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Supporting Information

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Synthesis, Structure and Chemoselective Reactivity of *N*-(2-Iodyl-phenyl)-acylamides, Novel Hypervalent Iodine Reagents Bearing Pseudo Six-membered Ring Scaffold**

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Experimental Section

General Information. All melting points were determined in an open capillary tube with a Mel-temp II® melting point apparatus and are uncorrected. Infrared spectra were recorded as a NaCl pellet on a Perkin-Elmer 1600 series FT-IR spectrophotometer; absorptions are reported in reciprocal centimeters. NMR spectra were recorded on a Varian ^{UNITY} INOVA 300 MHz NMR spectrometer at 300 MHz (^{1}H NMR), and 75.5 MHz (^{13}C NMR); chemical shifts are reported in parts per million (ppm). ^{1}H and ^{13}C chemical shifts are referenced relative to the tetramethylsilane. GC-MS analysis was carried out with a HP 5890A Gas Chromatograph using a 5970 Series mass selective detector. Mass-spectra were obtained with a Micromass Zabspec oaTOF or PE Biosystems Mariner TOF instrument. Dry column vacuum chromatography (DCVC) was performed according to the technique described earlier (D. S. Pedersen, C. Rosenbohm, *Synthesis* **2001**, 2431).

Materials. All commercial reagents were ACS reagent grade and used without further purification. Methylene chloride, 1,2-dichloroethane, diethyl ether and acetonirile were distilled from $CaCl_2$ and stored over molecular sieves (4 Å). THF was distilled from KOH immediately prior to use. 3,3-Dimethyldioxirane (as 0.1 M solution in acetone) was prepared from commercial acetone and Oxone (Aldrich) by a known method (W. Adam, J. Bialas, L. Hadjiarapoglou, *Chem. Ber.* **1991**, *124*, 3277). Reaction flasks were oven-dried at 200^{o}C and flushed with dry nitrogen prior to use.

General Procedure for the preparation of *N*-(2-iodophenyl)acylamides 9a-e and 10. To a stirred, cooled (0-5^{o}C) solution of 2-iodoaniline **8** (2.190 g, 10 mmol) and Et_3N (1.113 g, 1.55 ml, 11 mmol) in 20 ml of dry THF a solution of an appropriate acyl chloride (10 mmol) in 5 ml of dry THF was added dropwise within 10 min. Then ice bath was removed and the mixture was stirred vigorously for 30 min at room temperature. Then solid $Et_3N{\cdot}HCl{\cdot}$was filtered off and washed with THF (3×5 ml). The resulting organic fractions were combined and THF was removed under reduced pressure to yield crude amides **9a-e** and **10**. Recrystallization from hexanes/$CHCl_3$ and drying in vacuum afforded analytically pure compounds **9** and **10**.

***N*-(2-iodophenyl)acetamide 9a.** Recrystallization of the crude product from hexanes/$CHCl_3$ afforded 2.114 g (81%) of **9a** as an off-white solid. M.p. 111-112^{o}C; IR (NaCl): 3261, 3030, 1661, 1577, 1532, 1433, 1373, 1296, 1008 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.18 (dd, ^{3}J = 8.1 Hz, , ^{4}J=1.5 Hz, 1H), 7.77 (dd, ^{3}J = 8.1 Hz, , ^{4}J=1.2 Hz, 1H), 7.46 (br s, 1H), 7.34 (td, ^{3}J = 8.1 Hz, , ^{4}J=1.3 Hz, 1H), 6.84 (td, ^{3}J = 7.5 Hz, , ^{4}J=1.3 Hz, 1H), 2.24 (s, 3H); ^{13}C NMR (75.5 MHz, $CDCl_3$): δ 168.4, 138.9, 138.3, 129.3, 126.1, 122.3, 90.2, 24.8; ESI-MS: *m/z* (%) 284.0 (100) $[M+Na]^{+}$.

***N*-(2-iodophenyl)butyramide 9b.** Recrystallization of the crude product from hexanes/$CHCl_3$ afforded 2.177 g (73%) of **9b** as an off-white solid. M.p. 83-84^{o}C; IR (NaCl): 3272, 2952, 1653, 1527, 1432, 1283, 1194, 1010 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.22 (dd, ^{3}J = 7.2 Hz, , ^{4}J=1.6 Hz, 1H), 7.77 (dd, ^{3}J = 7.8 Hz, ^{4}J=1.5 Hz, 1H), 7.46 (br s, 1H), 7.33 (td, ^{3}J = 7.8 Hz, ^{4}J=1.2 Hz, 1H), 6.83 (td, ^{3}J = 7.8 Hz, ^{4}J=1.2 Hz, 1H), 2.41 (t, ^{3}J = 7.5 Hz, 2H), 1.80 (sextet, ^{3}J = 7.8 Hz, 2H), 1.04 (t, ^{3}J = 7.2 Hz, 3H); ^{13}C NMR (75.5 MHz, $CDCl_3$): δ 171.5, 139.0, 138.5, 129.5, 126.2, 122.4, 90.4, 40.1, 19.3, 14.1; ESI-MS: *m/z* (%) 312.0 (100) $[M+Na]^{+}$.

***N*-(2-iodophenyl)isobutyramide 9c**. Recrystallization of the crude product from hexanes/$CHCl_3$ afforded 2.398 g (83%) of **9c** as an off-white solid. M.p. 117-118^{o}C; IR (NaCl): 3245, 2970, 1661, 1522, 1433, 1275, 1204, 1096, 1010 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.26 (dd, ^{3}J = 8.1 Hz, 1H), 7.78 (dd, ^{3}J = 8.1 Hz, ^{4}J=1.4 Hz, 1H), 7.53 (br s, 1H), 7.34 (td, ^{3}J = 8.1 Hz, ^{4}J=1.5 Hz, 1H), 6.84 (t, ^{3}J = 7.8 Hz, ^{4}J=1.4 Hz, 1H), 2.62 (septet, ^{3}J = 6.9 Hz, 1H), 1.32 (d, ^{3}J = 6.9 Hz, 6H); ^{13}C NMR (75.5 MHz, $CDCl_3$): δ 175.4, 139.0, 138.5, 129.5, 126.1, 122.3, 90.4, 37.2, 19.9; ESI-MS: *m/z* (%) 312.0 (100) $[M+Na]^{+}$.

***N*-(2-iodophenyl)cyclohexanecarboxamide 9d**. Recrystallization of the crude product from hexanes/$CHCl_3$ afforded 2.465 g (75%) of **9d** as white needles. M.p. 139-140^oC; IR (NaCl): 3260, 2913, 2846, 1648, 1574, 1527, 1426, 1278, 1018 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 8.25 (dd, ^3J = 8.1 Hz, ^4J=1.2 Hz, 1H), 7.76 (dd, ^3J = 7.8 Hz, ^4J=1.2 Hz, 1H), 7.54 (br s, 1H), 7.32 (td, ^3J = 7.8 Hz, ^4J=1.3 Hz, 1H), 6.82 (td, ^3J = 7.5 Hz, ^4J=1.2 Hz, 1H), 2.32 (tt, $^3J_{ax}$ = 11.4 Hz, $^3J_{eq}$ = 3.3 Hz, 1H), 2.07-1.23 (m, 10H); ^{13}C NMR (75.5 MHz, $CDCl_3$): δ 174.3, 138.8, 138.3, 129.3, 125.8, 122.1, 90.2, 46.6, 29.8, 25.8; ESI-MS: *m/z* (%) 352.1 (100) $[M+Na]^+$.

***N*-(2-iodophenyl)pivalamide 9e**. Recrystallization of the crude product from hexanes afforded 2.485 g (82%) of **9e** as an off-white solid. M.p. 68-69^oC; IR (NaCl): 3272, 2959, 1653, 1467, 1174, 1013 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 8.29 (dd, ^3J = 8.4 Hz, ^4J=1.2 Hz, 1H), 7.81 (br s, 1H), 7.77 (dd, ^3J = 7.8 Hz, ^4J=1.2 Hz, 1H), 7.34 (td, ^3J = 8.7 Hz, ^4J=1.3 Hz, 1H), 6.83 (td, ^3J = 8.3 Hz, ^4J=1.4 Hz, 1H), 1.37 (s, 9H); ^{13}C NMR (75.5 MHz, $CDCl_3$): δ 177.0, 139.0, 138.6, 129.5, 125.9, 122.0, 90.4, 40.4, 28.0; ESI-MS: *m/z* (%) 326.1 (100) $[M+Na]^+$.

4-Chloro-*N*-(2-iodophenyl)butanamide 10. Recrystallization of the crude product from hexanes afforded 2.136 g (66%) of **10** as an off-white solid. M.p. 88-89^oC; IR (NaCl): 3261, 1650, 1570, 1516, 1427, 1277, 1204, 1016 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 8.18 (dd, ^3J = 7.8 Hz, ^4J=1.2 Hz, 1H), 7.78 (dd, ^3J = 8.1 Hz, ^4J=1.2 Hz, 1H), 7.48 (br s, 1H), 7.34 (td, ^3J = 7.5 Hz, ^4J=1.3 Hz, 1H), 6.86 (td, ^3J = 7.8 Hz, ^4J=1.3 Hz, 1H), 3.68 (t, ^3J = 6.3 Hz, 2H), 2.64 (t, ^3J = 7.2 Hz, 2H), 2.23 (quintet, ^3J = 6.6 Hz, 2H); ESI-MS: *m/z* (%) 346.0 (100) $[M+Na]^+$.

General Procedure for the preparation of *N*-(2-iodophenyl)-*N*-methylacylamides 11a-e. To a stirred suspension of NaH (0.132 g; 5.5 mmol) in 5 ml of dry THF at 0^oC the respective amide **9a-e** (5 mmol) dissolved in 10 ml of THF was added dropwise within 10 min. The reaction mixture was stirred until the solution became clear (30 min, hydrogen gas evolved), and the solution of MeI (0.9226 g; 0.405 ml; 6.5 mmol) in 5 ml of THF was added dropwise within 10 min. The solution was warmed up to room temperature and stirred for 3 h. Then, the reaction mixture was quenched with water (30 ml). The resulting solution was extracted with ethyl acetate (3×20 ml). Combined organic layers were washed with brine (1×20 ml) and dried over Na_2SO_4 (8 hours). Ethyl acetate was removed under reduced pressure to give crude **11a-e**. Recrystallization from hexanes or hexanes/$CHCl_3$ and drying in vacuum afforded analytically pure compounds **11.**

***N*-(2-iodophenyl)-*N*-methylacetamide 11a.** Recrystallization of the crude product from hexanes afforded 1.155 g (84%) of **11a** as a yellow solid. M.p. 61-62^{o}C; IR (NaCl): 2930, 1650, 1469, 1427, 1382, 1297, 1090, 1018 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.94 (dd, ^{3}J = 7.8 Hz, ^{4}J=1.3 Hz, 1H), 7.44 (td, ^{3}J = 7.5 Hz, ^{4}J=1.3 Hz, 1H), 7.30 (dd, ^{3}J = 7.8 Hz, ^{4}J=1.3 Hz, 1H), 7.09 (td, ^{3}J = 7.5 Hz, ^{4}J=1.5 Hz, 1H), 3.18 (s, 3H), 1.80 (s, 3H); ^{13}C NMR (75.5 MHz, $CDCl_3$): δ 170.3, 146.7, 140.3, 129.9, 128.9, 99.5, 35.9, 22.6; ESI-MS: *m/z* (%) 298.0 (100) $[M+Na]^{+}$.

***N*-(2-iodophenyl)-*N*-methylbutyramide 11b.** Recrystallization of the crude product from hexanes afforded 1.191 g (79%) of **11b** as a yellow solid. M.p. 62-63^{o}C; IR (NaCl): 2930, 1641, 1470, 1419, 1380, 1118, 1018 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.95 (dd, ^{3}J = 8.1 Hz, ^{4}J=1.5 Hz, 1H), 7.44 (td, ^{3}J = 7.8 Hz, ^{4}J=1.4 Hz, 1H), 7.28 (dd, ^{3}J = 7.8 Hz, ^{4}J=1.4 Hz, 1H), 7.09 (td, ^{3}J = 7.8 Hz, ^{4}J=1.4 Hz, 1H), 3.18 (s, 3H), 1.93 (t, ^{3}J = 7.2 Hz, 2H), 1.63 (sextet, ^{3}J = 6.9 Hz, 2H), 0.86 (t, ^{3}J = 7.2 Hz, 3H); ^{13}C NMR (75.5 MHz, $CDCl_3$): δ 172.7, 146.4, 140.3, 130.0, 129.8, 129.2, 99.9, 36.3, 35.9, 18.6, 14.1; ESI-MS: *m/z* (%) 326.0 (100) $[M+Na]^{+}$.

***N*-(2-iodophenyl)-*N*-methylisobutyramide 11c.** Recrystallization of the crude product from hexanes/$CHCl_3$ afforded 1.215 g (80%) of **11c** as a white solid. M.p. 97-98^{o}C; IR (NaCl): 2970, 1645, 1467, 1379, 1266, 1108, 1016 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.96 (dd, ^{3}J = 7.8 Hz, ^{4}J=1.2 Hz, 1H), 7.44 (td, ^{3}J = 8.1 Hz, ^{4}J=1.2 Hz, 1H), 7.29 (dd, ^{3}J = 7.5 Hz, ^{4}J=1.3 Hz, 1H), 7.09 (td, ^{3}J = 7.8 Hz, ^{4}J=1.3 Hz, 1H), 3.18 (s, 3H), 2.23 (septet, ^{3}J = 6.6 Hz, 1H), 1.14 (d, ^{3}J = 6.6 Hz, 3H), 1.00 (d, ^{3}J = 6.6 Hz, 3H); ^{13}C NMR (75.5 MHz, $CDCl_3$): δ 177.3, 146.4, 140.3, 129.9, 129.7, 128.9, 100.1, 36.2, 32.0, 20.0, 19.7; ESI-MS: *m/z* (%) 326.0 (100) $[M+Na]^{+}$.

***N*-(2-iodophenyl)-*N*-methylcyclohexanecarboxamide 11d.** Recrystallization of the crude product from hexanes/$CHCl_3$ afforded 1.442 g (84%) of **11d** as a white solid. M.p. 112-113^{o}C; IR (NaCl): 2930, 1641, 1464, 1383, 1271, 1106, 1018 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.96 (dd, ^{3}J = 8.1 Hz, ^{4}J=1.5 Hz, 1H), 7.44 (td, ^{3}J = 7.2 Hz, ^{4}J=1.4 Hz, 1H), 7.27 (dd, ^{3}J = 7.8 Hz, ^{4}J=1.4 Hz, 1H), 7.10 (td, ^{3}J = 7.5 Hz, ^{4}J=1.4 Hz, 1H), 3.16 (s, 3H), 1.94-0.86 (m, 11H); ^{13}C NMR (75.5 MHz, $CDCl_3$): δ 176.2, 146.4, 140.3, 129.9, 129.7, 128.9, 100.1, 42.3, 36.1, 29.9, 29.3, 25.7, 25.4; ESI-MS: *m/z* (%) 366.1 (100) $[M+Na]^{+}$.

***N*-(2-iodophenyl)-*N*-methylpivalamide 11e.** Recrystallization of the crude product from hexanes afforded 1.318 g (83%) of **11e** as a yellow solid. M.p. 132-133^{o}C; IR (NaCl): 2966, 1627, 1467, 1350, 1290, 1214, 1103, 1012 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.91 (dd, ^{3}J =

8.1 Hz, ^{4}J=1.4 Hz, 1H), 7.39 (td, ^{3}J = 7.8 Hz, ^{4}J=1.3 Hz, 1H), 7.28 (dd, ^{3}J = 7.8 Hz, ^{4}J=1.3 Hz, 1H), 7.04 (td, ^{3}J = 7.8 Hz, ^{4}J=1.4 Hz, 1H), 3.16 (s, 3H), 1.08 (s, 9H); ^{13}C NMR (75.5 MHz, $CDCl_3$): δ 178.1, 147.8, 140.4, 129.9, 129.7, 129.5, 101.2, 41.1, 40.2, 29.4; ESI-MS: *m/z* (%) 340.1 (100) $[M+Na]^+$.

General Procedure for the preparation of *N*-benzyl-*N*-(2-iodophenyl)acylamides 12a-e. To a stirred suspension of NaH (0.132 g; 5.5 mmol) in 5 ml of dry THF at 0^{o}C the respective amide **9a-e** (5 mmol) dissolved in 10 ml of THF was added dropwise within 10 min. The reaction mixture was stirred until the solution became clear (30 min, hydrogen gas evolved), and the solution of BnBr (1.112 g; 0.78 ml; 6.5 mmol) in 5 ml of THF was added dropwise within 10 min. The solution was warmed up to room temperature and stirred for 3 h. Then, the reaction mixture was quenched with water (30 ml). The resulting solution was extracted with ethyl acetate (3×20 ml). Combined organic layers were washed with brine (1×20 ml) and dried over Na_2SO_4 (8 hours). Ethyl acetate was removed under reduced pressure to give crude **12a-e**. Recrystallization from hexanes or purifying by DCVC (hexanes/EtOAc) and drying in vacuum afforded analytically pure compounds **12.**

***N*-benzyl-*N*-(2-iodophenyl)acetamide 12a.** Purification of the crude product by DCVC (hexanes with 10% EtOAc gradient) afforded 1.155 g (80%) of **12a** as a yellow oil. IR (neat): 3075, 3033, 2930, 1663, 1470, 1389, 1287, 1217, 1067, 1021 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.94 (dd, ^{3}J = 7.8 Hz, ^{4}J=1.2 Hz, 1H), 7.27-7.18 (m, 6H), 7.03 (td, ^{3}J = 7.8 Hz, ^{4}J=1.3 Hz, 1H), 6.72 (dd, ^{3}J = 7.8 Hz, ^{4}J=1.2 Hz, 1H), 5.67 (d, ^{2}J = 14.4 Hz, 1H), 3.93 (d, ^{2}J = 14.4 Hz, 1H), 1.82 (s, 3H); ^{13}C NMR (75.5 MHz, $CDCl_3$): δ 170.1, 144.5, 140.3, 137.0, 130.8, 129.9, 129.5, 129.3, 128.4, 127.6, 100.3, 51.5, 22.9; ESI-MS: *m/z* (%) 374.1 (100) $[M+Na]^+$

***N*-benzyl-*N*-(2-iodophenyl)butyramide 12b.** Purification of the crude product by DCVC (hexanes with 10% EtOAc gradient) afforded 1.459 g (77%) of **12b** as a yellow oil. IR (neat): 3057, 2960, 1666, 1467, 1392, 1278, 1251, 1016 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.94 (dd, ^{3}J = 8.0 Hz, ^{4}J=1.3 Hz, 1H), 7.27-7.17 (m, 6H), 7.02 (td, ^{3}J = 7.8 Hz, ^{4}J=1.2 Hz, 1H), 6.69 (dd, ^{3}J = 8.0 Hz, ^{4}J=1.2 Hz, 1H), 5.71 (d, ^{2}J = 14.1 Hz, 1H), 3.89 (d, ^{2}J = 14.4 Hz, 1H), 1.92 (t, ^{3}J = 7.6 Hz, 2H), 1.63 (m, 2H), 0.86 (t, ^{3}J = 7.5 Hz, 3H); ^{13}C NMR (75.5 MHz, $CDCl_3$): δ 172.5, 144.1, 140.2, 137.2, 130.9, 129.8, 129.4, 129.2, 128.4, 127.5, 100.6, 51.5, 36.5, 18.5, 14.0; ESI-MS: *m/z* (%) 402.1 (100) $[M+Na]^+$

***N*-benzyl-*N*-(2-iodophenyl)isobutyramide 12c.** Recrystallization of the crude product from hexanes afforded 1.631 g (86%) of **12c** as an off-white solid. M.p. 84-85^{o}C; IR (NaCl): 2970, 1648, 1462, 1409, 1244, 1197, 1078, 1024 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.95 (dd, ^{3}J = 7.8 Hz, ^{4}J=1.2 Hz, 1H), 7.27-7.17 (m, 6H), 7.02 (td, ^{3}J = 7.8 Hz, ^{4}J=1.2 Hz, 1H), 6.71 (dd, ^{3}J = 8.0 Hz, ^{4}J=1.3 Hz, 1H), 5.70 (d, ^{2}J = 14.4 Hz, 1H), 3.88 (d, ^{2}J = 14.4 Hz, 1H), 2.19 (septet, ^{3}J = 6.6 Hz, 1H), 1.17 (d, ^{3}J = 6.6 Hz, 3H), 1.00 (d, ^{3}J = 6.6 Hz, 3H); ^{13}C NMR (75.5 MHz, $CDCl_3$): δ 177.4, 144.4, 140.5, 137.6, 130.9, 130.1, 129.9, 129.3, 128.7, 127.7, 101.2, 51.8, 32.4, 20.4, 19.7; ESI-MS: *m/z* (%) 402.1 (100) $[M+Na]^+$

***N*-benzyl-*N*-(2-iodophenyl)cyclohexanecarboxamide 12d.** Recrystallization of the crude product from hexanes afforded 1.781 g (85%) of **12d** as a white solid. M.p. 136-137^{o}C; IR (NaCl): 2930, 2833, 1644, 1464, 1464, 1401, 1325, 1263, 1190 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.95 (dd, ^{3}J = 8.0 Hz, ^{4}J=1.4 Hz, 1H), 7.27-7.16 (m, 6H), 7.03 (td, ^{3}J = 8.0 Hz, ^{4}J=1.2 Hz, 1H), 6.70 (dd, ^{3}J = 8.0 Hz, ^{4}J=1.2 Hz, 1H), 5.69 (d, ^{2}J = 14.1 Hz, 1H), 3.86 (d, ^{2}J = 14.4 Hz, 1H), 1.90-0.81 (m, 11H); ^{13}C NMR (75.5 MHz, $CDCl_3$): δ 175.9, 144.0, 140.1, 137.3, 130.4, 129.5, 129.2, 128.9, 128.3, 127.3, 100.9, 51.3, 42.4, 30.0, 28.9, 25.6, 25.2; ESI-MS: *m/z* (%) 442.1 (100) $[M+Na]^+$

***N*-benzyl-*N*-(2-iodophenyl)pivalamide 12e.** Recrystallization of the crude product from hexanes afforded 1.591 g (81%) of **12e** as an off-white solid. M.p. 59-60^{o}C; IR (NaCl): 2960, 1636, 1470, 1392, 1277, 1184, 1079 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.94 (dd, ^{3}J = 7.8 Hz, ^{4}J=1.2 Hz, 1H), 7.26-7.13 (m, 6H), 7.00 (td, ^{3}J = 8.0 Hz, ^{4}J=1.3 Hz, 1H), 6.73 (dd, ^{3}J = 8.0 Hz, ^{4}J=1.2 Hz, 1H), 5.83 (d, ^{2}J = 14.4 Hz, 1H), 3.75 (d, ^{2}J = 14.4 Hz, 1H), 1.06 (s, 9H); ^{13}C NMR (75.5 MHz, $CDCl_3$): δ 177.2, 144.9, 140.1, 137.3, 131.8, 129.4, 129.2, 128.7, 128.2, 127.3, 102.1, 54.3, 41.1, 29.1; ESI-MS: *m/z* (%) 416.1 (100) $[M+Na]^+$

1-(2-Iodophenyl)pyrrolidin-2-one 13. To a stirred suspension of NaH (0.132 g; 5.5 mmol) in 5 ml of dry THF at 0^{o}C amide **10** (1.618 g, 5 mmol) dissolved in 10 ml of THF was added dropwise within 10 min. The reaction mixture was stirred for 30 min (clear solution), then ice bath was removed and the reaction mixture was stirred at room temperature for 3 h. Afterwards, the reaction was quenched with water (30 ml). The resulting solution was extracted with ethyl acetate (3×20 ml). Combined organic fractions were washed with brine (1×20 ml) and dried over Na_2SO_4 for 8 hours. Ethyl acetate was removed under reduced pressure to give crude **13**. Recrystallization from hexanes/$CHCl_3$ afforded 1.162 g (81%) of **11e** as a white solid. M.p. 82-83^{o}C; IR (NaCl): 2878, 1693, 1468, 1406, 1234, 1124, 1013 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$):

δ 7.91 (dd, ^{3}J = 8.1 Hz, ^{4}J=1.2 Hz, 1H), 7.41 (td, ^{3}J = 7.8 Hz, ^{4}J=1.2 Hz, 1H), 7.26 (dd, ^{3}J = 7.8 Hz, ^{4}J=1.3 Hz, 1H), 7.06 (td, ^{3}J = 7.8 Hz, ^{4}J=1.3 Hz, 1H), 3.76 (t, ^{3}J = 6.9 Hz, 2H), 2.60 (t, ^{3}J = 7.8 Hz, 2H), 2.27 (quintet, ^{3}J = 7.2 Hz, 2H); ^{13}C NMR (75.5 MHz, $CDCl_3$): δ 174.7, 141.5, 140.0, 129.8, 129.6, 129.0, 98.1, 50.3, 31.4, 19.2; ESI-MS: *m/z* (%) 310.0 (100) $[M+Na]^+$.

General Procedure for the preparation of *N*-(2-Iodyl-phenyl)-acylamides 14-17. A freshly prepared solution of 3,3-dimethyldioxirane in acetone (0.1 м, 30 mL, 3.0 mmol) was added to a stirred solution of amides **11-13** (1.0 mmol) in 5 ml of CH_2Cl_2 ($CHCl_3$ in case of amide **11d**) at 0^{o}C (ice bath). The reaction mixture was stirred for 1 h at 0^{o}C, then ice bath was removed and the solution was additionally stirred for 2 h at room temperature. Then, the solvent was removed under reduced pressure, the remained white solid was stirred with 5 ml of Et_2O (hexanes in case of compounds **16**) for 15 min, filtered, washed with cold Et_2O (cold hexanes in case of compounds **16**) (2×2.5 ml) and dried in vacuum to afford analytically pure **14-17** as white solids.

***N*-(2-iodylphenyl)acetamide 14a.** General procedure afforded 0.222 g (76%) of **14a** as a white solid. M.p. 160^{o}C (decomp.); IR (NaCl): 2990, 1648, 1614, 1597, 1539, 1464, 1422, 1367, 1248, 1036, 968, 778; ^{1}H NMR (300 MHz, [D_6]DMSO) d 11.00 (br s, 1H), 7.96 (dd, ^{3}J=8.1 Hz, ^{4}J=1.4 Hz, 1H), 7.54 (td, ^{3}J=7.8 Hz, ^{4}J=1.5 Hz, 1H, Ar), 7.34 (td, ^{3}J = 8.0 Hz, , ^{4}J=1.3 Hz, 1H), 7.19 (dd, ^{3}J = 8.1 Hz, , ^{4}J=1.5 Hz, 1H), 2.13 (s, 3H); ^{13}C NMR (75.5 MHz, [D_6]DMSO) d 171.6, 140.7, 135.9, 133.3, 128.6, 125.9, 122.7, 24.2; ESI-HRMS: *m/z* (%) 315.9460 (100) $[M+Na]^+$, 608.9635 (55) $[2M+Na]^+$.

***N*-(2-iodylphenyl)acetamide 14b.** General procedure afforded 0.280 g (87%) of **14b** as a white solid. M.p. 146^{o}C (decomp.); IR (NaCl): 2978, 1648, 1605, 1576, 1540, 1464, 1242, 1097, 941, 769 cm^{-1}; ^{1}H NMR (300 MHz, [D_6]DMSO) d 11.00 (br s, 1H), 7.97 (dd, ^{3}J=8.0 Hz, ^{4}J=1.4 Hz, 1H), 7.53 (td, ^{3}J=8.0 Hz, ^{4}J=1.2 Hz, 1H, Ar), 7.39 (td, ^{3}J = 8.2 Hz, , ^{4}J=1.4 Hz, 1H), 7.22 (dd, ^{3}J = 8.0 Hz, , ^{4}J=1.6 Hz, 1H), 2.37 (t, ^{3}J = 7.4 Hz, 2H), 1.64 (sextet, ^{3}J = 7.7 Hz, 2H), 0.94 (t, ^{3}J = 7.2 Hz, 3H); ^{13}C NMR (75.5 MHz, [D_6]DMSO) d 173.3, 139.7, 134.9, 132.3, 127.6, 124.8, 121.8, 37.5, 18.3, 13.5; ESI-HRMS: *m/z* (%) 343.9726 (100) $[M+Na]^+$, 665.0232 (61) $[2M+Na]^+$.

***N*-(2-iodylphenyl)isobutyramide 14c.** General procedure afforded 0.308 g (96%) of **14c** as a white solid. M.p. 161^{o}C (decomp.); IR (NaCl): 2954, 1653, 1611, 1575, 1539, 1464, 1088, 964, 760 cm^{-1}; ^{1}H NMR (300 MHz, [D_6]DMSO) d 10.92 (br s, 1H), 7.96 (dd, ^{3}J=8.2 Hz, ^{4}J=1.4 Hz, 1H), 7.53 (td, ^{3}J=8.2 Hz, ^{4}J=1.5 Hz, 1H, Ar), 7.39 (td, ^{3}J = 8.2 Hz, , ^{4}J=1.5 Hz, 1H), 7.24 (dd, ^{3}J

= 8.3 Hz, , ^{4}J=1.5 Hz, 1H), 2.70 (septet, ^{3}J = 7.0 Hz, 1H), 2.50 (d, ^{3}J = 7.0 Hz, 6H); ^{13}C NMR (75.5 MHz, [D$_6$]DMSO) d 177.9, 140.8, 136.1, 133.2, 128.6, 125.8, 122.9, 35.2, 20.0; ESI-HRMS: *m/z* (%) 343.9771 (100) [M+Na]$^{+}$, 665.0150 (48) [2M+Na]$^{+}$.

***N*-(2-iodylphenyl)cyclohexanecarboxamide 14d.** General procedure afforded 0.324 g (90%) of **14d** as a white solid. M.p. 174^{o}C (decomp.); IR (NaCl): 2930, 1645, 1608, 1572, 1533, 1462, 1422, 1268, 1226, 958, 775 cm^{-1}; ^{1}H NMR (300 MHz, [D$_6$]DMSO) d 10.87 (br s, 1H), 7.94 (dd, ^{3}J=8.0 Hz, ^{4}J=1.2 Hz, 1H), 7.53 (td, ^{3}J=8.0 Hz, ^{4}J=1.2 Hz, 1H, Ar), 7.38 (td, ^{3}J = 8.2 Hz, , ^{4}J=1.4 Hz, 1H), 7.22 (dd, ^{3}J = 8.0 Hz, , ^{4}J=1.5 Hz, 1H), 2.52-1.17 (m, 11H); ^{13}C NMR (75.5 MHz, [D$_6$]DMSO) d 176.9, 140.8, 136.1, 133.1, 128.5, 125.7, 122.8, 44.9, 29.6, 26.3, 25.9; ESI-HRMS: *m/z* (%) 384.0074 (42) [M+Na]$^{+}$, 745.1085 (100) [2M+Na]$^{+}$.

***N*-(2-iodylphenyl)pivalamide 14e.** General procedure afforded 0.293 g (87%) of **14e** as a white solid. M.p. 182^{o}C (decomp.); IR (NaCl): 3220, 2972, 1620, 1521, 1438, 1422, 1232, 1181, 928, 769 cm^{-1}; ^{1}H NMR (300 MHz, [D$_6$]DMSO) d 10.33 (br s, 1H), 7.93 (dd, ^{3}J=7.8 Hz, ^{4}J=1.3 Hz, 1H), 7.54 (td, ^{3}J=8.0 Hz, ^{4}J=1.5 Hz, 1H, Ar), 7.42-7.36 (m, 2H), 1.26 (s, 9H); ^{13}C NMR (75.5 MHz, [D$_6$]DMSO) d 179.3, 141.4, 136.5, 132.9, 128.7, 125.9, 123.9, 41.3, 27.7; ESI-HRMS: *m/z* (%) 357.9908 (63) [M+Na]$^{+}$, 693.0523 (100) [2M+Na]$^{+}$.

***N*-(2-iodylphenyl)-*N*-methylacetamide 15a.** General procedure afforded 0.243 g (79%) of **15a** as a white solid. M.p. 148^{o}C (decomp.); IR (NaCl): 3062, 1643, 1611, 1560, 1464, 1422, 1398, 1347, 1247, 1127, 1033, 883, 753 cm^{-1}; ^{1}H NMR (300 MHz, CD$_3$OD) d 8.05 (dd, ^{3}J=8.2 Hz, ^{4}J=1.5 Hz, 1H), 7.74 (td, ^{3}J=7.8 Hz, ^{4}J=1.5 Hz, 1H), 7.61-7.55 (m, 2H), 3.64 (s, 3H), 2.42 (s, 3H); ^{13}C NMR (75.5 MHz, CD$_3$OD) d 176.9, 146.7, 142.7, 135.5, 129.3, 128.9, 126.3, 41.1, 24.2; ESI-HRMS: *m/z* (%) 329.9607 (79) [M+Na]$^{+}$, 637.0042 (100) [2M+Na]$^{+}$.

***N*-(2-iodylphenyl)-*N*-methylbutyramide 15b.** General procedure afforded 0.301 g (90%) of **15b** as a white solid. M.p. 142-143^{o}C; IR (NaCl): 2954, 1668, 1611, 1572, 1464, 1395, 1322, 1232, 1124, 1091, 1036, 880, 736 cm^{-1}; ^{1}H NMR (300 MHz, CD$_3$OD) d 8.05 (dd, ^{3}J=8.0 Hz, ^{4}J=1.3 Hz, 1H), 7.72 (td, ^{3}J=7.9 Hz, ^{4}J=1.4 Hz, 1H), 7.58-7.53 (m, 2H), 3.61 (s, 3H), 2.68 (t, ^{3}J = 7.6 Hz, 2H), 1.78 (sextet, ^{3}J = 8.0 Hz, 2H), 1.07 (t, ^{3}J = 7.4 Hz, 3H);^{13}C NMR (75.5 MHz, CD$_3$OD) d 178.1, 146.0, 142.3, 134.8, 128.5, 128.1, 125.7, 39.7, 37.6, 19.1, 14.3; ESI-HRMS: *m/z* (%) 357.9920 (64) [M+Na]$^{+}$, 693.0614 (100) [2M+Na]$^{+}$.

***N*-(2-iodylphenyl)-*N*-methylisobutyramide 15c.** General procedure afforded 0.280 g (84%) of **15c** as a white solid. M.p. 170^{o}C (decomp.); IR (NaCl): 2923, 1611, 1572, 1455, 1395, 1316, 1227, 1076, 947, 781 cm^{-1}; ^{1}H NMR (300 MHz, CD_3OD) d 8.01 (dd, ^{3}J=7.8 Hz, ^{4}J=1.5 Hz, 1H), 7.69 (td, ^{3}J=7.8 Hz, ^{4}J=1.4 Hz, 1H), 7.57-7.50 (m, 2H), 3.65 (s, 3H), 3.16 (septet, ^{3}J = 7.2 Hz, 1H), 1.24 (d, ^{3}J = 6.8 Hz, 6H);^{13}C NMR (75.5 MHz, CD_3OD) d 181.6, 146.3, 142.6, 134.8, 128.4, 128.2, 125.9, 39.7, 32.9, 19.3; ESI-HRMS: *m/z* (%) 357.9917 (47) $[M+Na]^{+}$, 693.0682 (100) $[2M+Na]^{+}$.

***N*-(2-iodylphenyl)-*N*-methylcyclohexanecarboxamide 15d.** General procedure afforded 0.319 g (85%) of **15d** as a white solid. M.p. 163^{o}C (decomp.); IR (NaCl): 2923, 1614, 1572, 1464, 1401, 1340, 1220, 1148, 1103, 1006, 892, 766 cm^{-1}; ^{1}H NMR (300 MHz, CD_3OD) d 8.01 (dd, ^{3}J=7.7 Hz, ^{4}J=1.3 Hz, 1H), 7.69 (td, ^{3}J=7.9 Hz, ^{4}J=1.5 Hz, 1H), 7.56-7.50 (m, 2H), 3.63 (s, 3H), 2.87 (tt, $^{3}J_{ax}$ = 11.3 Hz, $^{3}J_{eq}$ = 3.8 Hz, 1H), 1.97-1.27 (m, 10H);^{13}C NMR (75.5 MHz, CD_3OD) d 179.2, 145.1, 141.3, 133.5, 127.0, 126.9, 124.7, 41.8, 38.3, 28.7, 25.8, 25.4; ESI-HRMS: *m/z* (%) 398.0223 (47) $[M+Na]^{+}$, 773.1306 (100) $[2M+Na]^{+}$.

***N*-(2-iodylphenyl)-*N*-methylpivalamide 15e.** General procedure afforded 0.300 g (86%) of **15e** as a white solid. M.p. 172^{o}C (decomp.); IR (NaCl): 2978, 1602, 1572, 1464, 1350, 1199, 1151, 1103, 1082, 1012, 760 cm^{-1}; ^{1}H NMR (300 MHz, CD_3OD) d 8.00 (dd, ^{3}J=8.0 Hz, ^{4}J=1.5 Hz, 1H), 7.69 (td, ^{3}J=7.7 Hz, ^{4}J=1.5 Hz, 1H), 7.51-7.59 (m, 2H), 3.68 (s, 3H), 1.43 (s, 9H); ^{13}C NMR (75.5 MHz, CD_3OD) d 182.0, 147.0, 144.7, 134.9, 128.4, 128.3, 127.0, 41.5, 41.0, 28.2; ESI-HRMS: *m/z* (%) 372.0075 (23) $[M+Na]^{+}$, 721.0684 (100) $[2M+Na]^{+}$.

***N*-benzyl-*N*-(2-iodylphenyl)acetamide 16a.** General procedure afforded 0.369 g (96%) of **16a** as a white solid. M.p. 118-119^{o}C; IR (NaCl): 2912, 1672, 1620, 1461, 1404, 1272, 1076, 1030, 974, 769 cm^{-1}; ^{1}H NMR (300 MHz, $[D_6]$DMSO-CD_3OD) δ 8.04 (dd, ^{3}J = 8.0 Hz, ^{4}J=1.2 Hz, 1H), 7.71 (td, ^{3}J = 7.9 Hz, ^{4}J=1.6 Hz, 1H), 7.51-7.18 (m, 6H), 6.81 (dd, ^{3}J = 8.2 Hz, ^{4}J=1.3 Hz, 1H), 5.62 (d, ^{2}J = 13.4 Hz, 1H), 3.93 (d, ^{2}J = 13.2 Hz, 1H), 1.85 (s, 3H); ^{13}C NMR (75.5 MHz, $[D_6]$DMSO-CD_3OD) d 171.6, 148.4, 140.7, 137.5, 133.3, 131.6, 130.2, 129.3, 128.1, 125.5, 52.0, 23.8 ; ESI-HRMS: *m/z* (%) 405.9937 (59) $[M+Na]^{+}$, 788.9952 (100) $[2M+Na]^{+}$.

***N*-benzyl-*N*-(2-iodylphenyl)butyramide 16b.** General procedure afforded 0.391 g (95%) of **16b** as a white solid. M.p. 110-112^{o}C; IR (NaCl): 2954, 1669, 1627, 1458, 1410, 1223, 1079, 766 cm^{-1}; ^{1}H NMR (300 MHz, $[D_6]$DMSO-CD_3OD) δ 8.02 (dd, ^{3}J = 8.2 Hz, ^{4}J=1.5 Hz, 1H), 7.70 (td, ^{3}J = 7.8 Hz, ^{4}J=1.5 Hz, 1H), 7.50-7.11 (m, 6H), 6.76 (dd, ^{3}J = 8.2 Hz, ^{4}J=1.3 Hz, 1H), 5.62 (d, ^{2}J =

13.6 Hz, 1H), 4.35 (d, ^{2}J = 13.8 Hz, 1H), 2.19 (t, ^{3}J = 7.4 Hz, 2H), 1.52 (m, 2H), 0.79 (t, ^{3}J = 7.9 Hz, 3H); ^{13}C NMR (75.5 MHz, [D_6]DMSO-CD_3OD) d 173.9, 148.4, 140.6, 137.6, 133.3, 131.5, 130.7, 129.3, 129.2, 128.1, 125.6, 52.1, 36.4, 18.4, 14.1; ESI-HRMS: *m/z* (%) 434.0258 (42) $[M+Na]^+$, 845.1103 (100) $[2M+Na]^+$.

***N*-benzyl-*N*-(2-iodylphenyl)butyramide 16c.** General procedure afforded 0.401 g (97%) of **16c** as a white solid. M.p. 158^{o}C (decomp.); IR (NaCl): 2984, 1671, 1620, 1464, 1410, 1392, 1223, 1088, 985, 772 cm^{-1}; ^{1}H NMR (300 MHz, [D_6]DMSO-CD_3OD) δ 8.06 (dd, ^{3}J = 8.0 Hz, ^{4}J=1.6 Hz, 1H), 7.71 (td, ^{3}J = 8.2 Hz, ^{4}J=1.2 Hz, 1H), 7.50-7.14 (m, 6H), 6.72 (dd, ^{3}J = 8.2 Hz, ^{4}J=1.2 Hz, 1H), 5.62 (d, ^{2}J = 14.6 Hz, 1H), 4.30 (d, ^{2}J = 14.8 Hz, 1H), 2.24 (m, 1H), 1.01 (m, 6H); ^{13}C NMR (75.5 MHz, [D_6]DMSO-CD_3OD) d 177.6, 147.9, 139.8, 139.8, 137.4, 132.7, 130.4, 129.7, 127.8, 125.2, 51.5, 31.8, 20.2, 19.2; ESI-HRMS: *m/z* (%) 434.0251 (42) $[M+Na]^+$, 845.1084 (100) $[2M+Na]^+$.

***N*-benzyl-*N*-(2-iodylphenyl)cyclohexanecarboxamide 16d.** General procedure afforded 0.439 g (97%) of **16d** as a white solid. M.p. 140-143^{o}C; IR (NaCl): 2923, 1668, 1611, 1452, 1419, 1358, 1202, 988, 766 cm^{-1}; ^{1}H NMR (300 MHz, [D_6]DMSO-CD_3OD) δ 8.07 (dd, ^{3}J = 8.2 Hz, ^{4}J=1.4 Hz, 1H), 7.72 (td, ^{3}J = 8.0 Hz, ^{4}J=1.4 Hz, 1H), 7.49-7.11 (m, 6H), 6.71 (dd, ^{3}J = 8.2 Hz, ^{4}J=1.2 Hz, 1H), 5.62 (d, ^{2}J = 14.0 Hz, 1H), 4.29 (d, ^{2}J = 14.4 Hz, 1H), 2.04-0.73 (m, 11H); ^{13}C NMR (75.5 MHz, [D_6]DMSO-CD_3OD) d 176.9, 148.4, 140.3, 137.9, 133.1, 130.8, 130.2, 129.2, 128.2, 125.7, 51.9, 42.1, 30.5, 28.6, 25.9, 25.2; ESI-HRMS: *m/z* (%) 474.0552 (62) $[M+Na]^+$, 925.1201 (100) $[2M+Na]^+$.

***N*-benzyl-*N*-(2-iodylphenyl)pivalamide 16e.** General procedure afforded 0.410 g (97%) of **16e** as a white solid. M.p. 105-106^{o}C; IR (NaCl): 2954, 1643, 1464, 1365, 1271, 1175, 974, 772 cm^{-1}; ^{1}H NMR (300 MHz, [D_6]DMSO-CD_3OD) δ 7.97 (dd, ^{3}J = 8.0 Hz, ^{4}J=1.5 Hz, 1H), 7.55 (td, ^{3}J = 8.1 Hz, ^{4}J=1.5 Hz, 1H), 7.46-7.20 (m, 6H), 7.03 (dd, ^{3}J = 8.0 Hz, ^{4}J=1.2 Hz, 1H), 5.15 (d, ^{2}J = 14.8 Hz, 1H), 4.08 (d, ^{2}J = 14.8 Hz, 1H), 1.14 (s, 9H); ^{13}C NMR (75.5 MHz, [D_6]DMSO-CD_3OD) d 179.1, 146.0, 141.1, 137.4, 132.6, 129.9, 129.3, 129.0, 127.9, 128.9, 126.8, 54.1, 41.5, 29.2; ESI-HRMS: *m/z* (%) 448.0370 (70) $[M+Na]^+$, 873.0806 (100) $[2M+Na]^+$.

1-(2-Iodylphenyl)pyrrolidin-2-one 17. General procedure afforded 0.247 g (77%) of **17e** as a snow-white solid. M.p. 180^{o}C (decomp.); IR (NaCl): 3274, 1643, 1581, 1455, 1410, 1329, 1233, 1088, 946, 768 cm^{-1}; ^{1}H NMR (300 MHz, CD_3OD) δ 7.96 (dd, ^{3}J = 7.8 Hz, ^{4}J=1.2 Hz, 1H), 7.61 (td, ^{3}J = 8.1 Hz, ^{4}J=1.2 Hz, 1H), 7.48 (td, ^{3}J = 8.0 Hz, ^{4}J=1.2 Hz, 1H), 7.38 (dd, ^{3}J = 8.2 Hz,

^{4}J=1.2 Hz, 1H), 4.00 (t, ^{3}J = 6.8 Hz, 2H), 2.59 (t, ^{3}J = 7.7 Hz, 2H), 2.12 (quintet, ^{3}J = 7.4 Hz, 2H); ^{13}C NMR (75.5 MHz, [D_6]DMSO-CD_3OD) d 176.4, 142.7, 135.6, 132.5, 128.2, 125.9, 121.2, 49.9, 32.2, 17.9; ESI-HRMS: *m/z* (%) 341.9619 (100) $[M+Na]^+$, 660.9937 (34) $[2M+Na]^+$.

General procedure for oxidation with amides 14-17. Solution/suspension of the respective iodylarene (0.05 mmol) and BnOH (10.3 μL, 0.1 mmol) or *p*-TolSMe (15.4 mg, 0.1 mmol) in 0.3 ml MeCN was brought to reflux on a preheated stirring plate. The reaction mixture was stirred for exactly 20 min. The resulting reaction mixture was quickly cooled with ice bath, and solution was analyzed by GC-MS. Conversions were determined after prior GC column calibration using authentic samples of reactants and products.

Preparation of aldehyde 27. A mixture of thioalcohol **26** (0.192 g, 1.25 mmol), amide **15d** (0.468, 1.25 mmol) and MeSEt (0.112 ml, 1.25 mmol) in 7.5 ml DCE was refluxed for 2.5 hours. Then DCE was removed under reduced pressure and the residue was separated by DCVC (hexanes/$CHCl_3$ solvent mixture) to give pure **27** (0.154 g, 81%) as a clear oil. ^{1}H NMR (300 MHz, $CDCl_3$): δ 9.92 (s, 1H), 7.78 (dd, ^{3}J = 6.6 Hz, ^{4}J=2.1 Hz, 2H), 7.32 (dd, ^{3}J = 6.9 Hz, ^{4}J=1.8 Hz, 2H), 2.54 (s, 3H); ^{13}C NMR (75.5 MHz, $CDCl_3$) d 191.6, 148.2, 133.2, 130.2, 129.1, 125.4, 14.9.

Preparation of aldehyde 29. A mixture of thioalcohol **28** (0.150 g, 0.151, 1.25 mmol), amide **15d** (0.468, 1.25 mmol) and MeSEt (0.112 ml, 1.25 mmol) in 7.5 ml DCE was refluxed for 2.5 hours. Then DCE was removed under reduced pressure and the residue was separated by DCVC (hexanes/$CHCl_3$ solvent mixture) to give pure **29** (0.126 g, 85%) as a clear oil with spicy odor. ^{1}H NMR (300 MHz, $CDCl_3$): δ 9.83 (t, ^{3}J = 1.2 Hz, 1H), 2.64-2.53 (m, 4H), 2.10 (s, 3H), 1.96 (quintet, ^{3}J = 7.2 Hz, 2H); ^{13}C NMR (75.5 MHz, $CDCl_3$) d 201.8, 42.6, 33.6, 21.4, 15.3.