Supplementary Material (ESI) for Chemical Communications

Supplementary Information

High yielding alkylations of unactivated sp^3 and sp^2 centres with alkyl-9-BBN reagents using an NHC-based catalyst: Pd-PEPPSI-IPr

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CONTENTS

General Experimental

All reagents were purchased from commercial sources and were used without further purification, unless indicated otherwise. 1,4-Dioxane was purchased from Fluka, stored over 4Å molecular sieves, and handled under Argon. Tetrahydrofuran (THF) was distilled from sodium benzophenone ketyl radical prior to use. All reaction vials (screw-cap threaded, caps attached, 17 × 60 mm) were purchased from Fischer Scientific. $CDCl_3$ was purchased from Cambridge Isotope Laboratories. Thin layer chromatography (TLC) was performed on Whattman 60 F_{254} glass plates and were visualized using UV light (254nm) and/or potassium permanganate. Column chromatography purifications were carried out using the flash technique on Silicycle silica gel 60 (230-400 mesh). NMR spectra were recorded on a Bruker 400 AV spectrometer. The chemical shifts (δ) for ^{1}H are given in ppm are referenced to the residual proton signal of the deuterated solvent. The chemical shifts (δ) for ^{13}C are referenced relative to the signal from the carbon of the deuterated solvent. ^{13}C APT spectra represent a positive set of peaks (indicated by (+)) for quaternary carbons as well as carbon atoms with even number of protons and a negative set of peaks (indicated by (-)) for carbon atoms with odd number of protons. Gas chromatography was performed on Varian Series GC/MS/MS 4000 System.

Synthetic Procedures

General Procedure for Hydroboration

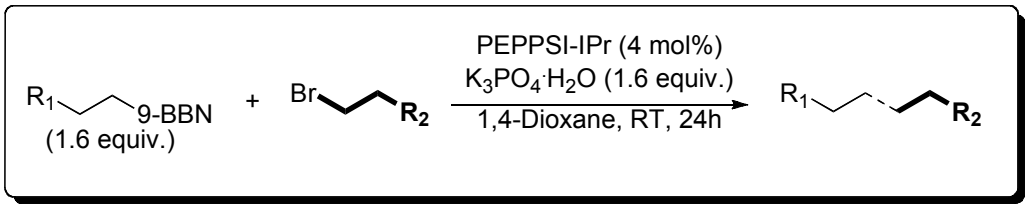
Procedure to make 10 mL of a 1.3 M solution: In air, 9-BBN dimer[1] (7.15 mmol, 1.74 g) was weighed into a flask equipped with a stir bar, sealed with a septum and purged with argon (3×). To this, 5 mL of anhydrous 1,4-dioxane was added, followed by the olefin (13 mmol). More 1,4-dioxane was added as required to deliver a final concentration of 1.3 M based on olefin (10 mL in total). The hydroboration was usually stirred overnight, resulting in a clear, homogeneous solution.

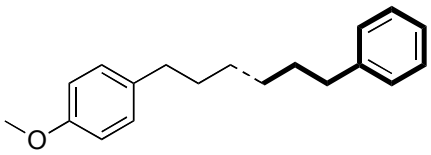
General Procedure for Cross-Coupling

In air, potassium phosphate monohydrate (1.6 mmol, 369 mg) and Pd-PEPPSI-IPr [(1,3-Diisopropylimidazol-2-ylidene)(3-chloropyridyl)palladium(II) dichloride] (0.04 mmol, 27 mg) were weighed into a 3 mL screw-cap threaded vial that was sealed with a septum and purged with argon (3×). With stirring, 1.25 mL of the previously prepared 1.3 M solution of alkyl-9-BBN in 1,4-dioxane (1.6 mmol) was added, followed by the alkyl bromide or aryl bromide/chloride (1.0 mmol). If the electrophile was a solid, it was introduced into the vial prior to purging with argon. The reaction was stirred for 16 hours at room temperature to generally give a dark brown viscous mixture, which was then diluted with 1.5 mL of CH_2Cl_2 and filtered through a plug of silica gel using CH_2Cl_2 as the eluent. The filtrate was concentrated *in vacuo* and the residue purified via silica gel flash chromatography. NOTE: For the optimization study (Table 1), 100 uL of undecane was added at the same time as 3-bromo-1-phenylpropane. After 16 hours, 100 uL of the reaction mixture were removed via syringe and passed through a plug of silica gel using

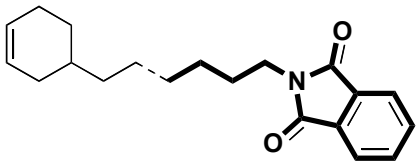
distilled hexane as the eluent to produce a final volume of 2 mL. From this, 40 uL was removed via syringe and added to a vial containing 2 mL of distilled hexane, and was subsequently analyzed by GC-MS.

Compound Data

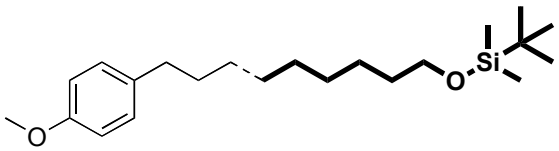




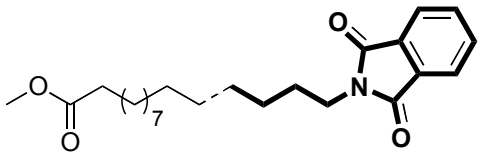
1-Methoxy-4-(6-phenylhexyl)benzene (**2**). Following the general procedure, 246 mg of **2** (92% yield) was isolated (R_f = 0.4, step gradient, two column volumes of 10% CH_2Cl_2 in pentane followed by 20% CH_2Cl_2 in pentane) as a colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.32-7.26 (m, 2H), 7.22-7.15 (m, 3H), 7.10 (d, *J* = 8.4 Hz, 2H), 6.84 (d, *J* = 8.8 Hz, 2H), 3.81 (s, 3H), 2.62 (t, *J* = 8.0 Hz, 2H), 2.56 (t, *J* = 8.0 Hz, 2H), 1.70-1.55 (m, 4H), 1.45-1.35 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 157.6 (+), 142.9 (+), 135.0 (+), 129.3 (-), 128.4 (-), 128.3 (-), 125.6 (-), 113.7 (-), 55.3 (-), 36.0 (+), 35.0 (+), 31.7 (+), 31.5 (+), 29.2 (+), 29.1 (+). Anal. Calcd. for $C_{19}H_{24}O$: C, 85.03; H, 9.01. Found: C, 85.14; H, 8.75.



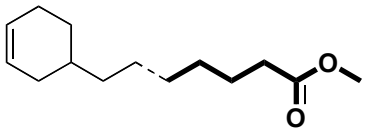
Methyl 2-(6-(cyclohex-3-enyl)hexyl)isoindoline-1,3-dione (**3**). Following the general procedure, 268 mg of **3** (86% yield) was isolated (R_f = 0.5, 60% CH_2Cl_2 in pentane) as a white solid (m.p. 52-54°C). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.90-7.80 (m, 2H), 7.75-7.65 (m, 2H), 6.70-6.60 (m, 2H), 3.68 (t, *J* = 7.2 Hz, 2H), 2.12-1.98 (m, 3H), 1.80-1.40 (m, 5H), 1.40-1.10 (m, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 168.5 (+), 133.8 (-), 132.2 (+), 127.0 (-), 126.7 (-), 123.1 (-), 38.0 (+), 36.6 (+), 33.4 (-), 31.9 (+), 29.4 (+), 28.9 (+), 28.6 (+), 26.9 (+), 26.7 (+), 25.3 (+). Anal. Calcd. for $C_{20}H_{25}NO_2$: C, 77.14; H, 8.09; N, 4.50. Found: C, 77.57; H, 8.12; N, 4.61.



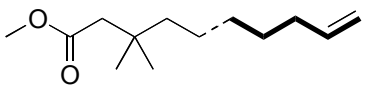
***tert*-Butyl(9-(4-methoxyphenyl)nonyloxy)-dimethylsilane** (**4**). Following the general procedure, 258 mg of **4** (71% yield) was isolated (R_f = 0.5, step gradient, two column volumes of 10% CH_2Cl_2 in pentane followed by 20% CH_2Cl_2 in pentane) as a colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.11 (d, *J* = 8.4 Hz, 2H), 6.84 (d, *J* = 8.4 Hz, 2H), 3.81 (s, 3H), 3.60 (t, *J* = 7.6 Hz, 2H), 2.56 (t, *J* = 7.6 Hz, 2H), 1.55-1.45 (m, 4H), 1.40-1.25 (m, 10H), 0.92 (s, 9H), 0.07 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 157.6 (+), 135.0 (+), 129.2 (-), 113.6 (-), 63.3 (+), 55.2 (-), 35.0 (+), 32.9 (+), 31.8 (+), 29.6 (+), 29.5 (+), 29.3 (+), 26.0 (-), 25.8 (+), 18.4 (+), -5.3 (-). Anal. Calcd. for $C_{22}H_{40}O_2Si$: C, 72.47; H, 11.06. Found: C, 72.12; H, 11.33.



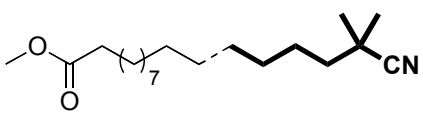
Methyl 15-(1,3-dioxoisoindolin-2-yl)pentadecanoate (**5**). Following the general procedure, **5** was collected (R_f = 0.3, 80% CH_2Cl_2 in pentane) along with a volatile impurity, which was removed by heating the mixture to 60°C *in vacuo* overnight, ultimately providing 324 mg of **5** (81% yield) as a white solid (m.p. 62-63°C). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85-7.78 (m, 2H), 7.72-7.65 (m, 2H), 3.70-3.60 (m, 5H), 2.28 (t, *J* = 7.6 Hz, 2H), 1.70-1.50 (m, 4H), 1.40-1.15 (m, 20H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 174.3 (+), 168.4 (+), 133.8 (-), 132.1 (+), 123.1 (-), 51.4 (-), 38.0 (+), 34.1 (+), 29.5 (+), 29.4 (+), 29.4 (+), 29.2 (+), 29.2 (+), 29.1 (+), 28.6 (+), 26.8 (+), 24.9 (+). Overlapping peaks in the methylene region account for the remaining ^{13}C resonances. Anal. Calcd. for $C_{24}H_{35}NO_4$: C, 71.79; H, 8.79; N, 3.16. Found: C, 71.97; H, 8.72; N, 3.16.



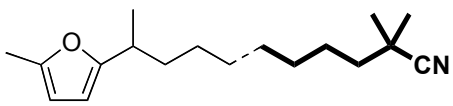
Methyl 7-(cyclohex-3-enyl)heptanoate (**6**). Following the general procedure, 138 mg of **6** (62% yield) was isolated (R_f = 0.3, step gradient, two column of 20% CH_2Cl_2 in pentane followed by 40% CH_2Cl_2 in pentane) as a colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 5.67-5.50 (m, 2H), 3.65 (s, 3H), 2.29 (t, *J* = 7.2 Hz, 2H), 2.10-1.95 (m, 3H), 1.75-1.55 (m, 4H), 1.55-1.42 (m, 1H), 1.40-1.10 (m, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 174.2 (+), 127.0 (-), 126.6 (-), 51.4 (-), 36.6 (+), 34.0 (+), 33.4 (-), 31.9 (+), 29.5 (+), 29.1 (+), 28.9 (+), 26.7 (+), 25.3 (+), 24.9 (+). HRMS *m/e* calcd. for $C_{14}H_{24}O_2$ (M^+) 244.1776, found: 244.1766. ^{1}H and ^{13}C specta are include to attest sample purity.



Methyl 3,3-dimethyldec-9-enoate (**7**). Following the general procedure, 172 mg of **7** (81% yield) was isolated (R_f = 0.4, 20% CH_2Cl_2 in pentane) as a colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 5.90-5.75 (m, 1H), 5.05-4.90 (m, 2H), 3.65 (s, 3H), 2.20 (s, 2H), 2.04 (q, *J* = 6.8 Hz, 2H), 1.45-1.35 (m, 2H), 1.30-1.22 (m, 6H), 0.98 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 172.8 (+), 139.1 (-), 114.2 (+), 51.0 (-), 45.8 (+), 42.2 (+), 33.8 (+), 33.2 (+), 29.8 (+), 28.9 (+), 27.2 (-), 23.9 (+). HRMS *m/e* calcd. for $C_{13}H_{24}O_2$ (M^+) 212.1776, found: 212.1770. ^{1}H and ^{13}C specta are include to attest sample purity.

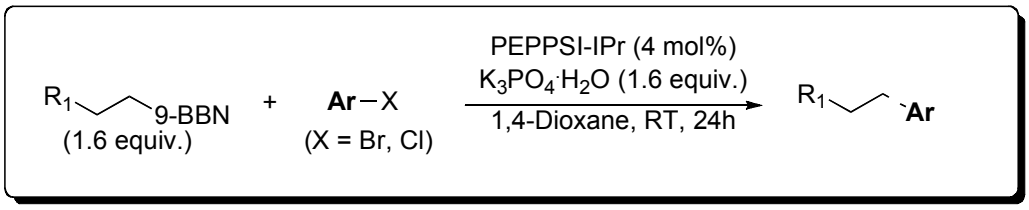


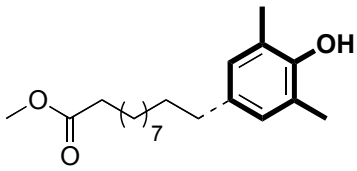
Methyl 16-cyano-16-methylheptadecanoate (**8**). Following the general procedure, **8** was collected (R_f = 0.4, 60% CH_2Cl_2 in pentane) along with a volatile impurity, which was removed by heating the mixture to 50°C *in vacuo* overnight, ultimately providing 235 mg of **8** (73% yield) as a white solid (m.p. 32-34°C). ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.69 (s, 3H), 2.32 (t, *J* = 7.2 Hz, 2H), 1.70-1.45 (m, 6H), 1.40-1.20 (m, 26H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 174.3 (+), 125.3 (+), 51.4 (-), 41.1 (+), 34.1 (+), 32.4 (+), 29.6 (+), 29.6 (+), 29.5 (+), 29.4 (+), 29.4 (+), 29.2 (+), 29.1 (+), 26.7 (-), 25.2 (+), 24.9 (+). Overlapping peaks in the methylene region account for the remaining ^{13}C resonances. Anal. Calcd. for $C_{20}H_{37}NO_2$: C, 74.25; H, 11.53. Found: C, 74.40; H, 11.45.



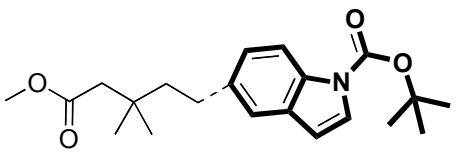
2,2-dimethyl-10-(5-methylfuran-2-yl)undecane-nitrile (**9**). Following the general procedure, 226 mg of **9** (82% yield) was isolated (R_f = 0.4, step gradient, two column volumes of 20%

CH_2Cl_2 in pentane followed by 40 % CH_2Cl_2 in pentane) as a colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 5.84 (s, 2H), 2.75 (quin., *J* = 6.8 Hz, 1H), 2.27 (s, 3H), 1.72-1.60 (m, 1H), 1.58-1.40 (m, 5H), 1.40-1.25 (m, 14H), 1.22 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.1 (+), 149.8 (+), 125.2 (+), 105.5 (-), 103.8 (-), 41.1 (+), 35.7 (+), 33.1 (-), 32.4 (+), 29.6 (+), 29.5 (+), 29.3 (+), 27.1 (+), 26.7 (-), 25.2 (+), 19.2 (-), 13.5 (-). Anal. Calcd. for $C_{18}H_{29}NO$: C, 78.49; H, 10.61. Found: C, 78.35; H, 10.80.

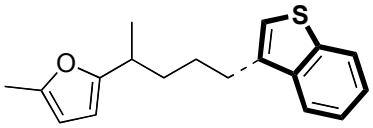




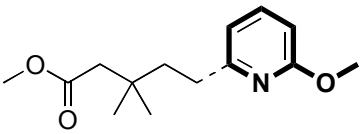
Methyl 11-(4-hydroxy-3,5-dimethylphenyl)undecanoate (**10**). Following the general procedure, 237 mg of **10** (74% yield) was isolated (R_f = 0.5, CH_2Cl_2) as a white solid (m.p. 66-68^{o}C). ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.81 (s, 2H), 4.51 (s, 1H), 3.69 (s, 3H), 2.48 (t, *J* = 7.6 Hz, 2H), 2.33 (t, *J* = 7.6 Hz, 2H), 2.25 (s, 6H), 1.70-1.50 (m, 4H), 1.40-1.20 (m, 12H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 174.5 (+), 150.1 (+), 134.5 (+), 128.4 (-), 122.8 (+), 51.5 (-), 35.1 (+), 34.1 (+), 31.9 (+), 29.5 (+), 29.4 (+), 29.4 (+), 29.3 (+), 29.2 (+), 25.0 (+), 16.0 (-). Overlapping peaks in the methylene region account for the remaining ^{13}C resonances. Anal. Calcd. for $C_{20}H_{32}O_3$: C, 74.96; H, 10.06. Found: C, 75.02; H, 10.34.



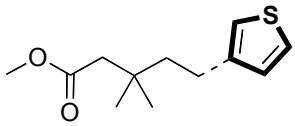
***tert*-Butyl 5-(5-methoxy-3,3-dimethyl-5-oxopentyl)-1*H*-indole-1-carboxylate** (**11**). Following the general procedure, 316 mg of **11** (88% yield) was isolated (R_f = 0.4, 80 % CH_2Cl_2 in pentane) as a colourless, viscous oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.07 (br. d, *J* = 6.4 Hz, 1H), 7.59 (d, *J* = 2.4 Hz, 1H), 7.40 (s, 1H), 7.18 (d, *J* = 8.4 Hz, 1H), 6.53 (d, *J* = 3.6 Hz, 1H), 3.71 (s, 3H), 2.80-2.70 (m, 2H), 2.34 (s, 2H), 1.75-1.65 (m, 2H), 1.70 (s, 9H), 1.12 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 172.7 (+), 149.8 (+), 137.3 (+), 133.5 (+), 130.8 (+), 126.0 (-), 124.9 (-), 120.2 (-), 115.0 (-), 107.1 (-), 83.4 (+), 51.2 (-), 45.7 (+), 45.0 (+), 33.5 (+), 30.6 (+), 28.2 (-), 27.4 (-). Anal. Calcd. for $C_{21}H_{29}NO_4$: C, 70.17; H, 8.13. Found: C, 69.93; H, 8.40.



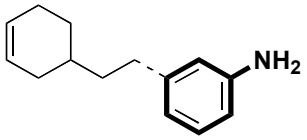
2-(5-(Benzo[*b*]thiophen-3-yl)pentan-2-yl)-5-methylfuran (**12**). Following the general procedure, 220 mg of **12** (77% yield) was isolated (R_f = 0.3, pentane) as a colourless, viscous oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.93 (d, *J* = 7.6 Hz, 1H), 7.81 (d, *J* = 7.6 Hz, 1H), 7.50-7.40 (m, 2H), 7.13 (s, 1H), 5.94 (s, 2H), 2.98-2.88 (m, 3H), 2.35 (s, 3H), 1.95-1.80 (m, 3H), 1.80-1.68 (m, 1H), 1.16 (d, *J* = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 158.7 (+), 150.1 (+), 140.5 (+), 139.2 (+), 137.0 (+), 124.1 (-), 123.8 (-), 122.9 (-), 121.8 (-), 121.0 (-), 105.7 (-), 104.2 (-), 35.8 (+), 33.1 (-), 28.6 (+), 26.7 (+), 19.4 (-), 13.6 (-). Anal. Calcd. for $C_{18}H_{20}OS$: C, 76.01; H, 7.09. Found: C, 75.76; H, 7.21.



Methyl 5-(6-methoxypyridin-2-yl)-3,3-dimethylpentanoate (**13**). Following the general procedure, **13** was collected (R_f = 0.2, 80% CH_2Cl_2 in pentane) along with a volatile impurity, which was removed by heating the mixture to 60°C *in vacuo* overnight, ultimately providing 223 mg of **13** (89% yield) as a colourless, viscous oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.45 (t, *J* = 7.6 Hz, 1H), 6.71 (d, *J* = 7.2 Hz, 1H), 6.52 (d, *J* = 8 Hz, 1H), 3.91 (s, 3H), 3.66 (s, 3H), 2.72-2.65 (m, 2H), 2.29 (s, 2H), 1.80-1.70 (m, 2H), 1.08 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 172.7 (+), 163.6 (+), 160.4 (+), 138.7 (-), 114.9 (-), 107.1 (-), 53.1 (-), 51.1 (-), 45.8 (+), 41.7 (+), 33.3 (+), 32.8 (+), 27.2 (-). Anal. Calcd. for $C_{14}H_{21}NO_3$: C, 66.91; H, 8.42. Found: C, 66.63; H, 8.62.

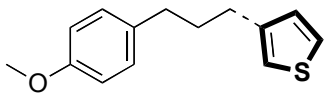


Methyl 3,3-dimethyl-5-(thiophen-3-yl)pentanoate (**14**). Following the general procedure, 179 mg of **14** (79% yield) was isolated (R_f = 0.3, 10% diethyl ether in pentane) as a colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.30-7.20 (m, 1H), 7.00-6.95 (m, 2H), 3.69 (s, 3H), 2.70-2.62 (m, 2H), 2.31 (s, 2H), 1.75-1.65 (m, 2H), 1.09 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 172.6 (+), 143.0 (+), 128.3 (-), 125.3 (-), 119.7 (-), 51.2 (-), 45.7 (+), 42.9 (+), 33.3 (+), 27.3 (-), 25.1 (+). Anal. Calcd. for $C_{12}H_{18}O_2S$: C, 63.68; H, 8.02. Found: C, 63.34; H, 8.32.

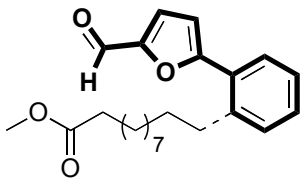


3-(2-(Cyclohex-3-enyl)ethyl)aniline (**15**). Following the general procedure, **15** was collected (R_f = 0.3, step gradient, two column volumes of 15% diethyl ether in pentane followed by 25% diethyl ether in pentane) along with a volatile impurity, which was removed by heating the mixture to

40°C *in vacuo* overnight, ultimately providing 153 mg of **15** (76% yield) as a light brown, viscous oil. Note: Compound decomposes in air. It is recommended that it is stored under argon and away from light. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.13 (t, *J* = 7.6 Hz, 1H), 6.67 (d, *J* = 7.2 Hz, 1H), 6.60-6.52 (m, 2H), 5.80-5.68 (m, 2H), 3.65 (br. s, 2H), 2.63 (t, *J* = 7.6 Hz, 2H), 2.30-2.00 (m, 3H), 1.90-1.55 (m, 5H), 1.40-1.25 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.4 (+), 144.3 (+), 129.2 (-), 127.1 (-), 126.6 (-), 118.8 (-), 115.3 (-), 112.6 (-), 38.5 (+), 33.3 (+), 33.2 (-), 31.9 (+), 28.9 (+), 25.3 (+). HRMS *m/e* calcd. for $C_{14}H_{19}N$ (M^+) 201.1517, found: 201.1515. Anal. Calcd. for $C_{14}H_{19}N$: C, 83.53; H, 9.51. Found: C, 83.24; H, 9.78.

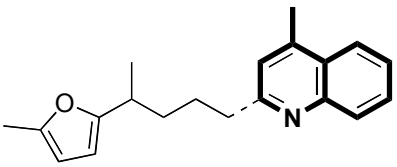


3-(3-(4-Methoxyphenyl)propyl)thiophene (**16**). Following the general procedure, 205 mg of **16** (88% yield) was isolated (R_f = 0.4, 20% CH_2Cl_2 in pentane) as a colourless, viscous oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.30-7.26 (m, 1H), 7.15 (d, *J* = 8.4 Hz, 2H), 7.00-6.95 (m, 2H), 6.88 (d, *J* = 8.0 Hz, 2H), 3.84 (s, 3H), 2.70 (t, *J* = 7.6 Hz, 2H), 2.65 (t, *J* = 7.6 Hz, 2H), 1.98 (quin., *J* = 7.6 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 157.7 (+), 142.7 (+), 134.3 (+), 129.3 (-), 128.2 (-), 125.2 (-), 120.0 (-), 113.7 (-), 55.3 (-), 34.5 (+), 32.3 (+), 29.7 (+). Anal. Calcd. for $C_{14}H_{16}OS$: C, 72.37; H, 6.94. Found: C, 71.91; H, 7.11.



Methyl 11-(2-(5-formylfuran-2-yl)phenyl)undecanoate (**17**). Following the general procedure, 246 mg of **17** (58% yield) was isolated (R_f = 0.5, CH_2Cl_2) as an amorphous, yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.70 (s, 1H), 7.70 (d, *J* = 7.6 Hz, 1H), 7.40-7.25 (m, 4H),

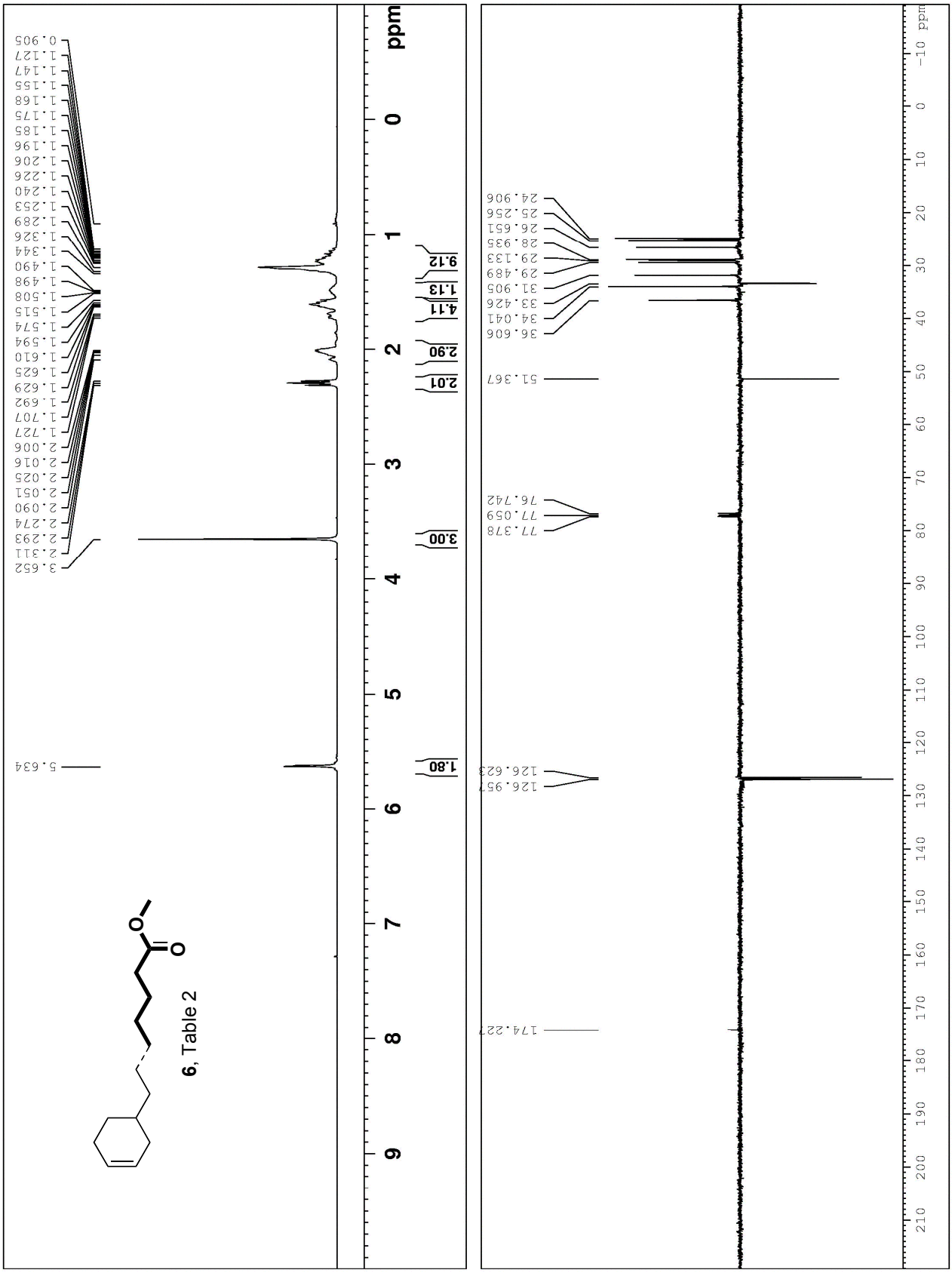
6.71 (d, *J* = 3.2 Hz, 1H), 3.69 (s, 3H), 2.86 (t, *J* = 7.6 Hz, 2H), 2.32 (t, *J* = 7.6 Hz, 2H), 1.70-1.55 (m, 4H), 1.45-1.20 (m, 12H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 177.3 (-), 174.3 (+), 159.7 (+), 151.9 (+), 141.2 (+), 130.5 (-), 129.6 (-), 129.0 (-), 128.1 (+), 126.1 (-), 110.8 (-), 51.4 (-), 34.3 (+), 34.1 (+), 31.1 (+), 29.6 (+), 29.5 (+), 29.4 (+), 29.4 (+), 29.2 (+), 29.1 (+), 24.9 (+). Overlapping peaks in the aromatic region account for the remaining ^{13}C resonances. Anal. Calcd. for $C_{23}H_{30}O_4$: C, 74.56; H, 8.16. Found: C, 74.49; H, 8.43.



4-Methyl-2-(4-(5-methylfuran-2-yl)pentyl)quinoline (**18**). Following the general procedure, 208 mg of **18** (71% yield) was isolated (R_f = 0.5, CH_2Cl_2) as a colourless, viscous oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.05 (d, *J* = 8.4 Hz, 1H), 7.96 (d, *J* = 8.4 Hz, 1H), 7.69 (t, *J* = 7.2 Hz, 1H), 7.52 (t, *J* = 7.2 Hz, 1H), 7.14 (s, 1H), 5.85 (s, 2H), 2.94 (t, *J* = 7.6 Hz, 2H), 2.82 (q, *J* = 6.8 Hz, 1H), 2.69 (s, 3H), 2.27 (s, 3H), 1.90-1.75 (m, 3H), 1.70-1.55 (m, 1H), 1.24 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 162.5 (+), 158.7 (+), 150.0 (+), 147.7 (+), 144.2 (+), 129.3 (-), 129.0 (-), 126.8 (+), 125.4 (-), 123.6 (-), 122.0 (-), 105.5 (-), 104.0 (-), 39.2 (+), 35.7 (+), 33.1 (-), 27.7 (+), 19.2 (-), 18.7 (-), 13.6 (-). Anal. Calcd. for $C_{20}H_{23}NO$: C, 81.87; H, 7.90. Found: C, 81.88; H, 8.13.

Reference

(1) 9-borabicyclo[3.3.1]nonane dimer (9-BBN dimer) was prepared according to the following procedure: Soderquist, J.A.; Negron, A. *Org. Synth.* **1992**, *70*, 169-76.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76.742
77.059
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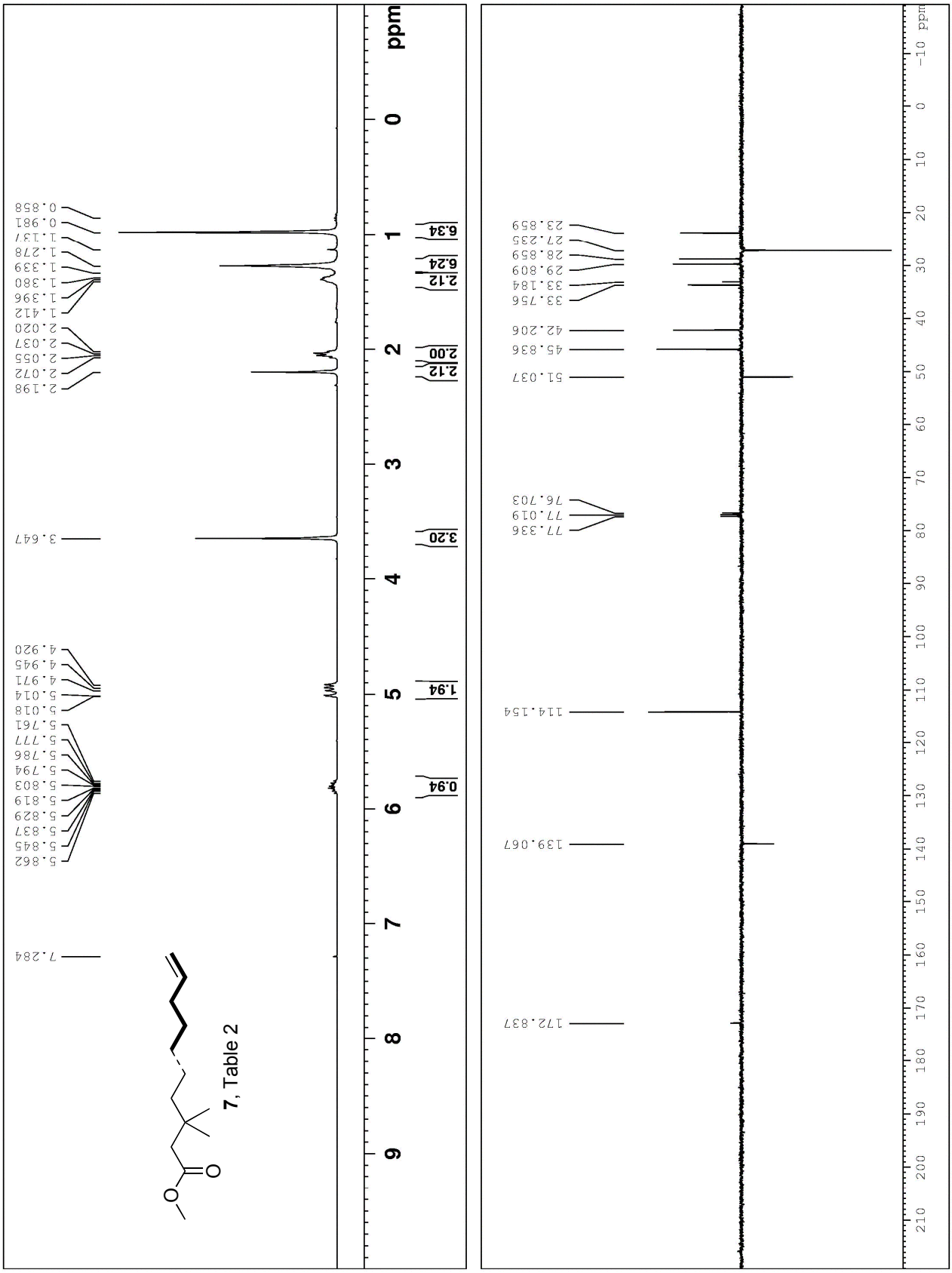
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