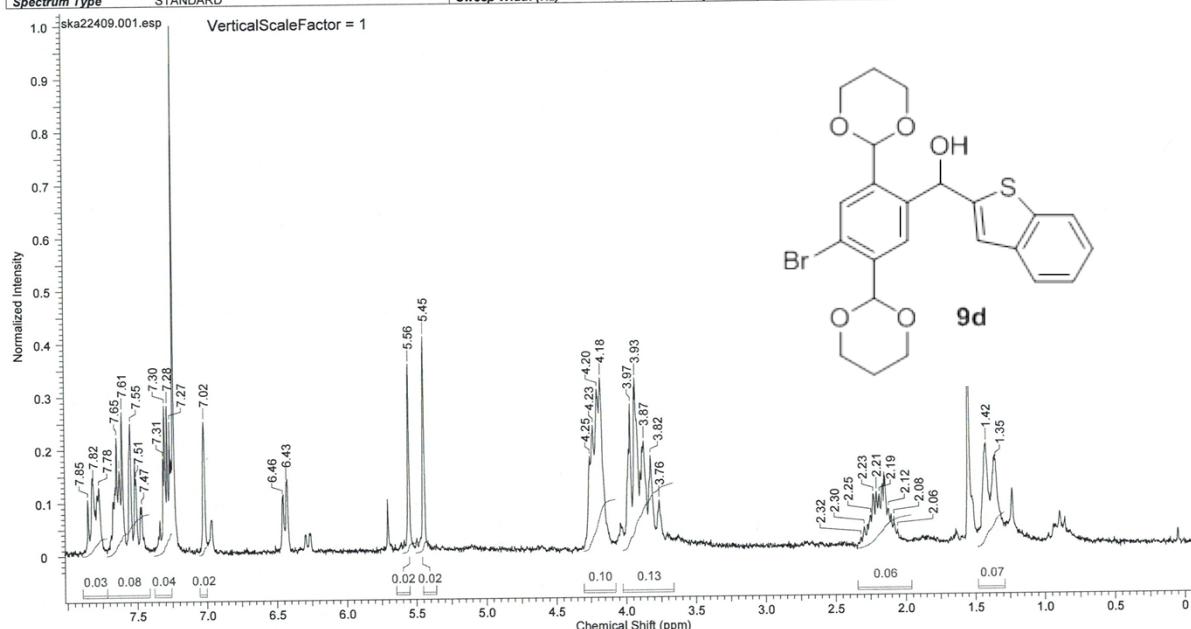




### p-BTFOH

9/24/2013 12:54:41 PM  
p-BTFOH

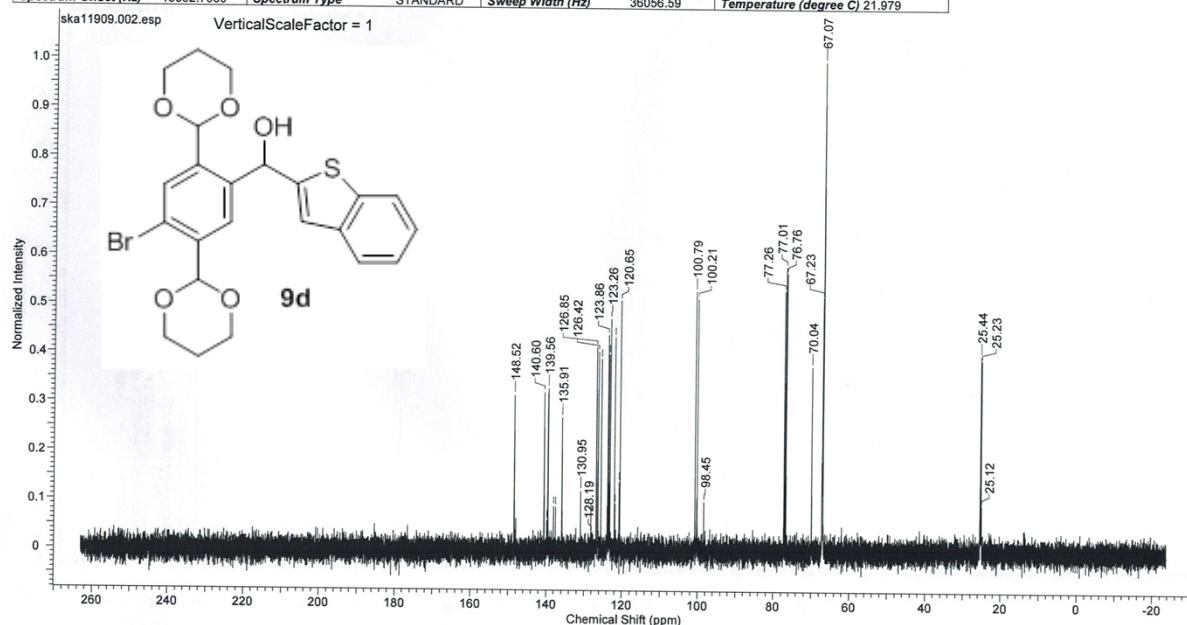
Acquisition Time (sec)	1.9792	Comment	p-BTFOH	Date	24 Sep 2013 12:44:16	Date Stamp	24 Sep 2013 12:44:16
File Name	C:\Users\User\Desktop\lska22409\1\fid	Frequency (MHz)	200.16	Nucleus	1H	Number of Transients	64
Origin	spect	Original Points Count	8192	Owner	root	Pulse Sequence	zg30
Receiver Gain	2298.80	SW(cyclical) (Hz)	4139.07	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	1696.7002
Spectrum Type	STANDARD	Sweep Width (Hz)	4138.57	Temperature (degree C)	19.200		

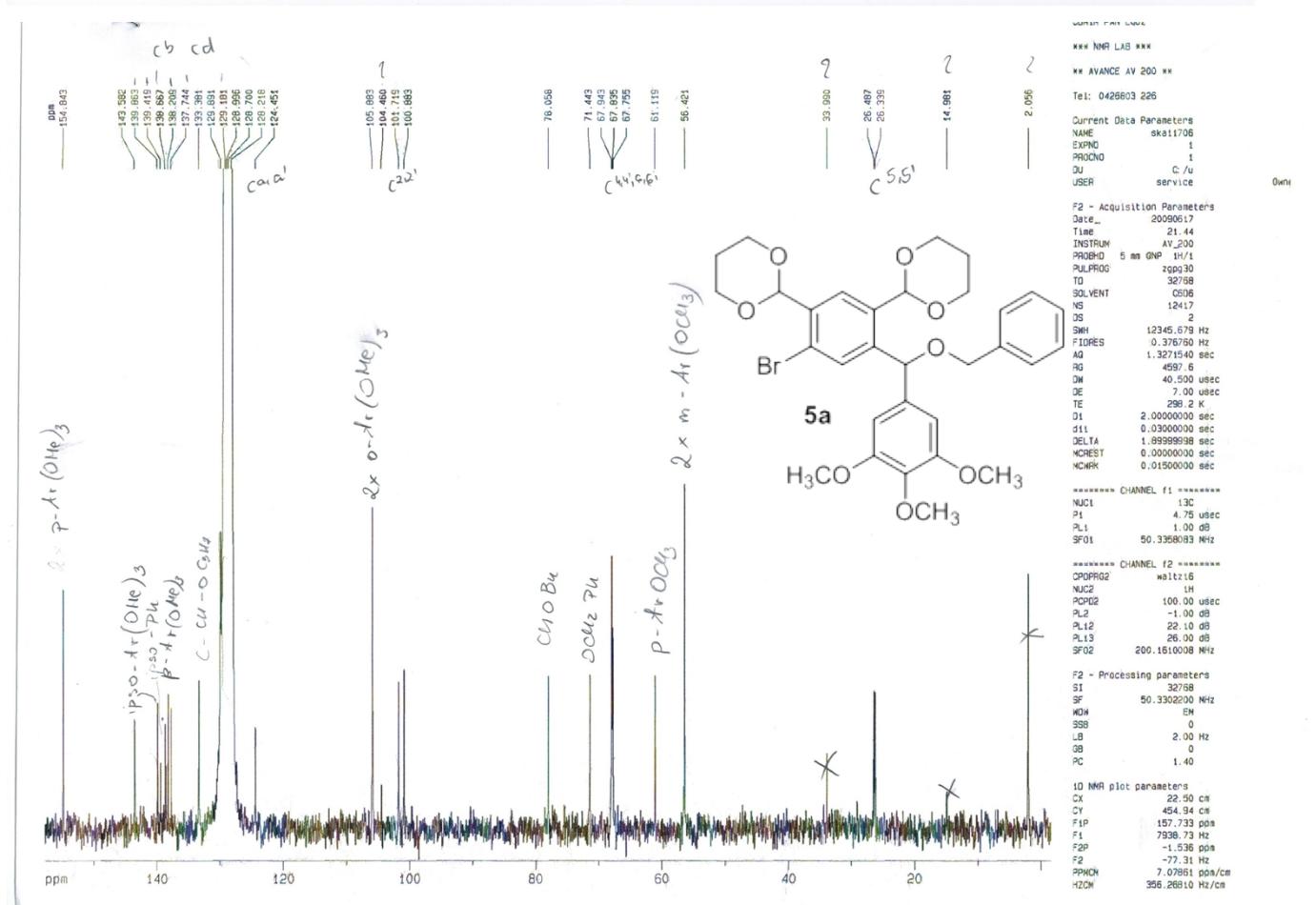
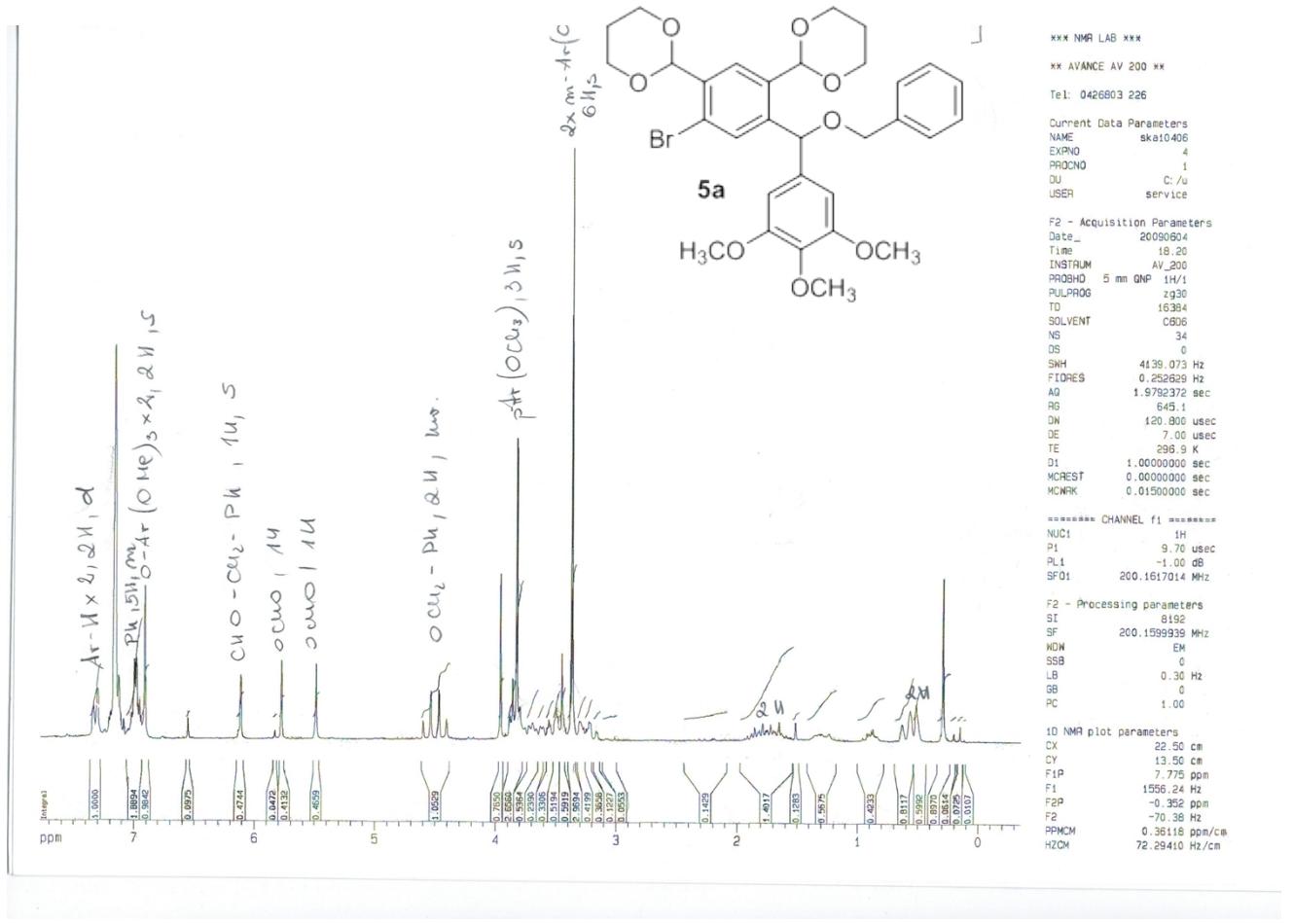


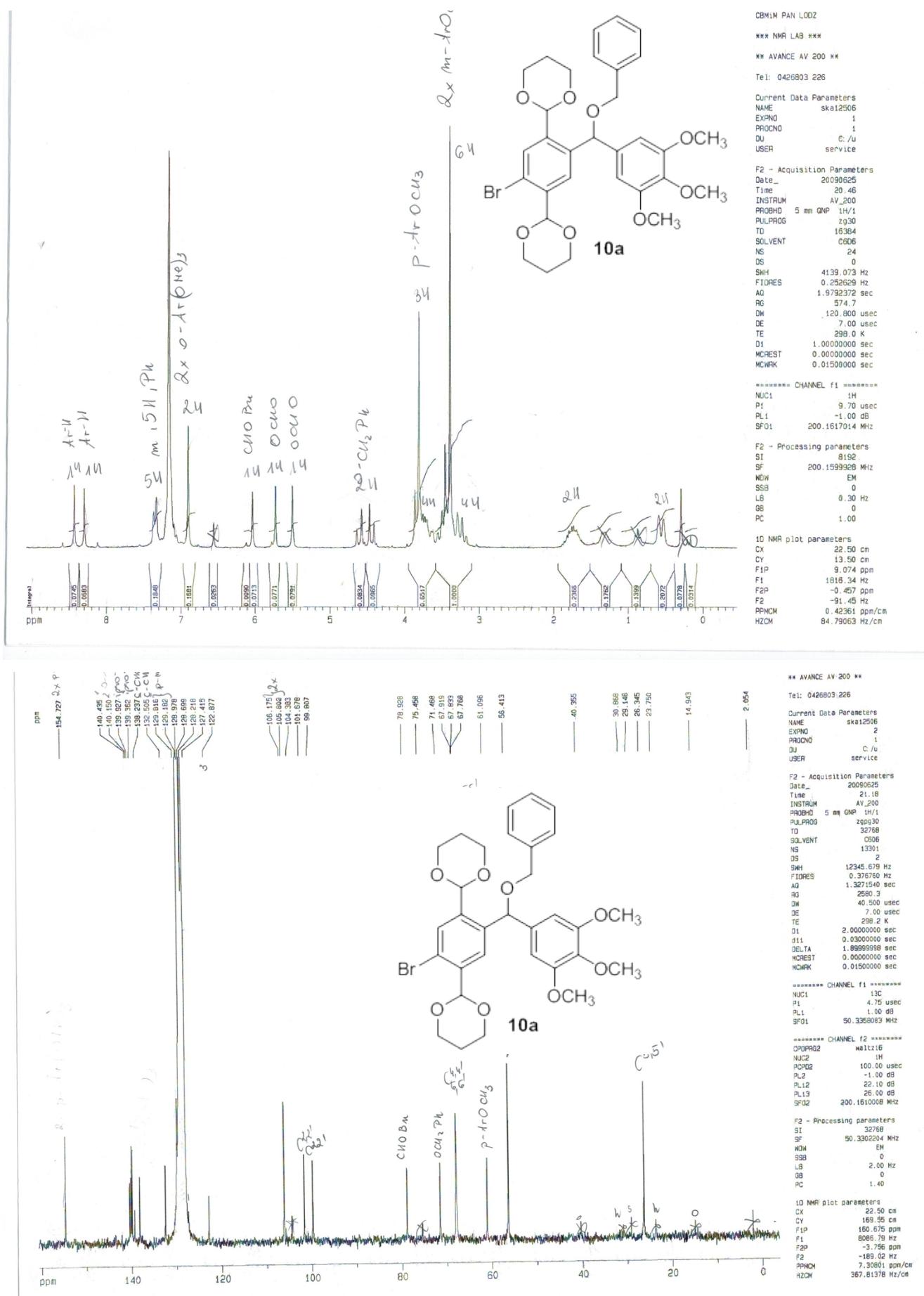
1

9/20/2013 11:16:56 AM  
j. skalik =p-btfoh= 13C{1H}

Acquisition Time (sec)	0.9088	Comment	j. skalik =p-btfoh= 13C{1H}	Date	19 Sep 2013 10:44:48	Frequency (MHz)	125.76		
Date Stamp	19 Sep 2013 10:44:48	File Name	C:\Users\User\Desktop\lska11909\2\fid	Origin	AV III 500	Number of Transients	128		
Nucleus	13C	Original Points Count	32768	Receiver Gain	2050.00	Pulse Sequence	zgpg30		
Points Count	32768	Spectrum Offset (Hz)	15052.7930 <th>Spectrum Type</th> <td>STANDARD</td> <th>SW(cyclical) (Hz)</th> <td>36057.69</td> <th>Temperature (degree C)</th> <td>21.979</td>	Spectrum Type	STANDARD	SW(cyclical) (Hz)	36057.69	Temperature (degree C)	21.979





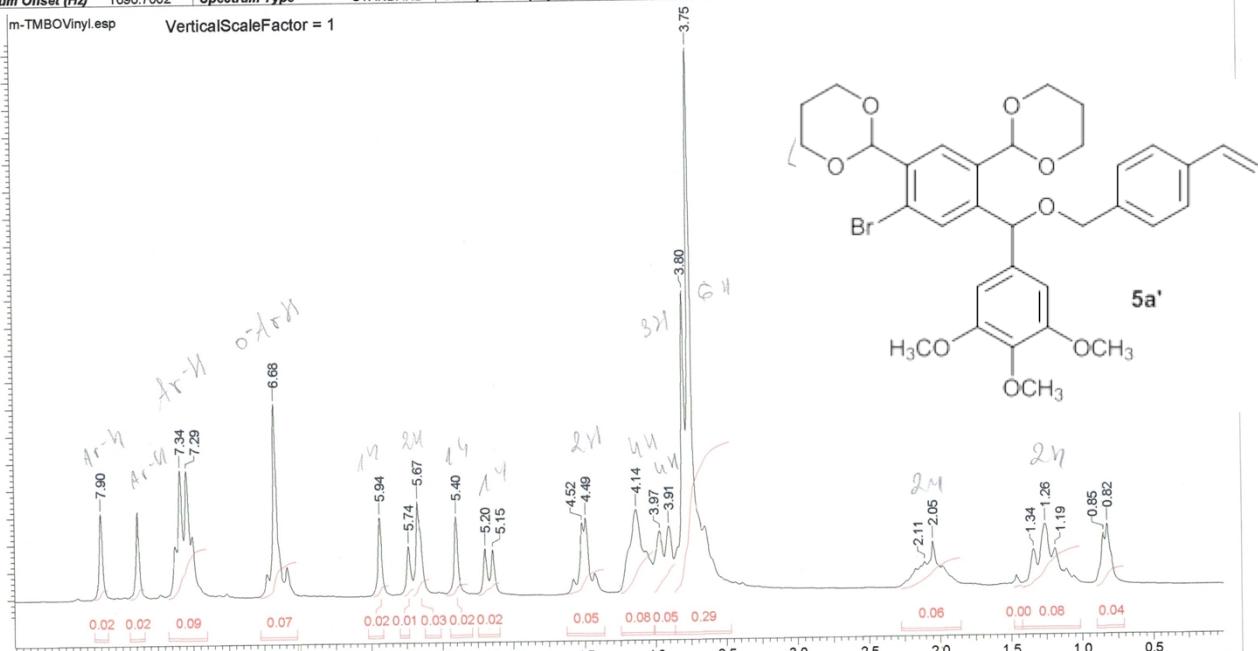


### m-TMBOBnVinyl

12/14/2011 9:56:48 AM  
m-TMBOBnVinyl

Acquisition Time (sec)	1.9792	Comment	Skalik Joanna Vinyl2	Date	01 Dec 2011 13:09:36		
Date Stamp	01 Dec 2011 13:09:36		File Name	C:\Users\Asia\Desktop\Wydruk\m-TMBOVinyl.fid	Frequency (MHz)	200.16	
Nucleus	1H	Number of Transients	64	Origin	spect	Owner	root
Points Count	8192	Pulse Sequence	zg30	Receiver Gain	22.60	Original Points Count	8192
Spectrum Offset (Hz)	1696.7002	Spectrum Type	STANDARD	Sweep Width (Hz)	4138.57	SW(cyclical) (Hz)	4139.07
						Temperature (degree C)	19.500

m-TMBOVinyl.esp VerticalScaleFactor = 1

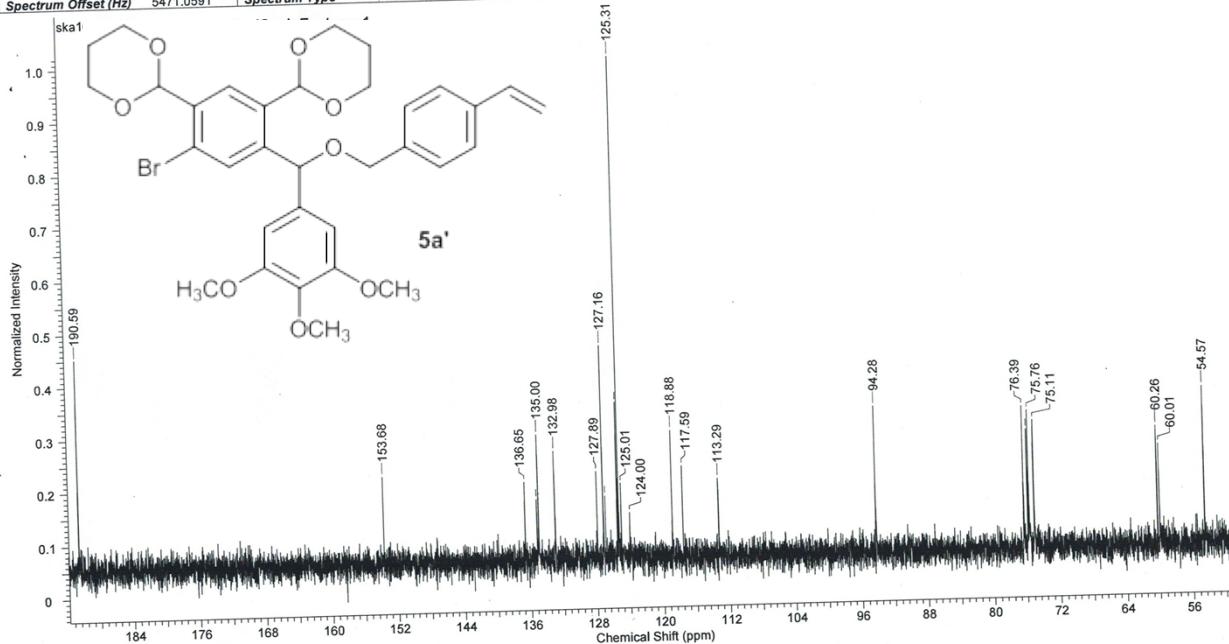


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### m-TMBOBnVinylC13

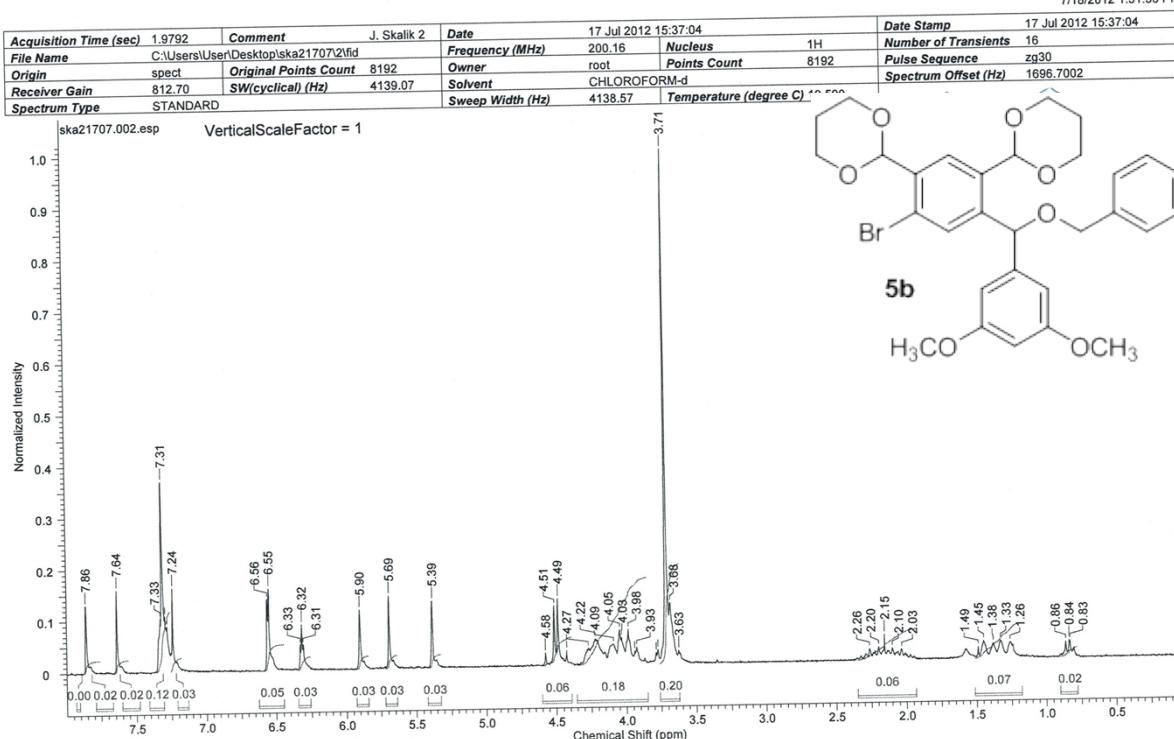
12/14/2011 10:28:05 AM  
m-TMBOBnVinylC13

Acquisition Time (sec)	1.3271	Comment	J. Skalik mTMBOBnVinyl	Date	09 Dec 2011 14:41:20		
Date Stamp	09 Dec 2011 14:41:20		File Name	C:\Users\Asia\Desktop\Wydruk\skal10912\2\fid	Frequency (MHz)	50.33	
Nucleus	<sup>13</sup> C	Number of Transients	512	Origin	spect	Owner	root
Points Count	16384	Pulse Sequence	zgpg30	Receiver Gain	1290.20	Original Points Count	16384
Spectrum Offset (Hz)	5471.0591	Spectrum Type	STANDARD	Sweep Width (Hz)	12344.92	SW(cyclical) (Hz)	12345.68
						Temperature (degree C)	19.700

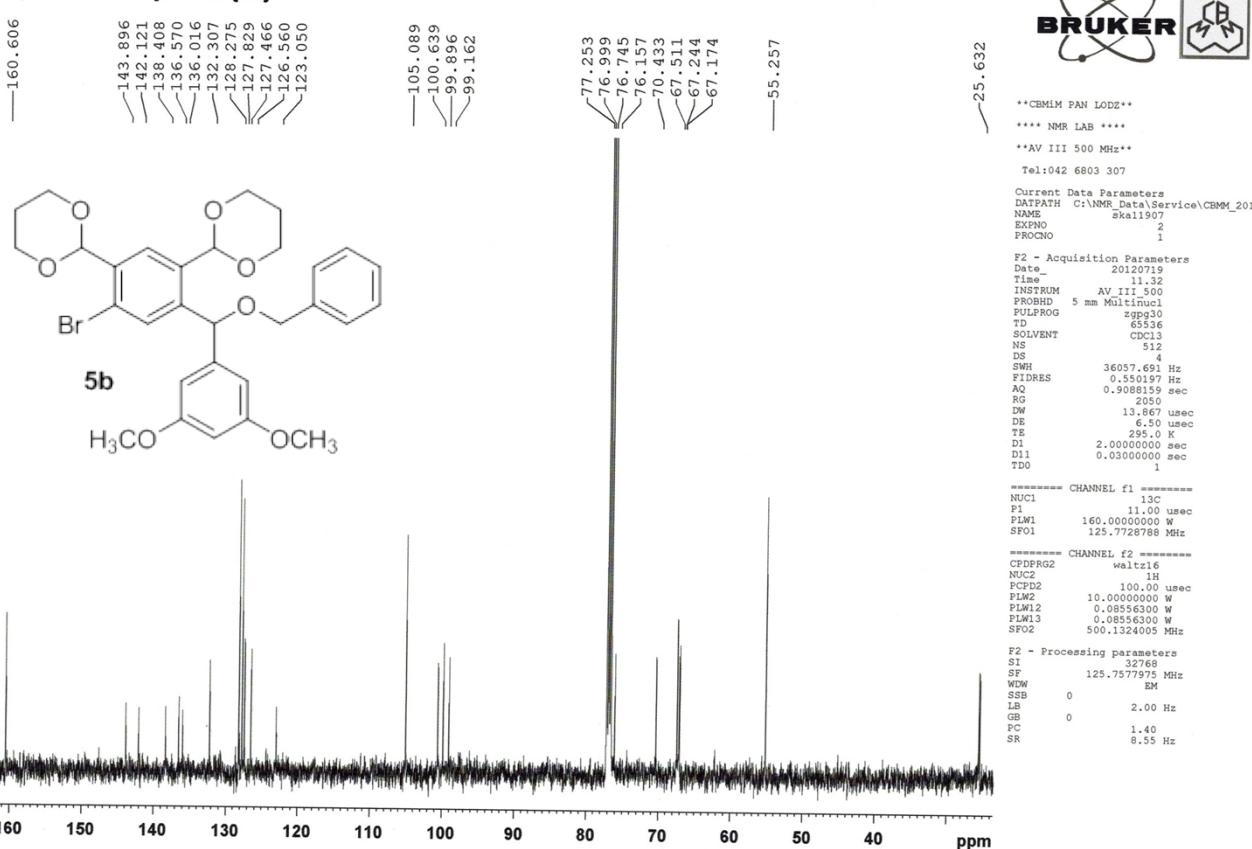


**194.JS**

7/18/2012 1:51:38 PM



j. skalik 194js 13C{1H}

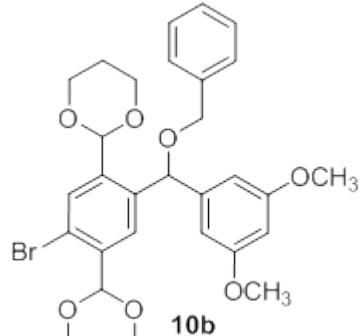
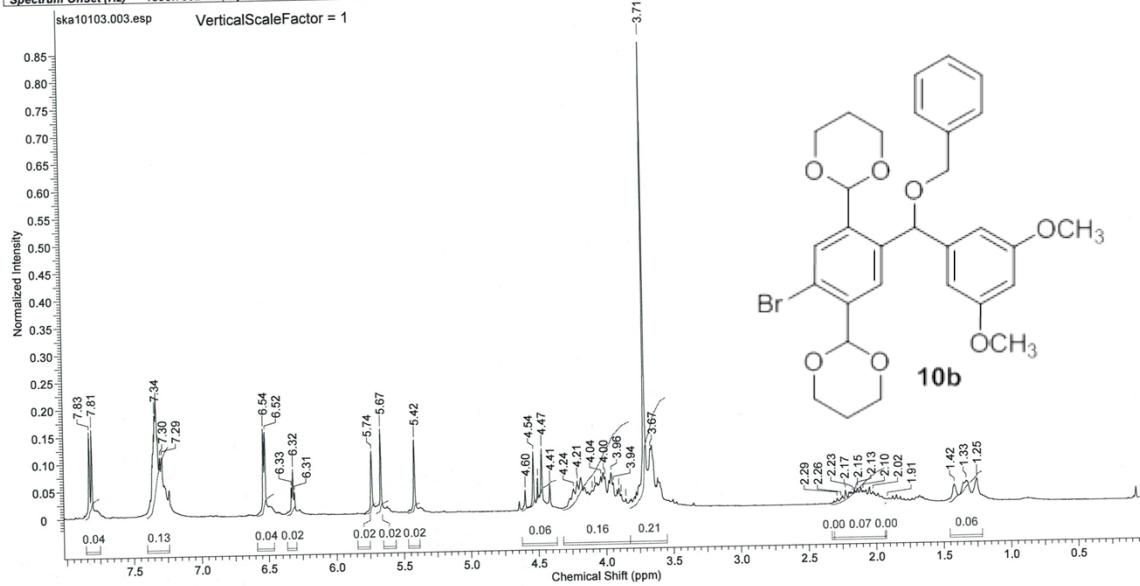


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## **benzylowany alkohol p-dimetoksy**

3/1/2013 9:29:12 AM  
2 JS para dimetoksy benzylowany alkohol

Acquisition Time (sec)	1.9792	Comment	JS para dimetoksy benzylowy alkohol	Date	01 Mar 2013 09:02:08	Frequency (MHz)	200.16
Date Stamp	01 Mar 2013 09:02:08	File Name	C:\Users\USER\Desktop\jska10103\3fid	Original Points Count	8192	Owner	root
Nucleus	1H	Number of Transients	64	Origin	spect	Solvent	CHLOROFORM-d
Points Count	8192	Pulse Sequence	zg30	Receiver Gain	322.50	SW(cyclical) (Hz)	4139.07
Spectrum Offset (Hz)	1696.7002	Spectrum Type	STANDARD	Sweep Width (Hz)	4138.57	Temperature (degree C)	18.500

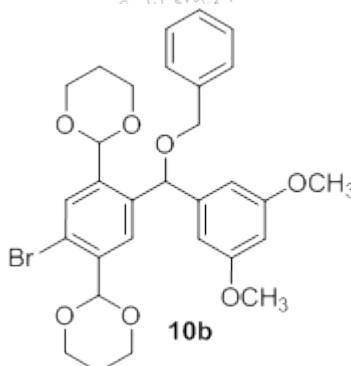
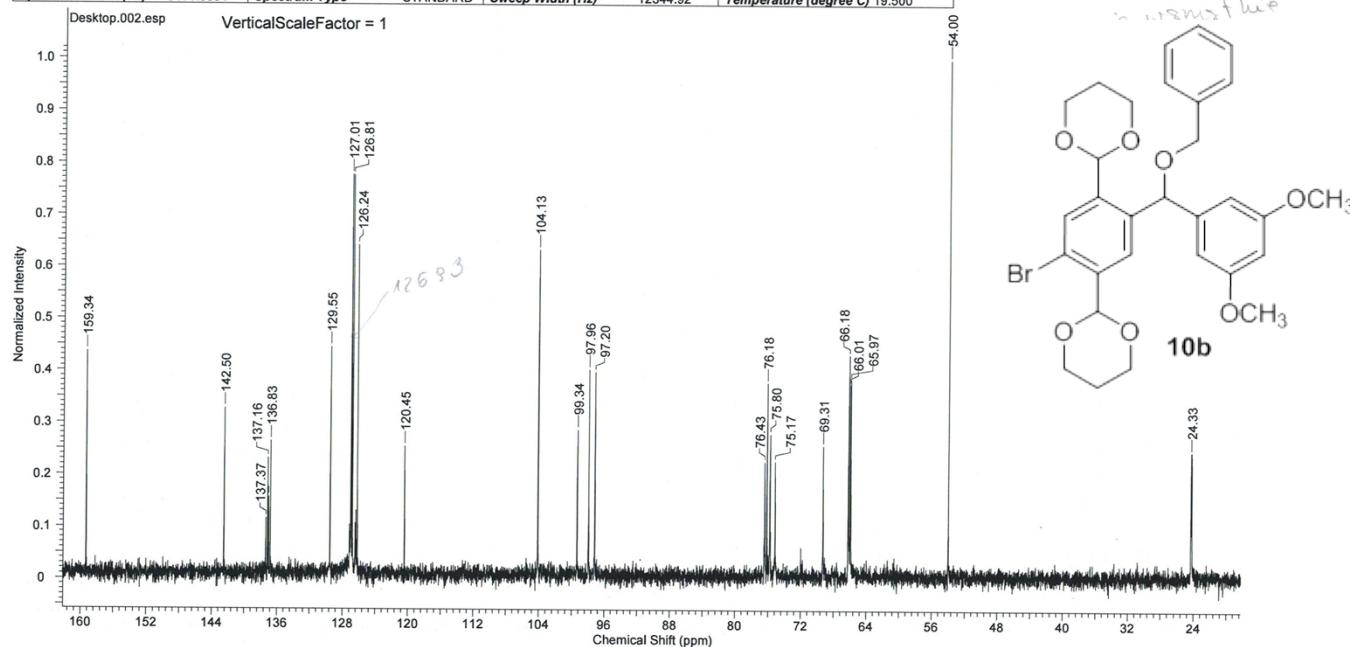


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### **benzylowany alkohol p-dimetoksy**

2/28/2013 4:06:44 PM  
2  
benzylowany p-dimetoksy

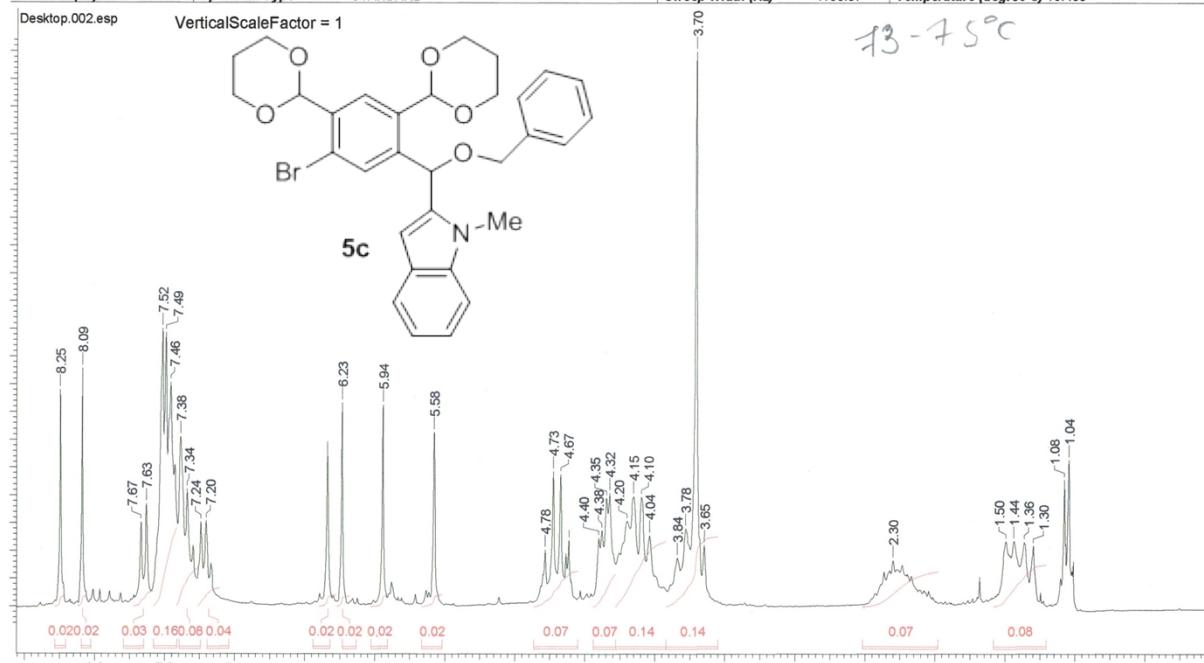
<b>Acquisition Time (sec)</b>	1.3271	<b>Comment</b>	benzylowany p-dimetoksy	<b>Date</b>	benzylowany p-dimetoksy	28 Feb 2013 15:58:08	
<b>Date Stamp</b>	28 Feb 2013 15:58:08	<b>File Name</b>	C:\Users\lUser\Desktop\2fid	<b>Frequency (MHz)</b>	50.33		
<b>Nucleus</b>	13C	<b>Number of Transients</b>	1024	<b>Origin</b>	spect		
<b>Points Count</b>	16384	<b>Pulse Sequence</b>	zgpg30	<b>Receiver Gain</b>	7298.20		
<b>Spectrum Offset (Hz)</b>	5471.0591	<b>Spectrum Type</b>	STANDARD	<b>Sweep Width (Hz)</b>	12344.92	<b>Temperature (degree C)</b>	19.500
<b>SW(cyclical) (Hz)</b>	12345.68	<b>Solvent</b>	CHLOROFORM-d				



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**benzylowany monoalkohol meta indole**

12/7/2011 9:59:28 AM

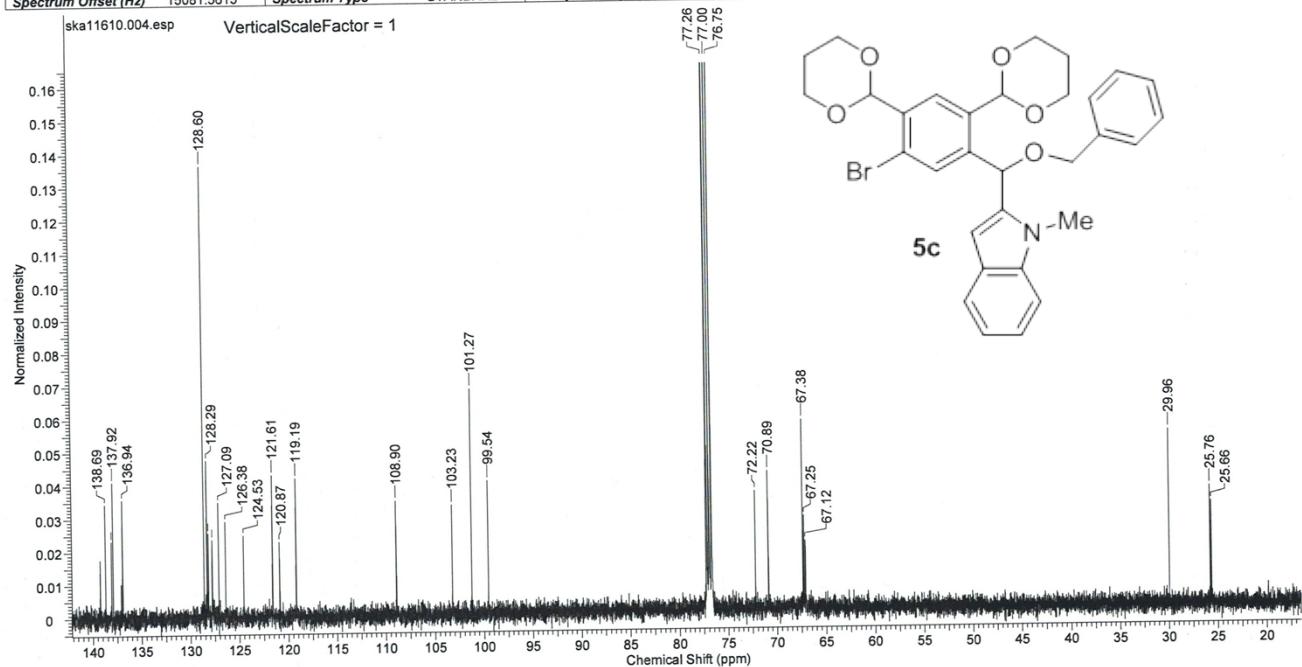
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Date Stamp	06 Dec 2011 14:26:24	File Name	C:\Users\Asia\Desktop\2\fid	Frequency (MHz)	200.16
Nucleus	1H	Number of Transients	32	Origin	spect
Original Points Count	8192	Pulse Sequence	zg30	Original Points Count	8192
Receiver Gain	22.60	SW(cyclical) (Hz)	4139.07	Owner	root
Spectrum Offset (Hz)	1696.7002	Spectrum Type	STANDARD	Solvent	CHLOROFORM-d
				Sweep Width (Hz)	4138.57
				Temperature (degree C)	19.400



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**m-IndOBn**

10/25/2013 2:46:46 PM  
j\_skalik =p-indobu /9/ = 13C{1H}

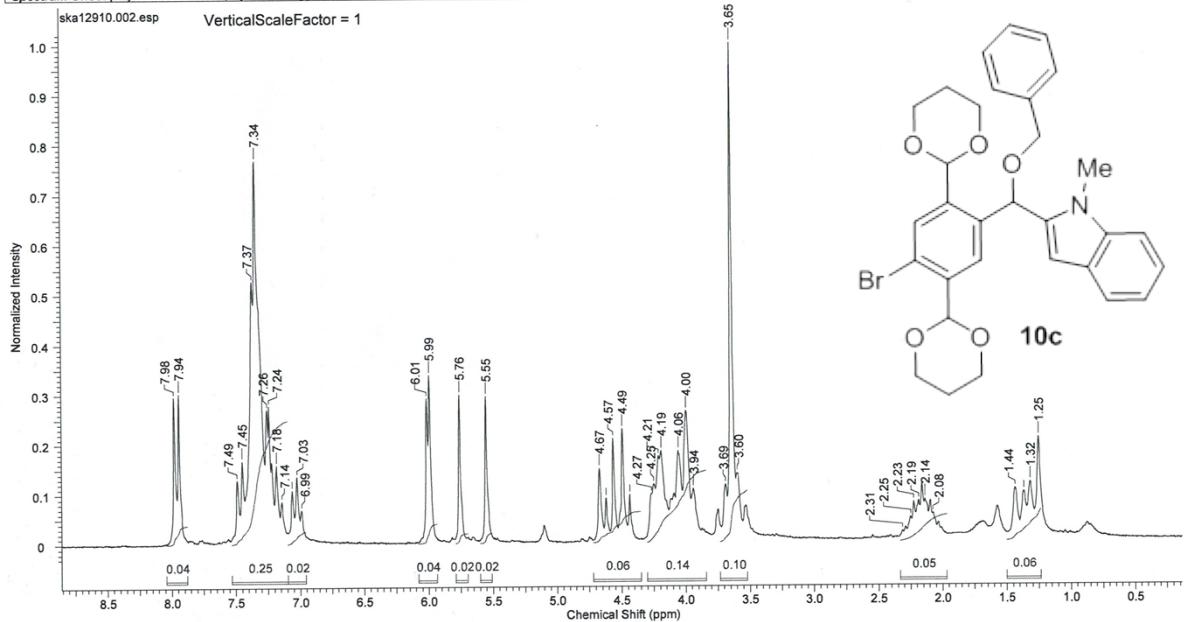
Acquisition Time (sec)	0.9088	Comment	j_skalik =p-indobu /9/ = 13C{1H}	Date	17 Oct 2013 03:10:24
Date Stamp	17 Oct 2013 03:10:24	File Name	C:\Users\User\Desktop\Nowy folder\skalik116104\fid	Frequency (MHz)	125.76
Frequency (MHz)	125.76	Nucleus	13C	Number of Transients	6776
Original Points Count	32768	Owner	nmrsu	Points Count	32768
Receiver Gain	2050.00	SW(cyclical) (Hz)	36057.69	Pulse Sequence	zgpg30
Spectrum Offset (Hz)	15081.5615	Spectrum Type	STANDARD	Solvent	CHLOROFORM-d
				Sweep Width (Hz)	36056.59
				Temperature (degree C)	22.001



### p-IndOBn

10/30/2013 11:23:12 AM  
Skalik J. p-IndOBn

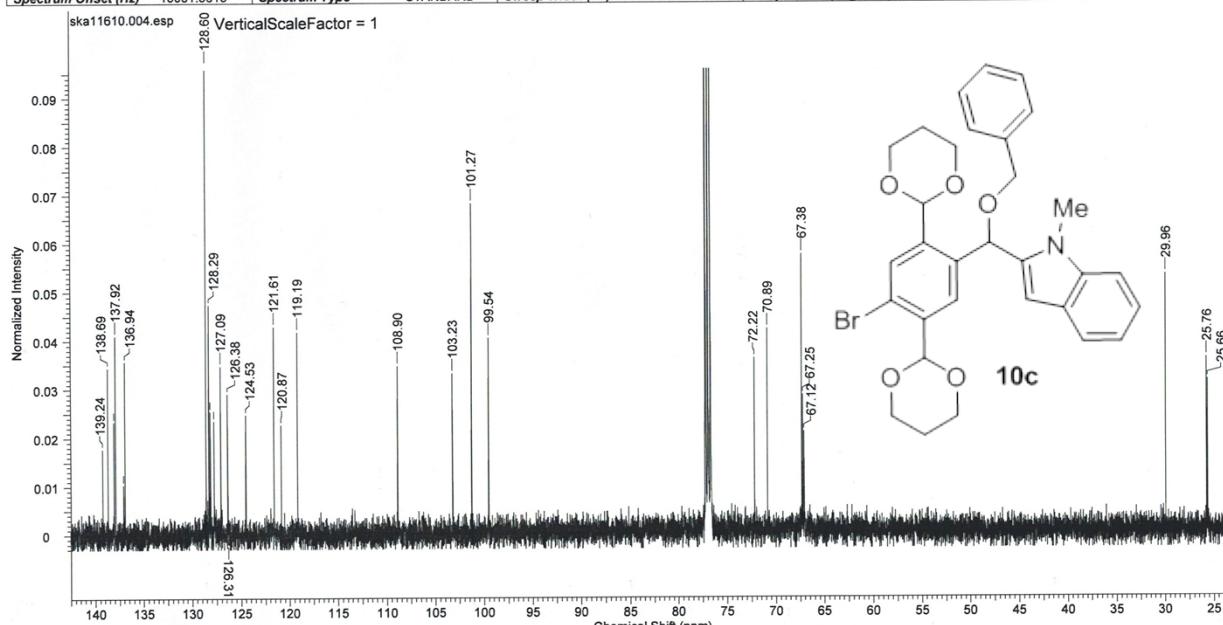
Acquisition Time (sec)	1.9792	Comment	Skalik J. p-IndOBn	Date	29 Oct 2013 15:49:36	Frequency (MHz)	200.16
Date Stamp	29 Oct 2013 15:49:36	File Name	C:\Users\User\Desktop\ska129102\fid	Origin	spect	Original Points Count	8192
Nucleus	1H	Number of Transients	64	Receiver Gain	645.10	SW(cyclical) (Hz)	4139.07
Points Count	8192	Pulse Sequence	zg30	Sweep Width (Hz)	4138.57	Temperature (degree C)	19.900
Spectrum Offset (Hz)	1696.7002	Spectrum Type	STANDARD	Vertical Scale Factor	1		

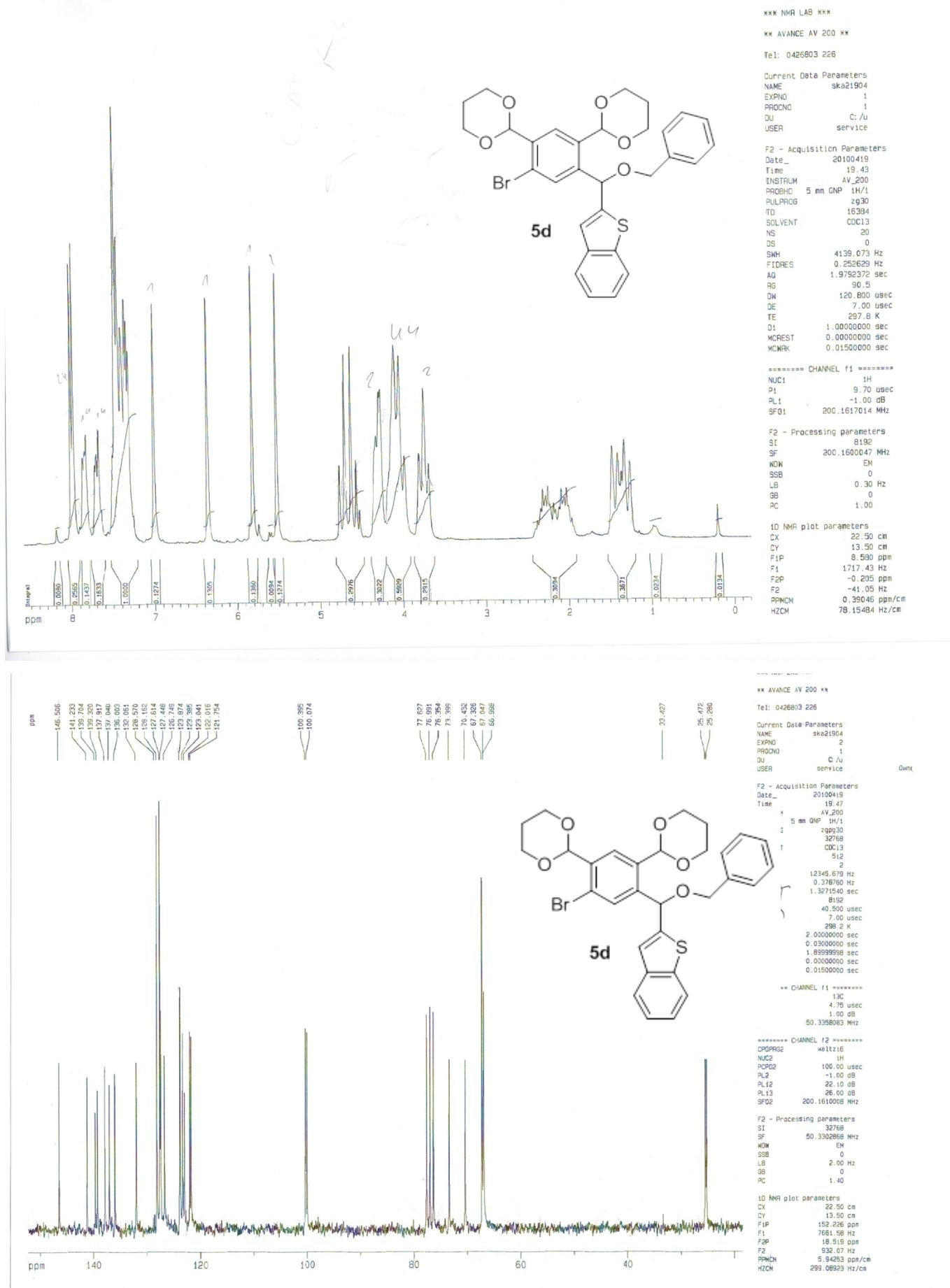


### p-IndOBn

10/30/2013 11:52:16 AM  
j. skalik =p-indobu /9/= 13C(1H)

Acquisition Time (sec)	0.9088	Comment	j. skalik =p-indobu /9/= 13C(1H)	Date	17 Oct 2013 03:10:24
Date Stamp	17 Oct 2013 03:10:24	File Name	C:\Users\User\Desktop\PRACA\ska116104\fid	Origin	AV_III_500
Frequency (MHz)	125.76	Nucleus	13C	Number of Transients	6776
Original Points Count	32768	Owner	rnmrsu	Points Count	32768
Receiver Gain	2050.00	SW(cyclical) (Hz)	36057.69	Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	15081.5615	Spectrum Type	STANDARD	Sweep Width (Hz)	36056.59
Vertical Scale Factor	1	Temperature (degree C)	22.001		

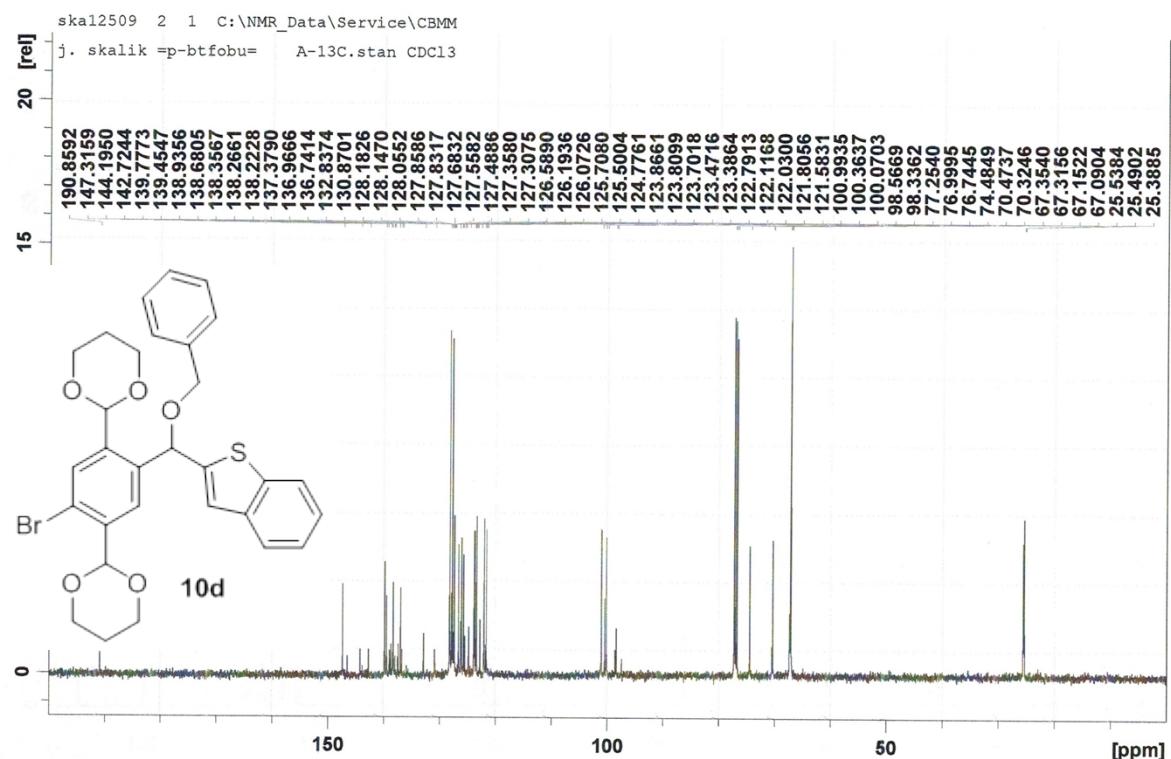
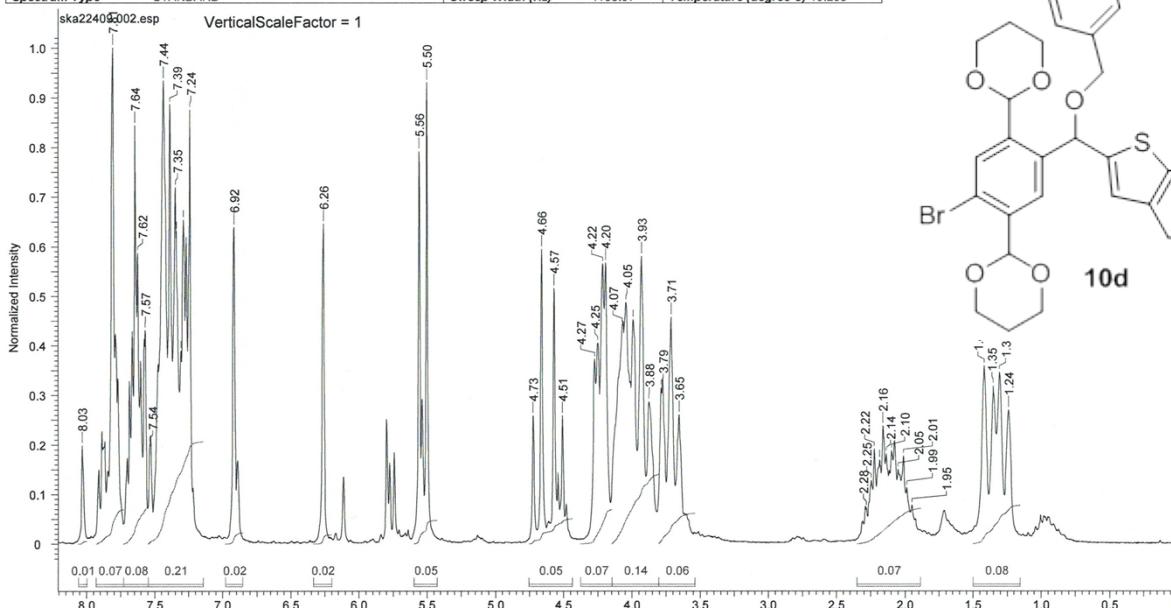




This report was created by ACD/NMR Processor Academic Edition. For more information go to [www.acdlabs.com/nmrproc/](http://www.acdlabs.com/nmrproc/)  
**p-BTFOBn**

9/24/2013 1:18:17 PM  
 p-BTFOBn

<b>Acquisition Time (sec)</b>	1.9792	<b>Comment</b>	p-BTFOBn	<b>Date</b>	24 Sep 2013 12:48:32	<b>Date Stamp</b>	24 Sep 2013 12:48:32
<b>File Name</b>	C:\Users\User\Desktop\sk22409\2\fid	<b>Frequency (MHz)</b>	200.16	<b>Nucleus</b>	1H	<b>Number of Transients</b>	32
<b>Origin</b>	spec1	<b>Original Points Count</b>	8192	<b>Owner</b>	root	<b>Points Count</b>	8192
<b>Receiver Gain</b>	181.00	<b>SW(cyclical) (Hz)</b>	4139.07	<b>Solvent</b>	CHLOROFORM-d	<b>Pulse</b>	
<b>Spectrum Type</b>	STANDARD	<b>Sweep Width (Hz)</b>	4138.57	<b>Temperature (degree C)</b>	19.200	<b>Spec</b>	

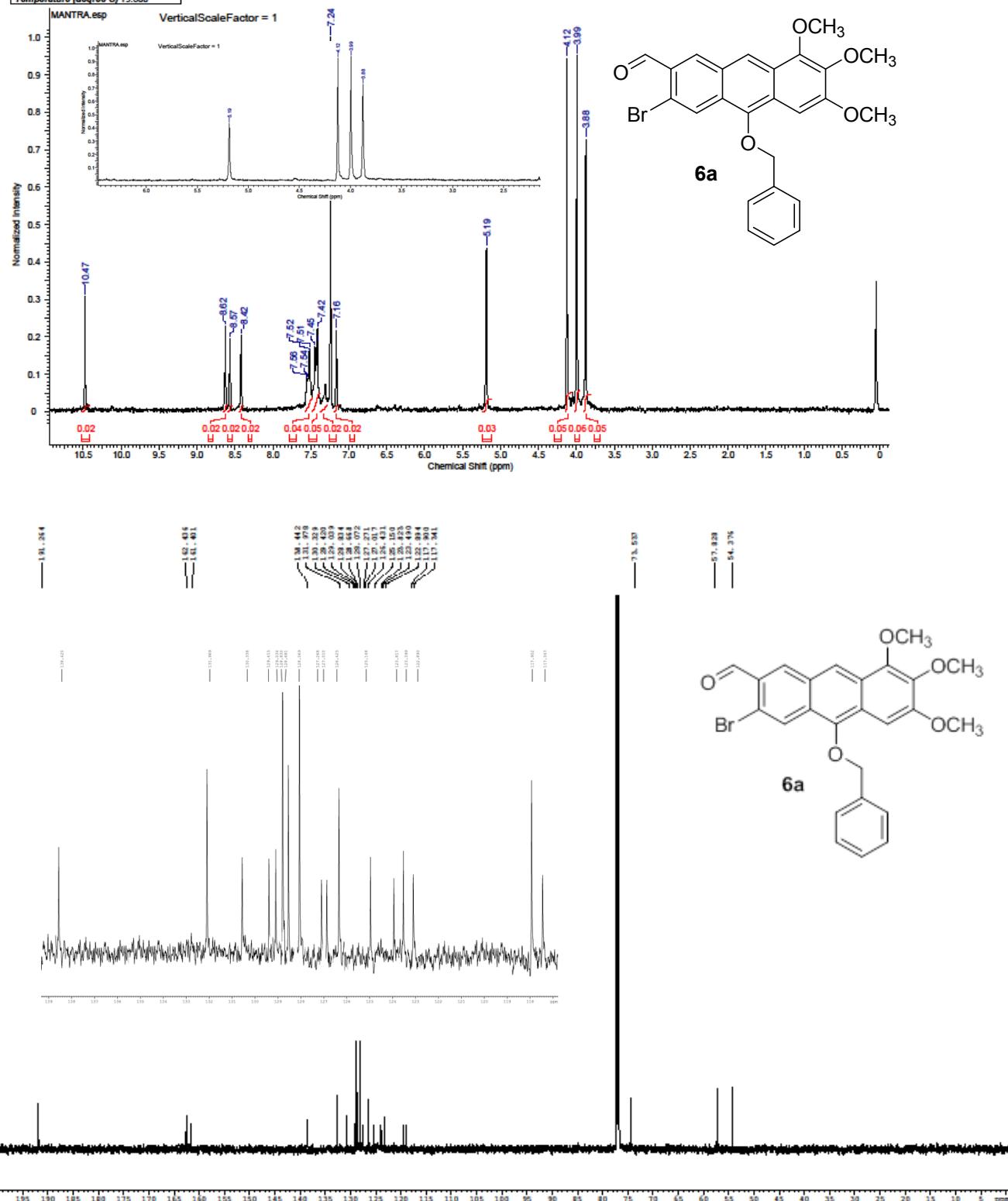


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## MANTRA

2/23/2015 5:23:28 PM

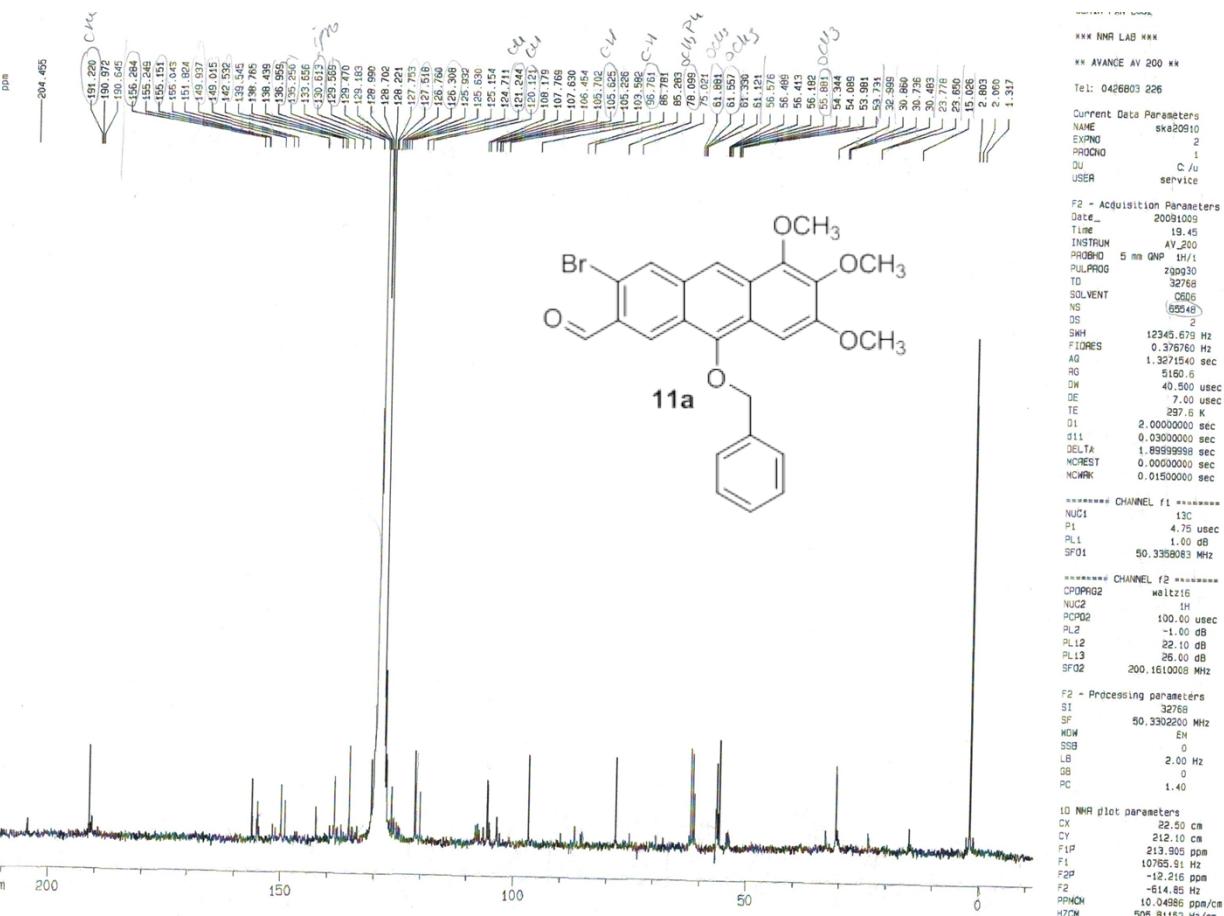
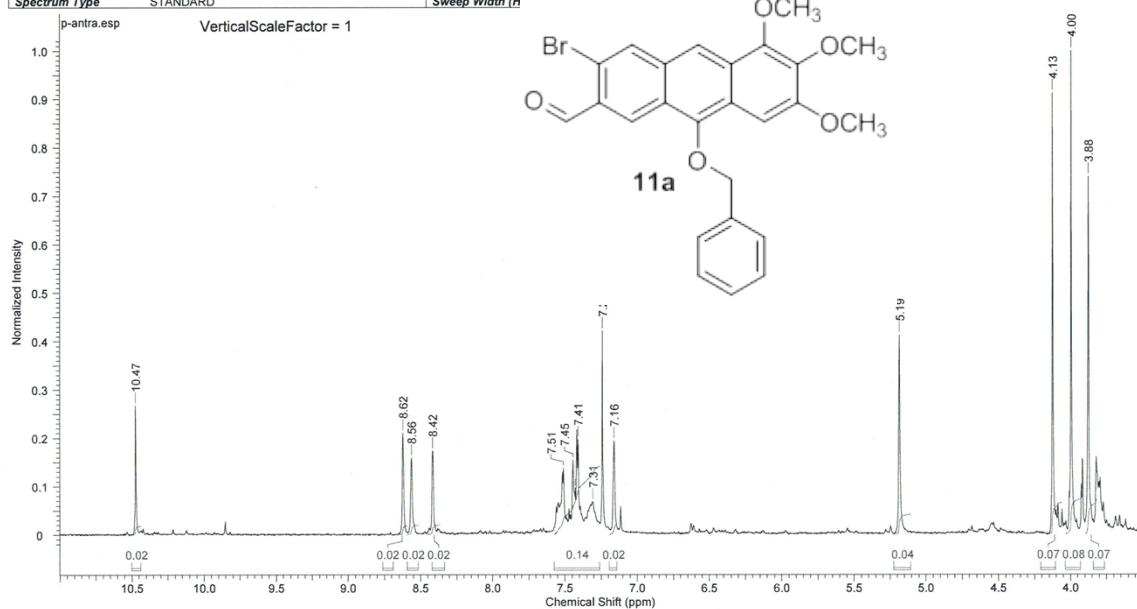
Acquisition Time (sec)	1.9792	Comment	MANTRA_2	Date	23 Jan 2015 15:49:36
Date Stamp	23 Jan 2015 15:49:36			File Name	C:\USERS\ASIA\Desktop\MISTI\POPRAWKI\MANTRA\FID
Frequency (MHz)	200.16	Nucleus	1H	Number of Transients	32
Owner	root	Points Count	8192	Origin	AV-200
Solvent	CHLOROFORM-d	Pulse Sequence	zg30	Receiver Gain	1824.60
		Spectrum Offset (Hz)	1696.7002	Original Points Count	8192
		Spectrum Type	STANDARD	SW(cyclical) (Hz)	4139.07
				Sweep Width (Hz)	4138.57
				Temperature (degree C)	19.500



**p-ANTRA**

10/11/2013 1:11:43 PM  
p-Antra

Acquisition Time (sec)	1.9792	Comment	p-Antra	Date	10 Oct 2013 15:13:36	Date Stamp	10 Oct 2013 15:13:36
File Name	C:\Users\User\Desktop\p-antralifd			Frequency (MHz)	200.16	Nucleus	1H
Origin	spect	Original Points Count	8192	Owner	root	Points Count	8192
Receiver Gain	1290.20	SW(cyclical) (Hz)	4139.07	Solvent	CHLOROFORM-d	Pulse Sequence	zg30
Spectrum Type	STANDARD			Sweep Width (Hz)		Spectrum Offset (Hz)	1696.7002

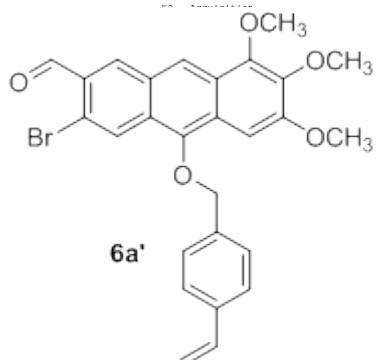
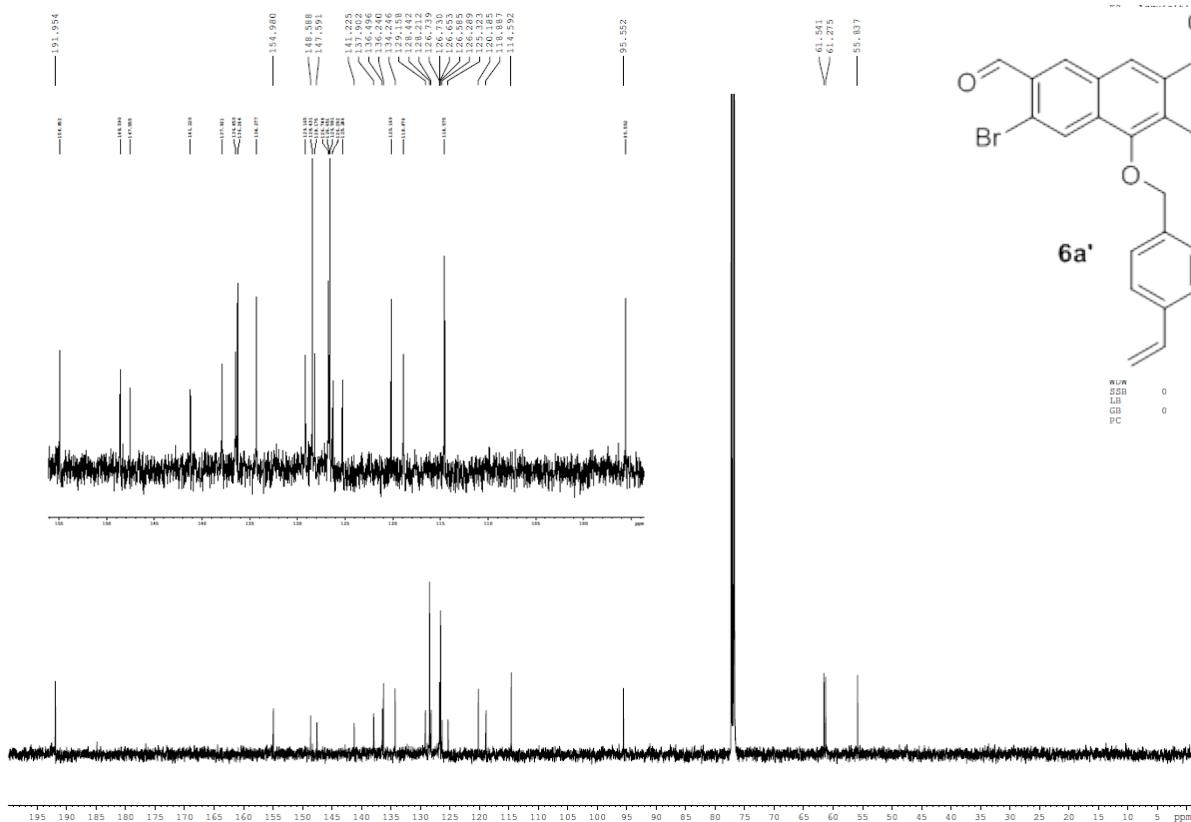
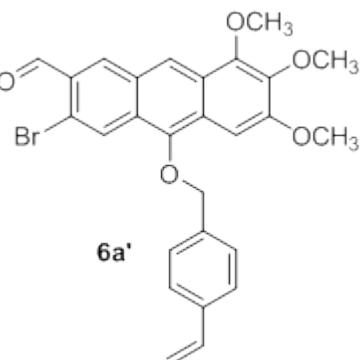
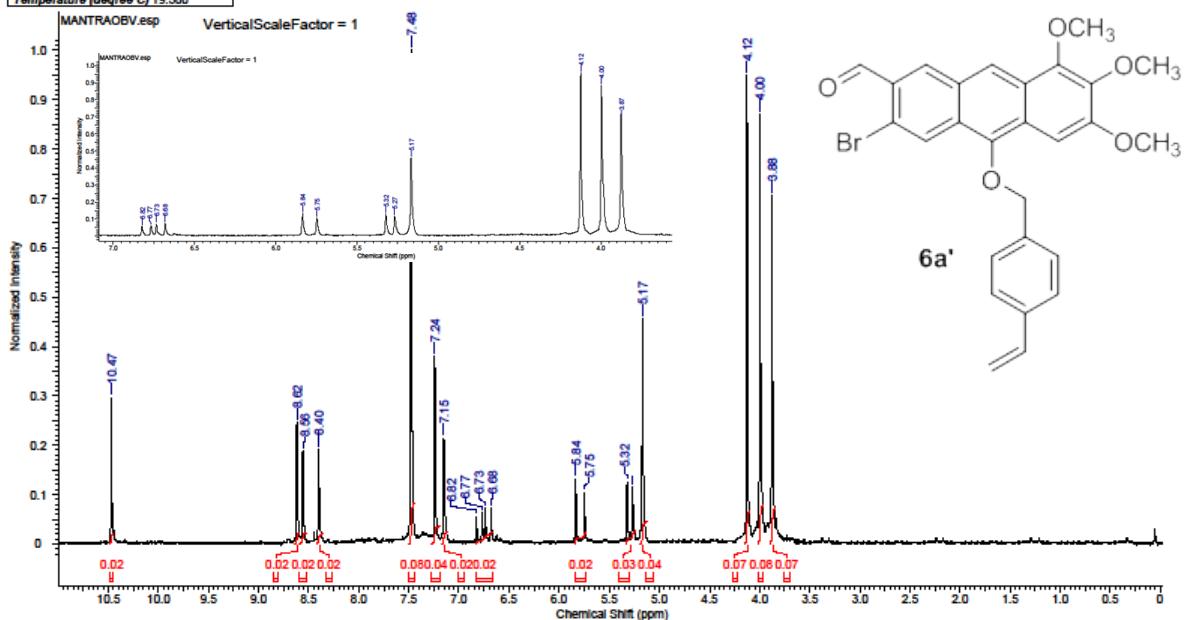


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MANTRA OBV

2/23/2015 5:54:53 PM

Acquisition Time (sec)	1.9792	Comment	Mantra0v2	Date	28 Jan 2015 16:36:32		
Date Stamp	28 Jan 2015 16:36:32			File Name	C:\USERS\ASIA\DESKTOP\TOPIMISTIPO\PRAWKIMANTRA0BV\FID		
Frequency (MHz)	200.16	Nucleus	1H	Number of Transients	32	Origin	
Owner	root	Points Count	8192	Pulse Sequence	zg30	Receiver Gain	1448.20
Solvent	CHLOROFORM-d			Spectrum Offset (Hz)	1656.7002	Spectrum Type	STANDARD
Temperature (degree C)	19.500					Sweep Width (Hz)	4138.57

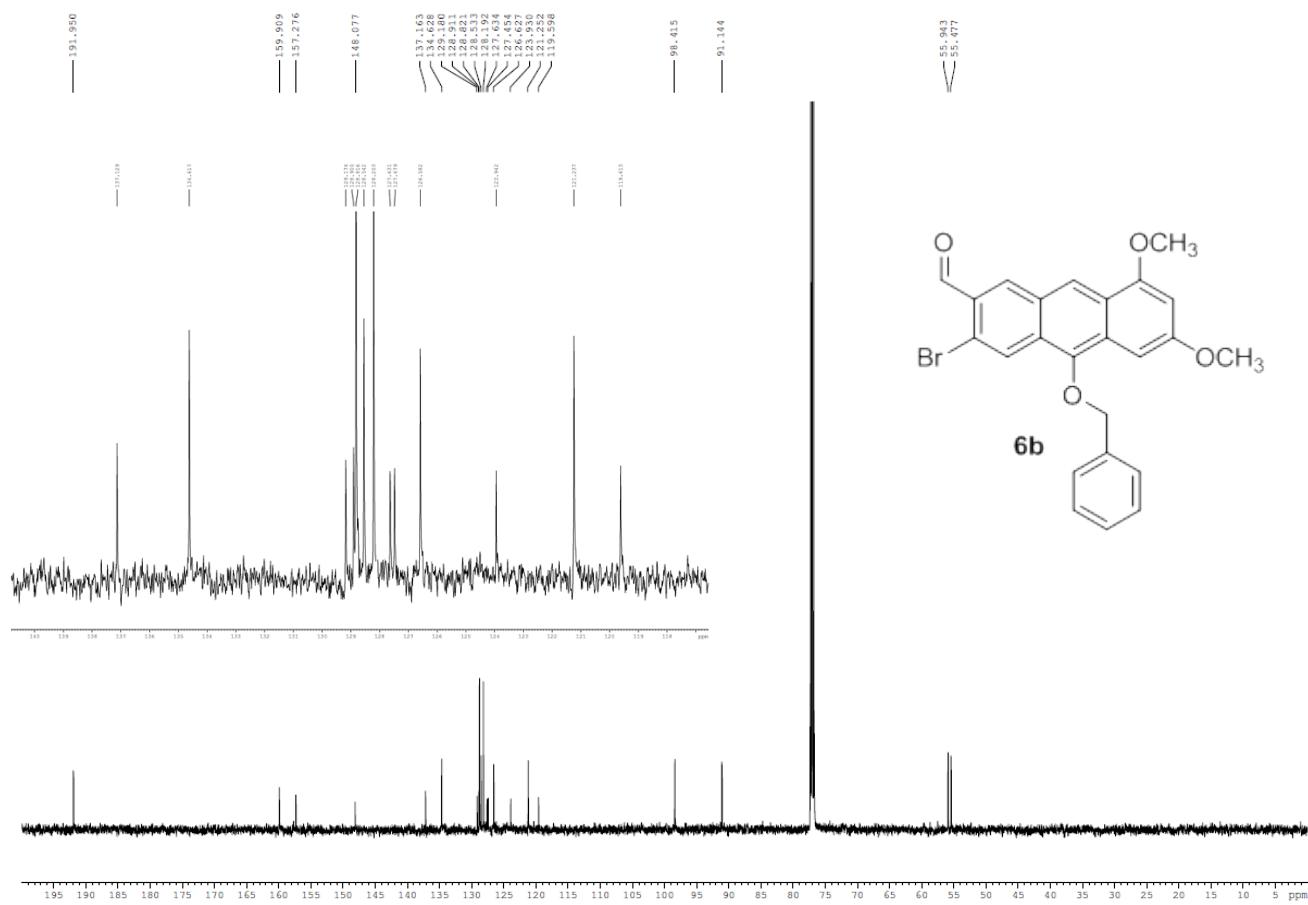
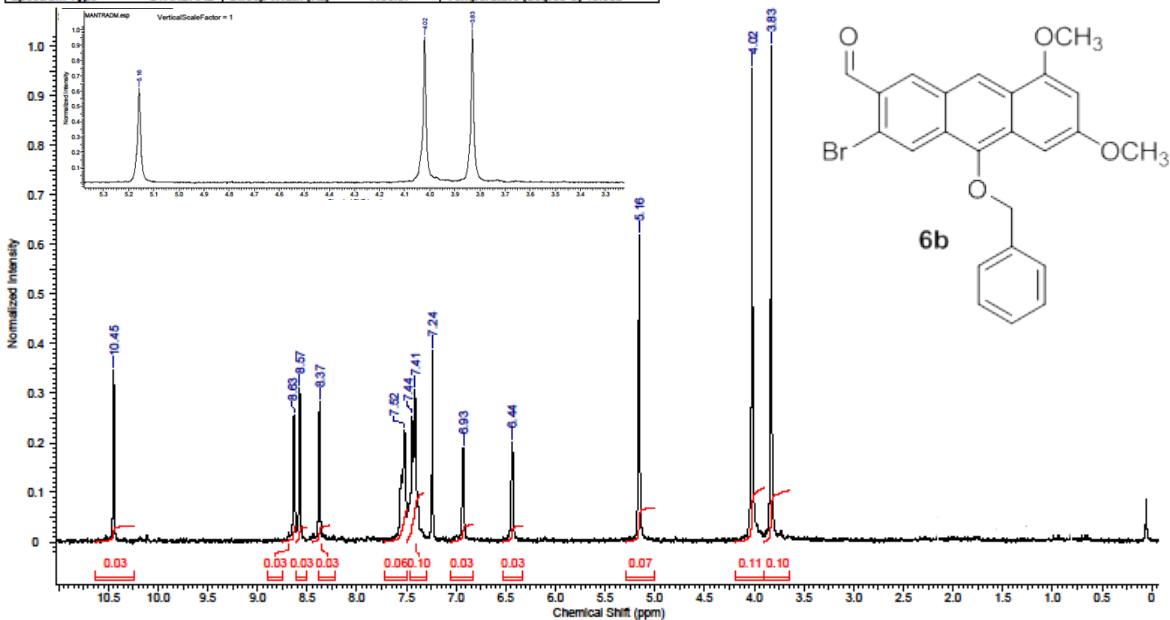


WJD  
SSB  
LB  
GB  
PC

# MANTRADM

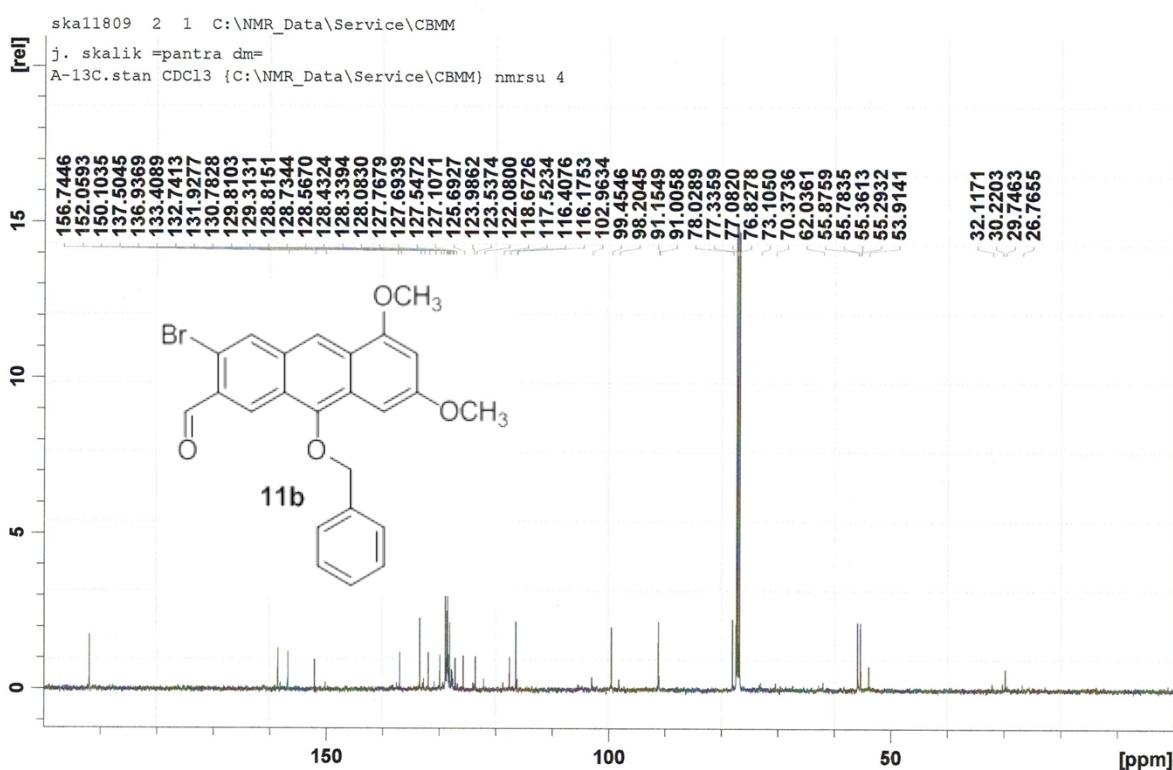
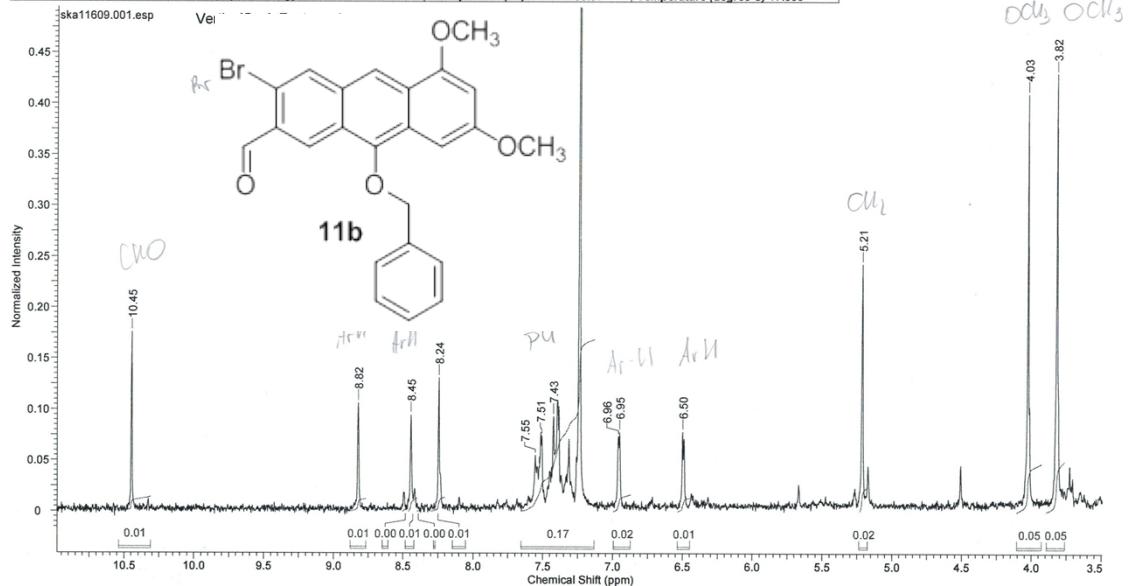
1/20/2015 3:50:15 PM  
3

Acquisition Time (sec)	1.9792	Comment	3	Date	16 Jan 2015 14:43:28	Date Stamp	16 Jan 2015 14:43:28
File Name	C:\USERS\USER\Desktop\NEW\SKA216013\FID	Frequency (MHz)	200.16	Nucleus	1H	Number of Transients	16
Origin	AV-200	Original Points Count	8192	Owner	root	Points Count	8192
Receiver Gain	1290.20	SW(cyclical) (Hz)	4139.07	Solvent	CHLOROFORM-d	Pulse Sequence	zg30
Spectrum Type	STANDARD	Sweep Width (Hz)	4138.57	Temperature (degree C)	19.500	Spectrum Offset (Hz)	1696.7002



9/16/2013 12:50:05 PM  
 cyklizacja p dimet

Acquisition Time (sec)	1.9792	Comment	cyklizacja p dimet	Date	16 Sep 2013 12:35:44
Date Stamp	16 Sep 2013 12:35:44	File Name	C:\Users\User\Desktop\skal11609\1fid	Frequency (MHz)	200.16
Nucleus	<sup>1</sup> H	Number of Transients	128	Origin	spect
Points Count	8192	Pulse Sequence	zg30	Original Points Count	8192
Spectrum Offset (Hz)	1696.7002	Receiver Gain	2298.80	SW(cyclical) (Hz)	4139.07
Spectrum Type	STANDARD	Sweep Width (Hz)	4138.57	Temperature (degree C)	17.300

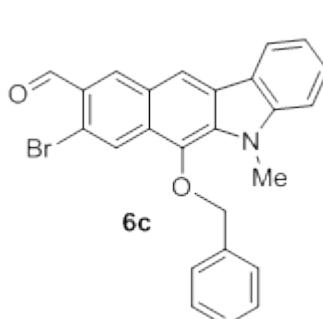
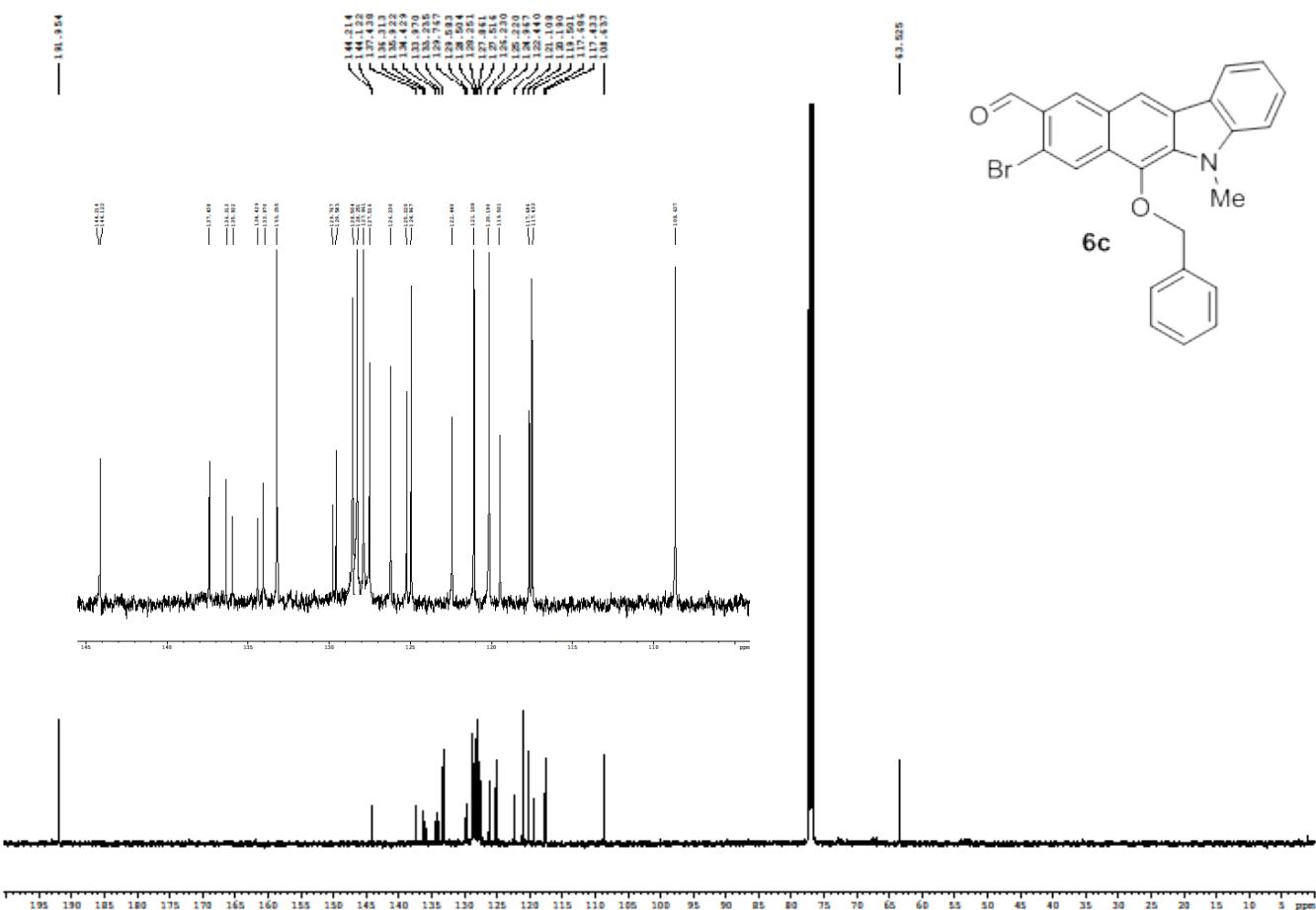
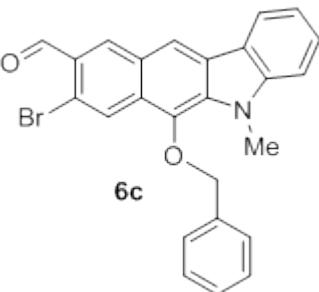
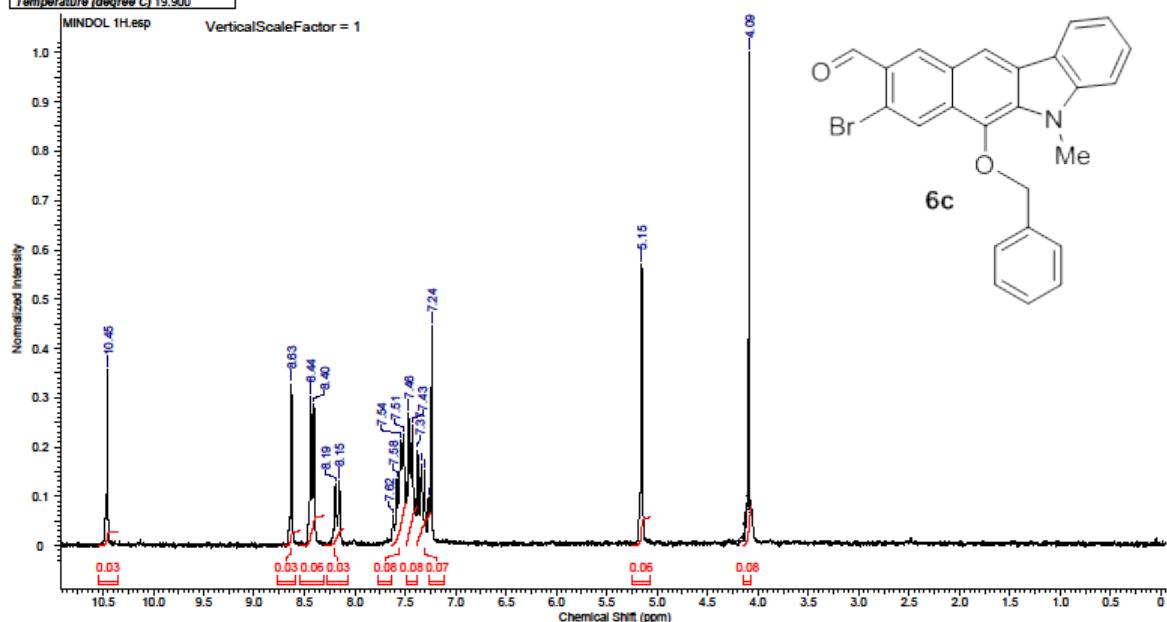


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MCARR

2/23/2015 5:01:19 PM

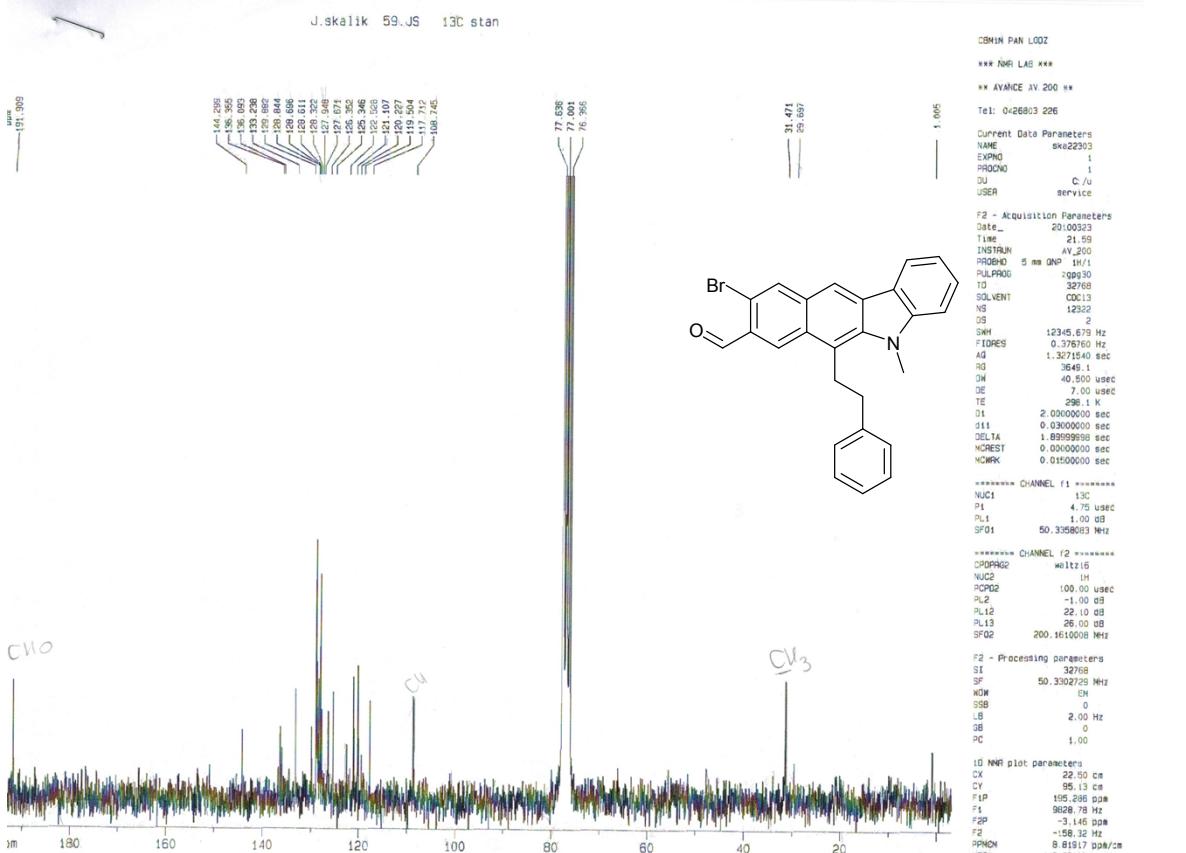
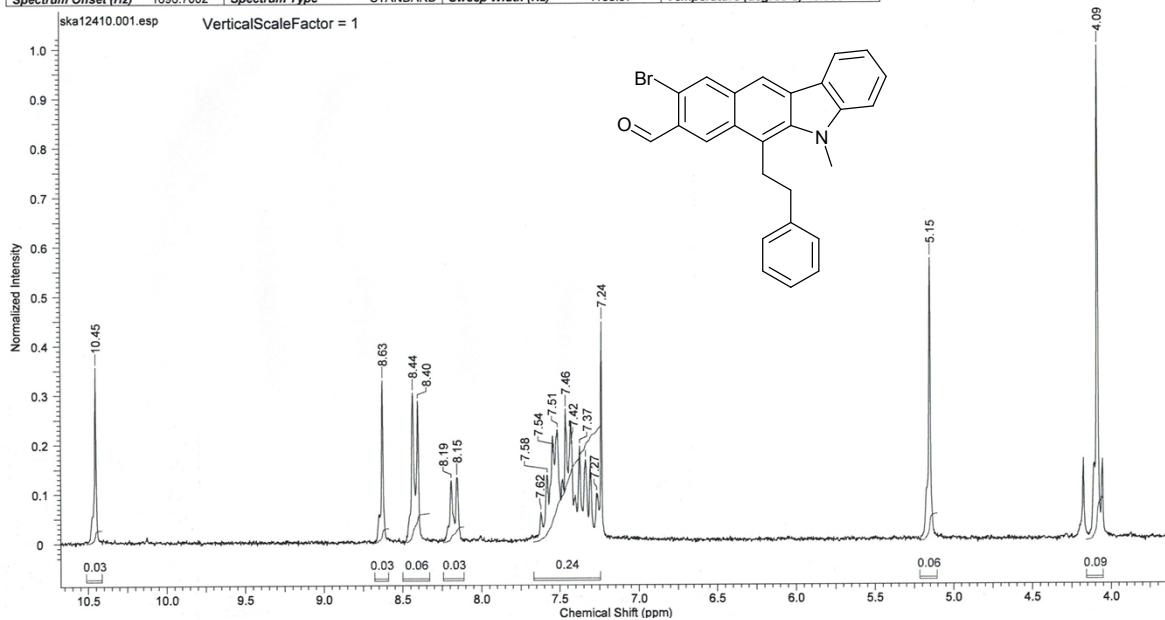
Acquisition Time (sec)	1.9792	Comment	J. Skalki	IND	Date	24 Oct 2014 13:54:40	2/23/2015 5:01:19 PM		
Date Stamp	24 Oct 2014 13:54:40				File Name	C:\USERS\ASIA\Desktop\MIST\PORAWKI\MINDOL 1H\FID			
Frequency (MHz)	200.16	Nucleus	1H	Number of Transients	32	Origin	spec	Original Points Count	8192
Owner	root	Points Count	8192	Pulse Sequence	zg30	Receiver Gain	1824.60	SW(cyclical) (Hz)	4139.07
Solvent	CHLOROFORM-d			Spectrum Offset (Hz)	1696.7002	Spectrum Type	STANDARD	Sweep Width (Hz)	4138.57
Total Transients	8192								



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**p-IND**

10/25/2013 9:50:29 AM  
J. Skalik p-IND

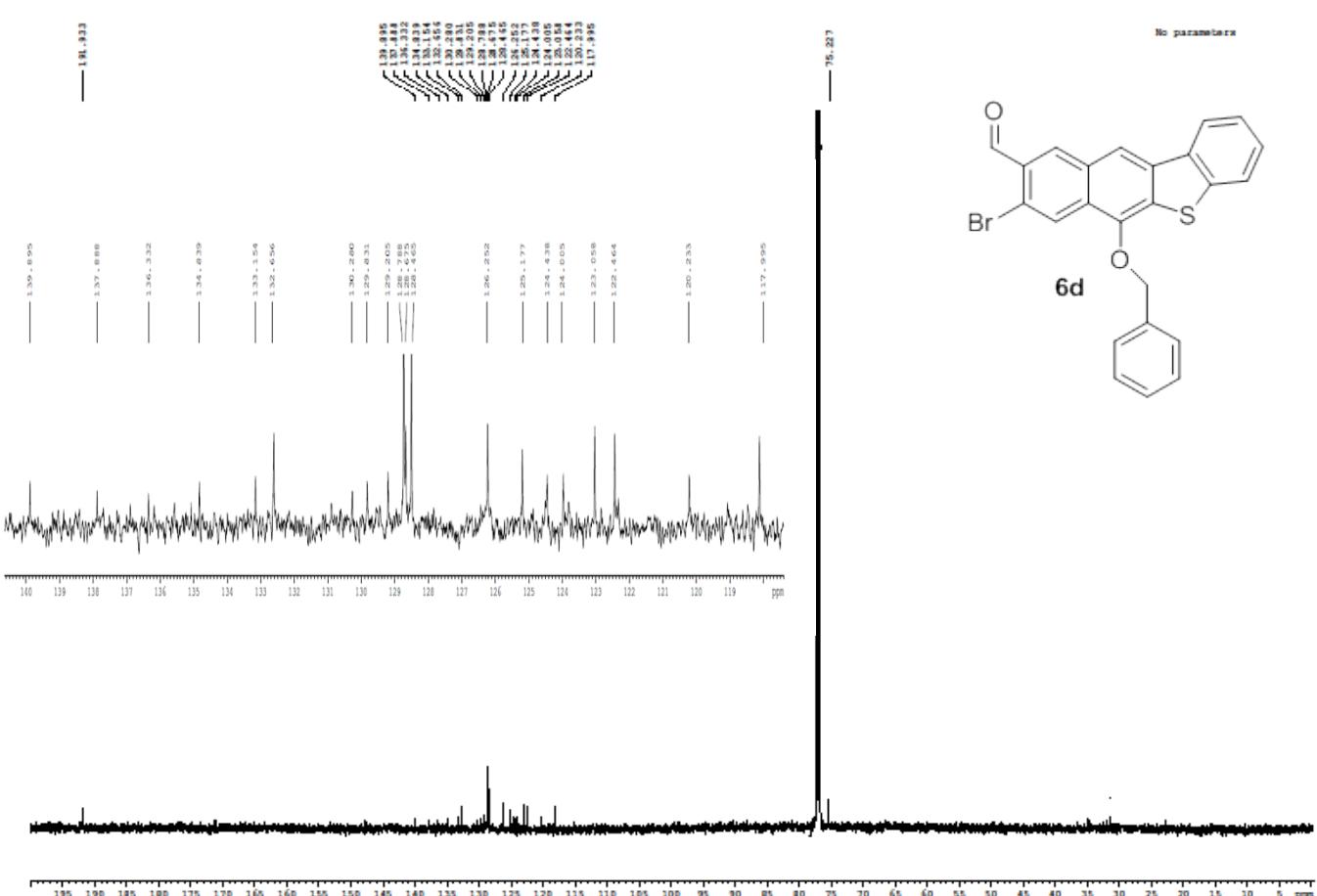
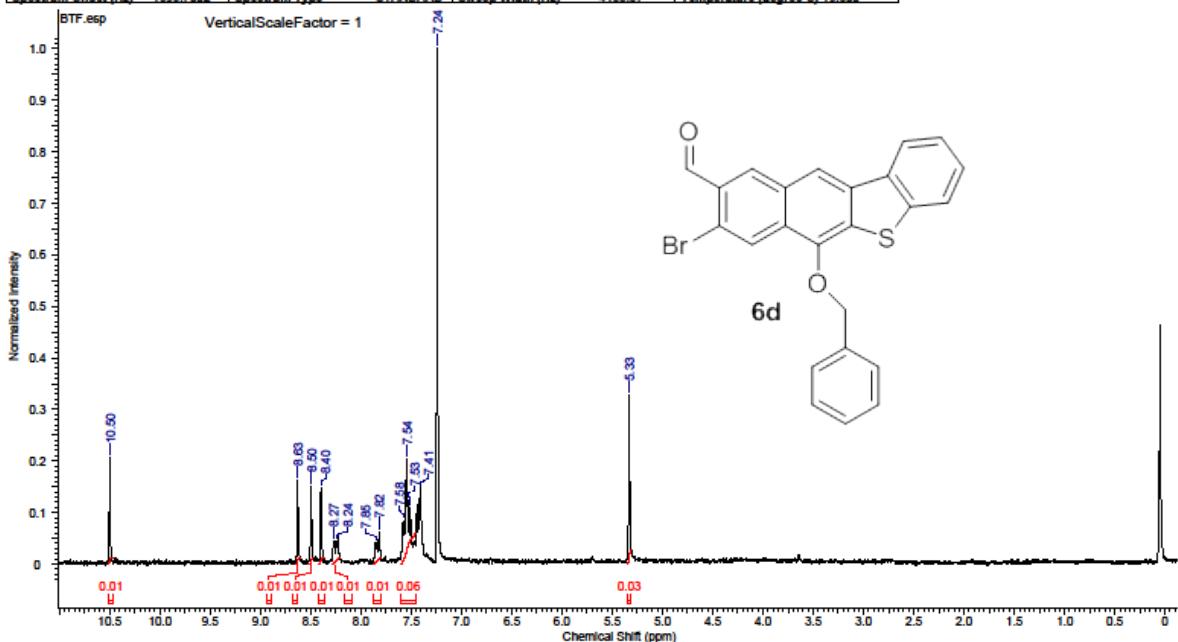
Acquisition Time (sec)	1.9792	Comment	J. Skalik p-IND	Date	24 Oct 2013 13:54:40	Frequency (MHz)	200.16
Date Stamp	24 Oct 2013 13:54:40 <th>File Name</th> <td>C:\Users\User\Desktop\skal124101\fid<th>Origin</th><td>spect</td><th>Original Points Count</th><td>8192</td></td>	File Name	C:\Users\User\Desktop\skal124101\fid <th>Origin</th> <td>spect</td> <th>Original Points Count</th> <td>8192</td>	Origin	spect	Original Points Count	8192
Nucleus	1H	Number of Transients	32	Receiver Gain	1824.60	Owner	root
Points Count	8192	Pulse Sequence	zg30	Sweep (cyclicall) (Hz)	4139.07	Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	1699.7002 <th>Spectrum Type</th> <td>STANDARD</td> <th>Sweep Width (Hz)</th> <td>4138.57<th>Temperature (degree C)</th><td>19.900</td></td>	Spectrum Type	STANDARD	Sweep Width (Hz)	4138.57 <th>Temperature (degree C)</th> <td>19.900</td>	Temperature (degree C)	19.900



**MBTF**

2/23/2015 5:14:37 PM

Acquisition Time (sec)	1.9732	Comments	b1F	Date	20 Feb 2015 14:13:36	Date Stamp	20 Feb 2015 14:13:36	
File Name	C:\USERS\ASIA\Desktop\PIMIST\POPRAWKI\BT\FID				Frequency (MHz)	200.16	Nucleus	1H
Number of Transients	64	Origin	AV-200	Original Points Count	8192	Owner	root	
Pulse Sequence	zq30	Receiver Gain	2048.00	SW(cyclical) (Hz)	4139.07	Solvent	CHLOROFORM-d	
Spectrum Offset (Hz)	1696.7002	Spectrum Type	STANDARD	Sweep Width (Hz)	4138.57	Temperature (degree C)	19.300	

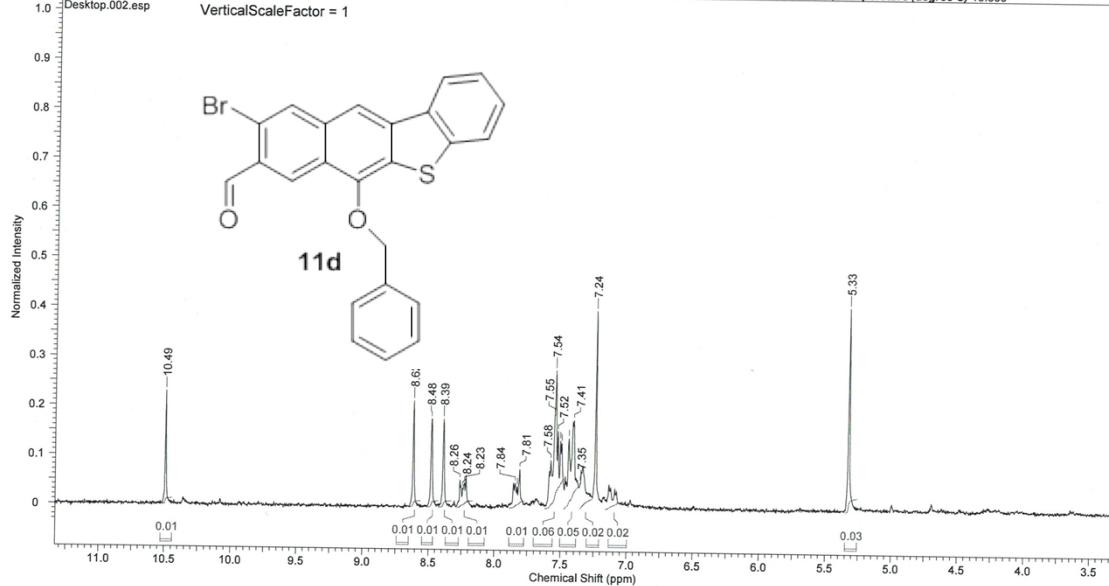


This report was created by ACD/NMR Processor Academic Edition. For more information go to [www.acdlabs.com/nmrproc/](http://www.acdlabs.com/nmrproc/).

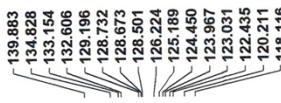
p-BTF

10/2/2013 9:14:51 AM  
Skalik J. p-BTF

Skalik J. p-BTF				Skalik J. p-BTF		
Acquisition Time (sec)	1.9792	Comment	Skalik J. p-BTF	Date	02 Oct 2013 08:51:44	
Date Stamp	02 Oct 2013 08:51:44	File Name	C:\Users\User\Desktop\2\fid <th>Frequency (MHz)</th> <td data-cs="2" data-kind="parent">200.16</td> <td data-kind="ghost"></td>	Frequency (MHz)	200.16	
Nucleus	1H	Number of Transients	32	Origin	spect	Owner
Points Count	8192	Pulse Sequence	zg30	Receiver Gain	1625.50	Solvent
Spectrum Offset (Hz)	1696.7002	Spectrum Type	STANDARD	SW(cyclical) (Hz)	4139.07	CHLOROFORM-d
				Sweep Width (Hz)	4138.57	Temperature (degree C)
						18.800



i. skalik =p-btf= 13C{1H}



**BRUKER** 

\*\*CBMiM PAN LODZ\*\*

\*\*\* NMR LAB \*\*\*

**\*\*AV III 500 MHz\*\***

Tel:042 6803 307

### Current Data Param

DATPATH C:\NMR\_Data\Service\CBMM  
NAME ska10210  
EXPNO 2  
PROCNO 1

```

F2 - Acquisition Parameters
Date       20131002
Time       9.50
INSTRUM   AV_1500
PROBHD   5 mm TXI1 PZ
PLPROG   zap030
TD        65536
SOLVENT   CDCl3
NS        3072
SWH      3605761.691 Hz
FIDRES   0.0550197 Hz
AQ        0.9087659 sec
RG        2050
DW        13.867 usec
DE        6.50 usec
TE        295.0 K
D1        2.0000000 sec
D11       0.03000000 sec
T90       6.

```

===== CHANNEL 11 ======  
SFO1 125.7728788 MHz  
NUC1 13C  
P1 11.50 usec  
PLW1 226.00000000 W

```
===== CHANNEL f2 =====
SFO2      500.1324005 MHz
NUC2      1H
CPDPRG[2] waltz16
PCPD2     100.00 usec
PLW2      9.00000000 W
PLW12     0.08398400 W
PLW13     0.08398400 W
```

F2 - Processing parameters  
 SI 32768  
 SF 125.7577956 MHz  
 WDW EM  
 SSB 0  
 LB 2.00 Hz  
 GB 0  
 PC 1.40  
 SR 6.61 Hz

