A New Synthesis of 2-Aryl-3-substituted Benzo[*b*]furans from Benzyl 2-Halophenyl Ethers

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General Considerations. All reactions involving air-sensitive compounds were carried out under an N₂ atmosphere in oven-dried glassware with magnetic stirring. Temperatures are reported as bath temperatures. THF was continuously refluxed and freshly distilled from sodium under nitrogen. Solvents used in extraction and purification were distilled prior to use. TLC was performed on aluminum-backed plates coated with silica gel 60 with F₂₅₄ indicator; the chromatograms were visualized by UV light (254 nm) and/or by staining with a Ce/Mo reagent, anisaldehyde or phosphomolybdic acid solution and subsequent heating. *R_f* values refer to silica gel. Flash column chromatography was carried out on silica gel 60, 230-400 mesh (Merck). Melting points were obtained on a Büchi-Tottoli apparatus using open capillary tubes and are uncorrected. ¹H and ¹³C NMR spectra were recorded on a Varian Inova-400 or Varian Mercury-Plus 300 spectrometer. ¹H NMR spectra were recorded at 400 MHz or at 300 MHz. Chemical shifts are reported in ppm from tetramethylsilane with the residual solvent resonance as the internal standard (CHCl₃; δ 7.16). Data are reported as follows: chemical shift, multiplicity (s: singlet, br s: broad singlet, d: doublet, dd: doublet of doublets, dt: doublet of triplets, t: triplet, td: triplet of doublets, q: quartet, m: multiplet), coupling constants (*J* in Hz) and integration. ¹³C NMR spectra were recorded at 100.6 MHz or at 75.4 MHz using broadband proton decoupling. Chemical shifts are reported in ppm with the solvent resonance as internal standard (CDCl₃; δ 77.16). Low-resolution electron impact mass spectra (EI-LRMS) were obtained at 70 eV on a HP 6890N/5973 or Micromass Autospec spectrometer and only the molecular ions and/or base peaks in MS are given. High-resolution mass spectrometry (HRMS) was carried out on a Micromass Autospec spectrometer. Elemental analyses were performed with Perkin Elmer and LECO CHNS-932 with VT-900 elemental analysers. HPLC analysis were carried out on a Agilent series 1100 with UV-Visible diode-array detector (G1315B) at λ = 254 nm and a Kromasil 100 SIL column (25 x 0.46 cm, Scharlab S.L.), with a flow rate = 1 mLmin⁻¹. In all the cases the percentage of total chromatogram integration represented by the product peak was larger than 95%. All commercially available reagents were used without purification unless otherwise indicated and were purchased from standard chemical suppliers.

Typical Procedure for the Synthesis of benzyl 2-haloaryl ethers 1 and 7. The corresponding commercially available *o*-halophenol or naphthol (10 mmol) and benzyl chloride derivative (10 mmol) were dissolved in acetonitrile (20 mL), potassium carbonate (1.52 g, 11 mmol) was added to the solution, and the resulting mixture was refluxed overnight. Then the mixture was cooled to room temperature, the remaining solid was filtered and the resulting solution was concentrated under reduced pressure. The residue was purified by flash chromatography (eluent: hexane/EtOAc) on silica gel to afford the corresponding ethers **1** and **7**.

Benzyl 2-iodophenyl ether (1a): Treatment of 2-iodophenol (2.20 g, 10 mmol) with benzyl chloride (1.27 g, 10 mmol) and potassium carbonate (1.52 g, 11 mmol) according to the typical procedure, and purification by column chromatography (eluent: hexane/EtOAc, 20/1) on silica gel, gave **1a** (2.54 g, 84%) as a colorless oil: R_f 0.38 (hexane/EtOAc, 10/1); ^1H NMR (300 MHz, CDCl_3) δ 7.88 (dd, $J = 7.7$, 1.4 Hz, 1H), 7.57 (d, $J = 7.4$ Hz, 2H), 7.50-7.28 (m, 4H), 6.91 (dd, $J = 8.3$, 1.1 Hz, 1H), 6.79 (td, $J = 7.4$, 1.1 Hz, 1H), 5.18 (s, 2H); ^{13}C NMR (75.4 MHz, CDCl_3) δ 157.1 (C), 139.5 (CH), 136.5 (C), 129.5 (CH), 128.6 (CH), 127.9 (CH), 127.0 (CH), 122.8 (CH), 112.7 (CH), 86.8 (C), 70.6 (CH_2); EI-LRMS m/z 310 (M^+ , 45), 91 (100).

Benzyl 2-bromo-4-methylphenyl ether (1b): Treatment of 2-bromo-4-methylphenol (1.87 g, 10 mmol) with benzyl chloride (1.27 g, 10 mmol) and potassium carbonate (1.52 g, 11 mmol) according to the typical procedure, and purification by column chromatography (eluent: hexane/EtOAc, 20/1) on silica gel, gave **1b** (1.98 g, 72%) as a white solid: mp 43-45 °C; ^1H NMR (300 MHz, CDCl_3) δ 7.51-7.43 (m, 2H), 7.42-7.33 (m, 3H), 7.33-7.3 (m, 1H), 7.03 (dd, $J = 8.3$, 1.4 Hz, 1H), 6.84 (d, $J = 8.3$ Hz, 1H), 5.14 (s, 2H), 2.28 (s, 3H); ^{13}C NMR (75.4 MHz, CDCl_3) δ 153.0 (C), 136.9 (C), 133.9 (CH), 132.0 (C), 128.9 (CH), 128.7 (CH), 128.0 (CH), 127.1 (CH), 114.0 (CH), 112.3 (C), 71.1 (CH_2), 20.3 (CH_3); EI-LRMS m/z 278 ($\text{M}^+ + 2$, 8), 276 (M^+ , 8), 91 (100). HRMS calcd. for $\text{C}_{14}\text{H}_{13}\text{BrO}$: 276.0150. Found: 276.0138.

1-Benzyl 2-bromo-4-chlorophenyl ether (1c): Treatment of 2-bromo-4-chlorophenol (2.07 g, 10 mmol) with benzyl chloride (1.27 g, 10 mmol) and potassium carbonate (1.52 g, 11 mmol) according to the typical procedure, and purification by column chromatography (eluent: hexane/EtOAc, 20/1) on silica gel, gave **1c** (2.68 g, 90%) as a white solid: mp 50-52 °C; ^1H NMR (300 MHz, CDCl_3) δ 7.57 (d, J = 2.7 Hz, 1H), 7.50-7.31 (m, 5H), 7.2 (dd, J = 8.8, 2.7 Hz, 1H), 6.84 (d, J = 8.8 Hz, 1H), 5.14 (s, 2H); ^{13}C NMR (75.4 MHz, CDCl_3) δ 153.9 (C), 136.1 (C), 133.0 (CH), 128.7 (CH), 128.3 (CH), 128.2 (CH), 127.1 (CH), 126.4 (C), 114.5 (CH), 113.1 (C), 71.2 (CH_2); EI-LRMS m/z 300 (M^+ +4, 3), 298 (M^+ +2, 10), 296 (M^+ , 8), 91 (100). HRMS calcd. for $\text{C}_{13}\text{H}_{10}\text{BrClO}$: 295.9604. Found: 295.9617.

***p*-Methylbenzyl 2-iodophenyl ether (1d):** Treatment of 2-iodophenol (2.20 g, 10 mmol) with 4-methylbenzyl chloride (1.41 g, 10 mmol) and potassium carbonate (1.52 g, 11 mmol) according to the typical procedure, and purification by column chromatography (eluent: hexane/EtOAc, 20/1) on silica gel, gave **1d** (2.53 g, 78%) as a white solid: mp 44-46 °C; ^1H NMR (300 MHz, CDCl_3) δ 7.82 (dd, J = 7.6, 1.6 Hz, 1H), 7.42 (d, J = 8.1 Hz, 2H), 7.29 (td, J = 7.6, 1.6 Hz, 1H), 7.23 (d, J = 8.1 Hz, 2H), 6.88 (dd, J = 8.3, 1.3 Hz, 1H), 6.74 (td, J = 7.6, 1.3 Hz, 1H), 5.13 (s, 2H), 2.39 (s, 3H); ^{13}C NMR (75.4 MHz, CDCl_3) δ 157.3 (C), 139.6 (CH), 137.7 (C), 133.5 (C), 129.5 (CH), 129.3 (CH), 127.2 (CH), 122.8 (CH), 112.8 (CH), 87.0 (C), 70.8 (CH_2), 21.3 (CH_3); EI-LRMS m/z 324 (M^+ , 36), 105 (100). HRMS calcd. for $\text{C}_{14}\text{H}_{13}\text{IO}$: 324.0011. Found: 324.0022.

2-Iodophenyl 1-naphthylmethyl ether (1e): Treatment of 2-iodophenol (2.20 g, 10 mmol) with 1-(chloromethyl)naphthalene (1.77 g, 10 mmol) and potassium carbonate (1.52 g, 11 mmol) according to the typical procedure, and purification by column chromatography (eluent: hexane/EtOAc, 20/1) on silica gel, gave **1e** (2.95 g, 82%) as a white solid: mp 56-58 °C; ^1H NMR (300 MHz, CDCl_3) δ 8.13 (d, J = 8.0 Hz, 1H), 8.00-7.87 (m, 3H), 7.79 (d, J = 7.2 Hz, 1H), 7.68-7.50 (m, 3H), 7.34 (td, J = 7.5, 1.2 Hz, 1H), 7.00 (d, J = 8.0 Hz, 1H), 6.80 (td, J = 7.5, 1.2 Hz, 1H), 5.57 (s, 2H); ^{13}C NMR (75.4 MHz, CDCl_3) δ 157.2 (C), 139.6 (CH), 133.6 (C), 131.7 (C), 131.0 (C), 129.5 (CH), 128.8 (CH), 128.7 (CH), 126.4

(CH), 125.9 (CH), 125.4 (CH), 123.6 (CH), 122.9 (CH), 112.7 (CH), 86.9 (C), 69.3 (CH₂); EI-LRMS: m/z 360 (M^+ , 8), 141 (100). HRMS calcd. for C₁₇H₁₃IO: 360.0011. Found: 360.0025.

2-Bromophenyl 2-fluorobenzyl ether (1f): Treatment of 2-bromophenol (1.73 g, 10 mmol) with 2-fluorobenzyl chloride (1.45 g, 10 mmol) and potassium carbonate (1.52 g, 11 mmol) according to the typical procedure, and purification by column chromatography (eluent: hexane/EtOAc, 15/1) on silica gel, gave **1f** (2.56 g, 91%) as a colorless oil: R_f 0.35 (hexane/EtOAc, 20/1); ¹H NMR (300 MHz, CDCl₃) δ 7.68 (td, J = 7.4, 1.2 Hz, 1H), 7.62-7.57 (m, 1H), 7.38-7.18 (m, 3H), 7.1 (t, J = 8.7 Hz, 1H), 7.00 (d, J = 8.2 Hz, 1H), 6.92-6.85 (m, 1H), 5.2 (s, 2H); ¹³C NMR (75.4 MHz, CDCl₃) δ 160.2 (d, J = 247.0 Hz, C), 154.8 (C), 133.5 (CH), 129.6 (d, J = 8.4 Hz, CH), 129.3 (d, J = 4.0 Hz, CH), 128.6 (CH), 124.5 (d, J = 3.4 Hz, CH), 123.8 (d, J = 14.0 Hz, C), 122.4 (CH), 115.3 (d, J = 20.8 Hz, CH), 113.7 (CH), 112.5 (C), 64.5 (d, J = 4.9 Hz, CH₂); EI-LRMS: m/z 282 (M^+ +2, 9), 280 (M^+ , 9), 109 (100). HRMS calcd. for C₁₃H₁₀BrFO: 279.9899. Found: 279.9906.

2-Iodophenyl 2-methoxybenzyl ether (1g): Treatment of 2-iodophenol (2.20 g, 10 mmol) with 2-methoxybenzyl chloride (1.57 g, 10 mmol) and potassium carbonate (1.52 g, 11 mmol) according to the typical procedure, and purification by column chromatography (eluent: hexane/EtOAc, 20/1) on silica gel, gave **1g** (2.45 g, 72%) as a white solid: mp 73-75 °C; ¹H NMR (300 MHz, CDCl₃) δ 7.80 (dd, J = 7.5, 1.3 Hz, 1H), 7.68 (d, J = 7.5 Hz, 1H), 7.36-7.24 (m, 2H), 7.03 (t, J = 7.5 Hz, 1H), 6.95-6.85 (m, 2H), 6.73 (t, J = 7.5 Hz, 1H), 5.20 (s, 2H), 3.88 (s, 3H); ¹³C NMR (75.4 MHz, CDCl₃) δ 157.3 (C), 156.3 (C), 139.5 (CH), 129.5 (CH), 128.7 (CH), 128.0 (CH), 125.0 (C), 122.6 (CH), 120.8 (CH), 112.7 (CH), 110.0 (CH), 86.8 (C), 66.0 (CH₂), 55.4 (CH₃); EI-LRMS: m/z 340 (M^+ , 3), 121 (100). HRMS calcd. for C₁₄H₁₃IO₂: 339.9960. Found: 339.9978.

Benzyl 1-bromo-2-naphthyl ether (7a): Treatment of 1-bromo-2-naphthol (2.23 g, 10 mmol) with benzyl chloride (1.27 g, 10 mmol) and potassium carbonate (1.52 g, 11 mmol) according to the typical procedure, and purification by column chromatography (eluent: hexane/EtOAc, 20/1) on silica gel, gave **7a** (2.50 g, 80%) as a white solid: mp 101-103 °C; ¹H NMR (300 MHz, CDCl₃) δ 8.25 (d, J = 8.5 Hz,

1H), 7.8-7.74 (m, 2H), 7.61-7.57 (m, 1H), 7.56-7.51 (m, 2H), 7.45-7.37 (m, 3H), 7.37-7.32 (m, 1H), 7.30-7.25 (m, 1H), 5.32 (s, 2H); ¹³C NMR (75.4 MHz, CDCl₃) δ 153.1 (C), 136.8 (C), 133.3 (C), 130.2 (C), 129.0 (CH), 128.8 (CH), 128.2 (CH), 127.8 (CH), 127.31 (CH), 127.27 (CH), 126.4 (CH), 124.7 (CH), 115.7 (CH), 110.1 (C), 71.9 (CH₂); EI-LRMS *m/z* 314 (M⁺+2, 9), 312 (M⁺, 9), 91 (100). HRMS calcd. for C₁₇H₁₃BrO: 312.0150. Found: 312.0154.

Benzyl 2-bromo-1-naphthyl ether (7b): Treatment of 2-bromo-1-naphthol¹ (2.23 g, 10 mmol) with benzyl chloride (1.27 g, 10 mmol) and potassium carbonate (1.52 g, 11 mmol) according to the typical procedure, and purification by column chromatography (eluent: hexane/EtOAc, 20/1) on silica gel, gave **7b** (1.0 g, 32%) as a white solid: mp 48-50 °C; ¹H NMR (300 MHz, CDCl₃) δ 8.22-8.15 (m, 1H), 7.89-7.83 (m, 1H), 7.71-7.69 (m, 1H), 7.66 (d, *J* = 8.8 Hz, 2H), 7.56 (d, *J* = 3.0 Hz, 1H), 7.55-7.40 (m, 5H), 5.17 (s, 2H); ¹³C NMR (75.4 MHz, CDCl₃) δ 152.0 (C), 137.0 (C), 134.0 (C), 130.2 (CH), 129.4 (C), 128.7 (CH), 128.4 (CH), 128.3 (CH), 128.1 (CH), 126.9 (CH), 126.7 (CH), 125.6 (CH), 122.3 (CH), 113.3 (C), 75.7 (CH₂); EI-LRMS *m/z* 314 (M⁺+2, 9), 312 (M⁺, 9), 91 (100). HRMS calcd. for C₁₇H₁₃BrO: 312.0150. Found: 312.0138.

Typical Procedure for the Double Lithiation of Benzyl *o*-Halophenyl Ethers **1 and **7**. Synthesis of Ethers **4** and 3-Hydroxy-2,3-Dihydrobenzo[*b*]furans **5**.** To a stirred solution of the corresponding starting ether **1** or **7** (1 mmol) in dry THF (2 mL) kept at -78 °C under N₂ atmosphere, *t*-BuLi (2 mL of a 1.5 M solution in pentane, 3 mmol) was added and the mixture was stirred at -78 °C for 30 min. The solution was warmed to -25 °C and the stirring was continued at this temperature for 40 min. Then, the corresponding electrophile (2.1 mmol for the preparation of difunctionalized ethers **4** or 1 mmol when organic carboxylates were used as electrophiles) was added at -78 °C and stirring continued at low temperature for further 30 min. The reaction mixture was then allowed to warm to room temperature and after quenching with H₂O it was extracted with EtOAc (3 x 25 mL). The combined organic layers were dried (Na₂SO₄), and evaporated under reduced pressure. The crude product was purified by column chromatography (eluent: hexane/EtOAc) on silica gel to afford compounds **4** and **5**.

Dihydrobenzofuranols **5** were obtained as mixture of diastereoisomers and in some cases the major diastereoisomer could be isolated by column chromatography.

α -Deuteriobenzyl 2-deuteriophenyl ether (4a): Treatment of **1a** (0.310 g, 1 mmol) according to the typical procedure, using deuterium oxide (excess) as electrophile and purification by column chromatography (eluent: hexane/EtOAc, 50/1) on silica gel, gave **4a** (0.167 g, 90%) as a white solid: mp 40-42 °C; ^1H NMR (300 MHz, CDCl_3) δ 7.6-7.4 (m, 7H), 7.2-7.1 (m, 2H), 5.19-5.12 (m, 1H); ^{13}C NMR (75.4 MHz, CDCl_3) δ 158.8 (C), 137.1 (C), 129.5 (CH), 129.4 (CH), 128.6 (CH), 128.5 (CH), 128.0 (CH), 127.6 (CH), 121.0 (CH), 114.9 (CH), 114.5 (t, $J = 32$ Hz, CD), 69.6 (t, $J = 23$ Hz, CHD); EI- LRMS m/z 186 (M^+ , 15), 92 (100). HRMS calcd. for $\text{C}_{13}\text{H}_{10}\text{D}_2\text{O}$: 186.1012. Found: 186.1005.

α -Phenylthiobenzyl 2-phenylthiophenyl ether (4b): Treatment of **1a** (0.310 g, 1 mmol) according to the typical procedure, using diphenyl disulfide (0.458 g, 2.1 mmol) as electrophile and purification by column chromatography (eluent: hexane/EtOAc, 20/1) on silica gel, gave **4b** (0.292 g, 73%) as a white solid: mp 52-54 °C; ^1H NMR (300 MHz, CDCl_3) δ 7.46-7.32 (m, 8H), 7.31-7.22 (m, 5H), 7.22-7.12 (m, 4H), 6.96-6.88 (m, 2H), 6.64 (s, 1H); ^{13}C NMR (75.4 MHz, CDCl_3) δ 154.6 (C), 138.2 (C), 134.9 (C), 134.7 (CH), 132.1 (CH), 131.9 (C), 131.8 (CH), 129.3 (CH), 128.7 (CH), 128.3 (CH), 128.3 (CH), 128.2 (CH), 128.0 (CH), 127.2 (CH), 126.3 (CH), 126.0 (C), 122.3 (CH), 114.8 (CH), 87.1 (CH); EI- LRMS m/z 400 (M^+ , 32), 197 (100). HRMS calcd. for $\text{C}_{25}\text{H}_{20}\text{OS}_2$: 400.0956. Found: 400.0969.

3-Hydroxy-3-methyl-2-phenyl-2,3-dihydrobenzo[*b*]furan (5a)²: Treatment of **1a** (0.310 g, 1 mmol) according to the typical procedure, using *t*-butyl acetate (0.116 g, 1 mmol) as electrophile and purification by column chromatography (eluent: hexane/EtOAc, 5/1) on silica gel, gave **5a** (0.147 g, 65%) as a colorless oil: R_f 0.21 (hexane/EtOAc, 5/1). Isolated as a (1.3:1, *trans*:*cis*) mixture of diastereoisomers; ^1H NMR (300 MHz, CDCl_3) δ 7.50-6.96 (m, ArH), 5.47 (s, 1H, *trans*), 5.32 (s, 1H, *cis*), 2.85 (s, OH, *trans*), 1.73 (s, 3H, *cis*), 1.70 (s, OH, *cis*), 1.10 (s, 3H, *trans*); ^{13}C NMR (75.4 MHz, CDCl_3) δ 159.3 (C), 159.0 (C), 137.3 (C), 134.8 (C), 132.5 (C), 131.9 (CH), 130.3 (CH), 130.2 (CH), 128.7 (C), 128.5 (CH), 128.3 (CH), 128.1 (CH), 127.1 (CH), 126.0 (CH), 123.9 (CH), 123.7 (CH),

121.4 (CH), 110.7 (CH), 110.4 (CH), 94.8 (CH), 92.9 (CH), 81.5 (C), 78.2 (C), 25.0 (CH₃), 24.3 (CH₃); EI-LRMS m/z 226 (M⁺, 52), 91 (100). HRMS calcd. for C₁₅H₁₄O₂: 226.0994. Found: 226.0985. HPLC (hexane): t_R = 4.54 min, 4.84 min.

***trans*-3-Hydroxy-2-phenyl-3-propyl-2,3-dihydrobenzo[*b*]furan (5b):** Treatment of **1a** (0.310 g, 1 mmol) according to the typical procedure, using ethyl butyrate (0.116 g, 1 mmol) as electrophile and purification by column chromatography (eluent: hexane/Et₂O, 5/1) on silica gel, gave the major *trans*-diastereoisomer ***trans*-5b** (0.163 g, 64%) as a colorless oil: R_f 0.27 (hexane/Et₂O, 3/1); ¹H NMR (300 MHz, CDCl₃) δ 7.48-7.43 (m, 2H), 7.41-7.37 (m, 3H), 7.37-7.28 (m, 2H), 7.04-6.97 (m, 2H), 5.47 (s, 1H), 2.84 (s, OH), 1.52-1.33 (m, 2H), 1.30-1.18 (m, 2H), 0.67 (t, J = 6.9 Hz, 3H); ¹³C NMR (75.4 MHz, CDCl₃) δ 158.9 (C), 136.5 (C), 132.0 (C), 129.9 (CH), 128.2 (CH), 128.1 (CH), 126.3 (CH), 124.6 (CH), 121.1 (CH), 110.7 (CH), 94.8 (CH), 83.5 (C), 39.0 (CH₂), 16.1 (CH₂), 14.3 (CH₃); EI-LRMS m/z 236 (M⁺-H₂O, 58), 207 (100). HRMS calcd. for C₁₇H₁₆O (M⁺-H₂O): 236.1201. Found: 236.1194. HPLC (hexane/2-PrOH, 99.6/0.4): t_R = 3.52 min.

***trans*-3-Hydroxy-3-isopropyl-2-phenyl-2,3-dihydrobenzo[*b*]furan (5c):** Treatment of **1a** (0.310 g, 1 mmol) according to the typical procedure, using ethyl isobutyrate (0.116 g, 1 mmol) as electrophile and purification by column chromatography (eluent: hexane/EtOAc, 5/1) on silica gel, gave the major *trans*-diastereoisomer ***trans*-5c** (0.102 g, 40%) as a colorless oil: R_f 0.44 (hexane/EtOAc, 5/1); ¹H NMR (400 MHz, CDCl₃) δ 7.63 (d, J = 7.6 Hz, 2H), 7.45-7.31 (m, 5H), 7.04-6.96 (m, 2H), 5.56 (s, 1H), 2.45 (s, OH), 1.81 (sept., J = 6.6 Hz, 1H), 0.97 (d, J = 6.6 Hz, 3H), 0.42 (d, J = 6.6 Hz, 3H); ¹³C NMR (75.4 MHz, CDCl₃) δ 159.3 (C), 136.1 (C), 130.4 (C), 129.9 (CH), 128.2 (CH), 127.7 (CH), 126.0 (CH), 125.0 (CH), 121.1 (CH), 110.9 (CH), 94.4 (CH), 86.0 (C), 33.0 (CH), 16.6 (CH₃), 16.4 (CH₃); EI-LRMS m/z 254 (M⁺, 4), 211 (100). HRMS calcd. for C₁₇H₁₈O₂: 254.1307. Found: 254.1316. HPLC (hexane/2-PrOH, 99.6/0.4): t_R = 3.94 min.

***trans*-3-Hydroxy-3-(2-methylpropyl)-2-phenyl-2,3-dihydrobenzo[*b*]furan (5d):** Treatment of **1a** (0.310 g, 1 mmol) according to the typical procedure, using ethyl isovalerate (0.130 g, 1 mmol) as

electrophile and purification by column chromatography (eluent: hexane/EtOAc, 5/1) on silica gel, gave the major *trans*-diastereoisomer **trans-5d** (0.126 g, 47%) as a white solid: mp 74-76 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.45-7.41 (m, 2H), 7.40-7.32 (m, 4H), 7.31-7.24 (m, 1H), 7.02-6.94 (m, 2H), 5.45 (s, 1H), 2.22 (br s, OH), 1.73-1.62 (m, 1H), 1.42 (dd, *J* = 14.6, 4.2 Hz, 1H), 1.07 (dd, *J* = 14.6, 7.6 Hz, 1H), 0.73 (d, *J* = 6.8 Hz, 3H), 0.65 (d, *J* = 6.8 Hz, 3H); ¹³C NMR (75.4 MHz, CDCl₃) δ 159.0 (C), 136.5 (C), 132.3 (C), 130.0 (CH), 128.3 (CH), 128.1 (CH), 126.4 (CH), 124.7 (CH), 121.2 (CH), 110.9 (CH), 95.6 (CH), 84.3 (C), 45.0 (CH₂), 25.0 (CH), 24.0 (CH₃), 23.6 (CH₃); EI-LRMS *m/z* 250 (M⁺-H₂O, 47), 207 (100). HRMS calcd. for C₁₈H₂₀O₂: 268.1463. Found: 268.1465. HPLC (hexane/2-PrOH, 99.6/0.4): *t*_R = 3.99 min.

3-*tert*-Butyl-3-hydroxy-2-phenyl-2,3-dihydrobenzo[*b*]furan (5e): Treatment of **1a** (0.310 g, 1 mmol) according to the typical procedure, using ethyl pivalate (0.130 g, 1 mmol) as electrophile and purification by column chromatography (eluent: hexane/EtOAc, 7/1) on silica gel, gave **cis-5e** (0.129 g, 48%) and **trans-5e** (0.040 g, 15%).

Data for **cis-5e**: White solid; mp 72-74 °C; ¹H NMR (300 MHz, CDCl₃) δ 7.36-7.28 (m, 5H), 7.26-7.21 (m, 2H), 7.02-6.94 (m, 2H), 5.8 (s, 1H), 1.37 (br s, 1H), 1.15 (s, 9H); ¹³C NMR (75.4 MHz, CDCl₃) δ 160.0 (C), 137.0 (C), 130.1 (CH), 129.2 (C), 128.6 (CH), 128.2 (CH), 126.71 (CH), 126.65 (CH), 120.7 (CH), 109.7 (CH), 87.5 (CH), 87.0 (C), 38.6 (C), 24.5 (CH₃); EI-LRMS *m/z* 250 (M⁺-H₂O, 42), 235 (100). HRMS calcd. for C₁₈H₂₀O₂: 268.1463. Found: 268.1455. HPLC (hexane/2-PrOH, 99.4/0.6): *t*_R = 6.97 min.

Data for **trans-5e**: Colorless oil; *R*_f 0.36 (hexane/EtOAc, 5/1); ¹H NMR (300 MHz, CDCl₃) δ 7.62 (d, *J* = 5.8 Hz, 2H), 7.40-7.02 (m, 7H), 5.53 (s, 1H), 2.34 (br s, 1H), 0.74 (s, 9H); ¹³C NMR (75.4 MHz, CDCl₃) δ 159.5 (C), 136.9 (C), 132.5 (C), 129.6 (CH), 128.4 (CH), 128.1 (CH), 125.8 (CH), 120.8 (CH), 110.9 (CH), 95.6 (CH), 87.7 (C), 38.3 (C), 25.3 (CH₃); EI-LRMS *m/z* 250 (M⁺-H₂O, 4), 133 (100).

3-Hydroxy-2-phenyl-3-(2-thienyl)-2,3-dihydrobenzo[*b*]furan (5f): Treatment of **1a** (0.310 g, 1 mmol) according to the typical procedure, using ethyl 2-thiophenecarboxylate (0.156 g, 1 mmol) as electrophile and purification by column chromatography (eluent: hexane/EtOAc, 5/1) on silica gel, gave **5f** (0.191 g, 65%) as a colorless oil; R_f 0.67 (hexane/EtOAc, 3/1). Isolated as a (2.5:1, *trans*:*cis*) mixture of diastereoisomers; ^1H NMR (300 MHz, CDCl_3) δ 7.50-7.00 (m, ArH), 6.73-6.66 (m, 1H), 6.32-6.27 (m, 1H), 5.8 (s, 1H, *cis*), 5.74 (s, 1H, *trans*), 3.34 (s, OH, *trans*), 2.43 (s, OH, *cis*); ^{13}C NMR (75.4 MHz, CDCl_3) δ 160.2 (C), 159.5 (C), 147.2 (C), 145.1 (C), 136.6 (C), 133.4 (C), 131.9 (C), 131.3 (CH), 131.1 (CH), 131.0 (C), 128.9 (CH), 128.4 (CH), 127.9 (CH), 127.7 (CH), 127.3 (CH), 127.2 (CH), 126.31 (CH), 126.27 (CH), 126.2 (CH), 125.9 (CH), 125.6 (CH), 125.3 (CH), 125.21 (CH), 125.17 (CH), 121.9 (CH), 121.8 (CH), 110.8 (CH), 110.7 (CH), 95.5 (CH), 94.9 (CH), 85.6 (C), 81.6 (C); EI-LRMS m/z 276 (M^+ , 100). HRMS calcd. for $\text{C}_{18}\text{H}_{14}\text{O}_2\text{S}$: 294.0714. Found: 294.0706. HPLC (hexane/2-PrOH, 99.4/0.6): t_R = 4.33 min, 5.36 min.

3,5-Dimethyl-3-hydroxy-2-phenyl-2,3-dihydrobenzo[*b*]furan (5g): Treatment of **1b** (0.277 g, 1 mmol) according to the typical procedure, using *t*-butyl acetate (0.116 g, 1 mmol) as electrophile and purification by column chromatography (eluent: hexane/EtOAc, 5/1) on silica gel, gave *cis*-**5g** (0.046 g, 19%) and *trans*-**5g** (0.103 g, 43%).

Data for *cis*-**5g**: Colorless oil; R_f 0.63 (hexane/EtOAc, 2/1); ^1H NMR (300 MHz, CDCl_3) δ 7.50-7.33 (m, 5H), 7.19 (d, J = 1.2 Hz, 1H), 7.12 (dd, J = 8.3, 1.2 Hz, 1H), 6.84 (d, J = 8.3 Hz, 1H), 5.31 (s, 1H), 2.36 (s, 3H), 1.72 (s, 3H), 1.4 (s, OH); ^{13}C NMR (75.4 MHz, CDCl_3) δ 157.3 (C), 135.0 (C), 131.9 (C), 130.9 (CH), 128.8 (CH), 128.6 (CH), 127.1 (CH), 124.3 (CH), 110.3 (CH), 93.1 (CH), 78.4 (C), 25.2 (CH_3), 21.0 (CH_3); EI-LRMS m/z 240 (M^+ , 57), 91 (100). HRMS calcd. for $\text{C}_{16}\text{H}_{16}\text{O}_2$: 240.1150. Found: 240.1158. HPLC (hexane/2-PrOH, 99.4/0.6): t_R = 4.60 min.

Data for *trans*-**5g**: Colorless oil; R_f 0.55 (hexane/EtOAc, 2/1); ^1H NMR (300 MHz, CDCl_3) δ 7.40-7.25 (m, 5H), 7.15-7.05 (m, 2H), 6.90-6.85 (m, 1H), 5.48 (s, 1H), 2.68 (br s, OH), 2.34 (s, 3H), 1.10 (s, 3H); ^{13}C NMR (75.4 MHz, CDCl_3) δ 157.0 (C), 137.5 (C), 132.4 (C), 130.89 (CH), 130.86 (CH), 128.4

(CH), 128.1 (CH), 126.1 (CH), 124.0 (C), 110.1 (CH), 95.1 (CH), 81.8 (C), 24.3 (CH₃), 21.0 (CH₃); EI-LRMS m/z 222 (M⁺-H₂O, 100). HPLC (hexane/2-PrOH, 99.4/0.6): t_R = 4.31 min.

5-Chloro-3-hydroxy-3-(2-methylpropyl)-2-phenyl-2,3-dihydrobenzo[*b*]furan (5h): Treatment of **1c** (0.297 g, 1 mmol) according to the typical procedure, using ethyl isovalerate (0.130 g, 1 mmol) as electrophile and purification by column chromatography (eluent: hexane/EtOAc, 5/1) on silica gel, gave **5h** (0.160 g, 53%) as a colorless oil: R_f 0.42 (hexane/EtOAc, 3/1). Isolated as a (5:1, *trans:cis*) mixture of diastereoisomers. Data for the *trans*-diastereoisomer: ¹H NMR (300 MHz, CDCl₃) δ 7.50-6.84 (m, 7H), 6.88 (d, J = 8.5 Hz, 1H), 5.44 (s, 1H), 2.42 (s, OH), 1.72-1.58 (m, 1H), 1.42 (dd, J = 14.4, 4.0 Hz, 1H), 1.06 (dd, J = 14.4, 7.6 Hz, 1H), 0.75 (d, J = 6.3 Hz, 3H), 0.67 (d, J = 6.3 Hz, 3H); ¹³C NMR (75.4 MHz, CDCl₃) δ 157.5 (C), 135.9 (C), 134.1 (C), 129.9 (CH), 128.7 (CH), 128.4 (CH), 127.1 (C), 126.3 (CH), 126.0 (C), 112.0 (CH), 96.0 (CH), 84.2 (C), 44.9 (CH₂), 25.0 (CH), 23.9 (CH₃), 23.5 (CH₃); EI-LRMS: m/z 302 (M⁺, 16), 91 (100). HRMS calcd. for C₁₈H₁₉ClO₂: 302.1074. Found: 302.1062.

5-Chloro-2,3-diphenyl-3-hydroxy-2,3-dihydrobenzo[*b*]furan (5i): Treatment of **1c** (0.297 g, 1 mmol) according to the typical procedure, using ethyl benzoate (0.150 g, 1 mmol) as electrophile and purification by column chromatography (eluent: hexane/EtOAc, 5/1) on silica gel, gave *cis*-**5i** (0.103 g, 32%) and *trans*-**5i** (0.074 g, 23%).

Data for *cis*-**5i**: Colorless oil; R_f 0.31 (hexane/EtOAc, 5/1); ¹H NMR (300 MHz, CDCl₃) δ 7.46-7.34 (m, 8H), 7.31 (dd, J = 8.5, 2.2 Hz, 1H), 7.24-7.16 (m, 2H), 7.06 (d, J = 2.2 Hz, 1H), 7.02 (d, J = 8.5 Hz, 1H), 5.72 (s, 1H), 2.05 (br s, OH); ¹³C NMR (75.4 MHz, CDCl₃) δ 158.7 (C), 142.0 (C), 134.5 (C), 133.3 (C), 130.6 (CH), 129.0 (CH), 128.6 (CH), 128.5 (CH), 128.0 (CH), 126.9 (CH), 126.6 (C), 125.6 (CH), 111.9 (CH), 95.8 (CH), 83.0 (C); EI-LRMS m/z 322 (M⁺, 67), 91 (100). HRMS calcd. for C₂₀H₁₅ClO₂: 322.0761. Found: 322.0765.

Data for *trans*-**5i**: Colorless oil; R_f 0.19 (hexane/EtOAc, 5/1); ¹H NMR (300 MHz, CDCl₃) δ 7.35 (dd, J = 8.6, 2.2 Hz, 1H), 7.12 (d, J = 2.2 Hz, 1H), 7.08-7.00 (m, 7H), 6.96-6.87 (m, 4H), 5.77 (s, 1H), 2.87 (s, OH); ¹³C NMR (75.4 MHz, CDCl₃) δ 159.2 (C), 139.8 (C), 136.4 (C), 133.9 (C), 130.8 (CH),

127.79 (CH), 127.76 (CH), 127.6 (CH), 127.6 (CH), 127.0 (CH), 126.6 (C), 126.2 (CH), 125.3 (CH), 111.9 (CH), 96.7 (CH), 87.0 (C); EI-LRMS m/z 322 (M^+ , 63), 91 (100). HRMS calcd. for $C_{20}H_{15}ClO_2$: 322.0761. Found: 322.0774. HPLC (hexane/2-PrOH, 99.4/0.6): t_R = 7.98 min.

Typical Procedure for the Synthesis of 2-Aryl-3-substituted Benzo[*b*]furans 6, Naphtho[2,1-*b*]furans 8 and Naphtho[1,2-*b*]furans 9. The procedure was the same as for the preparation of dihydrobenzofuranols 5 but after the addition of the organic carboxylate at low temperature and further evolution to room temperature, conc. HCl was added and the mixture was stirred at room temperature until complete dehydration (TLC and GC-MS analysis) of the intermediates 5. The crude product was purified by column chromatography (eluent: hexane/EtOAc) on silica gel to afford compounds 6 and 8,9. Alternatively, after the addition of the ester and evolution to room temperature, the mixture was quenched with water, extracted and the residue was dissolved in CH_2Cl_2 (2 mL). $InCl_3$ (5 mol%) was added and the resulting mixture was stirred at room temperature until consumption of the intermediate alcohols 5 (TLC and GC-MS analysis). The crude was purified by column chromatography on silica gel.

3-Methyl-2-phenylbenzo[*b*]furan (6a)⁴: Treatment of 1a (0.310 g, 1 mmol) according to the typical procedure, using *t*-butyl acetate (0.116 g, 1 mmol) as electrophile and purification by column chromatography (eluent: hexane) on silica gel, gave 6a (0.121 g, 58%) as a colorless oil (lit.⁵ mp 35-36 °C): R_f 0.40 (hexane/EtOAc, 50/1); 1H NMR (300 MHz, $CDCl_3$) δ 7.93-7.88 (m, 2H), 7.64-7.52 (m, 4H), 7.46-7.30 (m, 3H), 2.55 (s, 3H); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ 153.9 (C), 150.8 (C), 131.5 (C), 131.3 (C), 128.7 (CH), 128.0 (CH), 126.8 (CH), 124.4 (CH), 122.4 (CH), 119.4 (CH), 111.4 (C), 111.0 (CH), 9.6 (CH_3); EI-LRMS m/z 208 (M^+ , 100), 178 (23). HRMS calcd. for $C_{15}H_{12}O$: 208.0888. Found: 208.0884. HPLC (hexane): t_R = 6.45 min.

2-Phenyl-3-propylbenzo[*b*]furan (6b): Treatment of 1a (0.310 g, 1 mmol) according to the typical procedure, using ethyl butyrate (0.116 g, 1 mmol) as electrophile and purification by column chromatography (eluent: hexane) on silica gel, gave 6b (0.172 g, 73%) as a colorless oil: R_f 0.40 (hexane/EtOAc, 50/1); 1H NMR (300 MHz, $CDCl_3$) δ 7.90-7.82 (m, 2H), 7.66-7.60 (m, 1H), 7.56-7.49

(m, 3H), 7.46-7.39 (m, 1H), 7.39-7.25 (m, 2H), 2.97 (t, $J = 7.8$ Hz, 2H), 1.92-1.80 (m, 2H), 1.12 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (75.4 MHz, CDCl_3) δ 154.0 (C), 150.8 (C), 131.6 (C), 130.8 (C), 128.8 (CH), 128.1 (CH), 127.0 (CH), 124.4 (CH), 122.4 (CH), 119.8 (CH), 116.5 (C), 111.2 (CH), 26.4 (CH_2), 23.2 (CH_2), 14.5 (CH_3); EI-LRMS m/z 236 (M^+ , 57), 207 (100). HRMS calcd. for $\text{C}_{17}\text{H}_{16}\text{O}$: 236.1201. Found: 236.1192. HPLC (hexane): $t_{\text{R}} = 6.03$ min.

3-Isopropyl-2-phenylbenzo[*b*]furan (6c): Treatment of **1a** (0.310 g, 1 mmol) according to the typical procedure, using ethyl isobutyrate (0.116 g, 1 mmol) as electrophile and purification by column chromatography (eluent: hexane) on silica gel, gave **6c** (0.168 g, 71%) as a white solid: mp 63-65 °C; ^1H NMR (300 MHz, CDCl_3) δ 7.88-7.83 (m, 1H), 7.83-7.77 (m, 2H), 7.64-7.52 (m, 3H), 7.50-7.43 (m, 1H), 7.42-7.28 (m, 2H), 3.59 (sept., $J = 7.1$ Hz, 1H), 1.59 (d, $J = 7.1$ Hz, 6H); ^{13}C NMR (75.4 MHz, CDCl_3) δ 154.6 (C), 150.1 (C), 131.5 (C), 128.68 (CH), 128.65 (C), 128.4 (CH), 128.1 (CH), 124.0 (CH), 122.1 (CH), 121.8 (C), 121.5 (CH), 111.5 (CH), 25.7 (CH), 22.5 (CH_3); EI-LRMS m/z 236 (M^+ , 54), 221 (100). HRMS calcd. for $\text{C}_{17}\text{H}_{16}\text{O}$: 236.1201. Found: 236.1208. Anal. Calcd. for $\text{C}_{17}\text{H}_{16}\text{O}$: C, 86.40; H, 6.82. Found: C, 86.17; H, 6.81.

3-(2-Methylpropyl)-2-phenylbenzo[*b*]furan (6d): Treatment of **1a** (0.310 g, 1 mmol) according to the typical procedure, using ethyl isovalerate (0.130 g, 1 mmol) as electrophile and purification by column chromatography (eluent: hexane) on silica gel, gave **6d** (0.115 g, 46%) as a colorless oil: R_f 0.51 (hexane/EtOAc, 50/1); ^1H NMR (300 MHz, CDCl_3) δ 7.92-7.85 (m, 2H), 7.65-7.59 (m, 1H), 7.58-7.48 (m, 3H), 7.45-7.37 (m, 1H), 7.37-7.25 (m, 2H), 2.87 (d, $J = 7.1$ Hz, 2H), 2.2-2.30 (m, 1H), 1.06 (d, $J = 6.7$ Hz, 6H); ^{13}C NMR (75.4 MHz, CDCl_3) δ 153.9 (C), 151.2 (C), 131.7 (C), 131.1 (C), 128.7 (CH), 128.1 (CH), 127.0 (CH), 124.3 (CH), 122.4 (CH), 120.2 (CH), 115.9 (C), 111.1 (CH), 33.4 (CH_2), 29.3 (CH), 23.0 (CH_3); EI-LRMS m/z 250 (M^+ , 42), 207 (100). HRMS calcd. for $\text{C}_{18}\text{H}_{18}\text{O}$: 250.1358. Found: 250.1346. HPLC (hexane): $t_{\text{R}} = 5.82$ min.

3-Cyclopropyl-2-phenylbenzo[*b*]furan (6e): Treatment of **1a** (0.310 g, 1 mmol) according to the typical procedure, using ethyl cyclopropanecarboxylate (0.114 g, 1 mmol) as electrophile and

purification by column chromatography (eluent: hexane) on silica gel, gave **6e** (0.150 g, 64%) as a white solid: mp 34-36 °C; ^1H NMR (300 MHz, CDCl_3) δ 8.26-8.20 (m, 2H), 7.92-7.86 (m, 1H), 7.70-7.61 (m, 3H), 7.56-7.50 (m, 1H), 7.49-7.38 (m, 2H), 2.24-2.10 (m, 1H), 1.27-1.16 (m, 2H), 0.93-0.83 (m, 2H); ^{13}C NMR (75.4 MHz, CDCl_3) δ 153.8 (C), 152.6 (C), 131.0 (C), 130.9 (C), 128.4 (CH), 128.0 (CH), 127.1 (CH), 124.4 (CH), 122.5 (CH), 120.2 (CH), 116.8 (C), 111.1 (CH), 7.0 (CH_2), 5.9 (CH); EI-LRMS m/z 234 (M^+ , 100). HRMS calcd. for $\text{C}_{17}\text{H}_{14}\text{O}$: 234.1045. Found: 234.1040. Anal. Calcd. for $\text{C}_{17}\text{H}_{14}\text{O}$: C, 87.15; H, 6.02. Found: C, 87.00; H, 6.04.

2-Phenyl-3-(2-thienyl)benzo[*b*]furan (6f)⁶: Treatment of **1a** (0.310 g, 1 mmol) according to the typical procedure, using ethyl 2-thiophenecarboxylate (0.156 g, 1 mmol) as electrophile and purification by column chromatography (eluent: hexane) on silica gel, gave **6f** (0.149 g, 54%) as a white solid: mp 81-83 °C; ^1H NMR (300 MHz, CDCl_3) δ 7.82-7.74 (m, 2H), 7.64 (dd, $J = 7.7, 1.4$ Hz, 1H), 7.57 (dd, $J = 8.0, 0.8$ Hz, 1H), 7.45 (dd, $J = 5.0, 1.1$ Hz, 1H), 7.43-7.33 (m, 4H), 7.33-7.26 (m, 1H), 7.24-7.16 (m, 2H); ^{13}C NMR (75.4 MHz, CDCl_3) δ 153.9 (C), 151.8 (C), 133.4 (C), 130.4 (C), 130.3 (C), 128.9 (CH), 128.6 (CH), 127.8 (CH), 127.6 (CH), 127.4 (CH), 126.3 (CH), 125.0 (CH), 123.3 (CH), 120.3 (CH), 111.2 (CH), 110.8 (C); EI-LRMS m/z 276 (M^+ , 100). HRMS calcd. for $\text{C}_{18}\text{H}_{12}\text{OS}$: 276.0609. Found: 276.0617. HPLC (hexane): $t_R = 7.63$ min.

3,5-Dimethyl-2-phenylbenzo[*b*]furan (6g): Treatment of **1b** (0.277 g, 1 mmol) according to the typical procedure, using *t*-butyl acetate (0.116 g, 1 mmol) as electrophile and purification by column chromatography (eluent: hexane) on silica gel, gave **6g** (0.118 g, 53%) as a white solid: mp 69-71 °C (lit.⁷ mp 69.5-70 °C); ^1H NMR (300 MHz, CDCl_3) δ 7.94-7.88 (m, 2H), 7.60-7.53 (m, 2H), 7.50-7.44 (m, 2H), 7.41 (s, 1H), 7.20 (d, $J = 8.4$ Hz, 1H), 2.58 (s, 3H), 2.54 (s, 3H); ^{13}C NMR (75.4 MHz, CDCl_3) δ 152.3 (C), 150.8 (C), 131.8 (C), 131.7 (C), 131.3 (C), 128.7 (CH), 127.8 (CH), 126.7 (CH), 125.7 (CH), 119.2 (CH), 111.2 (C), 110.5 (CH), 21.5 (CH_3), 9.6 (CH_3); EI-LRMS: m/z 222 (M^+ , 100). HRMS calcd. for $\text{C}_{16}\text{H}_{14}\text{O}$: 222.1045. Found: 222.1036. Anal. Calcd. for $\text{C}_{16}\text{H}_{14}\text{O}$: C, 86.45; H, 6.35. Found: C, 86.62; H, 6.33.

3-Cyclopropyl-5-methyl-2-phenylbenzo[*b*]furan (6h): Treatment of **1b** (0.277 g, 1 mmol) according to the typical procedure, using ethyl cyclopropanecarboxylate (0.114 g, 1 mmol) as electrophile and purification by column chromatography (eluent: hexane) on silica gel, gave **6h** (0.112 g, 45%) as a white solid: mp 48-50 °C; ¹H NMR (300 MHz, CDCl₃) δ 8.15-8.05 (m, 2H), 7.60-7.50 (m, 3H), 7.48-7.40 (m, 2H), 7.17 (dd, *J* = 8.4, 1.9 Hz, 1H), 2.56 (s, 3H), 2-2.16 (m, 1H), 1.20-1.08 (m, 2H), 0.84-0.72 (m, 2H); ¹³C NMR (75.4 MHz, CDCl₃) δ 152.8 (C), 152.2 (C), 131.8 (C), 131.2 (C), 131.0 (C), 128.4 (CH), 128.0 (CH), 127.1 (CH), 125.5 (CH), 120.1 (CH), 116.6 (C), 110.6 (CH), 21.6 (CH₃), 7.1 (CH₂), 6.0 (CH); EI-LRMS: *m/z* 248 (M⁺, 100). HRMS calcd. for C₁₈H₁₆O: 248.1201. Found: 248.1192. Anal. Calcd. for C₁₈H₁₆O: C, 87.06; H, 6.49. Found: C, 86.91; H, 6.51.

5-Chloro-3-methyl-2-phenylbenzo[*b*]furan (6i): Treatment of **1c** (0.297 g, 1 mmol) according to the typical procedure, using *t*-butyl acetate (0.116 g, 1 mmol) as electrophile and purification by column chromatography (eluent: hexane) on silica gel, gave **6i** (0.155 g, 64%) as a white solid: mp 78-80 °C (lit.⁸ mp 80-81 °C); ¹H NMR (300 MHz, CDCl₃) δ 7.83-7.70 (m, 2H), 7.53-7.45 (m, 3H), 7.42-7.35 (m, 2H), 7.24 (dd, *J* = 9.0, 2.1 Hz, 1H), 2.44 (s, 3H); ¹³C NMR (75.4 MHz, CDCl₃) δ 152.2 (C), 132.6 (C), 131.0 (C), 128.8 (CH), 128.3 (CH), 128.1 (C), 126.8 (CH), 124.5 (CH), 119.0 (CH), 112.0 (CH), 111.0 (C), 9.5 (CH₃); EI-LRMS: *m/z* 242 (M⁺, 100). HRMS calcd. for C₁₅H₁₁ClO: 242.0498. Found: 242.0488. Anal. Calcd. for C₁₅H₁₁ClO: C, 74.23; H, 4.57. Found: C, 74.44; H, 4.56.

5-Chloro-2-phenyl-3-propylbenzo[*b*]furan (6j): Treatment of **1c** (0.297 g, 1 mmol) according to the typical procedure, using ethyl butyrate (0.116 g, 1 mmol) as electrophile and purification by column chromatography (eluent: hexane) on silica gel, gave **6j** (0.165 g, 61%) as a white solid: mp 65-67 °C; ¹H NMR (300 MHz, CDCl₃) δ 7.85-7.77 (m, 2H), 7.58-7.48 (m, 3H), 7.46-7.38 (m, 2H), 7.27 (dd, *J* = 8.6, 2.2 Hz, 1H), 2.56 (t, *J* = 7.6 Hz, 2H), 1.90-1.73 (m, 2H), 1.09 (t, *J* = 7.5 Hz, 3H); ¹³C NMR (75.4 MHz, CDCl₃) δ 152.4 (C), 152.2 (C), 132.2 (C), 131.0 (C), 128.8 (CH), 128.5 (CH), 128.0 (C), 127.0 (CH), 124.4 (CH), 119.4 (CH), 116.1 (C), 112.1 (CH), 26.2 (CH₂), 23.0 (CH₂), 14.4 (CH₃); EI-LRMS: *m/z* 270

(M^+ , 55), 241 (100). HRMS calcd. for $C_{17}H_{15}ClO$: 270.0811. Found: 270.0807. Anal. Calcd. for $C_{17}H_{15}ClO$: C, 75.41; H, 5.58. Found: C, 75.66; H, 5.60.

3-Isopropyl-2-*p*-tolylbenzo[*b*]furan (6k): Treatment of **1d** (0.324 g, 1 mmol) according to the typical procedure, using ethyl isobutyrate (0.116 g, 1 mmol) as electrophile and purification by column chromatography (eluent: hexane) on silica gel, gave **6k** (0.175 g, 70%) as a white solid: mp 61-63 °C; 1H NMR (300 MHz, $CDCl_3$) δ 7.78 (dd, $J = 7.0, 1.3$ Hz, 1H), 7.62 (d, $J = 8.0$ Hz, 2H), 7.52 (dd, $J = 7.6, 1.3$ Hz, 1H), 7.31 (d, $J = 7.6$ Hz, 2H), 7.28-7.20 (m, 2H), 3.49 (sept., $J = 7.2$ Hz, 1H), 2.45 (s, 3H), 1.52 (d, $J = 7.2$ Hz, 6H); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ 154.5 (C), 150.3 (C), 138.4 (C), 129.4 (CH), 128.73 (C), 128.66 (C), 128.0 (CH), 123.8 (CH), 122.0 (CH), 121.4 (CH), 121.3 (C), 111.4 (CH), 25.7 (CH), 22.5 (CH_3), 21.5 (CH_3); EI-LRMS: m/z 250 (M^+ , 53), 235 (100). HRMS calcd. for $C_{18}H_{18}O$: 250.1358. Found: 250.1370. Anal. Calcd. for $C_{18}H_{18}O$: C, 86.36; H, 7.25. Found: C, 86.18; H, 7.27.

3-(2-Thienyl)-2-*p*-tolylbenzo[*b*]furan (6l): Treatment of **1d** (0.324 g, 1 mmol) according to the typical procedure, using ethyl 2-thiophenecarboxylate (0.156 g, 1 mmol) as electrophile and purification by column chromatography (eluent: hexane) on silica gel, gave **6l** (0.168 g, 58%) as a white solid: mp 73-75 °C; 1H NMR (300 MHz, $CDCl_3$) δ 7.67 (d, $J = 8.2$ Hz, 2H), 7.63 (dd, $J = 7.8, 1.3$ Hz, 1H), 7.58 (d, $J = 7.8$ Hz, 1H), 7.45 (dd, $J = 4.9, 1.3$ Hz, 1H), 7.35 (td, $J = 7.8, 1.3$ Hz, 1H), 7.27 (td, $J = 7.4, 1.2$ Hz, 1H), 7.23-7.15 (m, 4H), 2.39 (s, 3H); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ 153.8 (C), 152.1 (C), 138.9 (C), 133.6 (C), 130.4 (C), 129.3 (CH), 127.7 (CH), 127.5 (C), 127.4 (CH), 127.3 (CH), 126.2 (CH), 124.8 (CH), 123.2 (CH), 120.2 (CH), 111.1 (CH), 110.2 (C), 21.5 (CH_3); EI-LRMS: m/z 290 (M^+ , 100). HRMS calcd. for $C_{19}H_{14}OS$: 290.0765. Found: 290.0783. HPLC (hexane): $t_R = 8.03$ min.

3-(2-Furyl)-2-*p*-tolylbenzo[*b*]furan (6m): Treatment of **1d** (0.324 g, 1 mmol) according to the typical procedure, using ethyl 2-furoate (0.140 g, 1 mmol) as electrophile and purification by column chromatography (eluent: hexane) on silica gel, gave **6m** (0.164 g, 60%) as a white solid: mp 45-47 °C; 1H NMR (300 MHz, $CDCl_3$) δ 7.91-7.87 (m, 1H), 7.82-7.76 (m, 2H), 7.64-7.58 (m, 2H), 7.44-7.33 (m, 2H), 7.31 (dd, $J = 8.0, 0.5$ Hz, 2H), 6.72-6.68 (m, 1H), 6.61 (dd, $J = 3.3, 3.0$ Hz, 1H), 2.5 (s, 3H); ^{13}C

NMR (75.4 MHz, CDCl₃) δ 154.0 (C), 152.2 (C), 147.2 (C), 141.9 (CH), 139.2 (C), 129.3 (CH), 128.6 (C), 127.8 (C), 127.6 (CH), 124.8 (CH), 123.2 (CH), 120.9 (CH), 111.4 (CH), 111.2 (CH), 108.6 (CH), 107.6 (C), 21.5 (CH₃); EI-LRMS: m/z 274 (M⁺, 100). HRMS calcd. for C₁₉H₁₄O₂: 274.0994. Found: 274.1002. Anal. Calcd. for C₁₉H₁₄O₂: C, 83.19; H, 5.14. Found: C, 83.45; H, 5.12.

3-Isopropyl-2-(1-naphthyl)benzo[*b*]furan (6n): Treatment of **1e** (0.360 g, 1 mmol) according to the typical procedure, using ethyl isobutyrate (0.116 g, 1 mmol) as electrophile and purification by column chromatography (eluent: hexane) on silica gel, gave **6n** (0.200 g, 70%) as a white solid: mp 96-98 °C; ¹H NMR (300 MHz, CDCl₃) δ 8.10-8.00 (m, 3H), 7.98-7.93 (m, 1H), 7.77-7.65 (m, 3H), 7.65-7.59 (m, 2H), 7.50-7.39 (m, 2H), 3.24 (sept., J = 7.1 Hz, 1H), 1.55 (d, J = 7.1 Hz, 6H); ¹³C NMR (75.4 MHz, CDCl₃) δ 155.1 (C), 149.9 (C), 133.8 (C), 132.8 (C), 129.8 (CH), 129.1 (CH), 128.8 (C), 128.4 (CH), 128.2 (C), 126.7 (CH), 126.3 (CH), 126.2 (CH), 125.2 (CH), 124.1 (CH), 123.9 (C), 122.3 (CH), 121.2 (CH), 111.7 (CH), 25.8 (CH), 22.6 (CH₃); EI-LRMS m/z 286 (M⁺, 78), 271 (100). HRMS calcd. for C₂₁H₁₈O: 286.1358. Found: 286.1339. Anal. Calcd. for C₂₁H₁₈O: C, 88.08; H, 6.34. Found: C, 87.85; H, 6.32.

2-(2-Fluorophenyl)-3-isopropylbenzo[*b*]furan (6o): Treatment of **1f** (0.281 g, 1 mmol) according to the typical procedure, using ethyl isobutyrate (0.116 g, 1 mmol) as electrophile and purification by column chromatography (eluent: hexane) on silica gel, gave **6o** (0.089 g, 35%) as a colorless oil: R_f 0.20 (hexane); ¹H NMR (300 MHz, CDCl₃) δ 7.80 (d, J = 7.8 Hz, 1H), 7.59 (td, J = 7.4, 1.6 Hz, 1H), 7.54 (d, J = 7.9 Hz, 1H), 7.47-7.39 (m, 1H), 7.37-7.17 (m, 4H), 3.19 (sept., J = 7.2 Hz, 1H), 1.46 (d, J = 7.2 Hz, 6H); ¹³C NMR (75.4 MHz, CDCl₃) δ 160.0 (d, J = 248.0 Hz, C), 155.3 (C), 144.8 (C), 131.6 (d, J = 2.4 Hz, CH), 130.8 (d, J = 8.1 Hz, CH), 128.1 (C), 124.3 (CH), 124.26 (d, J = 3.5 Hz, CH), 122.2 (CH), 121.5 (CH), 119.4 (d, J = 15.0 Hz, C), 116.3 (d, J = 23.0 Hz, CH), 111.6 (CH), 26.0 (d, J = 2.4 Hz, CH), 22.3 (CH₃); EI-LRMS m/z 254 (M⁺, 100). HRMS calcd. for C₁₇H₁₅FO: 254.1107. Found: 254.1118. HPLC (hexane): t_R = 7.04 min.

3-(2-Furyl)-2-(2-methoxyphenyl)benzo[*b*]furan (6p): Treatment of **1g** (0.340 g, 1 mmol) according to the typical procedure, using ethyl 2-furoate (0.140 g, 1 mmol) as electrophile and purification by column chromatography (eluent: hexane/AcOEt, 20/1) on silica gel, gave **6p** (0.136 g, 47%) as a colorless oil: R_f 0.43 (hexane/EtOAc, 5/1); ^1H NMR (300 MHz, CDCl_3) δ 8.08-8.00 (m, 1H), 7.66-7.54 (m, 2H), 7.52-7.43 (m, 2H), 7.40-7.32 (m, 2H), 7.10 (t, $J = 7.6$ Hz, 1H), 7.02 (d, $J = 8.5$ Hz, 1H), 6.48-6.43 (m, 1H), 6.36 (dt, $J = 3.3, 0.8$ Hz, 1H), 3.70 (s, 3H); ^{13}C NMR (75.4 MHz, CDCl_3) δ 157.5 (C), 154.5 (C), 149.2 (C), 148.4 (C), 141.4 (CH), 131.6 (CH), 131.2 (CH), 127.2 (C), 124.6 (CH), 123.1 (CH), 121.3 (CH), 120.6 (CH), 120.1 (C), 111.4 (CH), 111.3 (CH), 111.1 (CH), 110.3 (C), 106.8 (CH), 55.6 (CH_3); EI-LRMS m/z 290 (M^+ , 100). HRMS calcd. for $\text{C}_{19}\text{H}_{14}\text{O}_3$: 290.0943. Found: 290.0948. HPLC (hexane): $t_R = 7.73$ min.

3-Methyl-2-phenylnaphtho[2,1-*b*]furan (8a): Treatment of **7a** (0.313 g, 1 mmol) according to the typical procedure, using *t*-butyl acetate (0.116 g, 1 mmol) as electrophile and purification by column chromatography (eluent: hexane/AcOEt, 50/1) on silica gel, gave **8a** (0.147 g, 57%) as a white solid: mp 102-104 °C (lit.⁹ mp 104 °C); ^1H NMR (300 MHz, CDCl_3) δ 8.53 (d, $J = 8.3$ Hz, 1H), 8.03 (d, $J = 8.0$ Hz, 1H), 7.90-7.82 (m, 2H), 7.80-7.70 (m, 2H), 7.66 (td, $J = 7.7, 1.4$ Hz, 1H), 7.62-7.52 (m, 3H), 7.50-7.42 (m, 1H), 2.89 (s, 3H); ^{13}C NMR (75.4 MHz, CDCl_3) δ 151.8 (C), 150.9 (C), 131.3 (C), 130.9 (C), 129.20 (C), 129.16 (CH), 128.7 (CH), 127.9 (CH), 127.6 (CH), 126.2 (CH), 125.6 (CH), 124.1 (CH), 123.8 (C), 123.1 (CH), 113.6 (C), 112.4 (CH), 12.5 (CH_3); EI-LRMS: m/z 258 (M^+ , 100). HRMS calcd. for $\text{C}_{19}\text{H}_{14}\text{O}$: 258.1045. Found: 258.1034. Anal. Calcd. for $\text{C}_{19}\text{H}_{14}\text{O}$: C, 88.34; H, 5.46. Found: C, 88.08; H, 5.44.

2-Phenyl-3-(2-thienyl)naphtho[2,1-*b*]furan (8b): Treatment of **7a** (0.313 g, 1 mmol) according to the typical procedure, using ethyl 2-thiophenecarboxylate (0.156 g, 1 mmol) as electrophile and purification by column chromatography (eluent: hexane/AcOEt, 100/1) on silica gel, gave **8b** (0.231 g, 71%) as a white solid: mp 133-135 °C; ^1H NMR (300 MHz, CDCl_3) δ 8.10-7.95 (m, 1H), 7.84-7.72 (m, 5H), 7.66 (dd, $J = 5.0, 1.4$ Hz, 1H), 7.49 (td, $J = 7.5, 1.4$ Hz, 1H), 7.46-7.39 (m, 2H), 7.39-7.35 (m, 2H),

7.35-7.31 (m, 2H); ^{13}C NMR (75.4 MHz, CDCl_3) δ 152.0 (C), 151.4 (C), 134.9 (C), 131.0 (C), 130.5 (C), 129.0 (CH), 128.9 (CH), 128.6 (CH), 128.34 (CH), 128.27 (CH), 128.2 (C), 127.8 (CH), 126.4 (CH), 126.32 (CH), 126.29 (CH), 124.6 (CH), 124.1 (C), 123.0 (CH), 112.2 (CH), 111.7 (C); EI-LRMS: m/z 326 (M^+ , 100). HRMS calcd. for $\text{C}_{22}\text{H}_{14}\text{OS}$: 326.0765. Found: 326.0753. HPLC (hexane): t_R = 9.61 min.

3-Methyl-2-phenylnaphtho[1,2-*b*]furan (9a): Treatment of **7b** (0.313 g, 1 mmol) according to the typical procedure, using *t*-butyl acetate (0.116 g, 1 mmol) as electrophile and purification by column chromatography (eluent: hexane/AcOEt, 50/1) on silica gel, gave **9a** (0.144 g, 56%) as a white solid: mp 118-120 °C (lit.⁹ mp 118 °C); ^1H NMR (300 MHz, CDCl_3) δ 8.42 (d, J = 8.3 Hz, 1H), 8.0-7.9 (m, 3H), 7.76-7.60 (m, 3H), 7.60-7.48 (m, 3H), 7.45-7.37 (m, 1H), 2.60 (s, 3H); ^{13}C NMR (75.4 MHz, CDCl_3) δ 150.3 (C), 149.3 (C), 131.8 (C), 131.7 (C), 128.8 (CH), 128.5 (CH), 127.6 (CH), 126.5 (CH), 126.3 (CH), 125.0 (CH), 123.1 (CH), 121.3 (C), 120.3 (CH), 118.1 (CH), 112.6 (C), 9.8 (CH_3); EI-LRMS: m/z 258 (M^+ , 100). HRMS calcd. for $\text{C}_{19}\text{H}_{14}\text{O}$: 258.1045. Found: 258.1034. Anal. Calcd. for $\text{C}_{19}\text{H}_{14}\text{O}$: C, 88.34; H, 5.46. Found: C, 88.17; H, 5.45.

3-Isopropyl-2-phenylnaphtho[1,2-*b*]furan (9b): Treatment of **7b** (0.313 g, 1 mmol) according to the typical procedure, using ethyl isobutyrate (0.116 g, 1 mmol) as electrophile and purification by column chromatography (eluent: hexane/AcOEt, 100/1) on silica gel, gave **9b** (0.180 g, 63%) as a white solid: mp 119-121 °C; ^1H NMR (300 MHz, CDCl_3) δ 8.45 (d, J = 8.3 Hz, 1H), 7.99 (d, J = 7.8 Hz, 1H), 7.91 (d, J = 8.6 Hz, 1H), 7.88-7.82 (m, 2H), 7.66 (d, J = 8.6 Hz, 1H), 7.68-7.62 (m, 1H), 7.62-7.51 (m, 3H), 7.50-7.43 (m, 1H), 3.63 (sept., J = 7.0 Hz, 1H), 1.61 (d, J = 7.0 Hz, 6H); ^{13}C NMR (75.4 MHz, CDCl_3) δ 150.2 (C), 149.6 (C), 131.7 (C), 131.2 (C), 128.7 (C), 128.3 (CH), 128.1 (CH), 128.0 (CH), 126.3 (CH), 125.2 (CH), 124.1 (CH), 123.3 (C), 122.7 (CH), 121.6 (C), 120.3 (CH), 120.1 (CH), 25.8 (CH), 22.8 (CH_3); EI-LRMS: m/z 286 (M^+ , 48), 271 (100). HRMS calcd. for $\text{C}_{21}\text{H}_{18}\text{O}$: 286.1358. Found: 286.1344. Anal. Calcd. for $\text{C}_{21}\text{H}_{18}\text{O}$: C, 88.08; H, 6.34. Found: C, 87.79; H, 6.35.

Table: Comparative ^1H -NMR data for some of the dihydrobenzofuranols **5** showing the anisotropic effect of the C_2 -phenyl group for the C_3 -substituent

5a ($\text{R} = \text{Me}$, $\text{R}^1 = \text{H}$)	$\delta (\text{OH})$ 2.85 ppm $\delta (\text{Me})$ 1.10 ppm	$\delta (\text{OH})$ 1.70 ppm $\delta (\text{Me})$ 1.73 ppm
5c ($\text{R} = i\text{-Pr}$, $\text{R}^1 = \text{H}$) ^a	$\delta (\text{OH})$ 2.45 ppm $\delta (\text{CH})$ 1.81 ppm $\delta (\text{Me})$ 0.97 ppm $\delta (\text{Me})$ 0.42 ppm	$\delta (\text{OH})$ 1.46 ppm $\delta (\text{CH})$ 2.26 ppm $\delta (\text{Me})$ 1.08 ppm $\delta (\text{Me})$ 0.98 ppm
5e ($\text{R} = t\text{-Bu}$, $\text{R}^1 = \text{H}$)	$\delta (\text{OH})$ 2.34 ppm $\delta (\text{Me}_3)$ 0.74 ppm	$\delta (\text{OH})$ 1.37 ppm $\delta (\text{Me}_3)$ 1.15 ppm
5f ($\text{R} = 2\text{-Th}$, $\text{R}^1 = \text{H}$)	$\delta (\text{OH})$ 3.34 ppm	$\delta (\text{OH})$ 2.43 ppm
5g ($\text{R} = \text{Me}$, $\text{R}^1 = \text{Me}$)	$\delta (\text{OH})$ 2.68 ppm $\delta (\text{Me})$ 1.10 ppm	$\delta (\text{OH})$ 1.40 ppm $\delta (\text{Me})$ 1.72 ppm
5i ($\text{R} = \text{Ph}$, $\text{R}^1 = \text{Cl}$)	$\delta (\text{OH})$ 2.87 ppm	$\delta (\text{OH})$ 2.05 ppm

^aThe data for the *cis* isomer were collected from ref.[3].

¹H-NMR:

¹³C-NMR:

¹H-NMR:

¹³C-NMR:

¹H-NMR:

¹³C-NMR:

¹H-NMR:

¹³C-NMR:

¹H-NMR:

¹³C-NMR:

^1H -NMR:

^{13}C -NMR:

$^1\text{H-NMR}$:

$^{13}\text{C-NMR}$:

^1H -NMR:

(7a)

^{13}C -NMR:

¹H-NMR:

¹³C-NMR:

$^1\text{H-NMR}$:

$^{13}\text{C-NMR}$:

$^1\text{H-NMR}$:

$^{13}\text{C-NMR}$:

¹H-NMR:

¹³C-NMR:

¹H-NMR:

¹³C-NMR:

¹H-NMR:

¹³C-NMR:

¹H-NMR:

¹³C-NMR:

¹H-NMR:

¹³C-NMR:

¹H-NMR:

¹³C-NMR:

¹H-NMR:

(5f)

¹³C-NMR:

¹H-NMR:

¹³C-NMR:

¹H-NMR:

¹³C-NMR:

¹H-NMR:

¹³C-NMR:

¹H-NMR:

¹³C-NMR:

¹H-NMR:

¹³C-NMR:

(6a)

¹H-NMR:

¹³C-NMR:

¹H-NMR:

¹³C-NMR:

^1H -NMR:

^{13}C -NMR:

¹H-NMR:

¹³C-NMR:

¹H-NMR:

¹³C-NMR:

¹H-NMR:

¹³C-NMR:

¹H-NMR:

¹³C-NMR:

¹H-NMR:

¹³C-NMR:

¹H-NMR:

¹³C-NMR:

¹H-NMR:

¹³C-NMR:

^1H -NMR:

^{13}C -NMR:

¹H-NMR:

¹³C-NMR:

¹H-NMR:

¹³C-NMR:

¹H-NMR:

¹³C-NMR:

¹H-NMR:

¹³C-NMR:

$^1\text{H-NMR}$:

$^{13}\text{C-NMR}$:

(8b)

$^1\text{H-NMR}$:

$^{13}\text{C-NMR}$:

(9a)

$^1\text{H-NMR}$:

$^{13}\text{C-NMR}$:

¹H-NMR:

¹³C-NMR:

STANDARD 1H OBSERVE

Archive directory: /export/home/vnmr1/vnmrsys/data
Sample directory:

Pulse Sequence: NOESY

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
File: DL319-final-NOESY
INOVA-400 "carbone"

Relax. delay 1.000 sec
Mixing 0.500 sec
Acq. time 0.231 sec
Width 4439.5 Hz
2D Width 4439.5 Hz
32 repetitions
2 x 256 increments
OBSERVE H1, 399.9389071 MHz
DATA PROCESSING
Gauss apodization 0.107 sec
F1 DATA PROCESSING
Gauss apodization 0.053 sec
FT size 2048 x 2048
Total time 8 hr, 5 min, 6 sec

STANDARD 1H OBSERVE

Archive directory: /export/home/vnmr1/vnmrsys/data
Sample directory:
File: PROTON

Pulse Sequence: NOESY

Solvent: CDCl3
Ambient temperature
INOVA-400 "carbono"

Relax. delay 1.000 sec
Mixing 0.500 sec
Acq. time 0.171 sec
Width 3001.9 Hz
2D Width 3001.9 Hz
8 repetitions
2 x 256 increments
OBSERVE H1, 399.9389071 MHz
DATA PROCESSING
Gauss apodization 0.079 sec
F1 DATA PROCESSING
Gauss apodization 0.079 sec
F1 size 2048 x 2048
Total time 1 hr, 58 min, 46 sec

STANDARD 1H OBSERVE

Archive directory: /export/home/vnmr1/vnmrsys/data

Sample directory:

Pulse Sequence: NOESY

Solvent: CDCl₃

Ambient temperature

File: DL083-NOESY

INOVA-400 "carbono"

Relax. delay 1.000 sec

Mixing 0.500 sec

Acq. time 0.209 sec

Width 4898.4 Hz

2D Width 4898.4 Hz

8 repetitions

2 x 200 increments

OBSERVE H1, 399.9389071 MHz

DATA PROCESSING

Gauss apodization 0.097 sec

F1 DATA PROCESSING

Gauss apodization 0.038 sec

FT size 2048 x 2048

Total time 1 hr, 33 min, 53 sec

STANDARD 1H OBSERVE

Archive directory: /export/home/vnmr1/vnmr sys/data
Sample directory:

Pulse Sequence: NOESY

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
File: DL316-2f-NOESY
INOVA-400 "carbono"

Relax. delay 1.000 sec
Mixing 0.500 sec
Acq. time 0.129 sec
Width 3974.6 Hz
2D Width 3974.6 Hz
8 repetitions
2 x 256 increments
OBSERVE H1, 399.9389071 MHz
DATA PROCESSING
Gauss apodization 0.060 sec
F1 DATA PROCESSING
Gauss apodization 0.059 sec
FT size 2048 x 2048
Total time 1 hr, 55 min, 11 sec

STANDARD 1H OBSERVE

Archive directory: /export/home/vnmr1/vnmrSYS/data
Sample directory:
File: PROTON

Pulse Sequence: NOESY

Solvent: CDCl3
Ambient temperature
INOVA-400 "carbono"

Relax. delay 1.000 sec
Mixing 0.500 sec
Acq. time 0.142 sec
Width 3615.7 Hz
2D Width 3615.7 Hz
8 repetitions
2 x 200 increments
OBSERVE H1, 399.9389071 MHz
DATA PROCESSING
Gauss apodization 0.065 sec
F1 DATA PROCESSING
Gauss apodization 0.051 sec
FT size 2048 x 2048
Total time 1 hr, 30 min, 38 sec

STANDARD 1H OBSERVE

Archive directory: /export/home/vnmr1/vnmrSYS/data
Sample directory:
File: PROTON

Pulse Sequence: NOESY

Solvent: CDCl3
Ambient temperature
INOVA-400 "carbono"

Relax. delay 1.000 sec
Mixing 0.500 sec
Acq. time 0.150 sec
Width 3422.3 Hz
2D Width 3422.3 Hz
16 repetitions
2 x 256 increments
OBSERVE H1, 399.9389071 MHz
DATA PROCESSING
Gauss apodization 0.069 sec
F1 DATA PROCESSING
Gauss apodization 0.069 sec
F1 size 2048 x 2048
Total time 3 hr, 53 min, 4 sec

STANDARD 1H OBSERVE

Archive directory: /export/home/vnmr1/vnmrSYS/data
Sample directory:

Pulse Sequence: NOESY

Solvent: CDCl3
Ambient temperature
File: DL137-t30a42-NOESY
INOVA-400 "carbono"

Relax. delay 1.000 sec
Mixing 0.500 sec
Acq. time 0.151 sec
Width 3388.7 Hz
2D Width 3388.7 Hz
16 repetitions
2 x 128 increments
OBSERVE H1, 399.9389071 MHz
DATA PROCESSING
Gauss apodization 0.070 sec
F1 DATA PROCESSING
Gauss apodization 0.035 sec
FT size 2048 x 2048
Total time 1 hr, 55 min, 49 sec

REFERENCES.

- (1) Prepared by selective *o*-bromination with NBS of 1-naphthol following a reported procedure: Fujisaki, S.; Eguchi, H.; Omura, A.; Okamoto, A.; Nishida, A. *Bull. Chem. Soc. Jpn.* **1993**, *66*, 1576-1579.
- (2) Wagner, P. J.; Miador, M. A.; Park, B.-S. *J. Am. Chem. Soc.* **1990**, *112*, 5199-5211.
- (3) Sharshira, E. M.; Horaguchi, T. *J. Heterocycl. Chem.* **1997**, *34*, 1837-1850.
- (4) (a) Juhász, L.; Szilágyi, L.; Antus, S.; Visy, J.; Zsila, F.; Simonyi, M. *Tetrahedron* **2002**, *58*, 4261-4265. (b) Willis, M. C.; Taylor, D.; Gillmore, A. T. *Org. Lett.* **2004**, *6*, 4755-4757.
- (5) Angeloni, A. S.; Maurilio, T. *Ann. Chim.(Rome)* **1965**, *55*, 1028-1036.
- (6) Hu, Y.; Nawoschik, K. J.; Liao, Y.; Ma, J.; Fathi, R.; Yang, Z. *J. Org. Chem.* **2004**, *69*, 2235-2239.
- (7) Spetz, A. *Acta Chem. Scand.* **1956**, *10*, 1422-1426.
- (8) Bisagni, E.; Rivalle, C. *Bull. Soc. Chim. Fr.* **1969**, 2463-2468.
- (9) Royer, R.; Bisagni, E.; Hudry, C. *Bull. Soc. Chim. Fr.* **1960**, 1178-1180.