

Design and Synthesis of Three Naphthol Derivatives Using Several Strategies

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In this study, three naphthol derivatives were synthesized. The first stage, involved preparation of 3-[[2-(3-hydroxy-13-methyl-6,7,8,9,11,12,13,14,15,16-decahydro-cyclo-penta[a]phenanthren-17-ylideneamino)-ethylamino]-(3-hydroxy-naphtalen-2-yl)-methyl]-1,7,7-trimethyl-bicyclo[2.2.1]heptan-2-one (**4**) by the reaction of 1,7,7-trimethyl-bicyclo[2.2.1]heptan-2-one (**1**) with 17-(2-amino-ethylimino)-13-methyl-7,8,9,11,12,13,14,15,16,17-decahydro-6H-cyclopenta[a]phenanthren-3-ol (**2**) and 2-hydroxy-1-naphthaldehyde (**3**) using proline as catalyst. The second stage was achieved by the reaction of compund **1** with 1-[(2-amino-ethylamino)-phenyl-methyl]-naphthalen-2-ol to form the compound 1-{phenyl-[2-(1,7,7-trimethyl-bicyclo[2.2.1]hept-2-ylideneamino)ethyl-amino]-methyl]naphtalen-2-ol (**6**) using boric acid as catalyst. The third stage was achieved by the synthesis of N-[3-(1-hydroxy-10a,12a-dimethyl-2,2,3a,3b,4,5,10,10a,10b,11,12,12a-dodecahydro-1H-7-oxa-8-azadicyclopenta[a,h]phenanthren-1-ylethynylamino)-3-(3-hydroxy-naphtalen-2-yl)-propyl]-succinamic acid 3-allyl-5-methoxy-phenylester (**9**) using the three components system (2-hydroxy-1-naphthaldehyde, 4-allyl-2-methoxyphenyl 4-[(2-aminoethyl)amino]-4-oxobutanoate anddanazol). The structure of all compounds obtained was confirmed by elemental analysis, spectroscopy and spectrometry data.

Keywords: Naphthol derivative, Boric acid, Proline.

INTRODUCTION

There are several methods reported for synthesis of naphthol derivatives; for example, the synthesis of 4-isopropoxynaphthalene-1-carbaldehyde by the reaction of isopropyl o-ethynyl benzoate with butyl vinyl ether using PtCl₂ as catalyst¹. Other reports indicate the tandem pummerer Diels-Alder sequence for the preparation of α -thiosubstituted naphthalene derivatives². Additionally, other studies³ showed the synthesis of 1carbamato-alkyl-2-naphthol derivatives via a three-component condensation reaction between aryl aldehydes, 2-naphthol and carbamates in the presence of SiO2-NaHSO4 as a heterogeneous catalyst under thermal and solvent-free conditions³. Other reports indicate the synthesis of 1,8-diphenylnaphtalene and 1-iodo-8-phenylnaphtalene by the reaction of lithium diphenyl cuprate and aryl halides⁴. Also, there are studies⁵ which shown the development of (S)-1-(3-hydroxy-2-naphthylcarbonyl)pyrrolidine-2-carboxylic acid methyl ester by the reaction of 3-hydroxy-2-naphthoic acid and (S)-proline methyl ester in presence of SOCl₂. In addition, some carbamato-alkyl-naphthol derivatives^{6,7} have been synthetized by condensation of βnaphthol, aromatic aldehyde and methyl carbamate in ionic

liquid media. Other studies indicate that the compound N-(2,4-dibromonaphthyl)benzamide was prepared by benzoylation of 2,4-dibromonaphthylamine in pyridine⁸. Additionally, other naphthol derivative [N-{3-mercapto-5-(naphthalen-1-yl)-4H-1,2,4-triazol-4-yl}benzamide] was synthesized by the reaction of potassium 2-(2-naphthoyl)hydrazine carbodithioate with N-aminoarylcarboxamides in ethanol to reflux9. The compound N-(2-{[(2-hydroxy-naphtalen-1-yl)-phenyl-methyl]amino}-ethyl)-3,4-dinitrobenzamide was synthesized by the reaction of 1-[(2-amino-ethylamino)-phenyl-methyl]naphthalen-2-ol with 3,5-dinitrobenzoic acid using 1-ethyl-3-(3-dimethylaminopropyl)-carbodiimide or boric acid as catalyst¹⁰. All these experimental results show several procedures that are available for synthesis of naphthol derivatives. Nevertheless, expensive reagents and special conditions are required. Therefore, in this study three naphthol derivatives were synthesized using several chemical strategies.

EXPERIMENTAL

The compounds **2** (17-(2-amino-ethylimino)-13-methyl-7,8,9,11,12,13,14,15,16,17-deca-hydro-6*H*-cyclopenta[a]- phenanthren-3-ol), **5** (1-[[(2-aminoethyl)amino]-(phenyl) methyl]-2-naphthol) and **7** (4-allyl-2-methoxyphenyl 4-[(2-aminoethyl)amino]-4-oxobutanoate) were prepared according to a reported method^{10,11}. The other compounds used in this study were purchased from Sigma-Aldrich Co., Ltd. The melting points for the different compounds were determined on an Electrothermal (900 model). Infrared spectra (IR) were recorded using KBr pellets on a Perkin Elmer Lambda 40 spectrometer. ¹H and ¹³C NMR spectra were recorded on a Varian VXR-300/5 FT NMR spectrometer at 300 and 75.4 MHz in CDCl₃ using TMS as internal standard. EIMS spectra were obtained with a Finnigan Trace GCPolaris Q. spectrometer. Elementary analysis data were acquired from a Perkin Elmer Ser. II CHNS/0 2400 elemental analyzer.

Synthesis of 3-[[2-(3-hydroxy-13-methyl-6,7,8,9,11, 12,13,14,15,16-decahydro-cyclo- penta[a] phenanthren-17ylideneamino)ethylamino]-(3-hydroxy-naphthalen-2-yl)methyl]-1,7,7-trimethyl-bicyclo[2.2.1] heptan-2-one (4) (Fig. 1): A solution of 1,7,7-trimethylbicyclo[2.2.1]heptan-2-one (1) (50 mg, 0.32 mmol), compound 2 (100 mg, 0.32 mmol), 2hydroxy-1-naphthaldehyde (55 mg, 0.32 mmol) and proline (110.50 mg, 0.96 mmol) in 10 mL of methanol was stirring for 72 h to room temperature. The reaction mixture was evaporated to a smaller volume. After the mixture was diluted with water and extracted with chloroform. The organic phase was evaporated to dryness under reduced pressure, the residue was purified by crystallization from methanol: Water (3:1) yielding 75 % of product, m.p. 98-100 °C; IR (Kbr, v_{max}, cm⁻¹): 3410, 3338, 3320, 1718; ¹H NMR (300 MHz, CDCl₃)δ_H: 0.84 (s, 6H), 0.90 (s, 3H), 0.96 (s, 3H), 1.24 (m, 1H), 1.38-1.49 (m, 2H), 1.54 (m, 1_H), 1.58-1.62 (m, 2H), 1.70-1.78 (m, 3H), 1.86 (m, 1H), 1.88-2.24 (m, 6H), 2.48 (m, 1H), 2.52.2.85 (m, 4H), 2.87-3.17 (t, 2H, J = 6.44), 3.40 (t, 2H, J = 6.44), 4.26 (m, 1H), 4.79 (broad, 3H), 6.58-6.65 (m, 2H), 7.06-7.43 (d, 2H, *J* = 8.10), 7.45 (m, 2H), 7.50-7.71 (d, 4H, J = 8.10) ppm. ¹³C NMR (75.4 Hz, CDCl₃) $\delta_{\rm C}$: 9.35 (C-10), 15.70 (C-44), 19.02 (C-9), 19.34 (C-8), 20.99 (C-3), 21.90 (C-25), 25.78 (C-26), 26.00 (C-22), 27.50 (C-24),

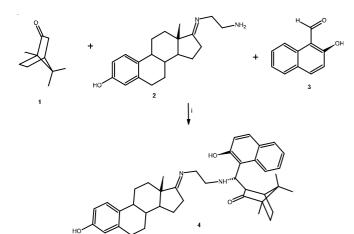


Fig. 1. Synthesis of 3-[[2-(3-hydroxy-13-methyl-6,7,8,9,11,12,13,14,15,16-decahydro-cyclopenta[a]phenanthren-17-ylideneamino)-ethylamino]-(3-hydroxy-naphthalen-2-yl)-methyl]-1,7,7-trimethyl-bicyclo[2.2.1]heptan-2-one (4). Reaction of 1,7,7-trimethyl-bicyclo[2.2.1]heptan-2-one (1) with 17-(2-amino-ethylimino)-13-methyl-7,8,9,11,12,13,14,15,16,17-decahydro-6H-cyclopenta[a]-phenanthren-3-ol (2) and 2-hydroxy-1-naphthaldehyde (3) to form 4. i = proline/room temperature

29.00 (C-27), 29.61 (C-4), 32.36 (C-23), 37.60 (C-20), 41.43 (C-18), 43.40 (C-23), 45.09 (C-1), 46.70 (C-14), 50.78 (C-2), 53.23 (C-15), 54.29 (C-19), 55.10 (C-12), 55.66 (C-7), 57.64 (C-5), 112.60 (C-31), 112.90 (C-42), 114.88 (C-33), 120.27 (C-40), 122.86 (C-36), 123.76 (C-38), 124.98 (C-43), 125.25 (C-34), 125.64 (C-30), 126.40 (C-39), 129.08 (C-37), 130.58 (C-29), 131.90 (C-35), 135.38 (C-28), 150.82 (C-41), 153.96 (C-32), 176.64 (C-17), 219.22 (C-6) ppm. EI-MS *m/z*: 618.30 (M⁺10). Anal. calcd. for C₄1H₅₀N₂O₃: C, 79.57; H, 8.14; N, 4.53. Found: C, 79.50; H, 8.10.

Synthesis of 1-{phenyl-[2-(1,7,7-trimethyl-bicyclo-[2.2.1]hept-2-ylideneamino)ethyl-amino]-methyl}naphthalen-2-ol (6) (Fig. 2): A solution of 1-[(2-aminoethylamino)-phenyl-methyl]-naphthalen-2-ol (100 mg, 0.34 mmol), 1,7,7-trimethylbicyclo[2.2.1]heptan-2-one (77 mg, 0.50 mmol) and boric acid (50 mg, 80 mmol) in 10 mL of methanol was stirring for 72 h to room temperature. The reaction mixture was evaporated to a smaller volume. After the mixture was diluted with water and extracted with chloroform. The organic phase was evaporated to dryness under reduced pressure, the residue was purified by crystallization from methanol: Water (3:1) yielding 60 % of product, m.p. 62-64 °C; IR (KBr, v_{max} , cm⁻¹): 3406, 3342, 3324; ¹H NMR (300 MHz, CDCl₃) δ_{H} : 0.84 (s, 3H), 0.91 (s, 3H), 0.93 (s, 3H), 1.27-2.58 (m 7H), 2.97-3.01 (t, 2H, J = 6.44), 3.59 (t, 2H, J = 6.44), 4.24 (broad, 2H), 5.15 (m, 1H), 6.88 (m, 2H), 7.07 (m, 1H), 7.12 (m, 2H), 7.27 (m, 1H), 7.41-7.78 (m, 5H) ppm. ¹³C NMR (75.4 Hz, CDCl₃)δ_C: 11.20 (C-10), 19.02 (C-9), 19.34 (C-8), 27.50 (C-3), 31.22 (C-4), 36.16 (C-7), 43.78 (C-2), 47.40 (C-1), 50.60 (C-13), 53.38 (C-12), 55.53 (C-15), 56.34 (C-5), 114.36 (C-18), 121.18 (C-22), 123.57 (C-24), 126.60 (C-23), 127.30 (C-29), 127.90 (C-16), 128.01 (C-20), 128.14 (C-19), 129.20 (C-27, C-31), 129.28 (C-25), 130.20 (C-28, C-30), 138.34 (C-21), 139.30 (C-26), 152.10 (C-17), 180.84 (C-6) ppm. EI-MS m/z: 426.20 (M⁺10). Anal. calcd. for C₂₉H₃₄N₂O: C, 65.00; H, 8.03; N, 6.57. Found: C, 64.98; H, 8.00.

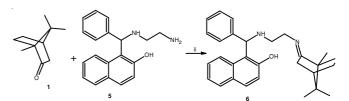


Fig. 2. Synthesis of 1-{phenyl-[2-(1,7,7-trimethyl-bicyclo[2.2.1]hept-2ylideneamino)ethyl-amino]-methyl}naphthalen-2-ol (6). Reaction of 1,7,7-trimethylbicyclo[2.2.1]heptan-2-one (1) with 1-[(2-aminoethylamino)-phenyl-methyl]-naphthalen-2-ol (5) to form 6. ii = boric acid/room temperature

Synthesis of N-[3-(1-hydroxy-10a,12a-dimethyl-2,2,3a,3b,4,5,10,10a,10b,11,12,12a-dodecahydro-1H-7-oxa-8-aza-dicyclopenta[a,h]phenanthren-1-ylethynylamino)-3-(3-hydroxy-naphthalen-2-yl)-propyl]-succinamic acid 3allyl-5-methoxy-phenyl ester (9) (Fig. 3): A solution of 2hydroxy-1-naphthaldehyde, 4-allyl-2-methoxyphenyl 4-[(2aminoethyl)amino]-4-oxobutanoate and danazol in 10 mL of ethanol was stirring for 72 h to room temperature. The reaction mixture was evaporated to a smaller volume. After the mixture was precipitated with water and separated, yielding 70 % of product, m.p. 140 °C; IR (KBr, v_{max} , cm⁻¹): 3378, 1718, 1188; ¹H NMR (300 MHz, CDCl₃) $\delta_{\rm H}$: 0.99 (s, 3H), 1.01 (s, 3H), 1.03-2.36 (m, 17H), 2.52-2.57 (t, 4H), 3.42 (t, 2H), 3.46 (t, 2H), 3.52 (t, 2H), 4.05 (s, 3H), 5.18-5.95 (t, 2H), 6.17 (d, 1H), 7.1-7.53 (d, 8H), 7.76 (d, 1H), 8.00 (broad, 3H), 8.10 (d, 1H) ppm. ¹³C NMR (75.4 Hz, CDCl₃) δ_{c} : 12.80 (C-22), 19.44 (C-21), 21.10 (C-19), 22.94 (C-13), 29.50 (C-43), 31.35 (C-17), 31.69 (C-18), 31.92 (C-42), 33.02 (C-20), 33.64 (C-5), 35.95 (C-6), 37.03 (C-20), 38.73 (C-12), 39.92 (C-56), 43.56 (C-28), 47.40 (C-10), 52.88 (C-7), 53.15 (C-9), 55.85 (C-55), 58.46 (C-27), 67.26 (C-24), 81.13 (C-11), 86.34 (C-25), 110.50 (C-15), 113.08 (C-48), 113.46 (C-34), 116.01 (C-58), 119.69 (C-38), 121.44 (C-4), 121.66 (C-51), 122.79 (C-50), 123.21 (C-40), 123.83 (C-35), 125.61 (C-39), 128.70 (C-41), 130.30 (C-36), 133.57 (C-37), 134.42 (C-32), 137.56 (C-57), 139.12 (C-49), 139.20 (C-46), 141.56 (C-16), 142.01 (C-33), 149.58 (C-3), 151.30 (C-47), 156.04 (C-14), 168.64 (C-44), 169.68 (C-30) ppm. EI-MS m/z: 783.36 (M⁺ 10). Anal. calcd. for C48H53N3O7: C, 73.54; H, 6.81; N, 5.36. Found: C, 73.50; H, 6.80.

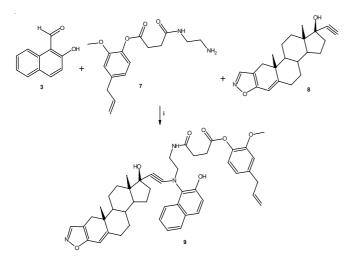


Fig. 3. Synthesis of N-[3-(1-hydroxy-10a,12a-dimethyl-2,2,3a,3b,4,5,10, 10a,10b,11,12,12a-dodecahydro-1H-7-oxa-8-aza-dicyclopenta-[a,h]phenanthren-1-ylethynylamino)-3-(3-hydroxy-naphthalen-2-yl)-propyl]-succinamic acid 3-allyl-5-methoxy-phenyl ester (9). Reaction of 2-hydroxy-1-naphthaldehyde (3), 4-Allyl-2-methoxy-phenyl 4-[(2-aminoethyl)amino]-4-oxobutanoate (7) and danazol (8) to form 9. iii = Ethanol/room temperature

RESULTS AND DISCUSSION

Several strategies are available for enantioselective catalysis, including heterogeneous catalysis¹²⁻¹⁴. For example, proline is used as catalyst in asymmetric reactions (three components system) from ketones, aldehydes and amines^{15,16}. Therefore, in this study we report a straight forward route for synthesis of 3-[[2-(3-hydroxy-13-methyl-6,7,8,9,11,12,13,14, 15,16-decahydro-cyclopenta[a]phenanthren-17-ylideneamino)-ethylamino]-(3-hydroxy-naphtalen-2-yl)-methyl]-1,7,7-trimethyl-bicyclo[2.2.1]heptan-2-one (4) by the reaction of 1,7,7-trimethylbicyclo[2.2.1]heptan-2-one with 17-(2amino-ethylimino)-13-methyl-7,8,9,11,12,13,14,15,16,17decahydro-6H-cyclopenta[a]phenanthren-3-ol and 2-hydroxy-1-naphthaldehyde using proline as catalyst. The¹H NMR spectrum of **4** shows signals at 0.84 and 0.90 ppm for methyl groups bound to bicyclic ring; at 0.96 ppm for methyl group bound to steroid nucleus; at 1.24, 1.54, 1.70-1.78, 1.88-2.24,

2.52-2.85, 6.58-6.65 and 7.45 ppm for steroid nucleus; at 1.38-1.49, 1.58-1.62, 1.86 and 2.48 ppm for protons involved in the bicyclic ring; at 2.87-3.17 and 3.40 ppm for arm bound to both imino and amino groups; at 4.26 ppm for methylene bound to both phenyl and amino groups; at 4.79 ppm for both amino and hydroxyl groups; at 7.06-7.71 ppm for phenyl groups.

On the other hand, the 13 C NMR spectrum of compound **4** contains peaks at 9.35, 19.02 and 19.34 ppm for methyl groups bound to bicyclic ring; at 15.70 ppm for methyl group of steroid fragment; at 20.99, 29.61, 45.09, 50.78, 55.10, 55.66 and 219.22 ppm for protons involved in the bicyclic ring; at 21.90, 25.78, 26.00, 27.50, 29.00, 32.36, 37.60, 41.43, 43.40, 54.29, 112.60, 114.88, 125.64, 130. 58, 135.38 153.32 and 176.64 ppm for steroid nucleus; at 46.70 and 53.23 ppm for arm bound to both imino and amino groups; 55.10 ppm for carbon bound to amino, phenyl and bicyclic ring; at 112.90, 120.27, 122.86-125.25, 126.40-129.08, 131.90 and 150.82 ppm for phenyl groups. Finally,the presence of compound **4** was further confirmed from mass spectrum which showed a molecular ion at m/z 618.30.

The second stage was achieved by the synthesis of 1-{phenyl-[2-(1,7,7-trimethyl-bicyclo[2.2.1]hept-2-ylideneamino)ethyl-amino]-methyl}naphthalen-2-ol (6); this compound has in the chemical structure an imino group. It is importantto mention that there are several procedures for thesynthesis of imines which are described in the literature^{10,17}. For example, the synthesis of imines by the reaction of benzaldehyde derivative with benzene-1,2-diamine using boric acid as catalyst¹⁸. Therefore, in this study the compound $\mathbf{6}$ was developed by the reaction of 1-[(2-amino-ethylamino)-phenylmethyl]-naphthalen-2-ol and 1,7,7-trimethyl-bicycle-[2.2.1]heptan-2-one using boric as catalyst to form an imino group involved in the compound 6. The ¹H NMR spectrum of 6 shows signals at 0.84, 0.91 and 0.93 ppm for methyl groups bound to bicyclic ring; at 1.27-2.58 ppm for protons involved in the bicyclic ring; 2.97-3.01 and 3.59 ppm for methylene groups bound to both imino and amino groups; at 4.29 ppm for both amino and hydroxyl groups; at 5.15 ppm for methylene group bound to both amino and phenyl groups; at 6.88-7.78 ppm for phenyl groups. The ¹³C NMR spectrum of 6 contains peaks at 11.20, 19.02, 19.34 ppm for methyl groups bound to bicyclic ring; at 27.50, 31.22, 36.16, 43.78, 47.40, 56.34 and 180.84 ppm for protons involved in the bicyclic ring; at 55.53 ppm for methylene group bound to both amino group and phenyl groups; at 50.60 and 53.38 ppm for methylene groups bound to imino and amino groups; at 114.36-152.10 ppm for phenyl groups. Finally, the presence of compound 6 was further confirmed from mass spectrum which showed a molecular ion at *m/z* 426.20.

The third stage was achieved by the synthesis of compound **9** using the three components(2-hydroxy-1-naphthaldehyde, 4allyl-2-methoxyphenyl4-[(2-aminoethyl) amino]-4-oxobutanoate and danazol). It is important to mention, that there are many procedures which have been used with this method in order to synthesize several compounds. The most widely practiced method employs boric acid¹⁹, silica sulfuric acid²⁰, poly(4-vinylpyridinecodivynylbenzene)-Cu(II)complex²¹, H₂SO₄²², silica triflate²³ and phosphorus pentoxide²⁴. Nevertheless, despite its wide scope, the former protocols suffer from several drawbacks *e.g.*, some reagents have a limited stability and its preparation can be dangerous. Analyzing these data, in this study a straight forward route is reported for the synthesis of **9**. The ¹H NMR spectrum of **9** shows signals at 0.99 and 1.01 ppm for methyl groups bound to steroid nucleus; at 1.03-2.52, 6.17 and 7.76 ppm for steroid nucleus; at 2.52-2.57 ppm for methylene groups bound to both amide and ester groups; at 3.42-3.46 ppm for methylene group bound to both amino groups; 3.52 ppm for methylene group bound to phenyl group; at 4.05 for methoxy group; 5.18-5.95 ppm for protons of alkene group; at 7.10-7.53 and 8.10 ppm for phenyl groups. Finally, other signal at 8.00 ppm for amino and hydroxyl groups were found.

On the other hand, the ¹³C NMR spectrum of **9** contains peaks at 12.80 and 19.44 ppm for methyl groups bound to steroid nucleus; at 21.10, 22.94, 31.35-31.69, 33.02-38.73, 47.40-53.15, 81.13, 110.50, 121.44, 141.56 and 149.58-156.04 ppm for steroid nucleus; at 29.50 and 31.92 ppm for carbons bound to both ester and amide groups; at 39.92 ppm for methylene group bound to phenyl group; at 43.56 and 58.46 ppm for carbons bound to amino groups; at 55.85 ppm for methoxy group; at 67.26 and 86.34 ppm for alkyne group; at 113.08-113.46, 119.69, 121.66-134.42, 139.12-139.20 and 142.01 ppm for phenyl groups; at 116.01 and 137.56 ppm for alkene group. Finally, other signals at 168.64 ppm for ester group; at 169.68 ppm for amide group. In addition, the presence of **9** was further confirmed from mass spectrum which showed a molecular ion at m/z 783.36.

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