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Supporting Information

Pd PEPPSI-IPr-Mediated Reactions in Metal-Coated Capillaries Under Microwave-Assisted, Continuous Flow Organic Synthesis (MACOS): The Synthesis of Indoles by Sequential Aryl Amination/Heck Coupling

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LC-MS of compound **3u** S62

Experimental

Microwave irradiation experiments:

All MACOS experiments were performed in 1180 μm borosilicate capillaries, using a single mode Biotage Smith Creator Synthesizer, operating at a frequency of 2.45 GHz with irradiation power from 0 to 300 W. The capillaries were fed reactants from Hamilton gastight syringes attached to a Harvard 22 syringe pump pre-set to the desired flow rate. The system was connected to a sealed collection vial, where a pressurized air line was attached to create backpressure (pressure inside the system reached 75 psi). The temperatures reported were measured by an IR sensor built into the microwave chamber that reads the temperature on the outer surface of capillaries. All reagents and solvents were purchased from commercial sources and used without additional purification. Column chromatography purifications were carried out using the flash technique on silica gel 60 (200 – 400 mesh). ^{1}H NMR spectroscopy was run using a Bruker Advance 400 MHz instrument and all spectra were calibrated to 7.26 ppm for the signal from the residual proton of the deuterated chloroform solvent.

Purity analyses were conducted on Waters 2695LC – Quattro Ultima Mass Spectrometer (MS) using an H_2O-CH_3CN solvent system; the MS was run in "ESI+, dual scan" mode (T = 350°C). The

LC columns used were Synergy 4 micron Hydro RP 150mm X 4.6mm (T = 20°C) and Gemini C18 4 micron 150mm X 4.6mm (T = 24°C).

General Procedure for creating the Pd- and Ag-film coating inside of 1180 micron (ID) capillaries.

Pd film coating. Borosilicate capillaries (1180 μm internal diameter) were filled with a 0.1 mmol/mL solution of palladium acetate in DMF, capped at both ends with Teflon tape and placed inside a muffle furnace; the temperature was increased to 120° C. After 10-30 min., metallic Pd was gradually released from the solution and deposited on the inner side of capillaries. Capillaries were drained, rinsed with fresh DMF and the tubes with their new thin films were calcinated (dry) in the same furnace for 1 min. at 400°C (X3) before use in MACOS.

Ag film coating (the "silver-mirror" approach). Tollen's reagent (0.5 mL) was mixed with 0.5 mL of 5% D-glucose solution into a 2 mL vial. The 1180 μm capillaries were filled with this mixture, capped at both ends with Teflon tape and left to develop at room temperature. After the Ag coating was fully developed (5-10 min), the remaining solution was poured out, the capillaries were rinsed with acetone and then they were calcinated at 400°C in a muffle furnace before use.

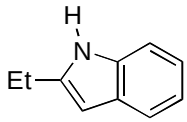
Tollen's reagent was prepared as follows: 1.5 mL of 4M NaOH was added drop-wise into 20 mL of a 5% $AgNO_3$ solution, forming a gray precipitate that was titrated with 4M NH_4OH until the solution became clear.

Ag film coating (the "colloidal silver" approach). Borosilicate capillaries (1180 μm internal diameter) were filled with a 0.5 mmol/mL colloidal solution of silver oxide in ethylene glycol, capped at both ends with Teflon tape and placed inside a muffle furnace; the temperature was gradually increased to 140° C. After the Ag coating was fully developed (30 min), capillaries were rinsed with acetone and placed inside a muffle furnace for calcination at 400°C before use.

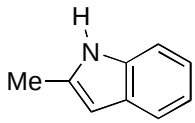
General Procedure for the Indole Synthesis.

A stock solution containing the substituted bromo alkene **1** (0.75 mmol, 1.5 equiv.), o-bromo aniline **2** (0.5 mmol, 1.0 equiv.), sodium t-butoxide (1.5 mmol, 3.0 equiv.), Pd PEPPSI-IPr (18 mg, 2.6 mol %.) in 0.8 mL toluene (total mixture volume is 1.0 mL) was prepared.

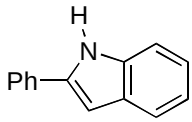
The continuous flow microwave system was primed with toluene and a 1 mL aliquot from the homogenous stock solution was taken up in a Hamilton gastight syringe that was connected to the reactor system with the aid of Microtight™ fittings. The syringe was placed in a Harvard 22 syringe pump that was set to deliver 15 μL/min and the single mode microwave was programmed so as to keep the temperature constant at the specified levels. The output (effluent) from the reactor was fed into a sealed, pressurized vial (75 psi) and then analyzed by ^{1}H NMR spectroscopy immediately after the reaction. Typically 0.5-0.8 mL of the crude reaction mixture was collected and the product was purified by silica gel column chromatography.



2-Ethyl-1H-indole (3a). Following the general procedure above for the preparation of indoles using MACOS, 0.6 mL of crude reaction mixture, derived from **1a** and **2a**, were collected and purification by flash chromatography (14% ethyl acetate in hexane) afforded 35.1 mg of **3a** in 81% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 (br s, 1H), 7.61 (d, J = 2.1 Hz, 1H), 7.32 (d, J = 8.0 Hz, 1H), 7.17 (m, 2H), 6.32 (s, 1H), 2.82 (q, J = 7.5 Hz, 2H), 1.39 (d, J = 7.5 Hz, 3H). Spectra matched that found in the literature.[3]

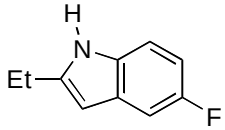


2-Methyl-1H-indole (3b). Following the general procedure above for the preparation of indoles using MACOS, 0.55 mL of crude reaction mixture, derived from **1b** and **2a**, were collected and purification by flash chromatography (20% acetone in hexane) afforded 26 mg of **3b** in 72% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (br s, 1H), 7.53 (d, J = 8.1 Hz, 1H), 7.31 (d, J = 8.0 Hz, 1H), 7.10 (m, 2H), 6.24 (s, 1H), 2.47 (s, 3H). Spectra matched that found in the literature.[1,2]

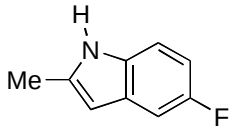


2-Phenyl-1H-indole (3c). Following the general procedure above for the preparation of indoles using MACOS, 0.7 mL of crude reaction mixture, derived from **1c** and **2a**, were collected and purification by flash chromatography (12% ethyl acetate in hexane) afforded 60 mg of **3c** in 74% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.35 (br s, 1H), 7.68 (m, 3H), 7.46 (m, 3H), 7.36 (t, J = 8.0 Hz,

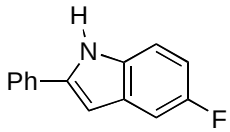
1H), 7.23 (t, J = 7.1 Hz, 1H), 7.15 (t, J = 7.1 Hz, 1H), 6.86 (s, 1H). Spectra matched that found in the literature.[1]



2-Ethyl-5-fluoro-1H-indole (3d). Following the general procedure above for the preparation of indoles using MACOS, 0.55 mL of crude reaction mixture, derived from **1a** and **2b**, were collected and purification by flash chromatography (30% dichloromethane in hexane) afforded 33.6 mg of **3d** in 76% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 (br s, 1H), 7.21 (m, 2H), 6.88 (t, $J_{^{1}H-^{19}F}$ = 9.0 Hz, 1H), 6.23 (s, 1H), 2.81 (q, J = 7.5 Hz, 2H), 1.37 (t, J = 7.5 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.9 ($^{1}J_{^{13}C-^{19}F}$ = 232.3 Hz), 143.2, 132.3, 129.2 ($^{3}J_{^{13}C-^{19}F}$ = 10.2 Hz), 110.6 ($^{3}J_{^{13}C-^{19}F}$ = 10.2 Hz), 109.0 ($^{2}J_{^{13}C-^{19}F}$ = 26.3 Hz), 104.7 ($^{2}J_{^{13}C-^{19}F}$ = 23.4 Hz), 99.2 ($^{4}J_{^{13}C-^{19}F}$ = 4.4 Hz), 21.5, 13.1. Anal. Calcd. for $C_{10}H_{10}NF$: C, 73.88; H, 6.19; N, 8.58. Found: C, 73.90; H, 6,28; N, 8.48. Compound **3b** has been reported previously[9] without ^{1}H NMR or ^{13}C NMR spectra, which are reported here.

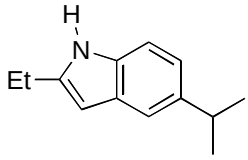


2-Methyl-5-fluoro-1H-indole (3e). Following the general procedure above for the preparation of indoles using MACOS, 0.58 mL of crude reaction mixture, derived from **1b** and **2b**, were collected and purification by flash chromatography (20% ethyl acetate in hexane) afforded 31.6 mg of **3e** in 73% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 (br s, 1H), 7.19 (m, 2H), 6.87 (dt, $J_{^{1}H-^{19}F}$ = 9.0, 2.0 Hz, 1H), 6.21 (s, 1H), 2.46 (s, 3H). Spectra matched that found in the literature.[5]

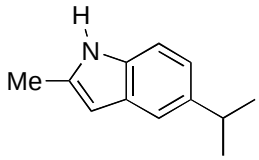


2-Phenyl-5-fluoro-1H-indole (3f). Following the general procedure above for the preparation of indoles using MACOS, 0.78 mL of crude reaction mixture, derived from **1c** and **2b**, were collected and purification by flash chromatography (30% dichloromethane in hexane) afforded 64.0 mg of **3f** in 78% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.33 (br s, 1H), 7.68 (d, J = 7.0 Hz, 2H), 7.48 (t, J = 8.0

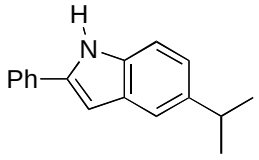
Hz, 2H), 7.35 (m, 3H), 6.97 (dt, J^{1H-19F} = 9.0, 3.0 Hz, 1H), 6.81 (s, 1H). Spectra matched that found in the literature.[5,7]



2-Ethyl-5-isopropyl-1H-indole (3g). Following the general procedure above for the preparation of indoles using MACOS, 0.61 mL of crude reaction mixture, derived from **1a** and **2c**, were collected and purification by flash chromatography (30% dichloromethane in hexane) afforded 47.4 mg of **3g** in 83% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68 (br s, 1H), 7.55 (s, 1H), 7.29 (d, J = 8.1 Hz, 1H), 7.18 (d, J = 8.1 Hz, 1H), 6.34 (s, 1H), 3.16 (septet, J = 7.1 Hz, 1H), 2.83 (q, J = 7.1 Hz, 2H), 1.45 (m, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 141.7, 140.2, 134.5, 129.0, 120.2, 116.9, 110.1, 98.5, 34.4, 24.9, 21.5, 13.5. HRMS *m/z* calcd for $C_{13}H_{17}N$: 187.1361; found: 187.1353.

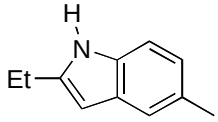


2-Methyl-5-isopropyl-1H-indole (3h). Following the general procedure above for the preparation of indoles using MACOS, 0.68 mL of crude reaction mixture, derived from **1b** and **2c**, were collected and purification by flash chromatography (30% dichloromethane in pentane) afforded 46.0 mg of **3h** in 79% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69 (br s, 1H), 7.44 (s, 1H), 7.24 (d, J = 8.1 Hz, 1H), 7.09 (d, J = 8.1 Hz, 1H), 6.24 (s, 1H), 3.07 (septet, J = 7.1 Hz, 1H), 2.45 (s, 3H), 1.38 (d, J = 7.1 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.4, 135.3, 134.6, 129.2, 120.1, 116.7, 110.0, 100.1, 34.2, 24.7, 13.7. HRMS *m/z* calcd for $C_{12}H_{15}N$: 173.1204; found: 173.1206.

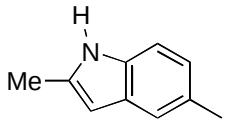


2-Phenyl-5-isopropyl-1H-indole (3i). Following the general procedure above for the preparation of indoles using MACOS, 0.72 mL of crude reaction mixture, derived from **1c** and **2c**, were collected and purification by flash chromatography (30% dichloromethane in pentane) afforded 68.0 mg of **3i** in 80% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.27 (br s, 1H), 7.70 (d, J = 7.1 Hz, 2H), 7.56 (s, 1H), 7.49 (t, J = 8.1 Hz, 2H), 7.38 (m, 2H), 7.18 (d, J = 8.1 Hz, 1H), 6.86 (s, 1H), 3.10 (septet, J = 7.1 Hz,

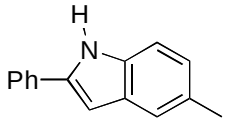
1H), 1.41 (d, J = 7.1 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.9, 138.0, 135.4, 132.6, 129.4, 128.9, 127.5, 125.1, 121.6, 117.5, 110.6, 99.8, 34.2, 24.6. HRMS *m/z* calcd for $C_{17}H_{17}N$: 235.1361; found: 235.1355.



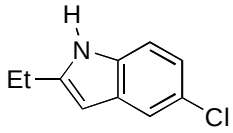
2-Ethyl-5-methyl-1H-indole (3j). Following the general procedure above for the preparation of indoles using MACOS, 0.66 mL of crude reaction mixture, derived from **1a** and **2d**, were collected and purification by flash chromatography (20% ethyl acetate in hexane) afforded 39.5 mg of **3j** in 75% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79 (br s, 1H), 7.35 (s, 1H), 7.21 (d, J = 8.0 Hz, 1H), 6.96 (d, J = 8.1 Hz, 1H), 6.19 (s, 1H), 2.80 (q, J = 8.0 Hz, 2H), 2.46 (s, 3H), 1.36 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 141.4, 134.6, 130.2, 128.7, 122.4, 119.5, 109.9, 98.2, 21.5, 21.3, 13.2. HRMS *m/z* calcd for $C_{11}H_{13}N$: 159.1048; found: 159.1050. Compound **3j** has been reported previously[9] without ^{1}H NMR or ^{13}C NMR spectra, which are reported here.



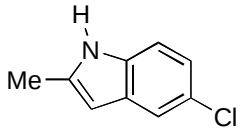
2-Methyl-5-methyl-1H-indole (3k). Following the general procedure above for the preparation of indoles using MACOS, 0.84 mL of crude reaction mixture, derived from **1b** and **2d**, were collected and purification by flash chromatography (20% acetone in hexane) afforded 45.8 mg of **3k** in 82% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76 (br s, 1H), 7.33 (s, 1H), 7.19 (d, J = 8.1 Hz, 1H), 6.96 (d, J = 9.1 Hz, 1H), 6.16 (s, 1H), 2.45 (s, 6H). Spectra matched that found in the literature.[6]



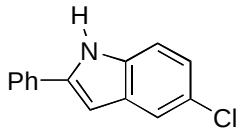
2-Phenyl-5-methyl-1H-indole (3l). Following the general procedure above for the preparation of indoles using MACOS, 0.75 mL of crude reaction mixture, derived from **1c** and **2d**, were collected and purification by flash chromatography (12% ethyl acetate in hexane) afforded 65.8 mg of **3l** in 85% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.26 (br s, 1H), 7.68 (m, 2H), 7.45 (m, 3H), 7.31 (m, 2H), 7.05 (d, J = 7.0 Hz, 1H), 6.78 (s, 1H), 2.48 (s, 3H). Spectra matched that found in the literature.[4]



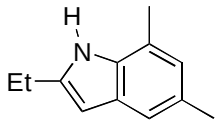
2-Ethyl-5-Chloro-1H-indole (3m). Following the general procedure above for the preparation of indoles using MACOS, 0.82 mL of crude reaction mixture, derived from **1a** and **2e**, were collected and purification by flash chromatography (20% ethyl acetate in hexane) afforded 51.4 mg of **3m** in 70% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (br s, 1H), 7.51 (s, 1H), 7.22 (d, J = 8.1 Hz, 1H), 7.08 (dd, J = 9.1, 2.0 Hz, 1H), 6.21 (s, 1H), 2.81 (q, J = 8.1 Hz, 2H), 1.37 (t, J = 8.1 Hz, 3H). Spectra matched that found in the literature.[2]



2-Methyl-5-Chloro-1H-indole (3n). Following the general procedure above for the preparation of indoles using MACOS, 0.7 mL of crude reaction mixture, derived from **1b** and **2e**, were collected and purification by flash chromatography (20% acetone in hexane) afforded 35.8 mg of **3n** in 62% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 (br s, 1H), 7.49 (d, J = 2.3 Hz, 1H), 7.20 (d, J = 8.7 Hz, 1H), 7.07 (dd, J = 8.7, 2.0 Hz, 1H), 6.19 (s, 1H), 2.49 (s, 3H). Spectra matched that found in the literature.[5]

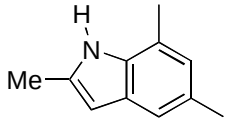


2-Phenyl-5-Chloro-1H-indole (3o). Following the general procedure above for the preparation of indoles using MACOS, 0.5 mL of crude reaction mixture, derived from **1c** and **2e**, were collected and purification by flash chromatography (16% ethyl acetate in hexane) afforded 37.4 mg of **3o** in 69% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.38 (br s, 1H), 7.68 (d, J = 8.0 Hz, 2H), 7.61 (d, J = 2.1 Hz, 1H), 7.48 (t, J = 7.0 Hz, 2H), 7.35 (m, 2H), 7.17 (dd, J = 9.1, 2.0 Hz, 1H), 6.79 (s, 1H). Spectra matched that found in the literature.[4]

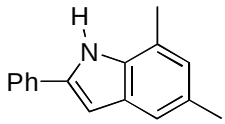


2-Ethyl-5,7-dimethyl-1H-indole (3p). Following the general procedure above for the preparation of indoles using MACOS, 0.45 mL of crude reaction mixture, derived from **1a** and **2f**, were collected

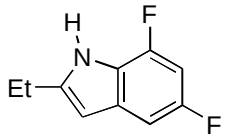
and purification by flash chromatography (10% acetone in hexane) afforded 27.0 mg of **3p** in 72% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72 (br s, 1H), 7.22 (s, 1H), 6.81 (s, 1H), 6.22 (s, 1H), 2.84 (q, J = 8.1 Hz, 2H), 2.48 (s, 3H), 2.46 (s, 3H), 1.39 (t, J = 8.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 141.1, 133.7, 129.0, 128.4, 123.4, 119.2, 117.6, 99.1, 21.8, 21.3, 15.7, 13.4. HRMS *m/z* calcd for $C_{12}H_{15}N$: 173.1204; found: 173.1205.



2-Methyl-5,7-dimethyl-1H-indole (3q). Following the general procedure above for the preparation of indoles using MACOS, 0.60 mL of crude reaction mixture, derived from **1b** and **2f**, were collected and purification by flash chromatography (20% acetone in hexane) afforded 37.5 mg of **3q** in 85% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70 (br s, 1H), 7.19 (s, 1H), 6.79 (s, 1H), 6.17 (s, 1H), 2.48 (s, 3H), 2.46 (s, 3H), 2.43 (s, 3H). Spectra matched that found in the literature.[10]

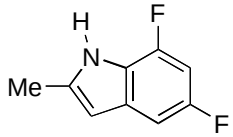


2-Phenyl-5,7-dimethyl-1H-indole (3r). Following the general procedure above for the preparation of indoles using MACOS, 0.40 mL of crude reaction mixture, derived from **1c** and **2f**, were collected and purification by flash chromatography (30% dichloromethane in hexane) afforded 36.4 mg of **3r** in 82% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 (br s, 1H), 7.71 (d, J = 7.0 Hz, 2H), 7.46 (m, 2H), 7.34 (m, 2H), 6.87 (s, 1H), 6.78 (s, 1H), 2.54 (s, 3H), 2.45 (s,3H). Spectra matched that found in the literature.[8]

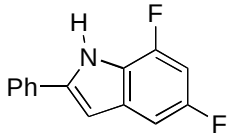


2-Ethyl-5,7-difluoro-1H-indole (3s). Following the general procedure above for the preparation of indoles using MACOS, 0.68 mL of crude reaction mixture, derived from **1a** and **2g**, were collected and purification by flash chromatography (10% ethyl ether in pentane) afforded 39.2 mg of **3s** in 64% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (br s, 1H), 7.01 (dd, $J_{^{1}H-^{19}F}$ = 9.0, 2.0 Hz, 1H), 6.68 (dt, $J_{^{1}H-^{19}F}$ = 10.0, 2.0 Hz, 1H), 6.28 (s, 1H), 2.82 (q, J = 7.5 Hz, 2H), 1.38 (t, J = 7.5 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.9 ($^{1}J_{^{13}C-^{19}F}$ = 235.6; 10.2 Hz), 147.9 ($^{1}J_{^{13}C-^{19}F}$ = 245.9; 14.6 Hz), 143.8, 131.3 ($^{2}J_{^{13}C-^{19}F}$ = 19.1 Hz), 120.4 ($^{3}J_{^{13}C-^{19}F}$ = 13.2 Hz), 100.5 ($^{2}J_{^{13}C-^{19}F}$ = 21.9;16.1 Hz), 99.9 ($^{4}J_{^{13}C-^{19}F}$ = 4.4

Hz), 95.9 ($^{2}J_{^{13}C-^{19}F}$ = 48.3; 20.5 Hz), 22.2, 13.2. HRMS *m/z* calcd for $C_{10}H_9NF_2$: 181.0703; found: 181.0701.



2-Methyl-5,7-difluoro-1H-indole (3t). Following the general procedure above for the preparation of indoles using MACOS, 0.60 mL of crude reaction mixture, derived from **1b** and **2g**, were collected and purification by flash chromatography (10% ethyl ether in pentane) afforded 32.7 mg of **3t** in 65% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 (br s, 1H), 6.98 (d, $J_{^{1}H-^{19}F}$ = 9.0 Hz, 1H), 6.66 (t, $J_{^{1}H-^{19}F}$ = 10.0 Hz, 1H), 6.24 (s, 1H), 2.48 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.3 ($^{1}J_{^{13}C-^{19}F}$ = 235.6; 10.2 Hz), 147.6 ($^{1}J_{^{13}C-^{19}F}$ = 244.4; 14.6 Hz), 138.2, 131.6 ($^{2}J_{^{13}C-^{19}F}$ = 17.5 Hz), 120.8 ($^{3}J_{^{13}C-^{19}F}$ = 11.7 Hz), 101.6 ($^{4}J_{^{13}C-^{19}F}$ = 4.4 Hz), 100.3 ($^{2}J_{^{13}C-^{19}F}$ = 23.4; 4.4 Hz), 96.1 ($^{2}J_{^{13}C-^{19}F}$ = 30.7; 20.5 Hz), 13.6. HRMS *m/z* calcd for $C_9H_7NF_2$: 167.0547; found: 167.0544.



2-Phenyl-5,7-difluoro-1H-indole (3u). Following the general procedure above for the preparation of indoles using MACOS, 0.50 mL of crude reaction mixture, derived from **1c** and **2g**, were collected and purification by flash chromatography (25% dichloromethane in pentane) afforded 36.0 mg of **3u** in 63% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.44 (br s, 1H), 7.69 (d, J = 8.1 Hz, 2H), 7.50 (t, J = 8.0 Hz, 2H), 7.41 (m, 1H), 7.11 (d, $J_{^{1}H-^{19}F}$ = 8.0 Hz, 1H), 6.83 (s, 1H), 6.77 (t, $J_{^{1}H-^{19}F}$ = 10.0 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.2 ($^{1}J_{^{13}C-^{19}F}$ = 237.1; 10.2 Hz), 148.4 ($^{1}J_{^{13}C-^{19}F}$ = 245.9; 14.6 Hz), 140.2, 131.7 ($^{3}J_{^{13}C-^{19}F}$ = 4.4 Hz), 131.2, 129.4, 128.1, 125.5, 121.6 ($^{2}J_{^{13}C-^{19}F}$ = 13.2 Hz), 101.0 ($^{2}J_{^{13}C-^{19}F}$ = 23.4; 4.4 Hz), 100.5, 97.4 ($^{2}J_{^{13}C-^{19}F}$ = 30.7; 20.5 Hz). HRMS *m/z* calcd for $C_{14}H_9NF_2$: 229.0703; found: 229.0692. Anal. Calcd. for $C_{14}H_9NF_2$: C, 73.36; H, 3.96; N, 6.11; F, 16.58. Found: C, 73.54; H, 3.58; N, 6.11; F, 16.38.

References

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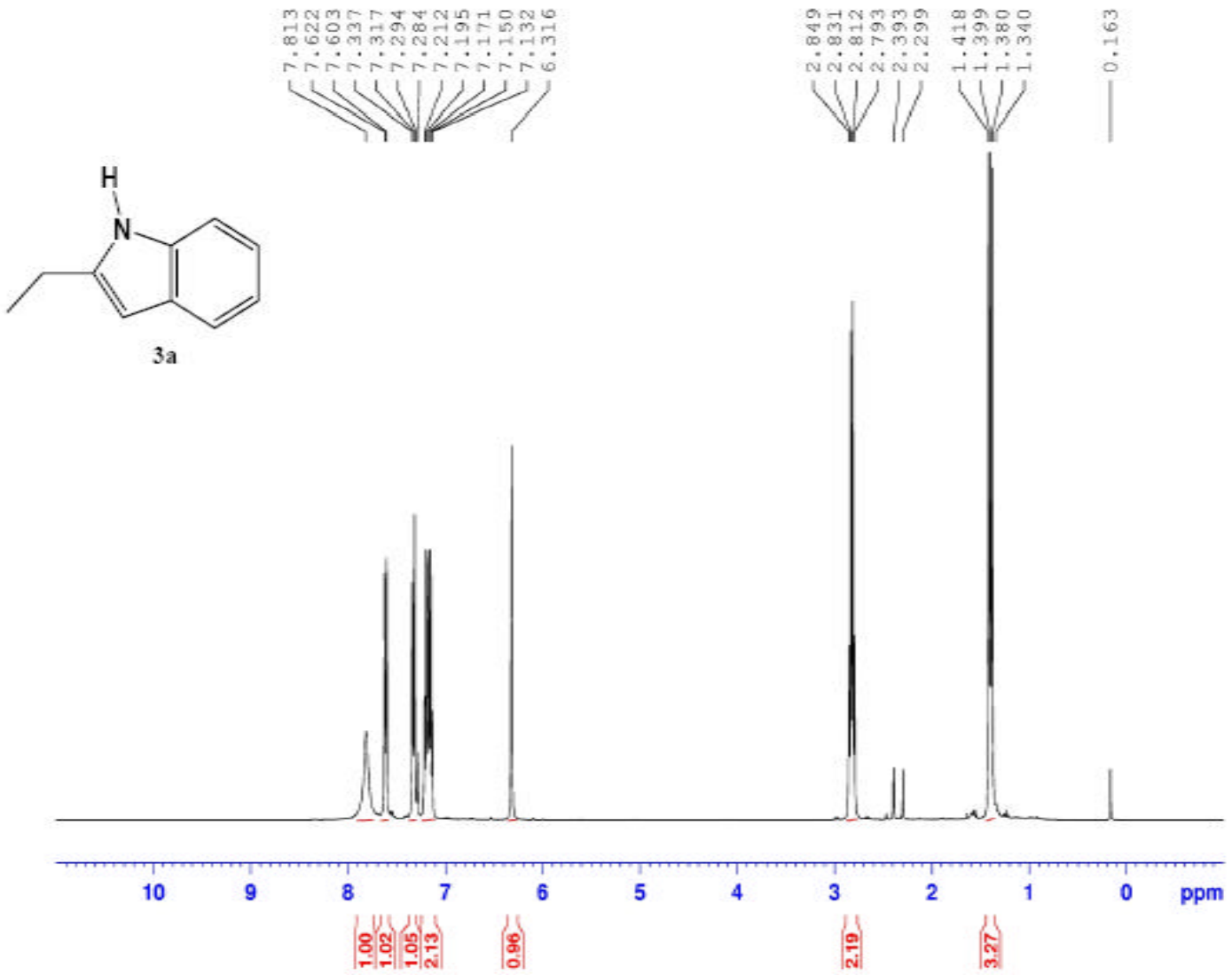
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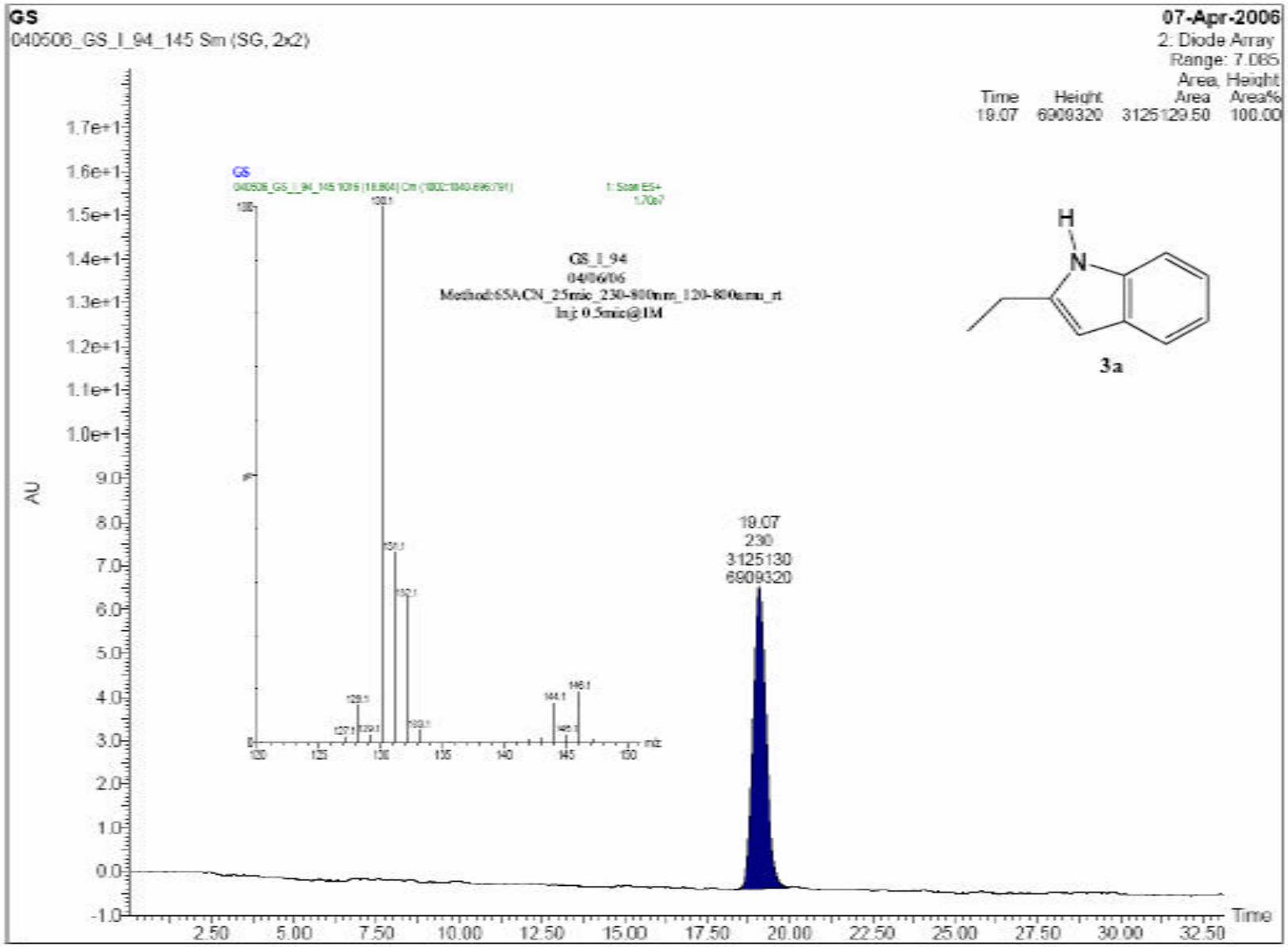
7.622
7.603
7.337
7.317
7.294
7.284
7.212
7.195
7.171
7.150
7.132
6.316
2.849
2.831
2.812
2.793
2.393
2.299
1.418
1.399
1.380
1.340
0.163
H
N
3a
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
1.02
1.05
2.13
0.96
2.19
3.27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07-Apr-2006
2: Diode Array
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Area, Height
Time Height Area Area%
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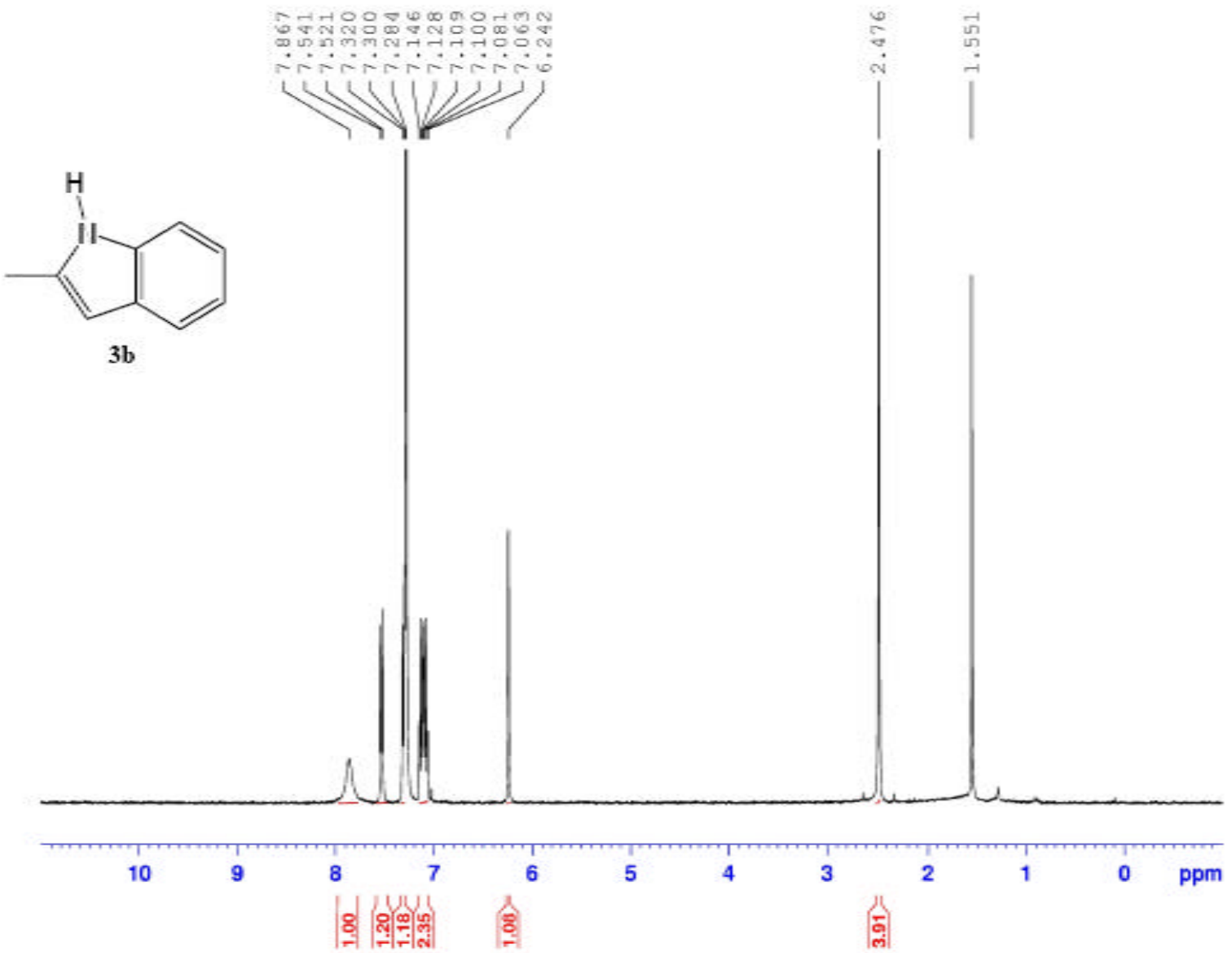
GS_I_94
04/06/06
Method:65ACN_25mic_230-800nm_120-800amu_rt
Inj: 0.5mic@1M
H
N
3a
19.07
230
3125130
6909320
AU
Time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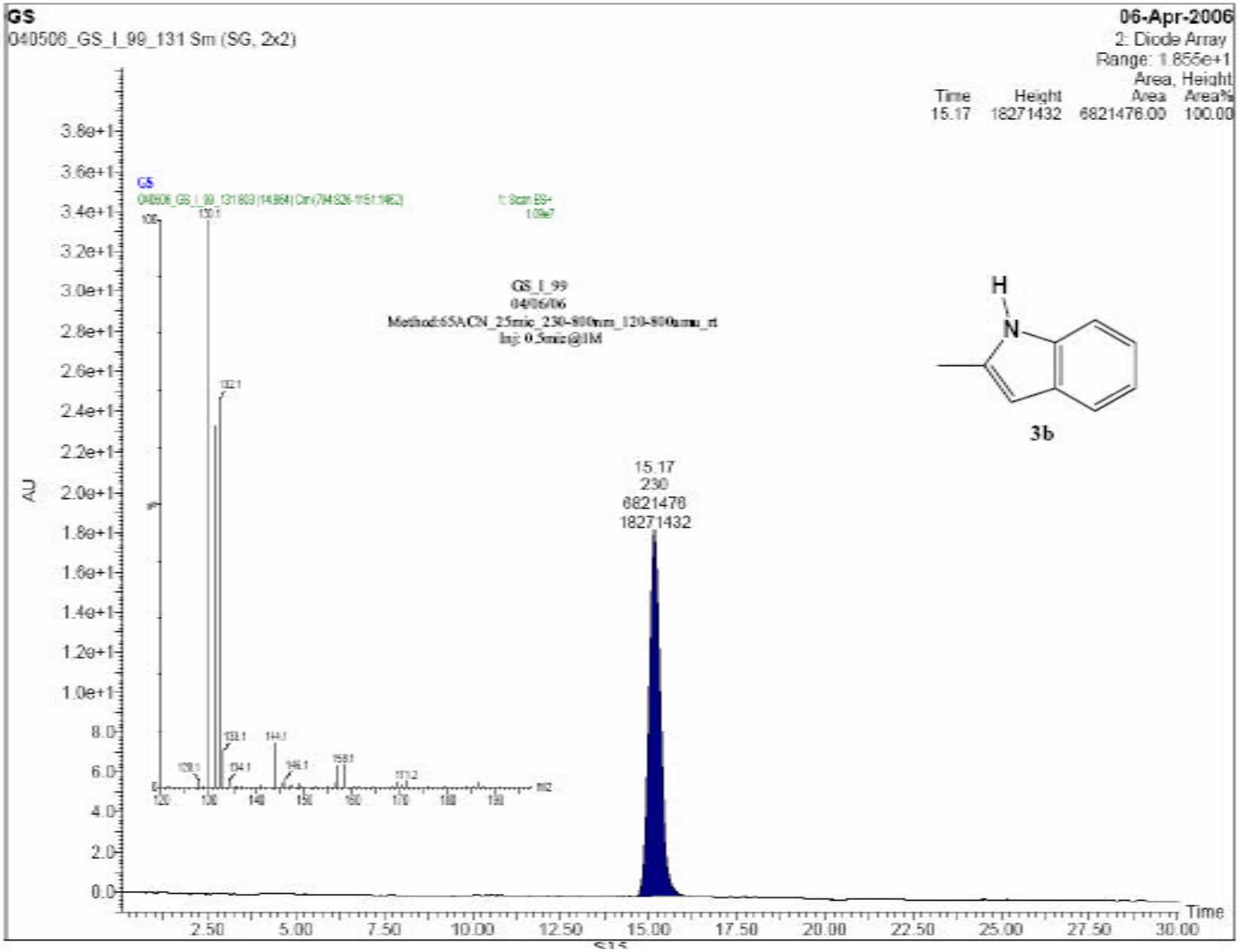
7.541
7.521
7.320
7.300
7.284
7.146
7.128
7.109
7.100
7.081
7.063
6.242
2.476
1.551
H
3b
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
1.20
1.18
2.35
1.06
3.91


GS
040506_GS_I_99_131 Sm (SG, 2x2)
06-Apr-2006
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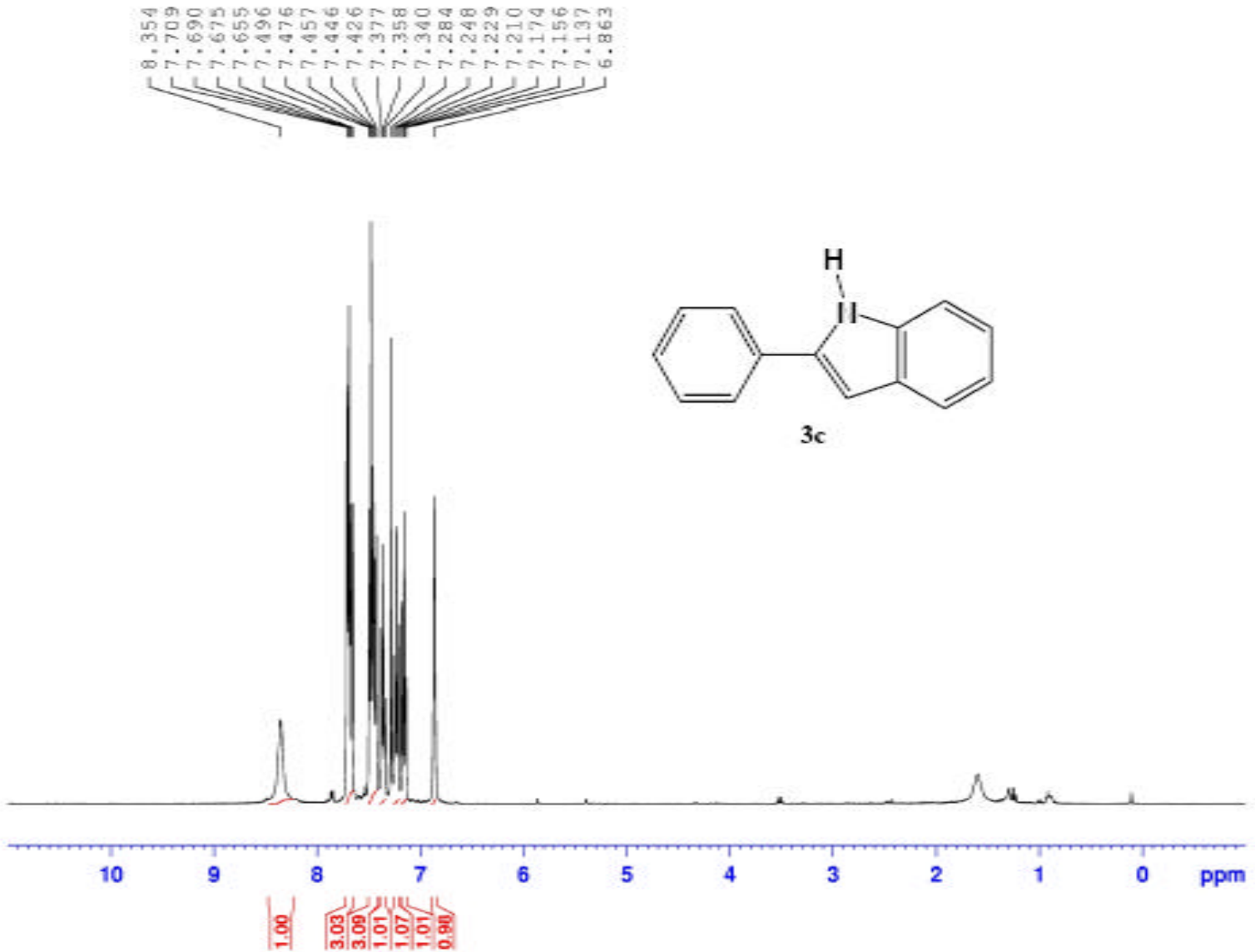
Range: 1.855e+1
Area, Height
Time Height Area Area%
15.17 18271432 6821476.00 100.00
GS
GS_I_99
04/06/06
Inj: 0.5mic@1M
H
N
3b
15.17
230
6821478
18271432
AU
Time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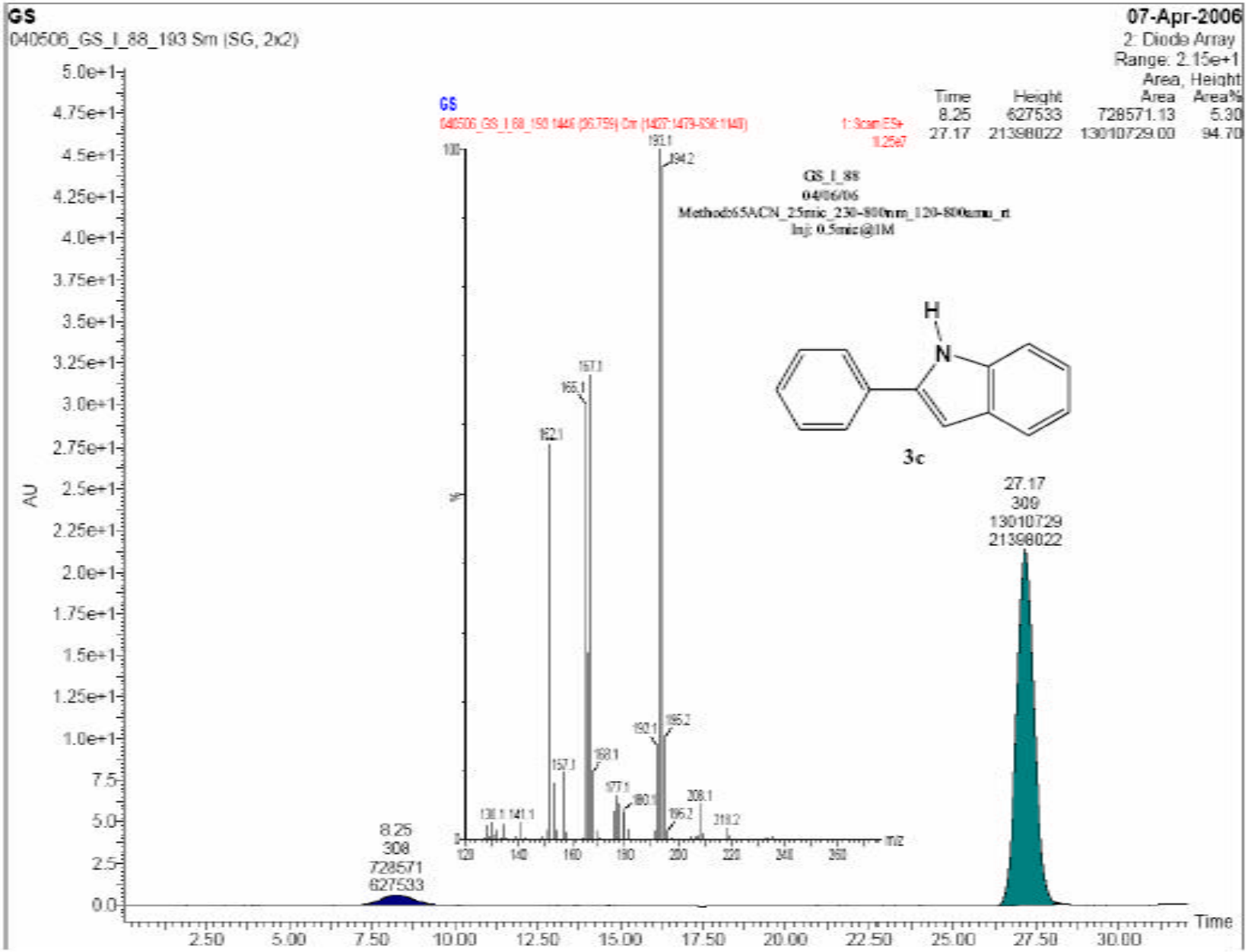
7.709
7.690
7.675
7.655
7.496
7.476
7.457
7.446
7.426
7.377
7.358
7.340
7.284
7.248
7.229
7.210
7.174
7.156
7.137
6.863
H
3c
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
3.03
3.09
1.01
1.07
1.01
0.98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040506_GS_I_88_193 Sm (SG, 2x2)
07-Apr-2006
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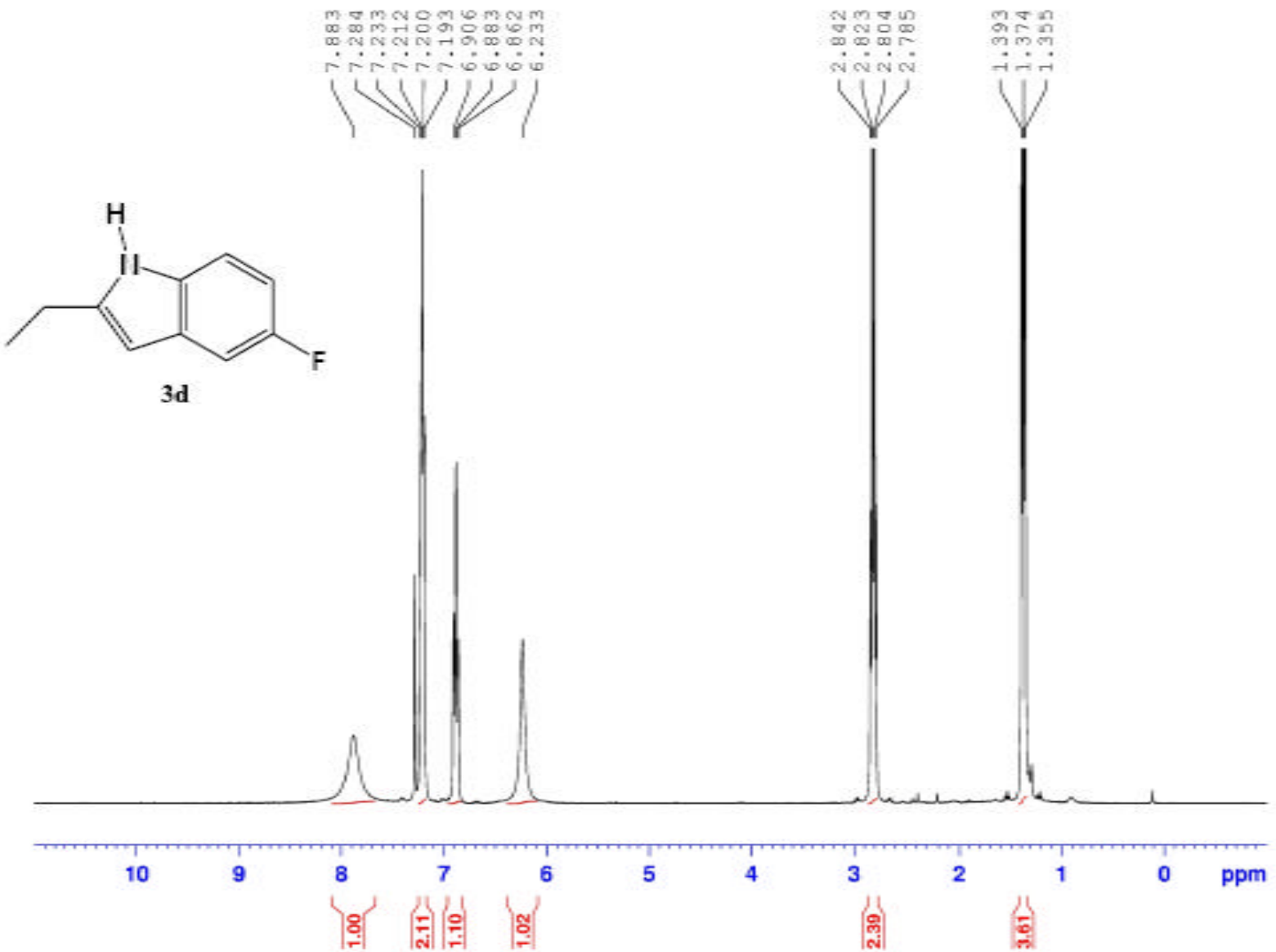
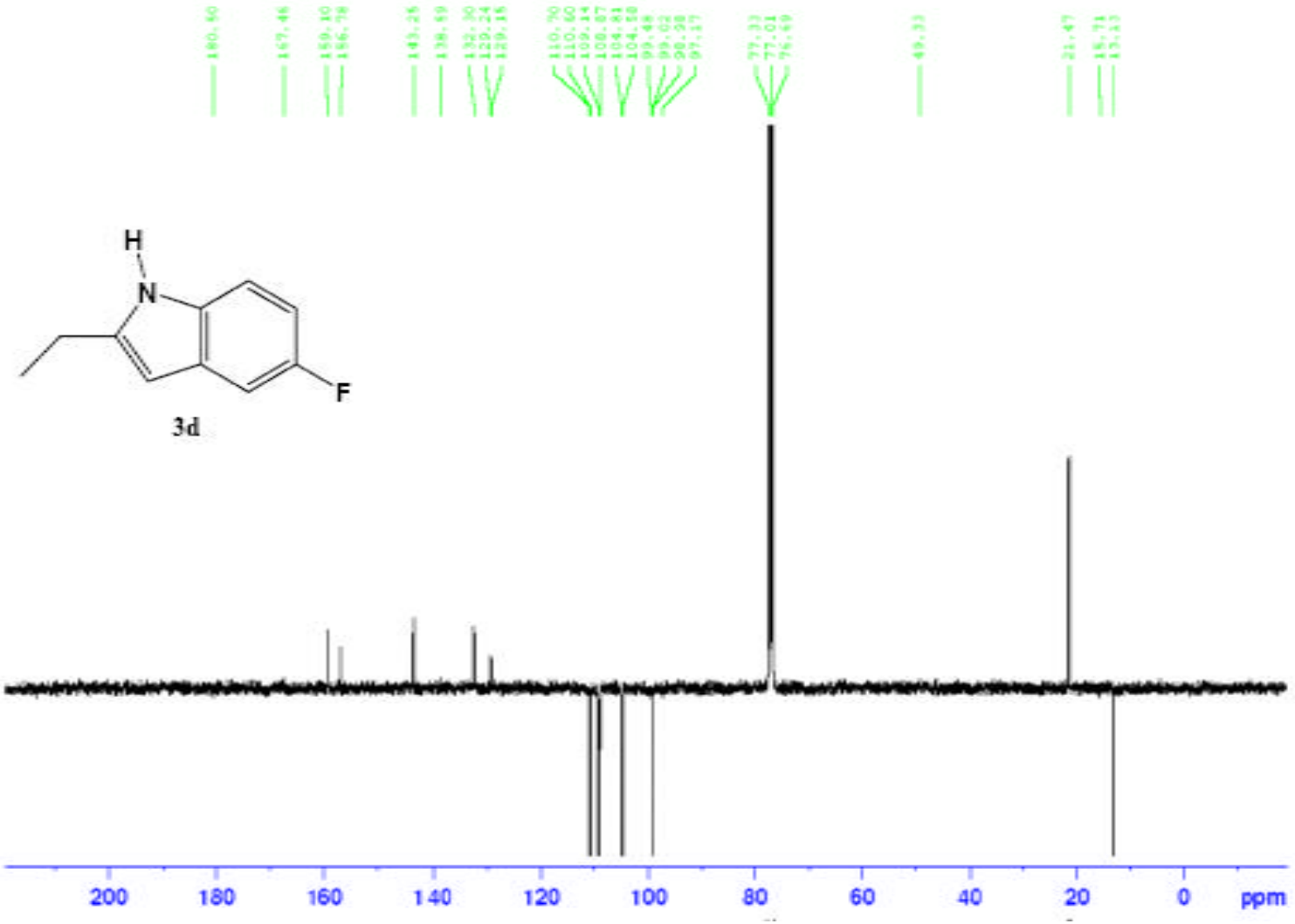
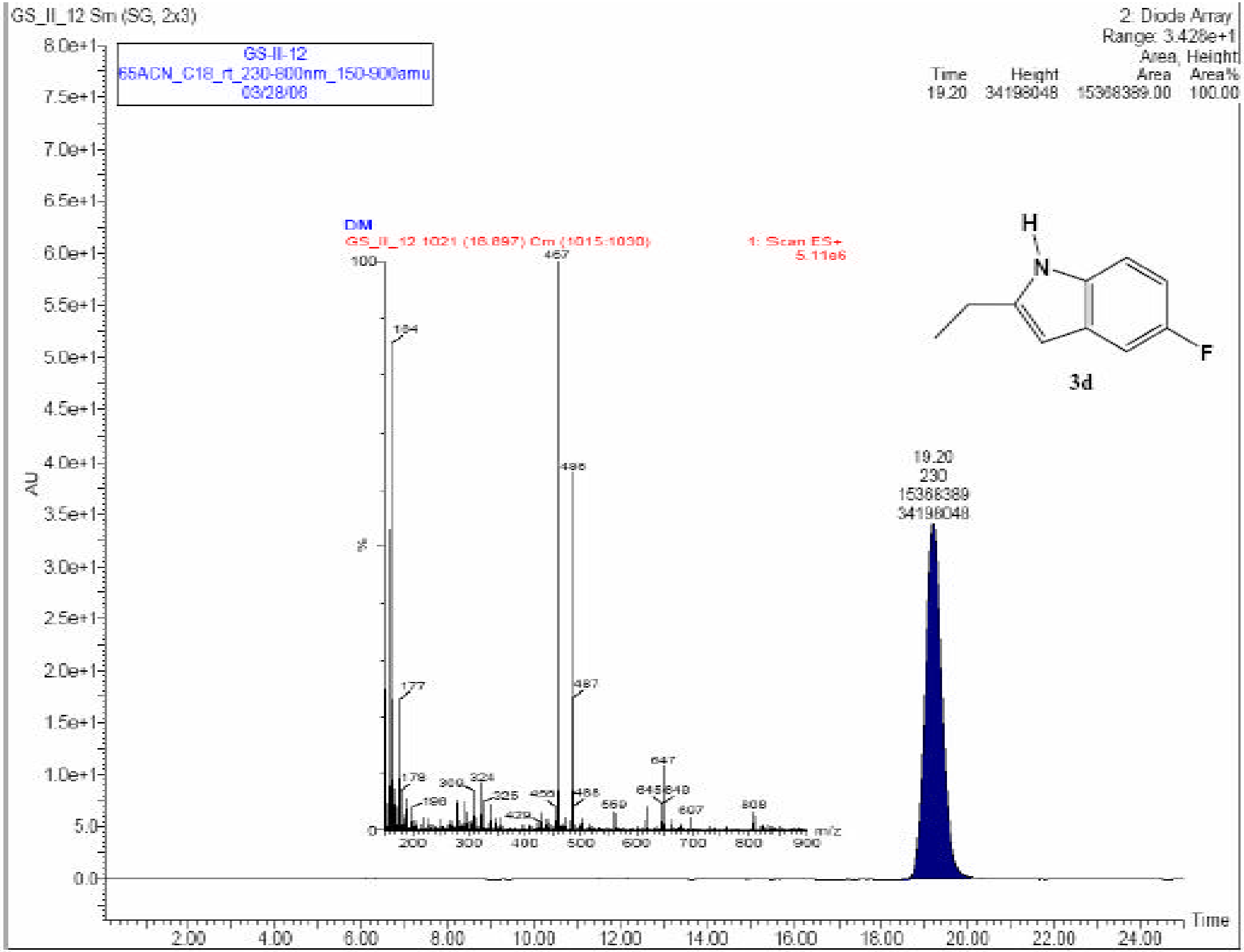
Area, Height
Time Height Area Area%
8.25 627533 728571.13 5.30
27.17 21398022 13010729.00 94.70
GS
040506_GS_I_88_150 1446 (26.759) Cm (1427:1479-936:1148)
1: Scan ES+
1.25e7
GS_I_88
04/06/06
Method65ACN_25min_230-800nm_120-800amu_rt
Inj: 0.5mic@1M
3c
8.25
308
728571
627533
27.17
309
13010729
21398022
AU
Time


7.883
7.284
7.233
7.212
7.200
7.193
6.906
6.883
6.862
6.233
2.842
2.823
2.804
2.785
1.393
1.374
1.355
H
F
3d
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
2.11
1.10
1.02
2.39
3.61


180.50
167.46
159.10
156.78
143.25
138.59
132.30
129.24
129.15
110.70
110.60
109.44
108.87
104.81
104.58
99.48
99.02
98.98
97.17
77.32
77.01
76.69
49.33
21.47
15.71
13.13
H
N
F
3d
200
180
160
140
120
100
80
60
40
20
0
ppm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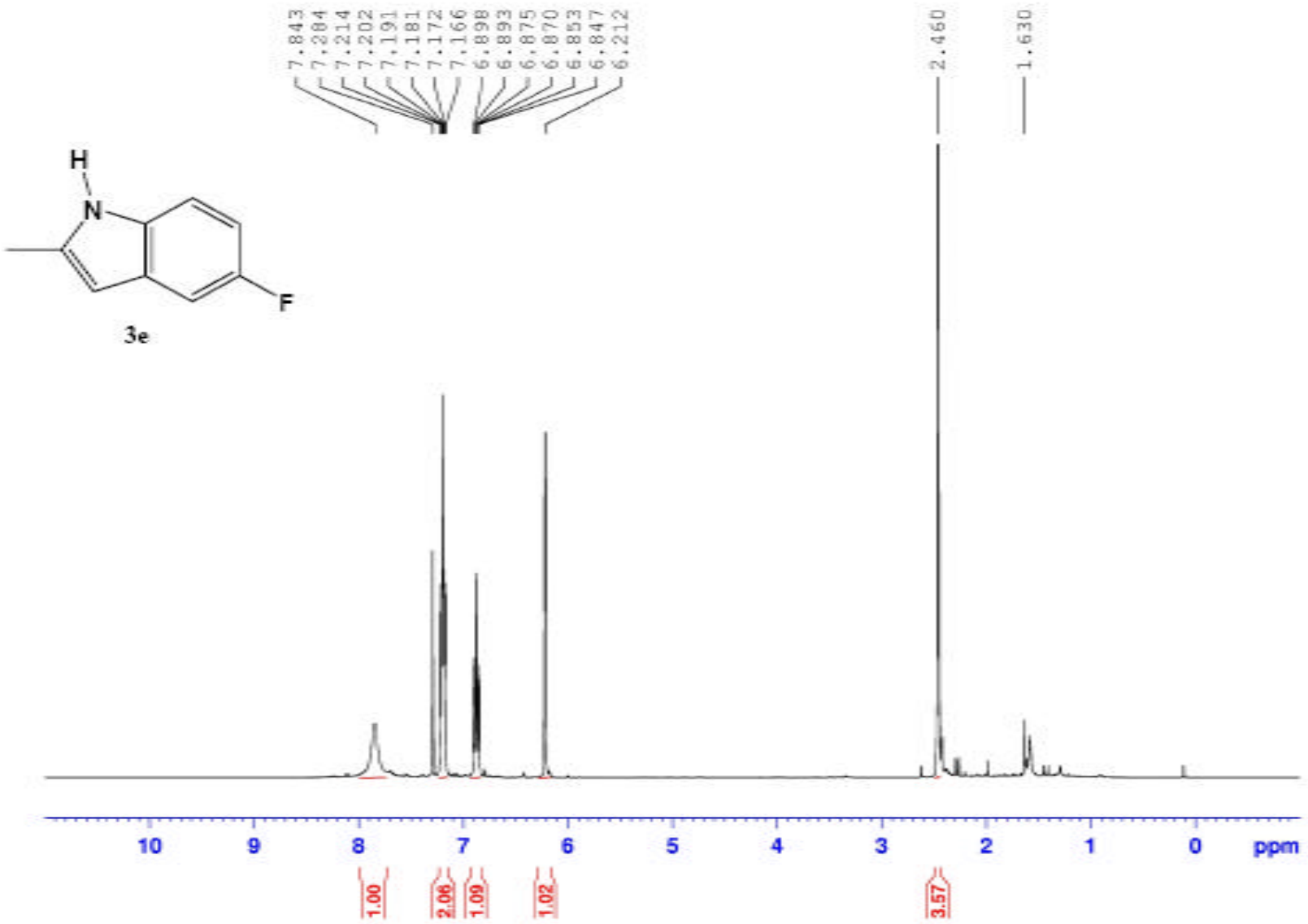
Area, Height
GS-II-12
65ACN_C18_rt_230-800nm_150-900amu
03/28/06
Time Height Area Area%
19.20 34198048 15368389.00 100.00
AU
Time
19.20
230
15368389
34198048
DM
GS_II_12 1021 (16.897) Cm (1015:1030)
1: Scan ES+
5.11e6
%
m/z
3d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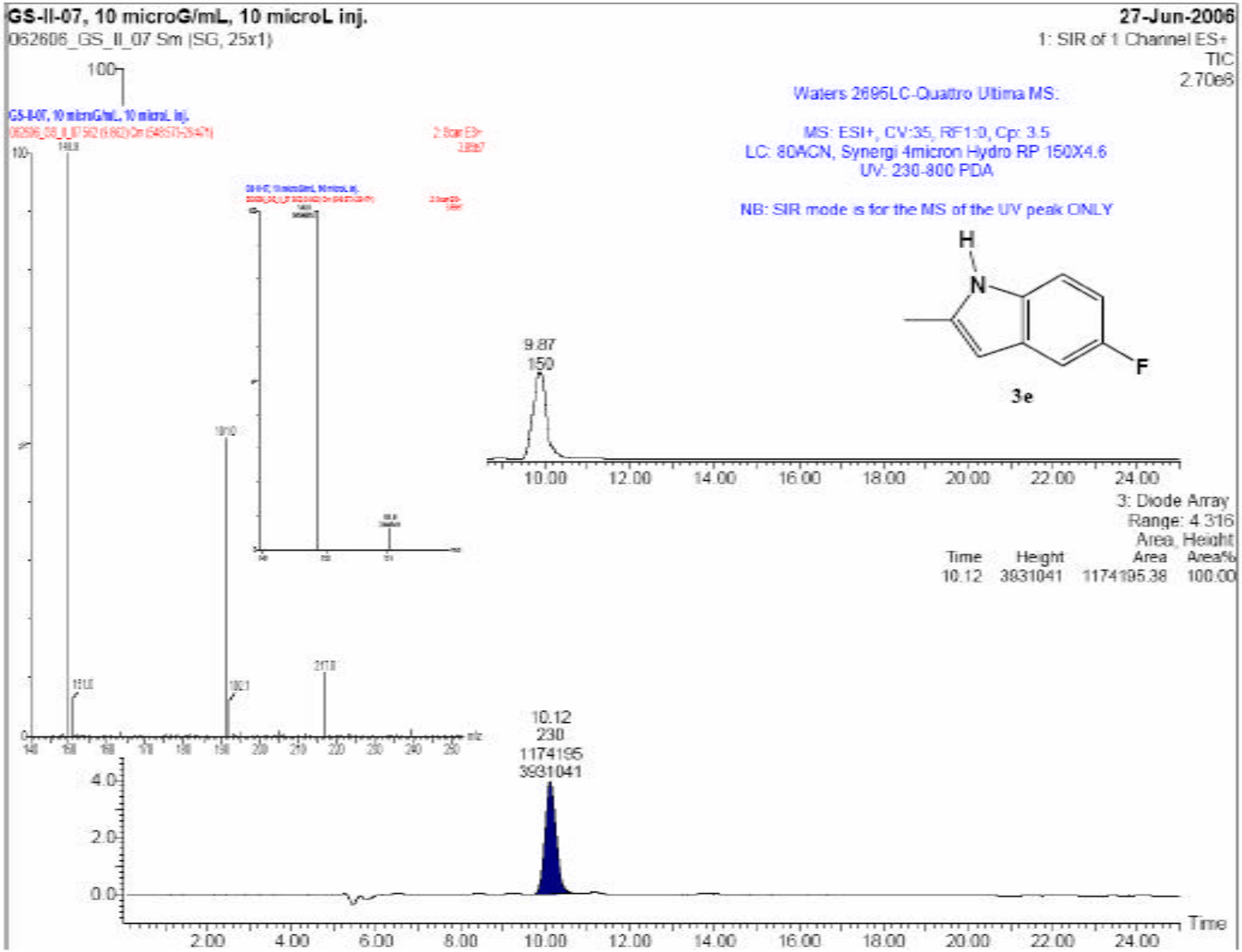
7.214
7.202
7.191
7.181
7.172
7.166
6.898
6.893
6.875
6.870
6.853
6.847
6.212
2.460
1.630
H
N
F
3e
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
2.06
1.09
1.02
3.57


GS-II-07, 10 microG/mL, 10 microL inj.
062606_GS_II_07 Sm (SG, 25x1)
27-Jun-2006
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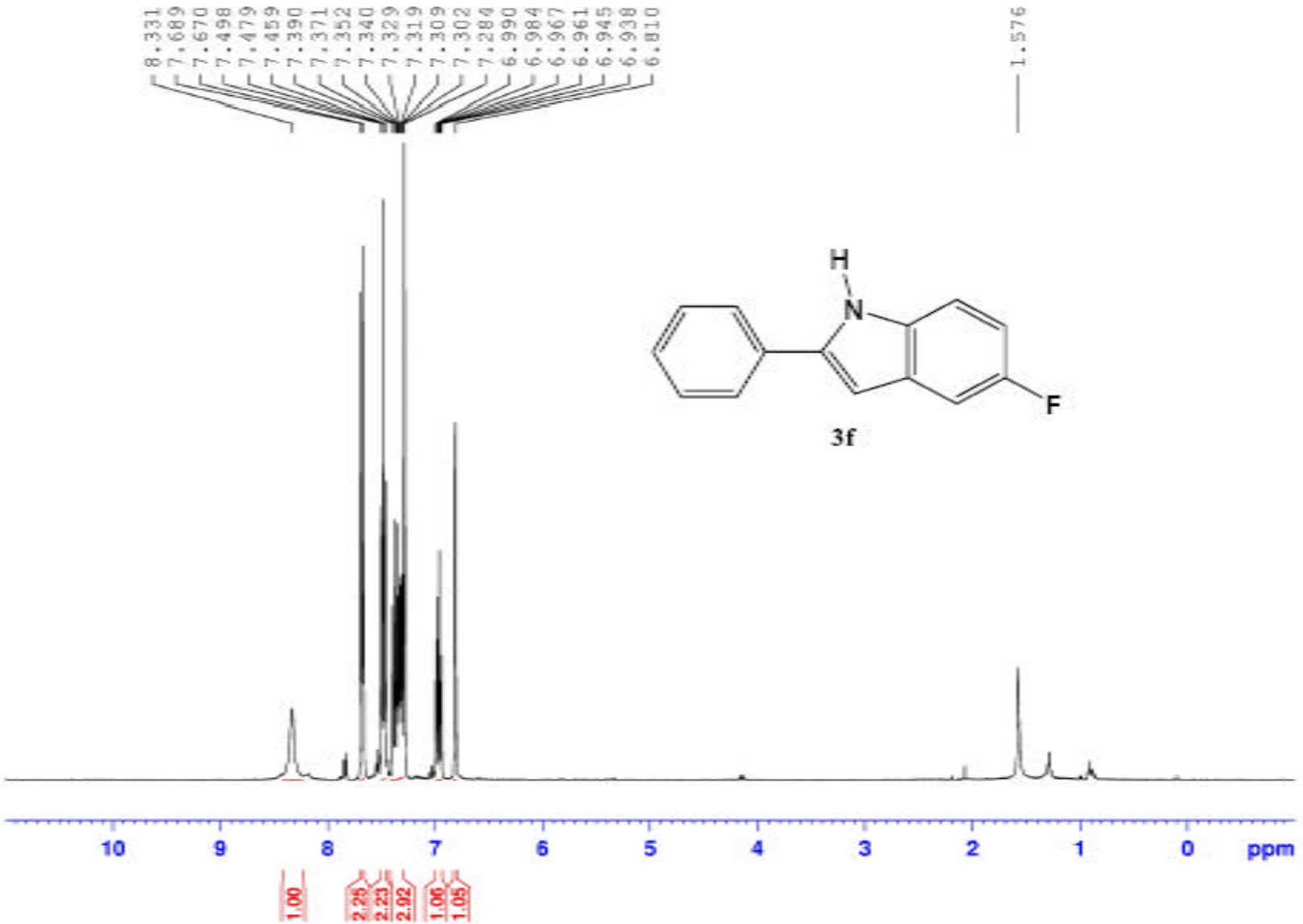
TIC
2.70e8
Waters 2695LC-Quattro Ultima MS:
MS: ESI+, CV:35, RF1:0, Cp: 3.5
LC: 80ACN, Synergi 4micron Hydro RP 150X4.6
UV: 230-800 PDA
NB: SIR mode is for the MS of the UV peak ONLY
H
N
F
3e
9.87
150
10.00
12.00
14.00
16.00
18.00
20.00
22.00
24.00
3: Diode Array
Range: 4.316
Area, Height
Time Height Area Area%
10.12 3931041 1174195.38 100.00
10.12
230
1174195
3931041
4.0
2.0
0.0
2.00
4.00
6.00
8.00
10.00
12.00
14.00
16.00
18.00
20.00
22.00
24.00
Time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7.498
7.479
7.459
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7.371
7.352
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7.329
7.319
7.309
7.302
7.284
6.990
6.984
6.967
6.961
6.945
6.938
6.810
1.576
H
N
F
3f
10
9
8
7
6
5
4
3
2
1
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ppm
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2.26
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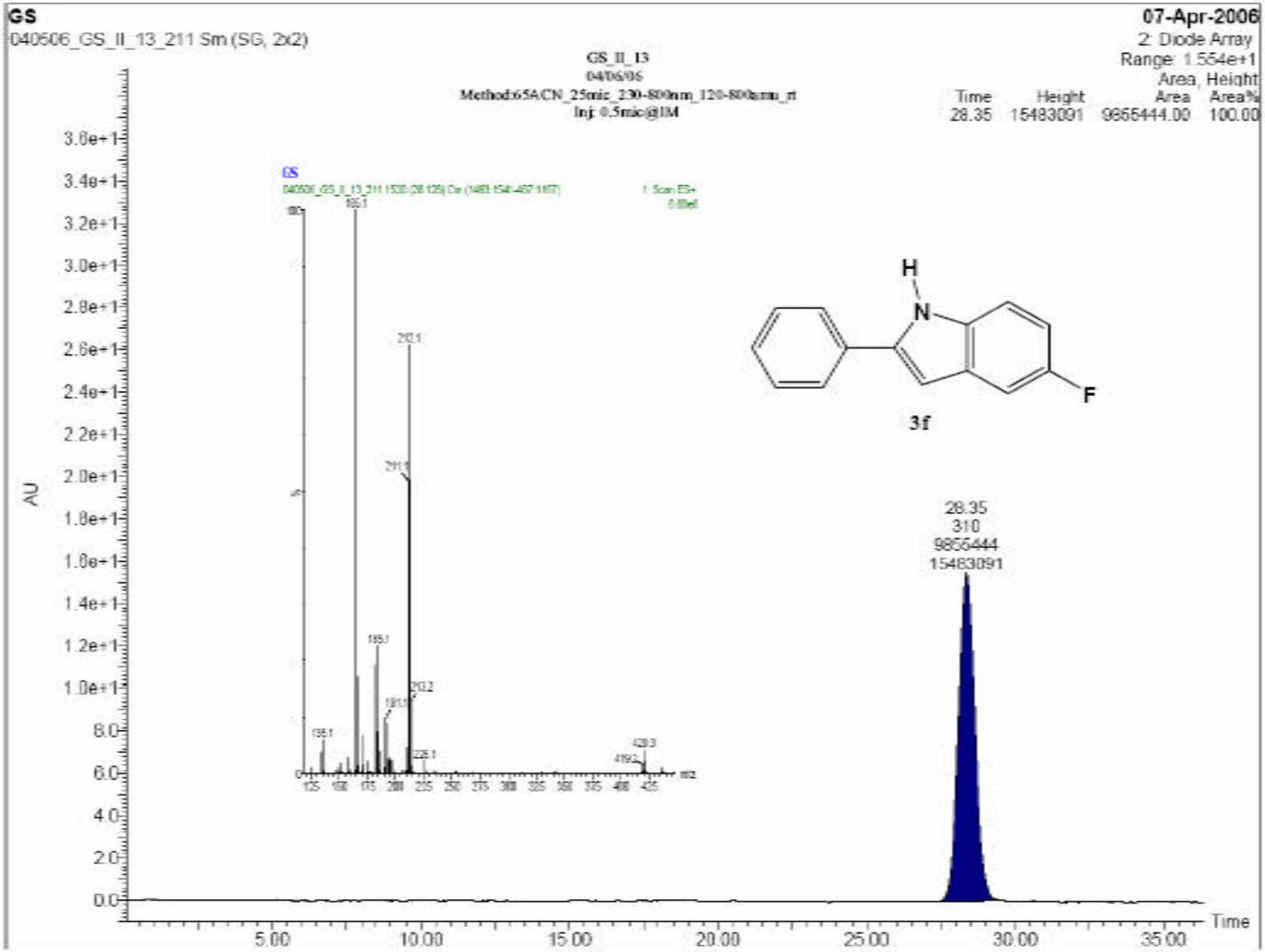
2.92
1.06
1.05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07-Apr-2006
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Range: 1.554e+1
GS_II_13
04/06/06
Method:65ACN_25mic_230-800nm_120-800amu_rt
Inj: 0.5mic@IM
Area, Height
Time Height Area Area%
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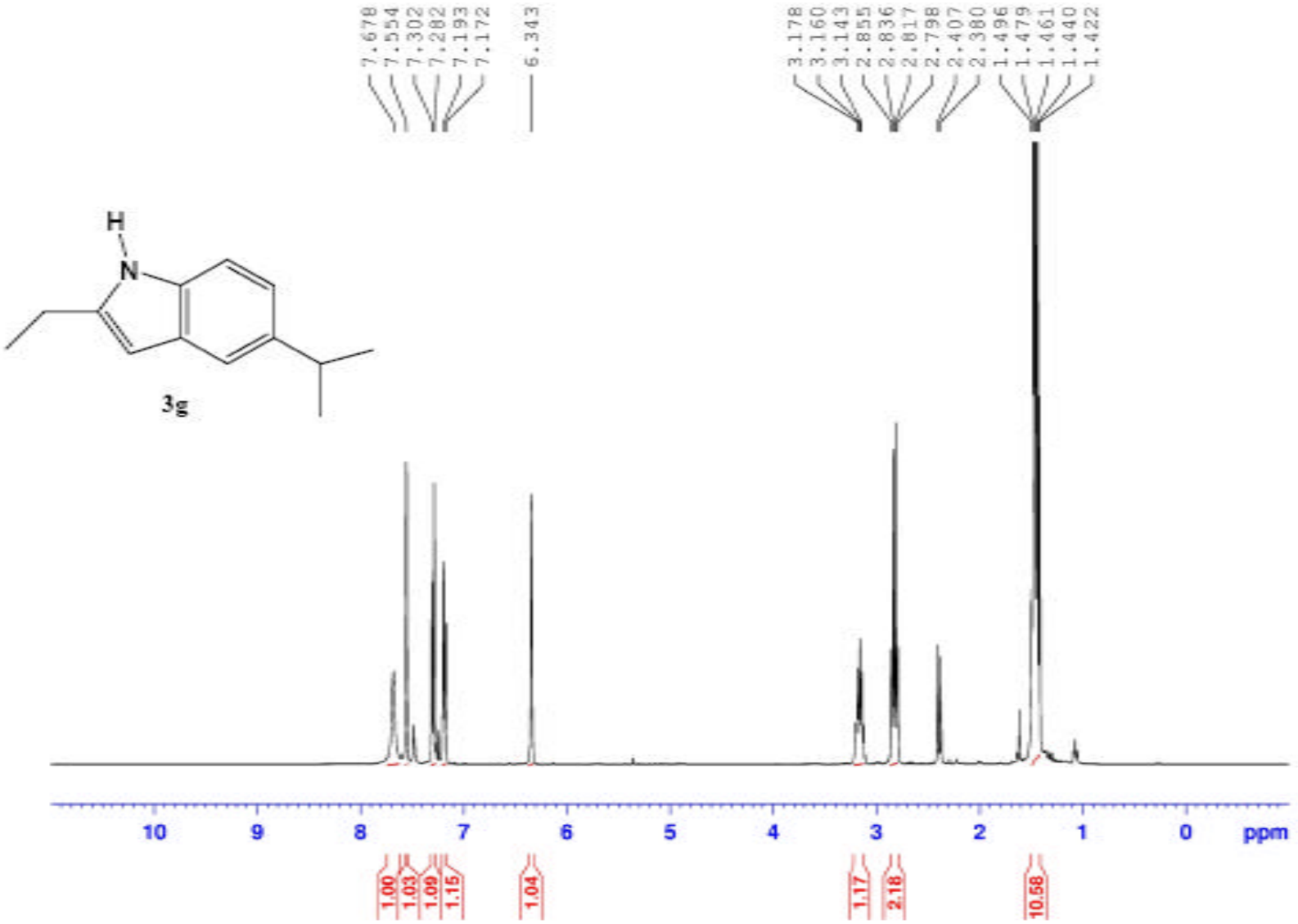
AU
Time
5.00
10.00
15.00
20.00
25.00
30.00
35.00
H
N
F
3f
28.35
310
9855444
15483091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7.302
7.282
7.193
7.172
6.343
3.178
3.160
3.143
2.855
2.836
2.817
2.798
2.407
2.380
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1.479
1.461
1.440
1.422
H
N
3g
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
1.03
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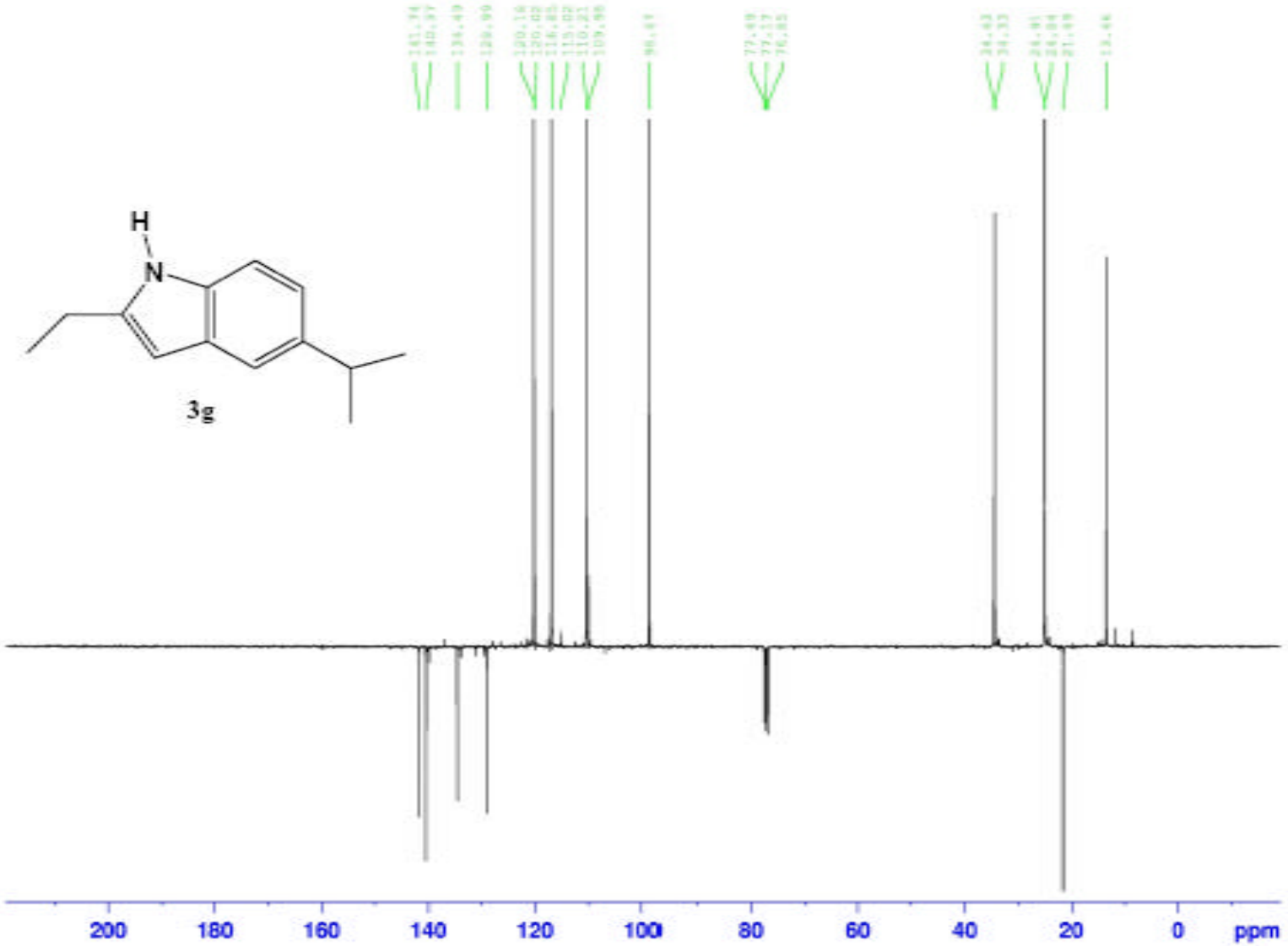
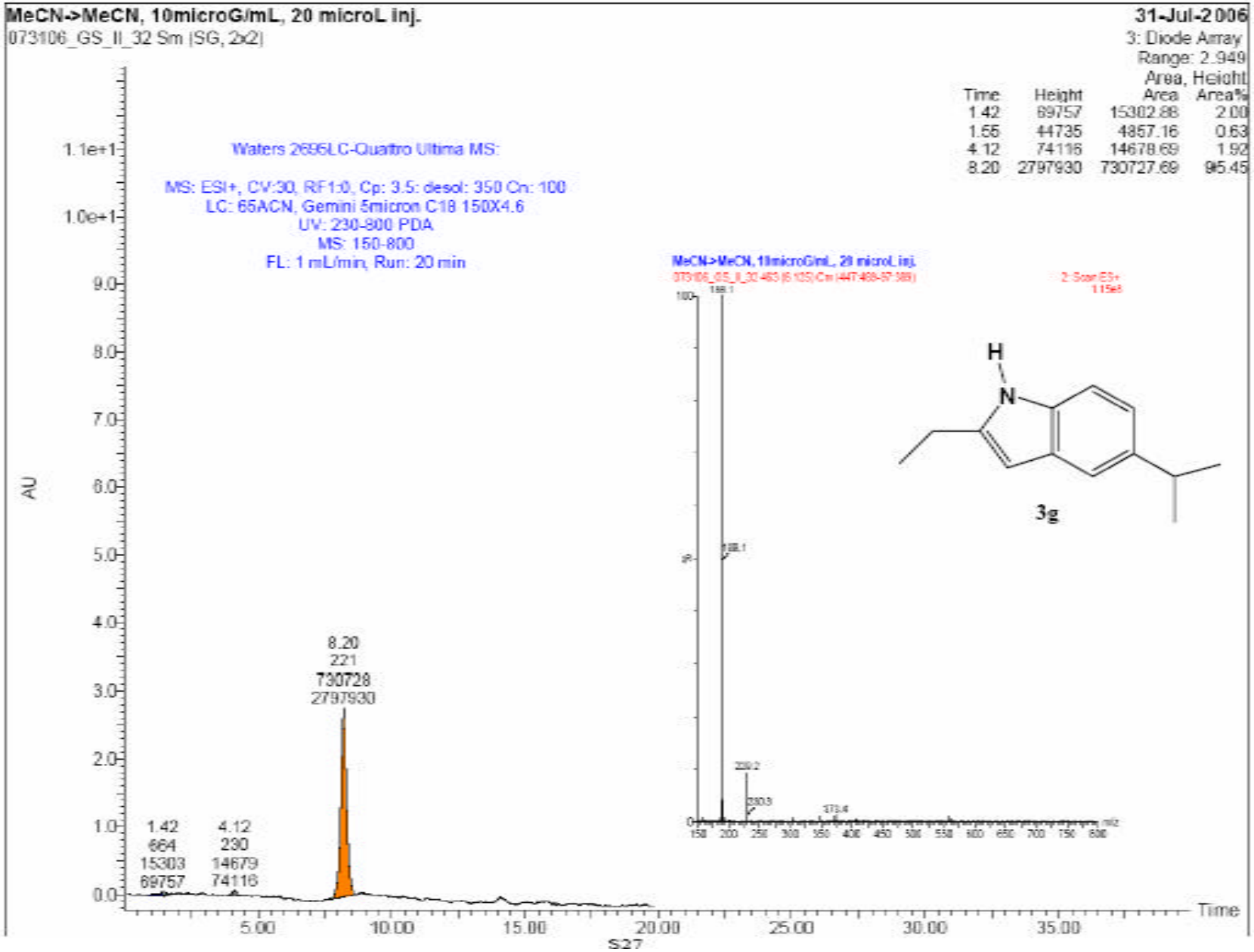
1.15
1.04
1.17
2.18
10.58


H
N
3g
200
180
160
140
120
100
80
60
40
20
0
ppm


MeCN->MeCN, 10microG/mL, 20 microL inj.
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31-Jul-2006
3: Diode Array
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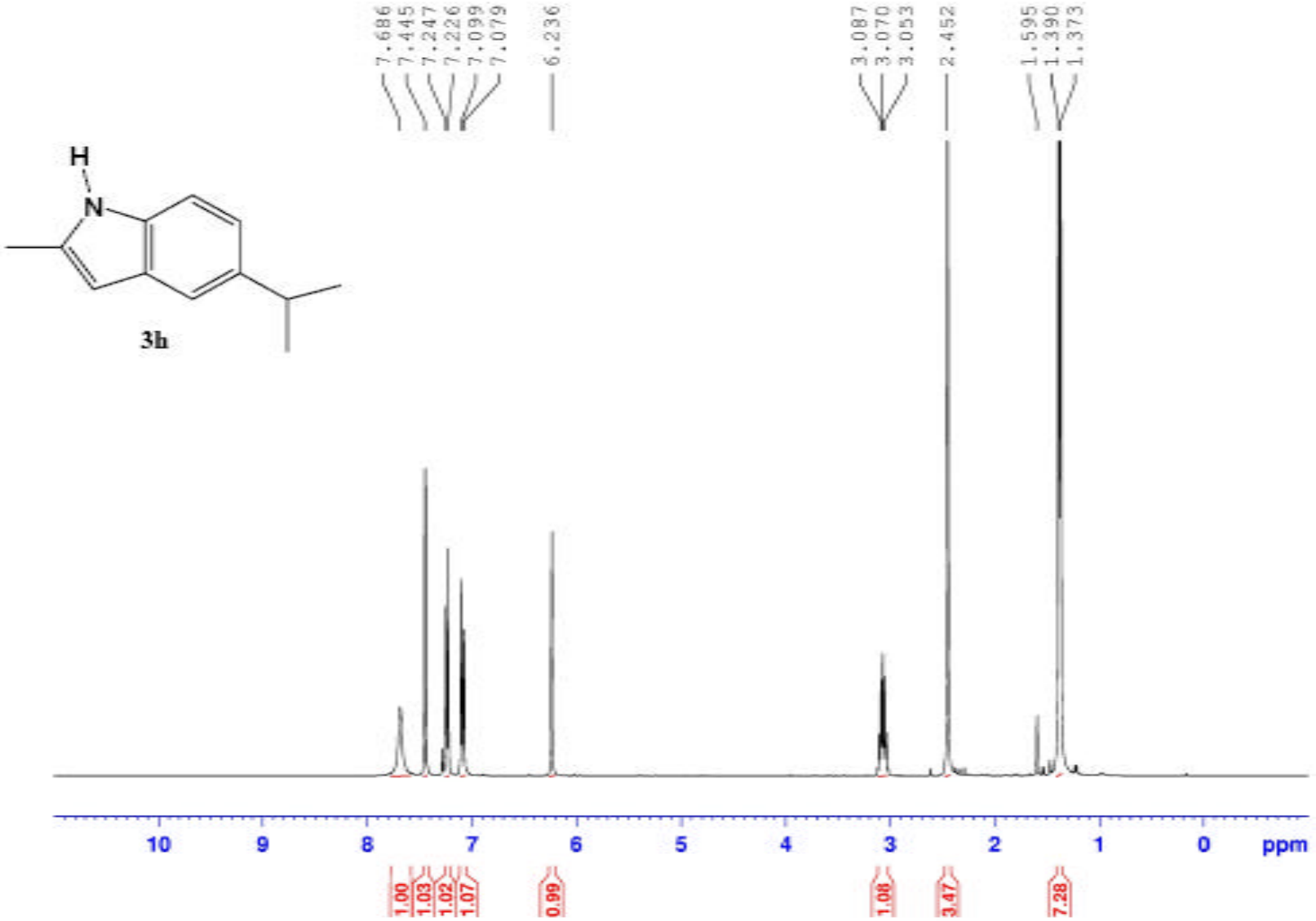
Area, Height
Time Height Area Area%
1.42 69757 15302.88 2.00
1.55 44735 4857.16 0.63
4.12 74116 14678.69 1.92
8.20 2797930 730727.69 95.45
Waters 2695LC-Quattro Ultima MS:
MS: ESI+, CV:30, RF1:0, Cp: 3.5: desol: 350 Cn: 100
LC: 65ACN, Gemini 5micron C18 150X4.6
UV: 230-800 PDA
MS: 150-800
FL: 1 mL/min, Run: 20 min
MeCN->MeCN, 10microG/mL, 20 microL inj.
2: Scan ES+
H
N
3g
AU
Time
1.42
664
15303
69757
4.12
230
14679
74116
8.20
221
730728
2797930
S27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7.099
7.079
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3.087
3.070
3.053
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1.595
1.390
1.373
H
N
3h
10
9
8
7
6
5
4
3
2
1
0
ppm
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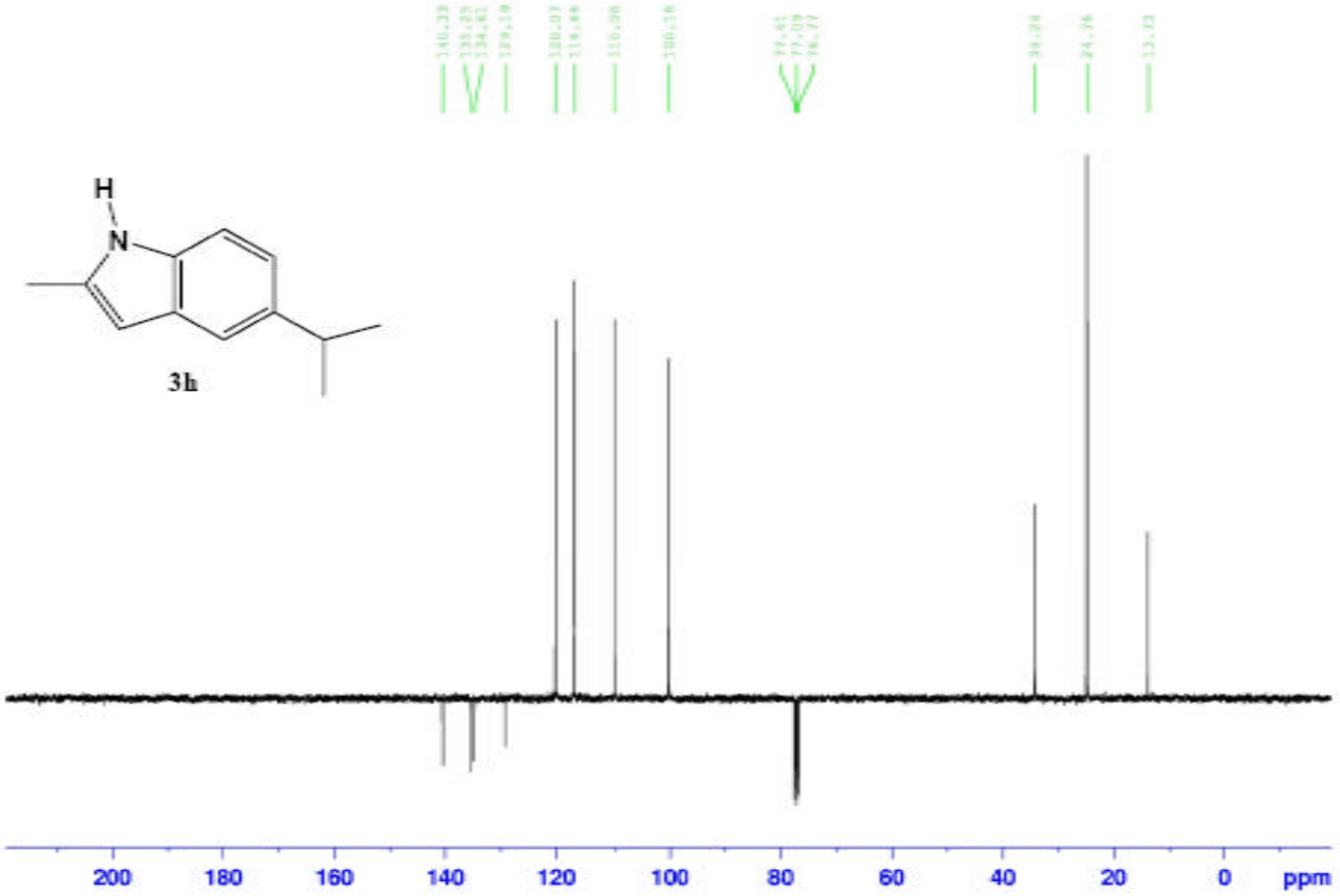
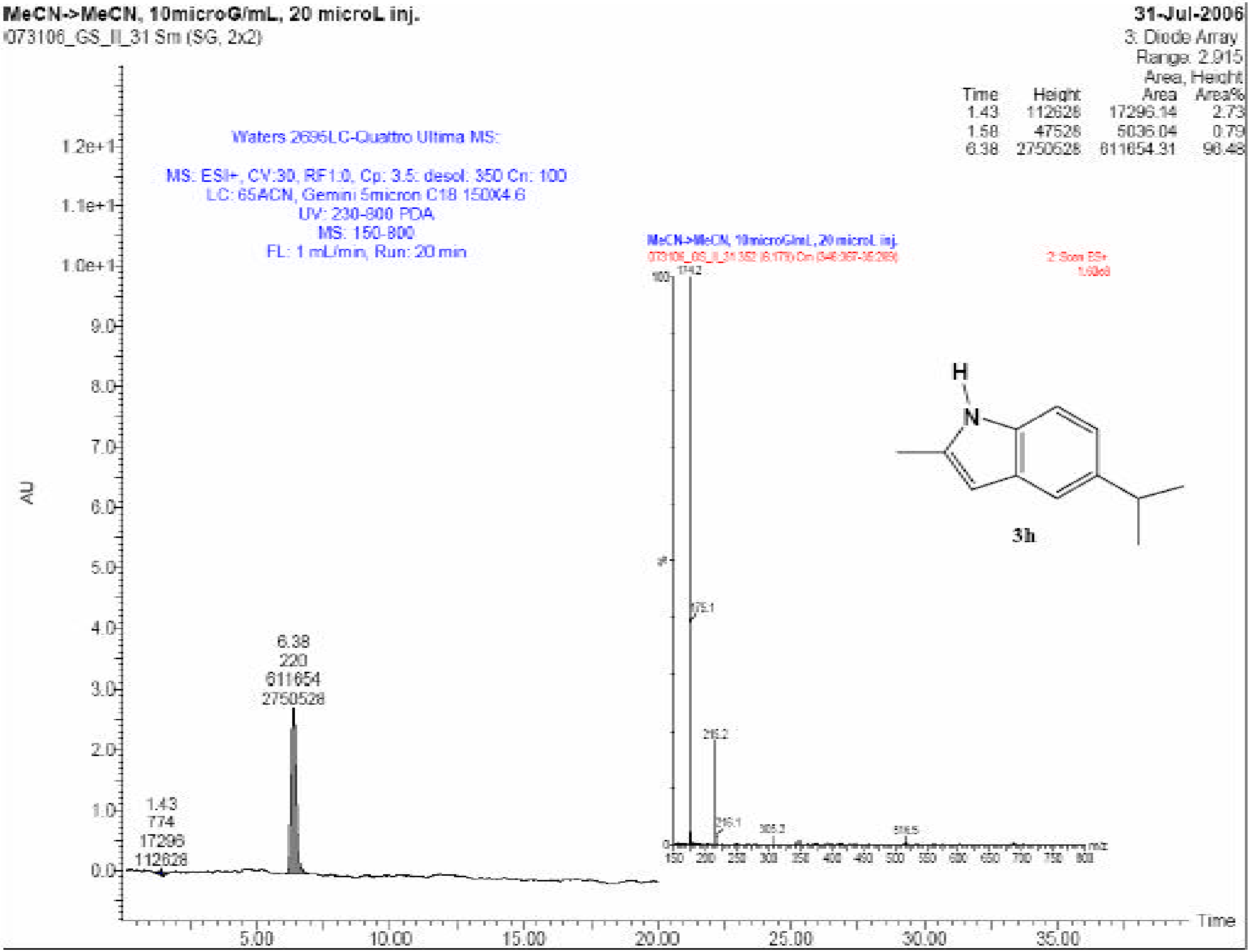
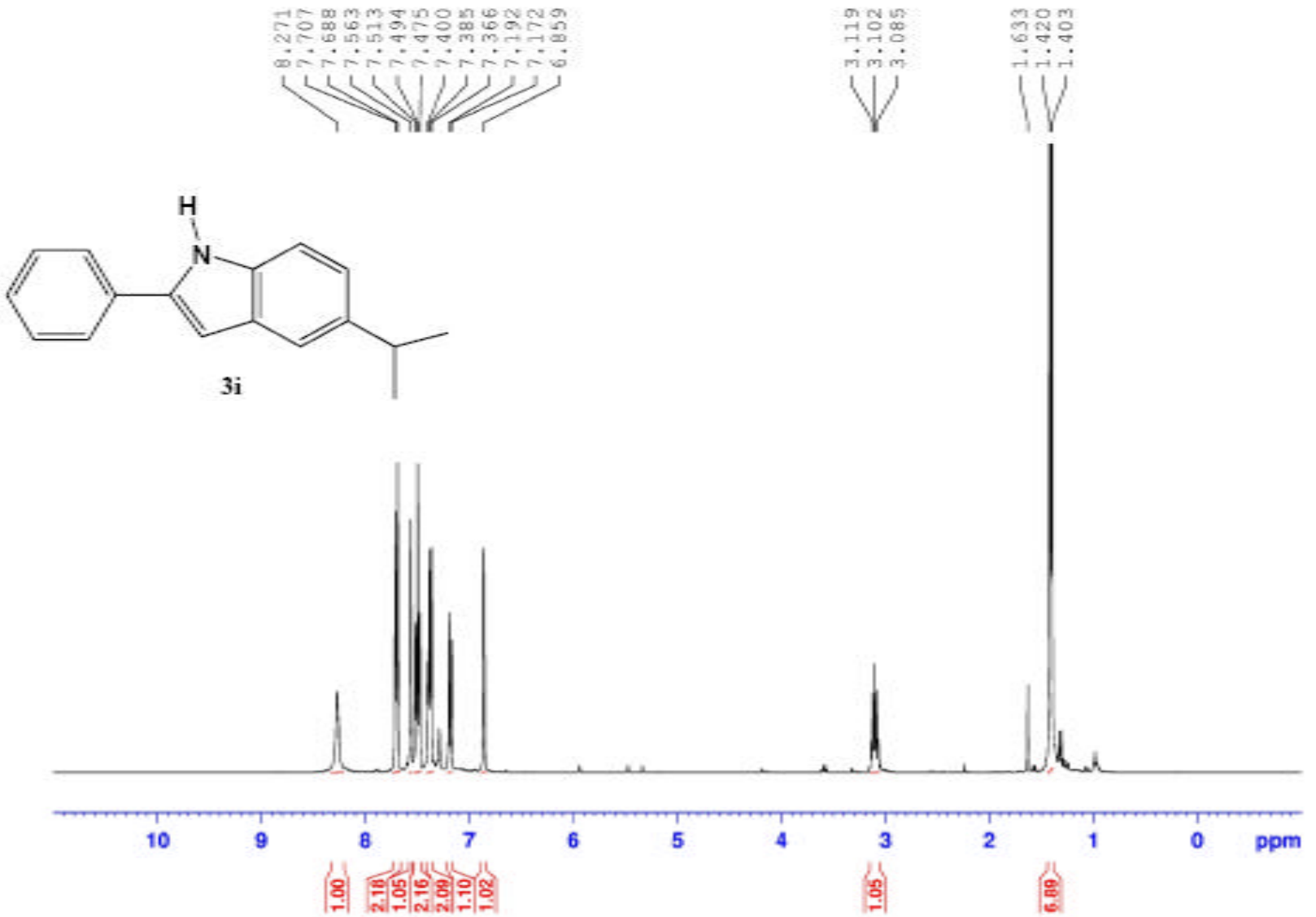
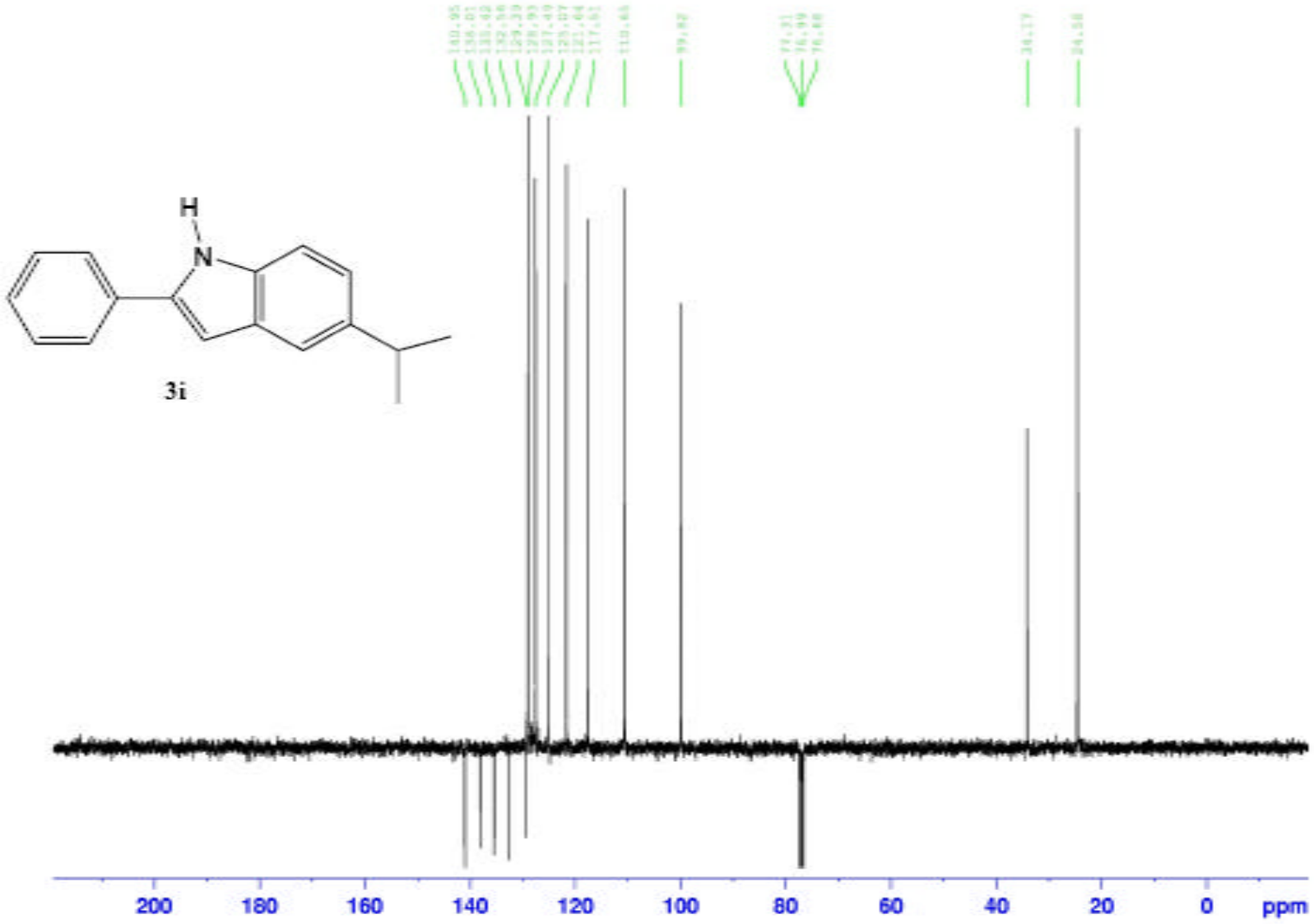
1.03
1.02
1.07
0.99
1.06
3.47
7.28

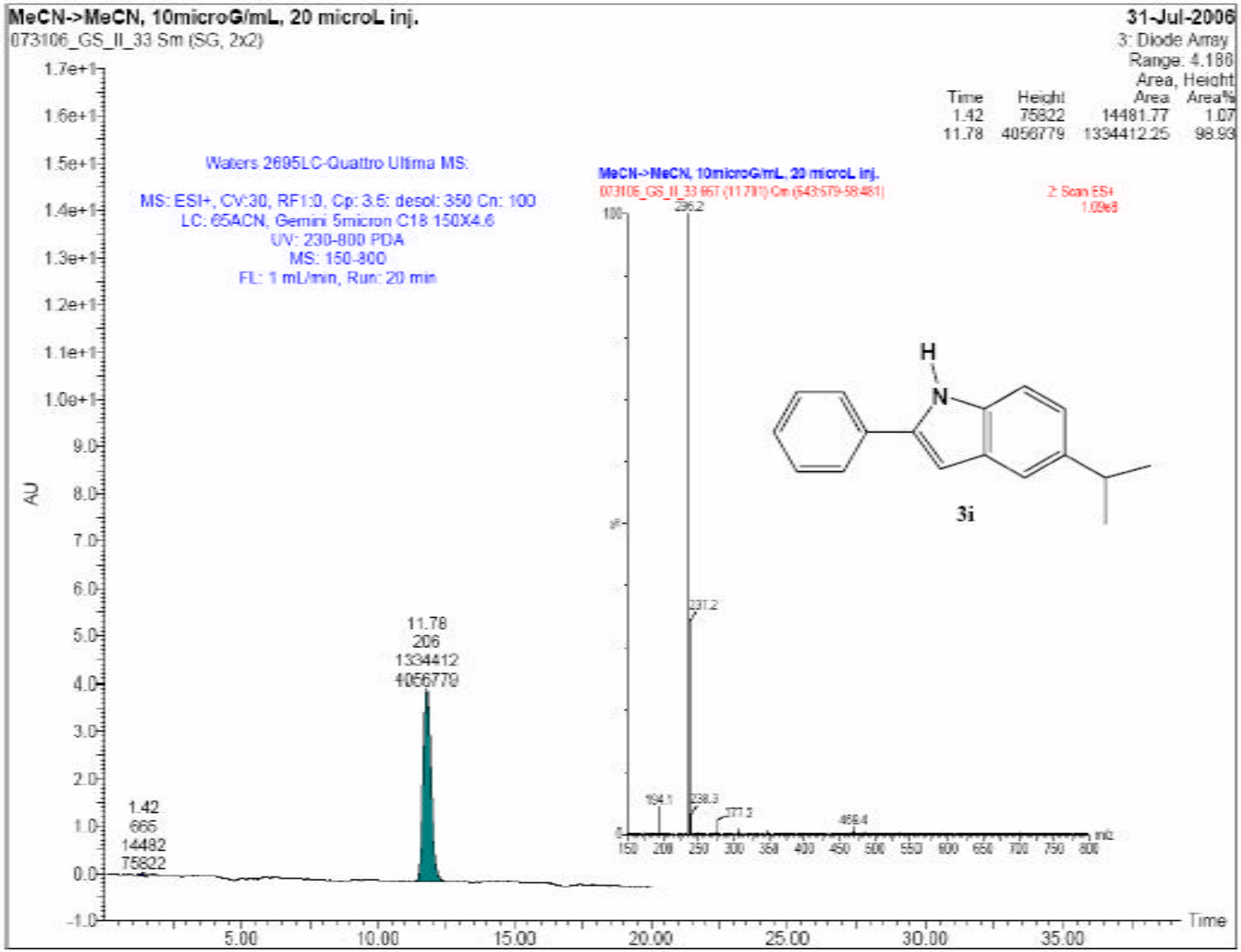

H
N
3h
200
180
160
140
120
100
80
60
40
20
0
ppm

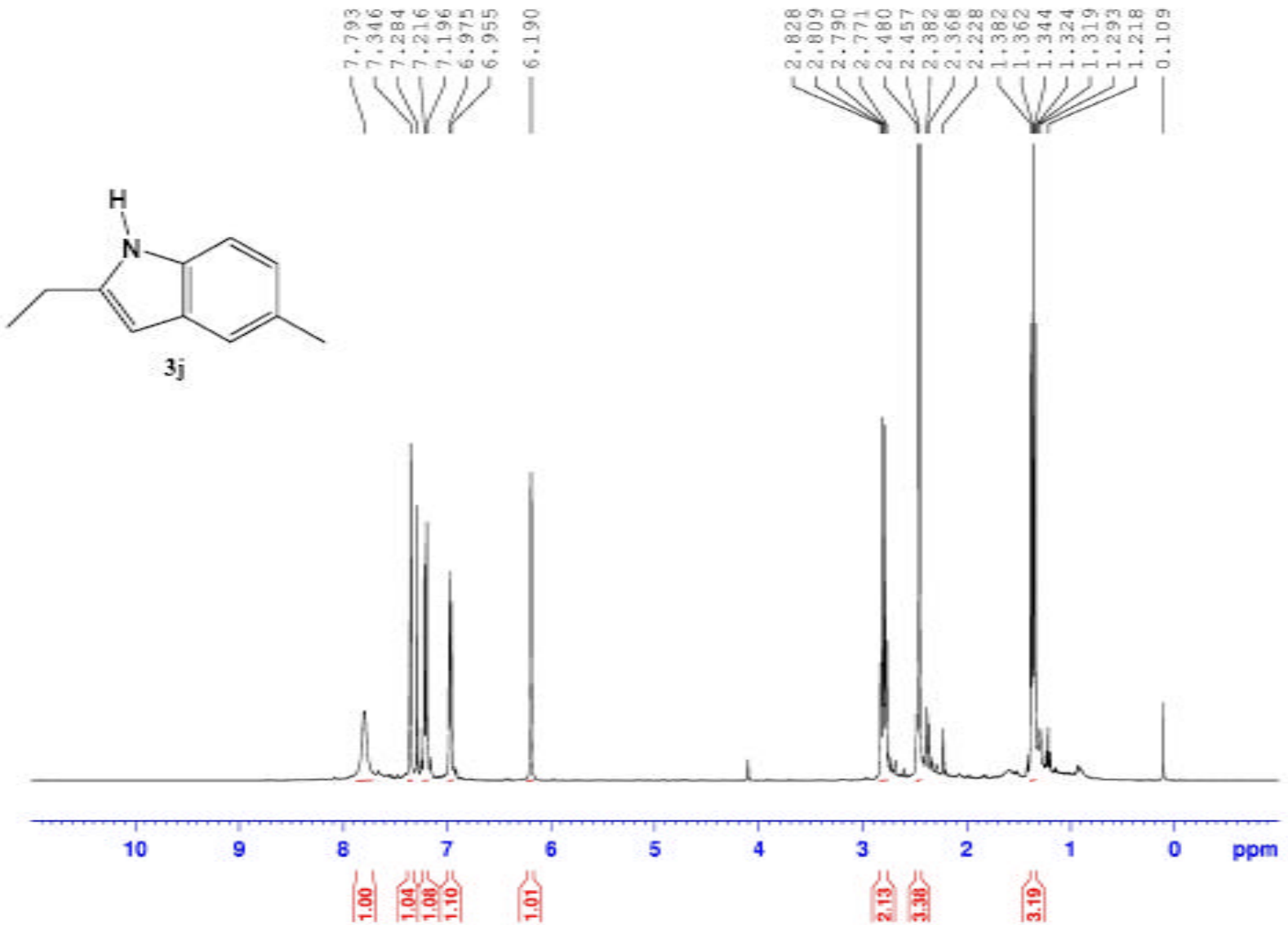


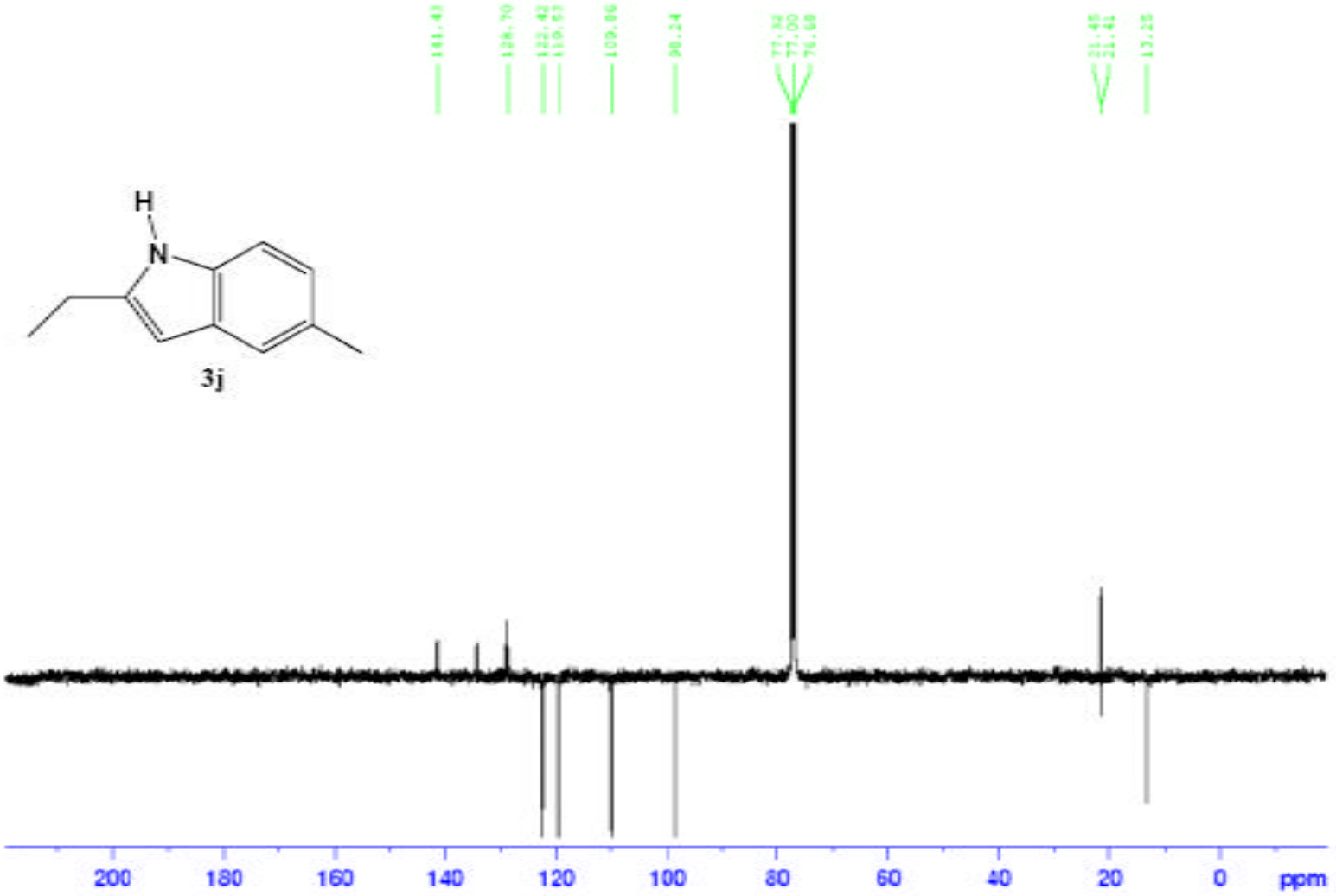
MeCN->MeCN, 10microG/mL, 20 microL inj.
073106_GS_II_31 Sm (SG, 2x2)
31-Jul-2006
3: Diode Array
Range: 2.915
Area, Height
Time Height Area Area%
1.43 112628 17296.14 2.73
1.58 47528 5036.04 0.79
6.38 2750528 611654.31 96.48
Waters 2695LC-Quattro Ultima MS:
MS: ESI+, CV:30, RF1.0, Cp: 3.5: desol: 350 Cn: 100
LC: 65ACN, Gemini 5micron C18 150X4.6
UV: 230-800 PDA
MS: 150-800
FL: 1 mL/min, Run: 20 min
6.38
220
611654
2750528
1.43
774
17296
112628
AU
Time
3h

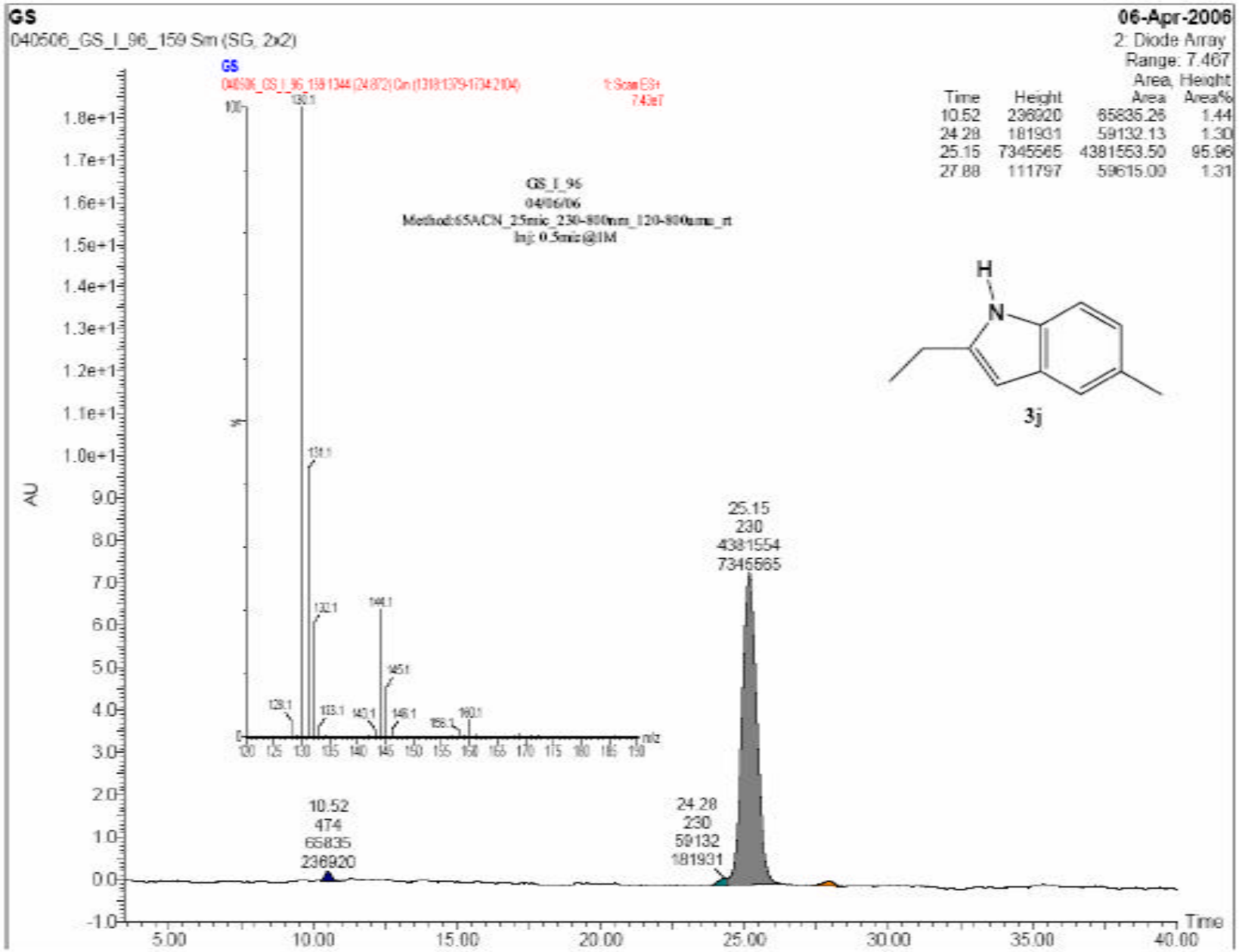

8.271
7.707
7.688
7.563
7.513
7.494
7.475
7.400
7.385
7.366
7.192
7.172
6.859
3.119
3.102
3.085
1.633
1.420
1.403
H
N
3i
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
2.18
1.05
2.16
2.09
1.10
1.02
1.05
6.09

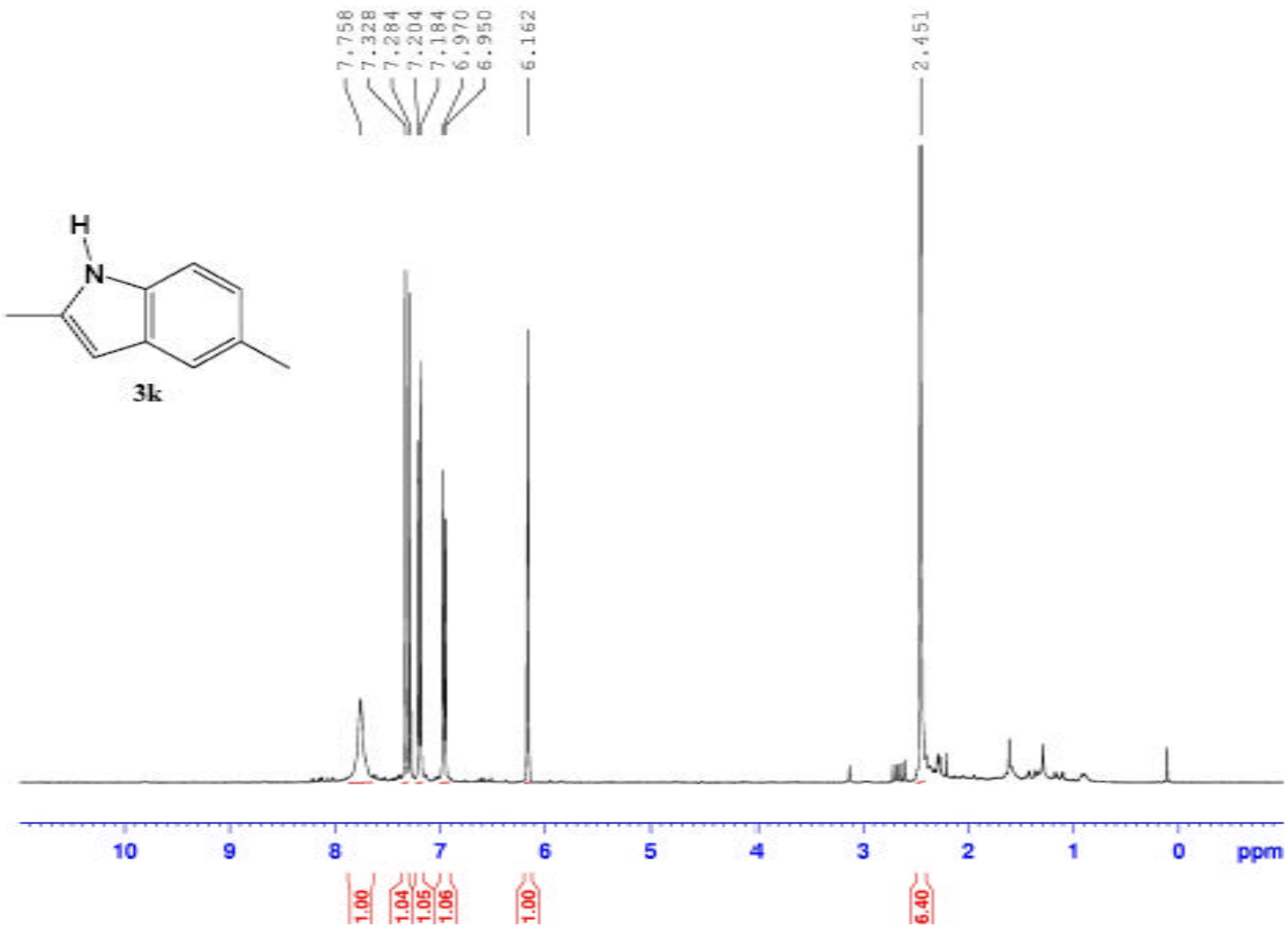

H
N
3i
200
180
160
140
120
100
80
60
40
20
0
ppm

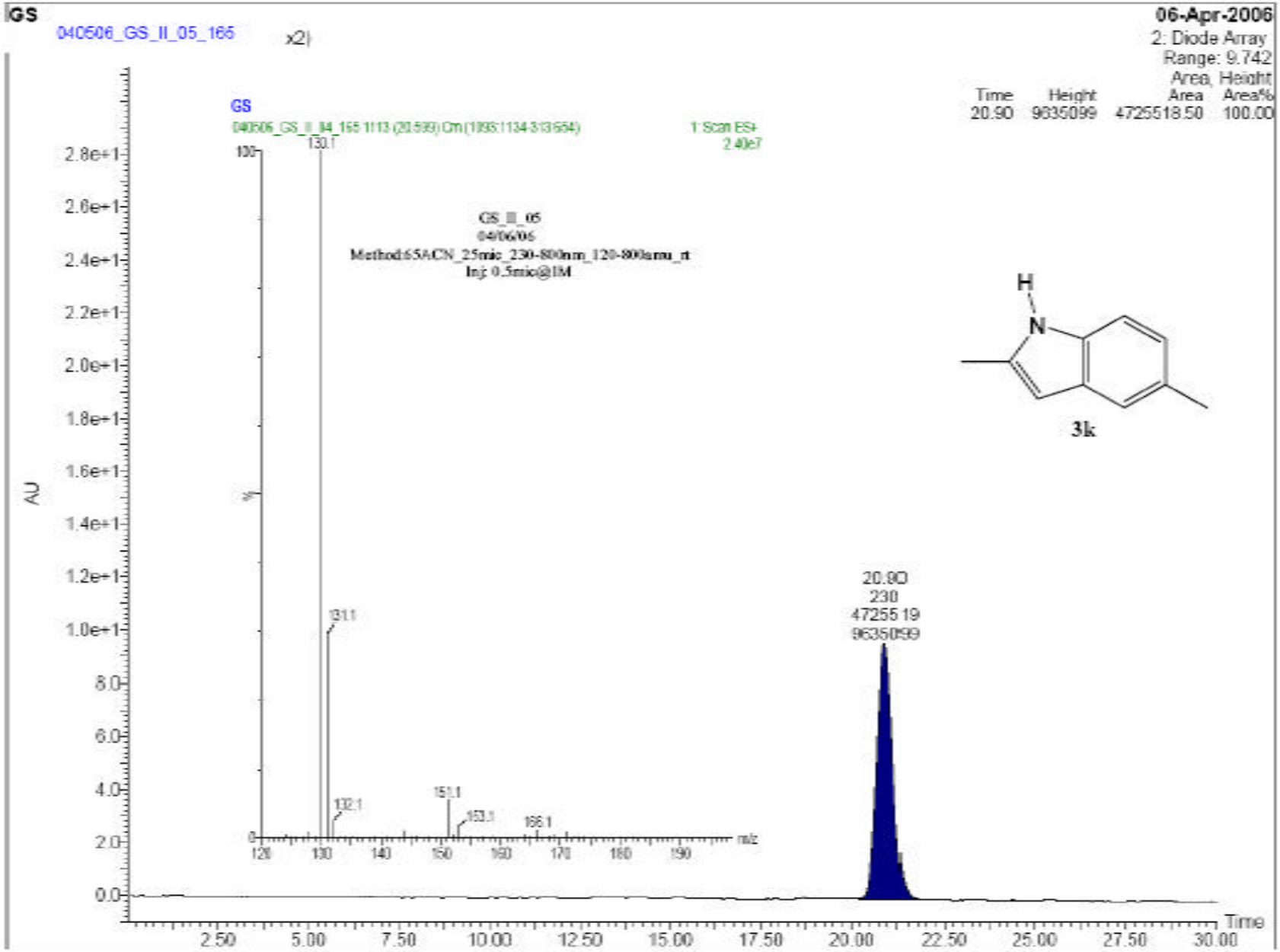

MeCN->MeCN, 10microG/mL, 20 microL inj.
073106_GS_II_33 Sm (SG, 2x2)
31-Jul-2006
3: Diode Array
Range: 4.186
Area, Height
Time Height Area Area%
1.42 75822 14481.77 1.07
11.78 4056779 1334412.25 98.93
Waters 2695LC-Quattro Ultima MS:
MS: ESI+, CV:30, RF1:0, Cp: 3.5: desol: 350 Cn: 100
LC: 65ACN, Gemini 5micron C18 150X4.6
UV: 230-800 PDA
MS: 150-800
FL: 1 mL/min, Run: 20 min
MeCN->MeCN, 10microG/mL, 20 microL inj.
2: Scan ES+
1.09e8
296.2
231.2
194.1
238.3
377.3
456.4
m/z
H
N
3i
11.78
206
1334412
4056779
1.42
665
14482
75822
AU
Time
5.00
10.00
15.00
20.00
25.00
30.00
35.00

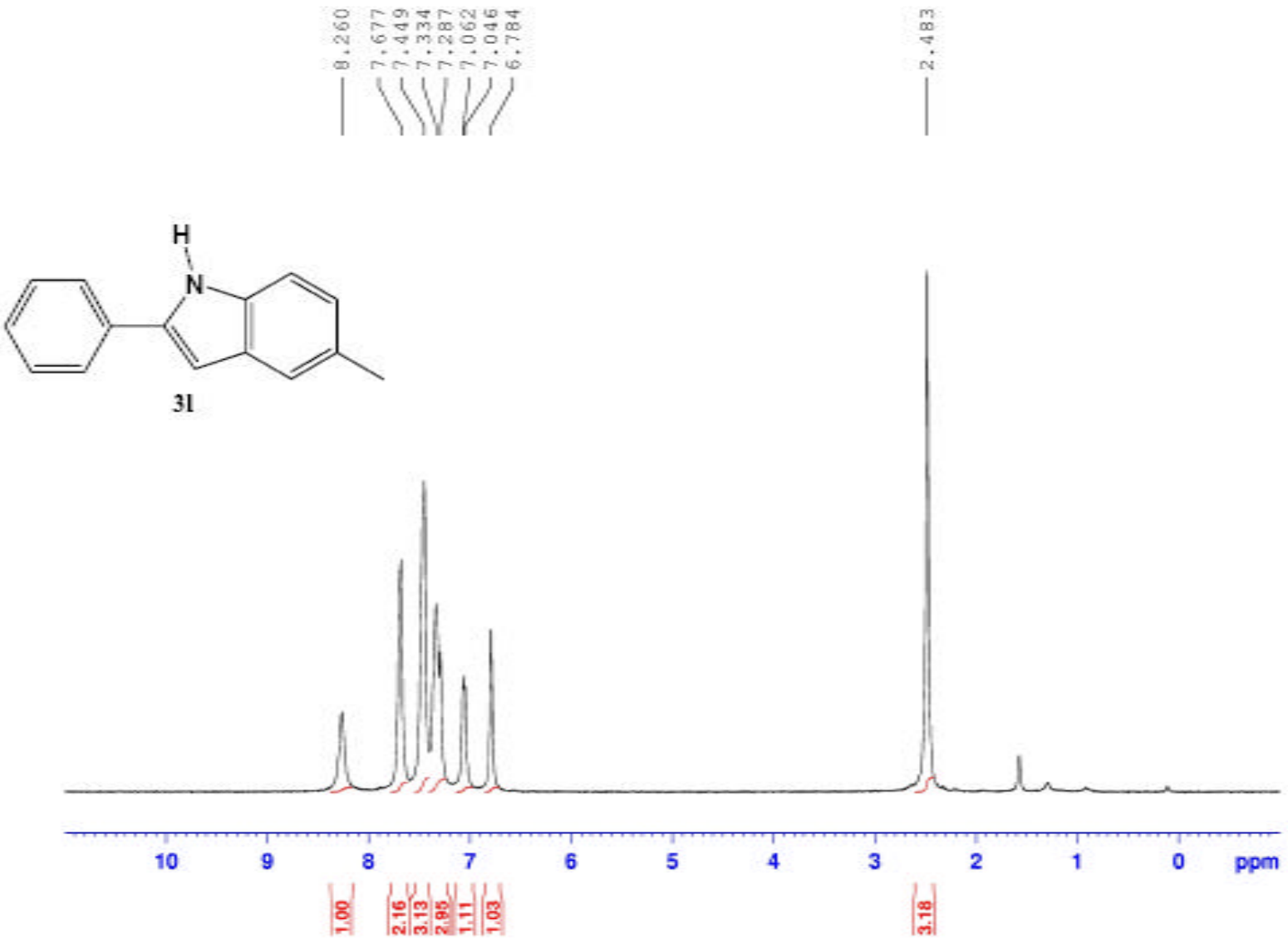

7.793
7.346
7.284
7.216
7.196
6.975
6.955
6.190
2.828
2.809
2.790
2.771
2.480
2.457
2.382
2.368
2.228
1.382
1.362
1.344
1.324
1.319
1.293
1.218
0.109
H
N
3j
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
1.04
1.08
1.10
1.01
2.13
3.36
3.19

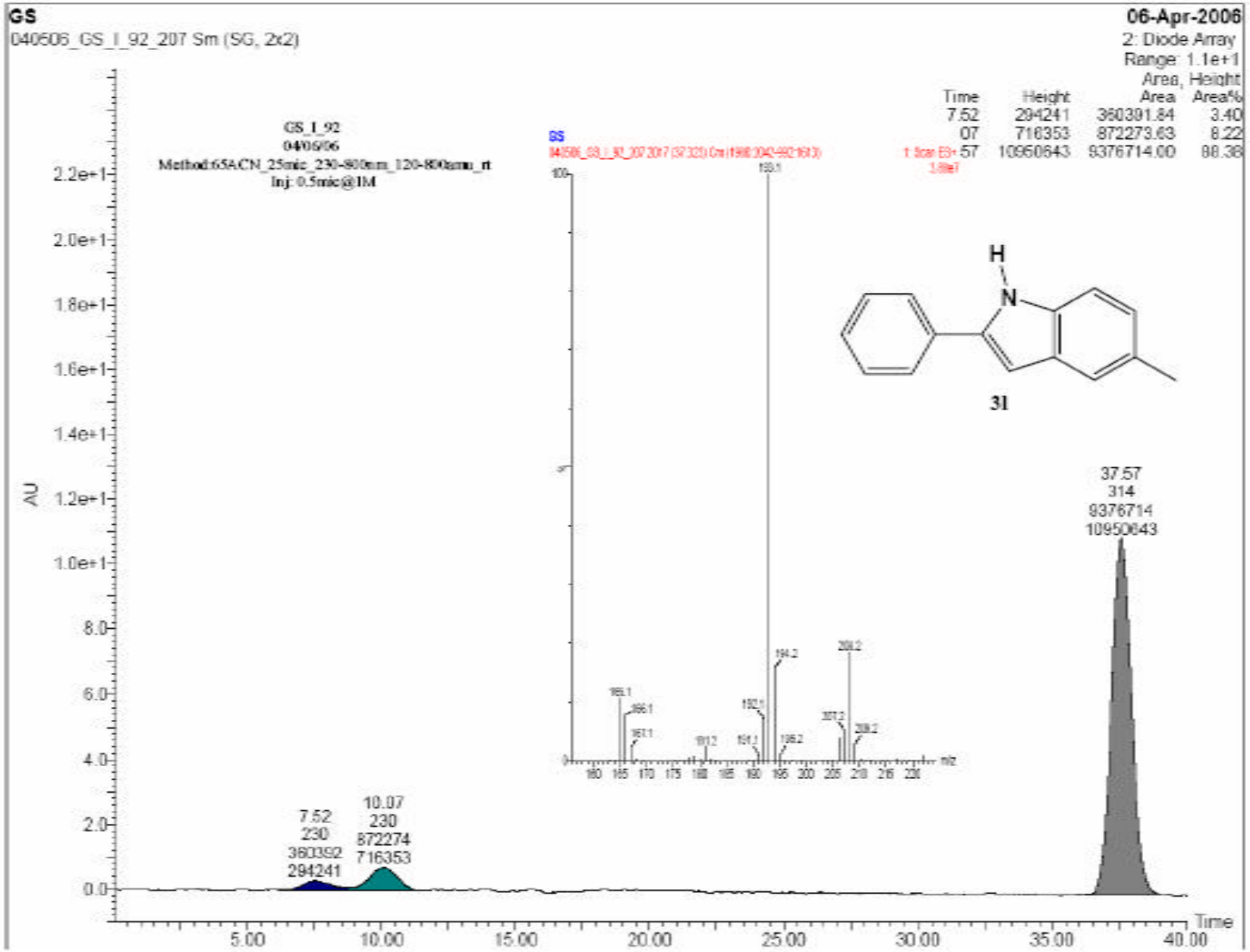

H
N
3j
141.43
128.70
122.42
119.67
109.86
98.24
77.32
77.00
76.68
21.45
21.41
13.28
200
180
160
140
120
100
80
60
40
20
0
ppm

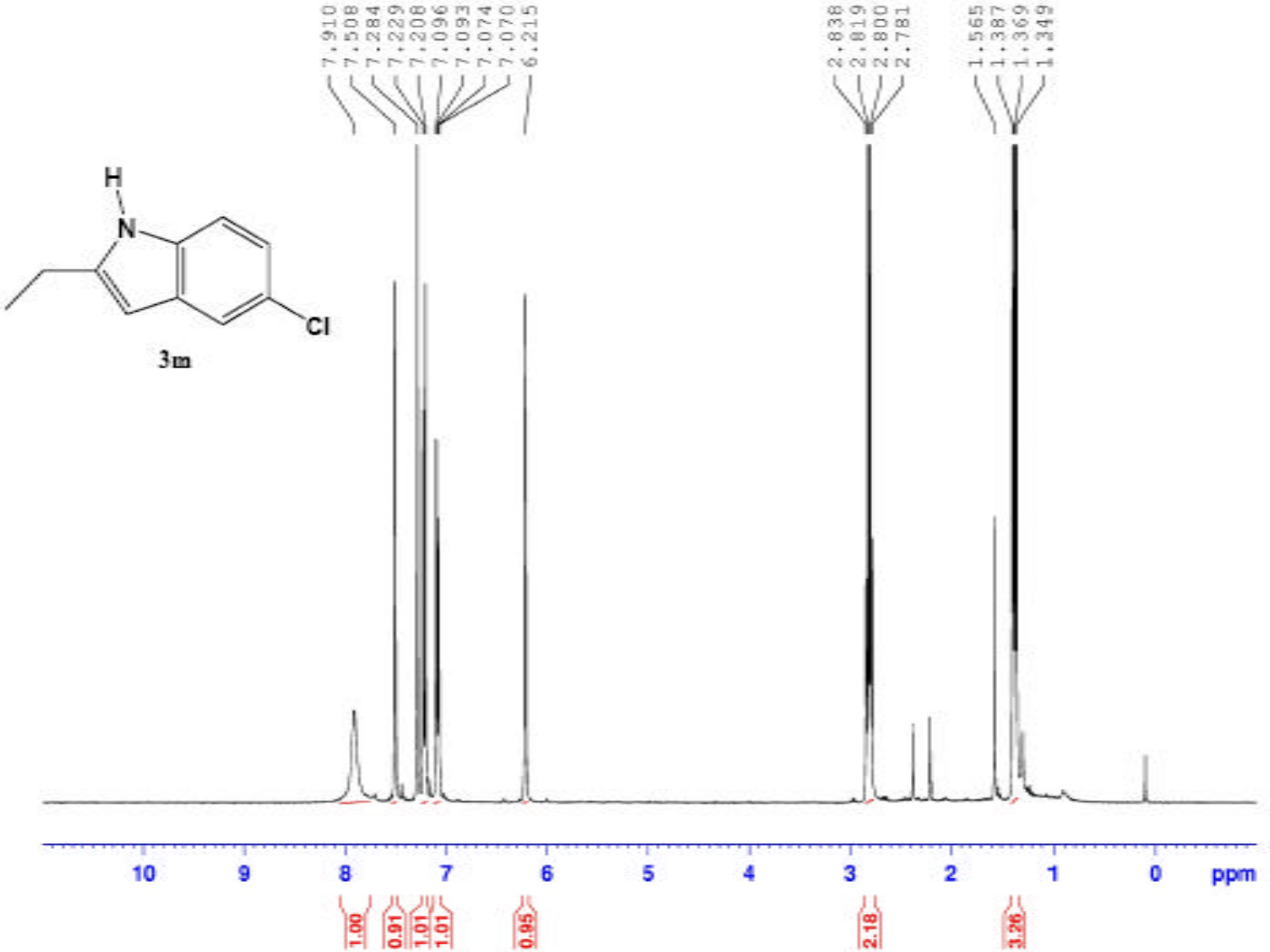

GS
040606_GS_I_96_159 Sm (SG, 2x2)
06-Apr-2006
2: Diode Array
Range: 7.467
Area, Height
Time Height Area Area%
10.52 236920 65835.26 1.44
24.28 181931 59132.13 1.30
25.15 7345565 4381553.50 95.96
27.88 111797 59615.00 1.31
GS_I_96
040606
Method:65ACN_25min_230-800nm_120-800nm_rt
Inj: 0.5mic@1M
3j
AU
Time
25.15
230
4381554
7346565
10.52
474
65835
236920
24.28
230
59132
181931

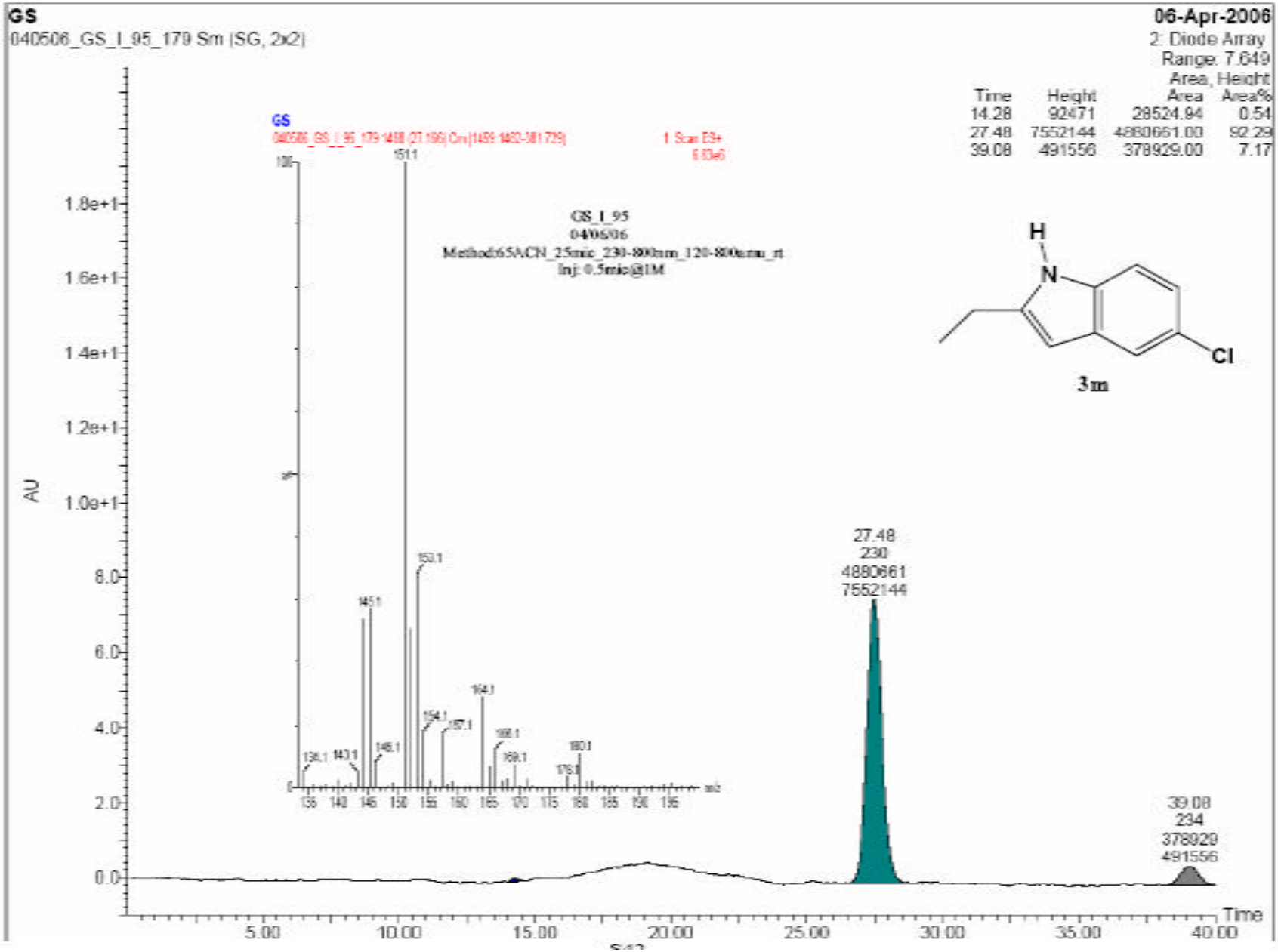

7.758
7.328
7.284
7.204
7.184
6.970
6.950
6.162
2.451
H
N
3k
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
1.04
1.05
1.06
1.00
6.40

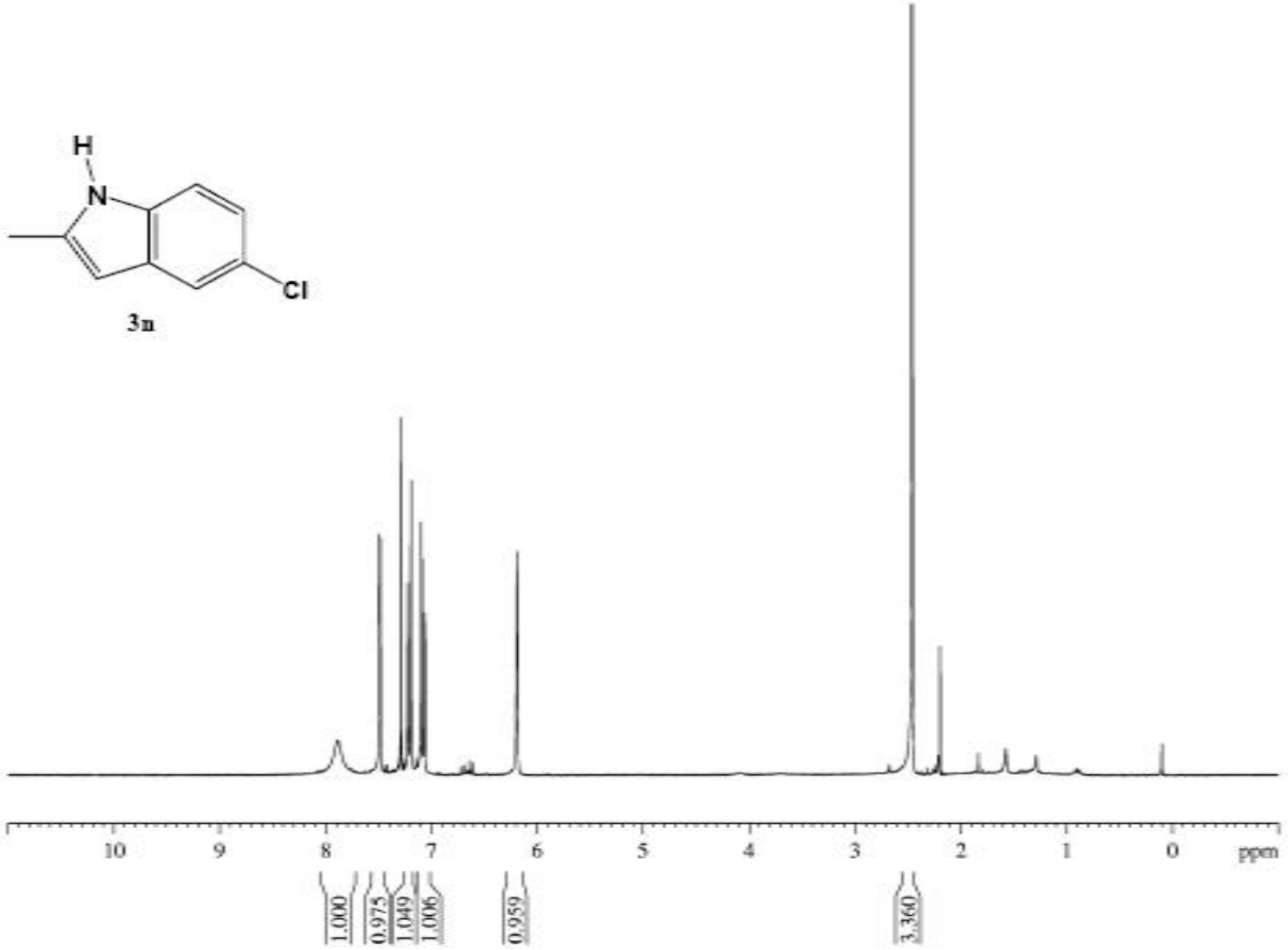

GS
040606_GS_II_05_165
x2
06-Apr-2006
2: Diode Array
Range: 9.742
Area, Height
Time Height Area Area%
20.90 9635099 4725518.50 100.00
GS
040606_GS_II_04_165 1:113 (20.599) Cm (1093:1134:313:654)
1: Scan ES+
2.40e7
130.1
GS_II_05
040606
Method:65ACN_25mic_230-800nm_120-800amu_rt
Inj: 0.5mic@1M
H
N
3k
131.1
132.1
151.1
153.1
166.1
m/z
20.90
230
4725519
9635099
AU
Time

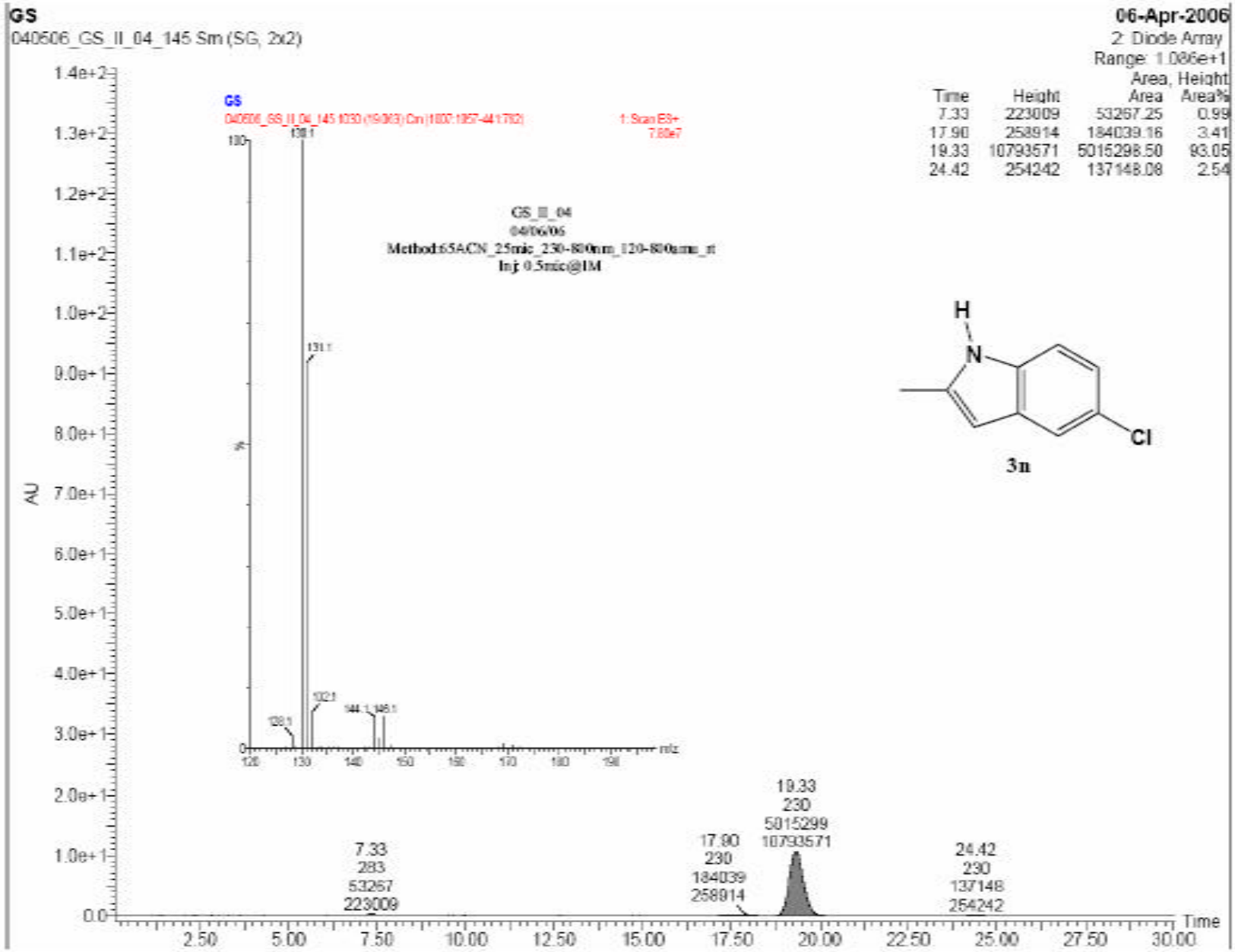

8.260
7.677
7.449
7.334
7.287
7.062
7.046
6.784
2.483
H
N
3l
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
2.16
3.13
2.95
1.11
1.03
3.18

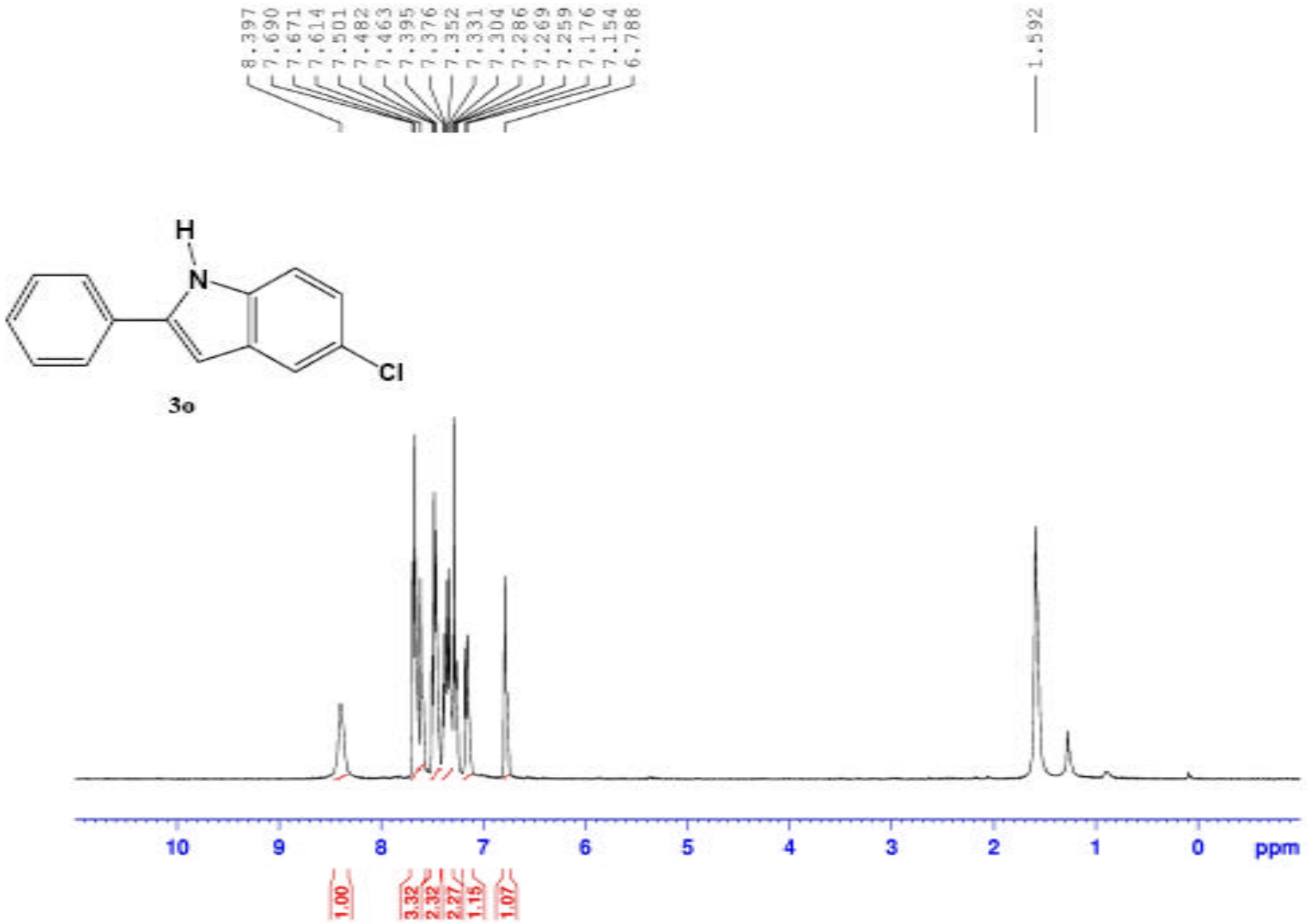

GS
040606_GS_I_92_207 Sm (SG, 2x2)
06-Apr-2006
2: Diode Array
Range: 1.1e+1
Area, Height
Time Height Area Area%
7.52 294241 360391.84 3.40
07 716353 872273.63 8.22
57 10950643 9376714.00 88.38
GS_I_92
040606
Method:65ACN_25min_230-800nm_120-800amu_rt
Inj: 0.5mic@1M
GS
H
N
31
37.57
314
9376714
10950643
7.52
230
360392
294241
10.07
230
872274
716353
AU
Time
5.00
10.00
15.00
20.00
25.00
30.00
35.00
40.00

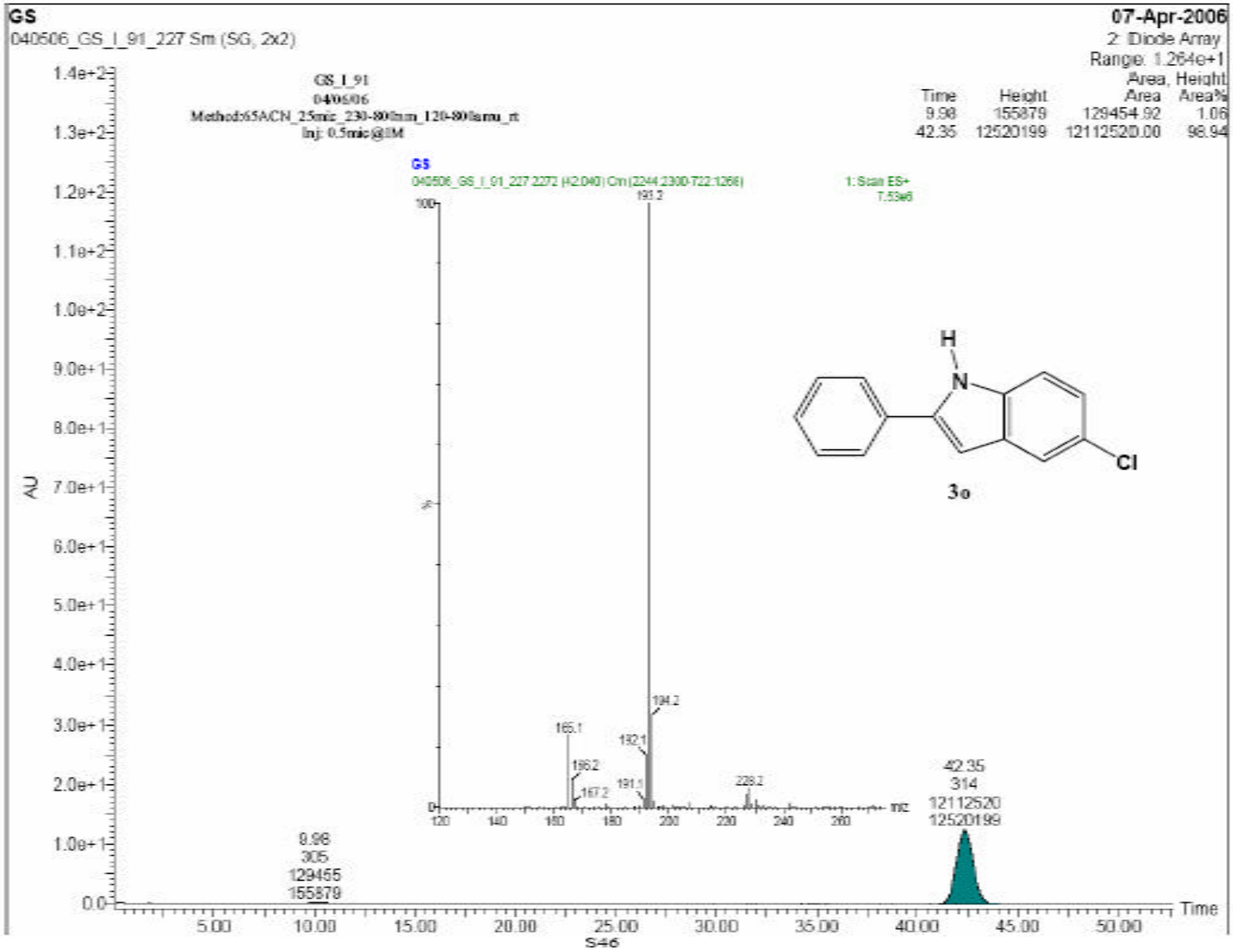

7.910
7.508
7.284
7.229
7.208
7.096
7.093
7.074
7.070
6.215
2.838
2.819
2.800
2.781
1.565
1.387
1.369
1.349
H
N
Cl
3m
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
0.91
1.01
1.01
0.95
2.18
3.26

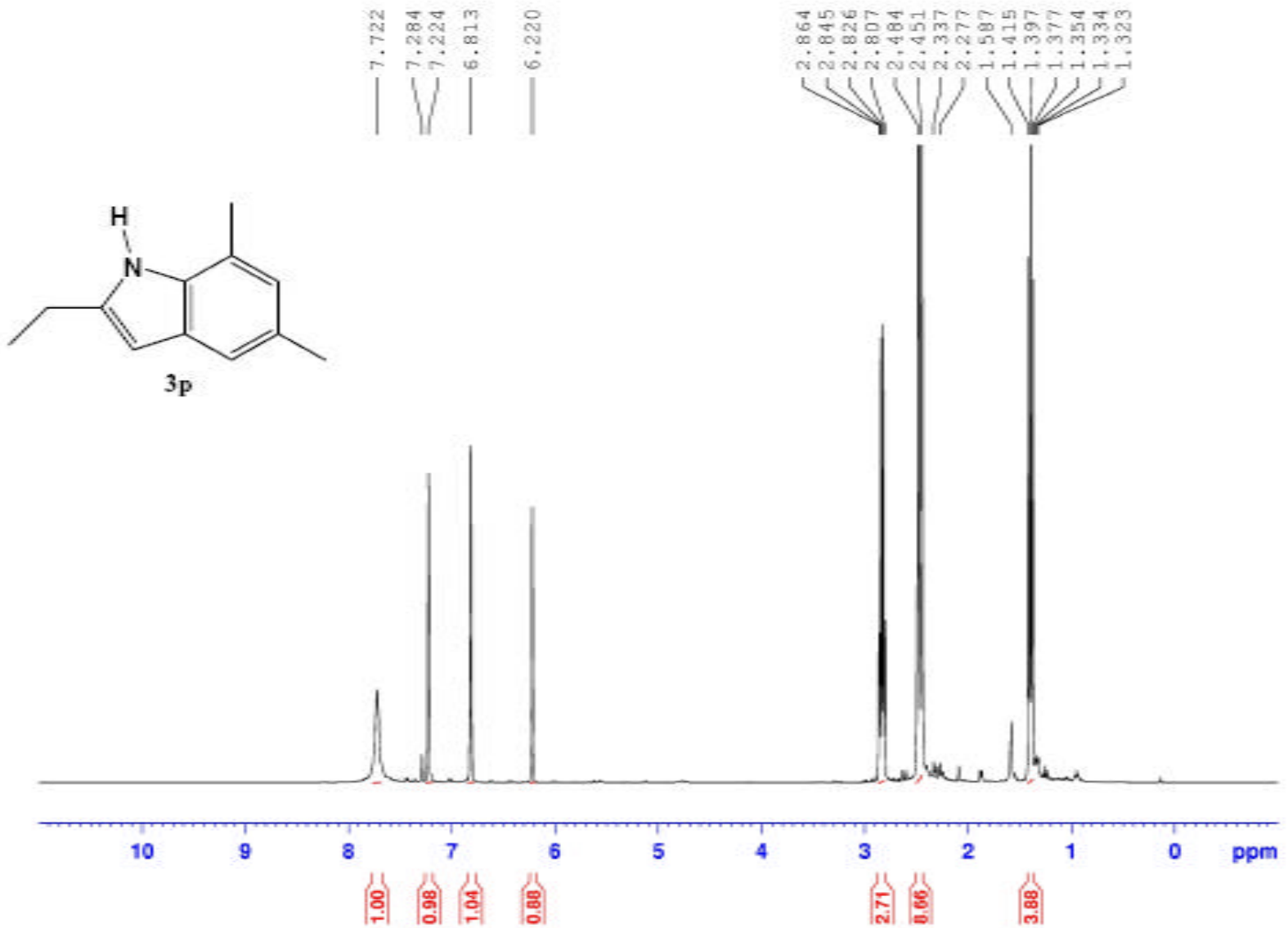

GS
040506_GS_I_95_179 Sm (SG, 2x2)
06-Apr-2006
2: Diode Array
Range: 7.649
Area, Height
Time Height Area Area%
14.28 92471 28524.94 0.54
27.48 7552144 4880661.00 92.29
39.08 491556 378929.00 7.17
GS
GS_I_95
040506
Method65ACN_25mic_230-800nm_120-800amu_rt
Inj: 0.5mic@1M
H
N
Cl
3m
27.48
230
4880661
7552144
39.08
234
378929
491556
AU
Time

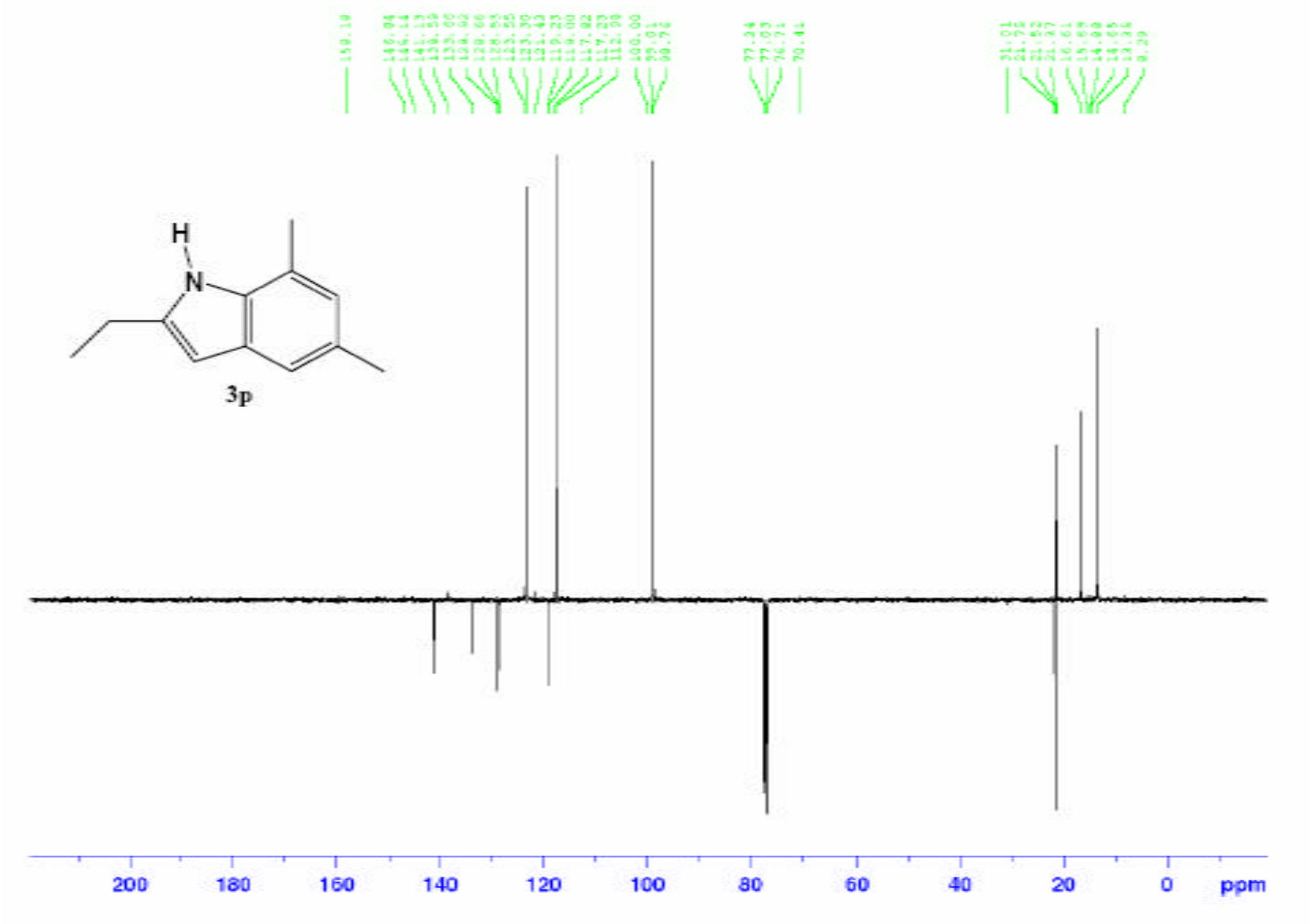

H
N
Cl
3n
10
9
8
7
6
5
4
3
2
1
0
ppm
1.000
0.975
1.049
1.006
0.959
3.360

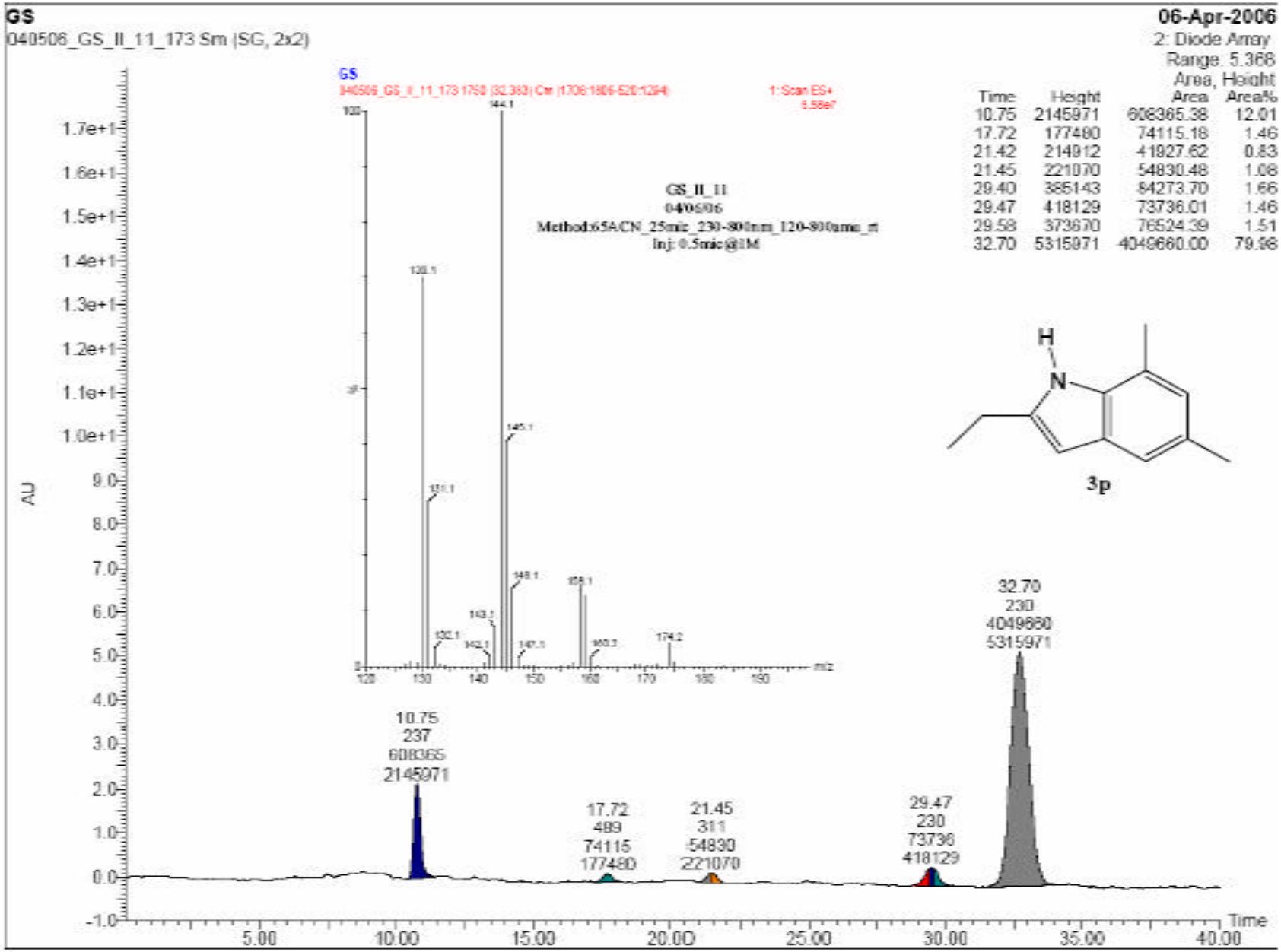

GS
040606_GS_II_04_145 Sm (SG, 2x2)
06-Apr-2006
2: Diode Array
Range: 1.086e+1
Area, Height
Time Height Area Area%
7.33 223009 53267.25 0.99
17.90 258914 184039.16 3.41
19.33 10793571 5015298.50 93.05
24.42 254242 137148.08 2.54
GS_II_04
04/06/06
Inj: 0.5mic@1M
H
N
Cl
3n
AU
Time
7.33
283
53267
223009
17.90
230
184039
258914
19.33
230
5015299
10793571
24.42
230
137148
254242

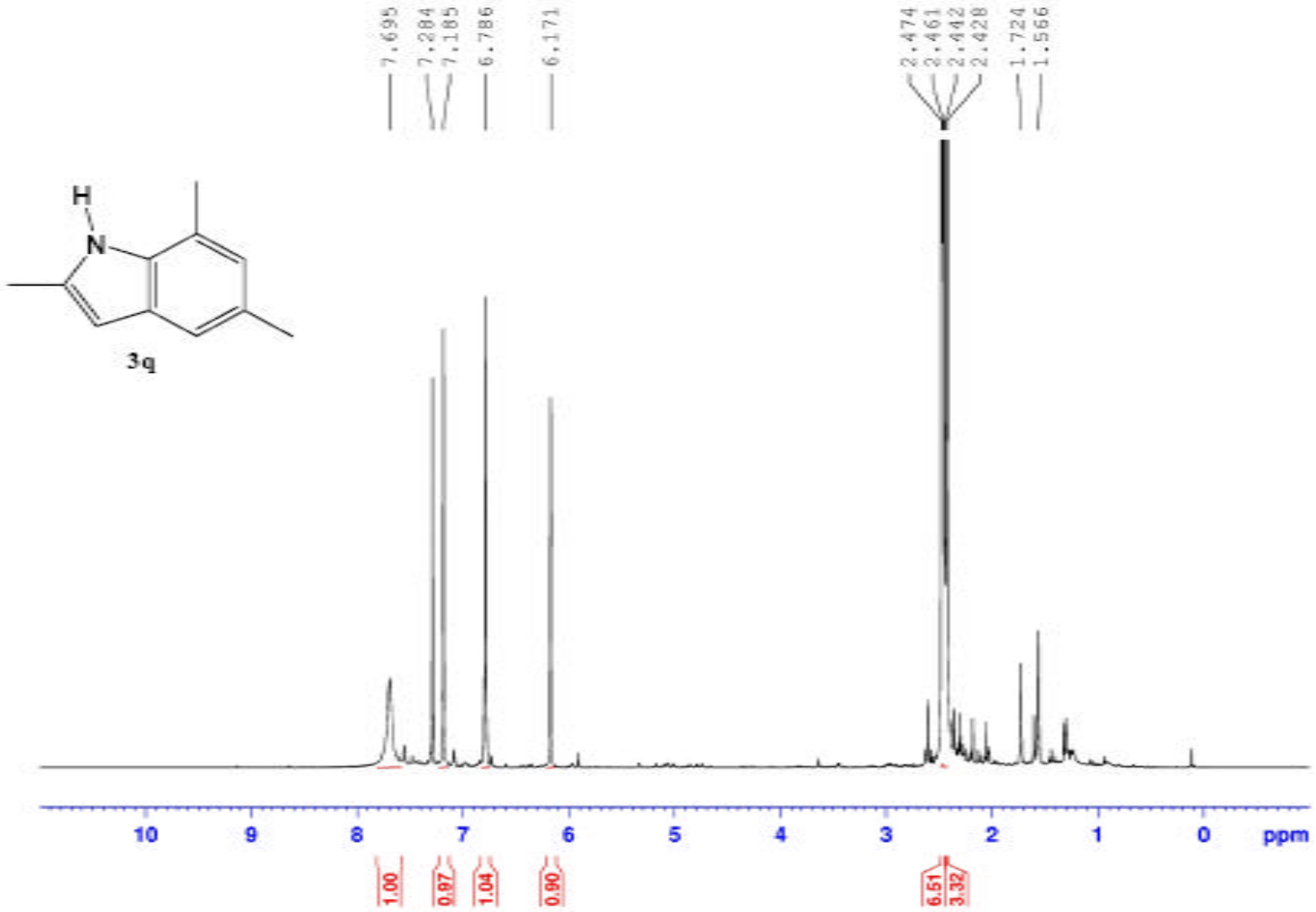

8.397
7.690
7.671
7.614
7.501
7.482
7.463
7.395
7.376
7.352
7.331
7.304
7.286
7.269
7.259
7.176
7.154
6.788
1.592
H
N
Cl
3o
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
3.32
2.32
2.27
1.15
1.07

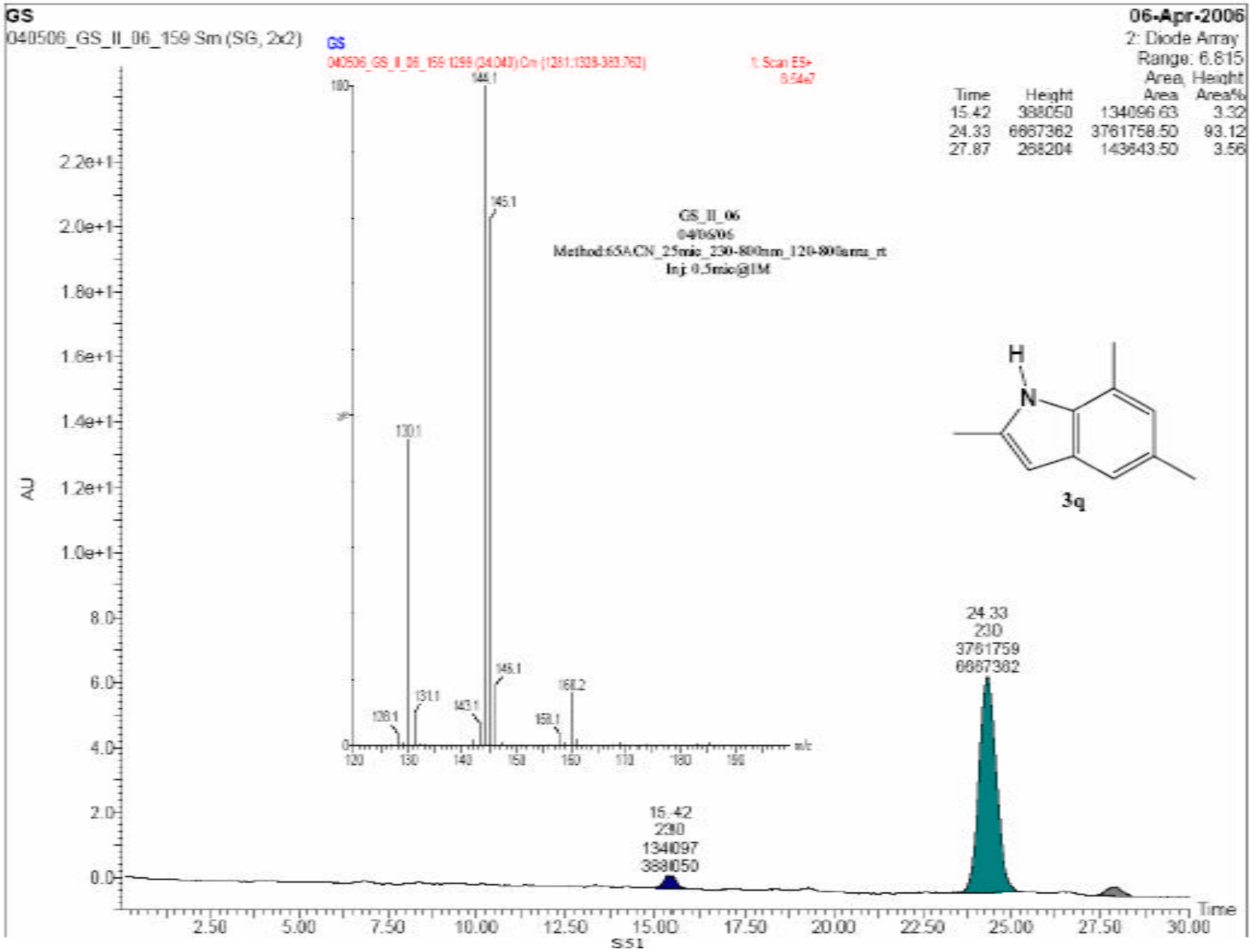

GS
040606_GS_I_91_227 Sm (SG, 2x2)
07-Apr-2006
2: Diode Array
Range: 1.264e+1
Area, Height
GS_I_91
040606
Method:65ACN_25min_230-800nm_120-80lamu_rt
Inj: 0.5mic@1M
Time Height Area Area%
9.98 155879 129454.92 1.06
42.35 12520199 12112520.00 98.94
GS
040606_GS_I_91_227 2272 (42.040) Cm (2244:2300-722:1266)
1: Scan ES+
7.53e6
193.2
194.2
165.1
192.1
166.2
191.1
167.2
228.2
m/z
H
N
Cl
3o
AU
9.98
305
129455
155879
42.35
314
12112520
12520199
Time
S46

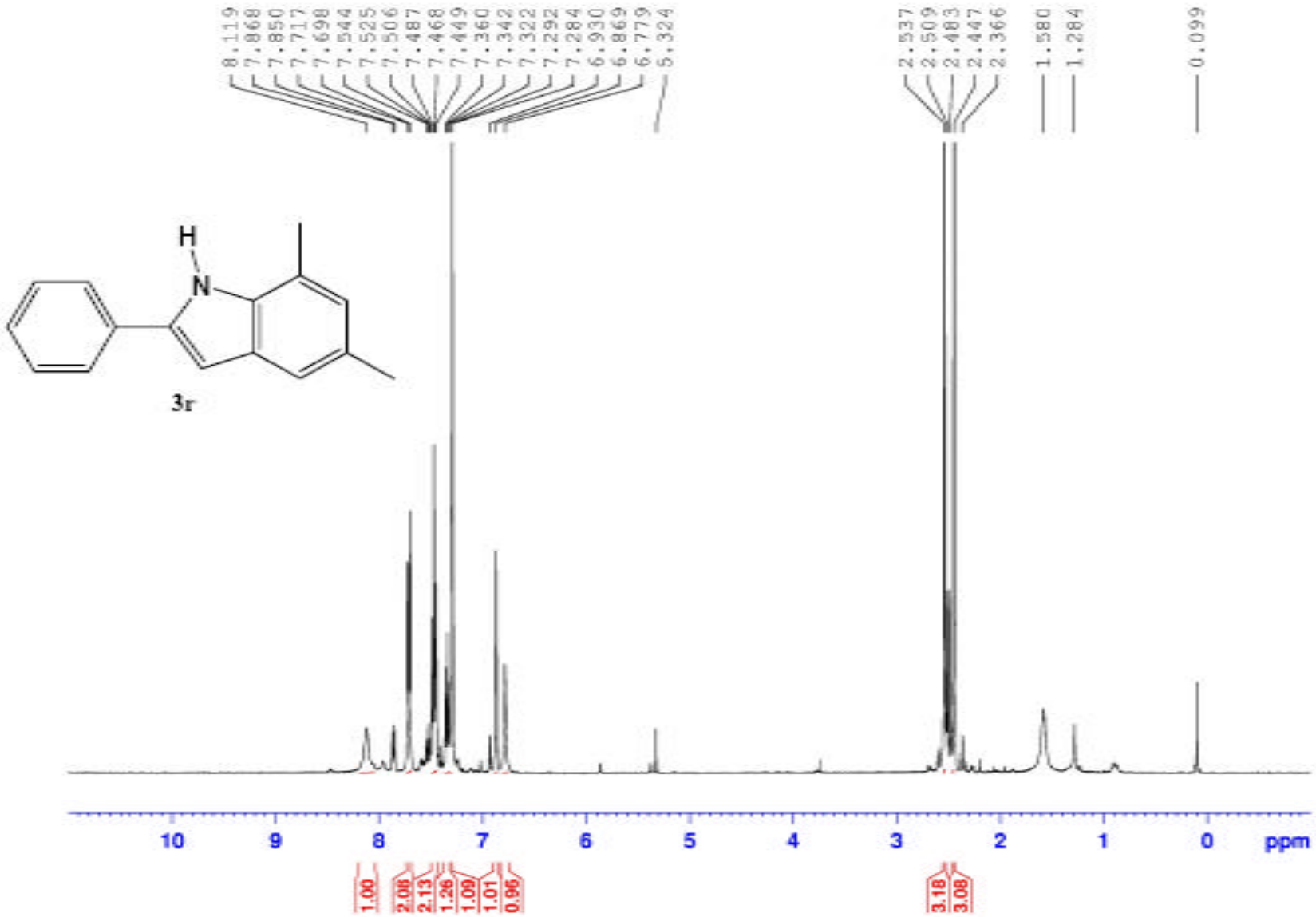

7.722
7.284
7.224
6.813
6.220
2.864
2.845
2.826
2.807
2.484
2.451
2.337
2.277
1.587
1.415
1.397
1.377
1.354
1.334
1.323
H
N
3p
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
0.98
1.04
0.88
2.71
6.66
3.88

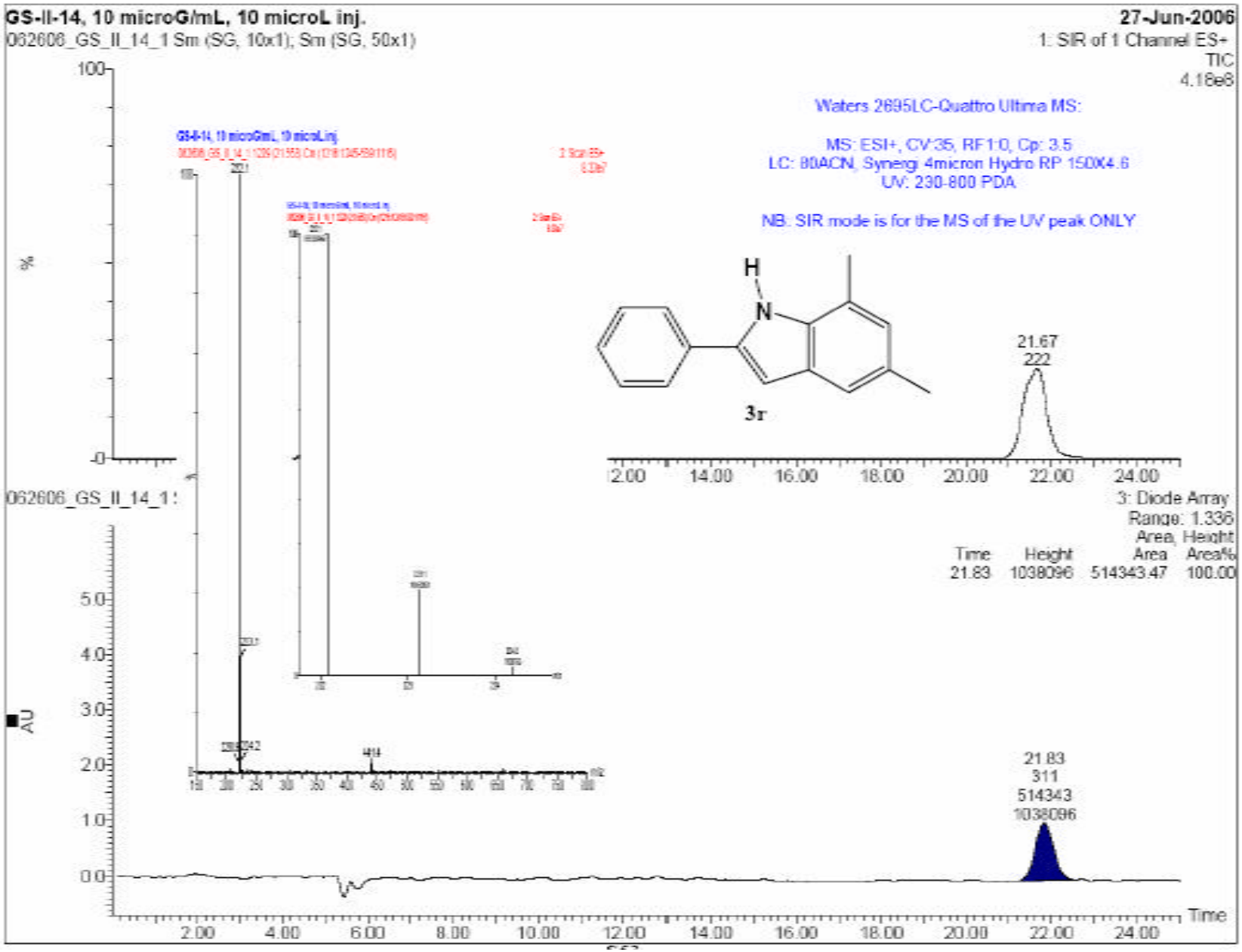

H
N
3p
200
180
160
140
120
100
80
60
40
20
0
ppm

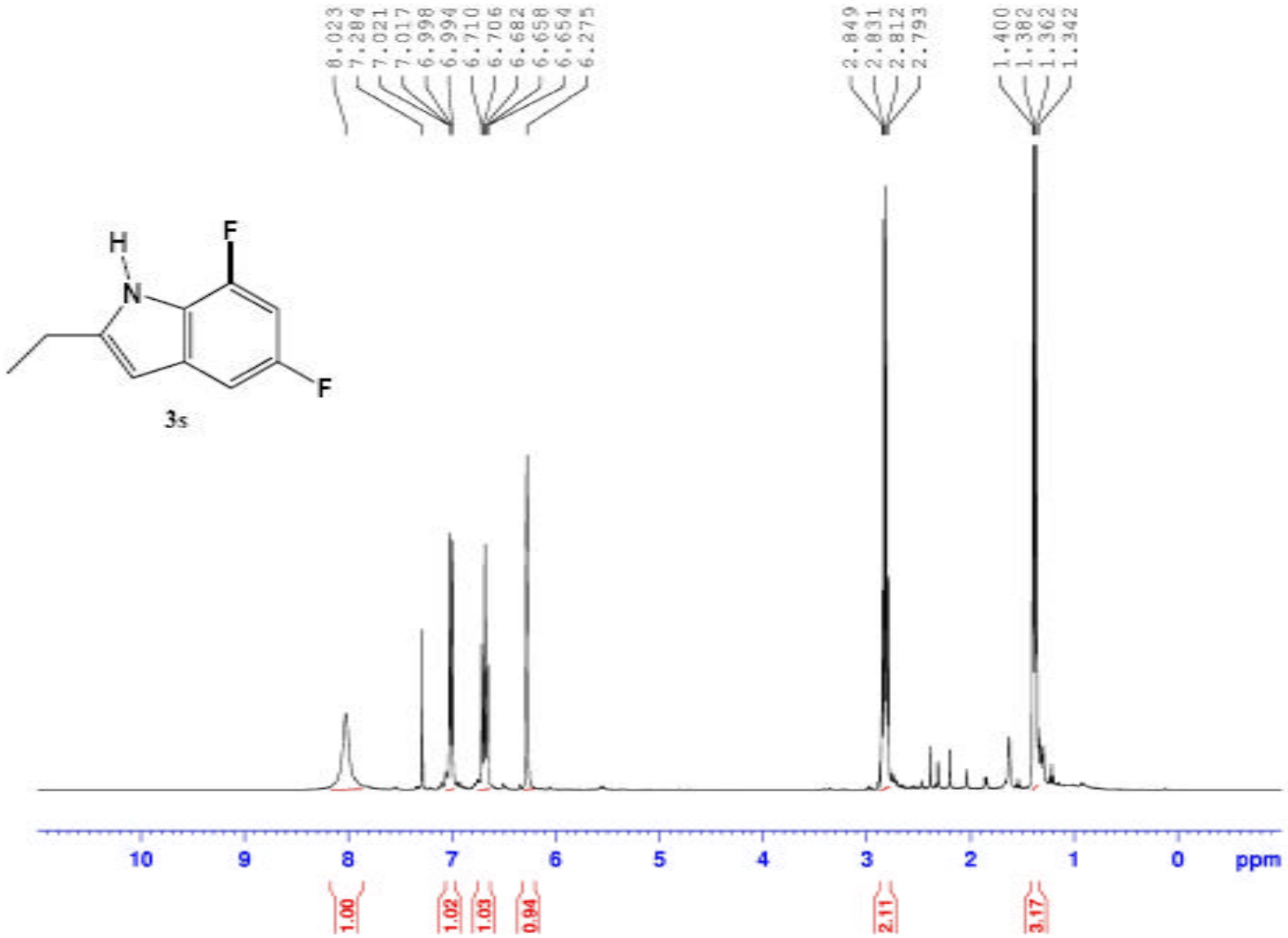

GS
040506_GS_II_11_173 Sm (SG, 2x2)
06-Apr-2006
2: Diode Array
Range: 5.368
Area, Height
Time Height Area Area%
10.75 2145971 608365.38 12.01
17.72 177480 74115.18 1.46
21.42 214912 41927.62 0.83
21.45 221070 54830.48 1.08
29.40 385143 84273.70 1.66
29.47 418129 73736.01 1.46
29.58 373670 76524.39 1.51
32.70 5315971 4049660.00 79.96
GS_II_11
040506
Method:65ACN_25min_230-800nm_120-800amu_rt
Inj: 0.5mic@1M
3p
AU
Time
10.75
237
608365
2145971
17.72
489
74115
177480
21.45
311
54830
221070
29.47
230
73736
418129
32.70
230
4049660
5315971

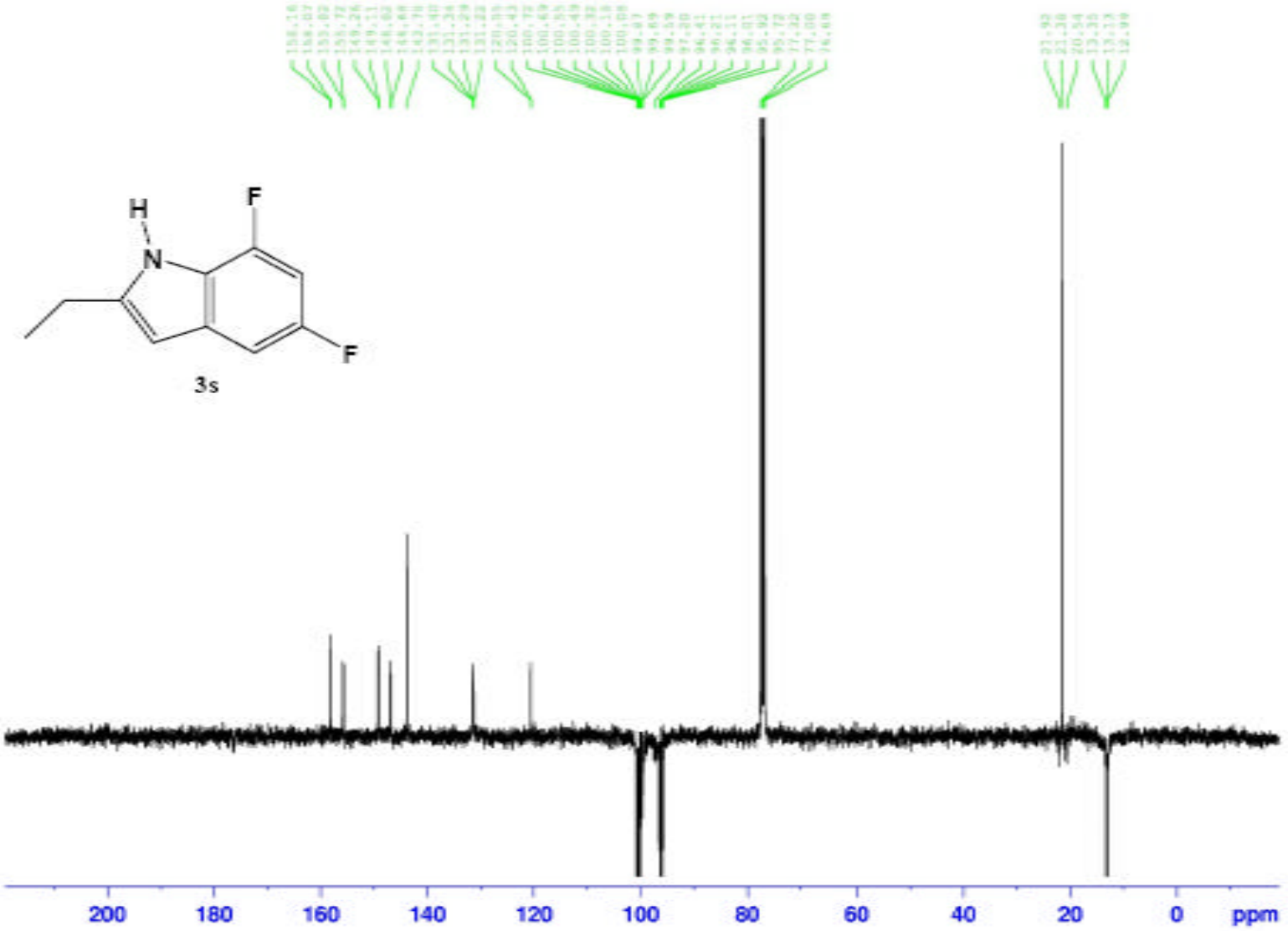

7.695
7.284
7.185
6.786
6.171
2.474
2.461
2.442
2.428
1.724
1.566
H
N
3q
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
0.97
1.04
0.90
6.51
3.32

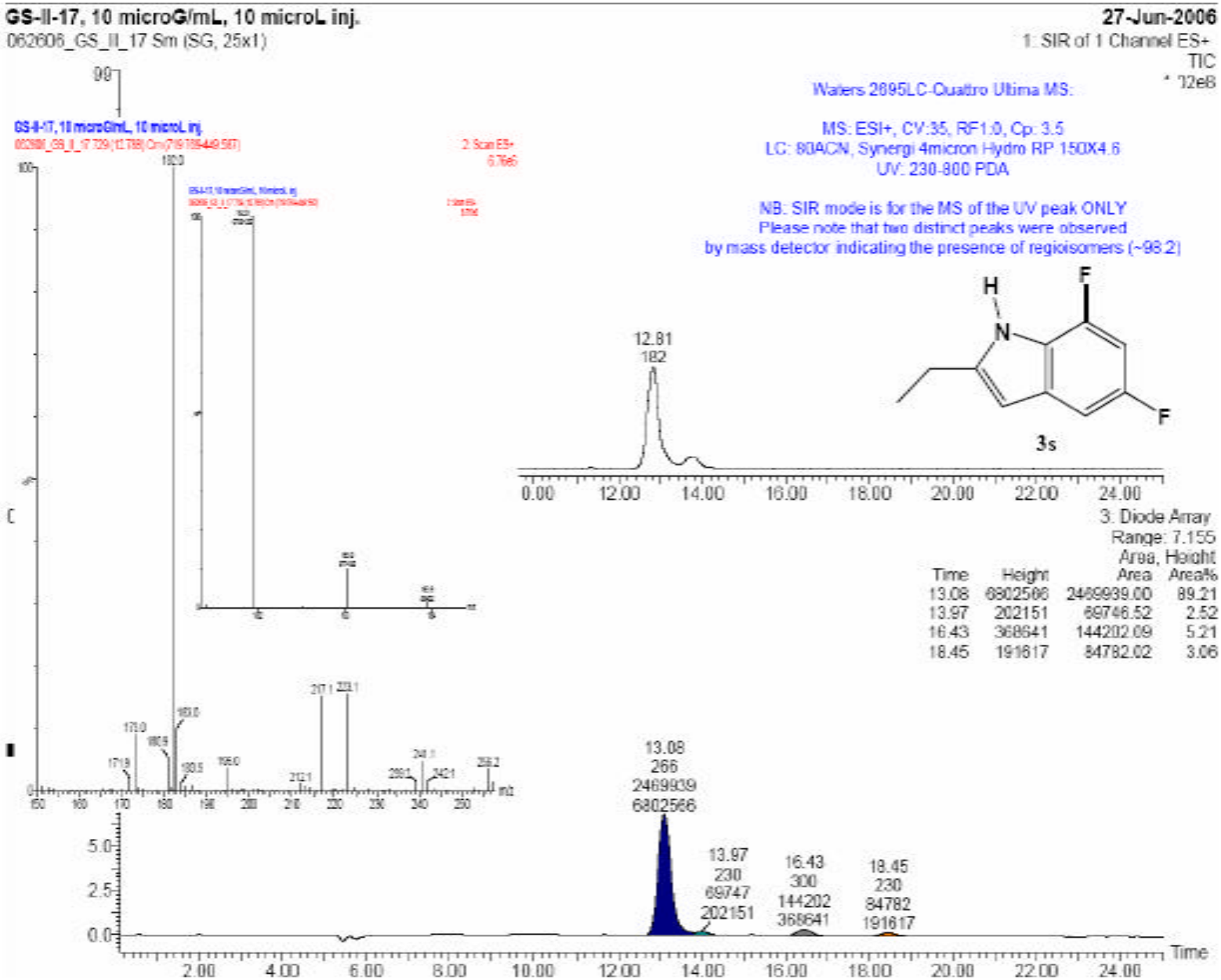

GS
040506_GS_II_06_159 Sm (SG, 2x2)
06-Apr-2006
2: Diode Array
Range: 6.815
Area, Height
Time Height Area Area%
15.42 388050 134096.63 3.32
24.33 6667362 3761758.50 93.12
27.87 268204 143643.50 3.56
GS_II_06
040506
Method:65ACN_25min_230-800nm_120-800amu_rt
Inj: 0.5mic@1M
3q
24.33
230
3761759
6667362
15.42
230
134097
388050
AU
Time

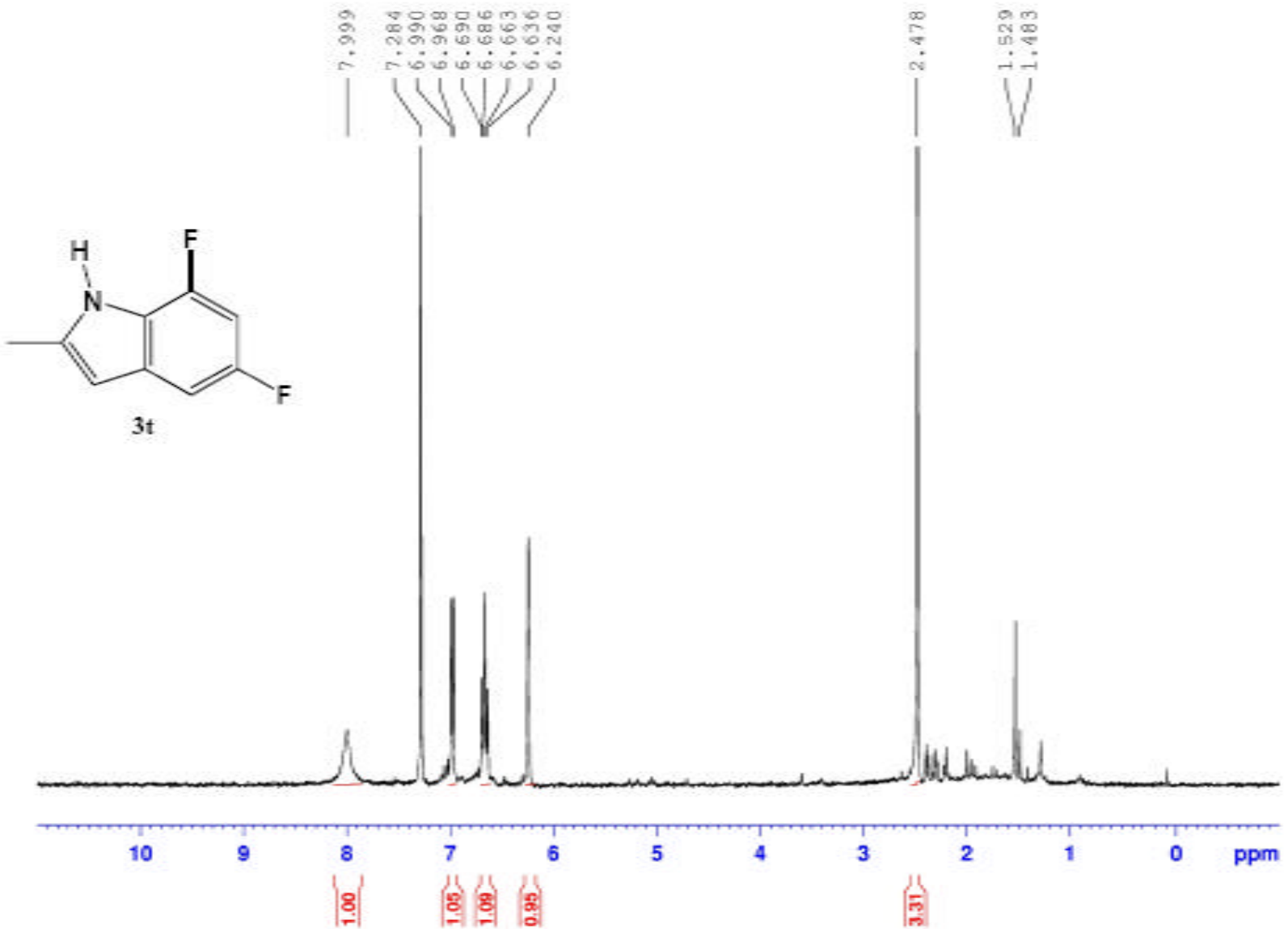

8.119
7.868
7.850
7.717
7.698
7.544
7.525
7.506
7.487
7.468
7.449
7.360
7.342
7.322
7.292
7.284
6.930
6.869
6.779
5.324
2.537
2.509
2.483
2.447
2.366
1.580
1.284
0.099
H
N
3r
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
2.08
2.13
1.26
1.09
1.01
0.96
3.16
3.08

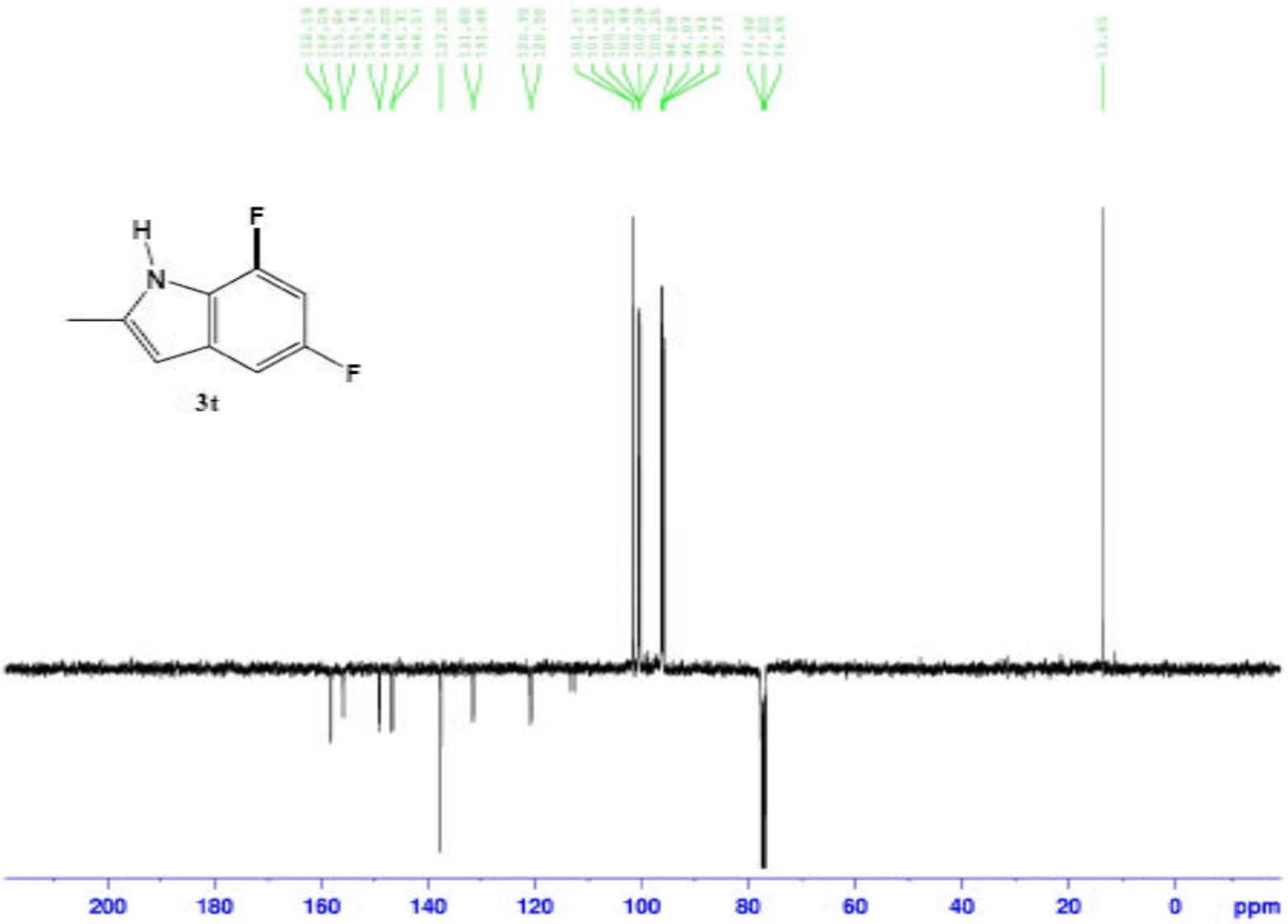

GS-II-14, 10 microG/mL, 10 microL inj.
27-Jun-2006
062606_GS_II_14_1 Sm (SG, 10x1); Sm (SG, 50x1)
1: SIR of 1 Channel ES+
TIC
4.18e8
Waters 2695LC-Quattro Ultima MS:
MS: ESI+, CV:35, RF1:0, Cp: 3.5
LC: 80ACN, Synergi 4micron Hydro RP 150X4.6
UV: 230-800 PDA
NB: SIR mode is for the MS of the UV peak ONLY
H
N
3r
21.67
222
062606_GS_II_14_1
3: Diode Array
Range: 1.336
Area, Height
Time Height Area Area%
21.83 1038096 514343.47 100.00
21.83
311
514343
1038096
AU
Time

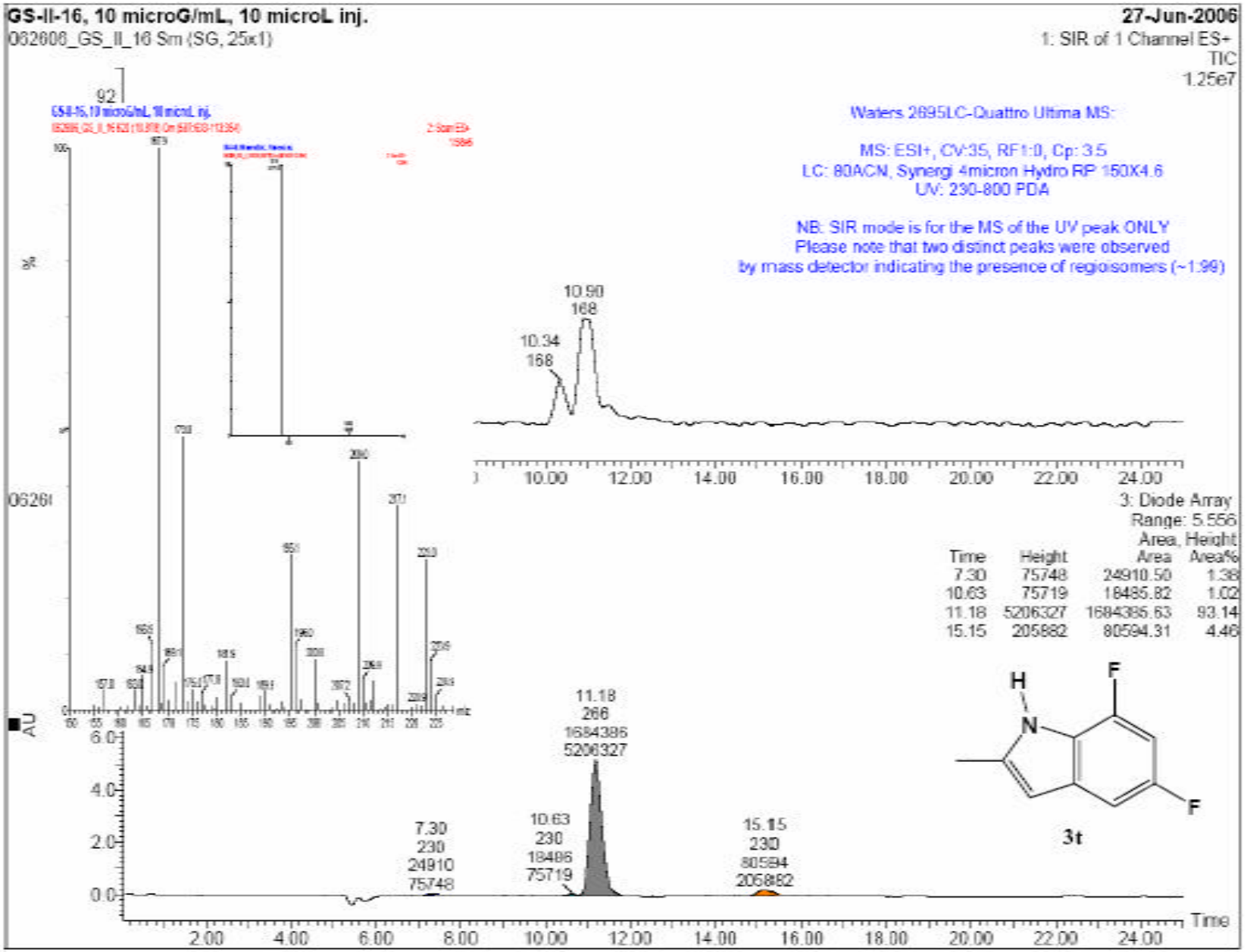

8.023
7.284
7.021
7.017
6.998
6.994
6.710
6.706
6.682
6.658
6.654
6.275
2.849
2.831
2.812
2.793
1.400
1.382
1.362
1.342
H
N
F
F
3s
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
1.02
1.03
0.94
2.11
3.17

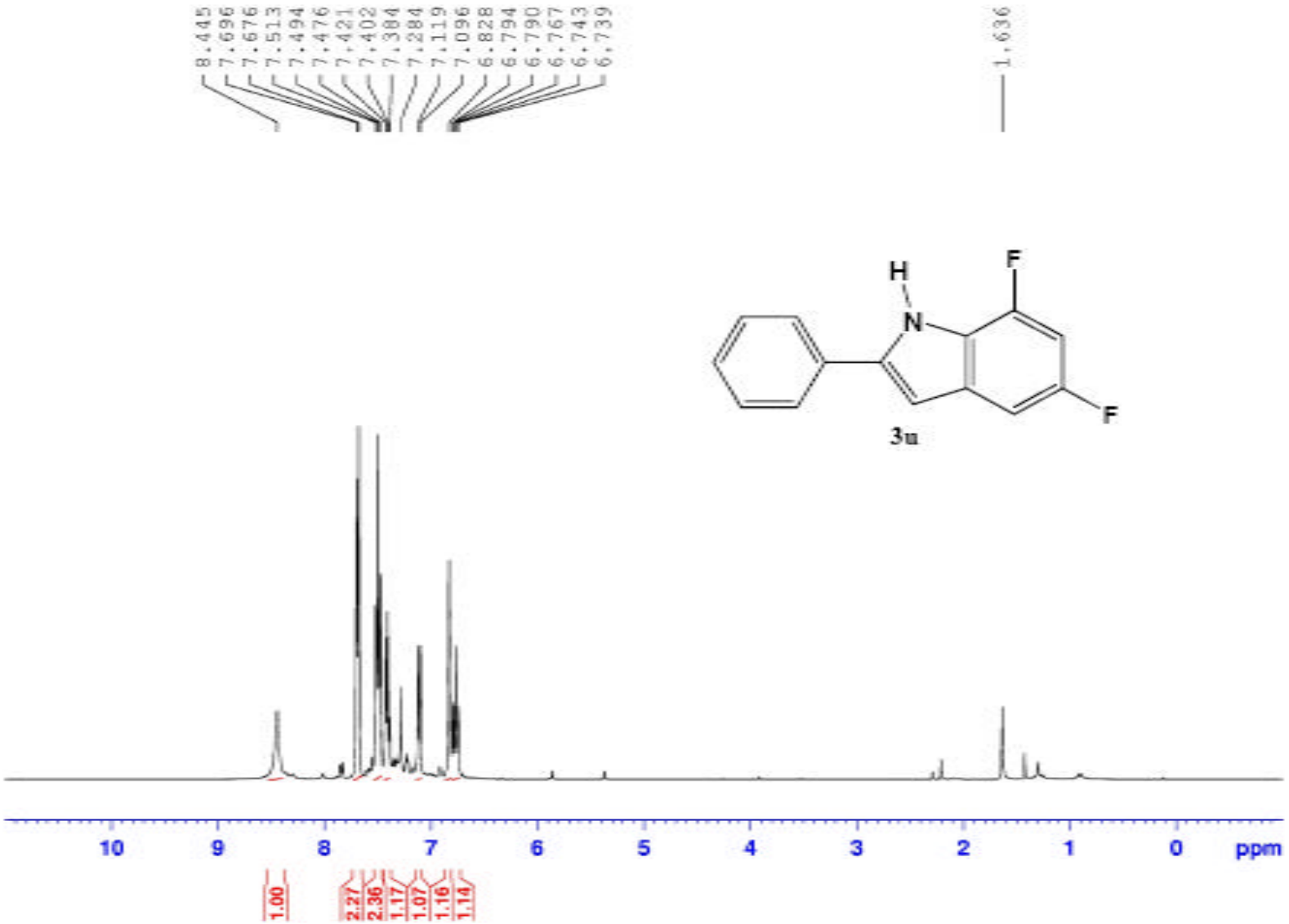

H
N
F
F
3s
200
180
160
140
120
100
80
60
40
20
0
ppm

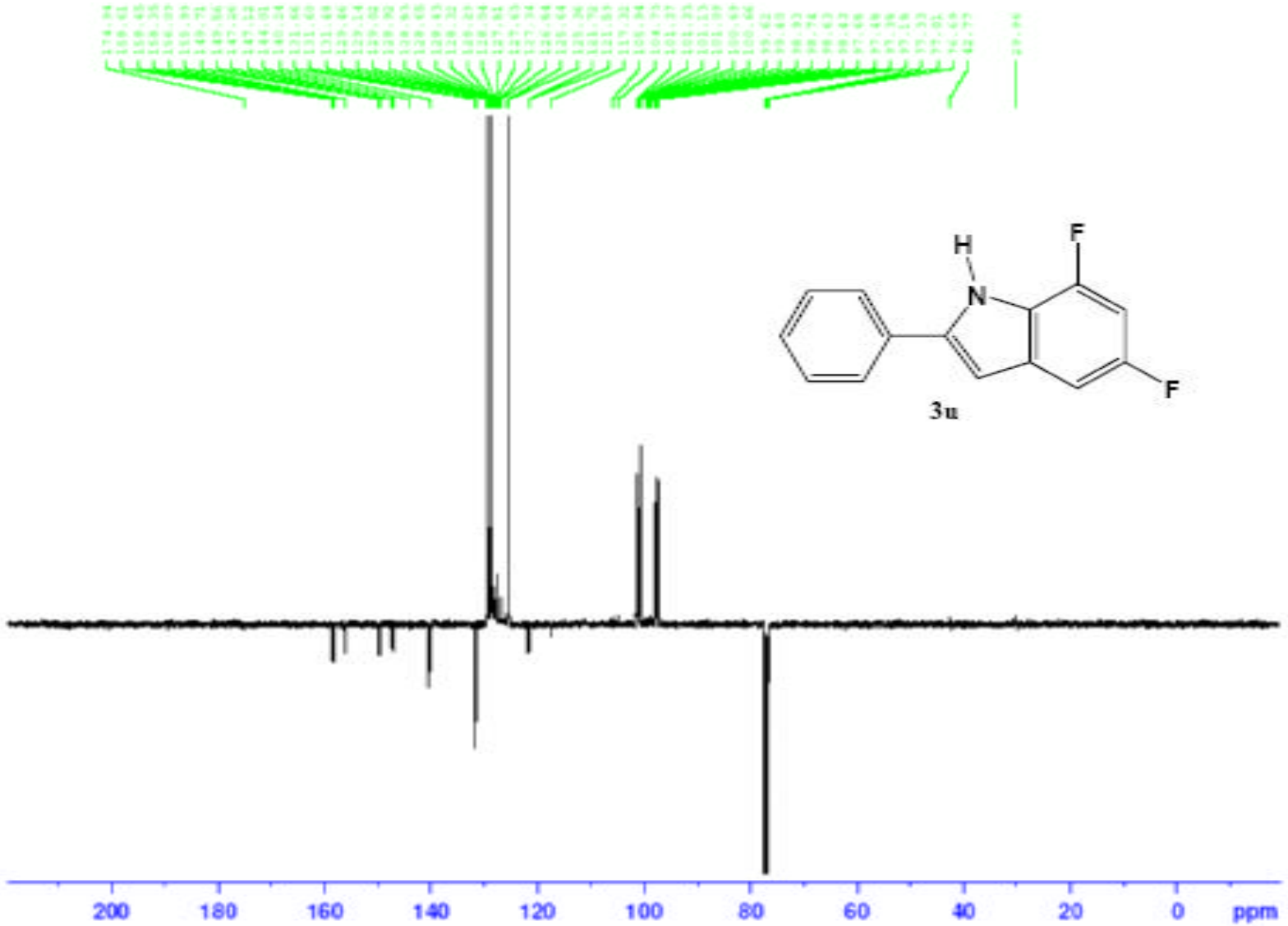

GS-II-17, 10 microG/mL, 10 microL inj.
062606_GS_II_17 Sm (SG, 25x1)
27-Jun-2006
1: SIR of 1 Channel ES+
TIC
Waters 2695LC-Quattro Ultima MS:
MS: ESI+, CV:35, RF1.0, Cp: 3.5
LC: 80ACN, Synergi 4micron Hydro RP 150X4.6
UV: 230-800 PDA
NB: SIR mode is for the MS of the UV peak ONLY
Please note that two distinct peaks were observed
by mass detector indicating the presence of regioisomers (~98:2)
12.81
182
3s
3: Diode Array
Range: 7.155
Area, Height
Time Height Area Area%
13.08 6802566 2469939.00 89.21
13.97 202151 69746.52 2.52
16.43 368641 144202.09 5.21
18.45 191617 84782.02 3.06
13.08
266
2469939
6802566
13.97
230
69747
202151
16.43
300
144202
368641
18.45
230
84782
191617
Time

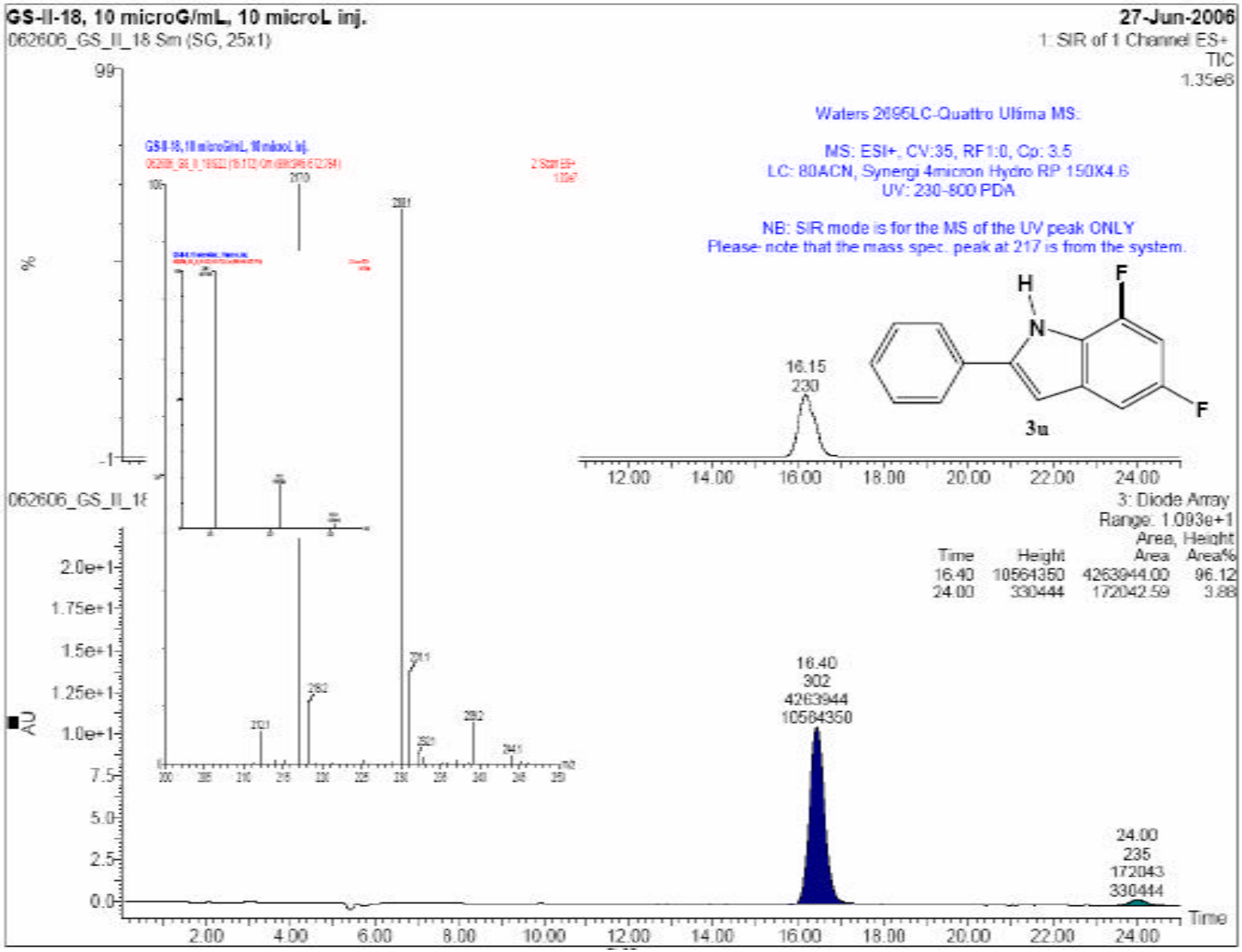

7.999
7.284
6.990
6.968
6.690
6.686
6.663
6.636
6.240
2.478
1.529
1.483
H
N
F
F
3t
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
1.05
1.09
0.95
3.31


H
N
F
F
3t
200
180
160
140
120
100
80
60
40
20
0
ppm


GS-II-16, 10 microG/mL, 10 microL inj.
062606_GS_II_16 Sm (SG, 25x1)
27-Jun-2006
1: SIR of 1 Channel ES+
TIC
1.25e7
Waters 2695LC-Quattro Ultima MS:
MS: ESI+, CV:35, RF1:0, Cp: 3.5
LC: 80ACN, Synergi 4micron Hydro RP 150X4.6
UV: 230-800 PDA
NB: SIR mode is for the MS of the UV peak ONLY
Please note that two distinct peaks were observed
by mass detector indicating the presence of regioisomers (~1.99)
10.90
168
10.34
168
3: Diode Array
Range: 5.556
Area, Height
Time Height Area Area%
7.30 75748 24910.50 1.38
10.63 75719 18486.82 1.02
11.18 5206327 1684386.63 93.14
15.15 205882 80594.31 4.46
11.18
266
1684386
5206327
7.30
230
24910
75748
10.63
230
18486
75719
15.15
230
80594
205882
H
N
F
F
3t
AU
Time


8.445
7.696
7.676
7.513
7.494
7.476
7.421
7.402
7.384
7.284
7.119
7.096
6.828
6.794
6.790
6.767
6.743
6.739
1.636
H
N
F
F
3u
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
2.27
2.36
1.17
1.07
1.16
1.14


H
N
F
F
3u
200
180
160
140
120
100
80
60
40
20
0
ppm


GS-II-18, 10 microG/mL, 10 microL inj.
062606_GS_II_18 Sm (SG, 25x1)
27-Jun-2006
1: SIR of 1 Channel ES+
TIC
1.35e6
Waters 2695LC-Quattro Ultima MS:
MS: ESI+, CV:35, RF1:0, Cp: 3.5
LC: 80ACN, Synergi 4micron Hydro RP 150X4.6
UV: 230-800 PDA
NB: SIR mode is for the MS of the UV peak ONLY
Please note that the mass spec. peak at 217 is from the system.
16.15
230
3u
3: Diode Array
Range: 1.093e+1
Area, Height
Time Height Area Area%
16.40 10564350 4263944.00 96.12
24.00 330444 172042.59 3.88
16.40
302
4263944
10564350
24.00
235
172043
330444
AU
%
Time