



Synthesis of 1-naphthoate and 2-naphthoate sulfide podandsbearing aliphatic long chains

Esmael Rostami^{2*}, Fatemeh Dehghani¹, Maryam Bagherzade¹, Akram Khodadadi¹,
Ay Soda Ghobadpoor¹

¹Department of Chemistry, Payame Noor University, PO BOX 19395-3697, Tehran, (IRAN)

²EsmaelRostami, Faculty of Chemistry, Bushehr Payame Noor University, P.O.Box 1698, Bushehr, (IRAN)

E-mail : Esmaelrostami@gmail.com; Esmrostami@yahoo.com

ABSTRACT

New receptors were prepared in good yields from the reaction of 1-naphthol and 2-naphthol derivatives with acid chlorides of palmitoyl, decanoyl and octanoylin NMP. All of the products well identified and characterized using conventional methods such as IR, ¹H NMR, ¹³C NMR and mass analysis.

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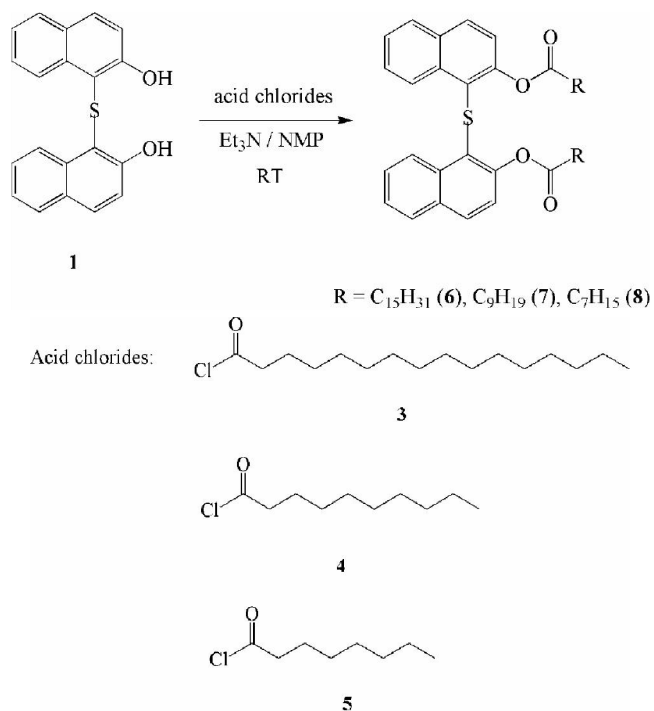
KEYWORDS

Receptor;
1-naphthol sulfide;
2-naphthol sulfide;
Diesterpodand;
Palmitoyl chloride;
Octanoyl chloride;
Decanoyl chloride.

INTRODUCTION

Crown ethers were synthesized by pederen and a large number of receptors were repoted^[1]. Diaza Crowns are an important class of host molecules^[2]. After synthesis and complexation studies of crown ethers, macrocyclescontaning ester and another functional groups were prepared^[3]. Their complexaton studies were performed and they showed strong complexation interaction between macrocycles and metal ions^[4]. Diesterpodandsand related receptors are another group of receptors which can be used as an important hosts in host-guest chemistry^[5]. They have been used as building blocks for the construction of a large number of supramolecular systems^[6].

Receptors bearing a polar core and long nonpolar tiles are important candidates for the study as phase transfer catalysts, membrane construction and as amphiphiles. Introduction of naphthalene ring in these receptors are important for studies under instrumental



Scheme 1 : Synthesis of 2-naphthol podands (6-8).

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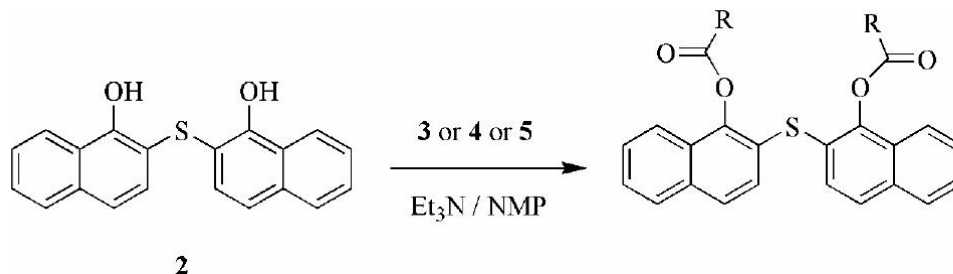
procedures such as fluorocene and on the other hand naphthalene is a nonpolar group and change the solubility of receptors.

In continuation of our research for the synthesis of new receptors^[7], in this research article new diesterpodands were prepared.

RESULTS AND DISCUSSION

(1) and (2) were reported previously^[7] podands (6-11) were prepared from the reaction of (1) and (2) with acid chlorides in NMP in the presence of triethylamine. Palmitoyl chloride, decanoyl chloride and octanoyl chloride were reacted with (1) and (2) to afford podands in good yield.

These podands are important candidate for stabilization of nanoparticles and nanostructures. On the other hand, these podands are important for study of micelles and realted aria. The long tiles in their structures are a capability for systems such as membrane, biological media and biothechnologica studies.



Scheme 2 : Synthesis of 1-naphthol podands (9-11).

General procedure for the synthesis of dinaphtho sulfidediesterpodands

To (1) or (2) (1mmol, 0.32g) in a round bottom flask, were added NMP (1-methyl-2-pyrrolidone) (30 ml), acid chloride (palmitoyl chloride, decanoyl chloride and octanoyl chloride, 1 mmol) and triethylamine (2 mmol). The mixture was stirred at room temperature for 48h. After completion of the reaction (monitored by TLC), water was added and the resulting precipitate was filtered. The precipitate was recrystallized in ethanol to afford the desired product.

Synthesis of 1,1'-thiobis (1-oxa-2-oxo-heptadecyl) naphthalene (6)

According to the general procedure (6) was pre-

EXPERIMENTAL

The reactions were carried out in an efficient hood cupboard. All the materials were purchased from Merck, Fluka, Across Organics and Aldrich chemical companies. Acetonitrile were distilled and stored over molecular sieves. DMF was distilled over molecular sieves under reduced pressure and stored over them. Merck silica gel 60 was used for column chromatography. Merck silica gel 60 F₂₅₄ TLC plates were used for thin layer chromatography (TLC). The melting points (uncorrected) were measured with an Electrothermal engineering LTD 9100 apparatus. Elemental analysis was performed by a CHN-O- Rapid Heraeus elemental analyzer. IR spectra were measured on a Perkin-Elmer model 543 and FT-IR BRUKER spectrometers. For some samples the ¹H NMR and ¹³C NMR spectra were obtained using BRUKER AVANCE DPX 300 MHz ¹H NMR and 75 MHz ¹³C NMR instrument. Mass spectra were obtained with Shimadzu GC-MS-QP 1100 EX model.

pared from the reaction of (1) and palmitoyl chloride in 91% yield and melting point of 115-116°C; IR (KBr): 3496, 3041, 3009, 2973, 2963, 2851, 1743, 1596, 1508, 1471, 1456, 1310, 1255, 1202, 1196, 1095, 1043, 811, 754cm⁻¹; ¹H NMR (300 MHz, DMSO-d₆) δ: 0.87 (t, J= 7Hz, 3H), 1.25-1.28 (m, 24H), 1.62 (m, 2H), 2.22-2.30 (m, 2H), 7.06-7.12 (2H, ddd, J = 8.45, J = 6.93, J = 1.31 Hz), 7.22-7.25 (2H, d, J = 9.05 Hz), 7.31-7.37 (2H, ddd, J = 7.97, J = 6.85, J = 1.15 Hz), 7.81-7.83 (2H, d, J = 7.92 Hz), 7.80-7.84 (2H, d, J = 9.01 Hz), 8.65-8.73 (2H, d, J = 8.57 Hz) ppm; ¹³C NMR (125MHz, DMSO-d₆) δ: 175.0, 155.7, 155.3, 133.7, 133.6, 130.4, 130.0, 129.5, 129.4, 128.7, 127.5, 127.1, 124.4, 124.3, 124.1, 124.0, 117.5, 115.2,

115.1, 35.5, 32.1, 30.7, 30.9, 29.5, 26.1, 23.1, 15.3 ppm; MS EI (electron impact) m/z (relative intensity): 794 $[M]^+$ (11%), 400 (31%), 358 (21%), 316 (46%), 239 (15%), 144 (39%), 98 (5%), 32 (100%).

Synthesis of 1,1'-thiobis (1-oxa-2-oxo-undecyl) naphthalene (7)

According to the general procedure (7) was prepared from the reaction of (1) and decanoyl chloride in 93% yield and melting point of 120-121°C; IR (KBr): 3042, 2976, 2951, 1745, 1502, 1436, 1204, 1197, 1041, 753 cm^{-1} ; ^1H NMR (300 MHz, DMSO-d_6): δ : 0.89 (t, $J=7\text{ Hz}$, 3H), 1.21-1.27 (m, 12H), 1.62-1.65 (m, 2H), 2.29 (t, $J=6\text{ Hz}$, 2H), 7.05-7.10 (2H, ddd, $J=8.45$, $J=6.92$, $J=1.33\text{ Hz}$), 7.23-7.26 (2H, d, $J=9.05\text{ Hz}$), 7.30-7.36 (2H, ddd, $J=7.95$, $J=6.82$, $J=1.15\text{ Hz}$), 7.80-7.83 (2H, d, $J=7.90\text{ Hz}$), 7.85-7.88 (2H, d, $J=9.01\text{ Hz}$), 8.65-8.73 (2H, d, $J=8.56\text{ Hz}$) ppm; ^{13}C NMR (125 MHz, DMSO-d_6): δ : 174.2, 155.7, 155.6, 133.0, 133.0, 130.1, 130.7, 129.6, 129.0, 128.1, 127.3, 127.2, 124.5, 124.2, 124.0, 124.0, 117.7, 115.0, 115.0, 51.3, 34.1, 32.1, 29.9, 29.4, 29.3, 25.2, 22.9, 14.1 ppm; MS EI (electron impact) m/z (relative intensity): 626 $[M]^+$ (32%), 400 (35%), 359 (12%), 316 (13%), 300 (8%), 155 (12%), 144 (29%), 87 (41%), 74 (100%), 29 (15%).

Synthesis of 1,1'-thiobis (1-oxa-2-oxo-nonyl) naphthalene (8)

According to the general procedure (8) was prepared from the reaction of (1) and octanoyl chloride in 95% yield and melting point of 122-123°C; IR (KBr): 3045, 3010, 2971, 2964, 2933, 1746, 1500, 1457, 1301, 1253, 1208, 1179, 1098, 1043 cm^{-1} ; ^1H NMR (300 MHz, DMSO-d_6): δ : 0.88 (t, $J=7\text{ Hz}$, 3H), 1.07-1.46 (m, 8H), 1.61-1.64 (m, 2H), 2.25 (t, $J=6\text{ Hz}$, 2H), 7.09-7.16 (2H, ddd, $J=8.45$, $J=6.92$, $J=1.31\text{ Hz}$), 7.24-7.28 (2H, d, $J=9.09\text{ Hz}$), 7.31-7.34 (2H, ddd, $J=7.97$, $J=6.81$, $J=1.19\text{ Hz}$), 7.83-7.81 (2H, d, $J=7.90\text{ Hz}$), 7.80-7.84 (2H, d, $J=9.01\text{ Hz}$), 8.63-8.79 (2H, d, $J=8.58\text{ Hz}$) ppm; ^{13}C NMR (125 MHz, DMSO-d_6): δ : 173.56, 155.2, 155.2, 133.8, 133.7, 130.3, 130.0, 129.6, 129.5, 128.7, 127.5, 127.2, 124.35, 124.2, 124.0, 124.0, 117.0, 11.30, 115.0, 60.1, 34.6, 31.5, 29.6, 29.1, 25.3, 22.1, 14.1, 13.9 ppm; MS EI (electron impact) m/z (relative intensity): 630 $[M]^+$ (51%), 400 (32%), 358 (10%), 316 (14%),

300 (11%), 144 (39%), 143 (5%), 101 (34%), 87 (100%), 73 (16%).

Synthesis of 2,2'-thiobis (1-oxa-2-oxo-heptadecyl) naphthalene (9)

According to the general procedure (9) was prepared from the reaction of (2) and palmitoyl chloride in 91% yield and melting point of 120-121°C; IR (KBr): 2963, 2947, 2778, 1741 (carbonyl), 1502, 1290, 1221, 1195, 1089, 1034, 805 cm^{-1} ; ^1H NMR (500 MHz, DMSO-d_6): δ : 0.88 (t, $J=7\text{ Hz}$, 3H), 1.25-1.28 (m, 24H), 1.61 (m, 2H), 2.25-2.31 (m, 2H), 7.26 (dd, $J=5$, 15 Hz, 4H, Ar), 7.40 (t, $J=10.1\text{ Hz}$, 2H, Ar), 7.80 (d, $J=5.2\text{ Hz}$, 2H, Ar), 7.84 (d, $J=10.4\text{ Hz}$, Ar), 2H, 8.55 (d, $J=10\text{ Hz}$, 2H, Ar) ppm; ^{13}C NMR (125 MHz, DMSO-d_6): δ : 175.06 (carbonyl), 157.1, 156.4, 135.0, 130.7, 130.1, 130.9, 129.4, 127.3, 126.2, 126.5, 124.7, 124.6, 118.4, 115.5, 35.62, 32.24, 30.74, 30.43, 29.7, 26.8, 23.8, 15.1 ppm; MS EI (electron impact) m/z (relative intensity): 794 $[M]^+$ (16%), 400 (35%), 358 (24%), 316 (41%), 239 (13%), 144 (32%), 98 (4%), 32 (100%).

Synthesis of 2,2'-thiobis (1-oxa-2-oxo-undecyl) naphthalene (10)

According to the general procedure (10) was prepared from the reaction of (2) and decanoyl chloride in 89% yield and melting point of 125-126°C; IR (KBr): 2955, 2928, 2776, 1745 (carbonyl), 1435, 1291, 1227, 1089, 1034, 805 cm^{-1} ; ^1H NMR (500 MHz, DMSO-d_6): δ : 0.87 (t, $J=6\text{ Hz}$, 3H), 1.24-1.27 (m, 12H), 1.63-1.67 (m, 2H), 2.26 (t, $J=7\text{ Hz}$, 2H), 7.22 (dd, $J=5$, 15.4 Hz, 4H, Ar), 7.41 (t, $J=10.2\text{ Hz}$, 2H, Ar), 7.83 (d, $J=5.2\text{ Hz}$, 2H, Ar), 7.84 (d, $J=10.4\text{ Hz}$, Ar), 2H, 8.53 (d, $J=10.1\text{ Hz}$, 2H, Ar) ppm; ^{13}C NMR (125 MHz, DMSO-d_6): δ : 173.25 (carbonyl), 157.5, 156.6, 135.0, 130.6, 130.1, 130.4, 129.4, 127.3, 126.9, 126.7, 124.5, 124.8, 118.2, 115.6, 51.34, 34.17, 32.14, 29.9, 29.4, 29.3, 25.4, 22.7, 14.9 ppm; MS EI (electron impact) m/z (relative intensity): 626 $[M]^+$ (37%), 400 (31%), 359 (11%), 316 (18%), 300 (5%), 154 (18%), 144 (25%), 74 (100%), 29 (12%).

Synthesis of 2,2'-thiobis (1-oxa-2-oxo-nonyl) naphthalene (11)

According to the general procedure (11) was prepared from the reaction of (2) and octanoyl chloride in

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93% yield and melting point of 119-120°C; IR (KBr): 2963, 2778, 1734 (carbonyl), 1455, 1221, 1179, 1085, 1032, 804 cm⁻¹; ¹H NMR (500 MHz, DMSO-d₆): δ 0.89 (t, J = 7 Hz, 3H), 1.04-1.45 (m, 8H), 1.61-1.64 (m, 2H), 2.28 (t, J = 6 Hz, 2H), 7.21 (dd, J = 5, 15 Hz, 4H, Ar), 7.43 (t, J = 10 Hz, 2H, Ar), 7.84 (d, J = 5 Hz, 2H, Ar), 7.89 (d, J = 10 Hz, Ar), 2H, 8.56 (d, J = 10 Hz, 2H, Ar) ppm; ¹³C NMR (125 MHz, DMSO-d₆): δ 173.56 (carbonyl), 157.1, 156.7, 135.4, 130.7, 130.2, 130.0, 129.5, 127.3, 126.4, 126.1, 124.5, 124.7, 118.0, 115.1, 60.7, 34.3, 31.7, 29.9, 29.3, 25.8, 22.5, 14.6, 13.3 ppm; MS EI (electron impact) m/z (relative intensity): 630 [M]⁺ (34%), 400 (22%), 358 (12%), 317 (12%), 301 (15%), 144 (31%), 101 (32%), 87 (100%), 73 (19%).

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