Uncatalyzed Friedel–Crafts Alkylation of Aromatic Compounds Through Reactive Benzyl Cations Generated From *N*-Sulfamoylcarbamates

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Supporting Information.

General Experimental

All reactions were performed in dried glassware under an inert (N₂) atmosphere. Standard reagents and solvents were purified according to known procedures.¹ Thin layer chromatographic (TLC) analyses were performed on silica gel plates Merck 60 F₂₅₄. Column chromatographic purifications (“flash chromatography”, FC) were performed as described.² Melting points were determined using a Büchi SMP-20 apparatus and are uncorrected. Optical rotations were measured with a JASCO DIP-1000 polarimeter. ¹H NMR and ¹³C NMR spectra were obtained using a Bruker ARX 300 instrument at 300.1 and 75.4 MHz, respectively. Unless otherwise stated, CDCl₃ was used as solvent. Infrared (IR) spectra were recorded with a Perkin-Elmer FT-IR 16 PC instrument either from KBr plates or neat. Ultraviolet (UV) spectra were obtained from alcoholic solutions utilizing a Shimadzu UV-160A or an ATI UNICAM UV 3 instrument. Mass spectra were achieved by electron impact (EI) method using a Finnigan MAT SSQ 710 instrument. Elemental analyses were performed on a LECO CHNS-932 instrument.

General procedure 1 (GP 1): Preparation of *N*-sulfamoylcarbamates. To a solution of 0.05 mol of the benzyl alcohol in THF (100 mL) was added 0.05 mol of CSI in such a way that the internal temperature was maintained below –5 °C (–60 °C for alkyl substituents). The mixture was stirred for 30 min at –5 °C (–60 °C) and 0.1 mol of the aniline was added at that

temperature. The solution was stirred for 2 h by which time the temperature raised to 5–10 °C (raised to –30 °C). An aqueous solution of 1N HCl was added until pH ~ 2 was obtained. The aqueous layer was extracted with Et₂O (3 x 20 mL). The combined organic extracts were dried over MgSO₄, filtered, and concentrated under reduced pressure. The residue was redissolved, filtered through a plug of silica gel and recrystallized from an appropriate solvent.

Benzyl *N*-phenyl-sulfamoylcarbamate (1). According to GP 1, benzyl alcohol (5.41 g, 50 mmol) was reacted with CSI (4.4 mL, 7.2 g, 51 mmol) and aniline (10.0 mL, 10.2 g, 110 mmol) in THF (100 mL) at –10 °C.

Recrystallized from EtOAc and light petroleum.

Yield of **1**: 11.9 g (78%). Colorless needles. M.p. 140–141 °C. – IR (KBr): $\tilde{\nu}$ 3237, 1716, 1600, 1452, 1158, 764 cm^{–1}. – ¹H NMR: 7.32–7.11 (m, 10H, H_{Ar}); 5.11 (s, 2H, CH₂O); 4.12 (br. s, 2H, NH). – ¹³C NMR: 151.69 (s), 136.23 (s), 134.79 (s), 128.90 (d), 128.19 (d), 128.10 (d), 127.69 (d), 124.93 (d), 120.91 (d), 67.49 (t). – MS (70eV): *m/z* (%) 306 (27), 262 (8), 198 (2), 181 (57), 91 (100). – UV (EtOH): λ_{max} () 231 (7700), 202 (28500) nm. – elemental analysis (%): calcd. for C₁₄H₁₄N₂O₄S (306.34): C 54.89, H 4.61, N 9.14, S 10.47; found C 54.75, H 4.64, N 9.11, S 10.51.

2,4,6-Trimethylbenzyl *N*-phenyl-sulfamoylcarbamate (2). According to GP 1, 2,4,6-trimethylbenzyl alcohol (3.75 g, 25 mmol) was reacted with CSI (2.3 mL, 3.74 g, 26 mmol) and aniline (5.6 mL, 5.7 g, 61 mmol) in THF (80 mL) at –9 °C.

Recrystallized from EtOAc and light petroleum.

Yield of **2**: 7.8 g (90%). Colorless solid. M.p. 172 °C. – IR (KBr): $\tilde{\nu}$ 3272, 2923, 1725, 1600, 1439, 1155, 843 cm^{–1}. – ¹H NMR (MeOH-d₄): 7.28–7.23 (m, 2H, H_{Ar}); 7.16–7.08 (m, 3H, H_{Ar}); 6.80 (s, 2H, H_{Ar}); 5.15 (s, 2H, CH₂O); 2.22 (s, 9H, CH₃). – ¹³C NMR (MeOH-d₄): 153.33 (s), 139.63 (s), 139.36 (s), 138.61 (s), 130.13 (d), 129.89 (d), 129.73 (s), 125.79 (d), 121.93 (d), 63.58 (t), 21.08 (q), 19.54 (q). – MS (70eV): *m/z* (%) 348 (0.7), 304 (0.5), 224 (1), 133 (100). – UV (EtOH): λ_{max} () = 225 (15200), 203 (66700) nm. – elemental analysis (%): calcd. for C₁₇H₂₀N₂O₄S (348.42): C 58.60, H 5.79, N 8.04, S 9.20; found C 58.29, H 6.01, N 7.97, S 9.22.

2-Methoxycarbonylmethoxybenzyl *N*-(4-chlorophenyl)-sulfamoylcarbamate (3).

According to GP 1, 2-Methoxycarbonylmethoxybenzyl alcohol (8.05 g, 41 mmol) was reacted with CSI (4.0 mL, 5.80 g, 41 mmol) and *p*-chloroaniline (10.5 g, 82 mmol) in THF (80 mL) at -5°C .

Recrystallized from EtOAc and light petroleum.

Yield of **3**: 12.6 g (72%). Colorless solid. M.p. 153°C . – ^1H NMR: 8.01 (br. s, 2H, NH); 7.32 (td, $J = 7.9, 1.6$, 1H, H_{Ar}); 7.25 (dd, $J = 7.4, 1.6$, 1H, H_{Ar}); 7.14 (d, $J = 8.7$ Hz, 2H, H_{Ar}); 6.96 (m, 1H, H_{Ar}); 6.95 (d, $J = 8.6$ Hz, 2H, H_{Ar}); 6.74 (d, $J = 8.3$ Hz, 1H, H_{Ar}); 5.23 (s, 2H, CH_2O); 4.65 (s, 2H, CH_2O); 3.74 (s, 3H, CH_3O). – ^{13}C NMR: 169.67 (s), 156.10 (s), 151.18(s), 134.40 (s), 131.28 (s), 131.21 (d), 130.53 (d), 129.33 (d), 123.37 (s), 123.19 (d), 121.66 (d), 111.33 (d) 64.91 (t), 64.37 (t), 52.44 (q). – MS (70eV): m/z (%) 430 (1), 428 (3), 234 (9), 232 (27), 208 (2), 206 (7), 179 (100), 151 (13), 126 (61), 124 (52).

2,4-Dichlorobenzyl *N*-(4-chlorophenyl)-sulfamoylcarbamate (4). According to GP 1, 2,4-dichlorobenzyl alcohol (8.47 g, 48 mmol) was reacted with CSI (4.3 mL, 7.0 g, 49 mmol) and *p*-chloroaniline (12.5 g, 98 mmol) in THF (100 mL) at -20°C .

Recrystallized from EtOAc and light petroleum.

Yield of **4**: 17.1 g (87%). Colorless solid. M.p. 160°C . – IR (KBr): $\tilde{\nu}$ 3244, 1714, 1593, 1451, 1166, 861, 598 cm^{-1} . – ^1H NMR (DMSO- d_6): 11.81 (br. s, 1H, NH); 10.76 (s, 1H, NH); 7.63 (s, 1H, H_{Ar}); 7.37 (m, 4H, H_{Ar}); 7.16 (d, $J = 8$ Hz, 2H, H_{Ar}); 5.15 (s, 2H, CH_2O). – ^{13}C NMR (DMSO- d_6): 150.80 (s), 136.44 (s), 133.99 (s), 133.70 (s), 132.18 (s), 131.42 (d), 129.08 (d), 128.97 (d), 127.92 (s), 127.49 (d), 120.80 (d), 63.70 (t). – MS (70eV): m/z (%) 412 (0.7), 410 (1.5), 408 (1.4), 318 (1.1), 316 (1.5), 287 (0.8), 285 (2), 283 (1.7), 234 (5), 232 (16), 160 (58), 159 (100), 126 (54), 99 (39). – UV (EtOH): λ_{max} () = 241 (11800), 203 (53500) nm. – elemental analysis (%): calcd. for $\text{C}_{14}\text{H}_{11}\text{Cl}_3\text{N}_2\text{O}_4\text{S}$ (409.68): C 41.05, H 2.71, N 6.84, S 7.83; found C 41.08, H 2.81, N 6.85, S 7.80.

General procedure 2 (GP 2): The Friedel–Crafts alkylation. A suspension of 2 mmol of *N*-Sulfamoylcarbamate in the aromatic solvent (2–4 mL) was heated to 110 – 125°C . Above 105°C a gas evolution started and the solid dissolved slowly. A colored precipitation was formed during heating and after 5 h at 110 – 125°C , no starting material was detected (TLC

analysis). The solvent was removed under reduced pressure, when possible, and the residue purified by FC on silica gel and recrystallization.

Methyl 2-[2-(2,4,6-trimethylbenzyl)-phenoxy]-acetate (6). According to GP 2, sulfamoylcarbamate **3** (2.00 g, 4.67 mmol) was heated in mesitylene (4.0 mL, 3.46 g, 29 mmol) at 130 °C.

FC (SiO₂, 3 x 30 cm, 11% EtOAc/light petroleum).

Yield of **6**: 0.71 g (51%). Colorless solid. M.p. 78–79 °C. – IR (KBr): $\tilde{\nu}$ 2926, 1763, 1600, 1584, 1488, 1454, 1203, 1115, 749 cm⁻¹. – ¹H NMR (DMSO-d₆): 7.12 (t, *J* = 7.7 Hz, 1H, H_{Ar}); 6.91 (d, *J* = 8.5 Hz, 1H, H_{Ar}); 6.88 (s, 2H, H_{Ar}); 6.76 (t, *J* = 7.5 Hz, 1H, H_{Ar}); 6.38 (d, *J* = 7.5 Hz, 1H, H_{Ar}); 4.91 (s, 2H, CH₂O); 3.91 (s, 2H, CH₂); 3.72 (s, 3H, CH₃O); 2.24 (s, 3H, CH₃); 2.09 (s, 6H, CH₃). – ¹³C NMR (DMSO-d₆): 169.41 (s), 155.49 (s), 136.55 (s), 134.90 (s), 132.99 (s), 128.66 (d), 127.88 (s), 127.00 (d), 126.86 (d), 121.05 (d), 111.35 (d), 64.85 (t), 51.84 (q), 27.87 (t), 20.56 (q), 19.47 (q). – MS (70eV): *m/z* (%) 298 (100), 283 (13), 225 (18), 209 (37), 179 (16), 133 (21). – UV (EtOH): $\lambda_{\max}$ (ε) = 271 (3800), 203 (62000) nm. – elemental analysis (%): calcd. for C₁₉H₂₂O₃ (298.39): C 76.48, H 7.43; found C 76.23, H 7.37.

2,4,6-Trimethylbenzylbenzene (7). According to GP 2, sulfamoylcarbamate **1** (0.92 g, 3.00 mmol) was heated in mesitylene (3.0 mL, 2.59 g, 22 mmol) at 127 °C.

FC (SiO₂, 3 x 20 cm, 0–100% EtOAc/hexane).

Yield of **1**: 0.45 g (71%). Colorless oil. Yield of **dibenzylmesitylene**: 0.05 g (9%). Pale yellow oil.

7: ¹H NMR: 7.23–7.18 (m, 2H, H_{Ar}); 7.12 (m, 1H, H_{Ar}); 7.00 (d, *J* = 7.4 Hz, 2H, H_{Ar}); 6.88 (s, 2H, H_{Ar}); 4.00 (s, 2H, CH₂); 2.28 (s, 3H, CH₃); 2.19 (s, 6H, CH₃). – ¹³C NMR: 140.06 (s), 136.97 (s), 135.61 (s), 133.75 (s), 128.86 (d), 128.31 (d), 127.81 (d), 125.62 (d), 34.67 (t), 20.88 (q), 20.10 (q). – MS (70eV): *m/z* (%) 210 (100), 195 (69), 165 (14).

dibenzylmesitylene: ¹H NMR: 7.25–7.20 (m, 4H, H_{Ar}); 7.14 (m, 2H, H_{Ar}); 7.03–7.00 (m, 4H, H_{Ar}); 6.96 (s, 1H, H_{Ar}); 4.07 (s, 4H, CH₂); 2.24 (s, 6H, CH₃); 2.08 (s, 3H, CH₃). – ¹³C NMR: 140.23 (s), 136.34 (s), 135.13 (s), 134.75 (s), 129.96 (d), 128.33 (d), 127.82 (d), 125.62 (d), 35.46 (t), 20.27 (q), 16.21 (q). – MS (70eV): *m/z* (%) 300 (100), 285 (10), 209 (65), 179 (23), 165 (11), 91 (35).

Dimesitylmethane (8). According to GP 2, sulfamoylcarbamate **2** (0.69 g, 1.98 mmol) was heated in mesitylene (1.7 mL, 1.47 g, 12 mmol) at 125 °C.

FC (SiO₂, 3 x 20 cm, 0–20% EtOAc/light petroleum).

Yield of **8**: 0.10 g (20%). Colorless oil. ¹H NMR: 6.88 (s, 4H, H_{Ar}); 4.10 (s, 2H, CH₂); 2.35 (s, 6H, CH₃); 2.19 (s, 12H, CH₃). – ¹³C NMR: 136.65 (s), 134.87 (s), 134.78 (s), 129.29 (d), 30.98 (t), 20.76 (q), 20.69 (q). – MS (70eV): *m/z* (%) 252 (100), 237 (33), 132 (82).

1,3-Dichlor-4-(2,4,6-trimethylbenzyl)-benzene (9). According to GP 8, sulfamoylcarbamate **4** (1.00 g, 2.44 mmol) was heated in mesitylene (3 mL, 1.29 g, 22 mmol) at 130 °C.

FC (SiO₂, 3 x 30 cm, 6% EtOAc/light petroleum).

Yield of **9**: 0.60 g (88%). Brownish crystals. M.p. 93.7 °C. – ¹H NMR (DMSO-d₆): 7.39 (d, *J* = 1.5 Hz, 1H, H_{Ar}); 6.99 (dd, *J* = 8.3, 1.5 Hz, 1H, H_{Ar}); 6.69 (s, 2H, H_{Ar}); 6.48 (d, *J* = 8.3 Hz, 1H, H_{Ar}); 3.96 (s, 2H, CH₂); 2.29 (s, 3H, CH₃); 2.12 (s, 6H, CH₃). – ¹³C NMR (DMSO-d₆): 137.10 (s), 136.20 (s), 136.13 (s), 134.84 (s), 132.06 (s), 131.99 (s), 129.01 (d), 128.83 (d), 127.01 (d), 31.84 (t), 20.90 (q), 19.79 (q). – MS (70eV): *m/z* (%) 280 (63), 278 (100), 265 (32), 263 (74), 192 (22), 178 (15), 132 (32), 91 (15).

Methyl 2-[2-(2-methylbenzyl)-phenoxy]-acetate (*o*-10) and methyl 2-[2-(4-methylbenzyl)-phenoxy]-acetate (*p*-10). According to GP 2, sulfamoylcarbamate **3** (2.00 g, 4.67 mmol) was heated in toluene (5.0 mL, 4.32 g, 47 mmol) at 111 °C.

FC (SiO₂, 3 x 30 cm, 11% EtOAc/light petroleum).

Yield of *p*-**10** and *o*-**10**: 0.48 g (38%). Colorless oil. Ratio of *p*-**10** and *o*-**10** = 1 : 2.5.

o-**10**: ¹H NMR: 7.20–6.69 (m, 8H, H_{Ar}); 4.59 (s, 2H, CH₂O); 4.00 (s, 2H, CH₂); 3.77 (s, 3H, CH₃O); 2.29 (s, 3H, CH₃). – ¹³C NMR: 169.45 (s), 155.46 (s), 137.60 (s), 135.12 (s), 130.53 (d), 129.79 (s), 128.85 (d), 127.17 (d), 121.55 (d), 111.33 (d), 65.41 (t), 52.02 (q), 35.32 (t), 20.92 (q). – MS (70eV): *m/z* (%) 270 (100), 255 (4), 210 (13), 197 (85), 181 (76), 165 (29), 152 (12), 105 (21).

p-**10**: ¹H NMR: 4.63 (s, 2H, CH₂O); 4.03 (s, 2H, CH₂); 2.25 (s, 3H, CH₃). – ¹³C NMR: 155.56 (s), 138.41 (s), 137.00 (s), 130.56 (d), 130.00 (d), 129.97 (d), 129.27 (s), 127.04 (d), 126.13 (d), 125.78 (d), 121.53 (d), 110.06 (d), 65.38 (t), 32.95 (t), 19.44 (q).

Methyl 2-[2-(2,4-dimethylbenzyl)-phenoxy]-acetate (*o,p*-11) and methyl 2-[2-(2,6-dimethylbenzyl)-phenoxy]-acetate (*o,p*-11). According to GP 2, sulfamoylcarbamate **3** (2.00 g, 4.67 mmol) was heated in *m*-xylene (5.0 mL, 4.34 g, 41 mmol) at 138 °C.

FC (SiO₂, 3 x 30 cm, 11% EtOAc/light petroleum).

Yield of *o,p*-11 and *o,o*-11: 0.82 g (62%). Colorless oil.

Ratio of *o,p*-11 and *o,o*-11 = 6 : 1.

o,p-11: ¹H NMR: 7.14–6.50 (m, 7H, H_{Ar}); 4.61 (s, 2H, CH₂O); 3.99 (s, 2H, CH₂); 3.74 (s, 3H, CH₃O); 2.28 (s, 3H, CH₃); 2.21 (s, 3H, CH₃). – ¹³C NMR: 169.38 (s), 155.55 (s), 136.47 (s), 135.46 (s), 135.28 (s), 130.81 (d), 129.92 (d), 129.77 (d), 129.65 (s), 126.91 (d), 126.40 (d), 121.48 (d), 111.02 (d), 65.37 (t), 51.95 (q), 32.51 (t), 20.82 (q), 19.33 (q). – MS (70eV): *m/z* (%) 284 (100), 269 (7), 211 (54), 195 (73), 179 (52), 165 (20), 119 (16).

o,o-11: ¹H NMR: 4.70 (s, 2H, CH₂O); 4.06 (s, 2H, CH₂); 3.77 (s, 3H, CH₃O); 2.19 (s, 6H, CH₃). – ¹³C NMR: 155.61 (s), 137.33 (s), 136.37 (s), 128.54 (s), 127.88 (d), 127.76 (d), 126.63 (d), 126.09 (s), 121.55 (d), 110.56 (d), 65.30 (t), 52.01 (q), 28.61 (t), 19.85 (q).

Methyl 2-(2- -naphthylmethyl)-phenoxy-acetate (-12) and methyl 2-(2- -naphthylmethyl)-phenoxy-acetate (-12). According to GP 2, sulfamoylcarbamate **3** (2.00 g, 4.67 mmol) was heated in naphthalene (5.0 g, 39 mmol) at 130 °C.

FC (SiO₂, 3 x 30 cm, 14% EtOAc/light petroleum).

Yield of -12 and -12: 0.81 g (57%). Colorless oil. Ratio of -12 and -12 = 4 : 1.

-12: ¹H NMR: 8.01 (m, 1H, H_{Ar}); 7.78 (m, 1H, H_{Ar}); 7.68 (d, *J* = 7.7 Hz, 1H, H_{Ar}); 7.40–7.24 (m, 4H, H_{Ar}); 7.06 (m, 1H, H_{Ar}); 6.88–6.63 (m, 3H, H_{Ar}); 4.59 (s, 2H, CH₂O); 4.48 (s, 2H, CH₂); 3.66 (s, 3H, CH₂O). – ¹³C NMR: 169.29 (s), 155.24 (s), 136.26 (s), 133.68 (s), 132.16 (s), 130.22 (d), 129.47 (s), 128.39 (d), 127.10 (d), 127.05 (d), 126.78 (d), 125.70 (d), 125.40 (d), 125.31 (d), 124.31 (d), 121.46 (d), 110.97 (d), 65.25 (t), 51.88 (q), 32.36 (t).

-12: ¹H NMR: 4.50 (s, 2H, CH₂O); 4.18 (s, 2H, CH₂); 3.64 (s, 3H, CH₂O). – ¹³C NMR: 169.18 (s), 155.47 (s), 138.19 (s), 133.46 (s), 131.87 (s), 130.62 (d), 130.05 (s), 127.73 (d), 127.60 (d), 127.39 (d), 127.30 (d), 125.60 (d), 124.97 (d), 121.51 (d), 111.28 (d), 51.84 (q), 35.91 (t).

1-Benzyl-naphthalene (**-13) and 2-benzyl-naphthalene (**-13**).** Similarly to GP 2, sulfamoylcarbamate **1** (307 mg, 1.00 mmol) was heated with naphthalene (195 mg, 1.52 mmol) in octane (3 mL) at 127 °C.

FC (SiO₂, 3 x 20 cm, 14% EtOAc/hexane).

Yield of **-13** and **-13**: 23 mg (10%). Colorless oil. Ratio of **-13** and **-13** = 8 : 1.

-13: ¹H NMR: 7.99 (m, 1H, H_{Ar}); 7.85 (m, 1H, H_{Ar}); 7.75 (m, 1H, H_{Ar}); 7.45–7.38 (m, 3H, H_{Ar}); 7.29–7.17 (m, 6H, H_{Ar}); 4.44 (s, 2H, CH₂). – MS (70eV): *m/z* (%) 218 (100), 202 (32), 189 (9), 141 (24), 115 (15).

-13: ¹H NMR: 4.13 (s, 2H, CH₂).

Diphenylmethane 15. Similarly to GP 2, sulfamoylcarbamate **1** (0.61 g, 2.0 mmol) was heated in benzene (5.0 mL, 4.37 g, 56 mmol) at 120 °C in a closed vessel.

FC (SiO₂, 2 x 22 cm, 0–20% EtOAc/hexane).

Yield of **15**: 87 mg (25%). Colorless oil. ¹H NMR: 7.42–7.30 (m, 10H, H_{Ar}); 4.10 (s, 2H, CH₂). – ¹³C NMR: 141.08 (s), 128.83 (d), 128.42 (d), 126.03 (d), 41.91 (t). – MS (70eV): *m/z* (%) 167 (100), 149 (70).

3-Benzyl-1H-indole 16. According to GP 2, sulfamoylcarbamate **1** (0.31 g, 1.0 mmol) was heated in indole (0.35 g, 3.0 mmol) at 125 °C.

FC (SiO₂, 2 x 25 cm, 25–100% EtOAc/hexane).

Yield of **16**: 50 mg (25%). Colorless oil. ¹H NMR: 7.49 (d, *J* = 7.9 Hz, 1H, H_{Ar}); 6.70 (br. s, 1H, H_{Ar}); 4.05 (s, 2H, CH₂). – MS (70eV): *m/z* (%) 207 (100), 206 (37), 130 (34).

2-Benzyl-4-bromoanisole (17). According to GP 2, sulfamoylcarbamate **1** (0.68 g, 2.2 mmol) was heated in 4-bromoanisole (2.0 mL, 3.0 g, 16 mmol) at 128 °C.

FC (SiO₂, 3 x 20 cm, 0–1% Et₂OAc/light petroleum).

Yield of **17**: 0.25 g (41%). Colorless oil. ¹H NMR: 7.23–7.14 (m, 7H, H_{Ar}); 6.65 (d, *J* = 8.6 Hz, 1H, H_{Ar}); 3.89 (s, 2H, CH₂); 3.71 (s, 3H, CH₃O). – ¹³C NMR: 156.35 (s), 139.98 (s), 132.78 (d), 131.93 (s), 129.94 (d), 128.83 (d), 128.32 (d), 126.00 (d), 112.63 (s), 111.98 (d), 55.44 (q), 35.56 (t). – MS (70eV): *m/z* (%) 278 (99), 276 (100), 263 (10), 261 (11), 197 (50), 182 (24), 181 (28), 165 (41), 152 (27).

2-Benzyl-4-chlorophenol (18) and 4-Chlorophenylbenzylether (21). According to GP 2, sulfamoylcarbamate **1** (0.61 g, 2.0 mmol) was heated in 4-chlorophenol (2.0 mL, 2.61 g, 20 mmol) at 120 °C.

FC (SiO₂, 2 x 22 cm, 20–50% EtOAc/hexane).

Yield of **18**: 83 mg (19%). Yield of **21**: 110 mg (25%). Colorless oils.

18: ¹H NMR: 7.32–7.11 (m, 7H, H_{Ar}); 6.68 (d, *J* = 8.9 Hz, 1H, H_{Ar}); 3.92 (s, 2H, CH₂). – ¹³C NMR: 152.24 (s), 139.02 (s), 133.02 (s), 130.47 (d), 129.00 (s), 128.69 (d), 128.66 (d), 127.43 (d), 126.53 (d), 116.60 (d), 36.04 (t). – MS (70eV): *m/z* (%) 220 (34), 218 (100), 183 (34), 165 (19), 152 (21).

21: ¹H NMR: 7.41–7.28 (m, 5H, H_{Ar}); 7.21/6.87 (AA'BB', *J* = 8.9 Hz, 2H, H_{Ar}); 5.00 (s, 2H, CH₂O). – ¹³C NMR (75 MHz, CDCl₃): = 157.30 (s), 136.54 (s), 129.30 (d), 128.59 (d), 128.05 (d), 127.40 (d), 125.75 (s), 116.11 (d), 70.20 (t).

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