

SUPPORTING INFORMATION

Palladium-Mediated Three-Component Synthesis of Furo[2,3-b]pyridones by One-Pot Coupling of 3-Iodopyridones, Alkynes, and Organic Halides.

Emmanuel Bossharth, Philippe Desbordes, Nuno Monteiro,
and Geneviève Balme.

General remarks: Unless otherwise noted, all reactions were carried out under a nitrogen atmosphere using standard syringe, cannula, and septa techniques. Commercially available reagents were used as purchased. THF and MeCN were distilled over calcium hydride. TLC was carried out on Merck silica 60/F-254 aluminium-backed plates. Flash chromatography was performed using Merck silica gel 60 (40-63 µm). Melting points were determined on a Büchi and are uncorrected. NMR spectra were recorded either on a Brucker ALS 300 or a Brucker AMS 200 spectrometers in the solvent indicated. Chemical shifts δ are reported in ppm relative to the internal reference. High resolution mass spectra were obtained on a ThermoFinnigan LCQ-advantage or on a ThermoFinnigan MAT95XL.

Iodination, general procedure: The 4-alkoxy-2-pyridone derivative (1.0 mmol) and N-iodosuccinimide (400 mg, 1.8 mmol) were dissolved in MeCN (20 mL). The reaction mixture was left to stir for 18h at room temperature and then concentrated *in vacuo*. The residue was purified by column chromatography (silica gel, AcOEt/Petroleum Ether 50/50) to afford the corresponding 3-iodo-2-pyridone.

4-Benzyl-3-iodo-1-methyl-2-pyridone (2a): White solid, Mol. Wt.: 341.1 g/mol. Mp: 103°C (decomp.). ^1H NMR (300 MHz, CDCl_3): δ 3.56 (s, 3H), 5.22 (s, 2H), 5.98 (d, $J = 7.5$ Hz, 1H), 7.26 (d, $J = 7.5$ Hz, 1H), 7.30-7.45 (m, 5H). ^{13}C NMR (75 MHz, CDCl_3): δ 38.80; 71.53; 95.79; 127.18; 128.66; 129.12; 135.81; 139.35; 161.82; 167.01. HRMS (CI): MH^+ , 341.9995; Calc. for $\text{C}_{13}\text{H}_{13}\text{INO}_2^+$: 341.9991.

4-Methoxy-3-iodo-1-methyl-2-pyridone (2b): Light yellow solid, Mol. Wt.: 265.0 g/mol. Mp: 108°C. ^1H NMR (300 MHz, CDCl_3): δ 3.60 (s, 3H), 3.93 (s, 3H), 6.01 (d, $J = 7.5$ Hz, 1H), 7.32 (d, $J = 7.5$ Hz, 1H). ^{13}C NMR (75 MHz, CDCl_3): δ 38.88; 57.30; 76.01; 94.44; 139.29; 161.79; 167.88. HRMS (CI): MH^+ , 265.9675; Calc. for $\text{C}_7\text{H}_9\text{INO}_2^+$: 265.9678.

1-Benzyl-4-benzyl-3-iodo-2-pyridone (2c): Yellow solid, Mol. Wt.: 417.2 g/mol. Mp: 119°C. ^1H NMR (300 MHz, CDCl_3): δ 5.13 (s, 2H), 5.19 (s, 2H), 6.01 (d, $J = 7.5$ Hz, 1H), 7.20-7.45 (m, 11H). ^{13}C NMR (50 MHz, CDCl_3): δ 53.08; 71.27; 76.74; 95.99; 127.02; 128.57, 128.70; 128.80; 129.16; 129.31; 135.49; 136.24; 138.40; 161.14; 166.61. HRMS (CI): MH^+ , 418.0303; Calc. for $\text{C}_{19}\text{H}_{17}\text{INO}_2^+$: 418.0304.

Sonogashira reaction, general procedure: The iodopyridone derivative (0.6 mmol), the terminal alkyne (0.8 mmol), $\text{Pd}(\text{PPh}_3)_2\text{Cl}_2$ (21 mg, 0.03 mmol) and CuI (5 mg, 0.03 mmol) were dissolved in a mixture of MeCN (4 mL) and TEA (0.5 mL). The reaction mixture was left to stir at 60°C for 24h and then concentrated *in vacuo*. The residue was purified by column chromatography (silica gel, ethyl acetate) to afford the corresponding 3-alkynyl-2-pyridone.

4-(4-Benzyl-1-methyl-2-oxo-1,2-dihydro-pyridin-3-ylethynyl)-benzoic acid methyl ester (4a): White solid, Mol. Wt.: 373.4 g/mol. Mp: 165°C. ¹H NMR (300 MHz, CDCl₃): δ 3.56 (s, 3H), 3.93 (s, 3H), 5.29 (s, 2H), 6.08 (d, J = 7.5 Hz, 1H), 7.30 (d, J = 7.6 Hz, 1H), 7.45 (m, 5H), 7.59 (d, J = 8.3 Hz, 2H), 8.00 (d, J = 8.3 Hz, 2H). ¹³C NMR (75 MHz, CDCl₃): δ 38.01; 52.54; 71.23; 85.85; 96.03; 98.22; 99.72; 127.16; 128.73; 129.12; 129.16; 129.37; 129.74; 131.79; 136.01; 139.33; 163.29; 167.09; 168.72. HRMS (CI): MH⁺, 374.1316; Calc. for C₂₃H₂₀NO₄⁺: 374.1314.

4-Benzyl-1-methyl-3-phenylethynyl-2-pyridone (4b): White solid, Mol. Wt.: 315.4 g/mol. Mp: 149.5°C. ¹H NMR (300 MHz, CDCl₃): δ 3.55 (s, 3H), 5.29 (s, 3H), 6.07 (d, J = 7.7 Hz, 1H), 7.24 (d, J = 7.7 Hz, 1H), 7.30-7.50 (m, 10H). ¹³C NMR (75 MHz, CDCl₃): δ 37.87; 71.04; 82.97; 96.13; 98.71; 99.65; 124.40; 127.17; 128.31; 128.56; 128.61; 129.02; 131.88; 136.19; 139.37; 163.39; 168.46. HRMS (CI): MH⁺, 316.1339; Calc. for C₂₁H₁₈NO₂⁺: 316.1338.

4-benzyl-3-hexynyl-1-methyl-2-pyridone (4c): Yellow oil, Mol. Wt.: 295.4 g/mol. ¹H NMR (300 MHz, CDCl₃): δ 0.89 (t, J = 7.3 Hz, 3H), 1.48 (m, 2H), 1.60 (m, 2H), 2.51 (t, J = 6.8 Hz, 2H), 3.49 (s, 3H), 5.21 (s, 2H), 5.98 (d, J = 7.6 Hz, 1H), 7.16 (d, J = 7.6 Hz, 1H), 7.40 (m, 5H). ¹³C NMR (75 MHz, CDCl₃): δ 14.06; 20.25; 22.31; 31.29; 37.86; 70.95; 72.99; 96.12; 100.24; 100.57; 127.20; 128.47; 128.94; 136.27; 138.02; 163.98; 167.98. Anal. Calcd. for C₁₉H₂₁NO₂: C 77.26%; H 7.17%; N 4.74%. Found: C 77.05%; H 7.00%; N 4.65%. HRMS (CI): MH⁺, 296.1648; Calc. for C₁₉H₂₂NO₂⁺: 296.1651.

4-(4-Methoxy-1-methyl-2-oxo-1,2-dihydro-pyridin-3-ylethynyl)-benzoic acid methyl ester (4d): Yellow solid, Mol. Wt.: 291.3 g/mol. Mp: 159°C. ¹H NMR (200 MHz, CDCl₃): δ 3.50 (s, 3H), 3.87 (s, 3H), 3.91 (s, 3H), 6.04 (d, J = 7.7 Hz, 1H), 7.33 (d, J = 7.7 Hz, 1H), 7.58 (d, J = 8.5 Hz, 2H), 7.94 (d, J = 8.5 Hz, 2H). ¹³C NMR (75 MHz, CDCl₃): δ 37.57; 52.15; 56.65; 85.47; 94.19; 97.31; 98.15; 128.72; 129.01; 129.32; 131.46; 139.36; 162.89; 166.65; 169.07. HRMS (CI): MH⁺, 298.1079; Calc. for C₁₇H₁₆NO₄⁺: 298.1079.

4-(1-Benzyl-4-benzyl-2-oxo-1,2-dihydro-pyridin-3-ylethynyl)-benzoic acid methyl ester (4e): White solid, Mol. Wt.: 449.5 g/mol. ¹H NMR (300 MHz, CDCl₃): δ 3.85 (s, 3H), 5.05 (s, 2H), 5.14 (s, 2H), 6.03 (d, J = 7.7 Hz, 1H), 7.35 (m, 11H), 7.53 (d, J = 8.3 Hz, 2H), 7.94 (d, J = 8.3 Hz, 2H). ¹³C NMR (75 MHz, CDCl₃): 52.40; 52.53; 71.18; 86.44; 96.37; 98.07; 99.43; 127.23; 128.48; 128.61; 128.70; 129.05; 129.16; 129.26; 129.35; 129.77; 131.72; 135.96; 136.55; 139.05; 162.88; 166.93; 168.77. HRMS (CI): MH⁺, 450.1707; Calc. for C₂₉H₂₄NO₄⁺: 450.1705.

Cyclization of 3-alkynyl-2-pyridones, general procedure: n-BuLi (2.5 M in hexanes,) was added dropwise to a suspension of Pd(PPh₃)₂Cl₂ (6.5 mg, 0.009 mmol) in THF (1 mL) until the mixture turned dark green. A heat gun was then used to gently heat the mixture and obtain a homogeneous dark red solution. MeCN (3 mL), the pyridone derivative (0.19 mmol) and the aromatic halide (0.27 mmol) were then successively added. The reaction mixture was left to stir for 48h at 60°C and concentrated *in vacuo*. The residue was purified by column chromatography (silica gel; acetone) to afford the corresponding furopyridone.

One pot coupling of 3-iodo-2-pyridones, terminal alkynes, and aryl halides, general procedure: The iodopyridone derivative (0.23 mmol), the terminal alkyne (0.28 mmol), Pd(PPh₃)₂Cl₂ (8.2 mg, 0.011 mmol) and CuI (2.8 mg, 0.011 mmol) were added to a MeCN (3.5 mL)/TEA (0.5 mL) mixture of solvents and the reaction mixture was left to stir at 60°C

for 24h. The aromatic halide (0.34 mmol) was then added, the heating was continued for 48h after which the reaction mixture was concentrated *in vacuo*. The residue was purified as described above.

2,3-Bis(4-methyloxycarbonylphenyl)-7-methyl-7H-furo[2,3-b]pyridin-4-one (7a): Yellow solid, Mol. Wt.: 417.41 g/mol. Mp: 288°C (decomp.). ¹H NMR (300 MHz, CDCl₃): δ 3.90 (s, 3H), 3.92 (s, 3H), 3.96 (s, 3H), 6.29 (d, *J* = 7.7 Hz, 1H), 7.19 (d, *J* = 7.7 Hz, 1H), 7.49 (d, *J* = 8.7 Hz, 1H), 7.64 (d, *J* = 8.5 Hz, 2H), 7.98 (d, *J* = 8.7 Hz, 2H), 8.09 (d, *J* = 8.5 Hz, 2H). ¹³C NMR (75 MHz, CDCl₃): δ 37.16, 52.54, 52.62, 112.62, 116.66, 122.09, 126.48, 129.94, 129.97, 130.22, 130.21, 130.92, 133.78, 135.90, 135.97, 144.61, 154.26, 166.80, 167.32, 175.78. Anal. Calcd. for C₂₄H₁₉NO₆: C 69.06%; H 4.59%; N 3.36%. Found: C 69.14%; H 4.53%; N 3.60%.

4-(7-Methyl-4-oxo-3-phenyl-4,7-dihydro-furo[2,3-b]pyridin-2-yl)-benzoic acid methyl ester (7b): Yellow solid, Mol. Wt.: 359.37 g/mol. Mp: 252°C. ¹H NMR (300 MHz, CDCl₃): δ 3.86 (s, 3H), 3.91 (s, 3H), 6.24 (d, *J* = 7.5 Hz, 1H), 7.16 (d, *J* = 7.5 Hz, 1H), 7.40 (m, 3H), 7.52 (m, 4H), 7.94 (d, *J* = 8.5 Hz, 2H). ¹³C NMR (75 MHz, CDCl₃): δ 37.11; 52.57; 112.87; 116.61; 123.18; 126.20; 128.69; 128.76; 129.50; 130.11; 130.71; 131.04; 134.26; 135.73; 144.12; 154.19; 166.94; 175.90. HRMS (CI): MH⁺, 360.1235; Calc. for C₂₂H₁₈NO₄⁺: 360.1235.

4-[3-(4-Iodo-phenyl)-7-methyl-4-oxo-4,7-dihydro-furo[2,3-b]pyridin-2-yl]-benzoic acid methyl ester (7c): Yellow solid, Mol. Wt.: 485.27 g/mol. Mp: 272°C (decomp.). ¹H NMR (300 MHz, DMSO-d₆, 323 K): δ 3.85 (s, 3H), 3.86 (s, 3H), 6.01 (d, *J* = 7.5 Hz, 1H), 7.25 (d, *J* = 8.3 Hz, 2H), 7.55 (d, *J* = 8.3 Hz, 2H), 7.62 (d, *J* = 7.5 Hz, 1H), 7.77 (d, *J* = 8.3 Hz, 2H), 7.92 (d, *J* = 8.3 Hz, 2H). ¹³C NMR (75 MHz, DMSO-d₆): δ 37.23; 52.99; 95.24; 111.97; 115.43; 121.85; 126.81; 129.77; 130.42; 131.35; 133.25; 134.23; 137.85; 138.15; 143.52; 154.72; 166.51; 174.93. HRMS (CI): MH⁺, 486.0202; Calc. for C₂₂H₁₇INO₄⁺: 486.0203

4-[7-Methyl-4-oxo-3-(3-trifluoromethyl-phenyl)-4,7-dihydro-furo[2,3-b]pyridin-2-yl]-benzoic acid methyl ester (7d): Yellow solid, Mol. Wt.: 427.37 g/mol. Mp: 242°C. ¹H NMR (300 MHz, CDCl₃): δ 3.89 (s, 3H), 3.92 (s, 3H), 6.25 (d, *J* = 7.5 Hz, 1H), 7.19 (d, *J* = 7.5 Hz, 1H), 7.49 (d, *J* = 8.5 Hz, 2H), 7.55 (t, *J* = 7.7 Hz, 1H), 7.65 (d, *J* = 7.7 Hz, 1H), 7.77 (d, *J* = 7.7 Hz, 1H), 7.83 (s, 1H), 7.97 (d, *J* = 8.5 Hz, 2H). ¹³C NMR (125 MHz, CDCl₃): δ 37.22; 52.89; 111.39; 115.49; 121.31; (121.76, 123.93, 126.10, 128.26); (125.42, 125.45, 125.48, 126.1); 126.92; (127.83, 127.86, 127.89, 128.02); (129.59, 129.84, 130.09, 130.34); 129.95; 130.10; 130.29; 132.85; 134.02; 135.07; 138.17; 144.13; 154.78; 166.47; 174.95. ¹⁹F NMR (188 MHz, CDCl₃): -63.12. HRMS (CI): MH⁺, 428.1106; Calc. for C₂₃H₁₇F₃NO₄⁺: 428.1109

4-(7-Methyl-4-oxo-3-thiophen-2-yl-4,7-dihydro-furo[2,3-b]pyridin-2-yl)-benzoic acid methyl ester (7e): Yellow solid, Mol. Wt.: 365.40 g/mol. Mp: 252°C (decomp.). ¹H NMR (300 MHz, CDCl₃): δ 3.84 (s, 3H), 3.92 (s, 3H), 6.23 (d, *J* = 7.5 Hz, 1H), 7.13 (m, 2H), 7.36 (d, *J* = 4.7 Hz, 1H), 7.41 (d, *J* = 4.7 Hz, 1H), 7.59 (d, *J* = 8.5 Hz, 2H), 7.98 (d, *J* = 8.5 Hz, 2H). ¹³C NMR (75 MHz, CDCl₃): δ 37.10; 52.61; 112.97; 116.01; 116.74; 126.74; 127.57; 127.71; 129.94; 130.03; 130.12; 131.18; 133.86; 135.81; 145.19; 154.08; 166.91; 175.71. HRMS (CI): MH⁺, 366.0800; Calc. for C₂₀H₁₆NO₄S⁺: 366.0800.

4-[3-(4-Methoxy-phenyl)-7-methyl-4-oxo-4,7-dihydro-furo[2,3-b]pyridin-2-yl]-benzoic acid methyl ester (7f): Yellow solid, Mol. Wt.: 389.40 g/mol. Mp: 292°C (decomp.). ¹H NMR (300 MHz, CDCl₃): δ 3.86 (s, 6H), 3.91 (s, 3H), 6.23 (d, *J* = 7.5 Hz, 1H), 6.95 (d, *J* =

8.5 Hz, 2H), 7.14 (d, J = 7.7 Hz, 1H), 7.46 (d, J = 8.5 Hz, 2H), 7.54 (d, J = 8.3 Hz, 2H), 7.94 (d, J = 8.3 Hz, 2H). ^{13}C NMR (75 MHz, CDCl_3): δ 37.32; 52.89; 55.98; 113.12; 114.98; 117.32; 123.74; 123.80; 126.92; 130.17; 130.93; 132.81; 135.29; 136.50; 144.76; 155.16; 161.03; 168.02; 177.12. HRMS (CI): MH^+ , 390.1339; Calc. for $\text{C}_{23}\text{H}_{20}\text{NO}_5^+$: 390.1341

4-[7-Methyl-3-(4-nitrophenyl)-4-oxo-4,7-dihydrofuro[2,3-b]pyridin-2-yl]-benzoic acid methyl ester (7g): Light yellow solid, Mol. Wt.: 404.37 g/mol. Mp: 293°C (decomp.). ^1H NMR (300 MHz, DMSO-d_6 , 383 K): δ 3.87 (s, 6H), 6.03 (d, J = 7.6 Hz, 1H), 7.52 (d, J = 8.5 Hz, 2H), 7.69 (d, J = 7.8 Hz, 1H), 7.74 (d, J = 8.5 Hz, 2H), 7.92 (d, J = 8.3 Hz, 2H), 8.22 (d, J = 7.2 Hz, 2H). ^{13}C NMR (75 MHz, DMSO-d_6 , 363 K): δ 36.87; 52.35; 111.90; 116.42; 120.53; 123.53; 126.43; 130.06; 130.12; 131.62; 132.95; 135.73; 137.73; 144.74; 147.65; 154.00; 166.31; 175.34. HRMS (CI): MH^+ , 405.1083; Calc. for $\text{C}_{22}\text{H}_{17}\text{N}_2\text{O}_6^+$: 405.1087

4-(7-Methyl-4-oxo-2-phenyl-4,7-dihydro-furo[2,3-b]pyridin-3-yl)-benzoic acid methyl ester (7h): Yellow solid, Mol. Wt.: 359.37 g/mol. Mp: 238°C. ^1H NMR (300 MHz, CDCl_3): δ 3.81 (s, 3H), 3.92 (s, 3H), 6.21 (d, J = 7.5 Hz, 1H), 7.12 (d, J = 7.5 Hz, 1H), 7.28 (m, 3H), 7.40 (m, 2H), 7.63 (d, J = 8.1 Hz, 2H), 8.02 (d, J = 8.1 Hz, 2H). ^{13}C NMR (75 MHz, CDCl_3): δ 37.10; 52.50; 112.47; 116.38; 119.90; 127.10; 128.95; 129.01; 129.58; 129.76; 131.08; 135.66; 136.47; 145.92; 154.00; 167.47; 175.82. HRMS (CI): MH^+ , 360.1236; Calc. for $\text{C}_{22}\text{H}_{18}\text{NO}_4^+$: 360.1235

3-(4-Fluoro-phenyl)-7-methyl-2-phenyl-7*H*-furo[2,3-b]pyridin-4-one (7i): Yellow solid, Mol. Wt.: 319.33 g/mol. Mp: 170°C. ^1H NMR (300 MHz, CD_2Cl_2): δ 3.74 (s, 3H), 6.07 (d, J = 7.7 Hz, 1H), 7.01 (t, J = 8.6 Hz, 2H), 7.08 (d, J = 7.5 Hz, 1H), 7.23 (m, 3H), 7.42 (m, 2H). ^{13}C NMR (50 MHz, CDCl_3): δ 36.71; 112.40; (115.02, 115.45); 116.11; 119.61; 126.57; (126.89, 126.96); 128.34; 128.61; 129.54; (132.29, 132.45); 135.08; 145.10; 153.57; (160.20, 165.10); 175.61. NMR ^{19}F (188 MHz, CDCl_3): -114.85. HRMS (CI): MH^+ , 320.1089; Calc. for $\text{C}_{20}\text{H}_{15}\text{FNO}_2^+$: 320.1087

4-(2-Butyl-7-methyl-4-oxo-4,7-dihydro-furo[2,3-b]pyridin-3-yl)-benzoic acid methyl ester (7j): Yellow oil, Mol. Wt.: 339.39 g/mol. ^1H NMR (200 MHz, CDCl_3): δ 0.89 (t, J = 7.1 Hz, 3H), 1.39 (hexuplet, J = 7.0 Hz, 2H), 1.68 (quintuplet, J = 7.1 Hz, 2H), 2.75 (t, J = 7.0 Hz, 2H), 3.77 (s, 3H), 3.92 (s, 3H), 6.21 (d, J = 7.5 Hz, 1H), 7.09 (d, J = 7.6 Hz, 1H), 7.60 (d, J = 8.1 Hz, 2H), 8.06 (d, J = 8.0 Hz, 2H). ^{13}C NMR (50 MHz, CDCl_3): δ 13.73; 22.33; 25.91; 30.53; 36.68; 52.07; 110.82; 115.86; 119.16; 128.77; 129.13; 130.20; 134.70; 136.09; 149.85; 153.37; 167.16; 175.25. HRMS (CI): MH^+ , 340.1550; Calc. for $\text{C}_{20}\text{H}_{22}\text{NO}_4^+$: 340.1548

2,3-bis(4-methyloxycarbonylphenyl)-7-benzyl-7*H*-furo[2,3-b]pyridin-4-one (7k): Light yellow solid, Mol. Wt.: 493.5 g/mol. Mp: 205°C (decomp.). ^1H NMR (300 MHz, CDCl_3): δ 3.89 (s, 3H), 3.92 (s, 3H), 5.30 (s, 2H), 6.23 (d, J = 7.5 Hz, 1H), 7.25 (d, J = 7.5 Hz, 1H), 7.45 (m, 7H), 7.60 (d, J = 8.1 Hz, 2H), 7.91 (d, J = 8.5 Hz, 2H), 8.05 (d, J = 8.5 Hz, 2H). ^{13}C NMR (75 MHz, CDCl_3): δ 52.55; 52.62; 54.06; 112.68; 116.87; 122.01; 126.44; 128.21; 129.41; 129.74; 129.97; 130.24; 130.92; 133.75; 134.56; 135.09; 135.97; 144.58; 153.97; 166.78; 167.30; 175.76. HRMS (CI): MH^+ , 494.1609; Calc. for $\text{C}_{30}\text{H}_{24}\text{NO}_6^+$: 494.1603

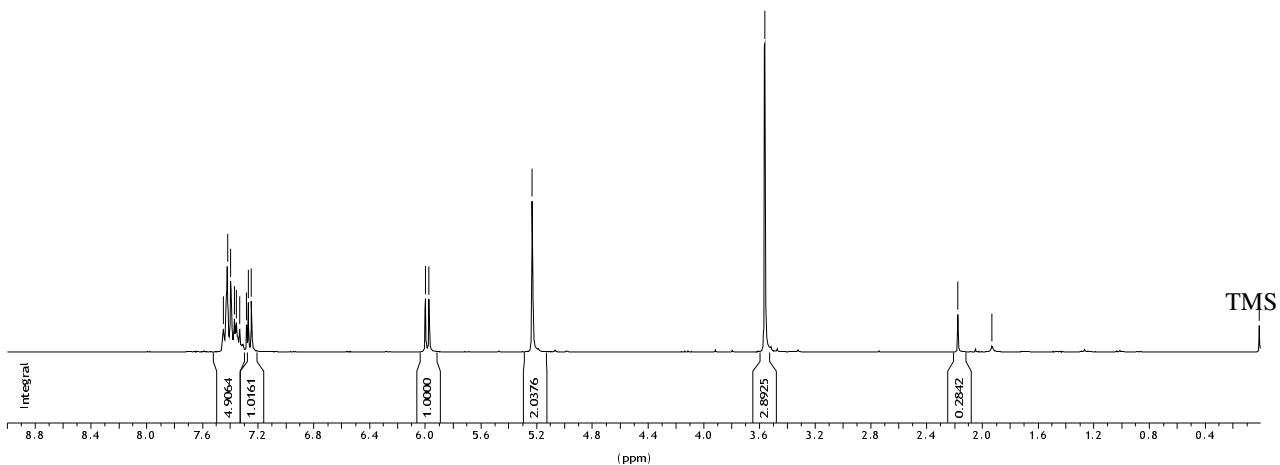
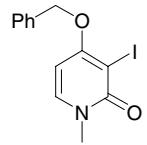
4-Benzyl-2,3-bis-(4-methoxycarbonyl-phenyl)-7-methyl-furo[2,3-b]pyridin-7-ium iodide (6a): Compound **6a** was isolated from cyclization of pyridone **4a** with methyl 4-iodobenzoate using the conditions described above. In this case the reaction mixture was concentrated after only 16h heating and purified by column chromatography (silica gel) using

ethanol as eluent. Yellow solid, Mol. Wt.: 635.45 g/mol. ^1H NMR (300 MHz, DMSO-d₆): δ 3.85 (s, 3H), 3.93 (s, 3H), 4.40 (s, 3H), 5.46 (s, 2H), 7.05 (d, J = 7.2 Hz, 2H), 7.19-7.37 (m, 3H), 7.62 (d, J = 8.1 Hz, 2H), 7.71 (d, J = 8.1 Hz), 8.01 (d, J = 6.9 Hz, 2H), 8.86 (d, J = 7.8 Hz, 1H). ^{13}C NMR (50 MHz, DMSO-d₆): δ 39.73; 52.31; 52.37; 72.36; 106.01; 113.52; 117.83; 126.93; 127.25; 128.15; 128.32; 129.61; 129.84; 130.41; 130.00; 130.69; 131.13; 133.92; 133.97; 142.43; 148.81; 153.90; 1165.32; 165.76. M+, 508.1764; calc. for C₃₁H₂₆NO₆⁺: 508.1760.

Crystal data for 7i : (C₂₀H₁₄FNO₂)

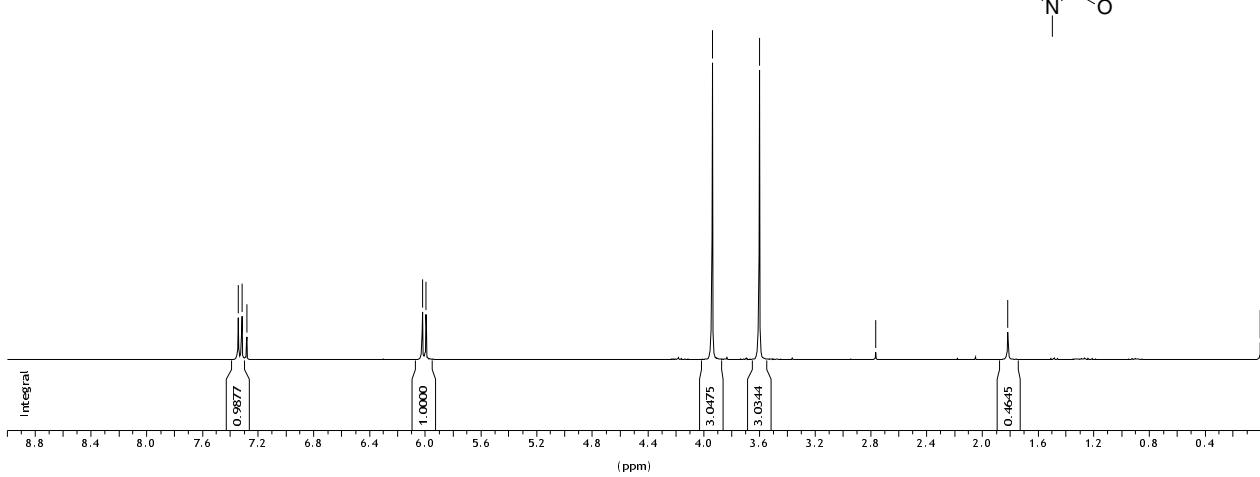
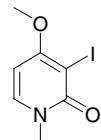
A suitable crystal was obtained from a solution of **7i** in CH₂Cl₂/EtOH. M=319.32 g/mol for C₂₀H₁₄FNO₂, monoclinic, P2(1)/c, a =12.532(3) Å, b =12.935(3) Å, c =9.831(2) Å, α =90°, β =92.51(3)°, γ =90°, V =1592.1(6) Å³, Z =4, $R_{gt}(F)$ =0.0664, $wR_{all}(F^2)$ =0.1897, T =293 K.

2a - CDCl₃



PROTONS

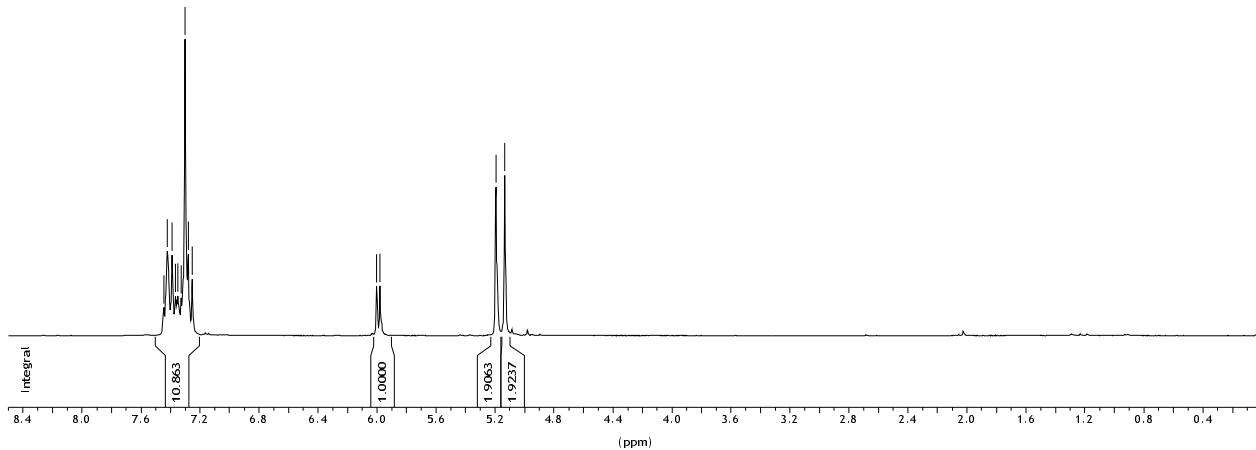
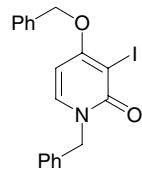
2b - CDCl₃



PROTO No
2c - CDCl₃

7.4458
7.4194
7.3893
7.3642
7.3304
7.3278
7.3027
7.2776
7.2519

6.0022
5.9771
5.1925
5.1354



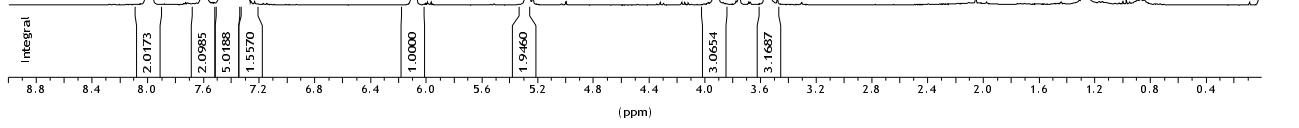
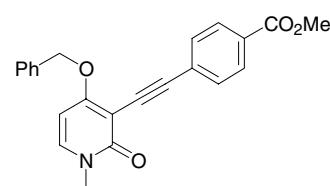
PROTO No

4a - CDCl₃

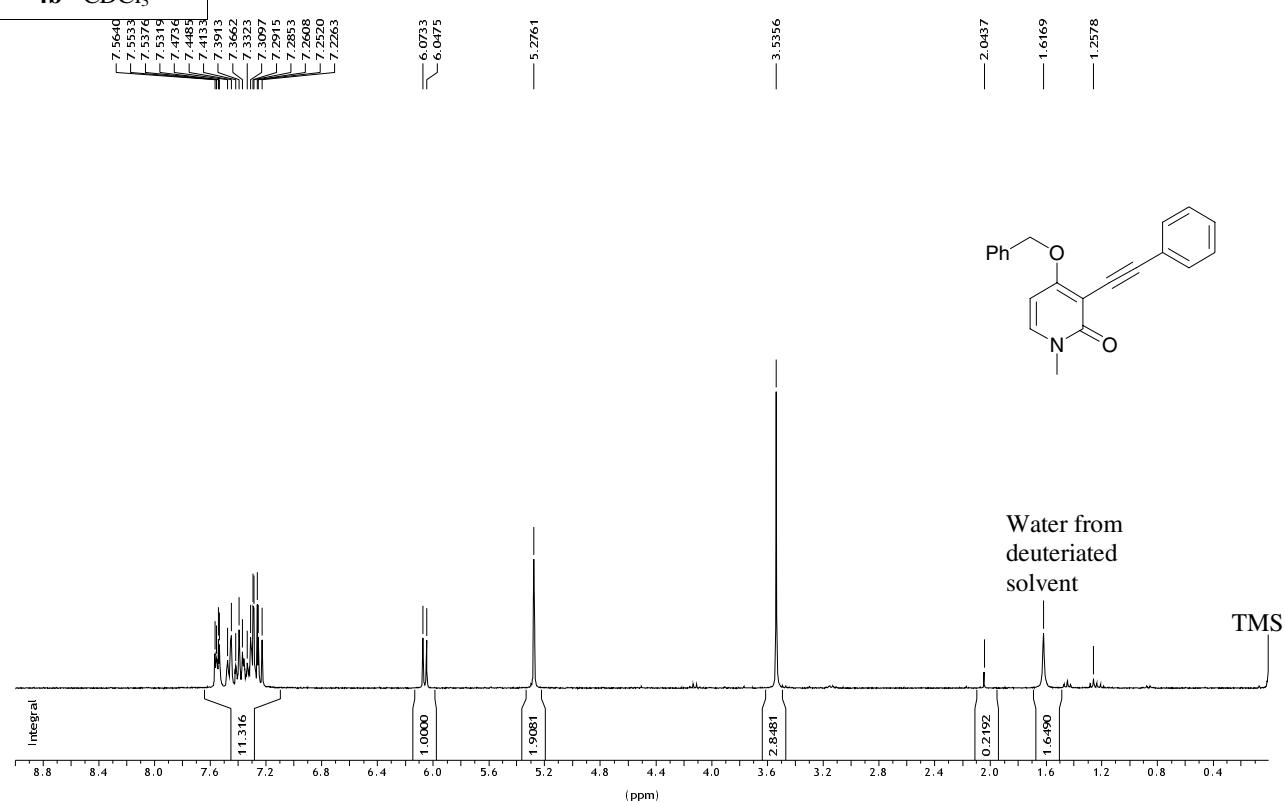
8.0000
7.9718
7.6027
7.5745
7.5747
7.4502
7.4351
7.4289
7.4075
7.3824
7.3761
7.3542
7.3083
7.2814

6.1007
6.0750
5.2710

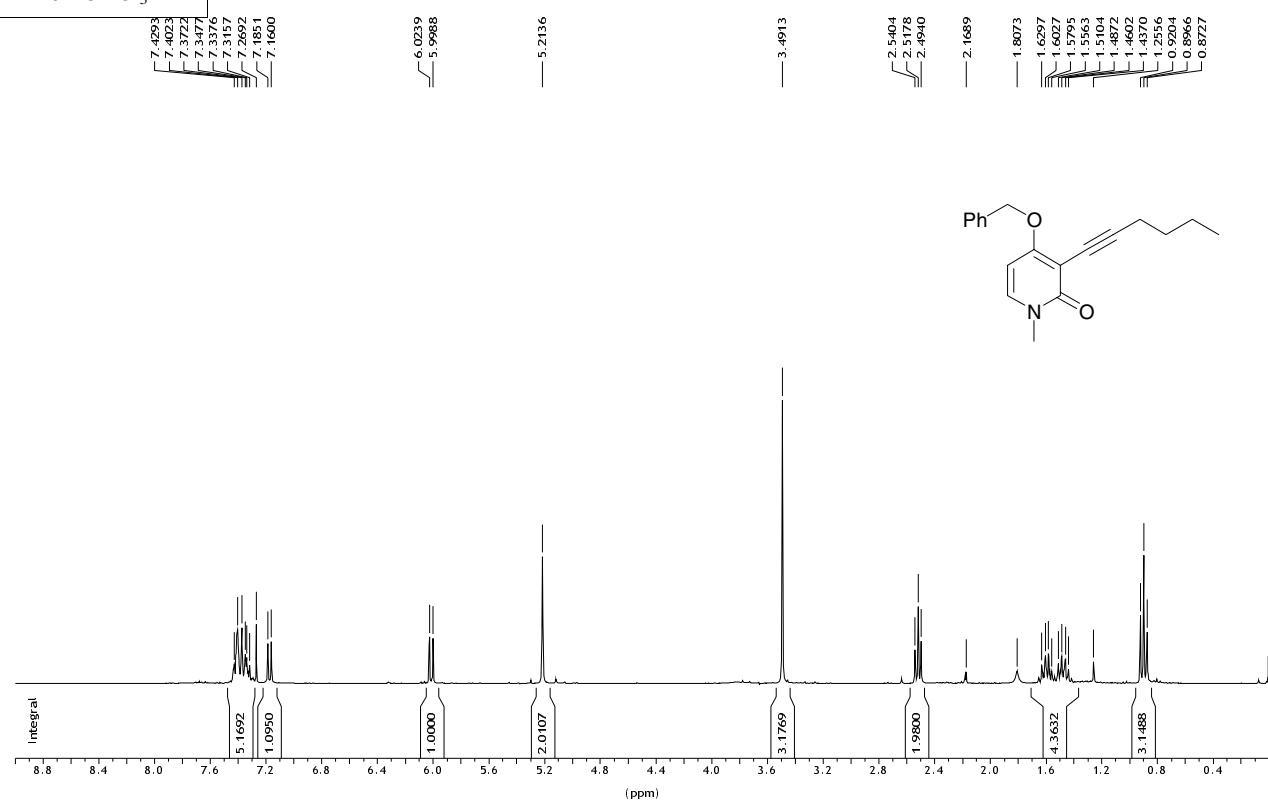
3.9190
3.5411



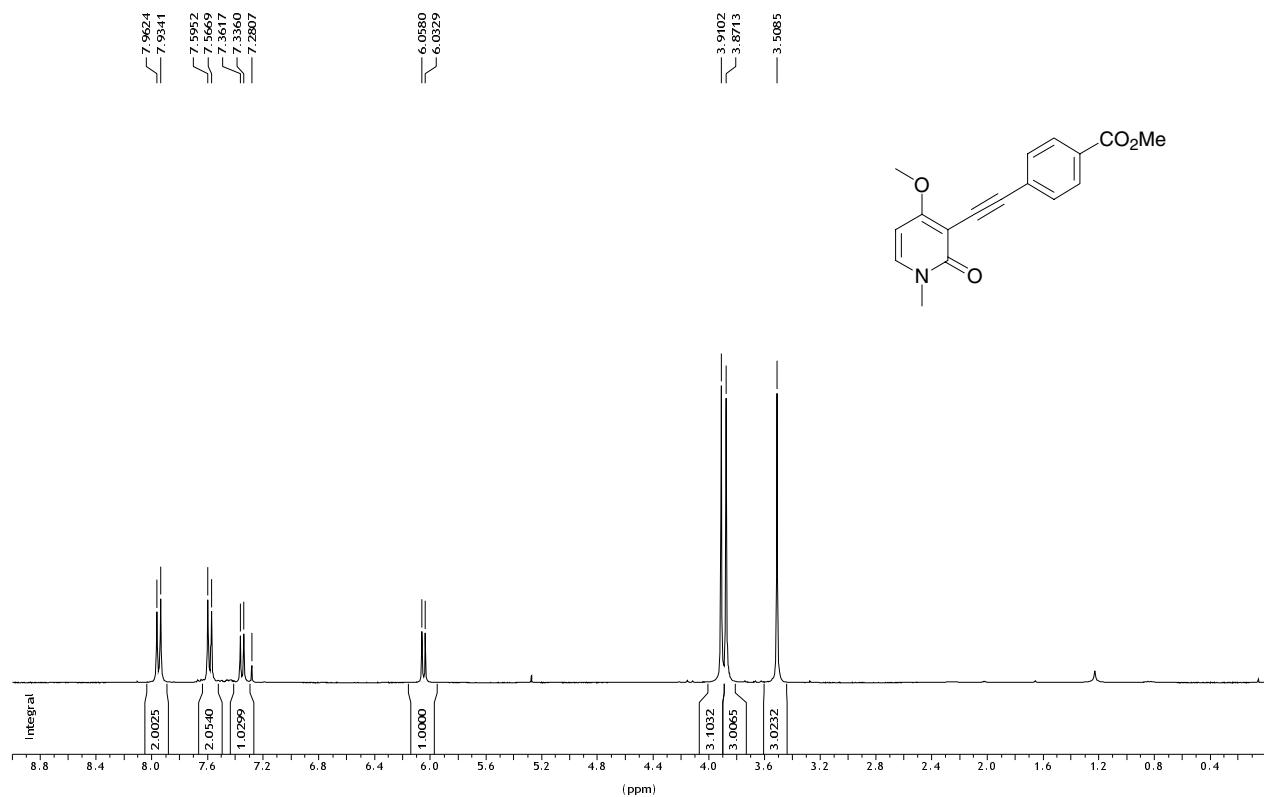
PROTO No

4b - CDCl₃

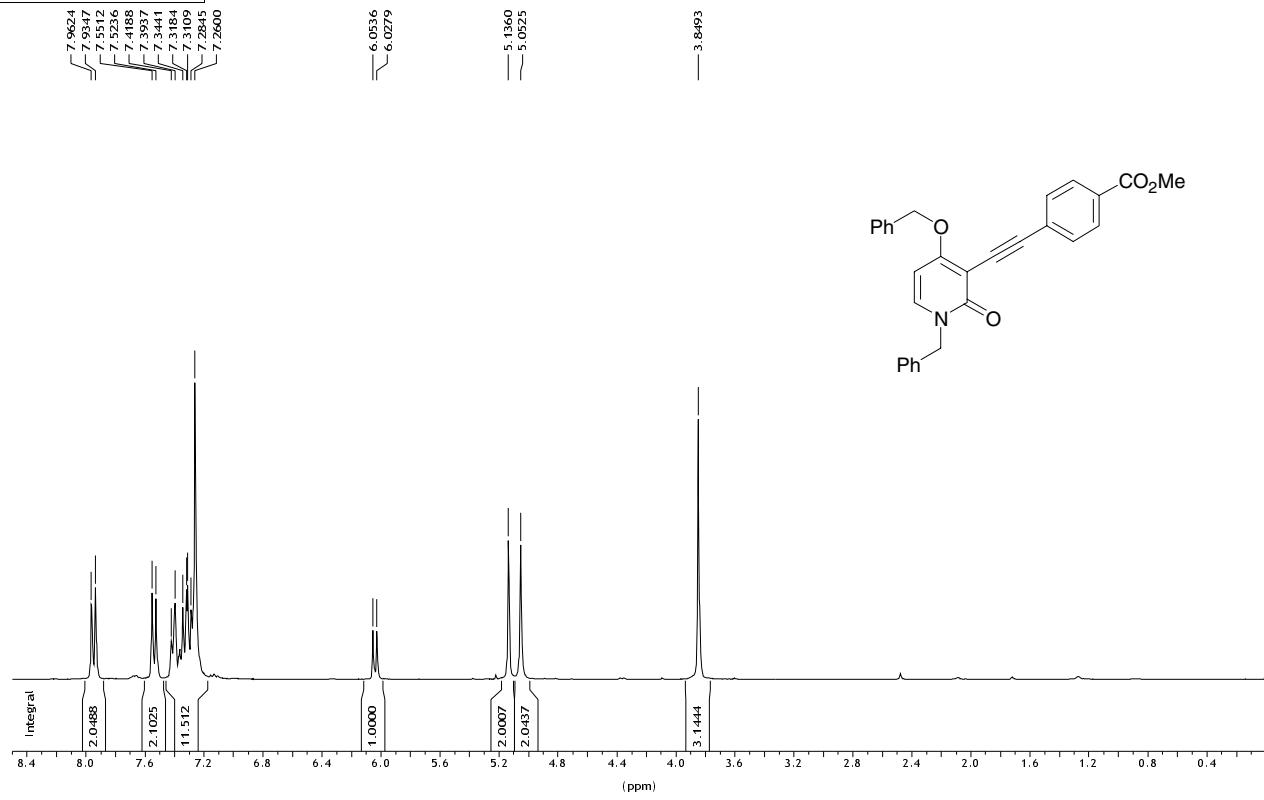
PROTO No

4c - CDCl₃

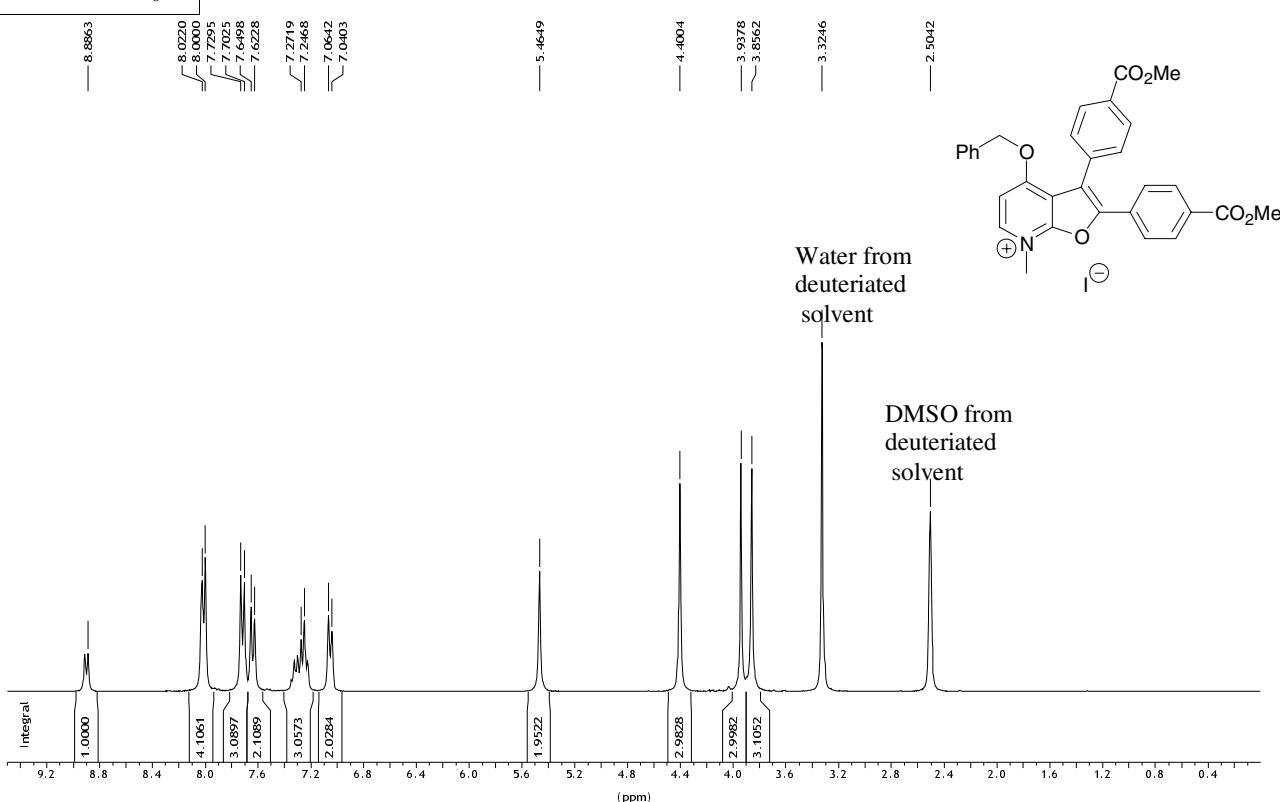
PROTO No
4d - CDCl₃



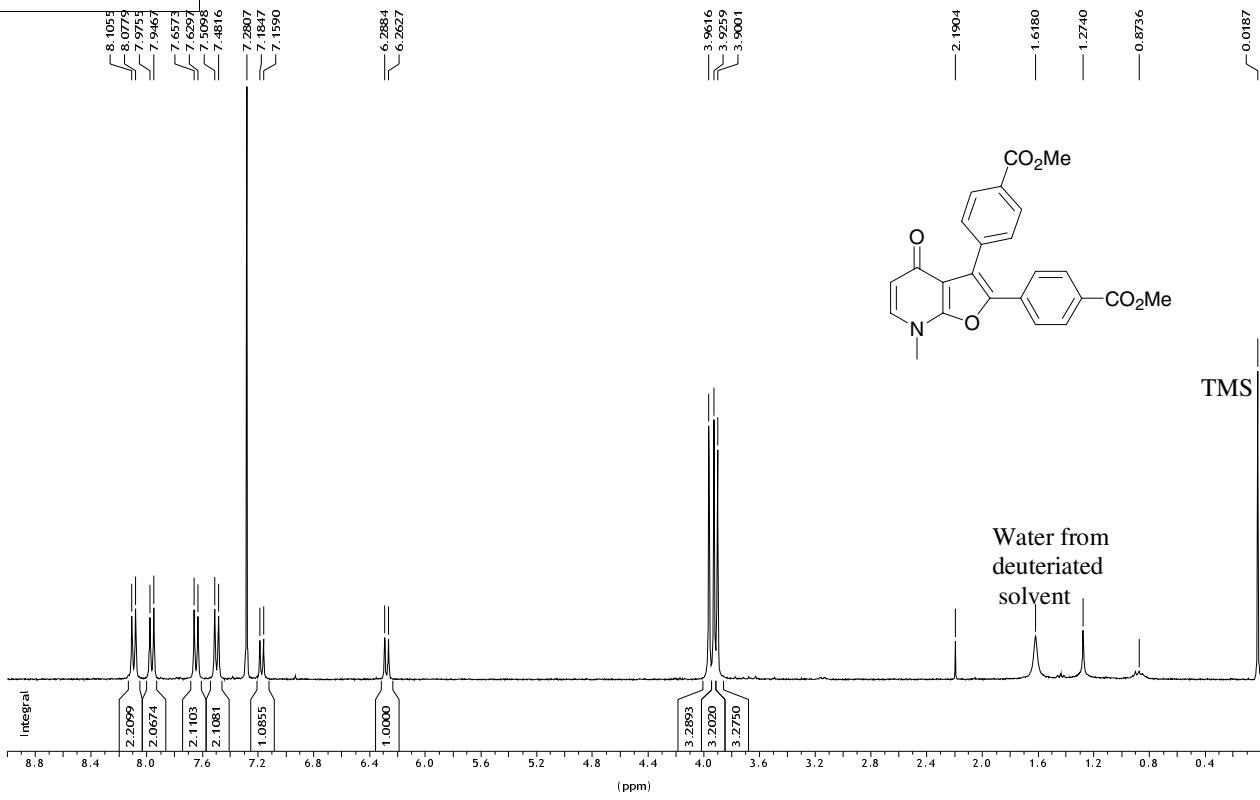
PROTO No
4e - CDCl₃



PROTO No

6a - DMSO-d₆

PROTO No

7a - CDCl₃

PROTO No

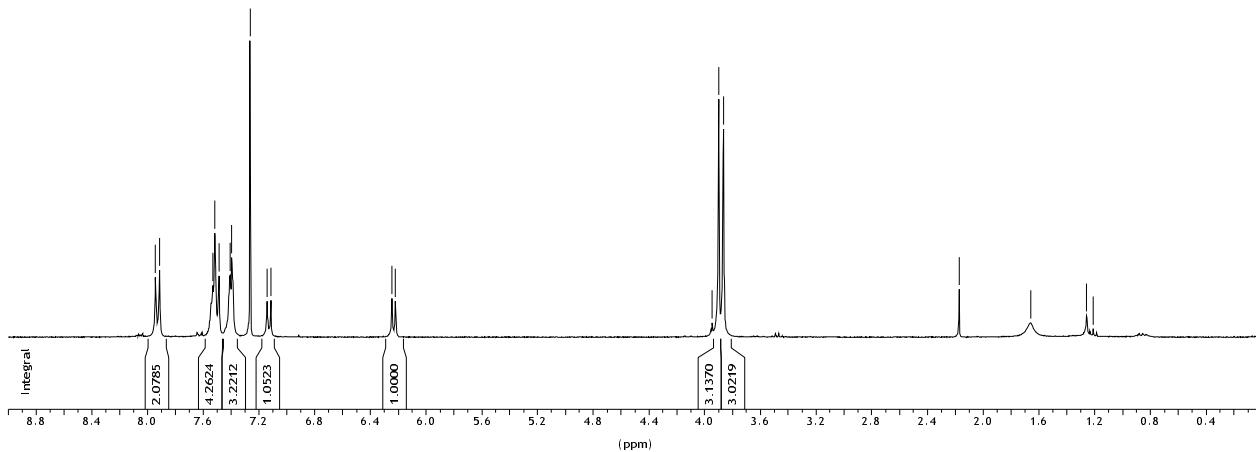
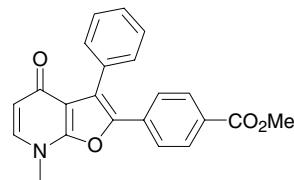
7b - CDCl₃

7.9418
7.9136
7.7904
7.7633
7.6375
7.6124
7.5638
7.5417
7.2694
7.2418

6.2452
6.2201

3.9455
3.8990
3.8626

2.1705
1.6583
1.2547
1.2082



spectre proton de EB332F3 a 50 degres □

7c - DMSO-d₆ at 323 K

7.9391
7.9115
7.7904
7.7633
7.6375
7.6124
7.5638
7.5417
7.2694
7.2418

6.0223
5.9972

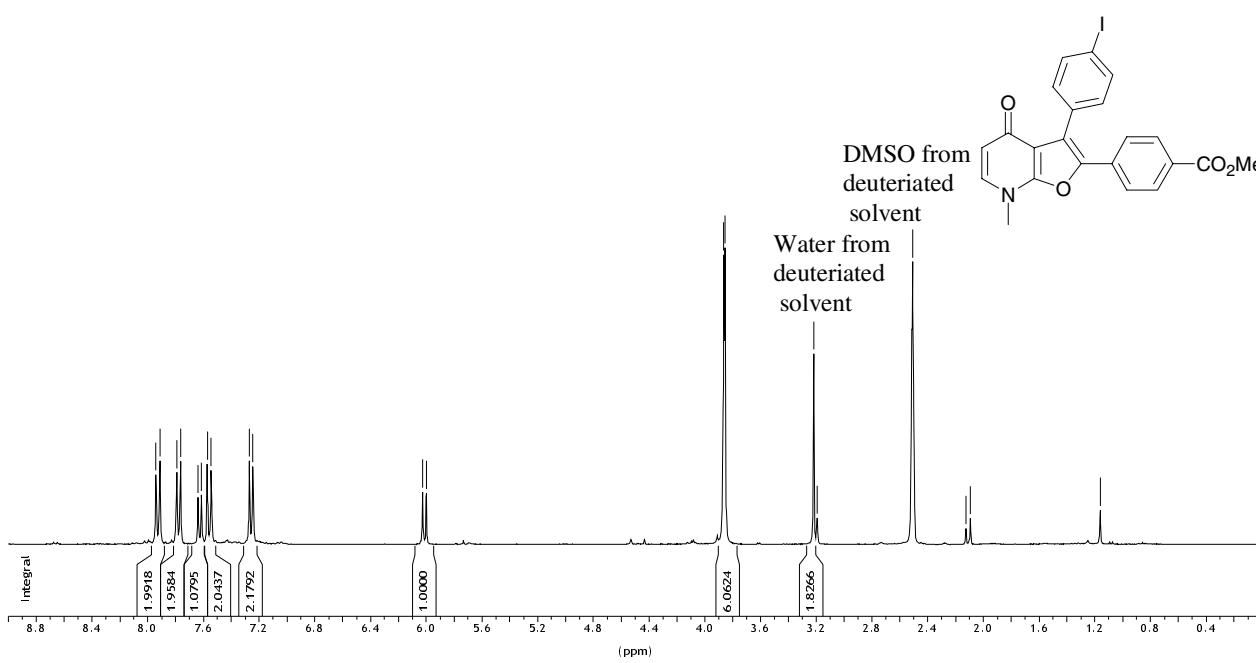
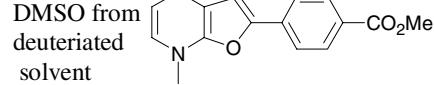
3.8606
3.8531

3.2166
3.1921

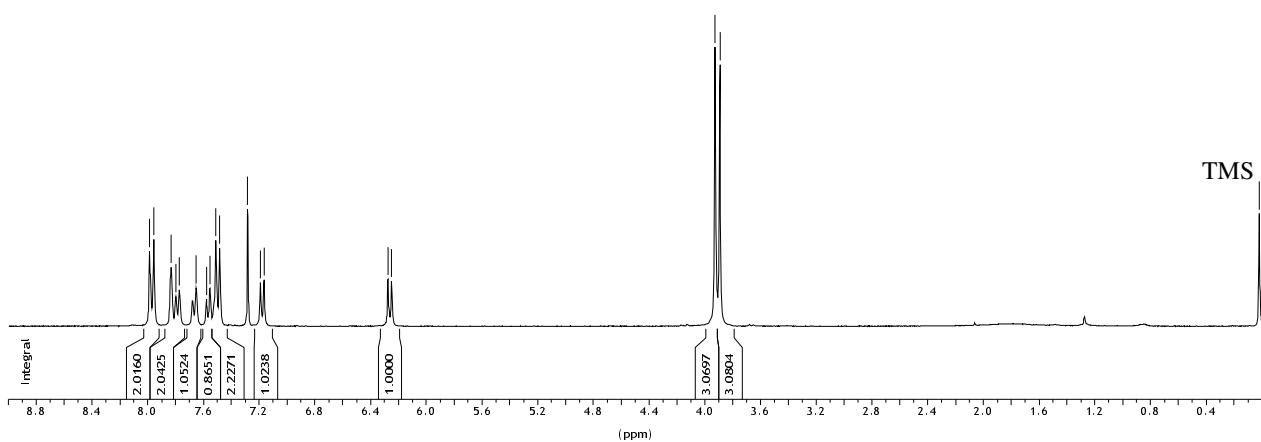
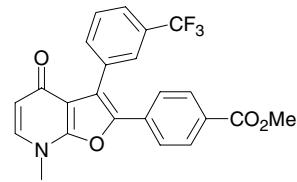
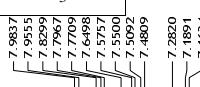
2.5057

2.1245
2.0931

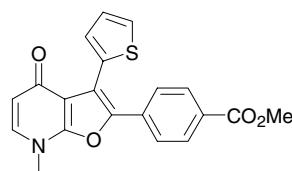
1.1598



PROTO No

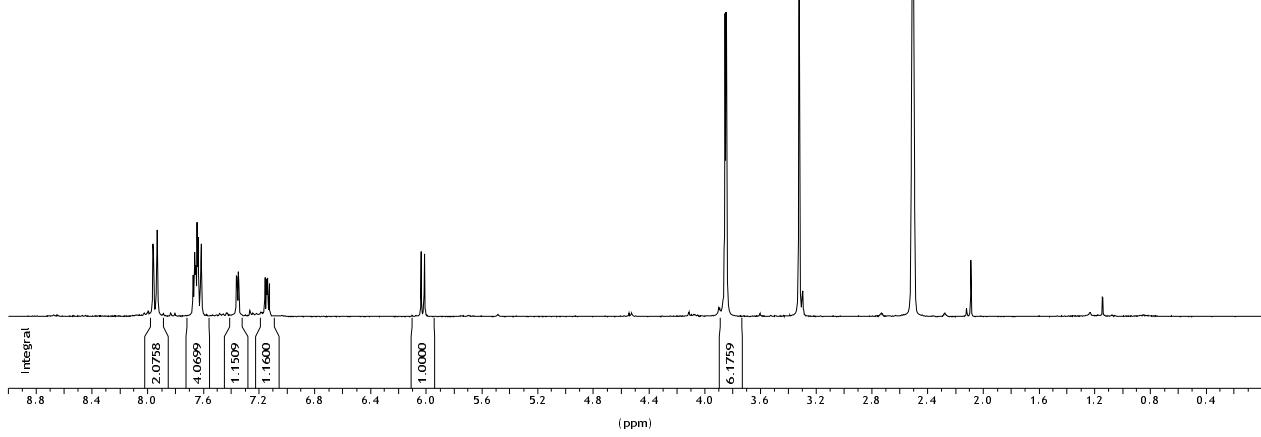
7d - CDCl₃

PROTO No

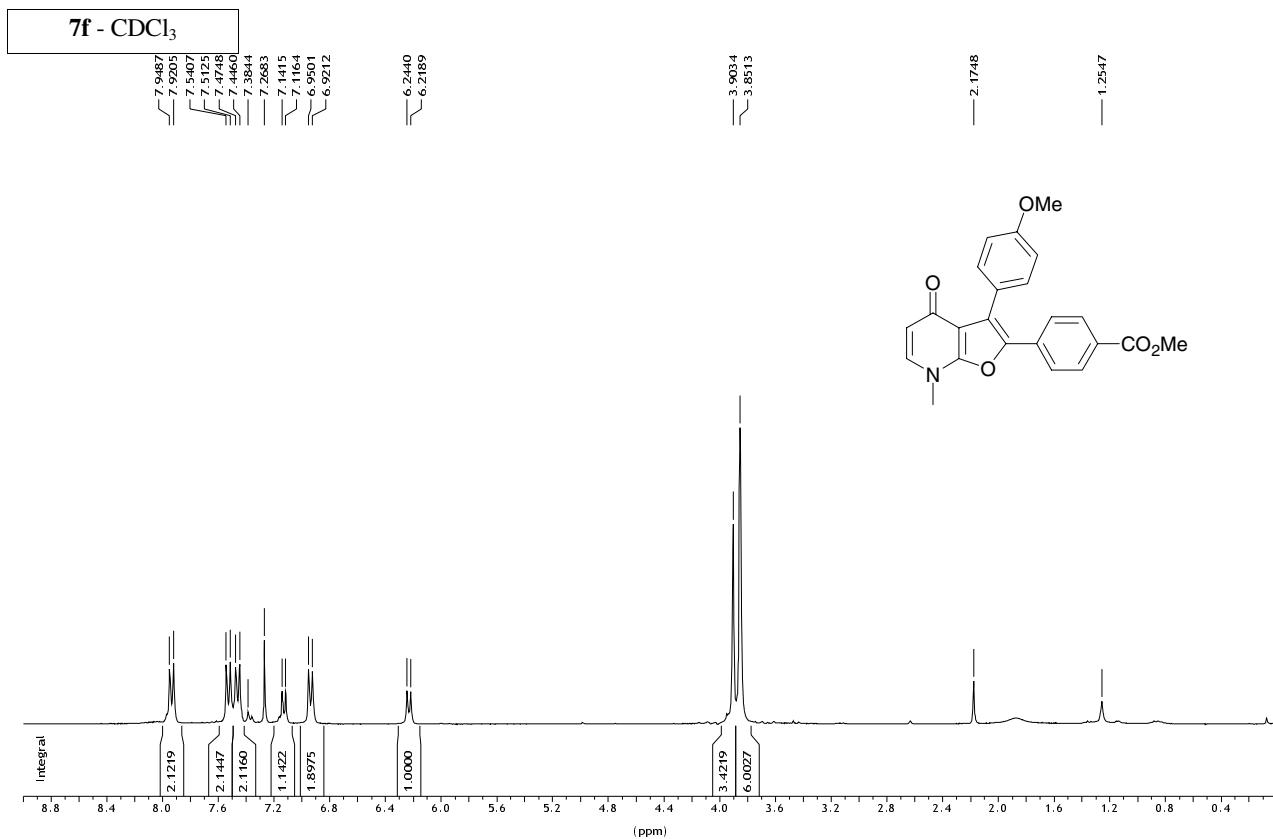
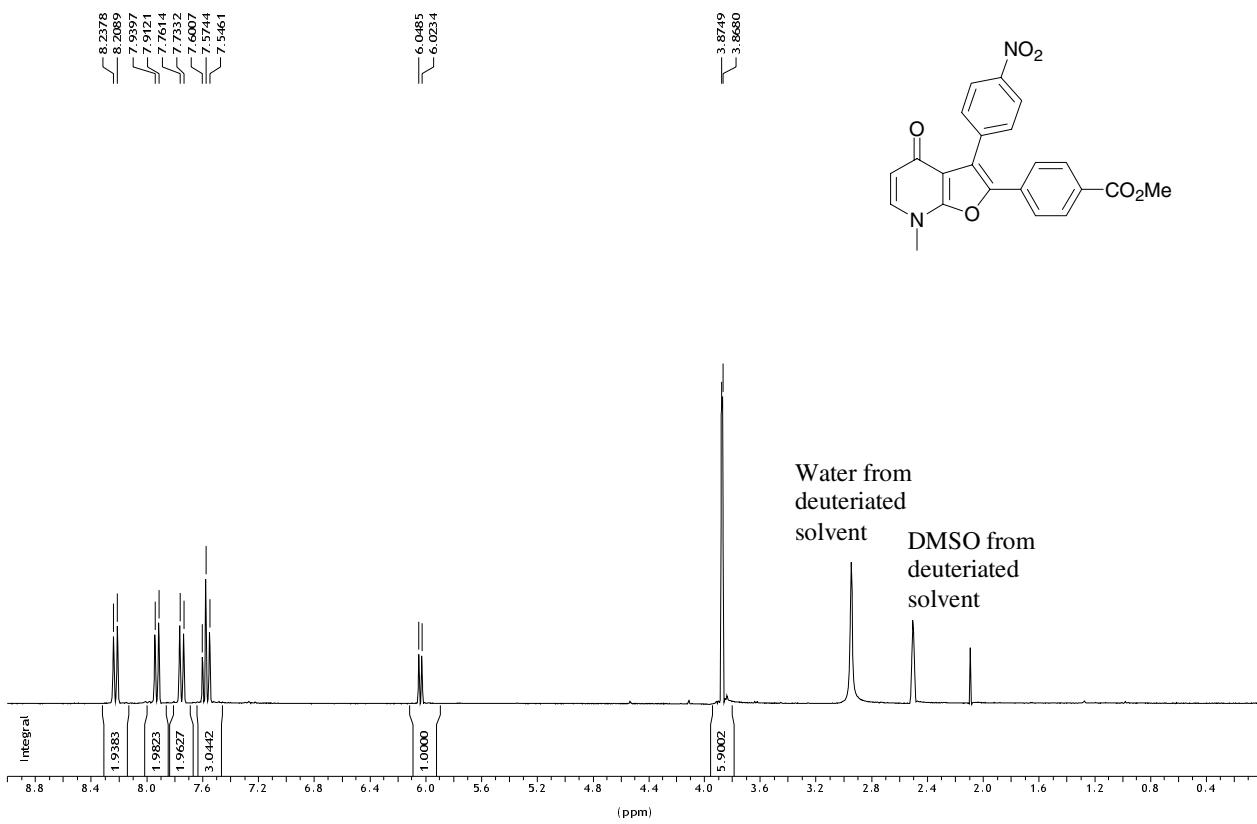
7e - DMSO-d₆

Water from deuteriated solvent

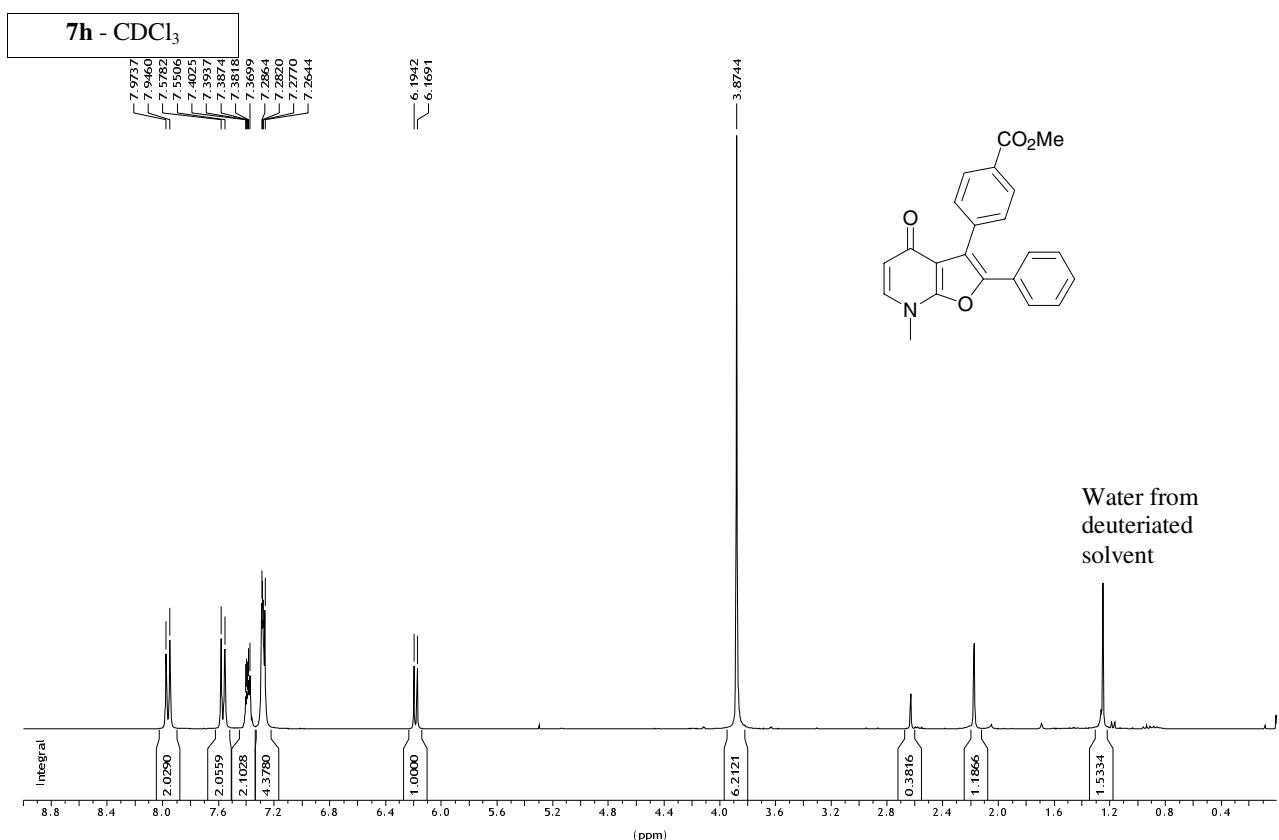
DMSO from deuteriated solvent



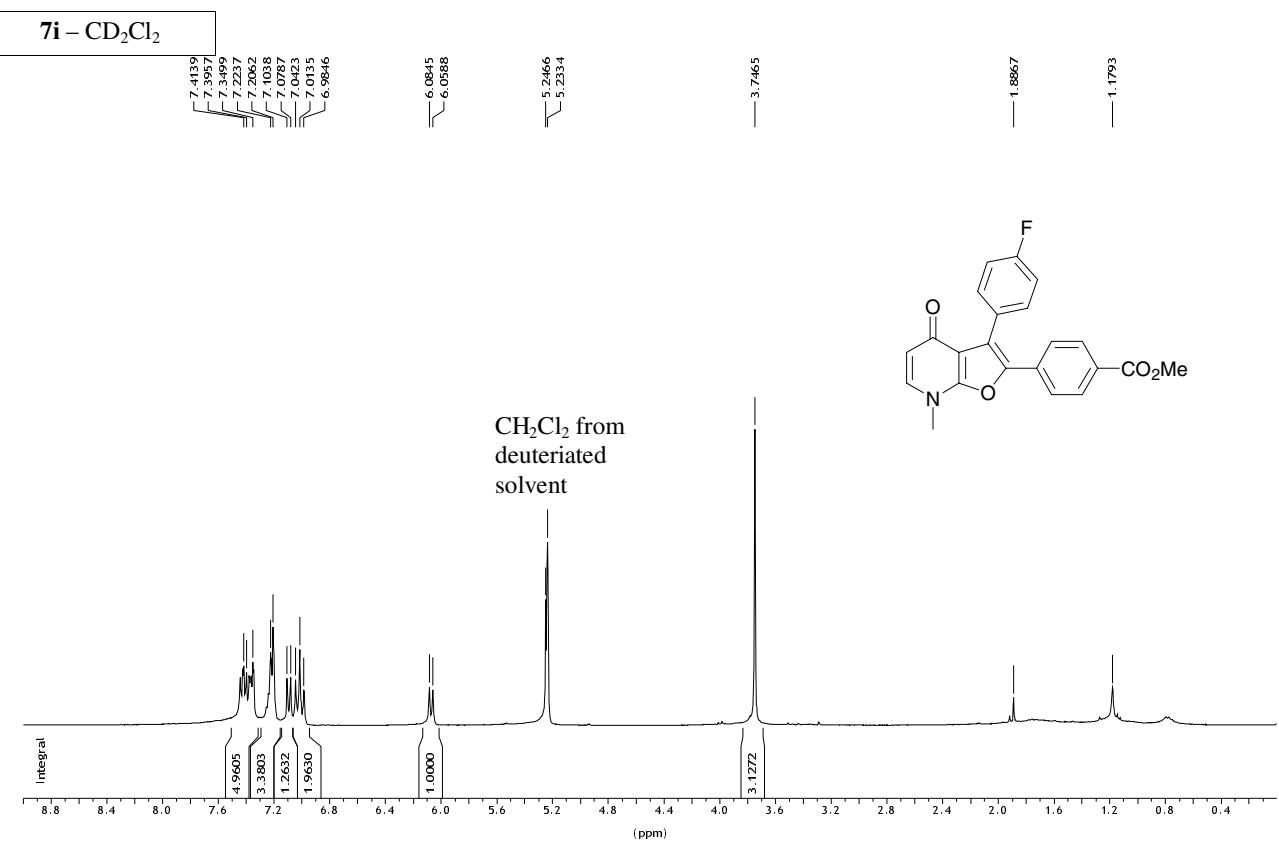
PROTO No

**7g – DMSO-d_6 at 383 K**

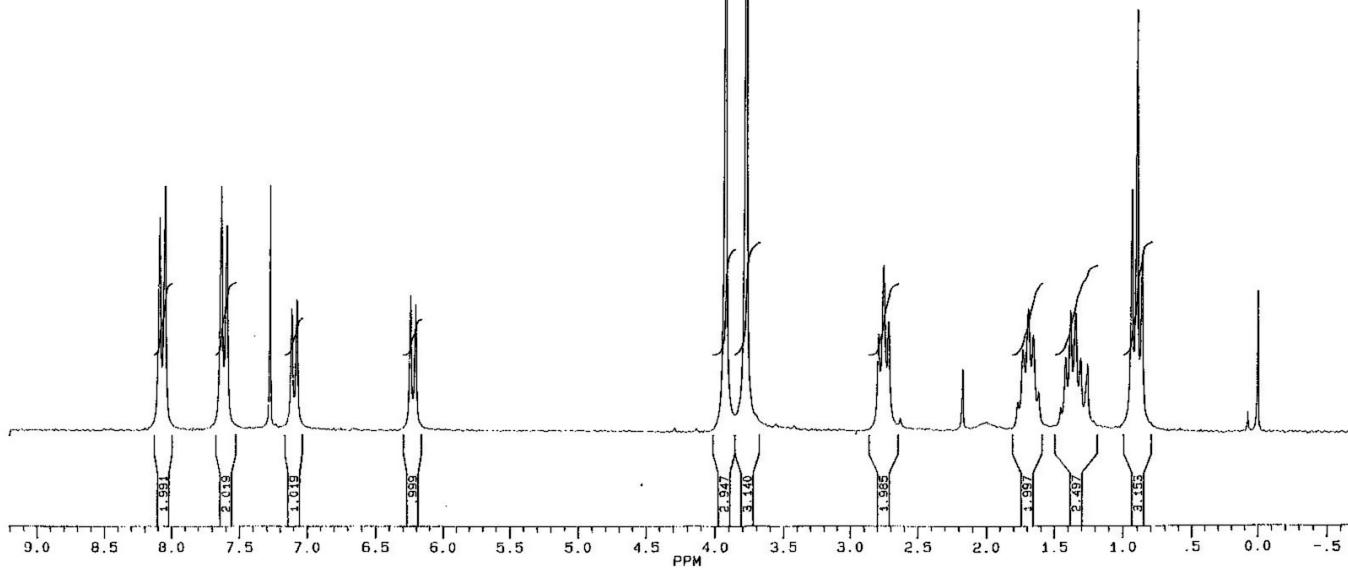
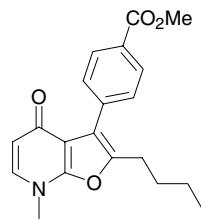
spectre proton de EB331a



PROTON128a



7j - CDCl₃



spectre proton de EB352B

7k - CDCl₃

