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Synthesis of Naphthalenecarboxylic and Naphthalenedicarboxylic Acids from Naphthalene, Carbon Tetrachloride, and Alcohols in the Presence of Iron Catalysts

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Abstract—Alkyl naphthalenecarboxylates and dialkyl naphthalenedicarboxylates have been synthesized by reactions of naphthalene and its derivatives with alcohols and carbon tetrachloride in the presence of iron catalysts.

Keywords: naphthalene, naphthalenecarboxylic acids, esters, carbon tetrachloride, aliphatic alcohols, iron catalysts

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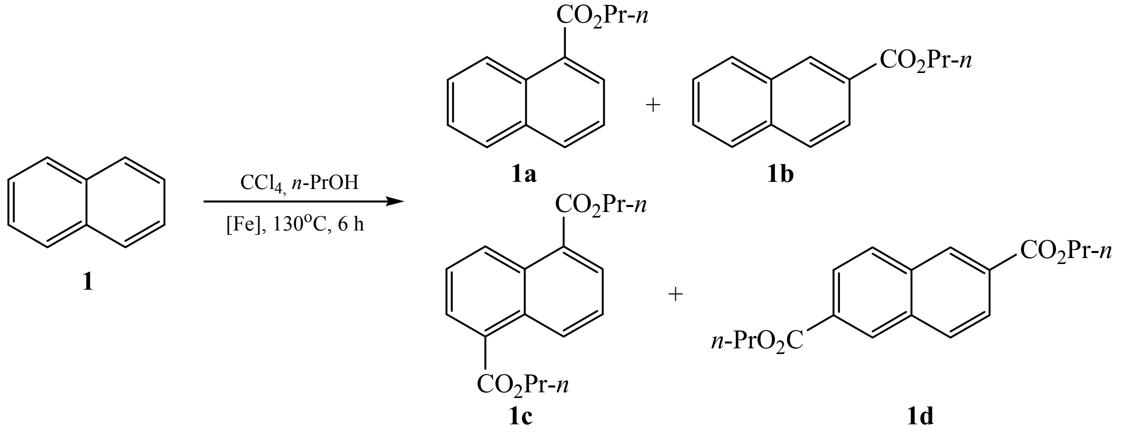
Naphthalenecarboxylic acids constitute an important class of aromatic carboxylic acids. They are widely used in the synthesis of herbicides, phytohormones, dyes, photographic materials, and polymers [1]. Naphthalenecarboxylic acid derivatives are also extensively used in medicine and cosmetology [2]. Several synthetic approaches to naphthalenecarboxylic acids are known. The classical method is based on the oxidation of alkylnaphthalenes with various oxidants such as nitric acid, hydrogen peroxide, sodium bromate, etc. [3–5]. Naphthalene-1- and 2-carboxylic acids can also be synthesized by carbonylation of halogen-substituted naphthalenes catalyzed by transition metal complexes (Co, Rh, Pd, Mo, Ni) [6, 7]. However, of particular interest is the synthesis of naphthalenecarboxylic acids directly from cheap and readily accessible naphthalene. For example, a mixture of naphthalene-1- and naphthalene-2-carboxylic acid was obtained by carboxylation of naphthalene with carbon dioxide in the presence of $Ph_3SiCl/AlBr_3$ as catalytic system [8–11].

In this article we report the synthesis of alkyl naphthalene-1- and naphthalene-2-carboxylates by reaction of naphthalene with carbon tetrachloride and alcohols catalyzed by iron compounds as well as by iron metal. The reaction conditions and concentrations of the reactants and catalyst were optimized for the reaction of naphthalene with CCl_4 and propan-1-ol in the presence of iron(III) acetylacetonate. The reaction of naphthalene (**1**) with carbon tetrachloride and propan-1-ol in the presence of $Fe(acac)_3$ at 130°C [6 h; molar ratio $Fe(acac)_3$–**1**–CCl_4–PrOH (5–15) : 100 : (500–1000) : (500–1000)] gave a mixture of propyl naphthalene-1-carboxylate (**1a**), propyl naphthalene-2-carboxylate (**1b**), dipropyl naphthalene-1,5-dicarboxylate (**1c**) and dipropyl naphthalene-2,6-dicarboxylate (**1d**) (Scheme 1).

The product yield and ratio depended on many factors. The yields of esters **1a**–**1d** at different catalyst concentrations are given in Table 1. No reaction occurred in the absence of $Fe(acac)_3$ (Table 1; run no. 1). As the catalyst concentration increased from 5 to 15 mol %, the conversion of naphthalene changed from 46 to 96%. The optimal $Fe(acac)_3$ concentration was 10%. Although the conversion of naphthalene increased by 6% when the catalyst concentration was raised to 15%, the selectivity of the reaction decreased due to formation of dipropyl naphthalene-1,5- and -2,6-dicarboxylates **1c** and **1d**.

A significant effect of carbon tetrachloride concentration on the yield of **1a**–**1d** was revealed

Scheme 1.



(Table 2). The conversion of naphthalene attained its maximum value at a **1**-to-CCl_4 ratio of 1 : 10. Despite increase of the naphthalene concentration, decrease of the amount of CCl_4 was accompanied by reduction of the conversion of naphthalene; simultaneously, dipropyl esters **1c** and **1d** were formed at a ratio of 1 : 1. The amount of propyl alcohol was also significant (Table 3). The maximum conversion of naphthalene was reached when 8 equiv of propyl alcohol was used; however, in this case dipropyl ethers **1c** and **1d** were obtained at a ratio of 2 : 1. The optimal ratio of propyl alcohol and naphthalene was 10 : 1; it favored selective formation of monoesters **1a** and **1b**.

Table 4 summarizes the results of the reactions of naphthalene with carbon tetrachloride and methanol, ethanol, and butan-1-ol in the presence of $Fe(acac)_3$ with formation of the corresponding naphthalene-1- and -2-carboxylic esters **1e**–**1j**. The product ratio depended on the alcohol nature. In the reaction with methanol, the major isomer was methyl naphthalene-2-carboxylate (**1e**).

In order to improve the reaction selectivity we examined the effect of some activating ligands. Such classical activating ligands as triphenylphosphine, triphenyl phosphite, triethylamine, pyridine, 1,10-phenanthroline, *N*,*N*-dimethylaniline, and acetonitrile insignificantly affected the chemoselectivity, reaction rate, and product yield.

We then tried naphthalene-1- and -2-carboxylic acids **2** and **3** as substrates in the reaction with carbon tetrachloride and proan-1-ol in the presence of $Fe(acac)_3$. The major products were the corresponding monoeesters **1a** and **1b**, whereas the yield of diesters (isomer mixtures) was 26% (Scheme 2).

1-Methyl- and 2-methylnaphthalenes **4** and **5**, as well as 1,3-, 1,6-, 1,7-, 2,7-, and 1,8-dimethylnaphthalenes **6**–**10**, reacted with carbon tetrachloride and

Table 1. Yields of propyl naphthalenecarboxylates **1a** and **1b** and dipropyl naphthalene dicarboxylates **1c** and **1g** in the reaction of naphthalene with carbon tetrachloride and propyl alcohol in the presence of $Fe(acac)_3$ at different concentrations[a]

Run no.	Molar ratio $Fe(acac)_3$–naphthalene–CCl_4–PrOH	Conversion of naphthalene, %	Yield, %			
			1a	**1b**	**1c**	**1d**
1	0:100:1000:1000	0	–	–	–	–
2	5:100:1000:1000	46	30	16	–	–
3	10:100:1000:1000	90	55	35	–	–
4	15:100:1000:1000	96	45	31	12	8

[a] 130°C, 6 h.

Table 2. Yields of esters **1a**–**1d** at different concentrations of carbon tetrachloride[a]

Run no.	Molar ratio $Fe(acac)_3$–naphthalene–CCl_4–PrOH	Conversion of naphthalene, %	Yield, %			
			1a	**1b**	**1c**	**1d**
1	10:100:0:1000	0	–	–	–	–
2	10:100:1000:1000	90	55	35	–	–
3	10:100:800:1000	84	61	15	4	4
4	10:100:500:1000	67	49	14	2	2

[a] 130°C, 6 h.

Table 3. Yields of esters **1a**–**1d** depending on the amount of propyl alcohol[a]

Run no.	Molar ratio Fe(acac)$_3$–naphthalene–CCl$_4$–PrOH	Conversion of naphthalene, %	Yield, %			
			1a	**1b**	**1c**	**1d**
1	10:100:1000:0	0	–	–	–	–
2	10:100:1000:1000	90	55	35	–	–
3	10:100:1000:800	96	62	13	16	7
4	10:100:1000:500	89	61	15	4	2

[a] 130°C, 6 h.

Table 4. Yields of esters **1a**, **1b**, and **1e**–**1j** in the reaction of naphthalene derivatives with carbon tetrachloride and different alcohols in the presence of Fe(acac)$_3$[a]

Run no.	Alcohol	Conversion of naphthalene, %	Yield, %	
			1-isomer	2-isomer
1	MeOH	42	15 (**1e**)	27 (**1f**)
2	EtOH	35	27 (**1g**)	8 (**1h**)
3	PrOH	90	55 (**1a**)	35 (**1b**)
4	1-BuOH	81	63 (**1i**)	18 (**1j**)

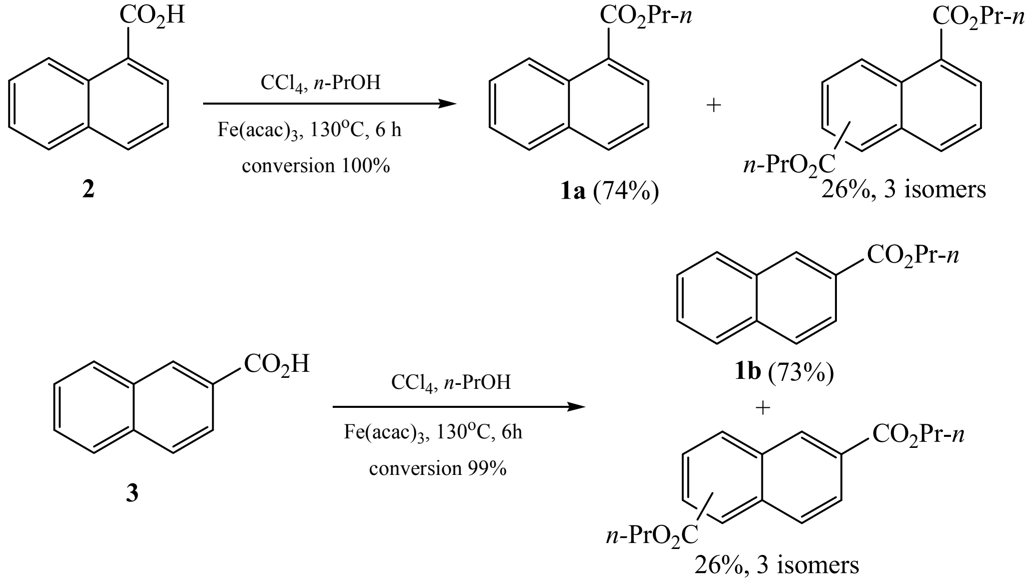
[a] 130°C, 6 h, Fe(acac)$_3$–naphthalene–CCl$_4$–alcohol 10:100:1000:1000.

popan-1-ol in the presence of Fe(acac)$_3$ to give the corresponding mono- and diesters. The reactions with **4** and **5** were characterized by high conversion, and the major products were monoesters **4a** and **4b** and **5a** and **5b**, respectively, as mixtures of isomers which were difficult to separate. Apart from **4a**, **4b** and **5a**, **5b**, the reaction mixtures contained small amounts (6–9%) of dipropyl esters **4c**, **4d** and **5c**, **4d** (Scheme 3).

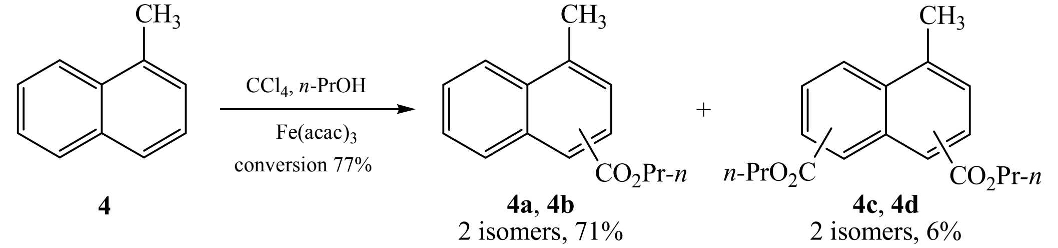
Under analogous conditions, dimethylnaphthalenes **6**–**9** were converted into the corresponding diemthylnaphthalenecarboxylates **6a**–**6d** and **7a**–**9c** as mixtures of isomers differing by the position of the ester group; the conversion of initial naphthalene **6**–**9** was 79, 74, 73, and 82%, respectively (Scheme 4). An exception was 1,8-dimethylnaphthalene (**10**) which was quantitatively converted to propyl 1,8-dimethylnaphthalene-4-carboxylate (**10a**) (Scheme 5).

The structure of ester **10a** was proved by one- (^{1}H, ^{13}C) and two-dimensional (COSY, HSQC, HMBC) NMR data. The ^{1}H NMR spectrum of **10a** displayed one triplet and four doublets in the aromatic region, which is consistent with structure **10a** with the ester group attached to C^4. Two three-proton singlets at δ 2.96 and 2.95 ppm were assigned to protons of the methyl groups on C^1 and C^8, and protons of the propyl group resonated as upfield multiplet and triplet signals. As follows from the COSY spectrum, the 6-H proton (δ 7.45 ppm, t, ^3J = 7.2 Hz) is coupled with 5-H (δ 8.77 ppm, ^3J = 8.4 Hz) and 7-H (7.35 ppm, ^3J = 8.0 Hz). The two doublets of 3-H (δ 7.94 ppm, ^3J = 7.2 Hz) and 2-H (δ 7.29 ppm, ^3J = 8.0 Hz) showed correlation with each other. The carbon signals in the ^{13}C NMR spectrum of **10a** were assigned on the basis of the HSQC and HMBC spectra.

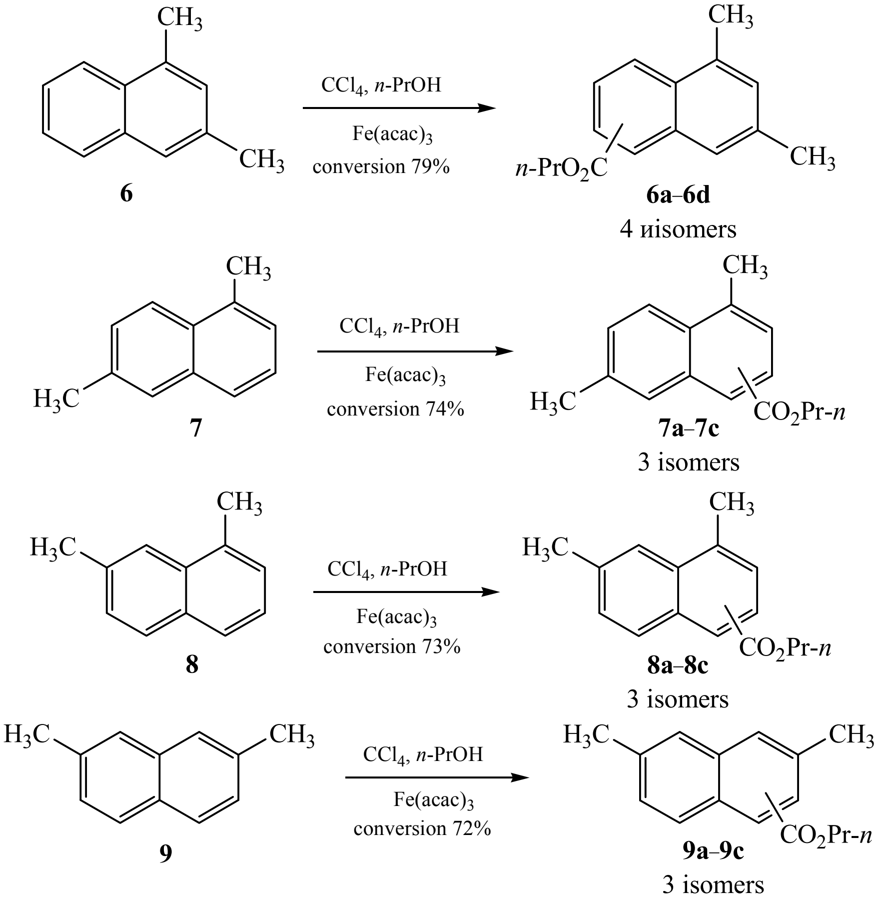
Scheme 2.



Scheme 3.

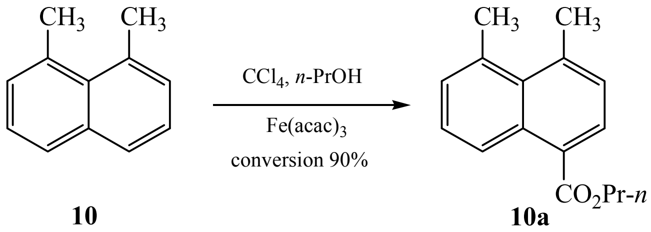


Scheme 4.

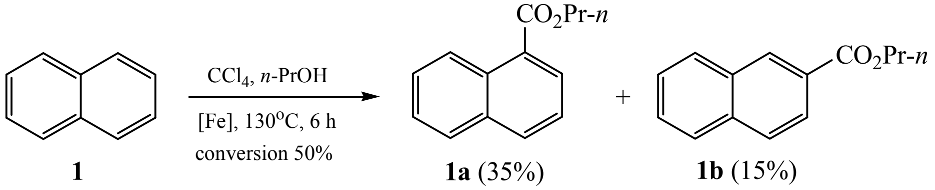


Apart from Fe(acac)$_3$, other iron compounds [FeCl$_3$, FeCl$_3$·6H$_2$O, FeBr$_2$, Fe, Fe$_2$(CO)$_9$] were tried as catalysts. However, the catalytic activity of all these compounds was lower than that of Fe(acac)$_3$. The behavior of iron metal was interesting. It showed an appreciable catalytic activity in the presence of acetylacetone [Fe–Hacac ratio 1 : (1–10)]. The reaction of naphthalene with carbon tetrachloride and propan-1-ol at 130°C (6 h) in the presence of iron metal (preliminarily activated with 5% HCl) and acetylacetone (molar ratio Fe–Hacac–**1**–CCl$_4$–PrOH 5 : 5 : 100 : 300 : 300) afforded a mixture of propyl

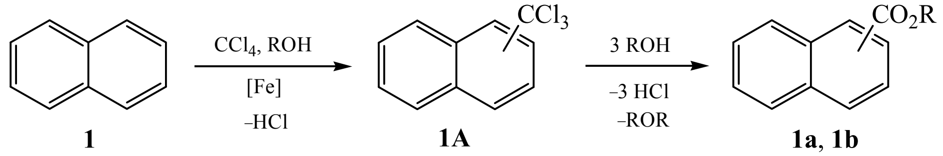
Scheme 5.



Scheme 6.



Scheme 7.



naphthalene-1- and -2-carboxylates **1a** and **2b** in an overall yield of 50% (Scheme 6). Iron metal gradually dissolved during the process; presumably, the catalytically active form was not Fe^0 but iron(III) acetylacetonate.

In all cases, the reactions were accompanied by formation of hydrogen chloride and the corresponding ethers as by-products. Taking these data into account, we propose the following mechanism of the formation of ester group. The first step is alkylation of naphthalene (**1**) with carbon tetrachloride, and the resulting trichloromethyl derivative **1A** undergoes alcoholysis to give ester **1a** or **1b** (Scheme 7).

The structure of **1a** and **1b** was confirmed by ^1H and ^{13}C NMR spectra. Ester **1a** displayed in the ^1H NMR spectrum five doublets corresponding to 2-H (δ 8.98 ppm, J = 8.8 Hz), 3-H (δ 7.65 ppm, J = 7.2 Hz), 4-H (δ 8.23 ppm, J = 7.2 Hz), 5-H (δ 8.03 ppm, J = 8.4 Hz), and 8-H (δ 7.90 ppm, J = 8 Hz) and one multiplet signal due to 6-H and 7-H (δ 7.53 ppm). In the upfield region we observed signals of the propyl group at δ 4.42 (J = 6.8 Hz, OCH_2), 1.89 ($OCH_2C\underline{H}_2$), and 1.12 ppm (J = 7.6 Hz, CH_3). The ^1H NMR spectrum of **1b** contained four doublets at δ 7.68 (J = 8 Hz, 3-H), 8.20 (J = 7 Hz, 4-H), 7.80 (J = 8 Hz, 5-H), and 8.04 ppm (J = 8 Hz, 8-H), one multiplet at δ 7.50 ppm (6-H, 7-H), one singlet at δ 8.60 ppm (1-H), and upfield signals typical of propoxy group.

Thus, we have demonstrated the possibility of synthesizing alkyl naphthalenecarboxylates and dialkyl naphthalenedicarboxylates by multicomponent reaction of naphthalene with carbon tetrachloride and alcohols catalyzed by iron compounds.

EXPERIMENTAL

The ^1H and ^{13}C NMR spectra were recorded on a Bruker Avance-400 spectrometer at 400.1 and 100.6 MHz, respectively, using $CDCl_3$ as solvent; the chemical shifts are given relative to tetramethylsilane. The mass spectra were obtained on a Shimadzu GCMS -QP2010Plus (SPB-5 capillary column, 30 m × 0.25 mm; carrier gas helium; oven temperature programming from 40 to 300°C at a rate of 8 deg/min, injector temperature 280°C, ion source temperature 200°C; electron impact, 70 eV). The reaction mixtures were analyzed by GLC on Shimadzu GC-9A and GC-2014 instruments using a 2-m × 3-mm column packed with 5% SE-30 on Chromaton N-AW-HMDS; oben temperature programming from 50 to 270°C at a rate of 8 deg/min; carrier gas helium, flow rate 47 mL/min.

GLC analyses were also done on a Shimadzu GCMS-QP2010 Ultra instrument (Supel-Q PLOT capillary column, 30 m × 0.53 mm; oven temperature programming from 37 to 250°C at a rate of 10 deg/min; carrier gas helium, flow rate 3 mL/min; ion source temperature 200°C; electron impact, 70 eV). All reagents and solvent were of analytical grade.

Alkyl naphthalenecarboxylates and dialkyl naphthalenedicarboxylates (*general procedure*). The reactions were carried out in a 10-mL glass ampule placed in a 17-ml stainless steel high pressure reactor with controlled heating. The ampule was charged under argon with 10 mmol of Fe(acac)$_3$, 0.1 mol of the corresponding naphthalene derivative, 1 mol of carbon tetrachloride, and 0.8–1.0 mol of the corresponding alcohol. The ampule was sealed and placed in the high-pressure reactor which was heated at 130°C for 6 h with continuous stirring. When the reaction was complete, the reactor was cooled to room temperature, the ampule was opened, and the reaction mixture was neutralized and filtered. The solvent was distilled off, and the residue was distilled under reduced pressure or recrystallized from ethanol.

Propyl naphthalene-1-carboxylate (1a). Yield 74%, bp 105–107°C (0.2 mm). ^{1}H NMR spectrum, δ, ppm: 1.12 t (3H, $CH_2C\underline{H}_3$, J = 7.6 Hz), 1.89 m (2H, $C\underline{H}_2CH_3$), 4.42 t (2H, OCH_2, J = 6.8 Hz), 8.98 d (1H, 2-H, J = 8.8 Hz), 7.65 d (1H, 3-H, J = 7.2 Hz), 8.23 d (1H, 4-H, J = 7.2 Hz), 8.03 d (1H, 5-H, J = 8.4 Hz), 7.53 m (2H, 6-H, 7-H), 7.90 d (1H, 8-H, J = 8 Hz). ^{13}C NMR spectrum, δ_C, ppm: 10.48 (CH_3), 21.87 ($\underline{C}H_2CH_3$), 66.68 (OCH_2), 125.24 (C^3), 126.53 (C^6), 126.62 (C^7), 127.21 (C^8), 128.48 (C^5), 130.06 (C^2), 130.25 (C^1), 132.48 (C^{4a}), 133.22 (C^4), 133.91 (C^{8a}), 168.93 (C=O). Mass spectrum, m/z (I_{rel}, %): 214 (72) $[M]^+$, 172 (100), 155 (95), 127 (56), 115 (5).

Propyl naphthalene-2-carboxylate (1b). Yield 73%, bp 107–109°C (0.2 mm). ^{1}H NMR spectrum, δ, ppm: 1.08 t (3H, $CH_2C\underline{H}_3$, J = 7.2 Hz), 2.03 m (2H, $C\underline{H}_2CH_3$), 4.30 t (2H, OCH_2, J = 6.8 Hz), 8.60 s (1H, 1-H), 7.68 d (1H, 3-H, J = 8.0 Hz), 8.20 d (1H, 4-H, J = 7.0 Hz), 7.80 d (1H, 5-H, J = 8.0 Hz), 7.50 m (2H, 6-H, 7-H), 8.04 d (1H, 8-H, J = 8 Hz). ^{13}C NMR spectrum, δ_C, ppm: 10.50 (CH_3), 22.15 ($\mathbf{C}H_2CH_3$), 66.61 (OCH_2), 125.26 (C^6), 125.80 (C^3), 127.22 (C^4), 127.51 (C^{8a}), 127.75 (C^7), 128.51 (C^5), 129.33 (C^1), 131.37 (C^8), 132.53 (C^{4a}), 135.49 (C^2), 167.63 (C=O). Mass spectrum, m/z (I_{rel}, %): 214 (48) $[M]^+$, 207 (29), 172 (94), 163 (13), 155 (100), 127 (59), 115 (7).

Dipropyl naphthalene-1,5-dicarboxylate (1c). Yield 16%, bp 160–162°C (0.2 mm). ^{1}H NMR spectrum, δ, ppm: 1.08 t (3H, $CH_2C\underline{H}_3$, J = 6.8 Hz), 1.65 m (2H, $C\underline{H}_2CH_3$), 4.23 t (2H, OCH_2, J = 7.2 Hz), 8.97 d (2H, 2-H, 6-H, J = 8.8 Hz), 7.68 m (1H, 3-H, 7-H), 8.25 d (2H, 4-H, 8-H, J = 7.2 Hz). ^{13}C NMR spectrum, δ_C, ppm: 10.58 (CH_3), 22.10 ($\underline{C}H_2CH_3$), 67.03 (OCH_2), 125.83 (C^4, C^8), 126.21 (C^3, C^7), 132.20 (C^2, C^6), 133.23 (C^{4a}, C^{8a}), 140.26 (C^1, C^5), 167.62 (C=O). Mass spectrum, m/z (I_{rel}, %): 300 (66) $[M]^+$, 258 (29), 241 (87), 221 (20), 216 (100), 199 (63), 150 (15), 126 (20).

Dipropyl naphthalene-2,6-dicarboxylate (1d). Yield 7%, bp 163–165°C (0.2 mm). ^{1}H NMR spectrum, δ, ppm: 1.13 t (3H, $CH_2C\underline{H}_3$, J = 6.8 Hz), 1.75 m (2H, $C\underline{H}_2CH_3$), 4.38 t (2H, OCH_2, J = 4.0 Hz), 8.76 s (1H, 1-H, 5-H), 8.24 d (2H, 3-H, 7-H, J = 8.0 Hz), 8.17 d (2H, 4-H, 8-H, J = 7.2 Hz). ^{13}C NMR spectrum, δ_C, ppm: 10.69 (CH_3), 22.19 ($\underline{C}H_2CH_3$), 66.90 (OCH_2), 127.65 (C^3, C^7), 130.51 (C^4, C^8), 131.72 (C^1, C^5), 134.06 (C^{4a}, C^{8a}), 140.28 (C^2, C^6), 167.64 (C=O). Mass spectrum, m/z (I_{rel}, %): 300 (45) $[M]^+$, 281 (23), 258 (25), 241 (84), 216 (100), 207 (51), 199 (50), 171 (30), 126 (19), 115 (27).

Methyl naphthalene-1-carboxylate (1e). Yield 15%, bp 155–157°C (10 mm); published data [12]: bp 165°C (17 mm). ^{1}H NMR spectrum, δ, ppm: 3.99 s (3H, CH_3), 8.7 d (1H, 2-H, J = 9.0 Hz), 7.69 d.d (1H, 3-H, J = 7.0, 9.0 Hz), 8.23 d (1H, 4-H, J = 7.0 Hz), 8.00 d (1H, 5-H, J = 8.0 Hz), 7.5 m (1H, 6-H), 7.58 m (1H, 7-H), 7.9 d (1H, 8-H, J = 8.0 Hz). ^{13}C NMR spectrum, δ_C, ppm: 52.19 (CH_3), 126.27 (C^6), 125.34 (C^3), 126.39 (C^7), 127.19 (C^8), 128.36 (C^5), 128.77 (C^1), 130.44 (C^2), 132.59 (C^{8a}), 132.69 (C^4), 133.47 (C^{4a}), 167.32 (C=O). Mass spectrum, m/z (I_{rel}, %): 186.17 (69) $[M]^+$, 155 (100), 127 (81), 126 (15), 77 (10), 75 (5).

Methyl naphthalene-2-carboxylate (1f). Yield 27%, mp 76–78°C; published data [12]: mp 77°C. ^{1}H NMR spectrum, δ, ppm: 3.93 s (3H, CH_3), 7.7 d (1H, 3-H, J = 7.0 Hz), 8.19 d (1H, 4-H, J = 7.0 Hz), 7.83 d (1H, 5-H, J = 8.0 Hz), 7.48 m (1H, 6-H), 7.4 m (1H, 7-H), 7.8 d (1H, 8-H, J = 8.0 Hz), 8.6 s (1H, 1-H). ^{13}C NMR spectrum, δ_C, ppm: 52.28 (CH_3), 125.34 (C^6), 125.43 (C^3), 126.80 (C^{4a}), 127.19 (C^4), 127.88 (C^7), 128.68 (C^5), 129.40 (C^1), 131.16 (C^8), 132.59 (C^{8a}), 135.57 (C^2), 167.90 (C=O). Mass spectrum, m/z (I_{rel}, %): 186.17 (69) $[M]^+$, 155 (100), 127 (80), 126 (15), 40 (16).

Ethyl naphthalene-1-carboxylate (1g). Yield 27%, bp 165–166°C (10 mm); published data [12]: bp

310°C. ^{1}H NMR spectrum, δ, ppm: 1.40 t (3H, CH_3, J = 7.6 Hz), 4.4 q (2H, OCH_2, J = 3.7 Hz), 8.97 d (1H, 2-H, J = 9.0 Hz), 7.63 d.d (1H, 3-H, J = 7.0, 9.0 Hz), 8.21 d (1H, 4-H, J = 7.0 Hz), 8.00 d (1H, 5-H, J = 8.0 Hz), 7.5 m (1H, 6-H), 7.5 m (1H, 7-H), 7.87 d (1H, 8-H, J = 8.0 Hz). ^{13}C NMR spectrum, δ_C, ppm: 14.40 (CH_3), 61.04 (CH_2O), 125.27 (C^3), 126.17 (C^6), 126.60 (C^7), 127.44 (C^8), 128.16 (C^5). 129.33 (C^1), 130.11 (C^2), 131.37 (C^{8a}), 133.23 (C^4), 133.85 (C^{4a}), 167.59 (C=O). Mass spectrum, m/z (I_{rel}, %): 200.10 (71) $[M]^+$, 172 (15), 156 (17), 155 (100), 127 (68), 126 (13).

Ethyl naphthalene-2-carboxylate (1h). Yield 8%, bp 165–166°C (10 mm); published data [12]: bp 178°C. ^{1}H NMR spectrum, δ, ppm: 1.37 t (3H, CH_3, J = 7.6 Hz), 4.4 q (2H, OCH_2, J = 3.8 Hz), 7.63 d (1H, 3-H, J = 7.0 Hz), 8.2 d (1H, 4-H, J = 7.0 Hz), 7.8 d (1H, 5-H, J = 8.0 Hz), 7.5 m (1H, 6-H), 7.4 m (1H, 7-H), 7.8 m (1H, 8-H), 8.5 s (1H, 1-H). ^{13}C NMR spectrum, δ_C, ppm: 14.40 (CH_3), 61.10 ($CH_3\underline{C}H_2$), 124.50 (C^6), 125.27 (C^3), 126.60 (C^{4a}), 127.44 (C^4), 127.76 (C^7), 128.16 (C^5), 128.55 (C^1), 130.96 (C^8), 131.37 (C^{8a}), 133.85 (C^2), 167.59 (C=O). Mass spectrum, m/z (I_{rel}, %): 200 (65) $[M]^+$, 186 (46), 156 (16), 155 (100), 126 (14), 44 (24).

Propyl 1,8-dimethylnaphthalene-4-carboxylate (10a). Yield 90%, bp 135–138°C (0.2 mm). ^{1}H NMR spectrum, δ, ppm: 1.11 t (3H, $CH_2C\underline{H}_3$, J = 7.6 Hz), 1.88 m (2H, $C\underline{H}_2CH_3$), 2.96 s (3H, 1-CH_3), 2.95 s (3H, 8-CH_3), 4.40 t (2H, OCH_2, J = 6.4 Hz), 7.29 d (1H, 2-H, J = 8.0 Hz), 7.35 d (1H, 7-H, J = 8.0 Hz), 7.45 t (2H, 6-H, J= 7.2 Hz), 7.94 d (1H, 3-H, J = 7.2 Hz), 8.77 d (1H, 5-H, J = 8.4 Hz). ^{13}C NMR spectrum, δ_C, ppm: 10.70 (CH_3), 21.20 ($\underline{C}H_2CH_3$), 26.50 (1-CH_3), 26.75 (8-CH_3), 66.80 (OCH_2), 124.86 (C^5), 126.53 (C^6), 128.27 (C^2), 128.72 (C^3), 130.00 (C^7), 133.14 (C^{8a}), 133,52 (C^{4a}), 133.96 (C^1), 135.71 (C^8), 140.86 (C^4), 168.10 (C=O). Mass spectrum, m/z (I_{rel}, %): 242 (97) $[M]^+$, 213 (7), 200 (77), 183 (92), 155 (48), 128 (21), 115 (13).

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