Supplementary Information

Palladium-Catalyzed Decarboxylative Acylation of *O*-Methyl Ketoximes with α-Keto Acids

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General methods

Commercially available reagents were used without additional purification, unless otherwise stated. Sealed tubes (13 x100 mm²) were purchased from Fischer Scientific and dried in oven for overnight and cooled at room temperature prior to use. Thin layer chromatography was carried out using plates coated with Kieselgel $60F_{254}$ (Merck). For flash column chromatography, E. Merck Kieselgel 60 (230-400 mesh) was used. Nuclear magnetic resonance spectra (¹H and ¹³C NMR) were recorded on a Bruker Unity 400, 500 and 700 MHz spectrometers for CDCl₃ solutions and chemical shifts are reported as parts per million (ppm) relative to, respectively, residual CHCl₃ $\delta_{\rm H}$ (7.24 ppm) and CDCl₃ $\delta_{\rm C}$ (77.0 ppm) as internal standards. Resonance patterns are reported with the notations s (singlet), d (doublet), t (triplet), q (quartet), and m (multiplet). In addition, the notation br is used to indicate a broad signal. Coupling constants (*J*) are reported in hertz (Hz). IR spectra were recorded on a Varian 2000 Infrared spectrophotometer and are reported as cm⁻¹. High-resolution mass spectra (HRMS) were recorded on a JEOL JMS-600 spectrometer.

General procedure for the synthesis of O-methyl ketoximes

O-Methyl ketoximes were prepared from the corresponding methyl ketones and methoxyl amine hydrochloride according to the reported procedure.¹

General procedure for the synthesis of O-methyl aldoximes

A solution of methoxylamine hydrochloride (1.34 g, 0.016 mol) in a mixture of water (15 mL) and THF (5 mL) was treated with sodium acetate (1.12 g, 0.014 mol) followed by the aldehydes (9.32 mmol, 1 equiv.) and the resulting mixture was stirred at room temperature for 4 h. The reaction mixture was then diluted with EtOAc, washed with brine, dried over Mg₂SO₄ and concentrated in vacuo. The residue was purified by flash column chromatography (*n*-hexanes/EtOAc) to afford *O*-methyl aldoximes **5a** and **5b**.

General procedure for the synthesis of a-keto acids

 α -Keto acids were prepared from the oxidation of corresponding aryl methyl ketones with SeO₂ and pyridine according to the reported procedure.²

⁽¹⁾ Tsai, A. S.; Brasse, M.; Bergman, R. G.; Ellman, J. A. Org. Lett. 2011, 13, 540.

⁽²⁾ Wadhwa, K.; Yang, C.-X.; West, P. R.; Deming, K. C.; Chemburkar, S. R.; Reddy, R. E. *Synth. Commun.* **2008**, *38*, 4434.

Typical procedure for the acylation of *O*-methyl ketoximes or *O*-methyl aldoximes

To an oven-dried sealed tube charged with 4-fluoroacetophenone *O*-methyl oxime (**1a**) (50.2 mg, 0.3 mmol, 1.0 equiv.), $Pd(OAc)_2$ (6.7 mg, 0.03 mmol, 10 mol %), and $(NH_4)_2S_2O_8$ (102.6 mg, 0.45 mmol, 1.5 equiv.) in diglyme (1 mL) was added phenylglyoxylic acid (**2a**) (67.6 mg, 0.45 mmol, 1.5 equiv.). The reaction mixture was allowed to stir at 70 °C for 3 h, and cooled to room temperature. The reaction mixture was diluted with EtOAc (3 mL) and concentrated in vacuo. The residue was purified by flash column chromatography (*n*-hexanes/EtOAc) to afford 58.9 mg of the acylated product **3a** in 72% yield.

Characterization data for all products (3a-k, 4b-m, 6a and 6b)

(5-Fluoro-2-(1-(methoxyimino)ethyl)phenyl)(phenyl)methanone (3a)



 $R_{\rm f} = 0.43$ (*n*-hexanes/EtOAc = 6:1); ¹H NMR (400 MHz, CDCl₃) δ 7.72 (d, J = 7.6 Hz, 2H), 7.56-7.42 (m, 4H), 7.25-7.19 (m, 2H), 3.68 (s, 3H), 2.04 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 195.9, 162.5 (d, $J_{\rm C-F} = 249.5$ Hz), 152.9, 140.9 (d, $J_{\rm C-F} = 6.4$ Hz), 137.5, 132.6 (d, $J_{\rm C-F} = 3.9$ Hz), 142.4, 129.6 (d, $J_{\rm C-F} = 8.0$ Hz), 129.2, 128.3, 117.0 (d, $J_{\rm C-F} = 21.5$ Hz), 116.1 (d, $J_{\rm C-F} = 22.4$ Hz), 61.7, 14.3; IR (KBr) υ 2937, 1671, 1600, 1578, 1492, 1408, 1369, 1318, 1274, 1177, 1069, 976, 828 cm⁻¹; HRMS (EI) Calcd for C₁₆H₁₄FNO₂ [M]⁺ 271.1009, found 271.1011.

(5-Chloro-2-(1-(methoxyimino)ethyl)phenyl)(phenyl)methanone (3b)



 $R_{\rm f} = 0.50$ (*n*-hexanes/EtOAc = 6:1); ¹H NMR (400 MHz, CDCl₃) δ 7.67 (d, J = 7.8 Hz, 2H), 7.51-7.37 (m, 6H), 3.62 (s, 3H), 1.99 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 195.9, 152.7, 140.4, 137.6, 134.8, 134.6, 132.8, 130.1, 129.2, 128.9, 128.8, 128.3, 61.7, 14.1; IR (KBr) υ 2936, 1671, 1594, 1479, 1449, 1369, 1315, 1283, 1179, 1105, 1048, 827 cm⁻¹; HRMS (EI) Calcd for C₁₆H₁₄CINO₂ [M]⁺ 287.0713, found 287.0713.

(2-(1-(Methoxyimino)ethyl)-5-(trifluoromethyl)phenyl)(phenyl)methanone (3c)



 $R_{\rm f} = 0.44$ (*n*-hexanes/EtOAc = 6:1); ¹H NMR (400 MHz, CDCl₃) δ 7.76 (d, J = 8.2 Hz, 1H), 7.70-7.67 (m, 3H), 7.61 (d, J = 8.2 Hz, 1H), 7.53-7.51 (m, 1H), 7.42-7.38 (m, 2H), 3.65 (s, 3H), 2.03 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 196.0, 152.6, 139.5, 137.4, 132.9, 130.5 (q, $J_{\rm C}$ - $_{\rm F} = 32.8$ Hz), 129.2, 128.4, 128.1, 126.8, 125.7, 124.9, 122.2 (q, $J_{\rm C-F} = 290.2$ Hz), 61.9, 14.1; IR (KBr) υ 2939, 1673, 1450, 1338, 1268, 1175, 1130, 1092, 1048, 840 cm⁻¹; HRMS (EI) Calcd for C₁₇H₁₄F₃NO₂ [M]⁺ 321.0977, found 321.0972.

(2-(1-(Methoxyimino)ethyl)phenyl)(phenyl)methanone (3d)



 $R_{\rm f} = 0.44$ (*n*-hexanes/EtOAc = 6:1); ¹H NMR (400 MHz, CDCl₃) δ 7.67 (d, J = 7.6 Hz, 2H), 7.36-7.51 (m, 7H), 3.66 (s, 3H), 2.01 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 197.5, 154.0, 138.9, 138.2, 136.4, 132.5, 130.1, 129.3, 129.0, 128.5, 128.2, 127.6, 61.6, 14.4; IR (KBr) υ 2935, 1668, 1597, 1449, 1367, 1314, 1286, 1154, 1048, 928 cm⁻¹; HRMS (EI) Calcd for C₁₆H₁₅NO₂ [M]⁺ 253.1103, found 253.1103.

(5-Methoxy-2-(1-(methoxyimino)ethyl)phenyl)(phenyl)methanone (3e)



 $R_{\rm f} = 0.30$ (*n*-hexanes/EtOAc = 6:1); ¹H NMR (400 MHz, CDCl₃) δ 7.69 (d, J = 7.6 Hz, 2H), 7.47-7.51 (m, 1H), 7.35-7.42 (m, 3H), 7.02 (d, J = 8.6 Hz, 1H), 6.97 (s, 1H), 3.83 (s, 3H), 3.61 (s, 3H), 1.97 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 197.2, 159.8, 153.3, 140.3, 138.1, 132.5, 129.2, 128.9, 128.6, 128.2, 115.9, 114.0, 61.5, 55.5, 14.1; IR (KBr) υ 2937, 1669, 1601, 1497, 1414, 1367, 1288, 1177, 1122, 1040, 896 cm⁻¹; HRMS (EI) Calcd for C₁₇H₁₇NO₃ [M]⁺ 283.1208, found 283.1212.

(3-(1-(Methoxyimino)ethyl)naphthalen-2-yl)(phenyl)methanone (3f)



 $R_{\rm f} = 0.40$ (*n*-hexanes/EtOAc = 6:1); ¹H NMR (400 MHz, CDCl₃) δ 7.85-7.95 (m, 3H), 7.75 (d, J = 7.6 Hz, 2H), 7.50-7.59 (m, 4H), 7.37-7.41 (m, 2H), 3.69 (s, 3H), 2.13 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 197.2, 153.8, 138.4, 136.6, 133.6, 133.5, 132.5, 132.4, 129.4, 129.3, 128.4, 128.2, 128.1, 127.8, 127.5, 127.4, 61.7, 14.2; IR (KBr) υ 2935, 1742, 1681, 1597, 1451, 1365, 1282, 1193, 1053, 872 cm⁻¹; HRMS (EI) Calcd for C₂₀H₁₇NO₂ [M]⁺ 303.1259, found 303.1261.

(4-Fluoro-2-(1-(methoxyimino)ethyl)phenyl)(phenyl)methanone (3g)



 $R_{\rm f} = 0.50$ (*n*-hexanes/EtOAc = 6:1); ¹H NMR (400 MHz, CDCl₃) δ 7.61 (d, J = 7.8 Hz, 2H), 7.46-7.39 (m, 2H), 7.33 (t, J = 7.4 Hz, 2H), 7.19-7.08 (m, 2H), 3.62 (s, 3H), 1.93 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 196.4, 163.4 (d, $J_{\rm C-F} = 248.7$ Hz), 153.1, 139.2 (d, $J_{\rm C-F} = 248.7$ Hz), 153.1, 153.1, 153.1, 153.1, 153.1, 153.1

8.0 Hz), 138.1, 134.9 (d, $J_{C-F} = 3.2$ Hz), 132.6, 131.3 (d, $J_{C-F} = 8.7$ Hz), 129.2, 128.3, 115.4 (d, $J_{C-F} = 21.3$ Hz), 114.9 (d, $J_{C-F} = 23.0$ Hz), 61.8, 14.3; IR (KBr) v 2937, 1669, 1604, 1448, 1369, 1283, 1203, 1148, 1047, 874 cm⁻¹; HRMS (EI) Calcd for $C_{16}H_{14}FO_2$ [M]⁺ 271.1009, found 271.1002.

(2-Fluoro-6-(1-(methoxyimino)ethyl)phenyl)(phenyl)methanone (3gg)



 $R_{\rm f} = 0.37$ (*n*-hexanes/EtOAc = 6:1); ¹H NMR (400 MHz, CDCl₃) δ 7.73 (d, J = 7.6 Hz, 2H), 7.33-7.47 (m, 4H), 7.24 (d, J = 7.8 Hz, 1H), 7.06-7.11 (m, 1H), 3.49 (s, 3H), 2.01 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 193.1, 160.9, 158.4 (d, $J_{\rm C-F} = 246.4$ Hz), 138.0, 137.5, 137.4 (d, $J_{\rm C-F} = 4.1$ Hz), 133.0, 132.9 (d, $J_{\rm C-F} = 2.8$ Hz), 130.7, 130.6 (d, $J_{\rm C-F} = 8.5$ Hz), 130.2, 129.0, 128.4, 123.3, 123.2 (d, $J_{\rm C-F} = 3.1$ Hz), 116.3, 116.1 (d, $J_{\rm C-F} = 22.0$ Hz), 61.6, 13.6; IR (KBr) v 2937, 1677, 1605, 1567, 1448, 1369, 1317, 1268, 1144, 1049, 945, 877 cm⁻¹; HRMS (EI) Calcd for C₁₆H₁₄FO₂ [M]⁺ 271.1009, found 271.1008.

(3-Fluoro-2-(1-(methoxyimino)ethyl)phenyl)(phenyl)methanone (3h)



 $R_{\rm f} = 0.47$ (*n*-hexanes/EtOAc = 6:1); ¹H NMR (400 MHz, CDCl₃) δ 7.73 (d, J = 7.7 Hz, 2H), 7.55-7.59 (m, 1H), 7.43-7.49 (m, 3H), 7.25-7.32 (m, 2H) 3.70 (s, 3H), 2.06 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 196.1, 196.0 (d, $J_{\rm C-F} = 2.7$ Hz), 161.9, 159.4 (d, $J_{\rm C-F} = 249.2$ Hz), 151.7, 141.7, 141.6 (d, $J_{\rm C-F} = 2.9$ Hz), 137.9, 132.8, 130.3, 130.0, 129.9 (d, $J_{\rm C-F} = 8.6$ Hz), 129.4,

128.3, 124.5, 124.4 (d, $J_{C-F} = 3.2 \text{ Hz}$), 118.0, 117.8 (d, $J_{C-F} = 22.0 \text{ Hz}$), 61.8, 16.1, 16.0 (d, $J_{C-F} = 4.3 \text{ Hz}$); IR (KBr) v 2937, 1671, 1599, 1451, 1367, 1315, 1284, 1177, 1059, 852 cm⁻¹; HRMS (EI) Calcd for C₁₆H₁₄FO₂ [M]⁺ 271.1009, found 271.1010.

(8-(Methoxyimino)-5,6,7,8-tetrahydronaphthalen-1-yl)(phenyl)methanone (3i)



 $R_{\rm f} = 0.61$ (*n*-hexanes/EtOAc = 6:1); ¹H NMR (500 MHz, CDCl₃) δ 7.71 (d, J = 7.8 Hz, 2H), 7.47 (t, J = 7.4 Hz, 1H), 7.39-7.33 (m, 3H), 7.27 (d, J = 8.7 Hz, 1H), 7.20 (d, J = 7.4 Hz, 1H), 3.54 (s, 3H), 2.81-2.78 (m, 2H), 2.59-2.56 (m, 2H), 1.88-1.84 (m, 2H); ¹³C NMR (125 MHz, CDCl₃) δ 198.1, 151.9, 140.8, 138.9, 138.5, 132.2, 129.8, 129.1, 129.0, 128.6, 128.3, 126.5, 61.8, 30.6, 24.4, 21.3; IR (KBr) υ 2932, 1666, 1596, 1446, 1313, 1280, 1166, 1040, 933, 808 cm⁻¹; HRMS (EI) Calcd for C₁₈H₁₇NO₂ [M]⁺ 279.1259, found 279.1257.

(3-Methoxy-8-(methoxyimino)-5,6,7,8-tetrahydronaphthalen-1-yl)(phenyl)methanone (3j)



 $R_{\rm f} = 0.34$ (*n*-hexanes/EtOAc = 6:1); ¹H NMR (400 MHz, CDCl₃) δ 7.70 (d, J = 7.8 Hz, 2H), 7.43-7.47 (m, 1H), 7.32-7.36 (m, 2H), 7.74 (d, J = 16.32 Hz, 2H), 3.79 (s, 3H), 3.48 (s, 3H), 2.72-2.75 (m, 2H), 2.50-2.53 (m, 2H), 1.79-1.84 (m, 2H); ¹³C NMR (100 MHz, CDCl₃) δ 197.3, 159.5, 151.4, 142.3, 140.2, 138.1, 132.0, 128.9, 128.1, 121.5, 114.6, 112.1, 61.3, 55.4, 30.6, 24.0, 21.1; IR (KBr) v 2936, 1671, 1595, 1470, 1354, 1289, 1214, 1175, 1087, 1013, 879 cm⁻¹; HRMS (EI) Calcd for C₁₉H₁₉NO₃ [M]⁺ 309.1365, found 309.1370.

(4-(Methoxyimino)chroman-5-yl)(phenyl)methanone (3k)



 $R_{\rm f} = 0.39$ (*n*-hexanes/EtOAc = 6:1); ¹H NMR (400 MHz, CDCl₃) δ 7.73 (d, J = 7.8 Hz, 2H), 7.48-7.29 (m, 4H), 6.99 (d, J = 8.2 Hz, 1H), 6.87 (d, J = 7.4 Hz, 1H), 4.19 (t, J = 6.2 Hz, 2H), 3.49 (s, 3H), 2.77 (t, J = 6.2 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃) δ 197.2, 156.8, 145.9, 138.8, 137.9, 132.3, 130.3, 129.0, 128.2, 120.9, 118.7, 116.2, 74.9, 71.7, 23.9; IR (KBr) υ 2937, 1673, 1595, 1469, 1318, 1279, 1145, 1079, 945, 850 cm⁻¹; HRMS (EI) Calcd for C₁₇H₁₅NO₃ [M]⁺ 281.1052, found 281.1049.

(8-(Methoxyimino)-5,6,7,8-tetrahydronaphthalen-1-yl)(4-methoxyphenyl)methanone (4b)



 $R_{\rm f} = 0.23$ (*n*-hexanes/EtOAc = 6:1); ¹H NMR (400 MHz, CDCl₃) δ 7.70 (d, J = 8.2 Hz, 2H), 7.36-7.25 (m, 2H), 7.18 (d, J = 7.4 Hz, 1H), 6.87 (d, J = 8.2 Hz, 2H), 3.85 (s, 3H), 3.57 (s, 3H), 2.82-2.79 (m, 2H), 2.62-2.59 (m, 2H), 1.90-1.83 (m, 2H); ¹³C NMR (175 MHz, CDCl₃) δ 196.9, 162.7, 151.7, 140.5, 138.9, 131.3, 131.1, 129.3, 128.6, 128.3, 126.2, 113.3, 61.6, 55.4, 30.4, 24.2, 21.1; IR (KBr) v 2937, 1663, 1578, 1460, 1419, 1311, 1256, 1169, 1049, 880 cm⁻¹; HRMS (EI) Calcd for C₁₉H₁₉NO₃ [M]⁺ 309.1365, found 309.1379.

(8-(Methoxyimino)-5,6,7,8-tetrahydronaphthalen-1-yl)(4-(trifluoromethyl)phenyl)methanone (4c)



 $R_{\rm f} = 0.41$ (*n*-hexanes/EtOAc = 6:1); ¹H NMR (400 MHz, CDCl₃) δ 7.79 (d, J = 8.1 Hz, 2H), 7.61 (d, J = 8.1 Hz, 2H), 7.37-7.27 (m, 2H), 7.17 (d, J = 7.4 Hz, 1H), 3.51 (s, 3H), 2.79 (t, J = 5.9 Hz, 2H), 2.55 (t, J = 5.9 Hz, 2H), 1.97-1.80 (m, 2H); ¹³C NMR (175 MHz, CDCl₃) δ 196.5, 151.8, 141.2, 140.7, 137.7, 133.5 (q, $J_{\rm C-F} = 32.2$ Hz), 130.1, 129.0, 128.7, 128.6, 126.3, 125.2 (q, $J_{\rm C-F} = 3.5$ Hz), 123.7 (q, $J_{\rm C-F} = 272.2$ Hz), 61.6, 30.3, 24.2, 20.9; IR (KBr) υ 2939, 1677, 1584, 1460, 1326, 1279, 1180, 1108, 1051, 857 cm⁻¹; HRMS (EI) Calcd for C₁₉H₁₆F₃NO₂ [M]⁺ 347.1133, found 347.1134.

(4-Fluorophenyl)(8-(methoxyimino)-5,6,7,8-tetrahydronaphthalen-1-yl)methanone (4d)



 $R_{\rm f} = 0.38$ (*n*-hexanes/EtOAc = 6:1); ¹H NMR (400 MHz, CDCl₃) δ 7.66-7.63 (m, 2H), 7.25 (d, J = 7.4 Hz, 1H), 7.19 (d, J = 7.2 Hz, 1H), 7.09 (d, J = 7.4 Hz, 1H), 6.96 (t, J = 8.4 Hz, 2H), 3.46 (s, 3H), 2.71 (t, J = 5.9 Hz, 2H), 2.50 (t, J = 6.6 Hz, 2H), 1.80-1.73 (m, 2H); ¹³C NMR (100 MHz, CDCl₃) δ 196.3, 165.0 (d, $J_{\rm C-F} = 251.6$ Hz), 151.7, 140.6, 138.3, 134.8 (d, $J_{\rm C-F} = 2.9$ Hz), 131.4 (d, $J_{\rm C-F} = 9.1$ Hz), 129.7, 128.6, 128.4, 126.2, 115.2 (d, $J_{\rm C-F} = 21.7$ Hz), 61.5, 30.3, 24.2, 21.0; IR (KBr) υ 2938, 1672, 1597, 1460, 1280, 1192, 1050, 1009, 850 cm⁻¹; HRMS (EI) Calcd for C₁₈H₁₆FNO₂ [M]⁺ 297.1165, found 297.1162.

(4-Chlorophenyl)(8-(methoxyimino)-5,6,7,8-tetrahydronaphthalen-1-yl)methanone (4e)



 $R_{\rm f} = 0.61$ (*n*-hexanes/EtOAc = 6:1); ¹H NMR (400 MHz, CDCl₃) δ 7.67 (d, J = 7.8 Hz, 2H), 7.38-7.30 (m, 4H), 7.19 (d, J = 7.4 Hz, 1H), 3.58 (s, 3H), 2.83-2.80 (m, 2H), 2.60 (t, J = 6.6 Hz, 2H), 1.89-1.85 (m, 2H); ¹³C NMR (100 MHz, CDCl₃) δ 196.6, 151.7, 140.6, 138.3, 138.1, 136.7, 130.2, 129.8, 128.7, 128.5, 128.4, 126.2, 71.6, 30.3, 24.2, 21.0; IR (KBr) υ 2937, 1673, 1587, 1459, 1399, 1281, 1171, 1050, 1008, 845 cm⁻¹; HRMS (EI) Calcd for C₁₈H₁₆CINO₂ [M]⁺ 313.0870, found 313.0874.

(4-Bromophenyl)(8-(methoxyimino)-5,6,7,8-tetrahydronaphthalen-1-yl)methanone (4f)



 $R_{\rm f} = 0.61$ (*n*-hexanes/EtOAc = 6:1); ¹H NMR (400 MHz, CDCl₃) δ 7.60-7.52 (m, 4H), 7.37-7.29 (m, 2H), 7.19 (d, J = 7.4 Hz, 1H), 3.58 (s, 3H), 2.83-2.80 (m, 2H), 2.60 (t, J = 6.6 Hz, 2H), 1.89-1.86 (m, 2H); ¹³C NMR (100 MHz, CDCl₃) δ 196.8, 151.7, 140.7, 138.0, 137.2, 131.4, 130.4, 129.8, 128.7, 128.5, 127.0, 126.2, 71.6, 30.3, 24.2, 21.0; IR (KBr) υ 2936, 1673, 1586, 1459, 1352, 1280, 1172, 1105, 1050, 842 cm⁻¹; HRMS (EI) Calcd for C₁₈H₁₆BrNO₂ [M]⁺ 357.0364, found 357.0363.

(8-(Methoxyimino)-5,6,7,8-tetrahydronaphthalen-1-yl)(m-tolyl)methanone (4g)



 $R_{\rm f} = 0.39$ (*n*-hexanes/EtOAc = 6:1); ¹H NMR (400 MHz, CDCl₃) δ 7.60 (s, 1H), 7.48 (d, J = 7.4 Hz, 1H), 7.39-7.21 (m, 5H), 3.58 (s, 3H), 2.83-2.80 (m, 2H), 2.59 (t, J = 6.6 Hz, 2H), 2.37 (s, 3H), 1.89-1.86 (m, 2H); ¹³C NMR (100 MHz, CDCl₃) δ 198.1, 151.7, 140.5, 138.8, 138.3, 137.8, 132.8, 129.5, 129.4, 128.8, 128.3, 127.9, 126.4, 126.3, 61.5, 30.3, 24.2, 21.3, 21.1; IR (KBr) υ 2937, 1670, 1585, 1458, 1283, 1143, 1050, 860 cm⁻¹; HRMS (EI) Calcd for C₁₉H₁₉NO₂ [M]⁺ 293.1416, found 293.1418.

(8-(Methoxyimino)-5,6,7,8-tetrahydronaphthalen-1-yl)(3-nitrophenyl)methanone (4h)



 $R_{\rm f} = 0.31$ (*n*-hexanes/EtOAc = 6:1); ¹H NMR (400 MHz, CDCl₃) δ 8.37 (s, 1H), 8.25 (d, J = 8.2 Hz, 1H), 8.01 (d, J = 8.2 Hz, 1H), 7.51 (t, J = 7.9 Hz, 1H), 7.35-7.26 (m, 2H), 7.18-7.15 (m, 1H), 3.45 (s, 3H), 2.77-2.74 (m, 2H), 2.48 (t, J = 6.6 Hz, 2H), 1.83-1.77 (m, 2H); ¹³C NMR (100 MHz, CDCl₃) δ 195.1, 152.0, 148.3, 141.0, 140.0, 137.0, 134.1, 130.5, 129.4, 128.9, 128.8, 126.5, 126.3, 123.6, 61.6, 30.2, 24.3, 20.9; IR (KBr) υ 2937, 1679, 1583, 1502, 1437, 1350, 1192, 1086, 1049, 887 cm⁻¹; HRMS (EI) Calcd for C₁₈H₁₆N₂O₄ [M]⁺ 324.1110, found 324.1118.

(3-Fluorophenyl)(8-(methoxyimino)-5,6,7,8-tetrahydronaphthalen-1-yl)methanone (4i)



 $R_{\rm f} = 0.61$ (*n*-hexanes/EtOAc = 6:1); ¹H NMR (400 MHz, CDCl₃) δ 7.38-7.36 (m, 2H), 7.28-7.22 (m, 3H), 7.11 (d, *J* = 7.8 Hz, 2H), 3.49 (s, 3H), 2.72 (t, *J* = 5.9 Hz, 2H), 2.50 (t, *J* = 6.6 Hz, 2H), 1.80-1.76 (m, 2H); ¹³C NMR (100 MHz, CDCl₃) δ 196.4, 162.6 (d, *J*_{C-F} = 245.5 Hz), 151.7, 140.6 (d, *J*_{C-F} = 5.9 Hz), 137.9, 129.9, 129.7, 128.7, 128.5, 126.3, 124.6 (d, *J*_{C-F} = 2.8 Hz), 119.0 (d, *J*_{C-F} = 21.3 Hz), 115.4 (d, *J*_{C-F} = 22.0 Hz), 61.6, 30.3, 24.2, 21.0; IR (KBr) υ 2937, 1675, 1609, 1588, 1442, 1284, 1172, 1131, 1050, 862 cm⁻¹; HRMS (EI) Calcd for C₁₈H₁₆FNO₂ [M]⁺ 297.1165, found 297.1162.

(8-(Methoxyimino)-5,6,7,8-tetrahydronaphthalen-1-yl)(naphthalen-2-yl)methanone (4j)



 $R_{\rm f} = 0.38$ (*n*-hexanes/EtOAc = 6:1); ¹H NMR (400 MHz, CDCl₃) δ 8.06 (s, 1H), 8.00 (d, J = 8.6 Hz, 1H), 7.89-7.82 (m, 3H), 7.58 (t, J = 7.0 Hz, 1H), 7.50 (t, J = 7.0 Hz, 1H), 7.41 (t, J = 7.4 Hz, 1H), 7.35-7.28 (m, 2H), 3.53 (s, 3H), 2.87-2.84 (m, 2H), 2.58 (t, J = 6.6 Hz, 2H), 1.90-1.87 (m, 2H); ¹³C NMR (100 MHz, CDCl₃) δ 197.9, 151.7, 140.6, 138.7, 135.8, 135.2, 132.5, 130.2, 129.7, 129.4, 128.9, 128.4, 128.0, 127.9, 127.7, 126.5, 126.4, 125.0, 61.6, 30.4, 24.2, 21.1; IR (KBr) v 2936, 1666, 1467, 1353, 1290, 1177, 1121, 1050, 866 cm⁻¹; HRMS (EI) Calcd for C₂₂H₁₉NO₂ [M]⁺ 329.1416, found 329.1417.

(8-(Methoxyimino)-5,6,7,8-tetrahydronaphthalen-1-yl)(naphthalen-1-yl)methanone (4k)



 $R_{\rm f} = 0.36$ (*n*-hexanes/EtOAc = 6:1); ¹H NMR (400 MHz, CDCl₃) δ 9.22 (d, J = 8.6 Hz, 1H), 7.89-7.96 (m, 2H), 7.67-7.71 (m, 1H), 7.56-7.59 (m, 1H), 7.28-7.48 (m, 5H), 3.35 (s, 3H), 2.78-2.81 (m, 2H), 2.46-2.50 (m, 2H), 1.79-1.85 (m, 2H); ¹³C NMR (100 MHz, CDCl₃) δ 199.3, 152.8, 141.0, 140.2, 135.5, 133.9, 132.7, 131.4, 129.7, 129.6, 129.5, 128.3, 128.2, 127.7, 127.6, 127.1, 126.2, 123.9, 61.6, 30.4, 24.4, 21.1; IR (KBr) υ 2937, 1646, 1591, 1437, 1309, 1249, 1152, 1074, 980, 883 cm⁻¹; HRMS (EI) Calcd for C₂₂H₁₉NO₂ [M]⁺ 329.1416, found 329.1415.

(2-Chlorophenyl)(8-(methoxyimino)-5,6,7,8-tetrahydronaphthalen-1-yl)methanone (4l)



 $R_{\rm f} = 0.33$ (*n*-hexanes/EtOAc = 6:1); ¹H NMR (400 MHz, CDCl₃) δ 7.48 (d, J = 7.8 Hz, 1H), 7.81-7.42 (m, 5H), 7.20-7.23 (m, 1H), 3.77 (s, 3H), 2.74-2.77 (m, 2H), 2.52-2.55 (m, 2H), 1.81-1.86 (m, 2H); ¹³C NMR (125 MHz, CDCl₃) δ 195.6, 152.7, 140.9, 139.1, 137.0, 133.3, 131.9, 131.8, 131.2, 130.0, 129.7, 128.5, 127.6, 126.0, 61.9, 30.3, 24.4, 21.1; IR (KBr) υ 2937, 1676, 1587, 1472, 1351, 1294, 1160, 1051, 1004, 824 cm⁻¹; HRMS (EI) Calcd for C₁₈H₁₆ClNO₂ [M]⁺ 313.0865, found 313.0874.

(8-(Methoxyimino)-5,6,7,8-tetrahydronaphthalen-1-yl)(thiophen-2-yl)methanone (4m)



 $R_{\rm f} = 0.37$ (*n*-hexanes/EtOAc = 6:1); ¹H NMR (400 MHz, CDCl₃) δ 7.59 (d, J = 4.8 Hz, 1H), 7.22-7.36 (m, 4H), 7.01-7.03 (m, 1H), 3.68 (s, 3H), 2.79-2.82 (m, 2H), 2.63-2.66 (m, 2H), 1.86-1.91 (m, 2H); ¹³C NMR (100 MHz, CDCl₃) δ 190.4, 151.6, 145.8, 140.8, 138.3, 132.6,

132.5, 129.7, 128.6, 128.2, 127.5, 126.1, 61.7, 30.3, 24.3, 21.0; IR (KBr) υ 2936, 1652, 1459, 1355, 1289, 1155, 1048, 854 cm⁻¹; HRMS (EI) Calcd for C₁₆H₁₅NO₂S [M]⁺ 285.0824, found 285.0829.

2-Benzoylbenzaldehyde O-methyl oxime (6a)



 $R_{\rm f} = 0.41$ (*n*-hexanes/EtOAc = 6:1); ¹H NMR (400 MHz, CDCl₃) δ 8.17 (s, 1H), 7.93 (d, J = 7.8 Hz, 1H), 7.80 (d, J = 7.7 Hz, 2H), 7.47-7.61 (m, 6H), 3.88 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 197.0, 146.3, 138.3, 137.5, 133.3, 131.0, 130.5, 130.1, 129.0, 128.9, 128.5, 127.2, 62.0; IR (KBr) υ 2937, 1663, 1597, 1448, 1315, 1268, 1180, 1054, 1001, 846 cm⁻¹; HRMS (EI) Calcd for C₁₅H₁₃NO₂ [M]⁺ 239.0946, found 239.0943.

Methyl 3-benzoyl-4-((methoxyimino)methyl)benzoate (6b)



 $R_{\rm f} = 0.28$ (*n*-hexanes/EtOAc = 3:1); ¹H NMR (400 MHz, CDCl₃) δ 8.13-8.14 (m, 1H), 8.05, (s, 1H), 7.98 (d, J = 8.2 Hz, 1H), 7.75 (d, J = 7.8 Hz, 1H), 7.56-7.60 (m, 1H), 7.42-7.46 (m, 2H), 3.89 (s, 3H), 3.86 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 196.1, 165.9, 145.5, 138.4, 137.0, 135.1, 133.6, 131.2, 130.4, 130.1, 130.0, 128.7, 127.3, 62.4, 52.4; IR (KBr) υ 2951, 1726, 1666, 1596, 1437, 1303, 1345, 1156, 1098, 1051, 860 cm⁻¹; HRMS (EI) Calcd for C₁₇H₁₅NO₄ [M]⁺ 297.1001, found 297.0999.



width: 24038.46 Hz = 238.8967 ppm = 0.366798 Hz/pt

S17



S18



freq. of 0 ppm: 100.612769 MHz processed size: 32768 complex points LB: 0.000 GF: 0.0000 Hz/cm: 864 955 ppm/cm: 8 59602









width: 24038.46 Hz = 238.8967 ppm = 0.366798 Hz/pt number of scans: 256

freq. of 0 ppm: 100.612769 MHz processed size: 32768 complex points LB: 0.000 GF: 0.0000 Hz/cm: 877.833 ppm/cm: 8.72400













width: 41666.67 Hz = 236.5193 ppm = 0.635783 Hz/pt number of scans: 32

processed size: 32768 complex points LB: 0.000 GF: 0.0000 Hz/cm: 1666.667 ppm/cm: 9.46077



S30



freq. of 0 ppm: 100.612769 MHz processed size: 32768 complex points LB: 0.000 GF: 0.0000 Hz/cm: 885.345 ppm/cm: 8.79865









S35





S37





S39





processed size: 32768 complex points LB: 0.000 GF: 0.0000 Hz/cm: 805.419 ppm/cm: 8.00434



S42