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A GENERAL AND REGIOSPECIFIC SYNTHESIS OF 5,8-DISUBSTITUTED α-TETRALONES

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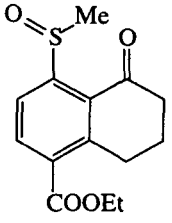
Abstract: A Pd[0]-mediated Stille coupling strategy was developed to prepare unsymmetrically 5,8-disubstituted α-tetralones in high yield. The compounds synthesized by this route are representative of the ease with which functionality may be introduced into the tetralone core at these positions.

Substituted α-tetralones are useful intermediates for the synthesis of a variety of compounds of biological and industrial importance.[2] During the course of a particular synthetic investigation, a series of 5,8-disubstituted α-tetralones were required (**1** and **2**). We envisioned that 5-bromo-8-methylthio-1-tetralone (**3**) could serve as a key intermediate for the synthesis of additional substituted tetralone derivatives.



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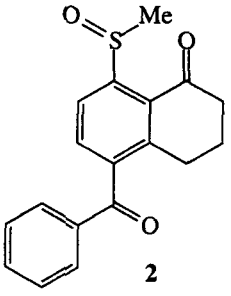

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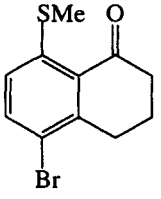

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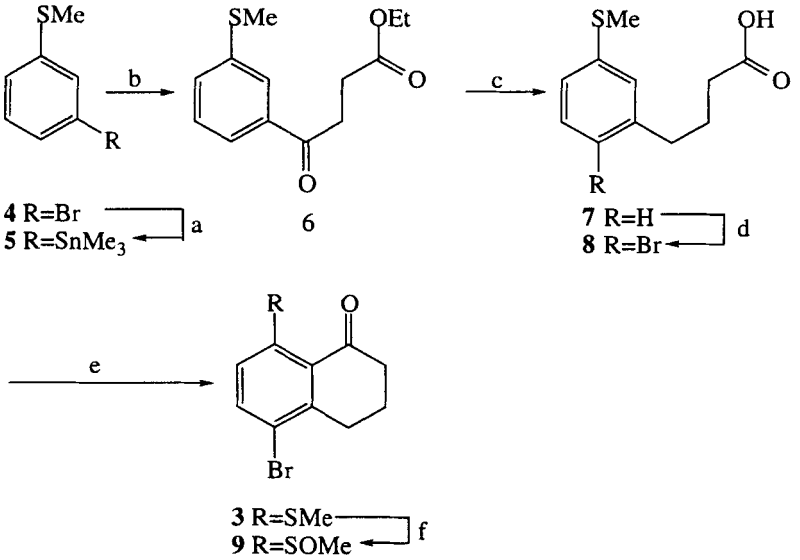
A search of the literature indicates that 6,8-, 5,7- and 6,7-disubstituted tetralones were readily prepared through the use of a Friedel-Crafts mediated ring closure.[3,4] The 5,8-disubstituted tetralones were not as common and no general synthetic pathway for preparing these derivatives existed.[5] Several groups have reported the preparation of the symmetrical 5,8-dimethoxy-1-tetralones[5a-e] and the synthesis of the 5-methoxy-8-halo-1-tetralones,[6] but the 5-halo-8-substituted compounds were not viable via this route.[7]

Recently, the synthesis of 5,6-methylenedioxy-1-tetralone was reported using an aryl Grignard approach to prepare the requisite phenyl ketone precursor.[8] A similar strategy was considered to control the regiochemistry in the synthesis of the key intermediate **7** (Scheme I). Our initial approach involved the reaction of the corresponding Grignard of **4** with succinyl anhydride to provide the keto acid



Scheme I

(a) Mg, ether, then $SnMe_3Cl$, 85%; (b) $Pd_2dba_3 \cdot CHCl_3$, ethyl succinyl chloride, DIEA, K_2CO_3, THF, 91%; (c) hydrazine, KOH, ethylene glycol, 88%; (d) Br_2, AcOH, 96%; (e) TFAA, $BF_3 \cdot Et_2O$, CH_2Cl_2, 91%; (f) mCPBA, $CHCl_3$, 88%.

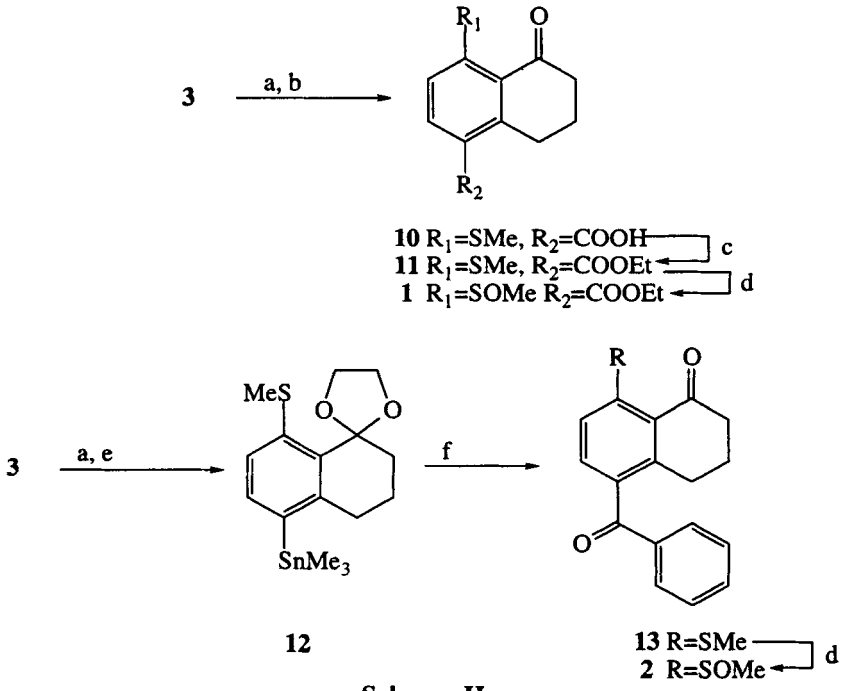
directly.[9] This reaction was successful, albeit the yields were low and the product was difficult to purify. To circumvent this difficulty, we found that a stepwise approach utilizing the Pd[0]-mediated Stille coupling[10] gave the desired tetralone precursor **6** in good yields. In addition, the mild conditions used in this type of coupling should increase the generality of this approach.

The synthesis began from the commercially available 3-bromothioanisole **4** which upon treatment with Mg followed by quenching with trimethyltin chloride gave the aryl stannane **5** in good yields. The Stille coupling was done using tris(dibenzylideneacetone)dipalladium[0]-chloroform[11] ($Pd_2dba_3 \cdot CHCl_3$) as the catalyst of choice. Reaction of **5**, ($Pd_2dba_3 \cdot CHCl_3$) and ethyl succinyl chloride provided the ketone **6** in >90% yield. The butyric acid derivative **7** was obtained by a Wolf-Kishner reduction of the aryl ketone[12] which also effected saponification of the ester. Bromination (Br_2, AcOH) provided the 4-bromo isomer **8** as the only detectable product in nearly quantitative yield. Cyclization was effected using trifluoroacetic anhydride/$BF_3 \cdot Et_2O$ to give the desired tetralone **3**, and proceeded smoothly even in the presence of the deactivating bromo substituent.

Having found a general and high yielding route to the tetralones with the requisite substitution pattern, we set out to complete the synthesis of the targeted compounds (Scheme II). Ketone **3** was first protected as its ethylene ketal, transmetallated at -78°C (nBuLi) and the resulting anion quenched with carbon dioxide or trimethyltin chloride to give compounds **10** and **12** respectively. The acid **10** was esterified under standard conditions (EtOH, H_2SO_4) to provide the ethyl ester **11**.

The utility of this approach is exemplified by the aryl stannane **12**, which can be reacted with a wide variety of substituted acyl chlorides. The reaction of **12** with benzoyl chloride ($Pd_2dba_3 \cdot CHCl_3$, cat) and hydrolysis of the ketal gave the 5-

benzoyl derivative **13** in 71% yield. As a final step, thioethers **3, 11** and **13** were oxidized to their respective sulfoxides **9, 1** and **2** with mCPBA in high yields.



Scheme II

(a) ethylene glycol, TsOH, benzene, reflux, 71%; (b) nBuLi, THF, -78°, then CO_2, HCl, 77%; (c) EtOH, H_2SO_4, reflux, 76%; (d) mCPBA, $CHCl_3$, 94%; (e) nBuLi, THF, -78°, then $SnMe_3Cl$, 96%; (f) $Pd_2dba_3 \cdot CHCl_3$, benzoyl chloride, DIEA, K_2CO_3, THF, then HCl, MeOH, H_2O 71%.

In summary, we have described a high-yielding, regiospecific synthesis of unsymmetrical 5,8-disubstituted-1-tetralones starting from readily available materials. We are currently in the process of further exploiting this chemistry in several of our lead optimization programs. The results will be reported in the near future.

Experimental

Commercial grade mCPBA was purified according to published procedures[13] and stored at -10°C. THF was distilled from sodium/benzophenone ketyl immediately prior to use. All other starting materials, reagents and solvents were purchased at highest commercial quality and used as received. NMR spectra were recorded on a Varian 300 instrument with TMS as an internal reference for proton spectra, $CDCl_3$ for ^{13}C. Electron impact mass spectra (EIMS) were recorded on a Hewlett-Packard 5890 GC/MS instrument and are reported as *m/z* (rel. int.). High-resolution mass spectra (HRMS) were obtained from Mass Consortium, San Diego, CA, 92122. Microanalyses were performed by Desert Analytics, Tucson, AZ 85717. IR spectra were taken on a Nicolet Impact 400D as thin films and melting points determined on a Mel-Temp II hot stage apparatus and are uncorrected.

3-Trimethylstannylthioanisole (5): A mixture of 3-bromothioanisole (6.0 g, 29.6 mmol), Mg (ribbon, 1.22 g, 50 mmol), Et_2O (100 mL) and I_2 (cat) was heated at reflux for 3 h under N_2. The mixture was allowed to cool to room temperature and then was transferred by cannula to a separate flask containing trimethyltin chloride (5.97 g, 30 mmol) in THF (60 mL) at -78°C. The reaction was stirred at -78°C for 10 min., allowed to slowly warm to room temperature and stirred for 18 h. The organic layer was removed, dried and concentrated. The residue was chromatographed (SiO_2, hexanes) to provide **5** (7.23 g, 86%) as an oil: ^1H NMR($CDCl_3$) δ 0.20 (s, 9H); 2.49 (s, 3H), 7.19-7.30 (m, 3H), 7.39 (m, 1H); HRMS Calc. MH^+ 289.0073. Found MH^+ 289.0070.

Ethyl 4-(3'-Methylthio)phenyl-4-oxobutyrate (6): To a mixture of Pd_2dba_3-$CHCl_3$ (0.90 g, 0.87 mmol), K_2CO_3 (0.15 g, 1.1 mmol), diisopropyl-

ethylamine (3.0 g, 23 mmol), THF (75 mL) and 3-trimethylstannylthioanisole (**5**, 5.0 g, 17.4 mmol) in an oven-dried flask under N_2, was added ethyl succinyl chloride (4.31 g, 26.1 g) dropwise via syringe. The reaction was stirred for 2 h, diluted with Et_2O and filtered. The Et_2O was washed with 10% K_2CO_3 (2x), H_2O (2x), brine, dried and concentrated to an oil. The residue was chromatographed (SiO_2, hexanes/EtOAc, 10:90 to 15:85) to provide **6** (4.0 g, 91%) as a pale yellow oil: ^1H NMR($CDCl_3$) δ 1.27 (t, 3H), 2.52 (s, 3H), 2.75 (t, 2H), 3.29 (t, 2H), 4.15 (q, 2H), 7.34-7.46 (m, 2H), 7.7 (d, 1H), 7.85 (t, 1H); MS 252 (20, M^+), 207 (20), 151 (100), 123 (30); Anal. calc. for $C_{13}H_{16}O_3S$: C, 61.88; H, 6.39. Found: C, 62.16; H, 6.64.

4-(3'-Methylthio)phenylbutyric acid (7): A mixture of ethyl 4-(3'-methylthio)phenyl-4-oxobutyrate (**6**, 5.11 g, 20.3 mmol), KOH (35.8 g, 64 mmol), hydrazine (85% in H_2O, 26 mL) and ethylene glycol (400 mL) was heated at reflux for 16 h. The reaction was diluted with H_2O (1.5 L) and cooled to 0°C. The solution was acidified with HCl (conc. to pH 1) and stirred at 0°C for 2 h. The resulting solid was collected by filtration and dried under vacuum to provide **7** (3.75 g, 88%) as a white solid: ^1H NMR($CDCl_3$) δ 1.97 (m, 2H), 2.39 (t, 2H), 2.41 (s, 3H), 2.66 (t, 2H), 6.97-7.25 (m, 4H); MS 210 (M^+, 89), 150 (100), 137 (22); IR 3050 (br.), 2921, 1705, 1421 cm^{-1}; Anal. calc. for $C_{11}H_{14}O_2S$: C, 62.83; H, 6.71. Found: C, 63.00; H, 6.81.

4-(2'-Bromo-5'-methylthio)phenylbutyric acid (8): To a solution of 4-(3'-methylthio)phenylbutyric acid (**7**, 2.42 g, 11.5 mmol) in acetic acid (60 mL) was added Br_2 (2.03 g, 12.7 mmol, dropwise over 10 min) in acetic acid (30 mL). The solution was stirred for 3 h at room temperature, poured into H_2O (0°C) and extracted with Et_2O. The Et_2O layer was washed with H_2O(4x), brine, dried and

concentrated to give **8** (3.19 g, 96%) as a light yellow solid which was sufficiently pure to be used in the next step: ^{1}H NMR ($CDCl_3$) δ 1.96 (m, 2H), 2.43 (t, 2H), 2.46 (s, 3H), 2.77 (t, 2H), 6.95, (dd, 1H), 7.09 (d, 1H), 7.42 (d, 1H); MS 290 (M^+, 72), 228 (100), 209 (18), 163 (22).

5-Bromo-8-methylthio-1-tetralone (3): To a solution of 4-(2'-bromo-5'-methylthio)phenylbutyric acid (**8**, 3.1 g, 10.7 mmol) in CH_2Cl_2 (200 mL) under N_2, was added BF_3-Et_2O (3.33 mL, 27 mmol) dropwise via syringe. Then $(CF_3CO)_2O$ (3.81 mL, 27 mmol) was carefully added. The resulting solution was stirred at room temperature for 2 h, and the H_2O (5 mL) was added. The reaction was concentrated and resulting residue dissolved in Et_2O. The Et_2O was washed with H_2O, 1N NaOH, H_2O, brine, dried and concentrated. Recrystallization (hexanes) provided **3** (2.65 g, 91%) as white needles: mp 51-52 °C; ^{1}H NMR ($CDCl_3$) δ 2.11 (m, 2H), 2.40 (s, 3H), 2.68 (t, 2H), 3.03 (t, 2H), 7.04 (d, 1H), 7.63 (d, 1H); MS 272 (M^+, 54), 255 (100), 244 (13), 216 (7), 174 (24); IR 2944, 1667, 1561, 1430, 1249 cm^{-1}; HRMS Calc MH^+ 270.9793. Found MH^+ 270.9795.

5-Bromo-8-methylsulfinyl-1-tetralone (9): A solution of 5-bromo-8-methylthio-1-tetralone (**3**, 0.10 g, 0.37 mmol) and $CHCl_3$ (10 mL) was cooled to -5°C, and mCPBA (0.070 g, mmol) in $CHCl_3$ (5 mL) was added dropwise over 5 min. The resulting mixture was stirred at -5°C for 10 min. and diluted with H_2O. The organic layer was washed with sat. $NaHSO_3$, 1N NaOH (2x), brine, dried and concentrated to a white solid. Recrystallization (hexanes/EtOAc) afforded **9** (0.095 g, 88%); mp 150-151°C: ^{1}H NMR ($CDCl_3$) δ 2.20 (m, 2H), 2.70 (m, 2H), 2.81 (s, 3H), 3.09 (m, 2H), 7.99 (d, 1H), 8.12 (d, 1H); MS 288 (M^+, 18), 271 (100), 255 (5), 243 (8), 225 (11), 207 (6); IR 1677, 1285, 1028 cm^{-1}; Anal. calc. for $C_{11}H_{11}BrO_2S$: C, 46.01; H, 3.86. Found: C, 45.92; H, 3.90.

8-Methylthio-1-tetralone-5-carboxylic acid (10): A mixture of 5-bromo-8-methylthio-1-tetralone (**3**, 1.50 g, 5.51 mmol), TsOH (cat), ethylene glycol (15 mL) and benzene (140 mL) was heated at reflux for 36 h.[14] The solution was cooled and poured into H_2O. The organic layer was dried, concentrated to a solid and recrystallized (hexanes) to provide 8-methylthio-5-bromo-(1,1-ethylenedioxy)-tetralone (0.50g, 71%) yield.

This material (0.500 g, 1.58 mmol) was immediately dissolved in THF (20 mL), under N_2 and cooled to -78°C. A solution of nBuLi (1.6M in hexane, 1.2 mL, 1.8 mmol) was added dropwise via syringe at -78°C and stirring was continued for 10 min. at -78°C . The solution was allowed to warm to 0°C and CO_2 was bubbled through the mixture for 10 min. The reaction mixture was allowed to warm to room temperature, diluted with H_2O and the organic layer washed with hexanes (2x). The aqueous layer was cooled to 0°C and acidified (pH 1) with HCl (0°C, 50%). The mixture was extracted with EtOAc and the organic layer was washed with H_2O, brine, dried and concentrated to provide **10** (0.29 g, 77%) as a white solid: ^{1}H NMR ($CDCl_3$) δ 2.09 (m, 2H), 2.45 (s, 3H), 2.72 (t, 2H), 3.43 (t, 2H), 7.28 (d, 1H), 8.12 (d, 1H).

Ethyl 8-methylthio-1-tetralone-5-carboxylate (11): A mixture of 8-methylthio-1-tetralone-5-carboxylic acid (**10**, 0.250 g, 1.05 mmol), EtOH (25 mL) and H_2SO_4 (cat) was heated at reflux for 48 h. The mixture was cooled and concentrated. The residue was partitioned between H_2O and EtOAc. The organic layer was dried, concentrated to a solid and recrystallized (hexanes) to provide **11** (0.21 g, 76%); mp 91-92°C: ^{1}H NMR ($CDCl_3$) δ 1.39 (t, 3H), 2.06 (m, 2H), 2.43 (s, 3H), 2.70 (t, 2H), 3.34 (t, 2H), 4.37 (q, 2H), 7.22 (d, 1H), 7.95 (d, 1H); ^{13}C NMR ($CDCl_3$) δ14.42, 16.42, 22.25, 28.58, 39.55, 61.24, 121.98, 125.43, 129.98, 134.01, 148.10, 150.67, 167.33, 198.96; MS 264 (M^+, 58),

249 (100), 221 (42), 203 (10), 175 (9); IR 2969, 2929, 1717, 1677, 1571, 1249, 1194, 1143 cm^{-1}; Anal. calc. for $C_{14}H_{16}O_3S$: C, 63.61; H, 6.10. Found: C, 63.44; H, 6.13.

Ethyl 8-methylsulfinyl-1-tetralone-5-carboxylate (1): To a solution of ethyl 8-methylthio-1-tetralone-5-carboxylate (**11**, 0.10 g, 0.38 mmol) in $CHCl_3$ (10 mL) was added mCPBA (0.071 g). The resulting mixture was stirred at -5°C for 10 min. and diluted with H_2O. The organic layer was washed with sat. $NaHSO_3$, 1N NaOH (2x), brine, dried and concentrated to a white solid. Recrystallization from (hexanes/EtOAc) afforded **1** (0.98 g, 92%) as white needles, mp = 94-95°C: ^1H NMR ($CDCl_3$) δ 1.43 (t, 3H), 2.16 (m, 2H), 2.73 (m, 2H), 2.83 (s, 3H), 3.35 (m, 2H), 4.41 (q, 2H), 8.25 (d, 1H), 8.33 (d, 1H); MS 280 (M^+, 15), 265 (100), 237 (9); IR 2959, 1712, 1682, 1249 cm^{-1}; Anal. calc. for $C_{14}H_{16}O_4S$: C, 59.98; H, 5.75. Found: C, 59.97; H, 5.80.

5-Trimethylstannyl-8-methylthio-(1,1-ethylenedioxy)-tetralone (12): A mixture of 5-bromo-8-methylthio-1-tetralone (**3**, 1.50 g, 3.67 mmol), TsOH (cat), ethylene glycol (15 mL) and benzene (140 mL) was heated at reflux for 36 h.[14] The solution was cooled and poured into H_2O. The organic layer was dried, concentrated to a solid and recrystallized (hexanes) to provide 8-methylthio-5-bromo-(1,1-ethylenedioxy)- tetralone (0.812 g, 71%) yield.

The ketal (0.812 g, 2.57 mmol) was dissolved in THF (20 mL) under nitrogen and cooled to -78°C, then nBuLi (1.6M in hexane, 1.9 mL, 3.0 mmol) was added dropwise by syringe. The solution was stirred at -78°C for 20 min., then trimethyltin chloride (1M in THF, 3.1 mL, 3.1 mmol) was added at once, and the reaction was allowed to warm slowly to room temperature and stirred 2 h. The reaction was then diluted with hexanes, washed with water (3x), brine, dried and

reduced to a thick yellow, acid-sensitive oil which was used without further purification in the subsequent step (0.98 g, 96%). MS 399 (M^+).

5-Benzoyl-8-methylthio-1-tetralone (13): A mixture of 5-trimethylstannyl-8-methylthio-(1,1-ethylene dioxy)-tetralone (**12**, 0.980 g, 2.46 mmol), Pd_2dba_3-$CHCl_3$ (0.13 g, 0.123 mmol), diisopropylethylamine (0.320 g, 2.46 mmol), K_2CO_3 (0.340 g, 2.46 mmol) and benzoyl chloride (0.42 g, 3.0 mmol) was stirred for 2 h at room temperature. The reaction was diluted with Et_2O and filtered. The Et_2O layer was washed with 10% K_2CO_3 (2x), H_2O (2x), brine, dried and concentrated. The residue was dissolved in MeOH (100 mL) and HCl (10 mL, 50%,) was added. The solution was stirred for 15h, concentrated and partitioned between EtOAc and H_2O. The organic layer was dried, concentrated and the residue was chromatographed (SiO_2, EtOAc/hexanes 15:85) to provide **13** (0.52 g, 71%) as a sticky white foam: ^1H NMR ($CDCl_3$) δ 2.02 (m, 2H), 2.45 (s, 3H), 2.71 (t, 2H), 2.95 (t, 2H), 7.24 (d, 1H), 7.40-7.51 (m, 3H), 7.61 (tt, 1H), 7.80 (m, 2H); MS 296 (M^+, 100), 281 (85), 267 (15); IR 2932, 1662, 1576, 1272 cm^{-1}; HRMS Calc. MH^+ 297.0949. Found MH^+ 297.0938.

5-Benzoyl-8-methylsulfinyl-1-tetralone (2): To a solution of 5-benzoyl-8-methylthio-1-tetralone (**13**, 0.300 g, 1.01 mmol) in $CHCl_3$ (10 mL) was added mCPBA (0.189 g). The resulting mixture was stirred at -5°C for 10 min. and diluted with H_2O. The organic layer was washed with sat. $NaHSO_3$, 1N NaOH (2x), brine, dried and concentrated to a white solid. Recrystallization (hexanes/EtOAc) afforded **2** (0.30 g, 95%); mp 125-126°C: ^1H NMR ($CDCl_3$) δ2.12 (m, 2H), 2.71 (m, 2H), 2.87 (s, 3H), 2.99 (t, 2H), 7.51 (tt, 2H), 7.66 (tt, 1H), 7.74 (d, 1H), 7.81 (m, 2H), 8.34 (d, 1H); MS 312 (M^+, 24), 297 (100), 282 (8), 255 (5); IR 3064, 2948, 1679, 1452, 1271, 1060, 964 cm^{-1}; Anal. calc. for $C_{18}H_{16}O_3S$: C, 69.21; H, 5.16. Found: C, 69.37; H, 5.20.

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