

Synthesis of 5-O -Methylsclerone

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Abstract

The total synthesis is of 5-methylsclerone, isolated from the fermentation broth of amidepsine - producing *Humicola* sp. FO-2942 has been realized, with an overall yield of 25%. A new synthesis of 8-methoxy-1-tetralone has also been discussed.

Keywords: Methylsclerone; Naphthalenone; Butylhydroperoxide; Pyridine; Tetrahydropyran

Introduction

5-O -methylsclerone (2,3-dihydro-4-hydroxy-5-methoxy-1-naphthalenone) 9 was isolated [1] from the fermentation broth of amidepsine-producing *Humicola* sp. FO-2942. The compound was also originally synthesized [2] from the metabolite Sclerone, isolated from the culture filtrate of *Sclerotinia sclerotiorum*. *Humicola* sp. FO-2942, originally discovered as a producing fungus of amidepsines, inhibits the accumulation of triglyceride [3,4] which can give origin of several diseases like obesity, arteriosclerosis etc.

In relation of our studies on terpenoid compounds, an attempt was sought to achieve a total synthesis of 5-O-methylsclerone 9. The synthetic details are described in Scheme 1. The already reported [5] alcohol 1 on benzylation with benzoyl chloride and pyridine yielded the benzoate 2 in 96 % yield. Benzylic oxidation [6] of the benzoate 2 with tert-butylhydroperoxide and pyridinium dichromate afforded the ketobenzoate 3 in 62 % yield. The oxidation was also attempted with chromium trioxide in acetic acid [7] but the yield was not superior to Figure 1.

It is necessary to mention here that due to the electronic and steric requirements resulting from the repulsion [8] occurring between the carbonyl oxygen atom and the bulky methoxy substituent oxidation at C-1 causes difficulty. The alcohol 4, 98% obtained by alkaline hydrolysis of the ketobenzoate 3, was converted to its tetrahydropyranyl derivative 5, in 94 % yielded by treatment with dihydropyran and a catalytic amount of p-TsOH.

Reduction of 5 with sodium borohydride in methanol followed by benzylation of the resulting alcohol with benzoyl chloride in pyridine afforded the benzoyl derivative 6. The transformation of 6 to the alcohol 7 was accomplished by heating ammonium

chloride in methanol [9]. Ketone 8, obtained in high yield by the chromic acid oxidation of the alcohol 7, yielded 8 which on alkaline hydrolysis afforded the desired product 5-O-methylsclerone 9 whose spectral data support the assigned structure. In relation to the present study an alternative attempt was made for the conversion of the ketobenzoate 8 to 8-methoxy-1-tetralone 11 [10,11]. The synthetic route is depicted in Figure 2.

The ketobenzoate 8 dissolved in ethanol was subjected to catalytic hydrogenation with platinum oxide (PtO₂). The resulting compound obtained in 68 % yield as liquid was identified as 5-methoxytetraline 10 which on oxidation with chromium trioxide in acetic acid yielded 8-methoxy-1-tetralone 11 as an oil in 14 % yield. The yield of the tetralone 11 is poor compared to most of other published methods [10,11]. In addition, commercially available 5-methoxy-1-tetralone 12 was obtained in 46 %. The other products obtained could not be characterized. In conclusion a total synthesis of 5-methylsclerone and a new synthesis of 8-methoxy-1-tetralone have been developed. For the nine steps shown in Scheme 1, the method provides an over-all yield of 25 % of 5-O- methylsclerone.

Experimental Section

Reagents were obtained from commercial sources and used without purification. Solvents were dried by distillation from appropriate drying agents immediately prior to use. For work-up, all organic extracts were dried over anhydrous MgSO₄, filtered and the solvent evaporated in vacuo. Flash column chromatography was performed on silica gel (0.04-0.63 mm) using the solvent system indicated. Unless otherwise stated, all melting points are uncorrected and were determined on an Electrothermal melting point apparatus IR spectra were taken on a Nicolet Fourier-

transform (FT) instrument. Mass spectra were recorded on a Thermo Scientific TSQ Quantum Ultra AM Triple Quadrupole mass spectrometer. NMR spectra were recorded on a Bruker AVANCE-500 spectrometer (at 500 MHz for H-NMR or 125 MHz

for C-NMR) in a suitable deuterated solvent, as specified below. Chemical shifts (δ) are expressed in ppm. Elemental analyses were performed on Carlo-Erba 1108 Elemental Analyser.

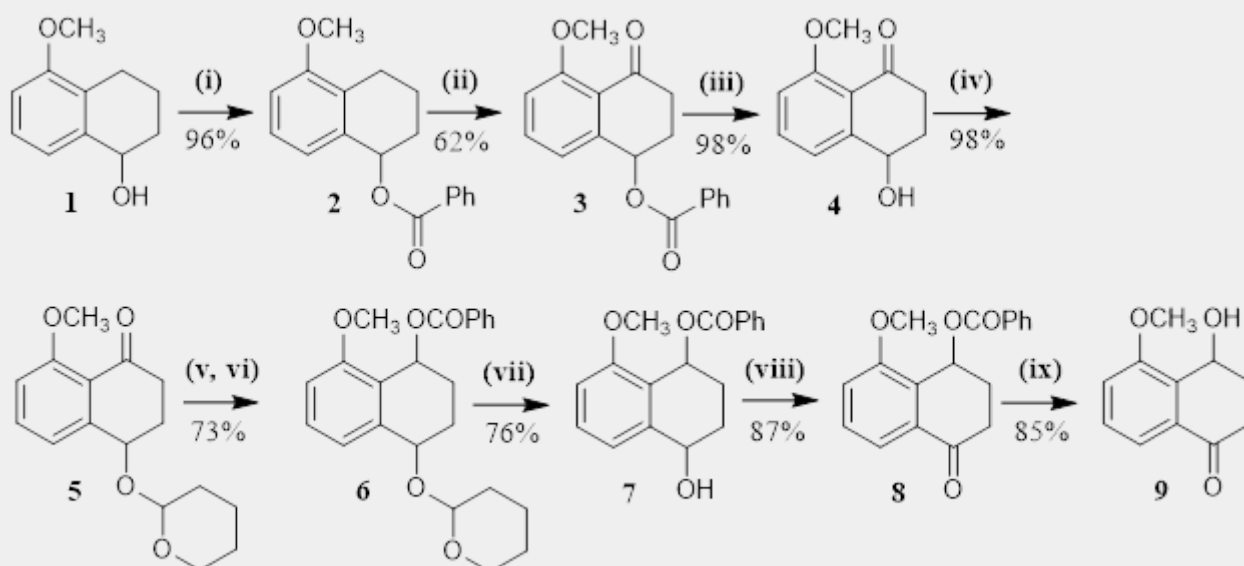


Figure 1: Preparation of the title compound 9. Reagents and conditions: (i) PhCOCl, pyridine, rt., 19h; (ii) PDC, Pyridine, TBHP, 70% H₂O₂, 24h, rt; (iii) EtOH, K₂CO₃, rt; (iv) DHP, p-TsOH, 1h, rt; (v) NaBH₄, EtOH, 3 h, rt; (vi) Py, PhCOCl, 18 h, rt; (vii) NH₄Cl, 4h, reflux; (viii) CrO₃, Py, 24h, rt; (ix) EtOH, K₂CO₃, rt.

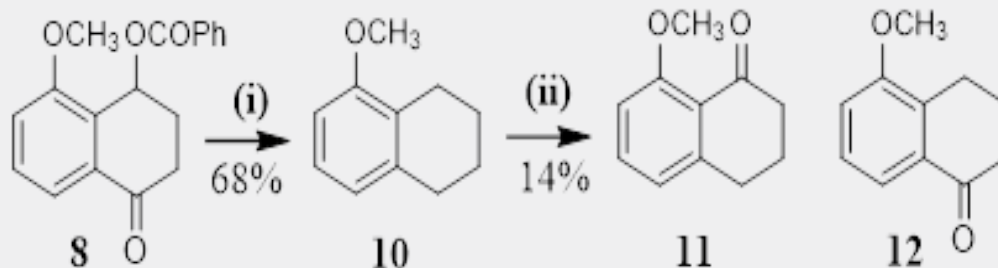


Figure 2: Reagents and Conditions: (i) H₂, PtO₂/MeCOOH, (ii) CrO₃, MeCOOH, H₂O (10 %, 19:1), 17-21°C, 3h.

1-Benzoyl-5-methoxytetralin (2) To the alcohol 1 (2.01 g, 11 mmol) dissolved in dry pyridine (25 mL) was added benzoyl chloride (7.26 g, 50 mmol) and stirred for 18 h at room temperature. To the resulting deep yellow solution was added cold water (70 mL) and extracted with ether (3 x 30 mL). The organic extracts were washed with hydrochloric acid (20 mL, 10%), copper sulfate solution (5 mL, 10%), sodium hydroxide solution (30 mL, 10%), with water, dried and evaporated to obtain a dark yellow liquid which was purified by column chromatography (eluant hexane)

to yield the benzoylated compound 2 (3.03 g, 96 %) as white solid; mp: 53-55°C (hexane); IR (cm⁻¹): 1713 (CO); MS(m/z): 160 (M-ArCOOH), 105 (ArCO); ¹H NMR (CDCl₃): δ 8.07 (dd, 2H, J = 7.7 Hz, 1.4 Hz; H-15, H-18), 7.56 (tt, 1H, J = 7.7 Hz, 1.4 Hz; H-16), 7.43 (t, 2H, J = 7.7 Hz; H-15, H-17), 7.21 (t, 1H, J = 8 Hz; H-8), 7.08 (d, 1H, J = 8 Hz; H-7), 6.78 (d, 1H, J = 8 Hz; H-6), 6.26 (t, 1H, J = 4.7 Hz; H-1), 3.87 (s, 3H, OMe), 2.89 (dt, 1H, J = 17.8 Hz; J = 8.4 Hz; H-4), 2.62 (dt, 1H, J = 17.8 Hz; J = 8.4 Hz; H-4), 2.12-1.91 (m, 4H, H-2, H-3); ¹³C NMR (CDCl₃): δ 166.16 (C-12), 156.97 (C-5), 135.72 (C-9), 132.78 (C-

13), 129.67 (C-14), 128.22 (C-15), 127.07 (C-10), 126.34 (C-7), 121.40 (C-8), 109.07 (C-6), 70.58 (C-1), 55.30 (OMe), 28.69 (C-2), 22.77 (C-4), 18.26 (C-3).

Anal. Calcd for C₁₈H₁₈O₃: C, 76.57; H, 6.43. Found: C, 76.82; H, 6.61.

4-Benzoyl-8-methoxy-1-tetralone (3)

To the benzoate 2 (520 mg, 1.84 mmol) in dry benzene (30 mL) was added celite (1.52 g), pyridinium dichromate (2.67 g, 7.12 mmol) followed by the addition of t-butyl hydroperoxide (4.50 mL, 70%) (TBHP) in water. The mixture was stirred at 100°C for 15 min and 24 h at room temperature. To the resulting solution was added diethyl ether (30 mL), filtered over celite, washed the celite three times with ether (20 mL x3). The organic extracts were dried, evaporated, chromatographed. The eluant (hexane: ether 6:4) afforded the tetralone 3 (340 mg, 62%) as white solid; mp 95°C (ether); IR (cm⁻¹): 1716, 1613 (CO); MS: 175 (M + 1-PhCOOH); ¹H NMR (CDCl₃): δ 8.04 (dd, 2H, J= 8Hz, 1.4 Hz, H-14, H-18), 7.61-7.42 (m, 4H, H-6, H-7, H-15, H-17), 7.14 (d, 1H, J=8 Hz, H-5), 7.04 (d, 1H, d=8 Hz, H-16), 6.33 (t, 1H, J=4.8 Hz, H-4), 3.96 (s, 3H, OMe), 2.74 (dt, 2H, J= 17.4, 8 Hz, H-2), 2.46-2.40 (m, 2H, CH₂); ¹³C NMR (CDCl₃): δ 195.97 (C-1), 165.75 (C-12), 159.99 (C-8), 143.00 (C-10), 134.62 (C-13), 133.22 (C-16), 129.86 (C-6), 129.67 (C-14), 128.40 (C-15), 121.42 (C-9), 120.29 (C-5), 112.62 (C-7), 70.31 (C-4), 56.13 (C-11), 35.70 (C-2), 27.91 (C-3).

Anal. Calcd for C₁₈H₁₆O₄: C, 72.96; H, 5.44. Found: C, 73.22; H, 5.59.

4-Hydroxy-8-Methoxy-1-Tetralone (4)

To a solution of the benzoate (200 mg, 0.68 mmol) in ethanol (20 mL) was added potassium carbonate (350 mg, 2.53 mmol) and stirred for 18 h at room temperature. The resulting solution was concentrated extracted with chloroform (2 x 15 mL). The organic extracts were combined, washed, dried and evaporated to obtain a brown liquid which was chromatographed (eluant hexane: ether 30:70) to obtain the oily tetralone 4 (130 mg, 98%); IR (cm⁻¹): 3412 (OH), 1664 (CO); MS: 193 [M + 1], 175 [M + 1-H₂O]; ¹H NMR (CDCl₃): δ 7.51 (t, 1H, J=8 Hz, H-6), 7.17 (d, 1H, J=8 Hz, H-5), 6.94 (d, 1H, J=8 Hz, H-7), 4.88 (dd, 1H, J=7.8 Hz, 3.8 Hz H-4) 3.90 (s, 3H, OMe), 2.94-2.84 (m, 1H), 2.62-2.52 (m, 2H), 2.32-2.17 (m, 1H), 2.15-2.12 (m, 1H) (H-2, H-3, H-4), 1.90 (s, br, OH, H-4); ¹³C NMR (CDCl₃): δ 197.08 (C-1), 159.82 (C-8), 148.13 (C-10), 134.70 (C-6), 120.29 (C-9), 118.88 (C-5), 111.56 (C-7), 68.06 (C-4), 55.95 (C-11), 36.39 (C-2), 31.11 (C-3).

Anal. Calcd for C₁₁H₁₂O₃: C, 68.73; H, 6.29. Found: C, 68.96; H, 6.43.

4-Tetrahydropyranyl-8-Methoxy-1-Tetralone (5)

To the hydroxytetralone 4 (630 mg, 3.28 mmol) dissolved in dihydropyran (4 mL, 21.33 mmol) was added p-toluenesulfonic acid (2 mg), stirred for 1 h room temperature, then added potassium carbonate (0.33g, 2.39 mmol) stirred for 2 min and extracted with ether (120 mL). The organic extract was washed,

dried and chromatographed (eluant hexane:ether 6:4) to obtain the oily tetralone 5 (850 mg, 94%); IR (cm⁻¹) 1680 (CO); MS (m/z): 277 [M + 1], 176 [M + -101]; ¹H NMR (CDCl₃): δ 7.46 (t, 1H, J= 8 Hz, C-6), 7.19 (d, 1H, J= 8 Hz, H-5), 6.91 (d, 1H, J= 8 Hz, H-7), 4.89 (td, 1H, J=4.2 Hz, H-12), 4.86 (dd, 1H, J=7Hz, 3.5 Hz, H-4), 3.87 (s, 3H, OMe), 3.85-3.78 (m, 1H, H-16), 3.51-3.46 (m, 1H, H-16), 2.89-2.78 (m, 1H, H-2), 2.57-2.46 (m, 1H, H-2), 2.21-2.08 (m, 1H, H-16), 1.80-1.72 (m, 2H, H-15), 1.62-1.47 (m, 4H, H-13, 14); ¹³C NMR (CDCl₃): δ 19.66 (C-1), 159.78 (C-8), 146.28 (C-10), 134.35 (C-6), 121.11 (C-9), 119.73 (C-5), 111.54 (C-7), 95.89 (C-12), 71.01 (C-4), 62.39 (C-16), 56.03 (C-11), 35.98 (C-2), 30.75 (C-13), 26.96 (C-15), 25.34 (C-3), 19.28 (C-14).

Anal. Calcd for C₁₆H₂₀O₄: C, 69.54; H, 7.30. Found: C, 69.77; H, 7.46.

1-Tetrahydropyranyl-4-Benzoyl-8-Methoxytetralin (6)

To the tetralone 5 (510 mg, 1.85 mmol) dissolved in ethanol (40 mL) was added sodium borohydride (10 mg, 2.64 mmol) and stirred for 3 h at room temperature. The alcohol was partly evaporated under pressure, added water and extracted with dichloromethane (80 mL). The organic extract was dried and evaporated to dryness. The crude product (535 mg) was dissolved in pyridine (3 mL) followed by the addition of benzoyl chloride (0.7 mL, 6 mmol). The reaction mixture was stirred for 20 h at room temperature, diluted with cold water and extracted with ether. The organic extract was washed with a solution of copper sulfate (10 mL, 20%), sodium hydroxide (15 mL, 10%), water, dried and evaporated. The residue was chromatographed (eluant hexane: ether 6:4) to obtain the tetralin 6 (520 mg, 73%); mp: 119°C; IR (cm⁻¹): 1712 (CO); MS (m/z): 159 (M + 1-PhCOOH-C₅H₉O₂); ¹H NMR (CDCl₃): δ 7.99 (dd, 2H, J= 7.4, 1.4 Hz, H-19, H-23), 7.50 (tt, 1H, J= 7.4, 1.4 Hz, C-8), 7.39-7.30 (m, 3H, H-20, H-21, H-22), 7.07 (d, 1H, J=8 Hz, H-7), 6.77 (d, 1H, J=8 Hz, H-6), 6.37 (t, 1H, J=3.5 Hz, H-4), 4.97 (t, 1H, J= 4.2 Hz, H-1), 4.64 (1H, dd J=10, 5.4 Hz, H-12), 4.03-3.96 (m, 1H, CH₂), 3.68 (s, 3H, OMe), 2.31-1.55 (m, 10H); ¹³C NMR: δ 165.93 (C-17), 158.14 (C-5), 141.07 (C-9), 132.50 (C-21), 130.89 (C-18), 129.66 (C-19), 129.43 (C-7), 128.10 (C-20), 122.81 (C-10), 118.80 (C-8), 109.78 (C-6), 101.15 (C-12), 64.97 (C-4), 62.48 (C-16), 55.58 (C-11), 30.94 (C-13), 26.82 (C-15), 26.76 (C-3), 25.47 (C-2), 19.61 (C-14).

Anal. Calcd for C₂₃H₂₆O₅: C, 72.23; H, 6.85. Found: C, 72.42; H, 6.99.

1-Hydroxy-4-benzoyl-5-methoxytetralin (7)

To the tetralin 6 (120 mg, 0.31 mmol) in methanol (5 mL) was added ammonium chloride (50 mg, 0.93 mmol) and was heated for 4 h. The resulting was diluted with water (20 mL) and extracted with ether (20 mL). The combined ethereal extracts were washed with water (10 mL), dried and evaporated. The resulting oil was chromatographed (eluant hexane: ether 6:4) to obtain the tetralin (67 mg, 76%); mp: 134°C (ether); IR (cm⁻¹): 3399 (OH), 1710 (CO); MS (m/z): 176 (M- C₆H₅COOH), 159 (M+1-C₆H₅COOH-

H₂O); ¹H NMR (CDCl₃): δ 7.99 (dd, 2H, J= 7.4,1.4 Hz, H-14,H-18), 7.50 (tt, 1H, J= 7.4, 1.4 Hz, H-16), 7.40-7.34 (m, 3H, H-8,H-15,H-17), 7.28 (d, 1H, J=8 Hz, H-7), 6.79 (d, 1H, J=3.4 Hz, H-6), 6.39 (t,1H, J=3.4.Hz, H-4), 4.81 (dd, 1H, J=10 .0, 5.4 Hz, H-4), 3.70 (s, 3H, OMe), 1.99-2.24 (m, 4H,H-2, H-3), 1.84-1.76 (s, br, 1H, OH); ¹³C NMR (CDCl₃): δ 165.89 (C-12), 157.66 (C-5), 142.55 (C-9), 132.58 (C-16), 130.81 (C-13), 130.04 (C-7), 129.81 (C-14), 128.15 (C-15), 122.40 (C-10), 118.43 (C-8), 109.30 (C-6), 65.09 (C-4), 64.97 (C-1), 55.59 (C-11), 28.27 (C-2), 26.36 (C-3).

Anal: Calcd for C₁₈H₁₈O₄: C, 72.46; H, 6.08. Found: C, 72. 69; H, 6.26.

4-Benzoyl-5-Methoxy-1-Tetralone (8)

To the Sarett reagent, prepared by the addition of chromic acid (200 mg, 20 mmol) to dry pyridine (2 mL) was added alcohol 7 (150 mg, 0.50 mmol) dissolved in dry pyridine (2 mL). The deep brown solution was stirred at room temperature for 24 h, diluted with ether (30 mL), filtered over celite-silica and washed celite-silica with ether (2x5 mL). The combined washed extract was washed with hydrochloric acid (15 mL, 30 %) and then with a solution of the copper sulfate (20 mL, 10%). The organic extract was washed, dried and evaporated. The resulting residue was chromatographed (eluant hexane: ether 7:3) to obtain the ketone 8 (129 mg, 87 %); mp: 116-117 oC (ether); IR (cm⁻¹): 1715, 1692 (CO); MS (m/z): 296 (M⁺),191 (M⁻ COC₆H₅); ¹H NMR (CDCl₃): δ 8.00 (dd, 2H, J= 8.0, 1.3 Hz, C-14, C-18), 7.70 (d, 1H, J= 8 Hz, H-16), 7.52 (tt, 1H, J= 8 .0, 1.3 Hz, H-7), 7.46 (t, 1H, J= 8 Hz,H-6), 7.39 (t, 2H, J= 8 Hz, H-15, H-17), 7.11 (d, 1H, J= 8 Hz,), 6.67 (t, 1H, J= 3 Hz, H-4), 3.80 (s, 3H, OMe), , 2.37-2.93(m,4H,H-2, H-3); ¹³CNMR (CDCl₃): δ 197.67 (C-1), 165.80 (C-12), 133.66 (C-13), (132.94 (C-16), 130.19 (C-9), 129.65 (C-14, C-9),128. 30 (C-15), 127.99 (C-10), 118.55 (C-8), 115.58 (C-6), 63.70 (C-4), 55.97 (C-11), 33.20 (C-2), 27.42 (C-3).

Anal. Calcd for C₁₈H₁₆O₄: C, 72.96; H, 5.44. Found: C, 73.12; H, 5.66

5-O-Methylsclerone (9)

To a solution of the tetralone 8 (60 mg, 0.31 mmol) in ethanol (20 mL) was added potassium carbonate (240 mg, 1.25 mmol) and stirred at room temperature for 24 h. The alcohol was evaporated and the residue was extracted with dichloromethane (20 mL). The organic extract was washed, dried, filtered and evaporated. The residue was (eluant hexane: ether 1:1) to obtain the 5-O-methylsclerone 9 (50.5 mg, 85 %) as oil; IR(cm⁻¹): 3420 (OH), 1683 (CO);MS(m/z): 192 (M⁺), 175 (M⁺-H₂O);¹HNMR (CDCl₃): δ 7.61 (dd, 1H, J= 8Hz, 1.1 Hz, H-6), 7.36 (t, 1H, J= 8 Hz, H-7), 7.09 (dd, 1H, j= 8.0, 1.1 Hz,H-8), 5.26 (t, 1H, J=8 Hz,, H-4), 3.90 (s, 3H, OMe), 3.01-2.25 (m ,4H, H-2,3) ; ¹³C NMR (CDCl₃): δ 198.08 (C-1), 156.98 (C-5), 132.47 (C-9), 132.33 (C-10), 129.11 (C-7), 118.92 (C-8), 115.30 (C-6), 62.12 (C-4), 55.80 (C-11), 33.65 (C-2), 29.37 (C-3). Anal. Calcd for C₁₁H₁₂O₃: C, 68.73; H, 6.29.

Found: C, 68.89; H, 6.43.

5-Methoxytetralin (10)

To a solution of the benzoate 8 (80 mg, 0.27 mmol) in alcohol (10 mL) was added platinum oxide (8 mg, 0.03 mmol) and stirred for 3 h under hydrogen. The progress of the reaction was observed by TLC. The reaction mixture was filtered and washed the residue with alcohol (3 mL). The combined filtrate was evaporated and the resulting residue was chromatographed (eluant hexane) to yield the tetralin 10 (30 mg, 68 %) as liquid; MS(m/z): 162 (M⁺); ¹H NMR (CDCl₃): δ 7.15 (t, 1H, J= 8Hz, C-8), 6.78 (d, 1H, J= 8Hz,H-7), 6.73 (d, 1H, J= 8Hz,H-6), 3.88 (s, 3H, OMe), 2.79 (dt, 4H, J= 280, 6 Hz), 1.92-1.80(m, 4H,H-2,3); ¹³C NMR (CDCl₃): δ 157.40 (C-5), 138.50 (C-9), 125.94 (C-10), 125.70 (C-7), 121.44 (C-8), 106.77(C-6), 55.19 (C-11), 29.66 (C-1), 23.11 (C-3), 22.90 (C-2).

Anal. Calcd for C₁₁H₁₄O: C, 81.44; H, 8.70. Found: C, 81.65; H, 8.88.

8-Methoxy-1-Tetralone (11)

To a solution of the tetralin 10 (500 mg, 3.09 mmol) in acetic acid (10 mL), cooled at 15 oC, was added a solution of chromic acid (7 mL) in acetic acid-water (19:1). The reaction mixture was stirred for h at 17-21 oC, filtered over silica gel and then washed the silica gel with acetic acid (3 mL). To the combined filtrate was added dichloromethane (30 mL) and stirred for 15 min. The organic extract was washed with a solution of sodium hydroxide (2 x 8 mL, 10 %), dried and evaporated to dryness. The residue was chromatographed (eluant hexane: ether 7:3) to obtain 8-methoxy-1-tetralone 11 (76 mg, 14 %); IR(cm⁻¹): 1672 (CO); MS(m/z): 176 (M⁺), 148 (M⁺-MeOH); ¹H NMR (CDCl₃): δ 7.35 (dd, 1H, J= 8.4, 7.6 Hz, H-5),6.81-6.78 (m,2H, H-6,7) ,3.87 (s, 3H ,OMe),2.89 (t, 2H, J=6.0 ,H-2),2.62 (t,2H ,J=6.0 Hz, H-3), 2.07-1.98 (m, 2H ,H-4); ¹³C NMR (CDCl₃): 197.55 (C-1), 160.33 (C-8), 147.11 (C-10), 133.88 (C-6), 122.19 (C-9), 120.68 (C-5), 109.89 (C-7), 55.92 (C-11), 40.89 (C-2), 30.78 (C-4), 22. 80 (C-3). Anal. Calcd for C₁₁H₁₂O₂: C, 74.97; H, 6.86. Found: C, 75.16; H, 6.94.

Further elusion (eluant hexane: ether 6:4) afforded 5-methoxy-1-tetralone 12 (249 mg, 46 %) whose mp. 87-89 oC was compared with commercially available tetralone 12 (mp. 85-89 oC) (Aldrich), the mixed mp. did not show any depression.

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