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Nickel(II)-catalyzed carbon–carbon bond formation reaction of functionalized organozinc reagents with aromatic aldehydes

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ABSTRACT

In the presence of a silylating reagent and catalytic amount of Ni(acac)$_2$, organozinc halides reacted with aromatic aldehydes to give the corresponding dialkylation products in good to excellent yields under mild conditions.

1. Introduction

Transition metal catalyzed carbon–carbon bond formation is one of the most useful reactions in synthetic organic chemistry, particularly in the synthesis of complex natural products with biological activity. Many methods[1] have been described for the reactions of carbon–carbon bond formation. The reaction of organozinc reagents with carbonyl compounds is also an important carbon–carbon bond formation reaction in organic chemistry.[2,3] Organozinc reagents are very useful and versatile organometallic reagents in organic chemistry because of their high functional group compatibility[4] and excellent reactivity in the presence of an appropriate transition metal catalyst.[5]

In 1978, Mukaiyama first reported that Et_2Zn could add smoothly to benzaldehyde in the presence of aminoalcochols.[6] Since then, much attention has been devoted to the reaction,[7] especially the enantioselective addition reaction[8] of dialkylzinc compounds with aldehydes and ketones. However, dialkylzinc compounds are relatively stable and poorly reactive organometallic compounds. Furthermore, in most cases only one of the two alkyl groups is transferred, wasting 50% of the precious reagent.

Alkylzinc halides, RZnX, which carry only one alkyl side chain, offer alternatives. However, these species are less reactive and often necessitate the use of additional catalyst. In 1988, Knochel[9] showed that functional organozinc iodides RZnI could be transmetallated into organozinccopper reagents RCu(CN)ZnI with THF soluble copper salt $CuCN\cdot 2LiCl$, and then coupled with acyl chlorides or conducted 1,4-additon reaction with enones. This report greatly broadened the scope of organozinc reagents in organic synthesis. Now these reagents have been successfully used in coupling reactions with organic halides, 1,2-addition reactions with aldehyde or ketones, and 1,4-addition reactions with β-unsaturated carbonyl compounds or nitro olefins.[2,7,10] But in these cases, stoichiometric $CuCN\cdot 2LiCl$ must be used in most of these reactions. Recently, Nickel-catalyzed coupling reaction of organic halides with organozinc reagents has been reported.[11,12] Addition of alkylzinc to carbonyl compounds to synthesize optically active secondary alcohols have also been reported.[8,13,14] We also reported[15] the transition metal catalyzed carbon–carbon bond formation reaction of functionalized organozinc reagents in the presence of a silylating reagent. However, we have found very few literatures on the dialkylation of aromatic aldehydes.[16,17] In our earlier communication,[18] we have showed Ni(II)-catalyzed dialkylation of aromatic aldehydes and organozinc reagents proceeded efficiently in the presence of a silylation reagent.

2. Results and discussion

We now report a novel carbon–carbon bond formation reaction via Ni(acac)$_2$-catalyzed dialkylation of aromatic aldehydes and organozinc reagents. Nickel(II)-catalyzed dialkylation of benzaldehyde with n-$C_8H_{17}ZnI$, affording 1-octyl-1-phenylnonane, was

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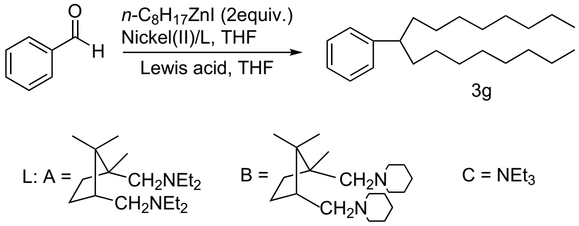
chosen as a model reaction to optimize the reaction conditions. The results are summarized in Table 1.

Among several catalysts screened, the best yield was obtained when [Ni(acac)$_2$] was employed (Table 1, entry 3). In the presence of [PdCl$_2$(PPh$_3$)$_2$], [CoCl$_2$(PPh$_3$)$_2$], [CoCl(PPh$_3$)$_3$], [NiCl$_2$(PPh$_3$)$_2$], [Ni(OAc)$_2$] or NiCl$_2$, the reaction was unsuccessful (Table 1, entries 14–19). The amount of [Ni(acac)$_2$] affected the yield and the use of 10 mol % of the catalyst gave the best results (entries 1–3). When A, B or C was employed as a ligand in the presence of Me$_3$SiCl and 10 mol % of [Ni(acac)$_2$], the yields of the corresponding 1-octyl-1-phenylnonane were 78, 76, and 82%, respectively (Table 1, entries 1–3). We have also investigated the effect of the reaction time and the temperature on this reaction. It was found that the high yield of 1-octyl-1-phenylnonane was obtained from −18 °C to room temperature for 12 h (Table 1, entries 1–3, 12, and 13). In the absence of [Ni(acac)$_2$] or Me$_3$SiCl, no product was formed. For example, no product was isolated after the reaction of benzaldehyde with n-C_8H_{17}ZnI without [Ni(acac)$_2$] and chlorotrimethylsilane (Table 1, entries 5 and 11).

In order to define the scope and the limitation of this new dialkylation reaction, the reaction of various aldehydes and organozinc reagents were examined as listed in Table 2.

As shown in Table 2, the reaction of substituted aromatic aldehydes with RZnI in the presence of the silylating reagent, a tertiary amine, and catalytic amount of Ni(acac)$_2$ from −18 °C to room temperature for 12 h gave the corresponding 1-alkyl-1-arylalkanes **3** in good to excellent yields (72–85%, Table 2, entries 1–12). Methoxyl substituent at *meta* or *para* position of the aromatic ring did not exert strong influence upon the dialkylation reaction, while the *ortho* substituted compound gave the dialkylated product in 50% yield[18] (Table 2, entries 2, 4, 5, 6, 9, 10, 12, 13, 16, and 17). Amazingly,

Table 1
Effects of the type and the amount of catalysts, the type of Lewis acid, and reaction conditions on the formation of **3g**[a]



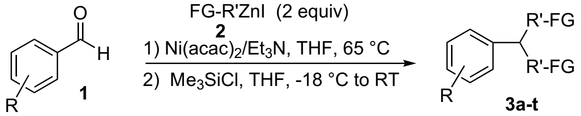
Entry	Catalyst (mol %)	L	Lewis acid	Conditions	Yield[b] (%)
1	Ni(acac)$_2$ (10)	A	(CH$_3$)$_3$SiCl	−18 °C→rt, 12 h	78
2	Ni(acac)$_2$ (10)	B	(CH$_3$)$_3$SiCl	−18 °C→rt, 12 h	76
3	Ni(acac)$_2$ (10)	C	(CH$_3$)$_3$SiCl	−18 °C→rt, 12 h	82
4	Ni(acac)$_2$ (5)	C	(CH$_3$)$_3$SiCl	−18 °C→rt, 12 h	31
5	—	C	(CH$_3$)$_3$SiCl	−18 °C→rt, 12 h	0
6	Ni(acac)$_2$ (10)	C	AlCl$_3$	−18 °C→rt, 12 h	0
7	Ni(acac)$_2$ (10)	C	BF$_3$·OEt$_2$	−18 °C→rt, 12 h	0
8	Ni(acac)$_2$ (10)	C	(CH$_3$)$_3$SiCl	−18 °C→rt, 12 h	60
9	Ni(acac)$_2$ (0.6)	C	(CH$_3$)$_3$SiCl	−18 °C→rt, 12 h	42
10	Ni(acac)$_2$ (10)	—	(CH$_3$)$_3$SiCl	−18 °C→rt, 12 h	18
11	Ni(acac)$_2$ (10)	C	—	−18 °C→rt, 12 h	0
12	Ni(acac)$_2$ (10)	C	(CH$_3$)$_3$SiCl	0 °C, 12 h	28
13	Ni(acac)$_2$(10)	C	(CH$_3$)$_3$SiCl	rt, 12 h	24
14	[PdCl$_2$(PPh$_3$)$_2$] (2)		(CH$_3$)$_3$SiCl	−18 °C→rt, 12 h	0[c]
15	[NiCl$_2$(PPh$_3$)$_2$] (10)		(CH$_3$)$_3$SiCl	−18 °C→rt, 12 h	0[c]
16	[CoCl$_2$(PPh$_3$)$_2$] (10)		(CH$_3$)$_3$SiCl	−18 °C→rt, 12 h	0[c]
17	[CoCl(PPh$_3$)$_3$] (10)		(CH$_3$)$_3$SiCl	−18 °C→rt, 12 h	0[c]
18	Ni(OAc)$_2$(10)	C	(CH$_3$)$_3$SiCl	−18 °C→rt, 12 h	0
19	NiCl$_2$(10)	C	(CH$_3$)$_3$SiCl	−18 °C→rt, 12 h	0

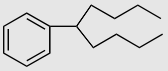
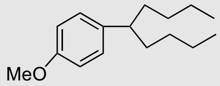
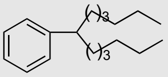
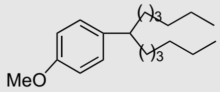
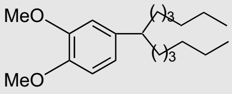
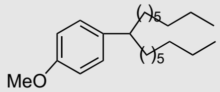
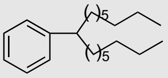
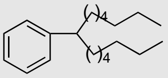
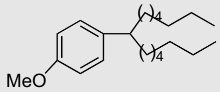
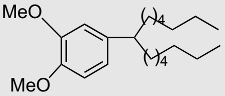
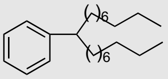
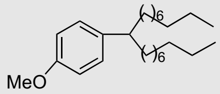
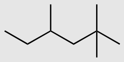
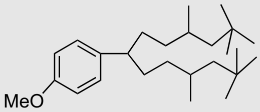
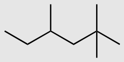
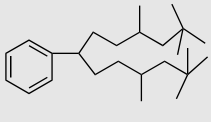
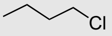
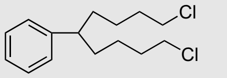
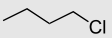
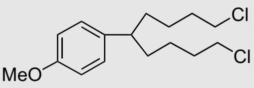
[a] All reactions were carried out using 12 mmol of benzaldehyde with 25 mmol of n-C_8H_{17}ZnI, 24 mmol of Lewis acid, and 4.8 mmol of L (2.4 mmol for A or B, 4.8 mmol for C) in 20 mL of THF.

[b] Isolated yield.

[c] The major product was *E*-alkene.

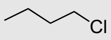
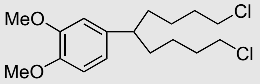
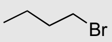
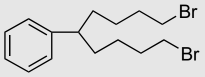
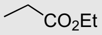
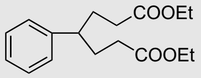
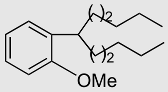
Table 2
Nickel(II)-catalyzed dialkylation reaction of aromatic aldehydes with functional organozinc reagents[a]



Entry	R	FG-R′	Product[b]		Yield[c] (%)
1	H	n-C_4H_9		**3a**	74
2	4-MeO	n-C_4H_9		**3b**	72
3	H	n-C_6H_{13}		**3c**	82
4	4-MeO	n-C_6H_{13}		**3d**	79
5	3,4-(MeO)$_2$	n-C_6H_{13}		**3e**	73
6	4-MeO	n-C_8H_{17}		**3f**	85
7	H	n-C_8H_{17}		**3g**	83
8	H	n-C_7H_{15}		**3h**	81
9	4-MeO	n-C_7H_{15}		**3i**	77
10	3,4-(MeO)$_2$	n-C_7H_{15}		**3j**	69
11	H	n-C_9H_{19}		**3k**	82
12	4-MeO	n-C_9H_{19}		**3l**	78
13	4-MeO			**3m**	74
14	H			**3n**	79
15	H			**3o**	77
16	4-MeO			**3p**	78

(*continued on next page*)

Table 2 (*continued*)

Entry	R	FG-R′	Product[b]		Yield[c] (%)
17	3,4-(MeO)$_2$			**3q**	67
18	H			**3r**	78
19	H			**3s**	63
20	2-MeO	n-C_5H_{11}		**3t**	50

[a] All reaction were conducted on the scale of 12 mmol of aldehydes with 25 mmol of organozinc reagents, 1.2 mmol of Ni(acac)$_2$, 24 mmol of Me_3SiCl, and 4.8 mmol of Et_3N in 20 mL of THF.

[b] All products were characterized by IR, ^{1}H NMR, ^{13}C NMR, and MS.

[c] Isolated yield.

we also found that these reactions tolerated various RZnI. The structure of alkyl group of RZnI has little effects on the yields (Table 2, entries 1, 3, 7, 8, 11, and 14). Under the same conditions, functionalized alkylzinc iodides gave the products in good yields (Table 2, entries 15–19). Functional groups such as chloro, bromo, and ester groups were introduced to the corresponding dialkylation products (Table 2, entries 17–19).

3. Conclusion

In conclusion, a novel, general, and efficient method of the [Ni(acac)$_2$]-catalyzed dialkylation of aromatic aldehydes with functionalized organozinc halides in the presence of a silylation reagent has been developed. The generality and synthetic usefulness of this method has been demonstrated in the efficient synthesis of the polyfunctionalized 1-alkyl-1-arylalkanes. This reaction uses more easily available organozinc halides rather than dialkylzinc reagents, gives higher yields of the products, tolerates multiple functional groups, and provides a new method to form dialkylated products from aldehydes.

4. Experimental

4.1. General remarks

^{1}H NMR spectra were recorded on a Brucker AM 400 MHz and Brucker AC-E 200 MHz spectrometers in $CDCl_3$ with TMS as an internal standard. ^{13}C NMR spectra were obtained on a Brucker AM-400 operating at 100 MHz or a Brucker AC-E 200 operating at 50 MHz. IR spectra were recorded on an Alpha Centauri FT-IR spectrometer. Mass spectra were recorded on a HP 5988A and GC/MS/DS instruments. Elemental analyses were carried out on Carlo Erba-1106 instruments. Purification of products was performed via flash chromatography with 200–400 mesh silica gel (10:1 petroleum/diethyl ether). All substrates and reagents were obtained commercially except RZnX and Ni(acac)$_2$, which were prepared by standard procedures. THF was distilled from sodium/benzophenone.

4.2. Typical experimental procedure for the synthesis of 3a–t

A mixture of zinc dust (1.7 g, 26 mmol), 1,2-dibromoethane (0.2 mL), and THF (2 mL) in a dry three-neck flask under argon atmosphere was heated to 65 °C for 2–3 min and then cooled to room temperature. Then chlorotrimethylsilane (0.2 mL) was added and the mixture was stirred at room temperature for 20 min. A solution of RI (25 mmol) in THF (10 mL) was added dropwise and the mixture was stirred for 12 h at 35–40 °C. The resulting solution of RZnI in THF was ready to use. In another three-neck flask, Ni(acac)$_2$ (1.2 mmol), Et_3N (0.48 g, 4.8 mmol), and THF (10 mL) were added and heated at 60 °C under argon for 10 min. After cooling to room temperature, the solution of RZnI in THF obtained above was added and the resulting mixture was cooled to −18 °C. A solution of aromatic aldehyde (12 mmol), chlorotrimethylsilane (24 mmol), and THF (10 mL) was added drop by drop and the temperature was allowed to rise to room temperature. After the reaction mixture was stirred for 12 h, saturated NH_4Cl in water solution (10 mL) and Et_2O (10 mL) were added, and the mixture was stirred for 10 min. The organic layer was separated, dried over anhydrous $MgSO_4$, and concentrated. Purification of the crude reaction mixture by chromatography on a silica gel column using petroleum/diethyl ether as the eluent gave the pure product. All compounds were characterized by IR, ^{1}H NMR, ^{13}C NMR, microanalyses, and mass spectroscopy.

4.2.1. *1-Butyl-1-phenylpentane* ***3a***

Oil; ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C, TMS): δ=7.34 (t, J=7.2 Hz, 2H, Ar–H), 7.23 (d, J=7.2 Hz, 1H), 7.20 (d, J=7.6 Hz, 2H), 2.55–2.50 (m, 1H), 1.72–1.62 (m, 2H), 1.60–1.58 (m, 2H), 1.34–1.28 (m, 4H), 1.22–1.16 (m, 4H), 0.89 (t, J=7.2 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ=146.4, 128.1, 127.6, 125.7, 46.0, 36.7, 29.8, 22.8, 14.0; DEPT (100 MHz, $CDCl_3$, 25 °C): δ=146.4, 128.1, 127.6, 125.7, 46.0, 36.7, 29.8, 22.8, 14.0; IR (neat): $\tilde{\nu}$=3027, 2958, 2926, 2858, 1465, 1378, 758, 700 cm^{-1}; MS (70 eV, EI): m/z (%): 204 (3.7) [M$^+$], 147 (20.1), 117 (2.8), 91 (100), 77 (5). Anal. Calcd (%) for $C_{15}H_{24}$ (204.4): C, 88.16; H, 11.84. Found: C, 88.05; H, 11.97.

4.2.2. *1-Butyl-1-(4-methoxyphenyl)pentane* ***3b***

Oil; ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ=7.04 (dt, J=6.4, 2.0 Hz, 2H), 6.83 (dt, J=6.4, 2.0 Hz, 2H), 3.78 (s, 3H), 2.44–2.38 (m, 1H), 1.62–1.49 (m, 4H), 1.20 (br s, 8H), 0.83 (t, J=7.2 Hz, 6H); ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C): δ=157.6, 138.5, 128.4, 113.5, 55.1, 45.1, 36.9, 29.8, 22.8, 14.0; IR (neat): $\tilde{\nu}$=2925, 2856, 1608, 1592, 1464, 1375, 1261, 1032, 765, 692 cm^{-1}; MS (70 eV, EI): m/z (%): 234 (8) [M$^+$], 177 (55), 121 (100), 91 (4), 77 (2). Anal. Calcd (%) for $C_{16}H_{26}O$ (234.4): C, 81.99; H, 11.18. Found: C, 81.92; H, 11.13.

4.2.3. *1-Hexyl-1-phenylheptane* ***3c***

Oil; ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C, TMS): δ=7.29 (q, J=7.2 Hz, 2H), 7.20 (d, J=7.2 Hz, 1H), 7.16 (d, J=7.6 Hz, 2H), 2.51–2.47 (m, 1H), 1.67–1.58 (m, 2H), 1.57–1.54 (m, 2H), 1.24 (br s, 12H), 1.17–1.12 (m, 4H), 0.88 (t, J=6.4 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ=146.4, 128.1, 127.6, 125.6, 46.1, 37.0, 31.8, 29.5, 27.6, 22.7, 14.1; IR (neat): $\tilde{\nu}$=3063, 3027, 2950, 2927, 2858, 1603, 1493, 1453, 1370, 1069, 759, 700 cm^{-1}; MS (70 eV, EI): m/z (%): 262 (26) [M$^+$+2], 176 (43), 134 (6), 120 (17), 92 (100). Anal. Calcd (%) for $C_{19}H_{32}$ (260.5): C, 87.62; H, 12.38. Found: C, 87.80; H, 12.38.

4.2.4. *1-Hexyl-1-(4-methoxyphenyl)heptane* ***3d***

Oil; ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C, TMS): δ=7.06 (dd, J=7.6, 2.0 Hz, 2H), 6.85 (dd, J=7.6, 2.0 Hz, 2H), 3.81 (s, 3H), 2.44–2.41 (m, 1H), 1.65–1.57 (m, 2H), 1.55–1.49 (m, 2H), 1.23 (br s, 12H), 1.22–1.07 (m, 4H), 0.87 (t, J=6.8 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ=157.5, 138.5, 128.4, 113.5, 55.1, 45.2, 37.2, 31.8, 29.4, 27.6, 22.7, 14.1; IR (neat): $\tilde{\nu}$=2924, 2854, 1611, 1578, 1510, 1461, 1374, 1300, 1247, 1176, 1106, 1040, 828, 724, 673, 574 cm^{-1}; MS (70 eV, EI): m/z (%): 292 (20) [M$^+$+2], 207 (74), 122 (100), 92 (4), 78 (2). Anal. Calcd (%) for $C_{20}H_{34}O$ (290.5): C, 82.69; H, 11.80. Found: C, 82.59; H, 11.68.

4.2.5. 1-Hexyl-1-(3,4-dimethoxyphenyl)heptane **3e**

Oil; ^{1}H NMR (400 MHz, CDCl$_3$, 25 °C, TMS): δ=6.79 (d, J=7.6 Hz, 1H), 6.67 (dd, J=8.8, 1.2 Hz, 2H), 3.88 (s, 3H), 3.87 (s, 3H), 2.43–2.37 (m, 1H), 1.64–1.53 (m, 2H), 1.52–1.47 (m, 2H), 1.24 (br s, 16H), 0.85 (t, J=6.9 Hz, 6H); ^{13}C NMR (100 MHz, CDCl$_3$, 25 °C): δ=148.6, 146.8, 139.0, 119.5, 110.8, 110.5, 55.7, 45.6, 37.0, 31.7, 29.4, 27.5, 22.6, 14.0; DEPT (100 MHz, CDCl$_3$, 25 °C): δ=148.6, 146.8, 139.0, 119.5, 110.8, 110.5, 55.7, 45.6, 37.0, 31.7, 29.4, 27.5, 22.6, 14.0; IR (neat): $\tilde{\nu}$=2996, 2927, 2855, 1605, 1592, 1515, 1464, 1410, 1376, 1325, 1261, 1235, 1150, 1143, 1031, 852, 806, 764, 692, 652 cm^{-1}; MS (70 eV, EI): m/z (%): 321 (2) [M$^+$+1], 320 (9) [M$^+$], 235 (22), 151 (100), 91 (12), 77 (4), 57 (2), 43 (9). Anal. Calcd (%) for C$_{21}$H$_{36}$O$_2$ (320.5): C, 78.69; H, 11.32. Found: C, 78.66; H, 11.26.

4.2.6. 1-Octyl-1-(4-methoxyphenyl)nonane **3f**

Oil; ^{1}H NMR (400 MHz, CDCl$_3$, 25 °C, TMS): δ=7.08 (d, J=8.8 Hz, 2H), 6.85 (d, J=8.8 Hz, 2H), 3.82 (s, 3H), 2.51–2.25 (m, 1H), 1.64–1.56 (m, 2H), 1.52–1.48 (m, 2H), 1.30–1.12 (m, 24H), 0.90 (t, J=6.4 Hz, 6H); ^{13}C NMR (100 MHz, CDCl$_3$, 25 °C): δ=157.5, 138.5, 128.4, 113.5, 55.1, 45.1, 37.1, 31.9, 29.8, 29.5, 29.3, 27.6, 22.7, 14.1; IR (neat): $\tilde{\nu}$=2926, 2854, 1611, 1504, 1511, 1464, 1377, 1247, 1177, 1041, 820 cm^{-1}; MS (70 eV, EI): m/z (%): 346 (11.6) [M$^+$], 203 (52), 161 (0.4), 147 (10.6), 133 (13.5), 119 (18), 105 (21), 91 (100), 57 (7.3). Anal. Calcd (%) for C$_{24}$H$_{42}$O (346.6): C, 83.17; H, 12.21. Found: C, 83.07; H, 12.16.

4.2.7. 1-Octyl-1-phenylnonane **3g**

Oil; ^{1}H NMR (400 MHz, CDCl$_3$, 25 °C, TMS): δ=7.34 (t, J=7.2 Hz, 2H), 7.23 (d, J=7.2 Hz, 1H), 7.17 (d, J=7.2 Hz, 2H), 2.53–2.48 (m, 1H), 1.68–1.59 (m, 2H), 1.58–1.54 (m, 2H), 1.26 (br s, 20H), 1.19–1.13 (m, 4H), 0.91 (t, J=6.8 Hz, 6H); ^{13}C NMR (100 MHz, CDCl$_3$, 25 °C): δ=146.4, 128.1, 127.7, 125.6, 46.0, 37.0, 31.9, 29.8, 29.5, 29.3, 27.6, 22.7, 14.1; DEPT (100 MHz, CDCl$_3$, 25 °C): δ=146.4, 128.1, 127.7, 125.6, 46.0, 37.0, 31.9, 29.8, 29.5, 29.3, 27.6, 22.7, 14.1; IR (neat): $\tilde{\nu}$=3062, 3027, 2956, 2926, 2855, 1937, 1865, 1603, 1493, 1465, 1377, 1204, 1170, 1110, 1029, 905, 760, 700 cm^{-1}; MS (70 eV, EI): m/z (%): 316 (5.2) [M$^+$], 233 (54), 121 (100), 91 (2), 77 (0.4). Anal. Calcd (%) for C$_{23}$H$_{40}$ (316.6): C, 87.26; H, 12.74. Found: C, 87.26; H, 12.71.

4.2.8. 1-Heptyl-1-phenyloctane **3h**

Oil; ^{1}H NMR (400 MHz, CDCl$_3$, 25 °C, TMS): δ=7.31 (q, J=7.2 Hz, 2H), 7.21 (d, J=7.2 Hz, 1H), 7.17 (d, J=7.2 Hz, 2H), 2.53–2.47 (m, 1H), 1.70–1.61 (m, 2H), 1.59–1.54 (m, 2H), 1.25 (br s, 16H), 1.17–1.10 (m, 4H), 0.90 (t, J=6.4 Hz, 6H); ^{13}C NMR (100 MHz, CDCl$_3$, 25 °C): δ=146.4, 128.1, 127.7, 125.7, 46.1, 37.0, 31.7, 29.8, 29.3, 27.7, 22.7, 14.1; IR (neat): $\tilde{\nu}$=3062, 3027, 2957, 2926, 2855, 1938, 1603, 1493, 1464, 1370, 1210, 1171, 1073, 759, 700 cm^{-1}; MS (70 eV, EI): m/z (%): 290 (21) [M$^+$+2], 191 (48), 148 (6), 134 (13), 106 (34), 91 (100), 78 (3). Anal. Calcd (%) for C$_{21}$H$_{36}$ (288.3): C, 87.42; H, 12.58. Found: C, 87.36; H, 12.43.

4.2.9. 1-Heptyl-1-(4-methoxyphenyl)octane **3i**

Oil; ^{1}H NMR (400 MHz, CDCl$_3$, 25 °C, TMS): δ=7.06 (dd, J=8.4, 1.2 Hz, 2H), 6.84 (dt, J=8.4, 2.0 Hz, 2H), 3.81 (s, 3H), 2.45–2.40 (m, 1H), 1.63–1.58 (m, 2H), 1.57–1.48 (m, 2H), 1.24 (br s, 16H), 1.16–1.10 (m, 4H), 0.87 (t, J=7.2 Hz, 6H); ^{13}C NMR (100 MHz, CDCl$_3$, 25 °C): δ=157.5, 138.5, 128.4, 113.4, 55.1, 45.1, 37.1, 31.9, 29.7, 29.2, 27.6, 22.6, 14.1; DEPT (100 MHz, CDCl$_3$, 25 °C): δ=157.5, 138.5, 128.4, 113.4, 55.1, 45.1, 37.1, 31.9, 29.7, 29.2, 27.6, 22.6, 14.1; IR (neat): $\tilde{\nu}$=2928, 2857, 1608, 1597, 1515, 1468, 1265, 1235, 1140, 1000, 867, 842, 812, 758 cm^{-1}; MS (70 eV, EI): m/z (%): 320 (0.1) [M$^+$+2], 319 (0.7) [M$^+$+1], 318 (3.1) [M$^+$], 220 (5.0), 219 (31), 147 (2.6), 134 (3.7), 121 (100), 91 (5.1), 77 (2.6), 43 (13.2). Anal. Calcd (%) for C$_{22}$H$_{38}$O (318.5): C, 82.95; H, 12.02. Found: C, 82.76; H, 11.87.

4.2.10. 1-Heptyl-1-(3,4-dimethoxyphenyl)octane **3j**

Oil; ^{1}H NMR (400 MHz, CDCl$_3$, 25 °C, TMS): δ=6.80 (d, J=8.0 Hz, 1H), 6.68 (d, J=8.0 Hz, 2H), 3.88 (s, 3H), 3.86 (s, 3H), 2.43–2.37 (m, 1H), 1.63–1.54 (m, 2H), 1.53–1.48 (m, 2H), 1.27–1.11 (m, 20H), 0.86 (t, J=7.2 Hz, 6H); ^{13}C NMR (100 MHz, CDCl$_3$, 25 °C): δ=148.6, 146.9, 139.0, 119.5, 110.9, 110.6, 55.7, 45.6, 37.1, 31.9, 29.7, 29.2, 27.6, 22.6, 14.1; IR (neat): $\tilde{\nu}$=2928, 2856, 1606, 1594, 1515, 1464, 1410, 1362, 1261, 1235, 1156, 1140, 1088, 1032, 867, 842, 809, 754 cm^{-1}; MS (70 eV, EI): m/z (%): 348 (0.03) [M$^+$], 318 (0.08), 249 (2.7), 219 (12.7), 179 (2.3), 167 (56.2), 151 (33.2), 147 (2.8), 139 (38.1), 134 (32.6), 121 (100), 108 (6.4), 91 (11.7), 77 (14.6), 41 (62.8). Anal. Calcd (%) for C$_{23}$H$_{40}$O$_2$ (348.3): C, 79.25; H, 11.57. Found: C, 79.30; H, 11.62.

4.2.11. 1-Honyl-1-phenyldecane **3k**

Oil; ^{1}H NMR (400 MHz, CDCl$_3$, 25 °C, TMS) δ=7.31 (t, J=7.2 Hz, 2H), 7.21 (d, J=7.2 Hz, 1H), 7.16 (d, J=7.2 Hz, 2H), 2.51–2.48 (m, 1H), 1.69–1.61 (m, 2H), 1.59–1.55 (m, 2H), 1.25 (br s, 28H), 0.91 (t, J=6.4 Hz, 6H); ^{13}C NMR (100 MHz, CDCl$_3$, 25 °C): δ=146.4, 128.1, 127.6, 125.6, 46.1, 37.0, 31.9, 29.8, 29.7, 29.6, 29.4, 27.6, 22.7, 14.1; IR (neat): $\tilde{\nu}$=3062, 3027, 2926, 2854, 1603, 1493, 1465, 1378, 1074, 760, 722, 700 cm^{-1}; MS (70 eV, EI): m/z (%): 345 (0.1) [M$^+$+1], 344 (0.5) [M$^+$], 217 (20), 147 (10), 133 (13), 119 (16), 91 (100), 77 (2), 43 (41). Anal. Calcd (%) for C$_{25}$H$_{44}$ (344.6): C, 87.13; H, 12.87. Found: C, 87.24; H, 12.70.

4.2.12. 1-Honyl-1-(4-methoxyphenyl)decane **3l**

Oil; ^{1}H NMR (400 MHz, CDCl$_3$, 25 °C, TMS): δ=7.06 (d, J=8.4 Hz, 2H), 6.84 (d, J=8.4 Hz, 2H), 3.81 (s, 3H), 2.51–2.25 (m, 1H), 1.64–1.56 (m, 2H), 1.52–1.48 (m, 2H), 1.30–1.12 (m, 28H), 0.90 (t, J=6.8 Hz, 6H); ^{13}C NMR (100 MHz, CDCl$_3$, 25 °C): δ=157.5, 138.5, 128.4, 113.5, 55.1, 45.1, 37.1, 31.9, 29.8, 29.7, 29.6, 29.4, 27.6, 22.7, 14.1; IR (neat): $\tilde{\nu}$=3028, 2925, 2854, 1611, 1582, 1511, 1462, 1349, 1246, 1174, 1097, 1040, 832, 723 cm^{-1}; MS (70 eV, EI): m/z (%): 374 (3.9) [M$^+$], 247 (14.8), 147 (4.7), 121 (100), 91 (4.8), 77 (1.5), 57 (47.8), 43 (7.8), 41 (11.3). Anal. Calcd (%) for C$_{26}$H$_{46}$O (374.6): C, 83.35; H, 12.38. Found: C, 83.44; H, 12.29.

4.2.13. 2,2,4,10,12,12-Hexamethyl-7-(4-methoxyphenyl)-tridecane **3m**

Oil; ^{1}H NMR (400 MHz, CDCl$_3$, 25 °C, TMS): δ=7.06 (d, J=8.4 Hz, 2H), 6.84 (d, J=8.4 Hz, 2H), 3.81 (s, 3H), 2.38–2.33 (m, 1H), 1.67–1.55 (m, 2H), 1.55–1.49 (m, 2H), 1.49–1.35 (m, 2H), 1.18–1.12 (m, 4H), 1.09–1.05 (m, 2H), 1.05–0.92 (m, 2H), 0.85 (br s, 24H); ^{13}C NMR (100 MHz, CDCl$_3$, 25 °C): δ=157.5, 138.5, 128.4, 113.5, 55.1, 51.3, 50.9, 45.8, 45.6, 37.3, 34.8, 34.5, 31.0, 30.0, 29.4, 29.2, 22.8, 22.5; IR (neat): $\tilde{\nu}$=3100, 2953, 2715, 1612, 1584, 1512, 1466, 1392, 1376, 1364, 1301, 1240, 1199, 1177, 1042, 959, 829, 808, 707, 605 cm^{-1}; MS (70 eV, EI): m/z (%): 374 (0.1) [M$^+$], 248 (0.5), 197 (0.6), 147 (30.9), 134 (7.4), 121 (17.6), 91 (8), 77 (3.9), 71 (100), 57 (5.6), 43 (20.3). Anal. Calcd (%) for C$_{26}$H$_{46}$O (374.6): C, 83.35; H, 12.38. Found: C, 83.48; H, 12.19.

4.2.14. 2,2,4,10,12,12-Hexamethyl-7-phenyltridecane **3n**

Oil; ^{1}H NMR (400 MHz, CDCl$_3$, 25 °C, TMS): δ=7.32 (dd, J=7.6, 7.2 Hz, 2H), 7.22 (d, J=7.2 Hz, 1H), 7.18 (d, J=8.0 Hz, 2H), 2.47–2.41 (m, 1H), 1.72–1.59 (m, 4H), 1.50–1.34 (m, 2H), 1.26–1.01 (m, 8H), 1.03–0.86 (m, 24H); ^{13}C NMR (100 MHz, CDCl$_3$, 25 °C): δ=146.4, 128.1, 127.6, 125.7, 51.3, 50.9, 46.5, 37.3, 34.7, 34.3, 31.0, 30.0, 29.4, 29.4, 22.8, 22.5; DEPT (100 MHz, CDCl$_3$, 25 °C): δ=146.4 (C), 128.1 (CH), 127.6 (CH), 125.7 (CH), 51.3 (CH$_2$), 50.9 (CH$_2$), 46.5 (CH), 37.3 (CH$_2$), 34.7 (CH$_2$), 34.3 (CH$_2$), 31.0 (C), 30.0 (CH$_3$), 29.4 (CH), 29.4 (CH), 22.8 (CH$_3$), 22.5 (CH$_3$); IR (neat): $\tilde{\nu}$=3062, 3027, 2945, 2716, 1602, 1492, 1467, 1393, 1377, 1364, 1247, 1203, 1170, 1070, 964, 846, 756, 700 cm^{-1}; MS (70 eV, EI): m/z (%): 345 (1) [M$^+$+1], 344 (4) [M$^+$], 217 (4), 161 (19), 147 (12), 133 (6), 119 (12), 105 (24), 91 (44), 77 (2), 71 (10), 57 (100), 43 (24). Anal. Calcd (%) for C$_{25}$H$_{44}$ (344.6): C, 87.13; H, 12.87. Found: C, 87.31; H, 12.74.

4.2.15. 5-Phenyl-1,9-dichlorononane **3o**

Oil; ^{1}H NMR (200 MHz, CDCl$_3$, 25 °C, TMS): δ=7.36–7.10 (m, 5H), 3.62–3.41 (m, 4H), 2.37 (q, J=6.8 Hz, 1H), 2.01–1.88 (m, 4H), 1.80–1.57 (m, 4H), 1.43–1.25 (m, 4H); ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C): δ=146.4, 128.1, 127.6, 125.7, 45.9, 31.8, 29.5, 27.6, 22.7; IR (neat): $\tilde{\nu}$=3080, 3058, 3025, 2955, 2930, 2848, 1663, 1598, 1493, 1444, 1383, 1305, 1274, 1070, 960, 741, 692 cm^{-1}; MS (70 eV, EI): *m*/*z* (%): 272 (1.2) [M$^+$], 183 (2.1), 155 (2.5), 145 (12.4), 117 (39.8), 92 (45), 105 (16.8), 91 (100), 77 (18.8). Anal. Calcd (%) for $C_{15}H_{22}Cl_2$ (273.2): C, 65.93; H, 8.12. Found: C, 65.79; H, 7.86.

4.2.16. 5-(4-Methoxyphenyl)-1,9-dichlorononane ***3p***

Oil; ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C): δ=7.28–6.80 (m, 4H), 3.77 (s, 3H), 3.59–3.40 (m, 4H), 2.33 (q, *J*=7.2 Hz, 1H), 1.95–1.23 (m, 12H); ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C): δ=157.8, 138.5, 128.4, 113.5, 55.6, 44.6, 44.6, 35.0, 32.4, 30.0, 24.6; IR (neat): $\tilde{\nu}$=2999, 2934, 2861, 2830, 1609, 1580, 1512, 1462, 1443, 1302, 1240, 1176, 1100, 1036, 960, 833, 808, 754, 725 cm^{-1}; MS (70 eV, EI): *m*/*z* (%): 302 (2) [M$^+$], 211 (45), 193 (17), 147 (5), 121 (100), 91 (31), 77 (4). Anal. Calcd (%) for $C_{16}H_{24}Cl_2O$ (303.3): C, 63.37; H, 7.98. Found: C, 63.58; H, 8.16.

4.2.17. 5-(3,4-Dimethoxyphenyl)-1,9-dichlorononane ***3q***

Oil; ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C, TMS): δ=7.26 (t, *J*=7.8 Hz, 1H), 6.91 (td, *J*=14.8, 7.2 Hz, 2H), 3.89 (s, 3H), 3.87 (s, 3H), 3.53–3.47 (m, 4H), 2.50–2.36 (m, 1H), 1.80–1.53 (m, 8H), 1.36–1.28 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ=155.6, 138.0, 129.5, 120.3, 111.2, 110.5, 55.7, 45.2, 44.9, 36.0, 32.5, 24.8; IR (neat): $\tilde{\nu}$=3045, 2935, 2862, 1930, 1837, 1701, 1598, 1505, 1471, 1419, 1365, 1257, 1232, 1164, 1145, 1070, 1025, 921, 886, 856, 811, 755, 692, 650, 509 cm^{-1}; MS (70 eV, EI): *m*/*z* (%): 334 (4.6) [M$^+$+2], 332 (6.4) [M$^+$], 164 (3.5), 151 (100), 107 (6.7), 91 (11.5), 77 (8.9), 55 (9.8). Anal. Calcd (%) for $C_{17}H_{26}Cl_2O_2$ (333.3): C, 61.26; H, 7.86. Found: C, 61.34; H, 7.78.

4.2.18. 5-Phenyl-1,9-dibromononane ***3r***

Oil; ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ=7.35–7.10 (m, 5H), 3.37–3.14 (m, 4H), 2.38–2.26 (m, 1H), 2.05–1.80 (m, 6H), 1.7–1.0 (m, 6H); ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C): δ=146.4, 128.1, 127.7, 125.7, 45.7, 37.1, 31.8, 29.4, 27.6; IR (neat): $\tilde{\nu}$=3058, 3025, 2927, 2851, 1651, 1598, 1492, 1440, 1253, 1236, 1216, 1204, 1160, 1072, 743, 693 cm^{-1}; MS (70 eV, EI): *m*/*z* (%): 360 (0.2) [M$^+$], 227 (1.7), 225 (1.8), 224 (2.7), 147 (1.9), 145 (13), 137 (1.9), 125 (1.9), 91 (100), 77 (13.3), 55 (29.5). Anal. Calcd (%) for $C_{15}H_{22}Br_2$ (362.1): C, 49.75; H, 6.12. Found: C, 49.87; H, 6.32.

4.2.19. Dietheyl 4-phenylheptanedioate ***3s***

Oil; ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C, TMS): δ=7.27 (t, *J*=7.2 Hz, 2H), 7.19 (d, *J*=6.8 Hz, 1H), 7.10 (d, *J*=6.0 Hz, 2H), 4.13–4.01 (m, 4H), 2.56–2.50 (m, 1H), 2.14–2.09 (m, 4H), 2.03–1.98 (m, 2H), 1.89–1.85 (m, 2H), 1.23 (t, *J*=6.8 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C, TMS): δ=173.4, 143.2, 128.5, 127.6, 126.5, 60.2, 60.1, 44.8, 32.3, 31.5, 24.3, 14.1; IR (neat): $\tilde{\nu}$=3061, 3029, 2981, 2932, 1736, 1601, 1496, 1458, 1371, 1350, 1336, 1275, 1245, 1212, 1157, 1116, 1097, 1068, 1027, 967, 750 cm^{-1}; MS (70 eV, EI): *m*/*z* (%): 293 (2) [M$^+$+1], 292 (10) [M$^+$], 246 (22), 229 (10), 205 (38), 159 (59), 145 (19), 132 (12), 128 (41), 117 (100), 91 (56), 55 (65). Anal. Calcd (%) for $C_{17}H_{24}O_4$ (292.4): C, 69.84; H, 8.27. Found: C, 69.73; H, 8.46.

4.2.20. 1-Pentyl-1-(2-methoxyphenyl)hexane ***3t***

Oil; ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ=7.06–6.95 (m, 4H), 3.82 (s, 3H), 2.43–2.40 (m, 1H), 1.66–1.50 (m, 4H), 1.23 (br s, 8H), 1.20–1.03 (m, 4H), 0.85 (t, *J*=6.8 Hz, 6H); ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C): δ=157.8, 139.5, 127.4, 127.0, 121.0, 113.5, 55.1, 45.2, 37.2, 31.8, 27.6, 22.7, 14.1; IR (neat): $\tilde{\nu}$=2925, 2857, 1615, 1580, 1513, 1463, 1378, 1305, 1250, 1180, 1109, 1042, 830 cm^{-1}; MS (70 eV, EI): *m*/*z* (%): 263 (0.8) [M$^+$+1], 262 (5) [M$^+$], 121 (100), 91 (8), 77 (3). Anal. Calcd (%) for $C_{18}H_{30}O$ (262.2): C, 82.38; H, 11.52. Found: C, 82.30; H, 11.61.

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