



Supporting Information

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Palladium-Catalyzed Intramolecular Nucleophilic Substitution at the Alkoxycarbonyl Group

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General Methods. ^1H - and ^{13}C NMR spectra were recorded in CDCl_3 solution, using Me_4Si as internal standard. ^{31}P NMR spectra were recorded in CDCl_3 with external H_3PO_4 as reference. Chemical shifts are reported in ppm downfield (δ) from Me_4Si . TLC was carried out on SiO_2 (silica gel 60 F₂₅₄, Merck), and the spots were located with UV light, iodoplatinate reagent or 1% aqueous KMnO_4 . Flash chromatography was carried out on SiO_2 (silica gel 60, SDS, 230-400 mesh ASTM). Drying of organic extracts during workup of reactions was performed over anhydrous MgSO_4 . Evaporation of solvents was accomplished with a rotatory evaporator. Microanalyses were performed by the Centro de Investigación y Desarrollo (CSIC), Barcelona.

Experimental procedures and characterization data for the starting materials:

Methyl 3-[*N*-(2-iodo-4-methylphenyl)-*N*-methylamino]propionate (**1a**)

A solution of *p*-toluidine (0.5 g, 4.67 mmol) and methyl acrylate (0.34 mL, 3.73 mmol) in acetic acid (0.25 mL) was stirred at 90°C in a sealed tube for 24 h.¹ The reaction mixture was poured into cooled (ice) water, basified with Na_2CO_3 , and extracted with Et_2O . The organic extracts were washed with saturated aqueous Na_2CO_3 solution, dried, and concentrated. The residue was dissolved in a CH_2Cl_2 (18 mL)-MeOH (9 mL) mixture, and CaCO_3 (0.53 g, 5.30 mmol) and BTMAICl_2 ² (1.57 g, 4.48 mmol) were added. The mixture was stirred at room temperature for 6 h and then filtered. The solution was concentrated and the residue was partitioned between water and EtOAc. The organic layer was washed with brine, dried and concentrated. The residue was purified by flash chromatography (SiO_2 , from hexanes to 4:1 hexanes-EtOAc) to give methyl 3-[*N*-(2-iodo-4-methylphenyl)amino]propionate (892 mg, 75%).

A mixture of methyl 3-[*N*-(2-iodo-4-methylphenyl)amino]propionate (892 mg, 2.80 mmol), K_2CO_3 (774 mg, 5.60 mmol), and iodomethane (1.42 mL, 22.4 mmol) in acetonitrile (5 mL) was stirred at 50°C in a sealed tube for 72 h. The solvent was removed *in vacuo*, and the residue was partitioned between water and CH_2Cl_2 . The organic layer was dried and concentrated. The residue was purified by flash chromatography (SiO_2 , CH_2Cl_2) to give methyl 3-[*N*-(2-iodo-4-methylphenyl)-*N*-methylamino]propionate³ (**1a**, 694 mg, 74%).

Methyl 3-[*N*-(2-bromo-4-methylphenyl)-*N*-methylamino]propionate (**1b**)

A solution of 2-bromo-4-methylaniline (0.33 mL, 2.6 mmol) and methyl acrylate (0.7 mL, 7.8 mmol) in acetic acid (0.25 mL) was stirred at 90°C in a sealed tube for 24 h. The reaction mixture was poured into cooled (ice) water, basified with Na_2CO_3 , and extracted with Et_2O .

¹ Johnson, W. S.; Woroch, E. L.; Buell, B. G. *J. Am. Chem. Soc.* **1949**, *71*, 1901-1905.

² Kajigaeshi, S.; Kakinami, T.; Yamasaki, H.; Fujisaki, S.; Okamoto, T. *Bull. Chem. Soc. Jpn* **1988**, *61*, 600-602.

³ Solé, D.; Díaz, S.; Solans, X.; Font-Bardia, M. *Organometallics* **2006**, *25*, 1995-2001.

The organic extracts were washed with saturated aqueous Na_2CO_3 solution, dried, and concentrated. The residue was dissolved in acetonitrile (6 mL) and K_2CO_3 (0.72 g, 5.2 mmol) and iodomethane (0.6 mL, 9.6 mmol) were added. The mixture was stirred at 50°C in a sealed tube for 5 days. The solvent was removed *in vacuo*, and the residue was partitioned between water and CH_2Cl_2 . The organic layer was dried and concentrated. The residue was purified by flash chromatography (SiO_2 , from hexanes to hexanes-EtOAc 4%) to give methyl 3-[*N*-(2-bromo-4-methylphenyl)-*N*-methylamino]propionate (**1b**, 0.3 g, 40%).

^1H NMR (CDCl_3 , 300 MHz) δ 2.27 (s, 3H), 2.54 (t, J = 7.5 Hz, 2H), 2.73 (s, 3H), 3.32 (t, J = 7.5 Hz, 2H), 3.64 (s, 3H), 6.99 (d, J = 8.1 Hz, 1H), 7.05 (dd, J = 8.1 and 1.8 Hz, 1H), 7.38 (d, J = 1.8 Hz, 1H). ^{13}C NMR (CDCl_3 , 75.4 MHz) δ 20.3 (CH_3), 32.5 (CH_2), 41.7 (CH_3), 51.5 (CH_3), 51.6 (CH_2), 120.5 (C), 122.0 (CH), 128.6 (CH), 134.1 (CH), 134.6 (C), 147.7 (C), 172.6 (C).

Methyl 3-[*N*-(2-iodo-4-methoxyphenyl)-*N*-methylamino]propionate (4a)

A solution of 2-iodo-4-methoxyaniline (0.5 g, 2 mmol) and methyl acrylate (0.54 mL, 6 mmol) in acetic acid (0.25 mL) was stirred at 90°C in a sealed tube for 24 h. The reaction mixture was poured into cooled (ice) water, basified with Na_2CO_3 , and extracted with Et_2O . The organic extracts were washed with saturated aqueous Na_2CO_3 solution, dried, and concentrated. The residue was purified by flash chromatography (SiO_2 , from hexanes to CH_2Cl_2) to give methyl 3-[*N*-(2-iodo-4-methoxyphenyl)amino]propionate (417 mg, 62%).

A mixture of methyl 3-[*N*-(2-iodo-4-methoxyphenyl)amino]propionate (417 mg, 1.24 mmol), K_2CO_3 (344 mg, 2.49 mmol), and iodomethane (0.62 mL, 9.95 mmol) in acetonitrile (6 mL) was stirred at 50°C in a sealed tube for 72 h. The solvent was removed *in vacuo*, and the residue was partitioned between water and CH_2Cl_2 . The organic layer was dried and concentrated. The residue was purified by flash chromatography (SiO_2 , from hexanes to 1:1 hexanes-EtOAc) to give methyl 3-[*N*-(2-iodo-4-methoxyphenyl)-*N*-methylamino]propionate (**4a**, 435 mg, quantitative).

^1H NMR (CDCl_3 , 300 MHz) δ 2.49 (t, J = 7.5 Hz, 2H), 2.64 (s, 3H), 3.22 (t, J = 7.5 Hz, 2H), 3.66 (s, 3H), 3.76 (s, 3H), 6.88 (dd, J = 8.7 and 3 Hz, 1H), 7.04 (d, J = 8.7 Hz, 1H), 7.38 (d, J = 3 Hz, 1H). ^{13}C NMR (CDCl_3 , 75.4 MHz) δ 32.9 (CH_2), 43.4 (CH_3), 51.7 (CH_3), 52.5 (CH_2), 55.6 (CH_3), 100.8 (C), 115.0 (CH), 122.5 (CH), 124.4 (CH), 146.4 (C), 156.8 (C), 172.7 (C). HRMS (ESI-TOF) calcd for $\text{C}_{12}\text{H}_{17}\text{INO}_3$: 350.0248 $[\text{M}+\text{H}]^+$; found: 350.0233.

Methyl 3-[*N*-(2-iodo-4-methoxycarbonylphenyl)amino]propionate

A solution of methyl *p*-aminobenzoate (1 g, 6.62 mmol) and methyl acrylate (0.66 mL, 7.28 mmol) in acetic acid (0.5 mL) was stirred at 90°C in a sealed tube for 24 h. The reaction mixture was poured into cooled (ice) water, basified with Na_2CO_3 , and extracted with Et_2O . The organic extracts were washed with saturated aqueous Na_2CO_3 solution, dried, and

concentrated. The residue was dissolved in a CH₂Cl₂ (12 mL)-MeOH (6 mL) mixture, and CaCO₃ (793 mg, 7.92 mmol) and BTMAICl₂ (2.55 g, 7.31 mmol) were added. The mixture was stirred at room temperature for 24 h and then filtered. The solution was concentrated and the residue was partitioned between water and EtOAc. The organic layer was washed with brine, dried, and concentrated. The residue was purified by flash chromatography (SiO₂, from hexanes to 3:1 hexanes-EtOAc) to give methyl 3-[*N*-(2-iodo-4-methoxycarbonylphenyl)amino]propionate (984 mg, 41%).

¹H NMR (CDCl₃, 300 MHz) δ 2.68 (t, *J* = 6.6 Hz, 2H), 3.57 (broad t, *J* = 6.6 Hz, 2H), 3.74 (s, 3H), 3.86 (s, 3H), 5.04 (broad, 1H), 6.53 (d, *J* = 8.7 Hz, 1H), 7.90 (dd, *J* = 8.7 and 2.1 Hz, 1H), 8.35 (d, *J* = 2.1 Hz, 1H). ¹³C NMR (CDCl₃, 75.4 MHz) δ 33.3 (CH₂), 39.2 (CH₂), 51.3 (CH₃), 51.9 (CH₃), 83.9 (C), 108.5 (CH), 120.0 (C), 131.4 (CH), 140.8 (CH), 149.9 (C), 165.7 (C), 172.0 (C).

Methyl 3-[*N*-(2-iodo-4-methoxycarbonylphenyl)-*N*-methylamino]propionate (4b**)**

A mixture of methyl 3-[*N*-(2-iodo-4-methoxycarbonylphenyl)amino]propionate (984 mg, 2.71 mmol), K₂CO₃ (750 mg, 5.42 mmol), and iodomethane (1.7 mL, 27.1 mmol) in acetonitrile (6 mL) was stirred at 90°C in a sealed tube for 72 h. The solvent was removed *in vacuo*, and the residue was partitioned between water and CH₂Cl₂. The organic layer was dried and concentrated. The residue was purified by flash chromatography (SiO₂, CH₂Cl₂) to give methyl 3-[*N*-(2-iodo-4-methoxycarbonylphenyl)-*N*-methylamino]propionate (**4b**, 645 mg, 63%) and methyl 3-[*N*-(2-iodo-4-methoxycarbonylphenyl)amino]propionate (266 mg, 27%).

¹H NMR (CDCl₃, 300 MHz) δ 2.61 (t, *J* = 7.5 Hz, 2H), 2.79 (s, 3H), 3.41 (t, *J* = 7.5 Hz, 2H), 3.66 (s, 3H), 3.89 (s, 3H), 7.05 (d, *J* = 8.4 Hz, 1H), 7.97 (dd, *J* = 8.4 and 2.1 Hz, 1H), 8.50 (d, *J* = 2.1 Hz, 1H). ¹³C NMR (CDCl₃, 75.4 MHz) δ 32.7 (CH₂), 41.8 (CH₃), 51.5 (CH₂), 51.8 (CH₃), 52.1 (CH₃), 96.1 (C), 120.9 (CH), 126.4 (C), 130.4 (CH), 141.8 (CH), 157.7 (C), 165.4 (C), 172.3 (C). HRMS (ESI-TOF) calcd for C₁₃H₁₇INO₄: 378.0196 [M+H]⁺; found: 378.0198.

Benzyl 3-[*N*-(4-methylphenyl)amino]propionate

A solution of *p*-toluidine (0.5 g, 4.67 mmol) and benzyl acrylate (758 mg, 4.67 mmol) in acetic acid (0.25 mL) was stirred at 90°C in a sealed tube for 24 h. The reaction mixture was poured into cooled (ice) water, basified with Na₂CO₃, and extracted with Et₂O. The organic extracts were washed with saturated aqueous Na₂CO₃ solution, dried, and concentrated. The residue was purified by flash chromatography (SiO₂, from hexanes to hexanes-EtOAc 6%) to give benzyl 3-[*N*-(4-methylphenyl)amino]propionate (635 mg, 51%).

¹H NMR (CDCl₃, 300 MHz) δ 2.23 (s, 3H), 2.64 (t, *J* = 6.3 Hz, 2H), 3.44 (t, *J* = 6.3 Hz, 2H), 3.84 (broad, 1H), 5.13 (s, 2H), 6.52 (d, *J* = 8.7 Hz, 2H), 6.98 (d, *J* = 8.7 Hz, 2H), 7.30-7.42 (m, 5H). ¹³C NMR (CDCl₃, 75.4 MHz) δ 20.3 (CH₃), 33.9 (CH₂), 39.8 (CH₂), 66.3 (CH₂), 113.2

(CH), 126.9 (C), 128.1 (CH), 128.2 (CH), 128.5 (CH), 129.7 (CH), 135.7 (C), 145.1 (C), 172.1 (C).

Benzyl 3-[*N*-(2-iodo-4-methylphenyl)amino]propionate

To a solution of benzyl 3-[*N*-(4-methylphenyl)amino]propionate (635 mg, 2.36 mmol) in a CH₂Cl₂ (12 mL)-MeOH (6 mL) mixture were added CaCO₃ (307 mg, 3.07 mmol) and BTMAICl₂ (904 mg, 2.60 mmol). The mixture was stirred at room temperature for 6 h and then filtered. The solution was concentrated and the residue was partitioned between water and EtOAc. The organic layer was washed with brine, dried and concentrated. The residue was purified by flash chromatography (SiO₂, from hexanes to 85:15 hexanes-EtOAc) to give benzyl 3-[*N*-(2-iodo-4-methylphenyl)amino]propionate (870 mg, 93%).

¹H NMR (CDCl₃, 300 MHz) δ 2.20 (s, 3H), 2.69 (t, *J* = 6.6 Hz, 2H), 3.49 (q, *J* = 6.6 Hz, 2H), 4.33 (broad t, *J* = 6.6 Hz, 1H), 5.16 (s, 2H), 6.50 (d, *J* = 8.1 Hz, 1H), 7.01 (dd, *J* = 8.1 and 1.5 Hz, 1H), 7.30-7.40 (m, 5H), 7.50 (d, *J* = 1.5 Hz, 1H). ¹³C NMR (CDCl₃, 75.4 MHz) δ 19.8 (CH₃), 34.0 (CH₂), 40.0 (CH₂), 66.6 (CH₂), 85.8 (C), 110.6 (CH), 128.3 (2 CH), 128.5 (C), 128.6 (CH), 130.0 (CH), 135.7 (C), 139.4 (CH), 144.5 (C), 171.8 (C).

Benzyl 3-[*N*-(2-iodo-4-methylphenyl)-*N*-methylamino]propionate (4c)

A mixture of benzyl 3-[*N*-(2-iodo-4-methylphenyl)amino]propionate (870 mg, 2.2 mmol), K₂CO₃ (608 mg, 4.4 mmol), and iodomethane (1.1 mL, 17.6 mmol) in acetonitrile (7 mL) was stirred at 50°C in a sealed tube for 72 h. The solvent was removed *in vacuo*, and the residue was partitioned between water and CH₂Cl₂. The organic layer was dried and concentrated. The residue was purified by flash chromatography (SiO₂, CH₂Cl₂) to give benzyl 3-[*N*-(2-iodo-4-methylphenyl)-*N*-methylamino]propionate (**4c**, 825 mg, 92%).

¹H NMR (CDCl₃, 300 MHz) δ 2.26 (s, 3H), 2.56 (t, *J* = 7.5 Hz, 2H), 2.67 (s, 3H), 3.29 (t, *J* = 7.5 Hz, 2H), 5.10 (s, 2H), 6.98 (d, *J* = 8.1 Hz, 1H), 7.10 (dd, *J* = 8.1 and 1.5 Hz, 1H), 7.30-7.38 (m, 5H), 7.67 (d, *J* = 1.5 Hz, 1H). ¹³C NMR (CDCl₃, 75.4 MHz) δ 20.2 (CH₃), 33.1 (CH₂), 43.0 (CH₃), 52.1 (CH₂), 66.3 (CH₂), 99.8 (C), 121.9 (CH), 128.1 (CH), 128.2 (CH), 128.5 (CH), 129.7 (CH), 135.8 (C), 135.9 (C), 140.2 (CH), 150.8 (C), 172.0 (C). HRMS (ESI-TOF) calcd for C₁₈H₂₁INO₂: 410.0612 [M+H]⁺; found: 410.0600.

Methyl 2-methyl-3-[*N*-(4-methylphenyl)amino]propionate

A solution of *p*-toluidine (1 g, 9.34 mmol) and methyl methacrylate (1.1 mL, 10.3 mmol) in acetic acid (0.5 mL) was stirred at 90°C in a sealed tube for 24 h. The reaction mixture was poured into cooled (ice) water, basified with Na₂CO₃, and extracted with Et₂O. The organic extracts were washed with saturated aqueous Na₂CO₃ solution, dried, and concentrated. The

residue was purified by flash chromatography (SiO₂, from hexanes to 1:1 hexanes-EtOAc) to give methyl 2-methyl-3-[N-(4-methylphenyl)amino]propionate (332 mg, 17%).

¹H NMR (CDCl₃, 300 MHz) δ 1.22 (d, *J* = 6.9 Hz, 3H), 2.23 (s, 3H), 2.80 (m, 1H), 3.19 (dd, *J* = 13.2 and 5.4 Hz, 1H), 3.39 (dd, *J* = 13.2 and 7.8 Hz, 1H), 3.69 (s, 3H), 3.82 (broad, 1H), 6.53 (d, *J* = 8.1 Hz, 2H), 6.98 (d, *J* = 8.1 Hz, 2H). ¹³C NMR (CDCl₃, 75.4 MHz) δ 15.0 (CH₃), 20.3 (CH₃), 39.2 (CH), 47.3 (CH₂), 51.7 (CH₃), 113.1 (CH), 126.7 (C), 129.7 (CH), 145.4 (C), 175.8 (C).

Methyl 3-[N-(2-iodo-4-methylphenyl)amino]-2-methylpropionate

To a solution of methyl 2-methyl-3-[N-(4-methylphenyl)amino]propionate (332 mg, 1.60 mmol) in a CH₂Cl₂ (10 mL)-MeOH (5 mL) mixture were added CaCO₃ (208 mg, 2.08 mmol) and BTMAI₂ (613 mg, 1.76 mmol). The mixture was stirred at room temperature for 6 h and then filtered. The solution was concentrated and the residue was partitioned between water and EtOAc. The organic layer was washed with brine, dried and concentrated. The residue was purified by flash chromatography (SiO₂, CH₂Cl₂) to give methyl 3-[N-(2-iodo-4-methylphenyl)amino]-2-methylpropionate (410 mg, 77%).

¹H NMR (CDCl₃, 300 MHz) δ 1.24 (d, *J* = 7.2 Hz, 3H), 2.20 (s, 3H), 2.83 (m, 1H), 3.24 (dd, *J* = 13.2 and 6 Hz, 1H), 3.43 (dd, *J* = 13.2 and 7.8 Hz, 1H), 3.72 (s, 3H), 4.40 (broad, 1H), 6.49 (d, *J* = 8.1 Hz, 1H), 7.01 (dd, *J* = 8.1 and 2.1 Hz, 1H), 7.49 (d, *J* = 2.1 Hz, 1H). ¹³C NMR (CDCl₃, 75.4 MHz) δ 15.0 (CH₃), 19.8 (CH₃), 38.9 (CH), 47.4 (CH₂), 51.9 (CH₃), 85.7 (C), 110.6 (CH), 128.4 (C), 130.0 (CH), 139.4 (CH), 144.5 (C), 175.4 (C).

Methyl 3-[N-(2-iodo-4-methylphenyl)-N-methylamino]-2-methylpropionate (7)

A mixture of methyl 3-[N-(2-iodo-4-methylphenyl)amino]-2-methylpropionate (410 mg, 1.23 mmol), K₂CO₃ (340 mg, 2.46 mmol), and iodomethane (0.6 mL, 9.84 mmol) in acetonitrile (5 mL) was stirred at 50°C in a sealed tube for 72 h. The solvent was removed *in vacuo*, and the residue was partitioned between water and CH₂Cl₂. The organic layer was dried and concentrated. The residue was purified by flash chromatography (SiO₂, CH₂Cl₂) to give methyl 3-[N-(2-iodo-4-methylphenyl)-N-methylamino]-2-methylpropionate (**7**, 400 mg, 94%).

¹H NMR (CDCl₃, 300 MHz) δ 1.18 (d, *J* = 6.9 Hz, 3H), 2.26 (s, 3H), 2.64 (s, 3H), 2.65 (m, 1H), 2.97 (dd, *J* = 12.6 and 6.6 Hz, 1H), 3.28 (dd, *J* = 12.6 and 8.1 Hz, 1H), 3.67 (s, 3H), 6.99 (d, *J* = 8.1 Hz, 1H), 7.11 (dd, *J* = 8.1 and 1.8 Hz, 1H), 7.67 (d, *J* = 1.8 Hz, 1H). ¹³C NMR (CDCl₃, 75.4 MHz) δ 15.5 (CH₃), 20.2 (CH₃), 38.5 (CH), 44.3 (CH₃), 51.8 (CH₃), 59.0 (CH₂), 99.8 (C), 122.0 (CH), 129.8 (CH), 135.8 (C), 140.2 (CH), 151.2 (C), 176.1 (C). HRMS (ESI-TOF) calcd for C₁₃H₁₉INO₂: 348.0455 [M+H]⁺; found: 348.0453.

Methyl 2,2-dimethyl-3-[N-(4-methylphenyl)amino]propionate

Following the previously described protocol for the preparation of closely related products,⁴ to a solution of SDS (625 mg, 2.16 mmol) in water (29 mL) were successfully added *p*-toluidine (580 mg, 5.41 mmol), formaldehyde (37 wt. % in water, 0.45 mL), 1-methoxy-1-trimethylsiloxy-2-methylpropene (3.3 mL, 14.3 mmol), and 48% aqueous HBF₄ solution (0.1 mL, 0.55 mmol) at 0°C. After being stirred at the same temperature for 30 min, Dowex 1-X8 (100-200 mesh, Cl⁻ form) and water were added to quench the reaction, and the reaction mixture was further stirred for 10 min. The mixture was extracted with CH₂Cl₂ and the combined organic extracts were washed with brine, dried, and concentrated to give methyl 2,2-dimethyl-3-[N-(4-methylphenyl)amino]propionate (1.20 g, quantitative), which was used in the next step without purification.

¹H NMR (CDCl₃, 300 MHz) δ 1.26 (s, 6H), 2.22 (s, 3H), 3.21 (s, 2H), 3.67 (s, 3H), 3.80 (broad, 1H), 6.55 (d, *J* = 8.1 Hz, 2H), 6.97 (d, *J* = 8.1 Hz, 2H). ¹³C NMR (CDCl₃, 75.4 MHz) δ 20.3 (CH₃), 23.5 (CH₃), 43.7 (C), 52.0 (CH₃), 53.2 (CH₂), 113.1 (CH), 126.5 (C), 129.6 (CH), 146.2 (C), 177.5 (C).

Methyl 3-[N-(2-iodo-4-methylphenyl)-N-methylamino]-2,2-dimethylpropionate (10a)

To a solution of methyl 2,2-dimethyl-3-[N-(4-methylphenyl)amino]propionate (1.20 g, 5.41 mmol) in a CH₂Cl₂ (23 mL)-MeOH (10 mL) mixture were added CaCO₃ (720 mg, 7.2 mmol) and BTMAICl₂ (1.88 g, 5.41 mmol). The mixture was stirred at room temperature for 6 h and then filtered. The solution was concentrated and the residue was partitioned between water and EtOAc. The organic layer was washed with brine, dried, and concentrated to give methyl 3-[N-(2-iodo-4-methylphenyl)amino]-2,2-dimethylpropionate (1.64 g, 87%), which was used in the next step without purification.

A mixture of methyl 3-[N-(2-iodo-4-methylphenyl)amino]-2,2-dimethylpropionate (1.53 g, 4.40 mmol), K₂CO₃ (1.22 g, 8.80 mmol), and iodomethane (2.2 mL, 35.3 mmol) in acetonitrile (15 mL) was stirred at 50°C in a sealed tube for 72 h. Iodomethane (2.2 mL, 35.3 mmol) was added and the mixture was stirred at 50°C in a sealed tube for an additional 72 h. The solvent was removed *in vacuo*, and the residue was partitioned between water and CH₂Cl₂. The organic layer was dried and concentrated. The residue was purified by flash chromatography (SiO₂, from hexanes to 8:2 hexanes-EtOAc) to give methyl 3-[N-(2-iodo-4-methylphenyl)-N-methylamino]-2,2-dimethylpropionate (**10a**, 1.10 g, 69%).

¹H NMR (CDCl₃, 300 MHz) δ 1.18 (s, 6H), 2.25 (s, 3H), 2.64 (s, 3H), 3.24 (s, 2H), 3.56 (s, 3H), 7.05-7.13 (m, 2H), 7.66 (m, 1H). ¹³C NMR (CDCl₃, 75.4 MHz) δ 20.2 (CH₃), 23.9 (CH₃), 44.6 (C), 46.8 (CH₃), 51.6 (CH₃), 65.1 (CH₂), 100.5 (C), 123.7 (CH), 129.9 (CH), 135.6 (C),

⁴ Akiyama, T.; Takaya, J.; Kagoshima, H. *Adv. Synth. Catal.* **2002**, *344*, 338-347.

140.0 (CH), 153.1 (C), 177.7 (C). HRMS (ESI-TOF) calcd for $C_{14}H_{21}INO_2$: 362.0612 $[M+H]^+$; found: 362.0604.

Methyl 3-[*N*-(2-iodo-4-methoxyphenyl)amino]-2,2-dimethylpropionate

To a solution of SDS (115 mg, 0.40 mmol) in water (5 mL) were successfully added 2-iodo-4-methoxyaniline (250 mg, 1 mmol), formaldehyde (37 wt. % in water, 80 μ L), 1-methoxy-1-trimethylsiloxy-2-methylpropene (0.6 mL, 3 mmol), and 48% aqueous HBF_4 solution (18 μ L, 0.1 mmol) at 0°C. After being stirred overnight at room temperature, Dowex 1-X8 (100-200 mesh, Cl^- form) and water were added to quench the reaction, and the reaction mixture was further stirred for 10 min. The mixture was extracted with CH_2Cl_2 and the combined organic extracts were washed with brine, dried, and concentrated. The residue was purified by flash chromatography (SiO_2 , CH_2Cl_2) to give methyl 3-[*N*-(2-iodo-4-methoxyphenyl)amino]-2,2-dimethylpropionate (192 mg, 53%).

1H NMR ($CDCl_3$, 300 MHz) δ 1.30 (s, 6H), 3.20 (s, 2H), 3.70 (s, 3H), 3.72 (s, 3H), 4.13 (broad, 1H), 6.55 (d, J = 8.7 Hz, 1H), 6.82 (dd, J = 8.7 and 3 Hz, 1H), 7.26 (d, J = 3 Hz, 1H). ^{13}C NMR ($CDCl_3$, 75.4 MHz) δ 23.6 (CH_3), 43.5 (C), 52.1 (CH_3), 53.9 (CH_2), 56.0 (CH_3), 85.8 (C), 111.6 (CH), 115.6 (CH), 124.4 (CH), 142.2 (C), 151.8 (C), 177.1 (C).

Methyl 3-[*N*-(2-iodo-4-methoxyphenyl)-*N*-methylamino]-2,2-dimethylpropionate (10b)

A mixture of methyl 3-[*N*-(2-iodo-4-methoxyphenyl)amino]-2,2-dimethylpropionate (192 mg, 0.53 mmol), K_2CO_3 (146 mg, 1.1 mmol), and iodomethane (0.26 mL, 4.2 mmol) in acetonitrile (5 mL) was stirred at 50°C in a sealed tube for 72 h. Iodomethane (0.26 mL, 4.2 mmol) was added and the mixture was stirred at 50°C in a sealed tube for an additional 72 h. The solvent was removed *in vacuo*, and the residue was partitioned between water and CH_2Cl_2 . The organic layer was dried and concentrated. The residue was purified by flash chromatography (SiO_2 , from hexanes to hexanes-EtOAc 2%) to give methyl 3-[*N*-(2-iodo-4-methoxyphenyl)-*N*-methylamino]-2,2-dimethylpropionate (**10b**, 140 mg, 70%).

1H NMR ($CDCl_3$, 300 MHz) δ 1.18 (s, 6H), 2.61 (s, 3H), 3.20 (s, 2H), 3.55 (s, 3H), 3.75 (s, 3H), 6.87 (dd, J = 8.7 and 3 Hz, 1H), 7.11 (d, J = 8.7 Hz, 1H), 7.36 (d, J = 3 Hz, 1H). ^{13}C NMR ($CDCl_3$, 75.4 MHz) δ 24.0 (CH_3), 44.5 (C), 47.0 (CH_3), 51.7 (CH_3), 55.6 (CH_3), 65.6 (CH_2), 101.2 (C), 115.2 (CH), 124.1 (CH), 124.4 (CH), 148.8 (C), 156.5 (C), 177.7 (C). HRMS (ESI-TOF) calcd for $C_{14}H_{21}INO_3$: 378.0561 $[M+H]^+$; found: 378.0553.

Methyl 3-[*N*-(4-methoxycarbonylphenyl)amino]-2,2-dimethylpropionate

To a solution of SDS (575 mg, 2 mmol) in water (25 mL) were successfully added methyl 4-aminobenzoate (750 mg, 4.96 mmol), formaldehyde (37 wt. % in water, 0.4 mL), 1-methoxy-1-trimethylsiloxy-2-methylpropene (3 mL, 15 mmol), and 48% aqueous HBF_4 solution (90 μ L,

0.5 mmol) at 0°C. After being stirred at room temperature for 4 h, Dowex 1-X8 (100-200 mesh, Cl⁻ form) and water were added to quench the reaction, and the reaction mixture was further stirred for 10 min. The mixture was extracted with CH₂Cl₂ and the combined organic extracts were washed with brine, dried, and concentrated. The residue was purified by flash chromatography (SiO₂, from hexanes to hexanes-EtOAc 7:3) to give methyl 3-[N-(4-methoxycarbonylphenyl)amino]-2,2-dimethylpropionate (507 mg, 39%).

¹H NMR (CDCl₃, 300 MHz) δ 1.26 (s, 6H), 3.29 (s, 2H), 3.67 (s, 3H), 3.83 (s, 3H), 4.53 (broad, 1H), 6.57 (d, *J* = 8.7 Hz, 2H), 7.83 (d, *J* = 8.7 Hz, 2H). ¹³C NMR (CDCl₃, 75.4 MHz) δ 23.4 (CH₃), 43.7 (C), 51.4 (CH₃), 51.5 (CH₂), 52.0 (CH₃), 111.4 (CH), 118.2 (C), 131.4 (CH), 152.1 (C), 167.1 (C), 177.1 (C).

Methyl 3-[N-(2-iodo-4-methoxycarbonylphenyl)amino]-2,2-dimethylpropionate

To a solution of methyl 3-[N-(4-methoxycarbonylphenyl)amino]-2,2-dimethylpropionate (507 mg, 1.9 mmol) in a CH₂Cl₂ (12 mL)-MeOH (5 mL) mixture were added CaCO₃ (250 mg, 2.5 mmol) and BTMAICl₂ (0.8 g, 2.3 mmol). After stirring at room temperature for 24 h, CaCO₃ (250 mg, 2.5 mmol) and BTMAICl₂ (0.4 g, 1.15 mmol) were added. The mixture was stirred at room temperature for 6 h and then filtered. The solution was concentrated and the residue was partitioned between water and EtOAc. The organic layer was washed with brine, dried, and concentrated. The residue was purified by flash chromatography (SiO₂, CH₂Cl₂) to give methyl 3-[N-(2-iodo-4-methoxycarbonylphenyl)amino]-2,2-dimethylpropionate (582 mg, 78%).

¹H NMR (CDCl₃, 300 MHz) δ 1.31 (s, 6H), 3.32 (s, 2H), 3.72 (s, 3H), 3.85 (s, 3H), 5.15 (broad, 1H), 6.54 (d, *J* = 8.4 Hz, 1H), 7.87 (dd, *J* = 8.4 and 1.8 Hz, 1H), 8.33 (d, *J* = 1.8 Hz, 1H). ¹³C NMR (CDCl₃, 75.4 MHz) δ 23.5 (CH₃), 43.5 (C), 51.7 (CH₃), 52.1 (CH₂), 52.3 (CH₃), 84.0 (C), 108.8 (CH), 119.7 (C), 131.4 (CH), 140.8 (CH), 150.6 (C), 165.9 (C), 176.8 (C).

Methyl 3-[N-(2-iodo-4-methoxycarbonylphenyl)-N-methylamino]-2,2-dimethylpropionate (10c)

A mixture of methyl 3-[N-(2-iodo-4-methoxycarbonylphenyl)amino]-2,2-dimethylpropionate (563 mg, 1.44 mmol), K₂CO₃ (400 mg, 2.89 mmol), and iodomethane (0.9 mL, 14.5 mmol) in acetonitrile (10 mL) was stirred at 80°C in a sealed tube for 72 h. Iodomethane (0.9 mL, 14.5 mmol) was added and the mixture was stirred at 80°C in a sealed tube for an additional 72 h. The solvent was removed *in vacuo*, and the residue was partitioned between water and CH₂Cl₂. The organic layer was dried and concentrated. The residue was purified by flash chromatography (SiO₂, from hexanes to hexanes-EtOAc 8%) to give methyl 3-[N-(2-iodo-4-methoxycarbonylphenyl)-N-methylamino]-2,2-dimethylpropionate (**10c**, 345 mg, 59%) and

methyl 3-[*N*-(2-iodo-4-methoxycarbonylphenyl)amino]-2,2-dimethylpropionate (225 mg, 40%).

¹H NMR (CDCl₃, 300 MHz) δ 1.18 (s, 6H), 2.78 (s, 3H), 3.38 (s, 2H), 3.55 (s, 3H), 3.88 (s, 3H), 7.18 (d, *J* = 8.4 Hz, 1H), 7.95 (dd, *J* = 8.4 and 2.1 Hz, 1H), 8.49 (d, *J* = 2.1 Hz, 1H). ¹³C NMR (CDCl₃, 75.4 MHz) δ 23.8 (CH₃), 44.7 (C), 46.1 (CH₃), 51.7 (CH₃), 52.8 (CH₃), 64.0 (CH₂), 97.5 (C), 122.8 (CH), 126.4 (C), 130.4 (CH), 141.7 (CH), 159.6 (C), 165.4 (C), 177.3 (C). HRMS (ESI-TOF) calcd for C₁₅H₂₁INO₄: 406.0510 [M+H]⁺; found: 406.0496.

Methyl 2-methyl-3-[*N*-(4-methylphenyl)amino]-2-phenylpropionate

To a solution of SDS (185 mg, 0.64 mmol) in water (8 mL) were successfully added *p*-toluidine (170 mg, 1.59 mmol), formaldehyde (37 wt. % in water, 0.13 mL), 1-methoxy-1-trimethylsiloxy-2-phenylpropene (1.5 g, 6.35 mmol), and 48% aqueous HBF₄ solution (30 μL, 0.17 mmol) at 0°C. After being stirred at the same temperature for 5 h, Dowex 1-X8 (100-200 mesh, Cl⁻ form) and water were added to quench the reaction, and the reaction mixture was further stirred for 10 min. The mixture was extracted with CH₂Cl₂ and the combined organic extracts were washed with brine and concentrated. The residue was dissolved in Et₂O and extracted with 2N HCl. The aqueous layer was alkalinized with Na₂CO₃ and extracted with CH₂Cl₂. The organic extracts were dried and concentrated to give methyl 2-methyl-3-[*N*-(4-methylphenyl)amino]-2-phenylpropionate (250 mg, 55%), which was used in the next step without purification.

¹H NMR (CDCl₃, 300 MHz) δ 1.68 (s, 3H), 2.21 (s, 3H), 3.45 (d, *J* = 12.6 Hz, 1H), 3.68 (s, 3H), 3.72 (d, *J* = 12.6 Hz, 1H), 6.50 (d, *J* = 8.4 Hz, 2H), 6.94 (d, *J* = 8.4 Hz, 2H), 7.24-7.40 (m, 5H). ¹³C NMR (CDCl₃, 75.4 MHz) δ 20.3 (CH₃), 21.5 (CH₃), 51.9 (C), 52.3 (CH₃), 52.6 (CH₂), 113.1 (CH), 126.1 (CH), 126.6 (C), 127.2 (CH), 128.6 (CH), 129.6 (CH), 141.3 (C), 146.1 (C), 179.9 (C).

Methyl 3-[*N*-(2-iodo-4-methylphenyl)amino]-2-methyl-2-phenylpropionate

To a solution of methyl 2-methyl-3-[*N*-(4-methylphenyl)amino]-2-phenylpropionate (250 mg, 0.88 mmol) in a CH₂Cl₂ (12 mL)-MeOH (5 mL) mixture were added CaCO₃ (114 mg, 1.14 mmol) and BTMAICl₂ (334 mg, 0.96 mmol). The mixture was stirred at room temperature for 6 h and then filtered. The solution was concentrated and the residue was partitioned between water and EtOAc. The organic layer was washed with brine, dried, and concentrated. The residue was purified by flash chromatography (SiO₂, from hexanes to hexanes-EtOAc 8%) to give methyl 3-[*N*-(2-iodo-4-methylphenyl)amino]-2-methyl-2-phenylpropionate (257 mg, 71%).

¹H NMR (CDCl₃, 300 MHz) δ 1.70 (s, 3H), 2.17 (s, 3H), 3.50 (dd, *J* = 12.6 and 5.1 Hz, 1H), 3.71 (m, 1H), 3.72 (s, 3H), 4.18 (broad t, *J* = 6 Hz, 1H), 6.47 (d, *J* = 8.4 Hz, 1H), 6.96 (dd, *J* =

8.4 and 1.8 Hz, 1H), 7.24-7.40 (m, 5H), 7.45 (d, J = 1.8 Hz, 1H). ^{13}C NMR (CDCl_3 , 75.4 MHz) δ 19.7 (CH_3), 21.4 (CH_3), 51.7 (C), 52.4 (CH_3), 52.6 (CH_2), 85.9 (C), 110.8 (CH), 126.2 (CH), 127.4 (CH), 128.1 (C), 128.7 (CH), 129.8 (CH), 139.2 (CH), 140.9 (C), 145.2 (C), 175.7 (C).

Methyl 3-[*N*-(2-iodo-4-methylphenyl)-*N*-methylamino]-2-methyl-2-phenylpropionate (10d)

A mixture of methyl 3-[*N*-(2-iodo-4-methylphenyl)amino]-2-methyl-2-phenylpropionate (257 mg, 0.63 mmol), K_2CO_3 (174 mg, 1.26 mmol), and iodomethane (0.4 mL, 6.3 mmol) in acetonitrile (5 mL) was stirred at 90°C in a sealed tube for 72 h. The solvent was removed *in vacuo*, and the residue was partitioned between water and CH_2Cl_2 . The organic layer was dried and concentrated. The residue was purified by flash chromatography (SiO_2 , from CH_2Cl_2 to CH_2Cl_2 -MeOH 2%) to give methyl 3-[*N*-(2-iodo-4-methylphenyl)-*N*-methylamino]-2-methyl-2-phenylpropionate (**10d**, 268 mg, quantitative).

^1H NMR (CDCl_3 , 300 MHz) δ 1.66 (s, 3H), 2.25 (s, 3H), 2.52 (s, 3H), 3.42 (d, J = 13.8 Hz, 1H), 3.60 (s, 3H), 3.88 (d, J = 13.8 Hz, 1H), 7.08 (m, 2H), 7.20-7.38 (m, 5H), 7.65 (s, 1H). ^{13}C NMR (CDCl_3 , 75.4 MHz) δ 20.2 (CH_3), 21.6 (CH_3), 47.2 (CH_3), 52.0 (CH_3), 52.6 (C), 65.1 (CH_2), 100.7 (C), 124.1 (CH), 126.2 (CH), 126.9 (CH), 128.3 (CH), 130.0 (CH), 136.6 (C), 140.0 (CH), 142.2 (C), 153.6 (C), 175.7 (C). HRMS (ESI-TOF) calcd for $\text{C}_{19}\text{H}_{23}\text{INO}_2$: 424.0768 $[\text{M}+\text{H}]^+$; found: 424.0752.

Methyl 3-[*N*-(2-iodo-5-methylphenyl)amino]-2,2-dimethylpropionate

To a solution of SDS (390 mg, 1.36 mmol) in water (18 mL) were successfully added 2-iodo-5-methylaniline (790 mg, 3.39 mmol), formaldehyde (37 wt. % in water, 0.28 mL), 1-methoxy-1-trimethylsiloxy-2-methylpropene (2 mL, 10 mmol), and 48% aqueous HBF_4 solution (62 μL , 0.34 mmol) at 0°C. After being stirred at room temperature for 24 h, Dowex 1-X8 (100-200 mesh, Cl^- form) and water were added to quench the reaction, and the reaction mixture was further stirred for 10 min. The mixture was extracted with CH_2Cl_2 and the combined organic extracts were washed with brine, dried, and concentrated. The residue was purified by flash chromatography (SiO_2 , from hexanes to hexanes-EtOAc 4%) to give methyl 3-[*N*-(2-iodo-5-methylphenyl)amino]-2,2-dimethylpropionate (198 mg, 17%).

^1H NMR (CDCl_3 , 300 MHz) δ 1.31 (s, 6H), 2.26 (s, 3H), 3.25 (s, 2H), 3.71 (s, 3H), 6.28 (dd, J = 7.8 and 2.1 Hz, 1H), 6.41 (s, 1H), 7.49 (d, J = 7.8 Hz, 1H). ^{13}C NMR (CDCl_3 , 100.5 MHz) δ 21.4 (CH_3), 23.6 (CH_3), 43.4 (C), 52.1 (CH_3), 52.8 (CH_2), 81.9 (C), 111.6 (CH), 119.8 (CH), 138.6 (CH), 139.4 (C), 147.1 (C), 177.1 (C).

Methyl 3-[*N*-(2-iodo-5-methylphenyl)-*N*-methylamino]-2,2-dimethylpropionate (12a**)**

A mixture of methyl 3-[*N*-(2-iodo-5-methylphenyl)amino]-2,2-dimethylpropionate (198 mg, 0.57 mmol), K₂CO₃ (158 mg, 1.14 mmol), and iodomethane (0.7 mL, 11.3 mmol) in acetonitrile (5 mL) was stirred at 90°C in a sealed tube for 68 h. The solvent was removed *in vacuo*, and the residue was partitioned between water and CH₂Cl₂. The organic layer was dried and concentrated. The residue was purified by flash chromatography (SiO₂, from hexanes to hexanes-EtOAc 2%) to give methyl 3-[*N*-(2-iodo-5-methylphenyl)-*N*-methylamino]-2,2-dimethylpropionate (**12a**, 140 mg, 68%).

¹H NMR (CDCl₃, 400 MHz) δ 1.19 (s, 6H), 2.28 (s, 3H), 2.66 (s, 3H), 3.26 (s, 2H), 3.56 (s, 3H), 6.61 (dd, *J* = 8 and 1.6 Hz, 1H), 7.00 (d, *J* = 1.6 Hz, 1H), 7.67 (d, *J* = 8 Hz, 1H). ¹³C NMR (CDCl₃, 100.5 MHz) δ 21.0 (CH₃), 23.9 (CH₃), 44.6 (C), 46.6 (CH₃), 51.6 (CH₃), 64.9 (CH₂), 96.2 (C), 125.1 (CH), 126.8 (CH), 139.2 (C), 139.4 (CH), 155.5 (C), 177.8 (C). HRMS (ESI-TOF) calcd for C₁₄H₂₁INO₂: 362.0611 [M+H]⁺; found: 362.0612.

Methyl 3-[*N*-(5-chloro-2-iodophenyl)-*N*-methylamino]-2,2-dimethylpropionate (12b**)**

Sodium (235 mg, 10.2 mmol) was slowly added to MeOH (10 mL). Once the evolution of hydrogen had ceased, 5-chloro-2-iodoaniline (515 mg, 2.03 mmol) was added and the resulting solution was poured onto a suspension of paraformaldehyde (85 mg, 2.83 mmol) in MeOH (5 mL). The resulting mixture was stirred at room temperature for 5 h and then hydrolyzed with cool water and extracted with Et₂O. The organic extracts were washed with water, dried, and concentrated in *vacuo*, taking care that the temperature remained below 25 °C. A 1:1 mixture of 5-chloro-2-iodoaniline and *N*-(methoxymethyl)-5-chloro-2-iodoaniline^{5,6} was obtained. This mixture was dissolved in dichloromethane (5 mL) and the solution was cooled to -78°C. A solution of BF₃·Et₂O (0.3 mL, 2.4 mmol) in dichloromethane (3 mL) was added dropwise, the resulting mixture was stirred at -78°C for 5 min and then a solution of 1-methoxy-1-trimethylsiloxy-2-methylpropene (0.25 mL, 1.25 mmol) in dichloromethane (3 mL) was added. After 4 h at -78°C, the reaction mixture was allowed to reach room temperature, poured into brine, and extracted with dichloromethane. The organic extracts were dried and concentrated to give a 1:5 mixture of 5-chloro-2-iodoaniline and methyl 3-[*N*-(5-chloro-2-iodophenyl)amino]-2,2-dimethylpropionate. The mixture was dissolved in CH₃CN (5 mL) and K₂CO₃ (0.25 g, 1.8 mmol) and iodomethane (0.56 mL, 9 mmol) were added. After 72 h at 90 °C in a sealed tube, iodomethane (1.5 mL, 24 mmol) was added, and heating was maintained for 24 h. The solvent was removed in *vacuo* and the residue was partitioned

⁵ Ha, H.J.; Suh, J.M.; Ahn, Y.-G.; Dong, Y.; Yun, H. *Heterocycles* **1999**, *50*, 203-214.

⁶ Barluenga, J.; Bayón, A. M.; Campos, P.; Asensio, G.; Gonzalez-Nuñez, E.; Molina Y. J. *Chem. Soc., Perkin Trans. 1* **1988**, 1631-1636.

between dichloromethane and water. The organic extracts were dried and concentrated. Chromatography (from hexanes to hexanes-EtOAc 1%) yielded **12b** (107 mg, 14%).

^1H NMR (CDCl_3 , 400 MHz) δ 1.19 (s, 6H), 2.68 (s, 3H), 3.26 (s, 2H), 3.57 (s, 3H), 6.79 (dd, J = 8.4 and 2 Hz, 1H), 7.15 (d, J = 2 Hz, 1H), 7.73 (d, J = 8.4 Hz, 1H). ^{13}C NMR (CDCl_3 , 100.5 MHz) δ 23.9 (CH_3), 44.6 (C), 46.5 (CH_3), 51.7 (CH_3), 64.6 (CH_2), 97.2 (C), 124.7 (CH), 125.8 (CH), 134.8 (C), 140.5 (CH), 156.8 (C), 177.5 (C).

Methyl 3-[*N*-(2-iodophenyl)-*N*-(methoxycarbonyl)amino]propionate (14a**)**

A solution of 2-iodoaniline (1 g, 4.56 mmol) and methyl acrylate (1.24 mL, 13.7 mmol) in acetic acid (0.5 mL) was stirred at 90°C in a sealed tube for 24 h. The reaction mixture was poured into cooled (ice) water, basified with Na_2CO_3 , and extracted with Et_2O . The organic extracts were washed with saturated aqueous Na_2CO_3 solution, dried, and concentrated. The residue was purified by flash chromatography (SiO_2 , from hexanes to 3:7 hexanes- CH_2Cl_2) to give methyl 3-[*N*-(2-iodophenyl)amino]propionate (245 mg, 18%).

A mixture of methyl 3-[*N*-(2-iodophenyl)amino]propionate (245 mg, 0.80 mmol) and K_2CO_3 (221 mg, 1.60 mmol) in methyl chloroformate (5 mL) was stirred at 60°C for 12 h. The mixture was poured into water and extracted with CH_2Cl_2 . The organic extracts were dried and concentrated. The residue was purified by flash chromatography (SiO_2 , from hexanes to 7:3 hexanes-EtOAc) to give methyl 3-[*N*-(2-iodophenyl)-*N*-(methoxycarbonyl)amino]propionate (**14a**, 269 mg, 93%).

^1H NMR (CDCl_3 , 300 MHz) δ 2.69 (t, J = 7.5 Hz, 2H), 3.63 (s, 3H), 3.65 (s, 3H), 3.66 (m, 1H), 4.13 (dt, J = 13.8 and 7.5 Hz, 1H), 7.03 (ddd, J = 7.8, 7.2, and 1.5 Hz, 1H), 7.20 (d, J = 7.5 Hz, 1H), 7.37 (ddd, J = 7.8, 7.5, and 1.5 Hz, 1H), 7.89 (d, J = 7.2 Hz, 1H). ^{13}C NMR (CDCl_3 , 75.4 MHz) δ 33.0 (CH_2), 46.1 (CH_2), 51.6 (CH_3), 53.1 (CH_3), 100.2 (C), 129.2 (CH), 129.5 (CH), 139.7 (CH), 143.6 (C), 155.2 (C), 171.7 (C). HRMS (ESI-TOF) calcd for $\text{C}_{12}\text{H}_{15}\text{INO}_4$: 364.0040 $[\text{M}+\text{H}]^+$; found: 364.0031.

Methyl 3-[*N*-(2-iodophenyl)-*N*-(*p*-toluenesulfonyl)amino]propionate (14b**)**

A solution of methyl 3-[*N*-(2-iodophenyl)amino]propionate (348 mg, 1.14 mmol) and *p*-toluenesulfonyl chloride (2.17 g, 11.4 mmol) in pyridine (12 mL) was stirred at reflux for 24 h. The mixture was poured into water and extracted with Et_2O . The organic extracts were washed with 1N hydrochloric acid and 10% aqueous KOH solution, dried and concentrated. The residue was purified by flash chromatography (SiO_2 , from CH_2Cl_2 to CH_2Cl_2 -MeOH 5%) to give methyl 3-[*N*-(2-iodophenyl)-*N*-(*p*-toluenesulfonyl)amino]propionate (**14b**, 407 mg, 78%).

^1H NMR (CDCl_3 , 300 MHz) δ 2.45 (s, 3H), 2.66 (m, 2H), 3.59 (s, 3H), 3.70 (ddd, J = 14.1, 9.3, and 6 Hz, 1H), 3.98 (ddd, J = 14.1, 9.6, and 6.3 Hz, 1H), 6.96 (dd, J = 7.8 and 1.5 Hz,

1H), 7.04 (ddd, $J = 7.8, 7.5$, and 1.5 Hz, 1H), 7.26-7.32 (m, 3H), 7.65 (d, $J = 8.4$ Hz, 2H), 7.92 (dd, $J = 7.8$ and 1.5 Hz, 1H). ^{13}C NMR (CDCl_3 , 75.4 MHz) δ 21.5 (CH_3), 33.4 (CH_2), 47.4 (CH_2), 51.6 (CH_3), 102.9 (C), 128.0 (CH), 128.8 (CH), 129.5 (CH), 130.0 (CH), 130.2 (CH), 135.6 (C), 140.4 (CH), 141.2 (C), 143.8 (C), 171.1 (C). HRMS (ESI-TOF) calcd for $\text{C}_{17}\text{H}_{19}\text{INO}_4\text{S}$: 460.0074 $[\text{M}+\text{H}]^+$; found: 460.0056.

Representative procedure for the Pd-catalyzed nucleophilic attack at the alkoxycarbonyl group:

A mixture of methyl 3-[*N*-(2-iodo-4-methylphenyl)-*N*-methylamino]propionate (**1a**, 75 mg, 0.23 mmol), K_3PO_4 (143 mg, 0.68 mmol), Et_3N (0.32 mL, 2.25 mmol), and $\text{Pd}(\text{PPh}_3)_4$ (52 mg, 0.045 mmol) in toluene (6 mL) was stirred at 110°C in a sealed tube for 72 h. The reaction mixture was poured into water and extracted with Et_2O . The organic extracts were washed with brine, dried, and concentrated. The residue was purified by flash chromatography (SiO_2 , from hexanes to hexanes-EtOAc 8%) to give 1,6-dimethyl-2,3-dihydro-1*H*-quinolin-4-one (**2**, 27 mg, 65%) and methyl 3-[*N*-methyl-*N*-(4-methylphenyl)amino]propionate (**3**, 14 mg, 30%).

1,6-Dimethyl-2,3-dihydro-1*H*-quinolin-4-one (2)

^1H NMR (CDCl_3 , 300 MHz) δ 2.25 (s, 3H), 2.72 (t, $J = 6.9$ Hz, 2H), 2.96 (s, 3H), 3.42 (t, $J = 6.9$ Hz, 2H), 6.65 (d, $J = 8.7$ Hz, 1H), 7.23 (dd, $J = 8.7$ and 2.1 Hz, 1H), 7.72 (d, $J = 2.1$ Hz, 1H). ^{13}C NMR (CDCl_3 , 75.4 MHz) δ 20.1 (CH_3), 38.5 (CH_2), 39.5 (CH_3), 51.7 (CH_2), 113.4 (CH), 119.8 (C), 126.4 (C), 127.7 (CH), 136.5 (CH), 151.0 (C), 193.9 (C). Anal. Calcd for $\text{C}_{11}\text{H}_{13}\text{NO}$: C, 75.40; H, 7.48; N, 7.99. Found: C, 75.70; H, 7.53; N, 8.01. HRMS (ESI-TOF) calcd for $\text{C}_{11}\text{H}_{14}\text{NO}$: 176.1069 $[\text{M}+\text{H}]^+$; found: 176.1069.

Methyl 3-[*N*-methyl-*N*-(4-methylphenyl)amino]propionate (3)

^1H NMR (CDCl_3 , 300 MHz) δ 2.25 (s, 3H), 2.56 (t, $J = 7.5$ Hz, 2H), 2.90 (s, 3H), 3.64 (t, $J = 7.5$ Hz, 2H), 3.67 (s, 3H), 6.67 (d, $J = 8.4$ Hz, 2H), 7.05 (d, $J = 8.4$ Hz, 2H). ^{13}C NMR (CDCl_3 , 75.4 MHz) δ 20.2 (CH_3), 31.4 (CH_2), 38.4 (CH_3), 49.0 (CH_2), 51.7 (CH_3), 113.0 (CH), 126.2 (C), 129.7 (CH), 146.6 (C), 172.8 (C).

6-Methoxy-1-methyl-2,3-dihydro-1*H*-quinolin-4-one (5a)

^1H NMR (CDCl_3 , 300 MHz) δ 2.73 (t, $J = 7.2$ Hz, 2H), 2.94 (s, 3H), 3.39 (t, $J = 7.2$ Hz, 2H), 3.79 (s, 3H), 6.71 (d, $J = 9$ Hz, 1H), 7.07 (dd, $J = 9$ and 3 Hz, 1H), 7.41 (d, $J = 3$ Hz, 1H). ^{13}C NMR (CDCl_3 , 75.4 MHz) δ 38.5 (CH_2), 39.7 (CH_3), 51.9 (CH_2), 55.7 (CH_3), 108.9 (CH), 115.0 (CH), 120.0 (C), 124.9 (CH), 148.3 (C), 151.6 (C), 193.6 (C). Anal. Calcd for $\text{C}_{11}\text{H}_{13}\text{NO}_2$: C, 69.09; H, 6.85; N, 7.32. Found: C, 68.83; H, 6.93; N, 7.13.

Methyl 3-[*N*-(4-methoxyphenyl)-*N*-methylamino]propionate (6a)

¹H NMR (CDCl₃, 300 MHz) δ 2.54 (t, *J* = 7.2 Hz, 2H), 2.86 (s, 3H), 3.59 (t, *J* = 7.2 Hz, 2H), 3.66 (s, 3H), 3.76 (s, 3H), 6.72-6.86 (m, 4H). ¹³C NMR (CDCl₃, 75.4 MHz) δ 31.4 (CH₂), 38.9 (CH₃), 49.9 (CH₂), 51.7 (CH₃), 55.7 (CH₃), 114.8 (CH), 115.1 (CH), 143.4 (C), 152.1 (C), 172.8 (C). HRMS (ESI-TOF) calcd for C₁₂H₁₈NO₃: 224.1281 [M+H]⁺; found: 224.1283.

Methyl 1-methyl-4-oxo-1,2,3,4-tetrahydroquinoline-6-carboxylate (5b)

¹H NMR (CDCl₃, 300 MHz) δ 2.77 (t, *J* = 7.2 Hz, 2H), 3.09 (s, 3H), 3.58 (t, *J* = 7.2 Hz, 2H), 3.87 (s, 3H), 6.71 (d, *J* = 9 Hz, 1H), 8.04 (dd, *J* = 9 and 2.4 Hz, 1H), 8.57 (d, *J* = 2.4 Hz, 1H). ¹³C NMR (CDCl₃, 75.4 MHz) δ 37.7 (CH₂), 39.3 (CH₃), 50.9 (CH₂), 51.8 (CH₃), 112.8 (CH), 118.4 (C), 118.7 (C), 130.5 (CH), 136.1 (CH), 154.7 (C), 166.7 (C), 192.6 (C). Anal. Calcd for C₁₂H₁₃NO₃: C, 65.74; H, 5.98; N, 6.39. Found: C, 65.97; H, 6.10; N, 5.85.

Methyl 3-[*N*-(4-methoxycarbonylphenyl)-*N*-methylamino]propionate (6b)

¹H NMR (CDCl₃, 300 MHz) δ 2.61 (t, *J* = 7.2 Hz, 2H), 3.03 (s, 3H), 3.68 (s, 3H), 3.74 (t, *J* = 7.2 Hz, 2H), 3.86 (s, 3H), 6.66 (d, *J* = 9 Hz, 2H), 7.90 (d, *J* = 9 Hz, 2H). ¹³C NMR (CDCl₃, 75.4 MHz) δ 31.6 (CH₂), 38.3 (CH₃), 48.0 (CH₂), 51.5 (CH₃), 51.8 (CH₃), 110.7 (CH), 117.4 (C), 131.3 (CH), 151.6 (C), 167.2 (C), 172.1 (C). HRMS (ESI-TOF) calcd for C₁₃H₁₈NO₄: 252.1230 [M+H]⁺; found: 252.1226.

Benzyl 3-[*N*-methyl-*N*-(4-methylphenyl)amino]propionate (6c)

¹H NMR (CDCl₃, 300 MHz) δ 2.24 (s, 3H), 2.60 (t, *J* = 7.2 Hz, 2H), 2.87 (s, 3H), 3.65 (t, *J* = 7.2 Hz, 2H), 5.10 (s, 2H), 6.66 (d, *J* = 8.4 Hz, 2H), 7.03 (d, *J* = 8.4 Hz, 2H), 7.30-7.38 (m, 5H). ¹³C NMR (CDCl₃, 75.4 MHz) δ 20.2 (CH₃), 31.7 (CH₂), 38.4 (CH₃), 49.0 (CH₂), 66.4 (CH₂), 113.1 (CH), 126.2 (C), 128.2 (CH), 128.5 (CH), 129.7 (CH), 135.8 (C), 146.6 (C), 172.2 (C). HRMS (ESI-TOF) calcd for C₁₈H₂₂NO₂: 284.1645 [M+H]⁺; found: 284.1639.

1,3,6-Trimethyl-2,3-dihydro-1*H*-quinolin-4-one (8)

¹H NMR (CDCl₃, 300 MHz) δ 1.21 (d, *J* = 7.2 Hz, 3H), 2.26 (s, 3H), 2.72 (m, 1H), 2.97 (s, 3H), 3.15 (t, *J* = 11.7 Hz, 1H), 3.39 (dd, *J* = 11.7 and 5.4 Hz, 1H), 6.64 (d, *J* = 8.7 Hz, 1H), 7.23 (ddd, *J* = 8.7, 2.4, and 0.6 Hz, 1H), 7.73 (dd, *J* = 2.4 and 0.6 Hz, 1H). ¹³C NMR (CDCl₃, 75.4 MHz) δ 12.6 (CH₃), 20.1 (CH₃), 39.4 (CH₃), 41.4 (CH), 58.3 (CH₂), 113.2 (CH), 119.1 (C), 126.3 (C), 127.9 (CH), 136.2 (CH), 150.7 (C), 196.6 (C). Anal. Calcd for C₁₂H₁₅NO: C, 76.16; H, 7.99; N, 7.40. Found: C, 76.30; H, 8.15; N, 7.22.

Methyl 2-methyl-3-[*N*-methyl-*N*-(4-methylphenyl)amino]propionate (9)

¹H NMR (CDCl₃, 300 MHz) δ 1.16 (d, *J* = 7.2 Hz, 3H), 2.24 (s, 3H), 2.90 (m, 1H), 2.91 (s, 3H), 3.27 (dd, *J* = 14.7 and 6.6 Hz, 1H), 3.62 (s, 3H), 3.64 (dd, *J* = 14.7 and 7.8 Hz, 1H), 6.63 (d, *J* = 8.4 Hz, 2H), 7.03 (d, *J* = 8.4 Hz, 2H). ¹³C NMR (CDCl₃, 75.4 MHz) δ 15.1 (CH₃), 20.2 (CH₃), 38.3 (CH), 39.2 (CH₃), 51.7 (CH₃), 56.7 (CH₂), 112.5 (CH), 125.8 (C), 129.6 (CH), 146.9 (C), 176.1 (C).

1,3,3,6-Tetramethyl-2,3-dihydro-1*H*-quinolin-4-one (11a)

¹H NMR (CDCl₃, 300 MHz) δ 1.16 (s, 6H), 2.26 (s, 3H), 2.99 (s, 3H), 3.13 (s, 2H), 6.64 (d, *J* = 8.7 Hz, 1H), 7.23 (dd, *J* = 8.7 and 2.4 Hz, 1H), 7.73 (dd, *J* = 2.4 Hz, 1H). ¹³C NMR (CDCl₃, 75.4 MHz) δ 20.1 (CH₃), 22.0 (CH₃), 39.3 (CH₃), 41.8 (C), 63.5 (CH₂), 112.9 (CH), 117.9 (C), 126.1 (C), 128.3 (CH), 136.1 (CH), 149.9 (C), 198.8 (C). Anal. Calcd for C₁₃H₁₇NO: C, 76.81; H, 8.43; N, 6.89. Found: C, 76.63; H, 8.48; N, 6.81.

6-Methoxy-1,3,3-trimethyl-2,3-dihydro-1*H*-quinolin-4-one (11b)

¹H NMR (CDCl₃, 300 MHz) δ 1.17 (s, 6H), 2.98 (s, 3H), 3.11 (s, 2H), 3.79 (s, 3H), 6.70 (d, *J* = 9 Hz, 1H), 7.07 (dd, *J* = 9 and 2.7 Hz, 1H), 7.43 (d, *J* = 2.7 Hz, 1H). ¹³C NMR (CDCl₃, 75.4 MHz) δ 22.0 (CH₃), 39.6 (CH₃), 41.9 (C), 55.7 (CH₃), 63.8 (CH₂), 109.5 (CH), 114.6 (CH), 118.1 (C), 124.5 (CH), 147.2 (C), 151.5 (C), 198.6 (C). Anal. Calcd for C₁₃H₁₇NO₂: C, 71.21; H, 7.81; N, 6.39. Found: C, 70.75; H, 7.96; N, 6.17.

Methyl 1,3,3-trimethyl-4-oxo-1,2,3,4-tetrahydroquinoline-6-carboxylate (11c)

¹H NMR (CDCl₃, 300 MHz) δ 1.15 (s, 6H), 3.10 (s, 3H), 3.26 (s, 2H), 3.85 (s, 3H), 6.69 (d, *J* = 9 Hz, 1H), 8.00 (dd, *J* = 9 and 2.1 Hz, 1H), 8.57 (d, *J* = 2.1 Hz, 1H). ¹³C NMR (CDCl₃, 75.4 MHz) δ 21.9 (CH₃), 39.2 (CH₃), 41.4 (C), 51.7 (CH₃), 62.7 (CH₂), 112.3 (CH), 117.0 (C), 118.4 (C), 131.2 (CH), 135.6 (CH), 153.6 (C), 166.7 (C), 197.5 (C). Anal. Calcd for C₁₄H₁₇NO₃: C, 68.00; H, 6.93; N, 5.66. Found: C, 67.88; H, 6.86; N, 5.48.

1,3,6-Trimethyl-3-phenyl-2,3-dihydro-1*H*-quinolin-4-one (11d)

¹H NMR (CDCl₃, 400 MHz) δ 1.46 (s, 3H), 2.23 (s, 3H), 2.99 (s, 3H), 3.44 (d, *J* = 12.8 Hz, 1H), 3.73 (d, *J* = 12.8 Hz, 1H), 6.54 (d, *J* = 8.4 Hz, 1H), 7.15-7.31 (m, 6H), 7.79 (s, 1H). ¹³C NMR (CDCl₃, 100.6 MHz) δ 20.1 (CH₃), 23.2 (CH₃), 39.1 (CH₃), 49.7 (C), 61.9 (CH₂), 113.0 (CH), 118.9 (C), 126.2 (C), 126.3 (CH), 126.9 (CH), 128.3 (CH), 128.5 (CH), 136.3 (CH), 140.9 (C), 149.7 (C), 196.5 (C). Anal. Calcd for C₁₈H₁₉NO: C, 81.47; H, 7.22; N, 5.28. Found: C, 79.86; H, 7.20; N, 5.13.

1,3,3,7-Tetramethyl-2,3-dihydro-1H-quinolin-4-one (13a)

¹H NMR (CDCl₃, 400 MHz) δ 1.15 (s, 6H), 2.33 (s, 3H), 3.01 (s, 3H), 3.15 (s, 2H), 6.50 (s, 1H), 6.57 (d, *J* = 8 Hz, 1H), 7.82 (d, *J* = 8 Hz, 1H). ¹³C NMR (CDCl₃, 100.6 MHz) δ 22.0 (CH₃), 23.3 (CH₃), 39.2 (CH₃), 41.6 (C), 63.5 (CH₂), 112.9 (CH), 116.0 (C), 118.5 (CH), 128.8 (CH), 145.9 (C), 151.7 (C), 198.4 (C). Calcd for C₁₃H₁₇NO: C, 76.81; H, 8.43; N, 6.89. Found: C, 76.63; H, 8.48; N, 6.81.

7-Chloro-1,3,3-trimethyl-2,3-dihydro-1H-quinolin-4-one (13b)

¹H NMR (CDCl₃, 300 MHz) δ 1.16 (s, 6H), 3.02 (s, 3H), 3.19 (s, 2H), 6.69 (s, 1H), 6.70 (dd, *J* = 7.8 and 1.8 Hz, 1H), 7.84 (d, *J* = 7.8 Hz, 1H). ¹³C NMR (CDCl₃, 100.6 MHz) δ 21.9 (CH₃), 39.2 (CH₃), 41.6 (C), 63.2 (CH₂), 112.5 (CH), 116.5 (C), 117.5 (CH), 130.3 (CH), 141.4 (C), 152.0 (C), 197.7 (C). Calcd for C₁₂H₁₄ClNO: C, 64.43; H, 6.31; N, 6.26. Found: C, 64.59; H, 6.20; N, 6.71.

Methyl 3-[*N*-(methoxycarbonyl)-*N*-phenylamino]propionate (15a)

¹H NMR (CDCl₃, 300 MHz) δ 2.61 (t, *J* = 7.5 Hz, 2H), 3.60 (s, 3H), 3.68 (s, 3H), 3.98 (t, *J* = 7.5 Hz, 2H), 7.15-7.40 (m, 5H). ¹³C NMR (CDCl₃, 75.4 MHz) δ 33.2 (CH₂), 46.5 (CH₂), 51.6 (CH₃), 52.9 (CH₃), 126.9 (CH), 127.3 (CH), 129.1 (CH), 141.3 (C), 155.8 (C), 171.7 (C). HRMS (ESI-TOF) calcd for C₁₂H₁₅NO₄Na: 260.0893 [M+Na]⁺; found: 260.0885.

Methyl 3-[*N*-phenyl-*N*-(*p*-toluenesulfonyl)amino]propionate (15b)

¹H NMR (CDCl₃, 300 MHz) δ 2.43 (s, 3H), 2.56 (t, *J* = 7.5 Hz, 2H), 3.60 (s, 3H), 3.84 (t, *J* = 7.5 Hz, 2H), 7.04 (m, 2H), 7.23-7.33 (m, 5H), 7.47 (d, *J* = 8.7 Hz, 2H). ¹³C NMR (CDCl₃, 75.4 MHz) δ 21.6 (CH₃), 34.0 (CH₂), 46.8 (CH₂), 51.7 (CH₃), 127.7 (CH), 128.1 (CH), 128.9 (CH), 129.1 (CH), 129.4 (CH), 135.1 (C), 139.0 (C), 143.5 (C), 171.3 (C). HRMS (ESI-TOF) calcd for C₁₇H₂₀NO₄S: 334.1108 [M+H]⁺; found: 334.1110.

Palladium complex 16

To a solution of methyl 3-[*N*-(2-iodo-4-methylphenyl)-*N*-methylamino]propionate (**1a**, 40 mg, 0.12 mmol) in benzene (10 mL) were added Pd₂(dba)₃ (77 mg, 0.084 mmol) and PPh₃ (44 mg, 0.168 mmol).⁷ The reddish reaction mixture was stirred at room temperature for 24 h. The solvent was evaporated, and the residue was purified by "flash" chromatography (SiO₂). Elution with hexane/EtOAc (75:25) afforded pure azapalladacycle **16** as an orange foam (115 mg, 73%).

⁷ Solé, D.; Vallverdú, L.; Solans, X.; Font-Bardia, M.; Bonjoch, J. *Organometallics*, **2004**, 23, 1438-1447.

^1H NMR (CDCl_3 , 300 MHz) δ 1.88 (s, 3H), 2.84 (m, 1H), 3.16 (d, J = 3 Hz, 3H), 3.28-3.68 (m, 3H), 3.55 (s, 3H), 5.54 (s, 1H), 6.71 (d, J = 7.8 Hz, 1H), 6.82 (d, J = 7.8 Hz, 1H), 7.30-7.50 (m, 9H), 7.65-7.75 (m, 6H). ^{13}C NMR (CDCl_3 , 100.5 MHz) δ 21.8 (CH_3), 34.0 (CH_2), 51.3 (CH_3), 51.6 (CH_3), 54.9 (CH_2), 118.9 (CH), 125.8 (d, J = 10.1 Hz, C), 126.1 (CH), 128.2 (d, J = 11.6 Hz, CH), 129.0 (d, J = 7.7 Hz, CH), 130.8 (d, J = 2.3 Hz, CH), 131.6 (d, J = 52.7 Hz, C), 135.0 (d, J = 12.4 Hz, CH), 158.6 (d, J = 3.9 Hz, C), 171.5 (C). One C was not observed. ^{31}P NMR (CDCl_3 , 121.5 MHz) δ 40.6. HRMS (ESI-TOF) calcd for $\text{C}_{30}\text{H}_{32}\text{INO}_2\text{PPd}$: 702.0245 $[\text{M}+\text{H}]^+$; found: 702.0234.

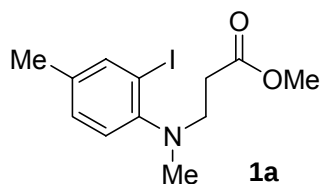
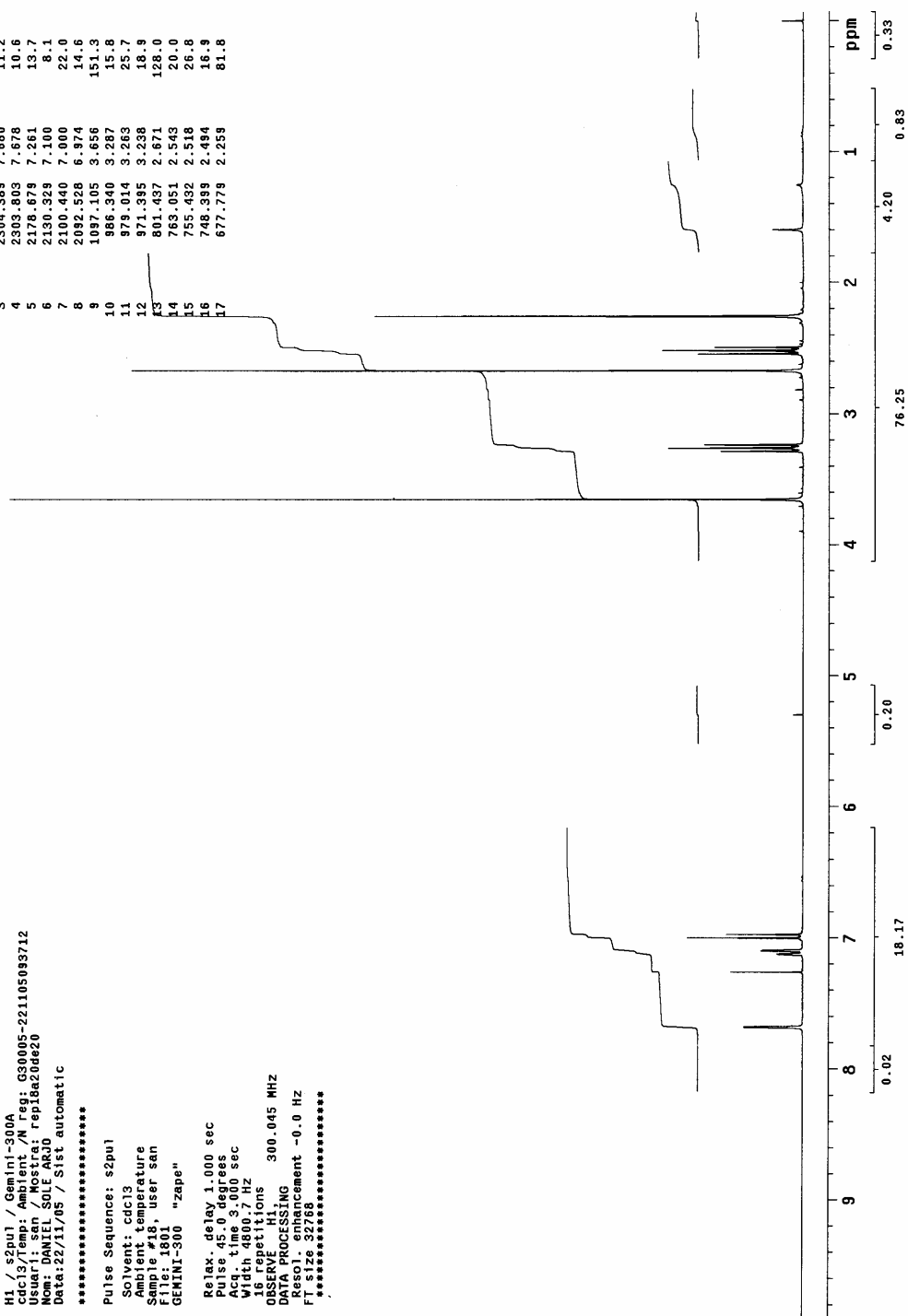
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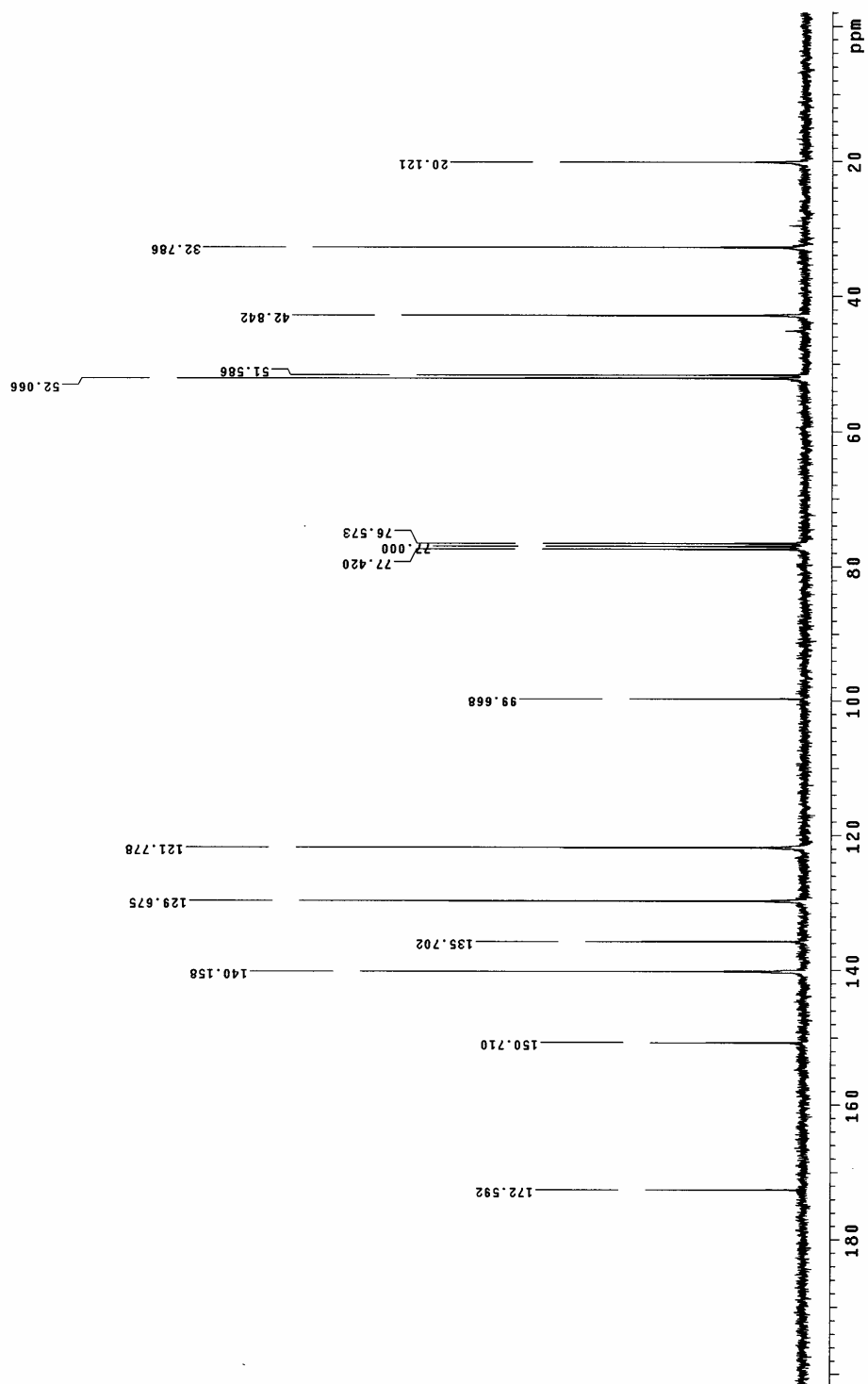
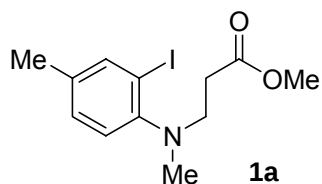
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Nom: DANIEL SOLE ARJO
Data:22/11/05 / Sist automatic

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Solvent: cdc13
Ambient temperature
Sample #18, user san
File: 1801
GEMINI-300 "Zape"

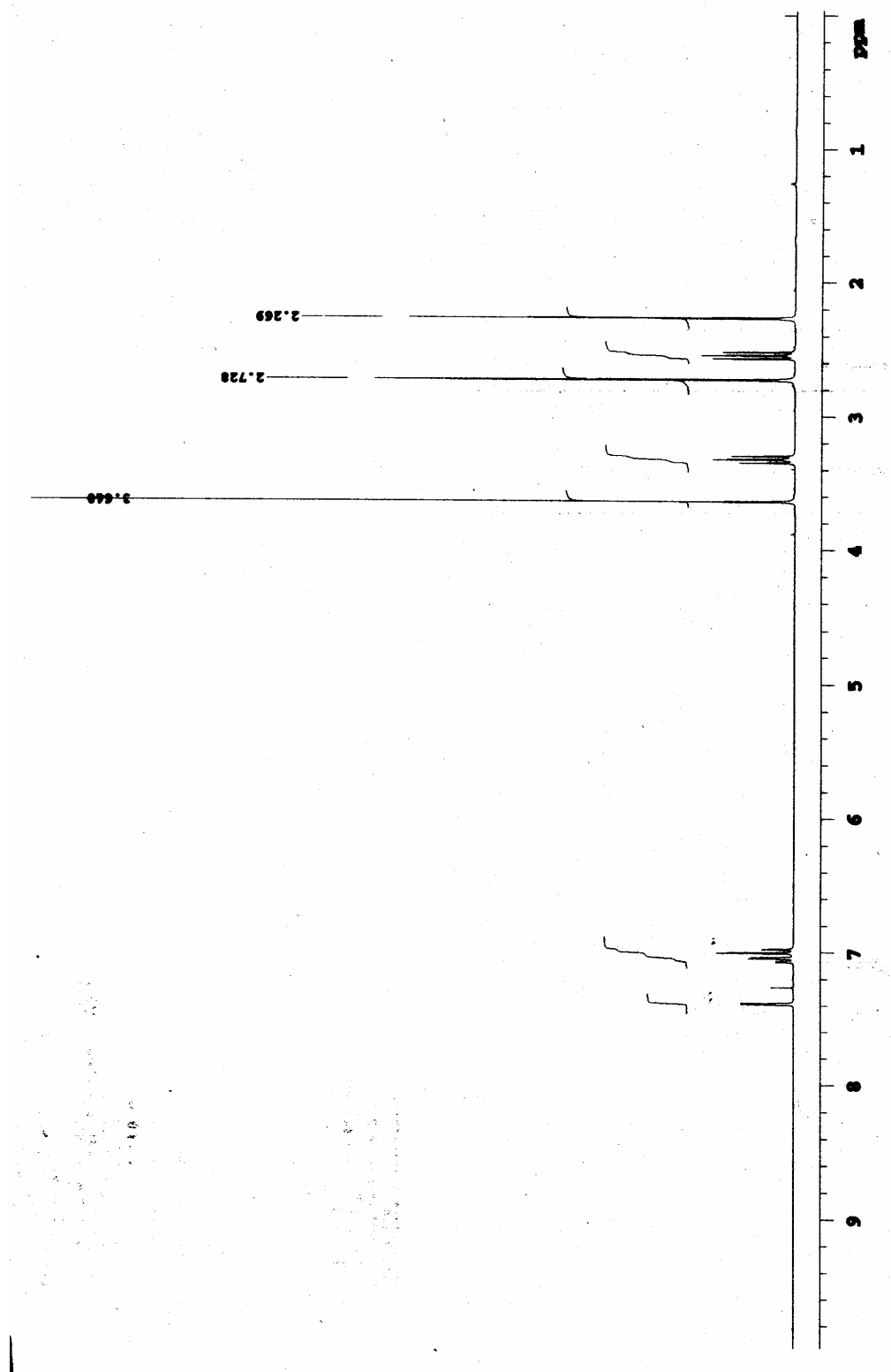
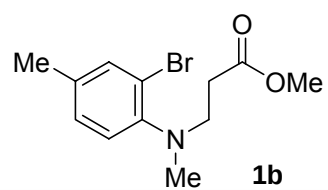
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DATA PROCESSING 300.045 MHz
Resol. enhancement -0.0 Hz
FT size 32768

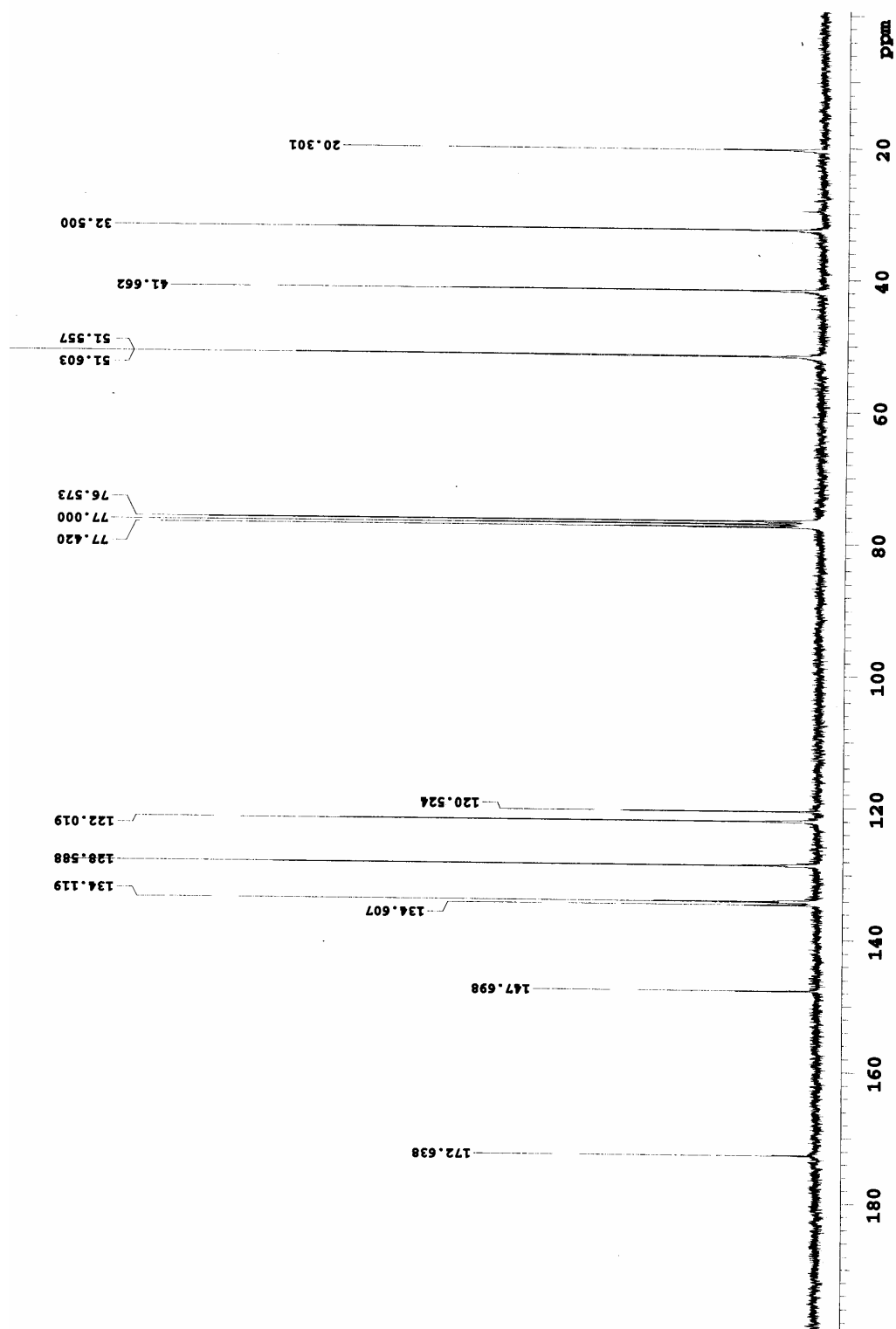
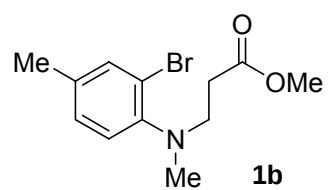
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4	2303.803	7.678	10.6
5	2178.679	7.261	13.7
6	2130.329	7.100	8.1
7	2100.440	7.000	22.0
8	2092.528	6.974	14.6
9	1097.105	3.656	151.3
10	886.340	3.287	15.8
11	879.014	3.263	25.7
12	871.395	3.238	18.9
13	801.437	2.671	128.0
14	763.051	2.543	20.0
15	755.432	2.518	26.8
16	748.389	2.494	16.9
17	677.779	2.259	81.8





C13 / s2pul / Gemini-300A
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 Usuari: san / Mostra: b24sd2_cru
 Nom: SANDRA DIAZ FITE
 Data:30/05/05 / Sist automatic
 Pulse Sequence: s2pul





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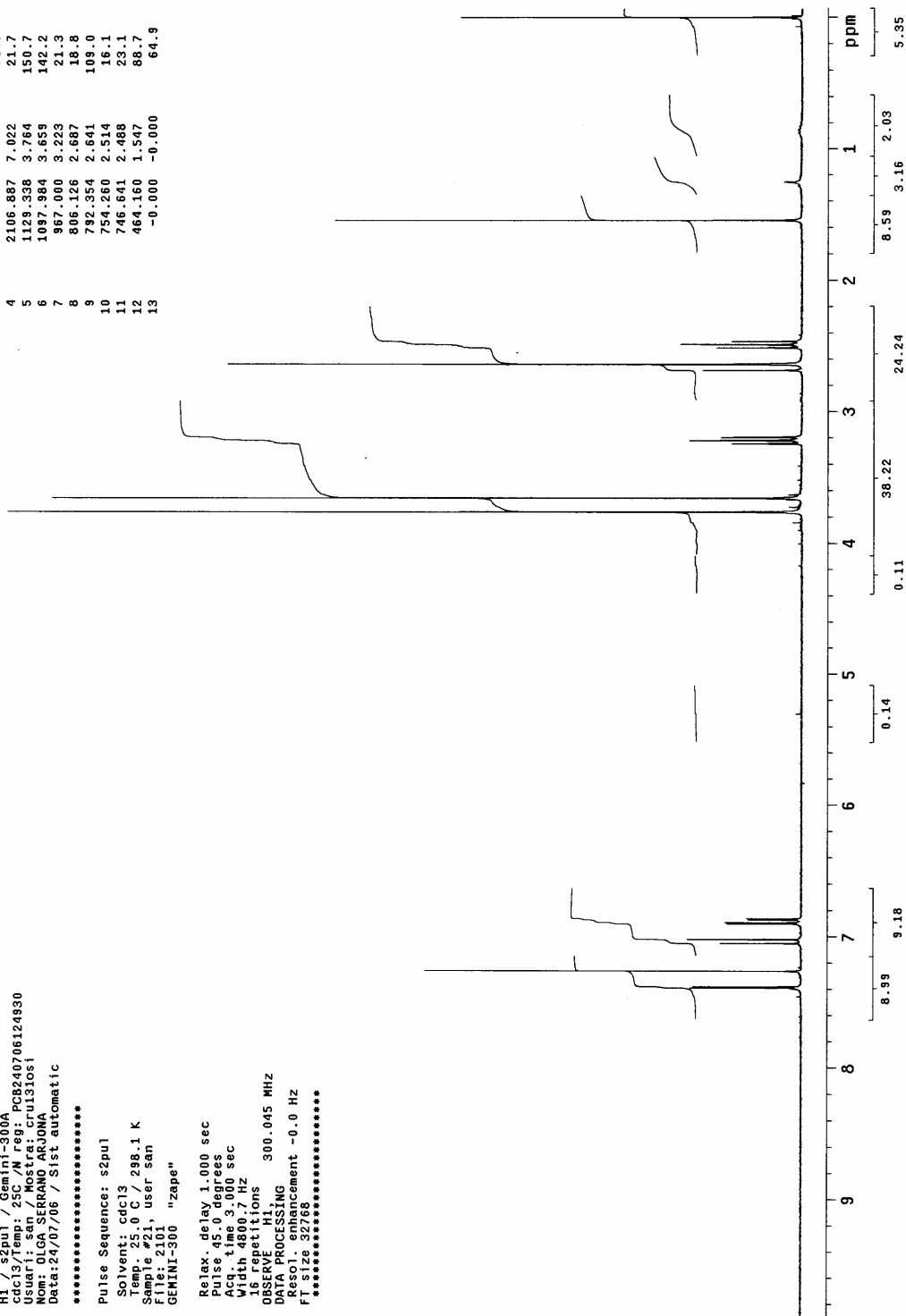
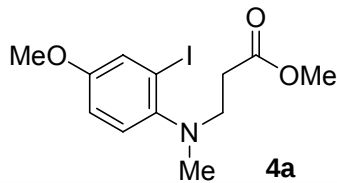
H1 / s2pul / Gemini-300A
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Data: 24/07/06 / Sist automatic

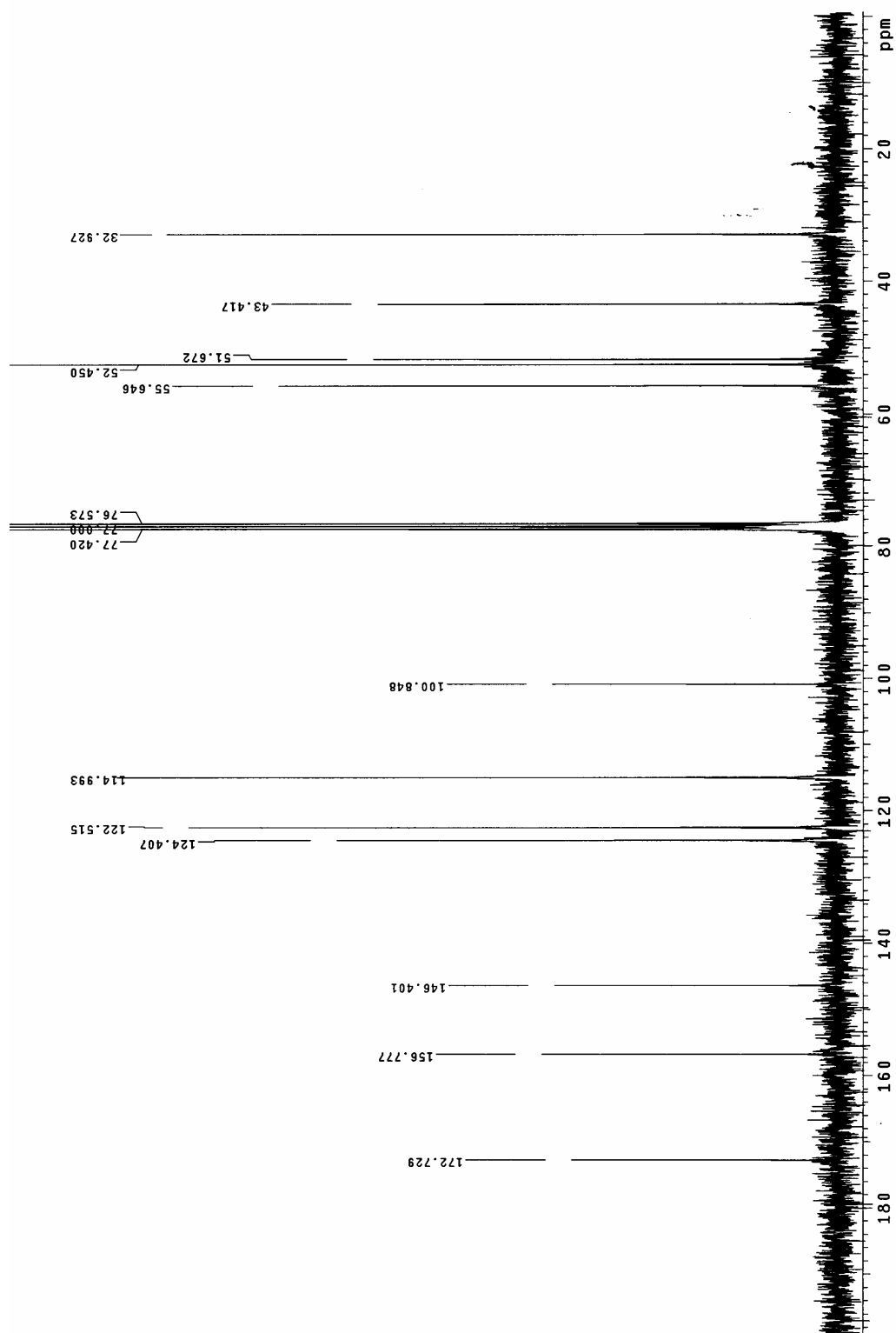
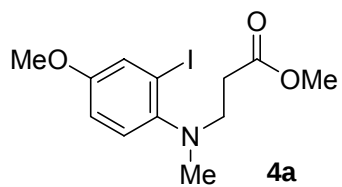
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Solvent: cdc13
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Sample #21, user san
File: 2101
GEMINI-300 "zape"

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Acq. time 5.00 sec
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16 repetitions
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DATA PROCESSING
Resol. enhancement -0.0 Hz
FT size 32768

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5	1129.338	3.764	150.7
6	1097.984	3.659	142.2
7	967.000	3.223	21.3
8	866.126	2.687	18.8
9	792.354	2.641	109.0
10	754.260	2.514	16.1
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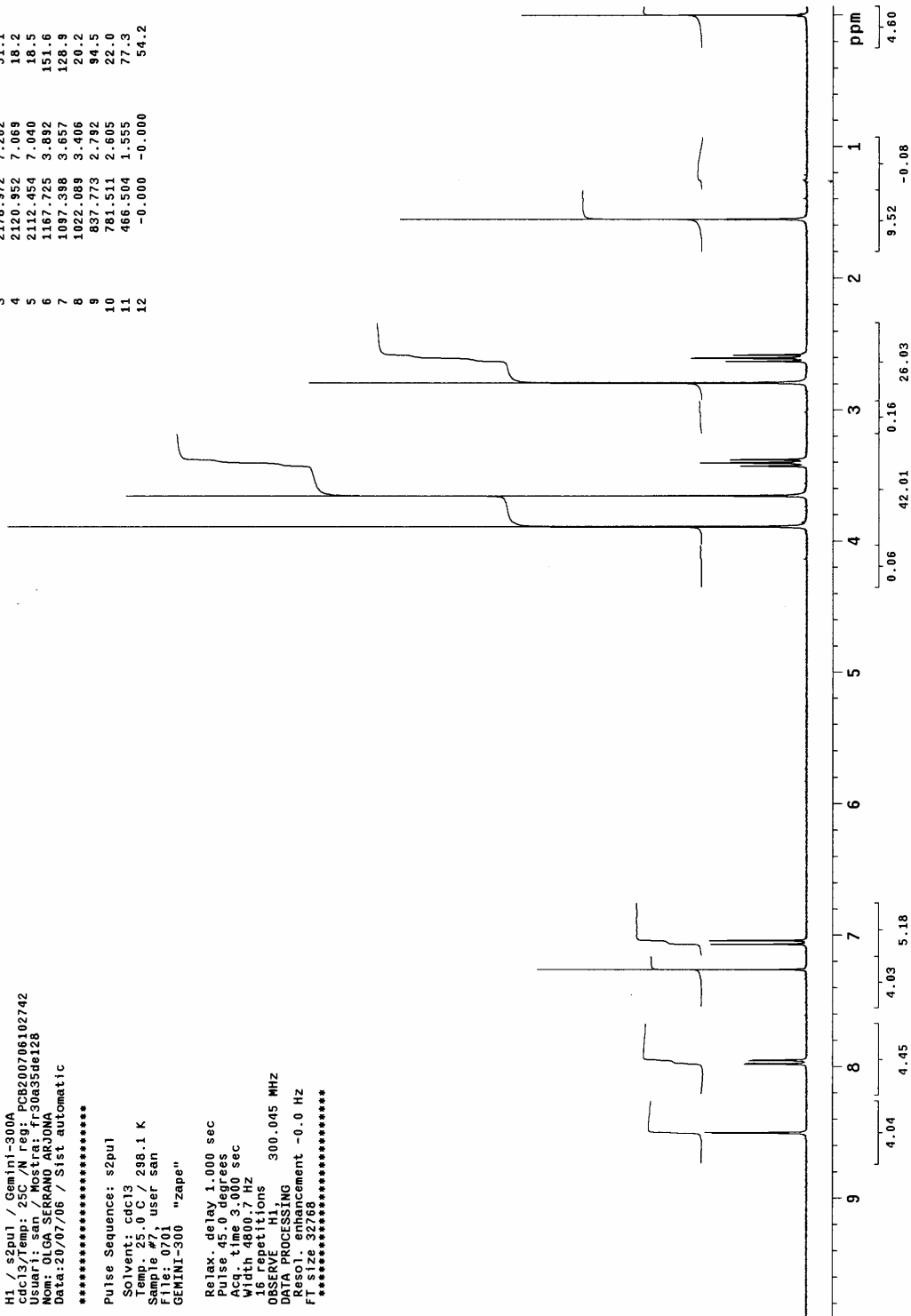
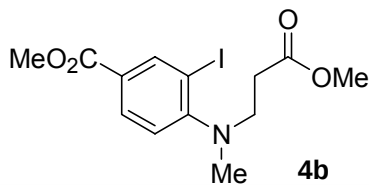
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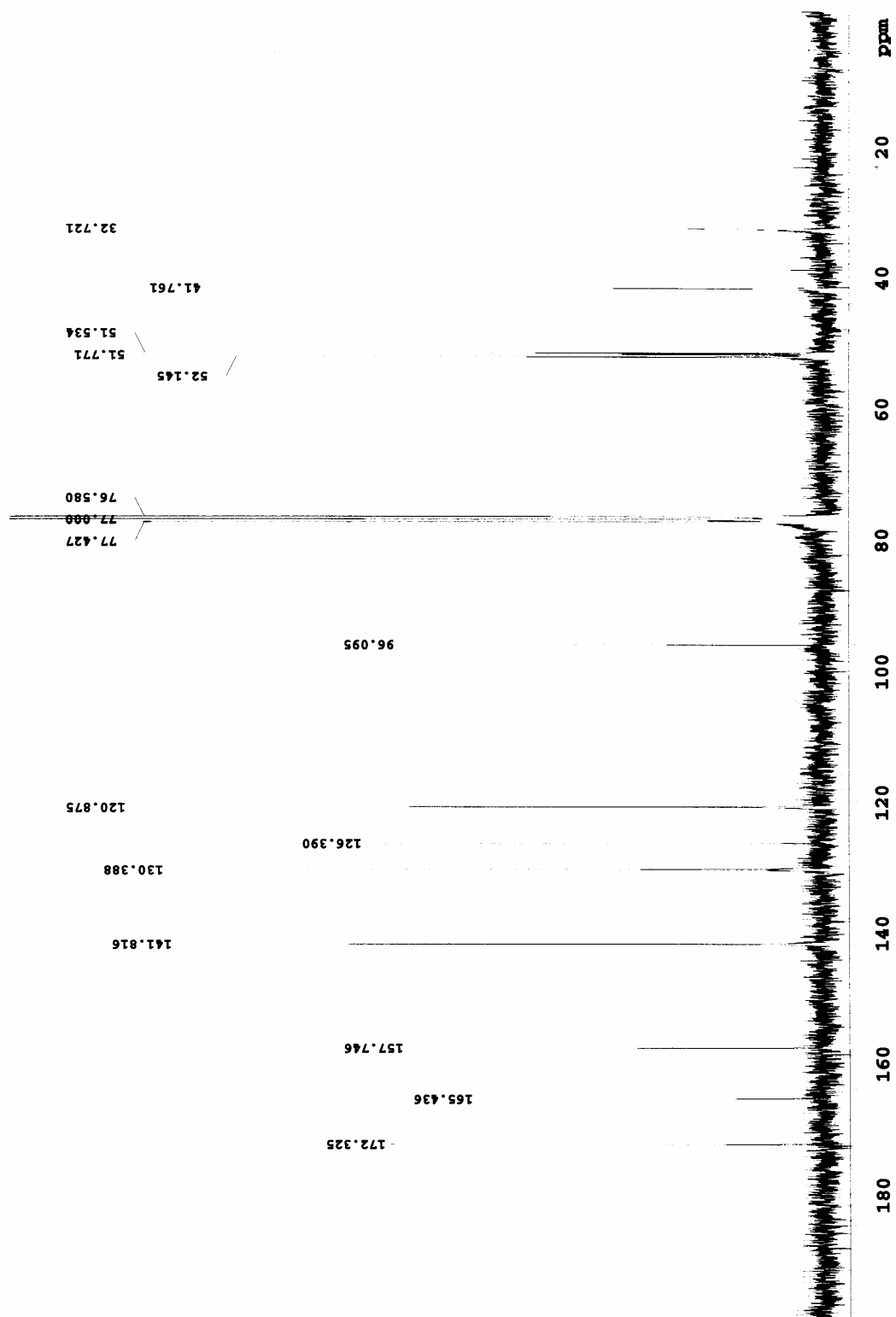
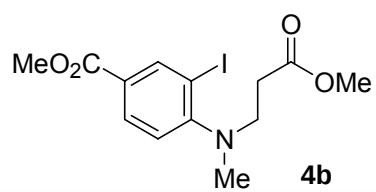
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Nom: OLGA SERRANO ARJONA
Data:20/07/06 / Sist automatic

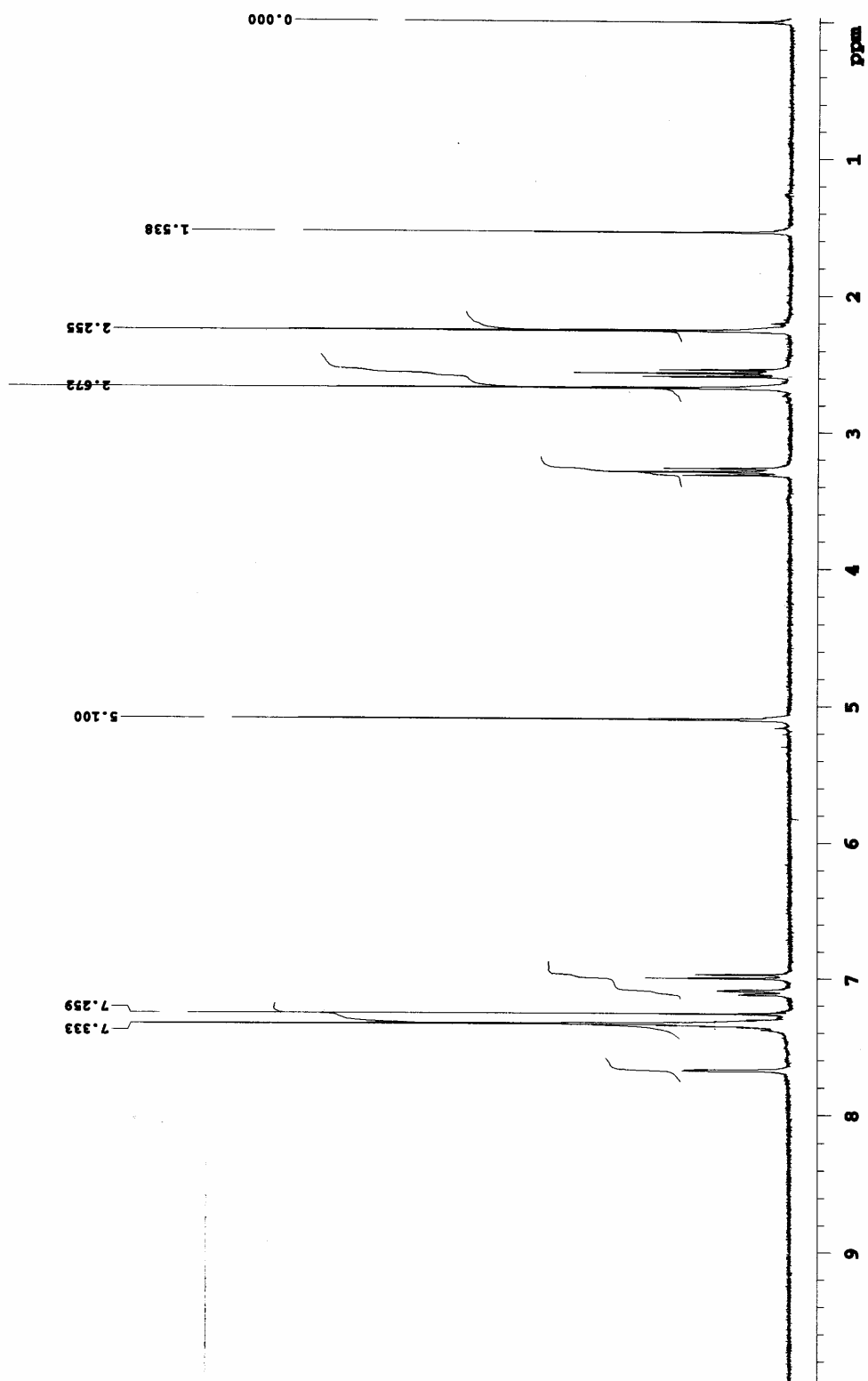
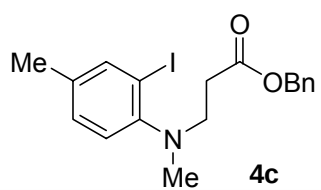
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Sample #: user san
File: 0701
GEMINI-300 "Zape"

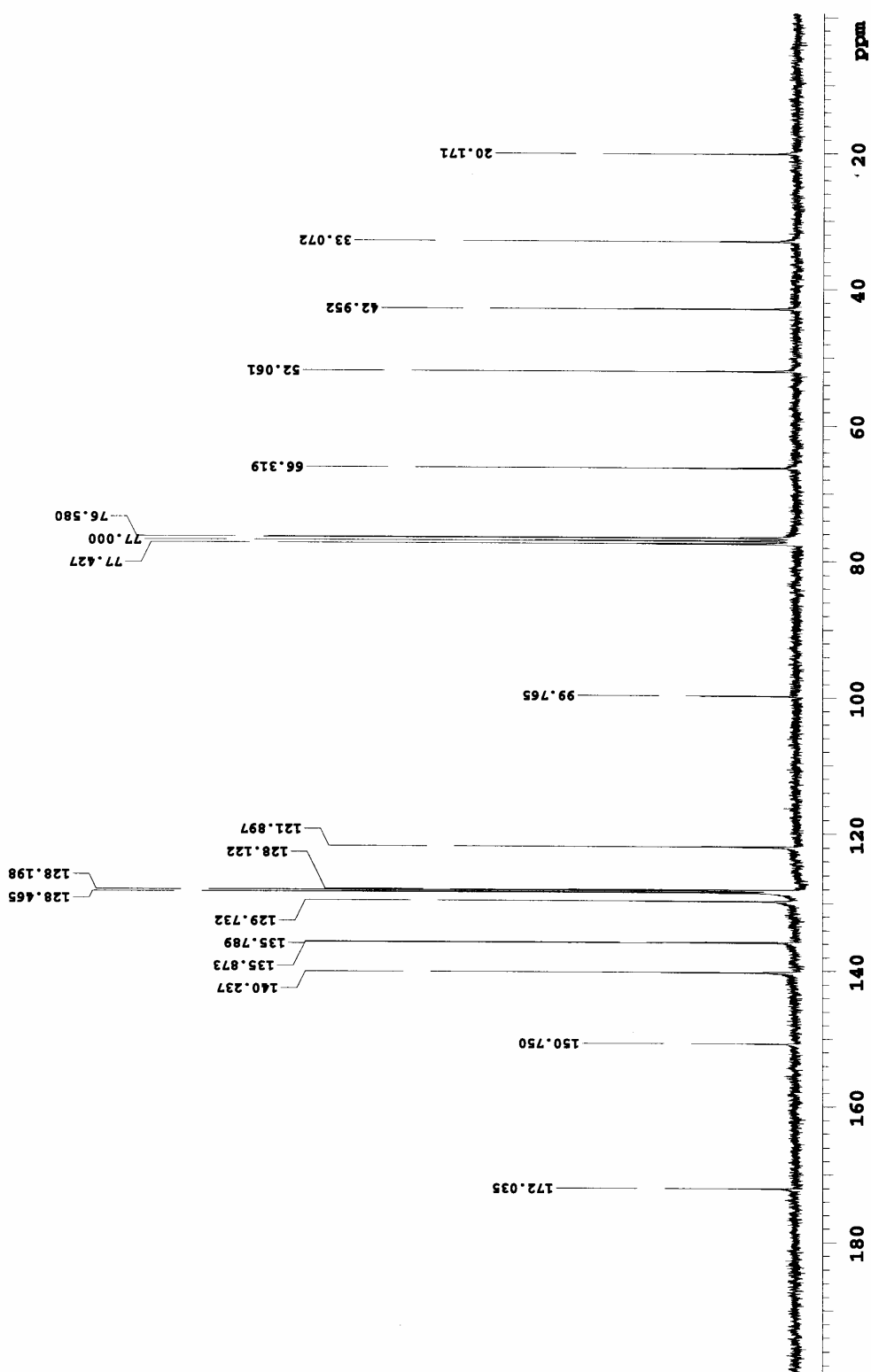
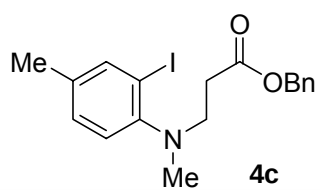
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DATA PROCESSING
Recoupling phase -0.0 Hz
FT size 32768

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5	2112.454	7.040
6	1167.725	3.892
7	1097.398	3.657
8	1022.089	3.406
9	837.773	2.792
10	781.511	2.605
11	466.504	1.555
12	-0.000	-0.000









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H1 / s2pul / Gemini-300A
cdcl3/Temp: 25C / N rg: PCB260
Acq. time 3.000 sec / 267del
No. of scans 16 / MS
Data: 26/05/06 / Sist automàtic

Pulse Sequence: s2pul

Solvent: cdcl3
Temp: 25.0 C / 298.1 K
Sample #34, user san
File: s2pul
GEMINI-300 "zapc"

Relax. delay 1.000 sec
Pulse: 45.0 degrees
Acq. time 3.000 sec
Width 4800.7 Hz
16 repetitions

OBSERVE H1, 300.045 MHz
DATA PROCESSING
Resol: 0.0768
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Phase shift: 0.0

