

Synthesis of 3-(Aminomethylene)-2-oxoindolines by Palladium Catalyzed Oxidative Tandem Reactions of 3-Chloro-2-iodo-*N*-arylacrylamides with Amides

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Supporting Information

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(A) Typical Experimental Procedure

Typical Experimental Procedure for the Synthesis of

3-Chloro-2-iodo-N-arylacrylamides (**1**):

To a three-neck flask were added *N*-arylpropiolamides (3 mmol) and CH₂Cl₂ (5 mL). Then ICl (4 mmol)/CH₂Cl₂ (2 mL) solution was dropped in the flask at 0 °C for about 1 h. The mixture was stirred at room temperature for about 1-5 h until complete consumption of starting material as monitored by TLC analysis. After the reaction was finished, the reaction mixture was diluted in diethyl ether, and washed with saturated Na₂S₂O₃ and brine. The aqueous phase was extracted with diethyl ether. The combined organic extracts were dried over Na₂SO₄ and concentrated in vacuum, and the resulting residue was purified by silica gel column chromatography (hexane/ethyl acetate) to afford 3-chloro-2-iodo-*N*-arylacrylamides (**1**).

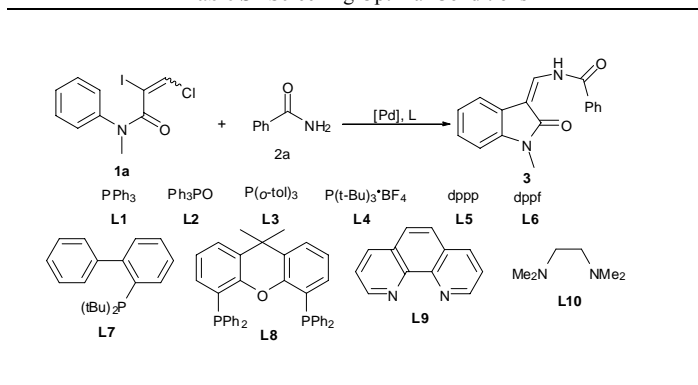
Typical Experimental Procedure for Palladium-Catalyzed Synthesis

3-(Aminomethylene)-2-oxoindolines:

To a Schlenk tube were added 3-Chloro-2-iodo-*N*-arylacrylamides **1** (0.3 mmol), amides **2** (0.45 mmol), Pd(dba)₂ (10 mol %), Ag(OAc)₂ (2 equiv), dimethylbis(diphenylphosphino)ethane (**L8**, 10 mol %), Na₂CO₃ (2 equiv), and toluene (2 mL). Then the tube was charged with argon, and was stirred at 50 °C (oil bath temperature) for the indicated time until complete consumption of starting material as monitored by TLC and GC-MS analysis. After the reaction was finished, the reaction mixture was diluted in diethyl ether, and washed with brine. The aqueous phase was extracted with diethyl ether.

The combined organic extracts were dried over Na₂SO₄ and concentrated in vacuum, and the resulting residue was purified by silica gel column chromatography (hexane/ethyl acetate) to afford the product.

Table S1 Screening Optimal Conditions^d

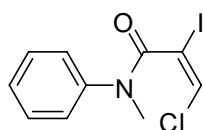


| Entry | [Pd] (mol%) | ligand | [Ag] (equivi) | base | Yield (%) ^b |
|-----------------|--|------------|--|---------------------------------|------------------------|
| 1 | Pd(OAc) ₂ | — | AgOAc (1) | Cs ₂ CO ₃ | trace |
| 2 | Pd(OAc) ₂ | L1 | AgOAc (1) | Cs ₂ CO ₃ | 20 |
| 3 | Pd(OAc) ₂ | L2 | AgOAc (1) | Cs ₂ CO ₃ | trace |
| 4 | Pd(OAc) ₂ | L3 | AgOAc (1) | Cs ₂ CO ₃ | 30 |
| 5 | Pd(OAc) ₂ | L4 | AgOAc (1) | Cs ₂ CO ₃ | 21 |
| 6 | Pd(OAc) ₂ | L5 | AgOAc (1) | Cs ₂ CO ₃ | trace |
| 7 | Pd(OAc) ₂ | L6 | AgOAc (1) | Cs ₂ CO ₃ | trace |
| 8 | Pd(OAc) ₂ | L7 | AgOAc (1) | Cs ₂ CO ₃ | 30 |
| 9 | Pd(OAc) ₂ | L7 | AgOAc (1) | Na ₂ CO ₃ | 35 |
| 10 | Pd(OAc) ₂ | L8 | AgOAc (1) | Na ₂ CO ₃ | 45 |
| 11 | Pd(OAc) ₂ | L9 | AgOAc (1) | Na ₂ CO ₃ | 25 |
| 12 | Pd(OAc) ₂ | L10 | AgOAc (1) | Na ₂ CO ₃ | trace |
| 13 | PdCl ₂ | L8 | AgOAc (1) | Na ₂ CO ₃ | 67 |
| 14 | Pd(PPh ₃) ₂ Cl ₂ | L8 | AgOAc (1) | Na ₂ CO ₃ | 65 |
| 15 | Pd(PPh ₃) ₄ | L8 | AgOAc (1) | Na ₂ CO ₃ | 66 |
| 16 | Pd(dba) ₂ | L8 | AgOAc (1) | Na ₂ CO ₃ | 68 |
| 17 | Pd(dba) ₂ | L8 | CF ₃ CO ₂ Ag (1) | Na ₂ CO ₃ | 46 |
| 18 | Pd(dba) ₂ | L8 | Ag ₂ O (1) | Na ₂ CO ₃ | 56 |
| 19 | Pd(dba) ₂ | L8 | Ag ₂ CO ₃ (1) | Na ₂ CO ₃ | 50 |
| 20 | Pd(dba) ₂ | L8 | AgOAc (1.5) | Na ₂ CO ₃ | 79 |
| 21 | Pd(dba) ₂ | L8 | AgOAc (2) | Na ₂ CO ₃ | 85 |
| 22 | Pd(dba) ₂ | L8 | AgOAc (3) | Na ₂ CO ₃ | 80 |
| 23 ^c | Pd(dba) ₂ | L8 | AgOAc (1) | Na ₂ CO ₃ | 86 |
| 24 ^d | Pd(dba) ₂ | L8 | AgOAc (1) | Na ₂ CO ₃ | 72 |

| | | | | | |
|------------------|----------------------|-----------|-----------|---------------------------------|---------|
| 25 ^c | Pd(dba) ₂ | L8 | AgOAc (1) | Et ₃ N | trace |
| 26 ^c | Pd(dba) ₂ | L8 | AgOAc (1) | NaHCO ₃ | 30 |
| 27 ^{ce} | Pd(dba) ₂ | L8 | AgOAc (1) | Na ₂ CO ₃ | 83 |
| 28 ^{cf} | Pd(dba) ₂ | L8 | AgOAc (1) | Na ₂ CO ₃ | 58 |
| 29 ^{cg} | Pd(dba) ₂ | L8 | AgOAc (1) | — | 0 |
| 30 ^{ch} | Pd(dba) ₂ | L8 | AgOAc (1) | Na ₂ CO ₃ | mixture |

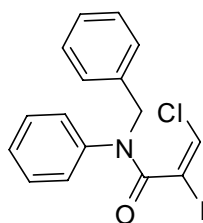
^a Reaction conditions: **1a** (0.3 mmol), **2a** (0.45 mmol), [Pd] (10 mol %), ligand (10 mol %), [Ag] (1 equiv), base (2 equiv), and solvent (2 mL) at 80 °C for 8 h. ^b Isolated yield. ^c At 50 °C for 13 h. ^d At room temperature for 96 h. ^e In THF. ^f 5 mol % of Pd(dba)₂ was added. ^g Without base. ^h Without Ag salt.

(B) Analytical data for **1** and **3-24**



3-Chloro-2-iodo-N-methyl-N-phenylacrylamide (1a):¹

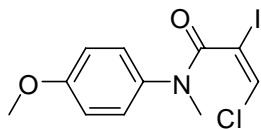
White solid, mp 114.5-116.2 °C (uncorrected); ¹H NMR (500 MHz) δ: 7.40-7.35 (m, 5H), 6.26 (s, 1H), 3.35 (s, 3H); ¹³C NMR (125 MHz) δ: 164.1, 142.0, 129.2, 128.4, 126.5, 122.4, 86.9, 37.5; IR (KBr, cm⁻¹): 1648; LRMS (EI, 70 eV) *m/z* (%): 323 (M⁺+2, 0.3), 321 (M⁺, 0.9), 194 (100), 159 (55); HRMS *m/z* (EI) calcd for C₁₀H₉ClINO (M⁺) 320.9417, found 320.9413.



N-Benzyl-3-chloro-2-iodo-N-phenylacrylamide (1b):¹

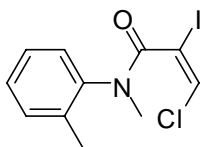
Colorless oil; ¹H NMR (500 MHz) δ: 7.32-7.16 (m, 10H), 6.22 (s, 1H), 4.93 (s, 2H); ¹³C NMR (125 MHz) δ: 164.0, 140.0, 136.3, 128.8, 128.6, 128.5, 128.4, 127.5, 122.1, 86.7, 53.1; IR (KBr, cm⁻¹): 1646; LRMS (EI, 70 eV) *m/z* (%): 399 (M⁺+2, 0.4), 397 (M⁺, 1), 361

(9), 216 (11), 91 (100); HRMS m/z (EI) calcd for $C_{16}H_{13}ClINO$ (M^+) 396.9730, found 396.9727.



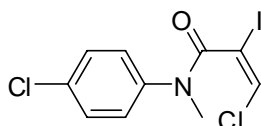
3-Chloro-2-iodo-N-(4-methoxyphenyl)-N-methylacrylamide (1c):¹

Colorless oil; 1H NMR (500 MHz) δ : 7.16 (d, $J = 9.0$ Hz, 2H), 6.84 (d, $J = 10.0$ Hz, 2H), 3.76 (s, 3H), 3.24 (s, 3H); ^{13}C NMR (125 MHz) δ : 164.3, 159.4, 134.7, 127.7, 120.0, 114.3, 87.0, 55.4, 37.6; IR (KBr, cm^{-1}): 1650; LRMS (EI, 70 eV) m/z (%): 353 ($M^+ + 2$, 4), 351 (M^+ , 12), 189 (83), 136 (100); HRMS m/z (EI) calcd for $C_{11}H_{11}ClINO_2$ (M^+) 350.9523, found 350.9520.



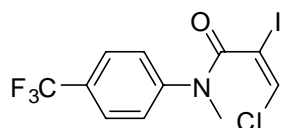
3-Chloro-2-iodo-N-methyl-N-o-tolylacrylamide (1d):¹

Colorless oil; 1H NMR (500 MHz) δ : 7.38-7.07 (m, 4H), 6.25 (s, 1H), 3.18 (s, 3H), 2.30 (s, 3H); ^{13}C NMR (125 MHz) δ : 164.4, 140.4, 135.2, 131.7, 129.1, 127.7, 126.8, 122.9, 86.3, 36.6, 18.6; IR (KBr, cm^{-1}): 1646; LRMS (EI, 70 eV) m/z (%): 337 ($M^+ + 2$, 0.2), 335 (M^+ , 0.7), 208 (100), 173 (42); HRMS m/z (EI) calcd for $C_{11}H_{11}ClINO$ (M^+) 334.9574, found 334.9573.



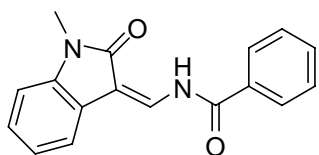
3-Chloro-N-(4-chlorophenyl)-2-iodo-N-methylacrylamide (1e):¹

Colorless oil; ^1H NMR (500 MHz) δ : 7.37 (d, $J = 9.0$ Hz, 2H), 7.30 (d, $J = 9.0$ Hz, 2H), 6.28 (s, 1H), 3.32 (s, 3H); ^{13}C NMR (125 MHz) δ : 163.8, 140.3, 134.0, 129.3, 127.7, 122.5, 86.5, 37.3; IR (KBr, cm^{-1}): 1646; LRMS (EI, 70 eV) m/z (%): 357 ($\text{M}^+ + 2$, 12), 355 (M^+ , 39), 193 (67), 105 (100); HRMS m/z (EI) calcd for $\text{C}_{10}\text{H}_8\text{Cl}_2\text{INO}$ (M^+) 354.9028, found 354.9026.



3-Chloro-2-iodo-N-methyl-N-(4-(trifluoromethyl)phenyl)acrylamide (1f):¹

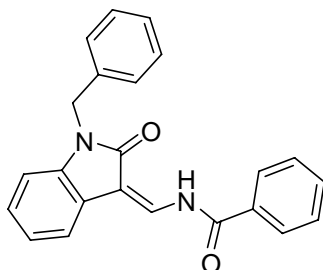
Colorless oil; ^1H NMR (500 MHz) δ : 7.65 (d, $J = 8.0$ Hz, 2H), 7.47 (d, $J = 8.0$ Hz, 2H), 6.31 (s, 1H), 3.36 (s, 3H); ^{13}C NMR (125 MHz) δ : 163.7, 145.0, 127.1, 126.7, 126.3 (3C), 126.2, 123.0, 86.4, 83.3, 37.2; IR (KBr, cm^{-1}): 1659; LRMS (EI, 70 eV) m/z (%): 391 ($\text{M}^+ + 2$, 0.14), 389 (M^+ , 0.44), 354 (3), 315 (54), 262 (100); HRMS m/z (EI) calcd for $\text{C}_{11}\text{H}_8\text{ClI}_3\text{NO}$ (M^+) 388.9291, found 388.9295.



(Z)-N-((1-Methyl-2-oxoindolin-3-ylidene)methyl)benzamide (3):

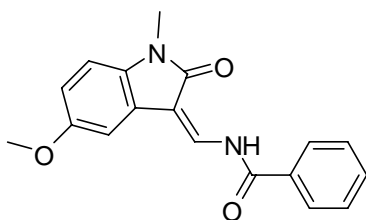
Yellow solid, mp 160.2-162.6 °C (uncorrected); ^1H NMR (500 MHz) δ : 12.2 (d, $J = 10.0$ Hz, 1H), 8.34 (d, $J = 10.5$ Hz, 1H), 8.05 (d, $J = 7.5$ Hz, 2H), 7.61 (t, $J = 7.5$ Hz, 1H), 7.53 (t, $J = 7.5$ Hz, 2H), 7.47 (d, $J = 7.5$ Hz, 1H), 7.24 (d, $J = 7.5$ Hz, 1H), 7.08 (t, $J = 7.5$ Hz, 1H), 6.88 (d, $J = 8.0$ Hz, 1H), 3.32 (s, 3H); ^{13}C NMR (125 MHz) δ : 169.2, 164.6, 140.8, 133.1, 131.9, 129.0, 127.9, 127.3, 122.3, 122.0, 118.6, 108.4, 108.1, 105.8, 25.8; IR (KBr,

cm^{-1}): 1686, 1636; LRMS (EI, 70 eV) m/z (%): 278 (M^+ , 30), 105 (100), 77 (42); HRMS m/z (EI) calcd for $\text{C}_{17}\text{H}_{14}\text{N}_2\text{O}_2$ (M^+) 278.1055, found 278.1052.



(Z)-N-((1-Benzyl-2-oxoindolin-3-ylidene)methyl)benzamide (4):

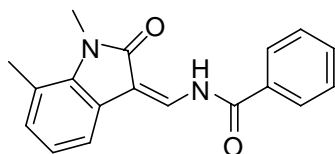
Yellow solid, mp 178.1-180.9 °C (uncorrected); ^1H NMR (500 MHz) δ : 12.21 (d, $J = 10.5$ Hz, 1H), 8.41 (d, $J = 11.0$ Hz, 1H), 8.07 (d, $J = 7.5$ Hz, 2H), 7.61 (t, $J = 7.5$ Hz, 1H), 7.54-7.48 (m, 3H), 7.31 (d, $J = 5.5$ Hz, 4H), 7.27-7.25 (m, 1H), 7.16-7.13 (m, 1H), 7.07-7.04 (m, 1H), 6.80 (d, $J = 9.0$ Hz, 1H), 5.03 (s, 3H); ^{13}C NMR (125 MHz) δ : 169.2, 164.6, 139.9, 135.8, 133.1, 131.8, 128.9, 128.8, 127.9, 127.6, 127.2, 127.1, 122.3, 122.1, 118.6, 109.3, 107.8, 43.3; 1638; LRMS (EI, 70 eV) m/z (%): 354 (M^+ , 43), 105 (100), 77 (33); HRMS m/z (EI) calcd for $\text{C}_{23}\text{H}_{18}\text{N}_2\text{O}_2$ (M^+) 354.1368, found 354.1366.



(Z)-N-((5-Methoxy-1-methyl-2-oxoindolin-3-ylidene)methyl)benzamide (5):

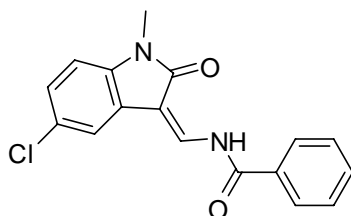
Yellow solid, mp 170.2-172.4 °C (uncorrected); ^1H NMR (500 MHz) δ : 12.20 (d, $J = 10.5$ Hz, 1H), 8.30 (d, $J = 10.5$ Hz, 1H), 8.04 (d, $J = 7.5$ Hz, 2H), 7.61 (t, $J = 7.5$ Hz, 1H), 7.22 (t, $J = 7.5$ Hz, 2H), 7.05 (d, $J = 10.5$ Hz, 1H), 6.79-6.75 (m, 2H), 3.81 (s, 3H), 3.28 (s, 3H); ^{13}C NMR (125 MHz) δ : 168.9, 164.5, 155.8, 134.7, 133.1, 131.8, 131.3, 128.9, 127.9, 122.9, 112.8, 108.7, 108.4, 104.8, 55.8, 25.7; IR (KBr, cm^{-1}): 1671, 1635; LRMS (EI, 70

eV) m/z (%): 308 (M^+ , 46), 105 (100), 77 (37); HRMS m/z (EI) calcd for $C_{18}H_{16}N_2O_3$ (M^+) 308.1161, found 308.1163.



(Z)-N-((1,7-Dimethyl-2-oxoindolin-3-ylidene)methyl)benzamide (6):

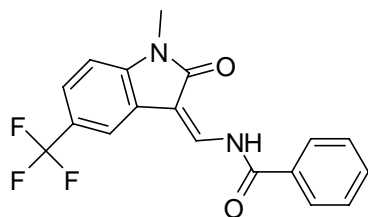
Yellow solid, mp 186.8-188.6 °C (uncorrected); 1H NMR (500 MHz) δ : 12.21 (d, J = 10.0 Hz, 1H), 8.30 (d, J = 11.0 Hz, 1H), 8.05 (d, J = 8.0 Hz, 2H), 7.61 (d, J = 9.0 Hz, 1H), 7.53 (t, J = 8.0 Hz, 2H), 7.34-7.31 (m, 1H), 6.95 (d, J = 5.0 Hz, 2H), 3.59 (s, 3H), 2.57 (s, 3H); ^{13}C NMR (125 MHz) δ : 169.5, 164.5, 138.6, 133.0, 131.9, 130.9, 130.8, 128.9, 127.9, 122.4, 122.2, 120.1, 116.4, 107.9, 28.9, 18.9; IR (KBr, cm^{-1}): 1687, 1618; LRMS (EI, 70 eV) m/z (%): 292 (M^+ , 38), 105 (100), 77 (33); HRMS m/z (EI) calcd for $C_{18}H_{16}N_2O_2$ (M^+) 292.1212, found 292.1211.



(Z)-N-((5-Chloro-1-methyl-2-oxoindolin-3-ylidene)methyl)benzamide (7):

Yellow solid, mp 188.3-190.8 °C (uncorrected); 1H NMR (500 MHz) δ : 12.15 (d, J = 10.5 Hz, 1H), 8.33 (d, J = 11.0 Hz, 1H), 8.03 (d, J = 7.5 Hz, 2H), 7.62 (t, J = 7.5 Hz, 1H), 7.53 (t, J = 7.5 Hz, 2H), 7.42 (s, 1H), 7.20-7.18 (m, 1H), 6.79 (d, J = 8.5 Hz, 1H), 3.31 (s, 3H); ^{13}C NMR (125 MHz) δ : 168.9, 164.5, 139.1, 133.3, 132.6, 131.6, 129.1, 127.9, 126.9, 123.5, 118.7, 109.2, 107.0, 25.9; IR (KBr, cm^{-1}): 1679, 1626; LRMS (EI, 70 eV) m/z (%):

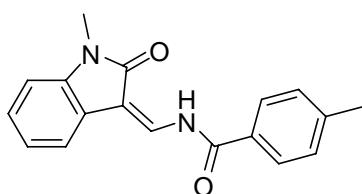
314 (M^{+2} , 10), 312 (M^{+} , 26), 105 (100), 77 (40); HRMS m/z (EI) calcd for $C_{17}H_{13}ClN_2O_2$ (M^{+}) 312.0666, found 312.0662.



(Z)-N-((1-Methyl-2-oxo-5-(trifluoromethyl)indolin-3-ylidene)methyl)benzamide

(8):

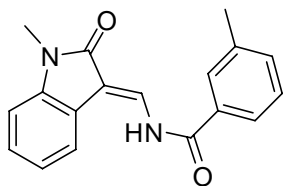
Yellow solid, mp 224.3-226.4 °C (uncorrected); 1H NMR (500 MHz) δ : 12.14 (d, J = 10.5 Hz, 1H), 8.45 (d, J = 11.0 Hz, 1H), 8.05 (d, J = 7.5 Hz, 2H), 7.71 (s, 1H), 7.63 (t, J = 7.5 Hz, 1H), 7.56-7.51 (m, 3H), 6.96 (d, J = 8.0 Hz, 1H), 3.37 (s, 3H); ^{13}C NMR (125 MHz) δ : 169.2, 164.5, 142.9, 133.4, 133.1, 131.5, 129.1, 127.9, 124.4, 124.3, 122.4, 115.5, 115.4, 108.1, 106.6, 25.9; IR (KBr, cm^{-1}): 1683, 1635; LRMS (EI, 70 eV) m/z (%): 346 (M^{+} , 28), 105 (100), 77 (34); HRMS m/z (EI) calcd for $C_{18}H_{13}F_3N_2O_2$ (M^{+}) 346.0929, found 346.0927.



(Z)-4-Methyl-N-((1-methyl-2-oxoindolin-3-ylidene)methyl)benzamide (9):

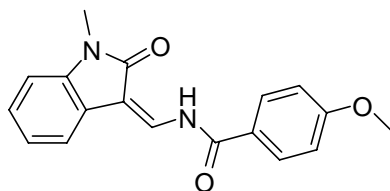
Yellow solid, mp 188.2-190.1 °C (uncorrected); 1H NMR (500 MHz) δ : 12.13 (d, J = 10.5 Hz, 1H), 8.35 (d, J = 10.5 Hz, 1H), 7.95 (d, J = 8.0 Hz, 2H), 7.47 (t, J = 7.0 Hz, 1H), 7.32 (d, J = 8.0 Hz, 1H), 7.24 (d, J = 7.5 Hz, 2H), 7.08 (t, J = 7.5 Hz, 1H), 6.89 (d, J = 7.5 Hz, 1H), 3.33 (s, 3H), 2.44 (s, 3H); ^{13}C NMR (125 MHz) δ : 169.2, 164.5, 143.9, 140.6, 131.6, 129.6, 129.0, 127.9, 127.1, 122.2, 122.1, 118.4, 108.3, 107.7, 25.7, 21.6; IR (KBr,

cm⁻¹): 1675, 1630; LRMS (EI, 70 eV) *m/z* (%): 292 (M⁺, 35), 119 (100), 91 (43); HRMS *m/z* (EI) calcd for C₁₈H₁₆N₂O₂ (M⁺) 292.1212, found 292.1210.



(Z)-3-Methyl-N-((1-methyl-2-oxoindolin-3-ylidene)methyl)benzamide (10):

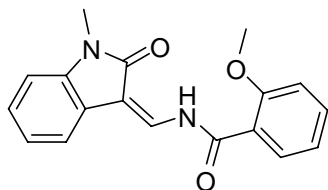
Yellow solid, mp 136.5-138.1 °C (uncorrected); ¹H NMR (500 MHz) δ: 12.10 (d, *J* = 10.5 Hz, 1H), 8.35 (d, *J* = 10.5 Hz, 1H), 7.86-7.82 (m, 2H), 7.47 (t, *J* = 7.5 Hz, 1H), 7.42-7.41 (m, 1H), 7.27-7.23 (m, 1H), 7.10-7.07 (m, 1H), 6.89 (d, *J* = 8.0 Hz, 1H), 3.33 (s, 3H), 2.46 (s, 3H); ¹³C NMR (125 MHz) δ: 169.1, 164.8, 140.7, 138.9, 133.9, 131.8, 131.4, 128.8, 128.6, 127.2, 124.9, 122.3, 122.0, 118.5, 108.3, 107.9, 25.7, 21.4; IR (KBr, cm⁻¹): 1680, 1631; LRMS (EI, 70 eV) *m/z* (%): 292 (M⁺, 35), 119 (100), 91 (42); HRMS *m/z* (EI) calcd for C₁₈H₁₆N₂O₂ (M⁺) 292.1212, found 292.1211.



(Z)-4-Methoxy-N-((1-methyl-2-oxoindolin-3-ylidene)methyl)benzamide (11):

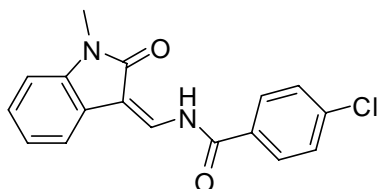
Yellow solid, mp 128.9-130.6 °C (uncorrected); ¹H NMR (500 MHz) δ: 12.12 (d, *J* = 10.5 Hz, 1H), 8.35 (d, *J* = 11.0 Hz, 2H), 8.02 (d, *J* = 9.0 Hz, 2H), 7.47 (d, *J* = 7.5 Hz, 1H), 7.24 (d, *J* = 7.5 Hz, 1H), 7.09 (t, *J* = 7.5 Hz, 1H), 7.00 (d, *J* = 8.5 Hz, 1H), 6.89 (d, *J* = 8.5 Hz, 1H), 3.89 (s, 3H), 3.34 (s, 3H); ¹³C NMR (125 MHz) δ: 169.2, 163.9, 163.5, 140.6, 131.8, 130.0, 127.0, 124.1, 122.2, 122.1, 118.4, 114.2, 108.3, 107.4, 55.5, 25.7; IR (KBr,

cm⁻¹): 1687, 1606; LRMS (EI, 70 eV) *m/z* (%): 308 (M⁺, 32), 135 (100), 77 (17); HRMS *m/z* (EI) calcd for C₁₈H₁₆N₂O₃ (M⁺) 308.1161, found 308.1163.



(Z)-2-Methoxy-N-((1-methyl-2-oxoindolin-3-ylidene)methyl)benzamide (12):

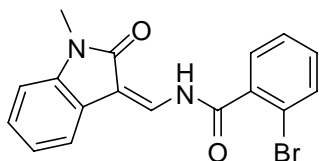
Yellow solid, mp 202.2-204.1 °C (uncorrected); ¹H NMR (500 MHz) δ: 12.73 (d, *J* = 11.0 Hz, 1H), 8.40 (d, *J* = 10.5 Hz, 1H), 8.29-8.28 (m, 1H), 7.56-7.52 (m, 1H), 7.46 (d, *J* = 7.5 Hz, 1H), 7.22 (t, *J* = 7.5 Hz, 1H), 7.11 (d, *J* = 7.5 Hz, 1H), 7.09-7.04 (m, 2H), 6.84 (d, *J* = 7.5 Hz, 1H), 4.19 (s, 3H), 3.30 (s, 3H); ¹³C NMR (125 MHz) δ: 168.1, 163.8, 158.5, 140.8, 134.5, 132.9, 130.5, 126.9, 122.6, 121.8, 121.4, 119.4, 118.2, 111.6, 107.9, 107.8, 55.8, 25.6; IR (KBr, cm⁻¹): 1687, 1622; LRMS (EI, 70 eV) *m/z* (%): 308 (M⁺, 19), 135 (55), 43 (100); HRMS *m/z* (EI) calcd for C₁₈H₁₆N₂O₃ (M⁺) 308.1161, found 308.1160.



(Z)-4-Chloro-N-((1-methyl-2-oxoindolin-3-ylidene)methyl)benzamide (13):

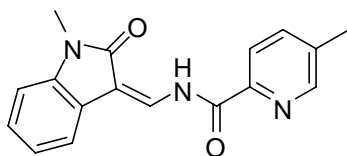
Yellow solid, mp 180.6-182.2 °C (uncorrected); ¹H NMR (500 MHz) δ: 12.19 (d, *J* = 10.5 Hz, 1H), 8.31 (d, *J* = 10.5 Hz, 1H), 7.99-7.97 (m, 2H), 7.51-7.46 (m, 3H), 7.28-7.24 (m, 1H), 7.09 (d, *J* = 7.5 Hz, 1H), 6.90 (d, *J* = 7.5 Hz, 1H), 3.33 (m, 3H); ¹³C NMR (125 MHz) δ: 169.2, 163.5, 140.8, 139.6, 131.1, 130.3, 129.3, 127.4, 122.4, 121.8, 118.6, 108.4, 108.4, 25.7; IR (KBr, cm⁻¹): 1679, 1622; LRMS (EI, 70 eV) *m/z* (%): 314 (M⁺+2, 8), 312

(M⁺, 26), 105 (100), 77 (34); HRMS *m/z* (EI) calcd for C₁₇H₁₃ClN₂O₂ (M⁺) 312.0666, found 312.0663.



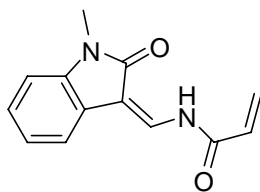
(Z)-2-Bromo-N-((1-methyl-2-oxoindolin-3-ylidene)methyl)benzamide (14):

Yellow solid, mp 182.6-184.3 °C (uncorrected); ¹H NMR (500 MHz) δ: 11.58 (d, *J* = 10.5 Hz, 1H), 8.28 (d, *J* = 11.0 Hz, 1H), 7.69-7.66 (m, 2H), 7.47-7.42 (m, 2H), 7.39-7.25 (m, 1H), 7.09 (t, *J* = 7.5 Hz, 1H), 6.88 (d, *J* = 7.5 Hz, 1H), 3.28 (s, 3H); ¹³C NMR (125 MHz) δ: 168.6, 165.5, 140.9, 135.5, 134.1, 132.5, 129.9, 127.7, 127.5, 122.3, 121.8, 120.0, 118.6, 108.8, 108.3, 25.7; IR (KBr, cm⁻¹): 1683, 1634; LRMS (EI, 70 eV) *m/z* (%): 356 (M⁺, 58), 358 (58) 183 (100), 155 (20); HRMS *m/z* (EI) calcd for C₁₇H₁₃BrN₂O₂ (M⁺) 356.0160, found 356.0164.



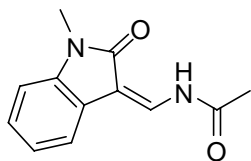
(Z)-5-Methyl-N-((1-methyl-2-oxoindolin-3-ylidene)methyl)picolinamide (15):

Yellow solid, mp 191.6-193.0 °C (uncorrected); ¹H NMR (500 MHz) δ: 12.20 (d, *J* = 10.5 Hz, 1H), 9.19 (d, *J* = 7.5 Hz, 1H), 8.32 (d, *J* = 7.5 Hz, 1H), 8.18-8.16 (m, 1H), 7.48 (d, *J* = 7.5 Hz, 1H), 7.32-7.25 (m, 1H), 7.11-7.08 (m, 1H), 6.90 (d, *J* = 8.0 Hz, 1H), 3.33 (s, 3H), 2.67 (s, 3H); ¹³C NMR (125 MHz) δ: 169.1, 163.5, 163.2, 149.2, 140.8, 135.3, 130.7, 127.5, 124.9, 123.2, 122.4, 121.7, 118.7, 108.6, 108.4, 25.8, 24.8; IR (KBr, cm⁻¹): 1687, 1634; LRMS (EI, 70 eV) *m/z* (%): 293 (M⁺, 48), 120 (100), 92 (38); HRMS *m/z* (EI) calcd for C₁₇H₁₅N₃O₂ (M⁺) 293.1164, found 293.1161.



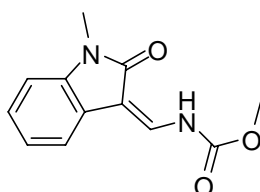
(Z)-N-((1-Methyl-2-oxoindolin-3-ylidene)methyl)acrylamide (16):

Yellow solid, mp 172.7-174.3 °C (uncorrected); ¹H NMR (500 MHz) δ: 11.28 (d, *J* = 9.5 Hz, 1H), 8.20 (d, *J* = 11.0 Hz, 1H), 7.44 (d, *J* = 7.5 Hz, 1H), 7.27-7.24 (m, 1H), 7.08 (t, *J* = 7.5 Hz, 1H), 6.88 (d, *J* = 8.0 Hz, 1H), 6.53 (d, *J* = 17.5 Hz, 1H), 6.36-5.94 (m, 1H), 5.93 (d, *J* = 10.5 Hz, 1H), 3.30 (s, 3H); ¹³C NMR (125 MHz) δ: 168.9, 163.4, 140.9, 130.6, 130.2, 129.8, 127.4, 122.3, 121.9, 118.6, 108.4, 108.1, 25.7; IR (KBr, cm⁻¹): 1687, 1634; LRMS (EI, 70 eV) *m/z* (%): 228 (M⁺, 63), 174 (100), 55 (62); HRMS *m/z* (EI) calcd for C₁₃H₁₂N₂O₂ (M⁺) 228.0899, found 228.0896.



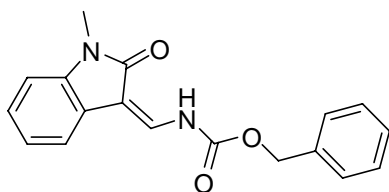
(Z)-N-((1-Methyl-2-oxoindolin-3-ylidene)methyl)acetamide (17):²

Yellow solid, mp 181.6-183.2 °C (uncorrected); ¹H NMR (500 MHz) δ: 11.02 (d, *J* = 9.0 Hz, 1H), 8.10 (d, *J* = 11.0 Hz, 1H), 7.24 (t, *J* = 7.5 Hz, 1H), 7.06 (t, *J* = 7.5 Hz, 1H), 6.87 (d, *J* = 8.0 Hz, 1H), 3.29 (s, 3H), 2.25 (s, 3H); ¹³C NMR (125 MHz) δ: 168.8, 168.6, 140.8, 130.5, 127.2, 122.2, 122.0, 118.4, 108.3, 107.3, 25.7, 23.7; IR (KBr, cm⁻¹): 1687, 1626; LRMS (EI, 70 eV) *m/z* (%): 216 (M⁺, 41), 174 (100), 158 (38).



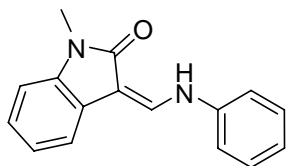
(Z)-Methyl-N-((1-methyl-2-oxoindolin-3-ylidene)methyl)carbamate (18):

Yellow solid, mp 150.5-152.2 °C (uncorrected); ¹H NMR (500 MHz) δ: 10.37 (s, 1H), 7.90 (d, *J* = 11.0 Hz, 1H), 7.39 (d, *J* = 7.5 Hz, 1H), 7.22 (t, *J* = 7.5 Hz, 1H), 7.50 (t, *J* = 7.5 Hz, 2H), 6.86 (d, *J* = 7.5 Hz, 1H), 3.87 (s, 3H), 3.28 (s, 3H); ¹³C NMR (125 MHz) δ: 168.5, 154.1, 140.5, 132.6, 126.8, 122.0, 121.0, 117.9, 108.1, 106.0, 53.4, 25.7; IR (KBr, cm⁻¹): 1687, 1630; LRMS (EI, 70 eV) *m/z* (%): 232 (M⁺, 95), 200 (100), 158 (44); HRMS *m/z* (EI) calcd for C₁₂H₁₂N₂O₃ (M⁺) 232.0848, found 232.0850.



(Z)-Benzyl-N-(1-methyl-2-oxoindolin-3-ylidene)methylcarbamate (19):

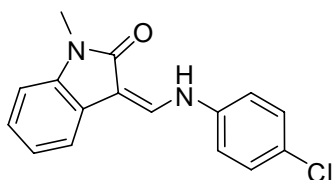
Yellow solid, mp 129.2-131.8 °C (uncorrected); ¹H NMR (500 MHz) δ: 10.43 (d, *J* = 9.5 Hz, 1H), 7.90 (d, *J* = 10.5 Hz, 1H), 7.42-7.35 (m, 6H), 7.22 (t, *J* = 7.5 Hz, 1H), 7.21 (t, *J* = 7.5 Hz, 1H), 6.85 (d, *J* = 7.5 Hz, 1H), 5.27 (s, 2H), 3.27 (m, 3H); ¹³C NMR (125 MHz) δ: 168.5, 140.5, 135.1, 132.6, 128.7, 128.6, 128.6, 128.3, 126.8, 122.0, 121.9, 117.9, 108.1, 68.2, 25.6; IR (KBr, cm⁻¹): 1683, 1636; LRMS (EI, 70 eV) *m/z* (%): 308 (M⁺, 23), 247 (11), 91 (100); HRMS *m/z* (EI) calcd for C₁₈H₁₆N₂O₃ (M⁺) 308.1161, found 308.1163.



(Z) and (E)-1-Methyl-3-((phenylamino)methylene)indolin-2-one (20):³

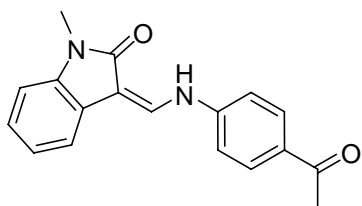
Z/E = 1:1.8; Yellow solid, mp 143.4-145.1 °C (uncorrected); ¹H NMR (500 MHz) δ: 10.70 (d, *J* = 12.0 Hz, 0.53H), 8.05 (d, *J* = 14.0 Hz, 1H), 7.98 (d, *J* = 12.5 Hz, 0.55H), 7.36 (t, *J* = 7.5 Hz, 4.51H), 7.28 (t, *J* = 12.5 Hz, 1.52H), 7.21-7.02 (m, 8.06H), 6.90-6.85 (m,

1.53H), 3.34 (s, 1.69H), 3.29 (s, 3H); ^{13}C NMR (125 MHz) δ : 169.5, 168.9, 140.7, 140.1, 139.9, 138.8, 136.4, 135.9, 129.9, 129.8, 125.7, 124.4, 123.8, 123.5, 123.4, 122.1, 121.3, 121.2, 119.2, 116.1, 115.9, 107.9, 107.8, 101.8, 25.9, 25.6; IR (KBr, cm^{-1}): 1667; LRMS (EI, 70 eV) m/z (%): 250 (M^+ , 78), 158 (61), 43 (100).



(Z)-3-((4-Chlorophenylamino)methylene)-1-methylindolin-2-one (21):³

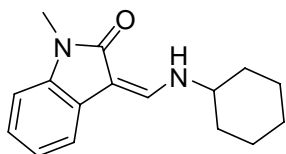
Yellow solid, mp 157.7-159.5 °C (uncorrected); ^1H NMR (500 MHz) δ : 10.72 (d, $J = 12.5$ Hz, 1H), 7.89 (d, $J = 12.5$ Hz, 1H), 7.36 (d, $J = 7.5$ Hz, 1H), 7.32 (t, $J = 8.0$ Hz, 2H), 7.16 (t, $J = 7.5$ Hz, 1H), 7.08-7.03 (m, 3H), 6.89 (d, $J = 9.0$ Hz, 1H), 3.34 (s, 3H); ^{13}C NMR (125 MHz) δ : 169.0, 138.9, 138.6, 135.8, 129.8, 124.7, 123.1, 121.1, 117.0, 116.1, 107.9, 100.3, 25.6; IR (KBr, cm^{-1}): 1674; LRMS (EI, 70 eV) m/z (%): 286 ($\text{M}^+ + 2$, 20), 284 (M^+ , 61), 158 (59), 43 (100).



(Z)-3-((4-Acetylphenylamino)methylene)-1-methylindolin-2-one (22):

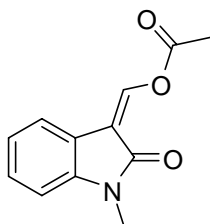
Yellow solid, mp 219.4-221.1 °C (uncorrected); ^1H NMR (500 MHz) δ : 10.90 (d, $J = 12.5$ Hz, 1H), 7.98 (t, $J = 9.0$ Hz, 3H), 7.40 (d, $J = 7.5$ Hz, 1H), 7.21-7.15 (m, 3H), 7.06 (t, $J = 7.5$ Hz, 1H), 6.90 (d, $J = 7.5$ Hz, 1H), 3.34 (s, 3H), 2.58 (s, 3H); ^{13}C NMR (125 MHz) δ : 196.4, 168.9, 143.8, 139.3, 134.3, 131.9, 130.6, 125.3, 121.6, 116.6, 114.9, 108.1, 101.9,

26.3, 25.6; IR (KBr, cm^{-1}): 1667, 1597; LRMS (EI, 70 eV) m/z (%): 292 (M^+ , 36), 105 (100), 77 (34); HRMS m/z (EI) calcd for $\text{C}_{18}\text{H}_{16}\text{N}_2\text{O}_2$ (M^+) 292.1212, found 292.1210.



(Z)-3-((Cyclohexylamino)methylene)-1-methylindolin-2-one (23):

Yellow solid, ^1H NMR (500 MHz) δ : 8.82 (s, 1H), 7.50 (d, $J = 13.5$ Hz, 1H), 7.24 (d, $J = 7.5$ Hz, 1H), 7.07 (t, $J = 7.5$ Hz, 1H), 6.99 (t, $J = 7.5$ Hz, 1H), 6.87 (d, $J = 7.5$ Hz, 1H), 3.33 (s, 2H), 2.00 (d, $J = 5.0$ Hz, 2H), 1.83-1.81 (m, 2H), 1.47-1.34 (m, 7H); ^{13}C NMR (125 MHz) δ : 168.9, 144.6, 137.5, 124.3, 122.6, 120.7, 114.6, 107.5, 94.8, 57.4, 34.1, 25.2, 24.5; IR (KBr, cm^{-1}): 1659; LRMS (EI, 70 eV) m/z (%): 256 (M^+ , 96), 158 (27), 43 (100); HRMS m/z (EI) calcd for $\text{C}_{16}\text{H}_{20}\text{N}_2\text{O}$ (M^+) 256.1576, found 256.1573.



(Z)-(1-Methyl-2-oxoindolin-3-ylidene)methyl acetate (24):

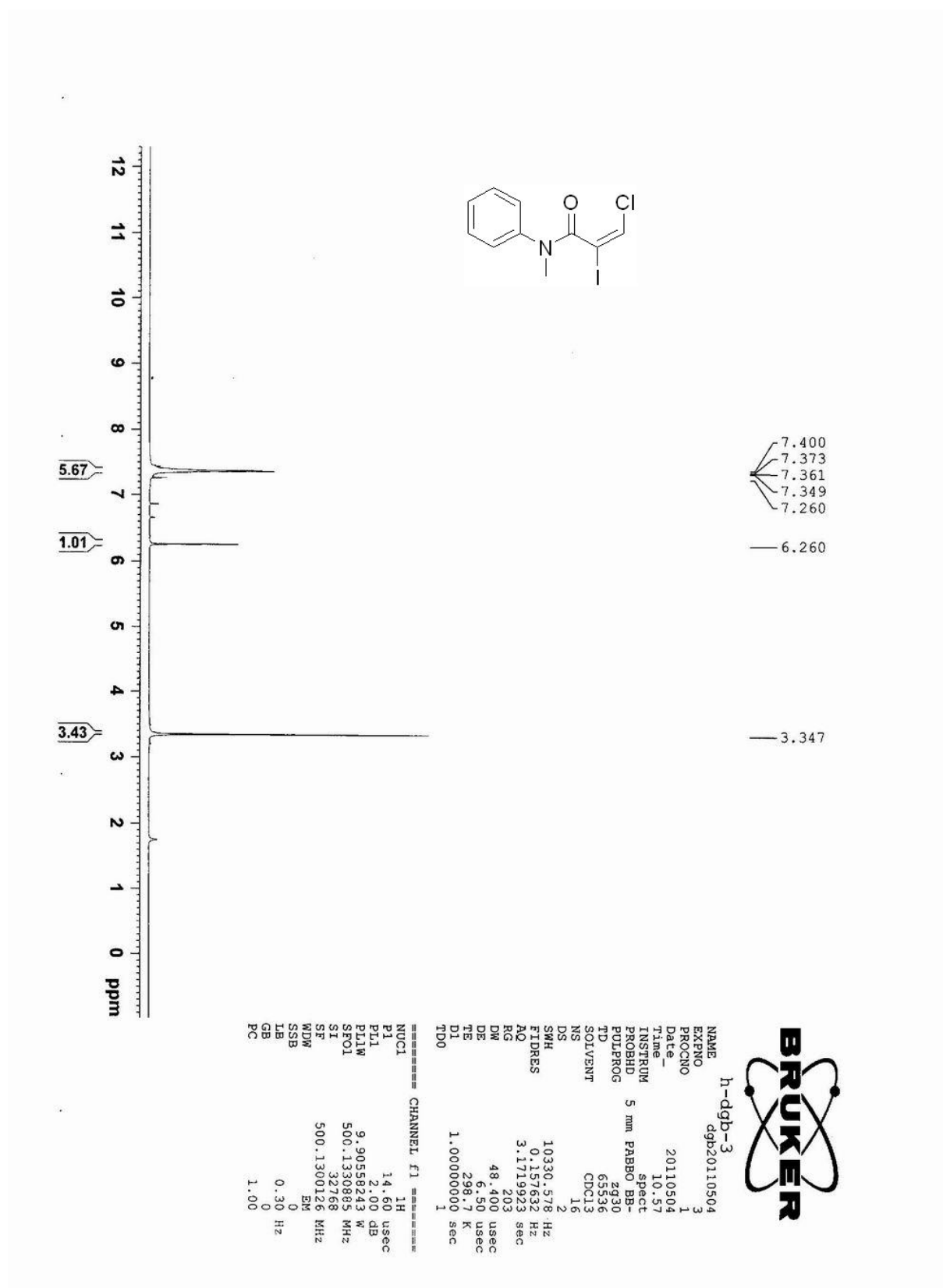
Yellow solid; mp 152.2-153.7 $^{\circ}\text{C}$ (uncorrected); ^1H NMR (500 MHz, CDCl_3) δ : 8.46 (s, 1H), 7.67 (d, $J = 7.5$ Hz, 1H), 7.29 (t, $J = 7.5$ Hz, 1H), 7.06 (t, $J = 7.5$ Hz, 1H), 6.82 (d, $J = 7.5$ Hz, 1H), 3.24 (s, 3H), 2.40 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ : 168.3, 166.3, 142.7, 141.4, 129.1, 124.1, 122.2, 120.2, 113.1, 108.0, 26.0, 20.7; LRMS (EI, 70 eV) m/z (%): 217 (M^+ , 6), 175 (100), 118 (60), 44 (57); HRMS m/z (EI) calcd for $\text{C}_{12}\text{H}_{11}\text{NO}_3$ (M^+) 217.0739, found 217.0734.

(C) References

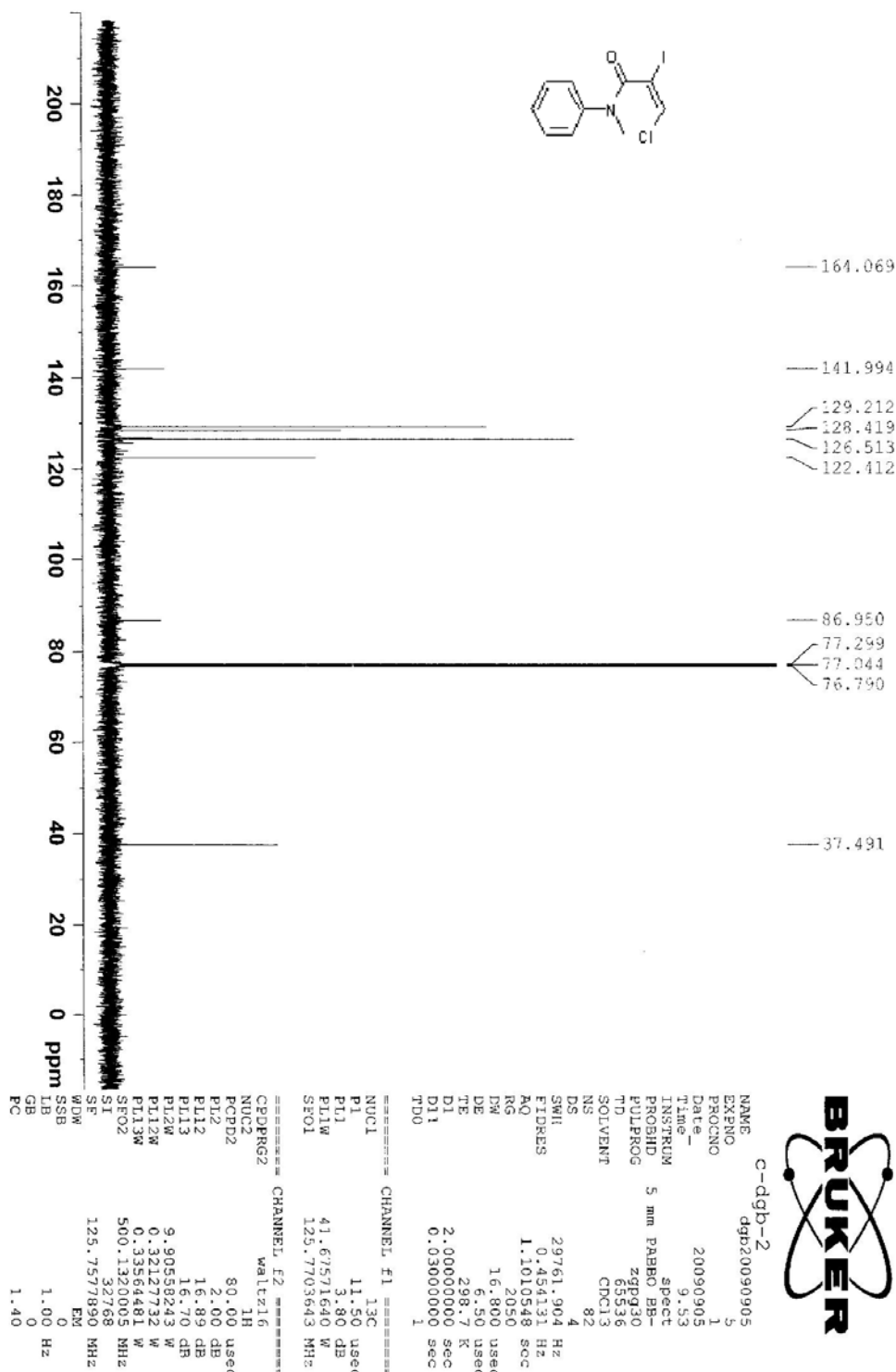
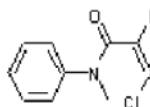
- (1) The stereochemistry of the products was assigned on the basis of the chemical shift of the olefinic proton according to the authoritative ^1H NMR spectra, see: Jiang, H.; Ma, S.; Zhu, G.; Lu, X. *Tetrahedron* **1996**, *52*, 10945.
- (2) (a) Long, D. R.; Richards, C. G.; Ross, M. S. F. *J. Heterocycl. Chem.* **1978**, *15*, 633. (b) Pedras, M. S. C.; Zaharia, I. L. *Org. Lett.* **2001**, *3*, 1213.
- (3) Corsico Coda, A.; Desimoni, G.; Quadrelli, P.; Righetti, P. P.; Tacconi, G. *Gazzetta Chimica Italiana* **1987**, *117*, 301 (*Chem. Abstr.* CAN 107:189491).

(D) Spectra

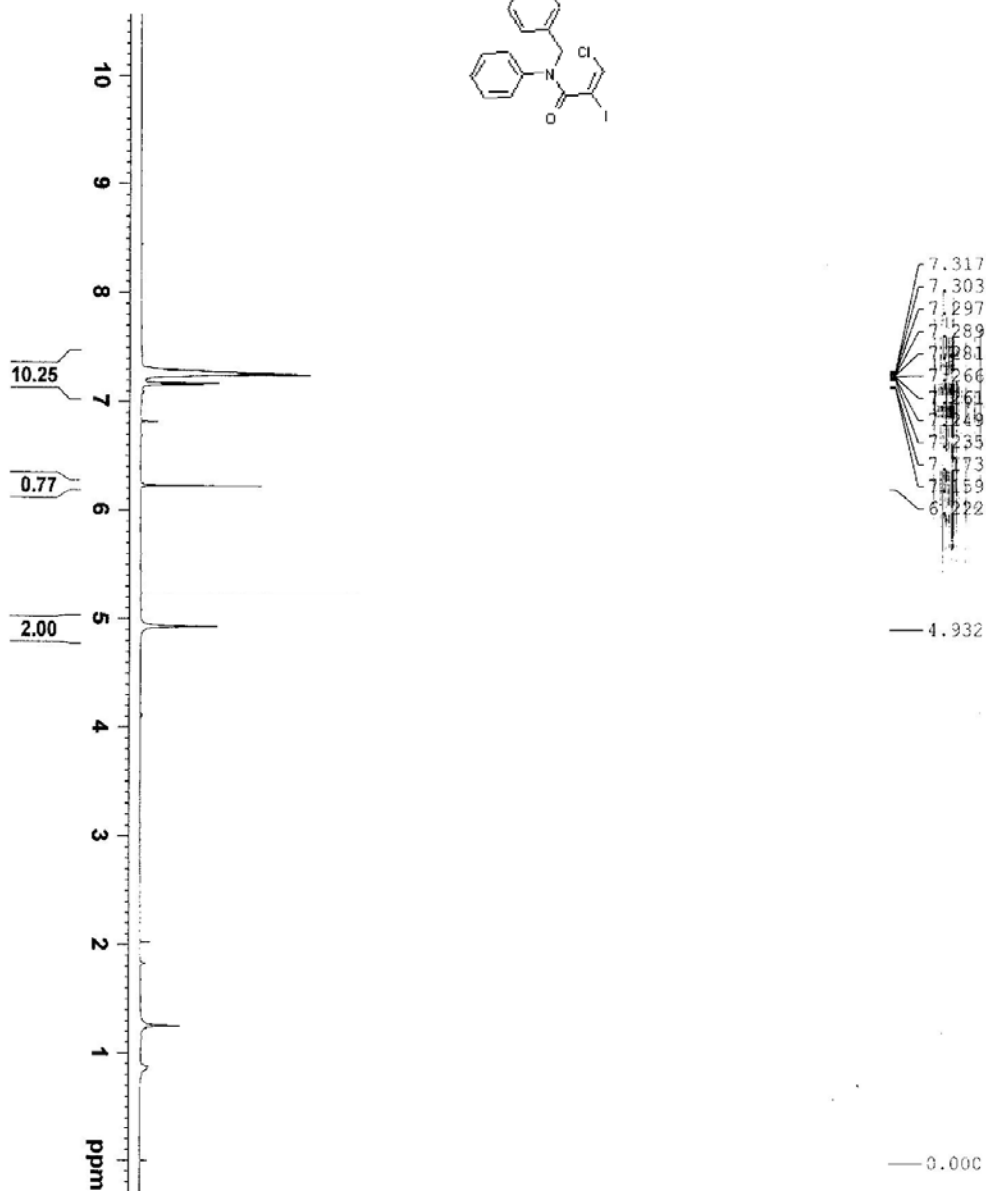
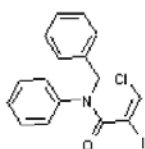
3-Chloro-2-iodo-N-methyl-N-phenylacrylamide (1a)



3-Chloro-2-iodo-N-methyl-N-phenylacrylamide (1a)



***N*-Benzyl-3-chloro-2-iodo-*N*-phenylacrylamide (1b)**

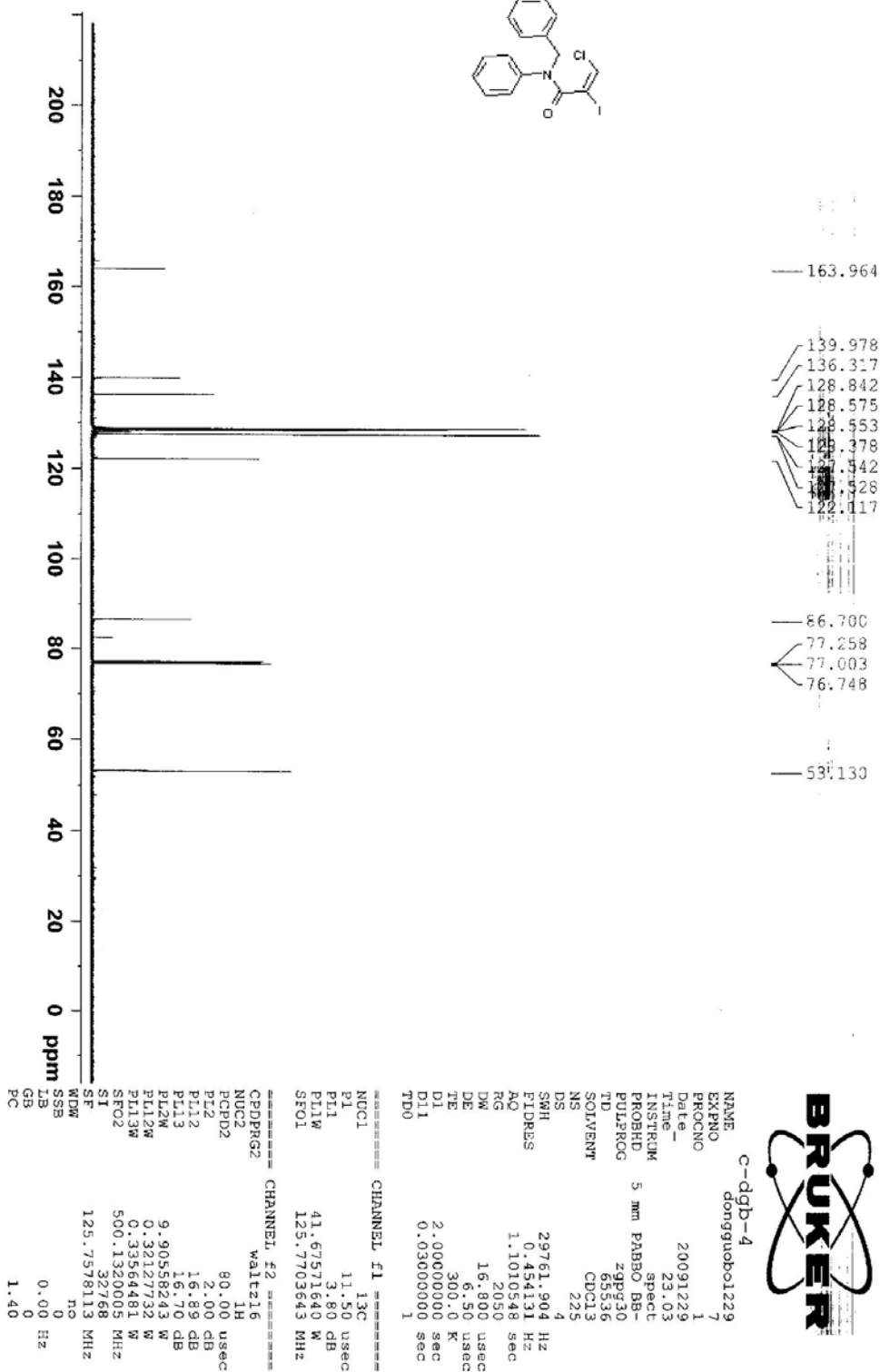
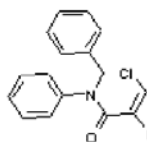


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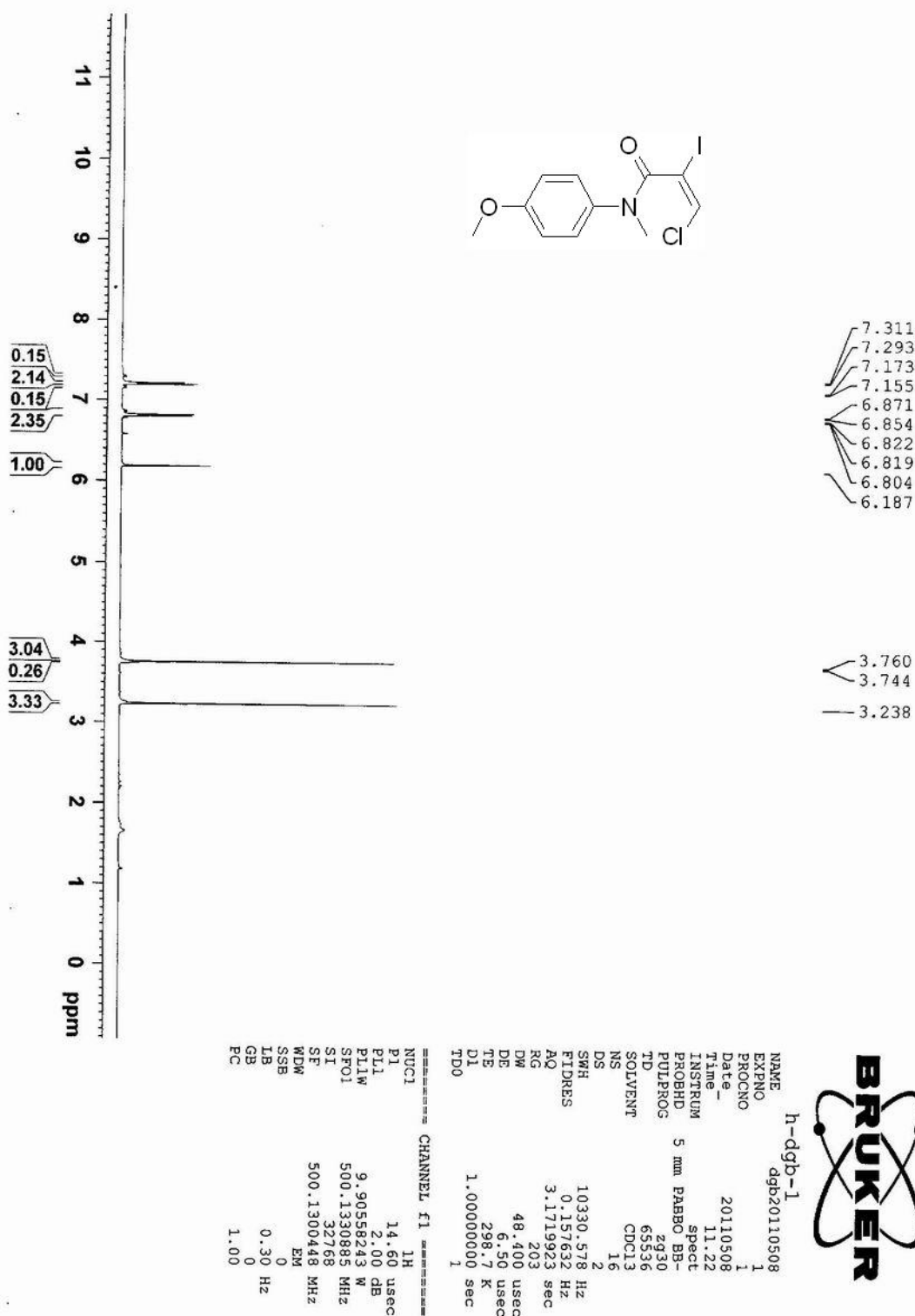
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AQ 3.171923 sec
RG 512
DE 48.400 usec
TE 300.0 K
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TD0 1

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PC 1.00
    
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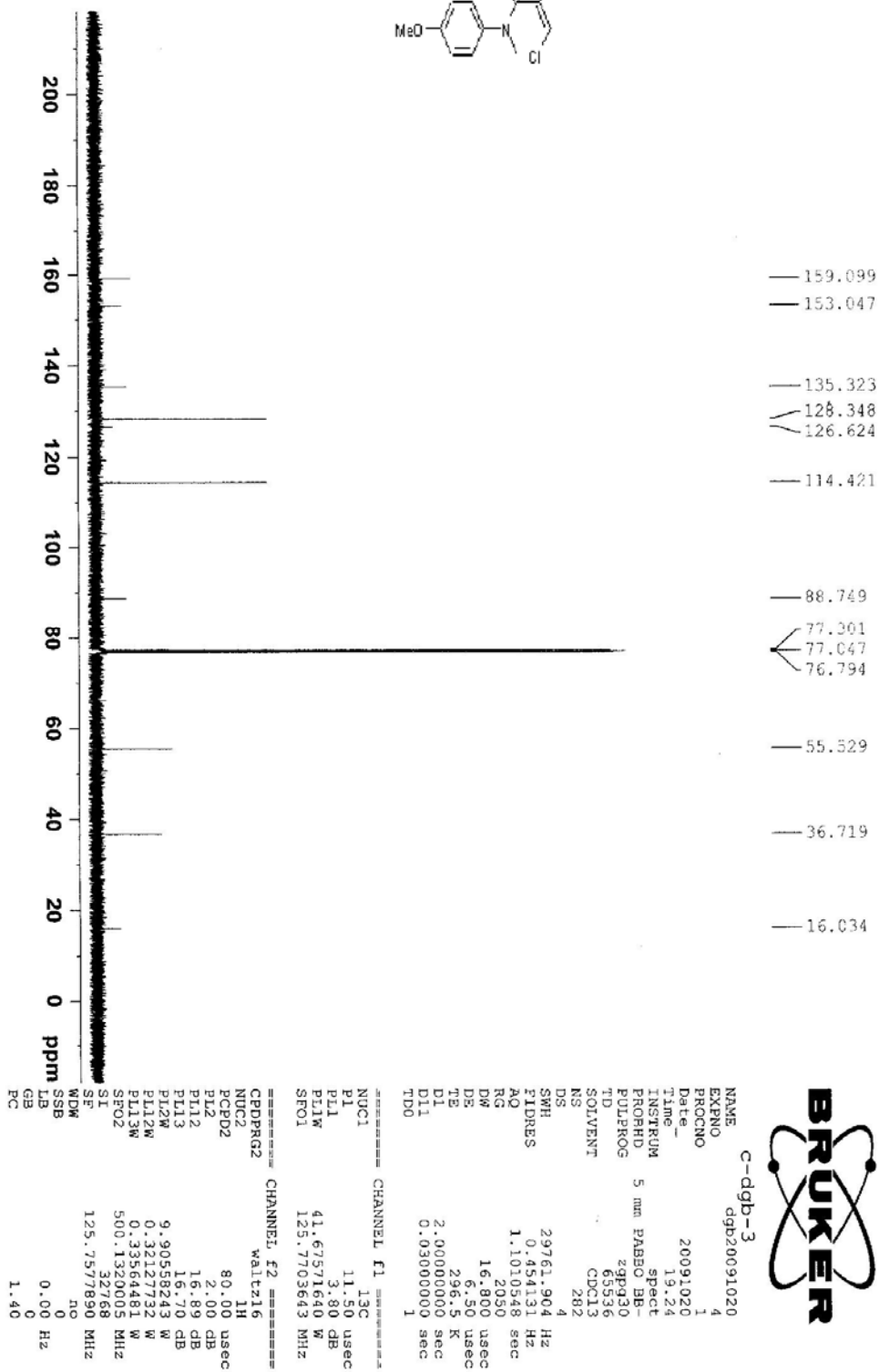
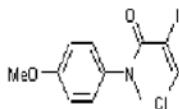
***N*-Benzyl-3-chloro-2-iodo-*N*-phenylacrylamide (1b)**



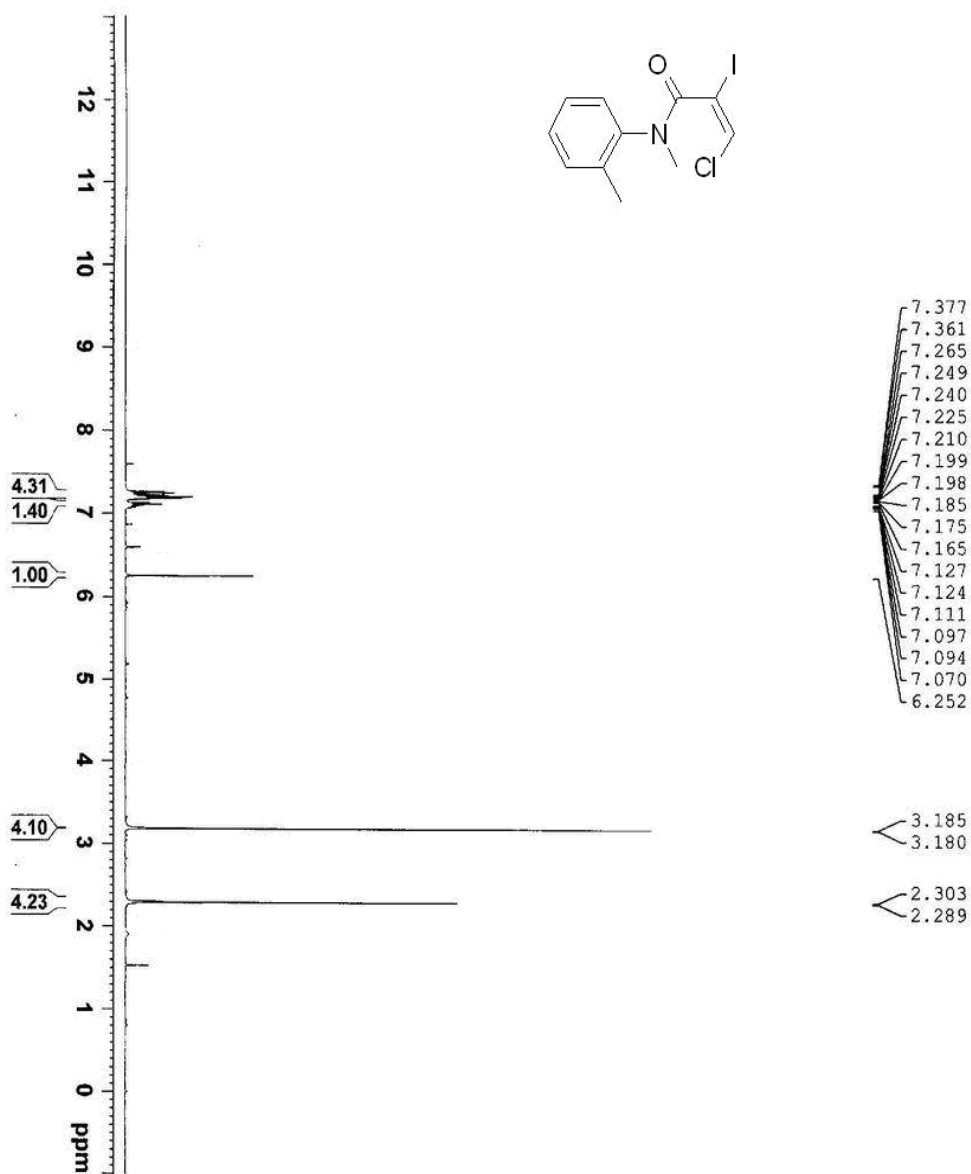
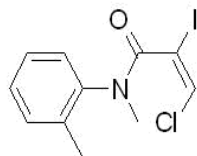
3-Chloro-2-iodo-N-(4-methoxyphenyl)-N-methylacrylamide (1c)



3-Chloro-2-iodo-N-(4-methoxyphenyl)-N-methylacrylamide (1c)



3-Chloro-2-iodo-N-methyl-N-o-tolylacrylamide (1d)

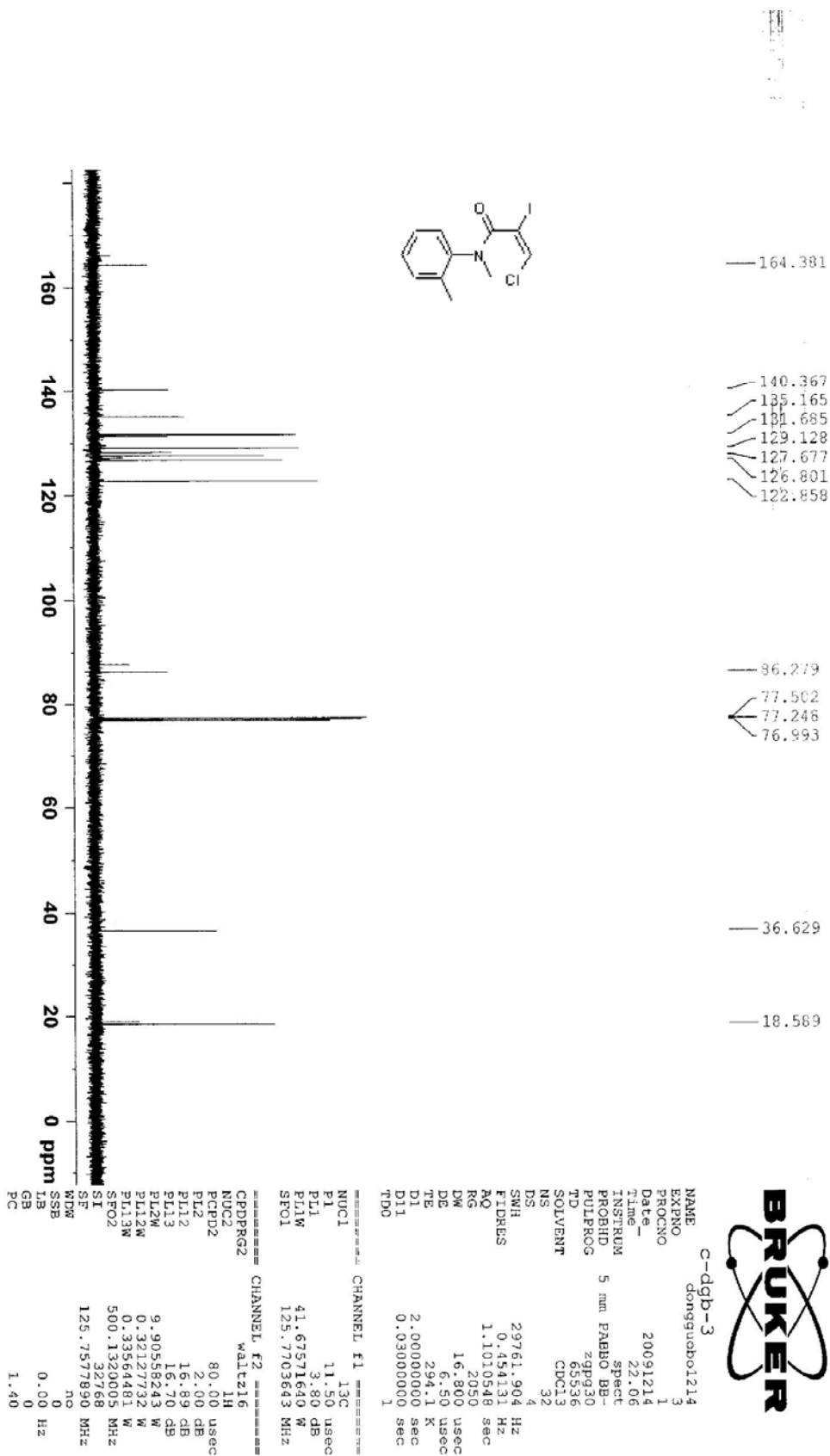
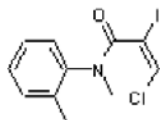


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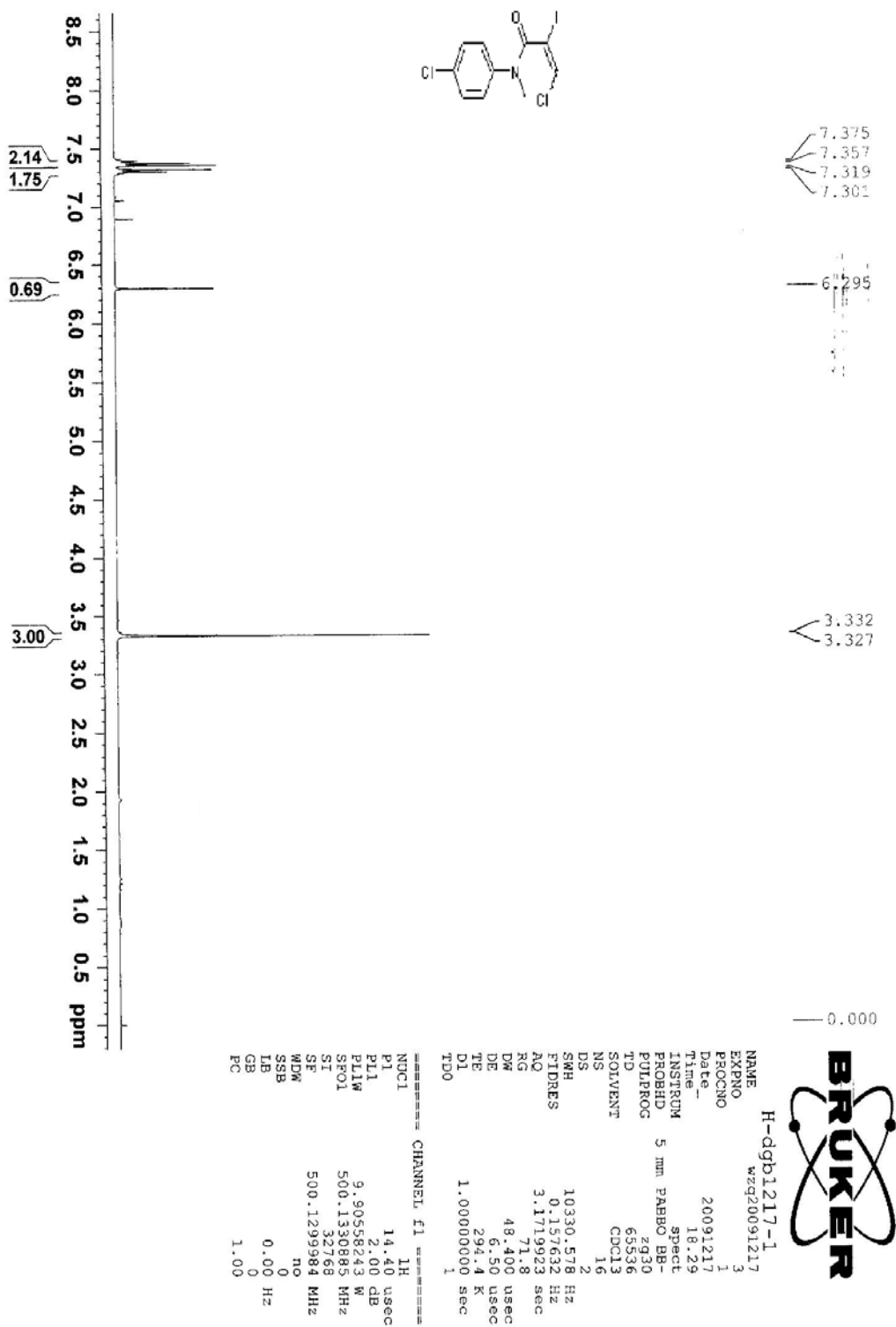
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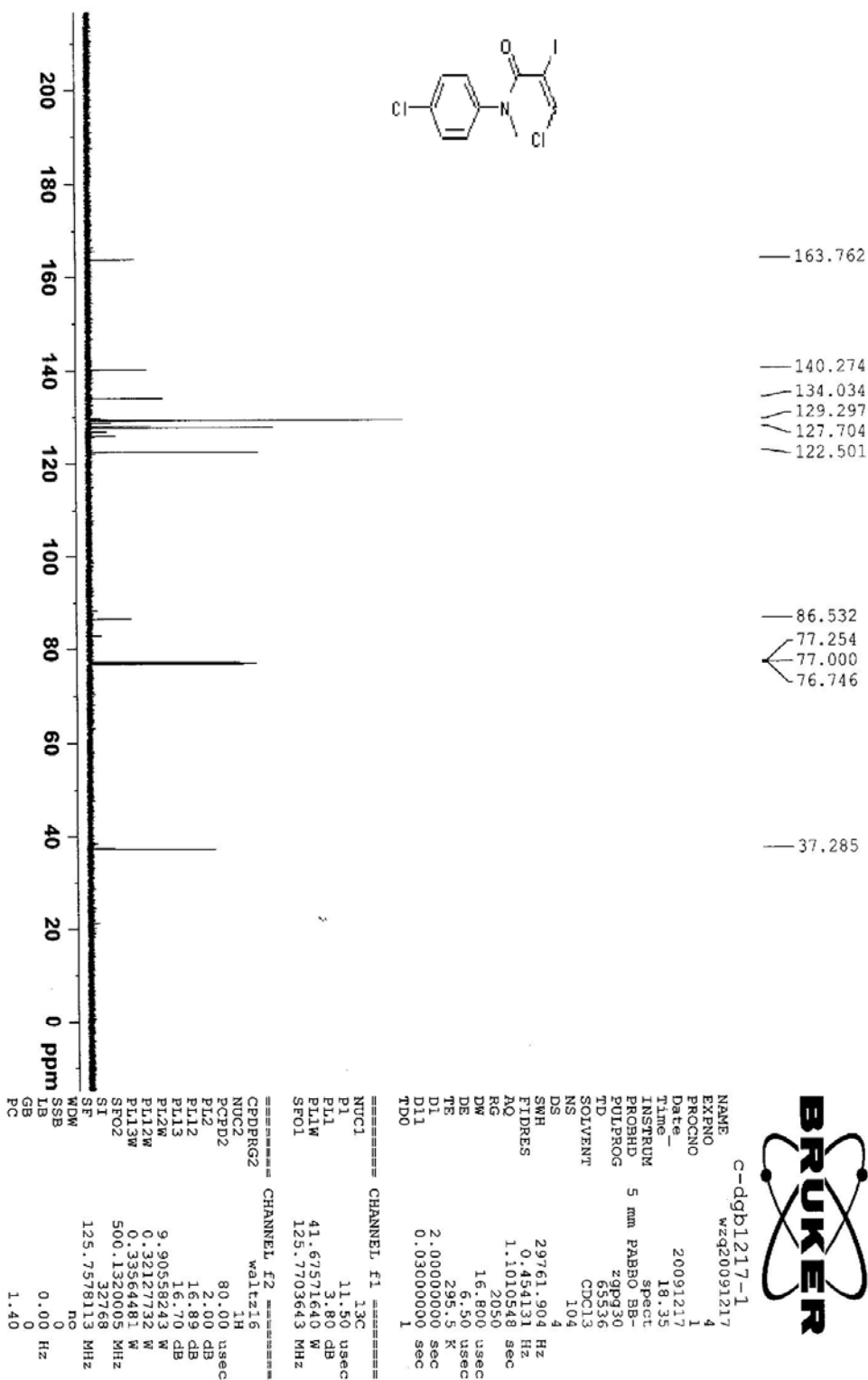
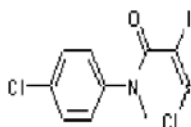
3-Chloro-2-iodo-N-methyl-N-o-tolylacrylamide (1d)



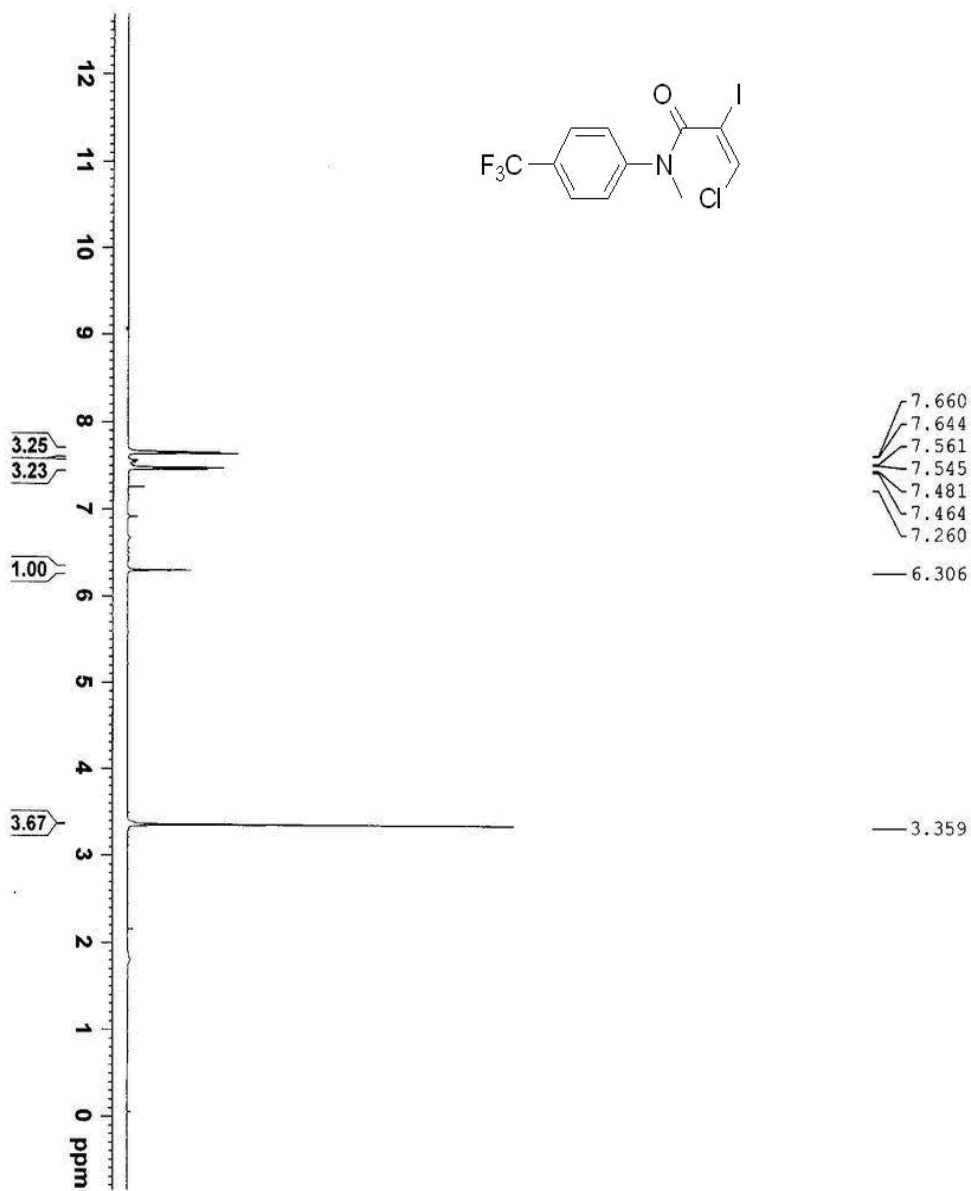
3-Chloro-N-(4-chlorophenyl)-2-iodo-N-methylacrylamide (1e)



3-Chloro-N-(4-chlorophenyl)-2-iodo-N-methylacrylamide (1e)



3-Chloro-2-iodo-N-methyl-N-(4-(trifluoromethyl)phenyl)acrylamide (1f)

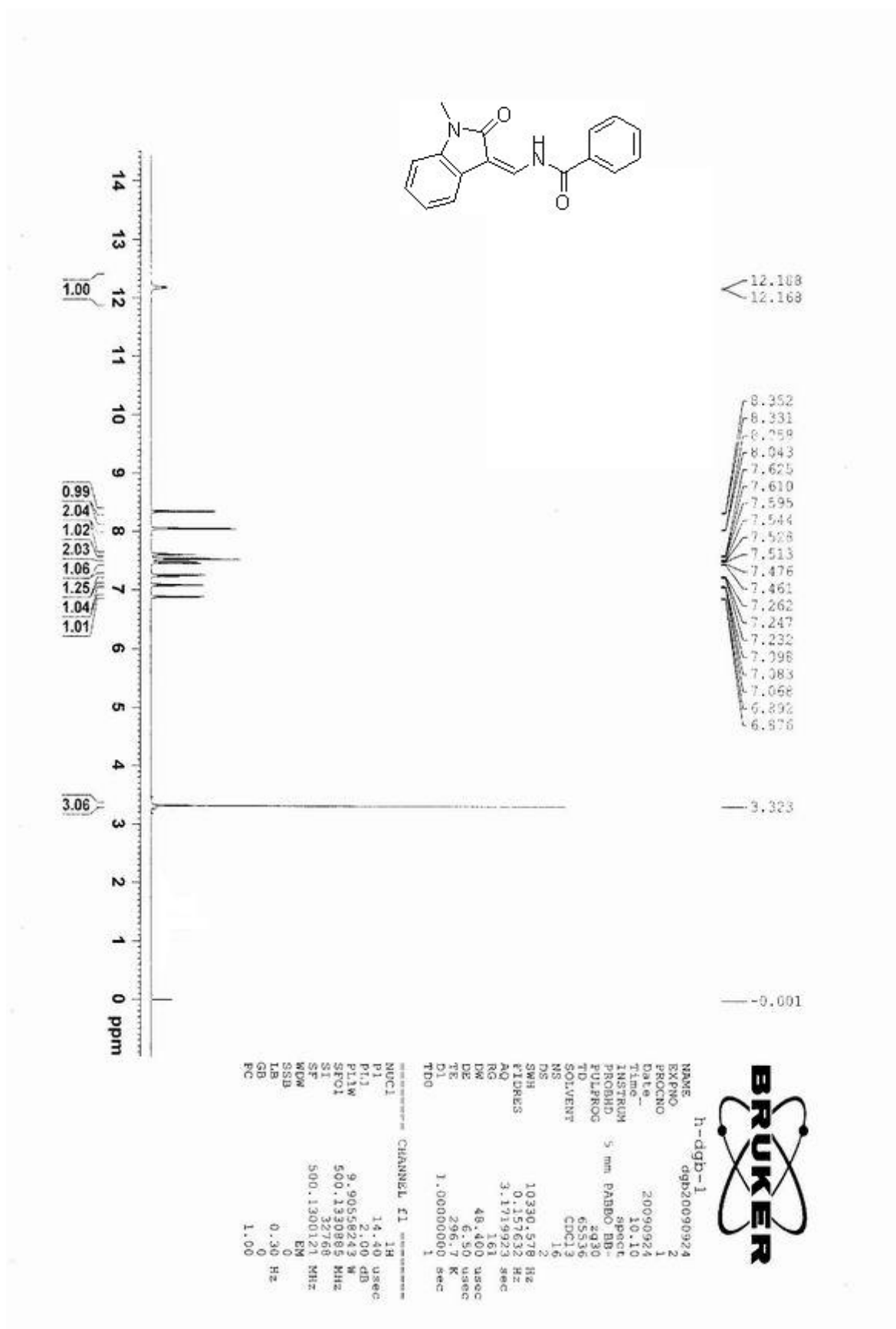


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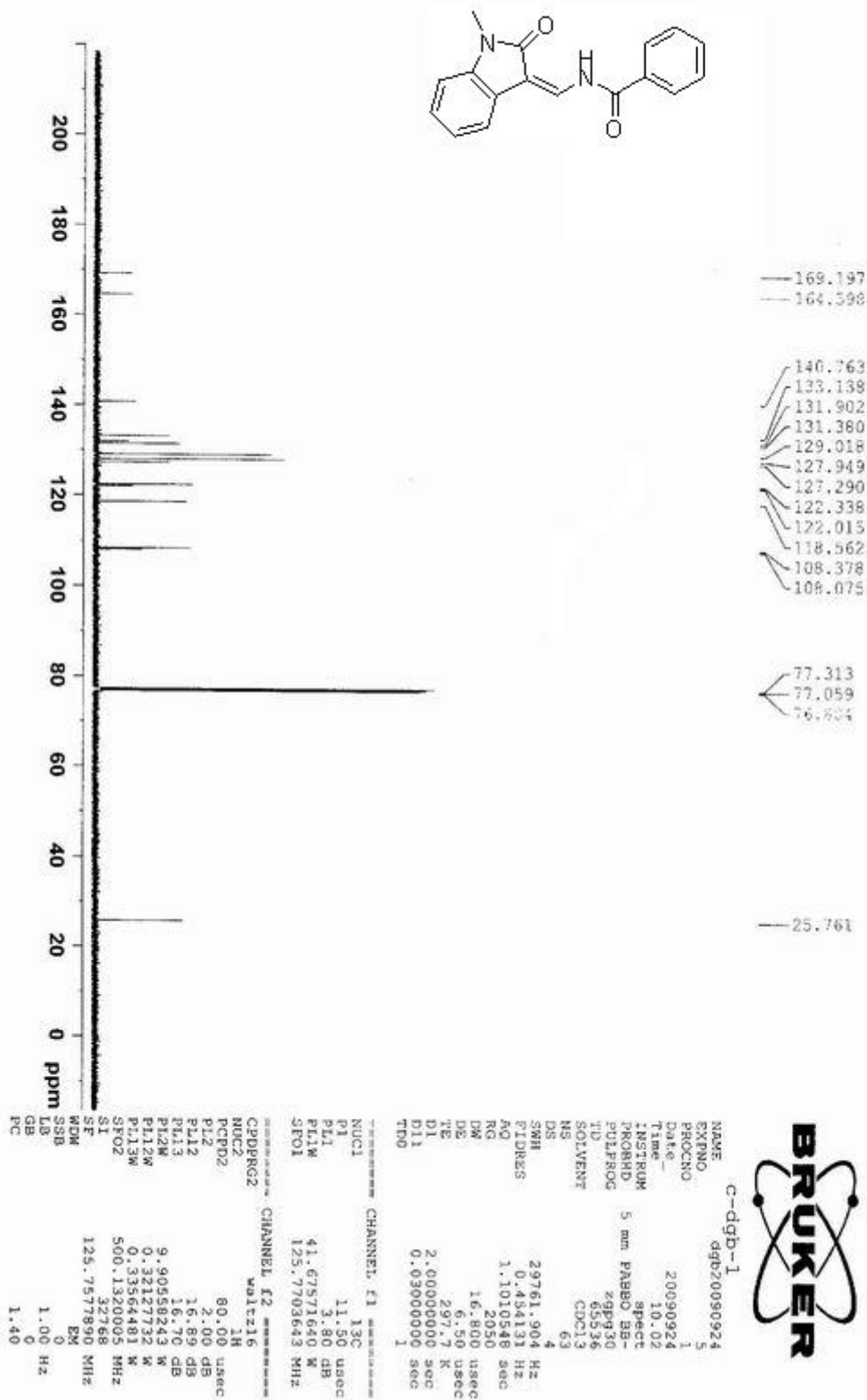
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PROCNO 3
Date_ 20110508
Time_ 11.33
INSTRUM spect
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TD 65536
SOLVENT CDCl3
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DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 128
DE 48.400 usec
TE 298.4 K
D1 1.00000000 Sec
TDO 1

===== CHANNEL f1 =====
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P1 14.60 usec
PL1 2.00 dB
PL1W 9.90558243 W
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PC 1.00
    
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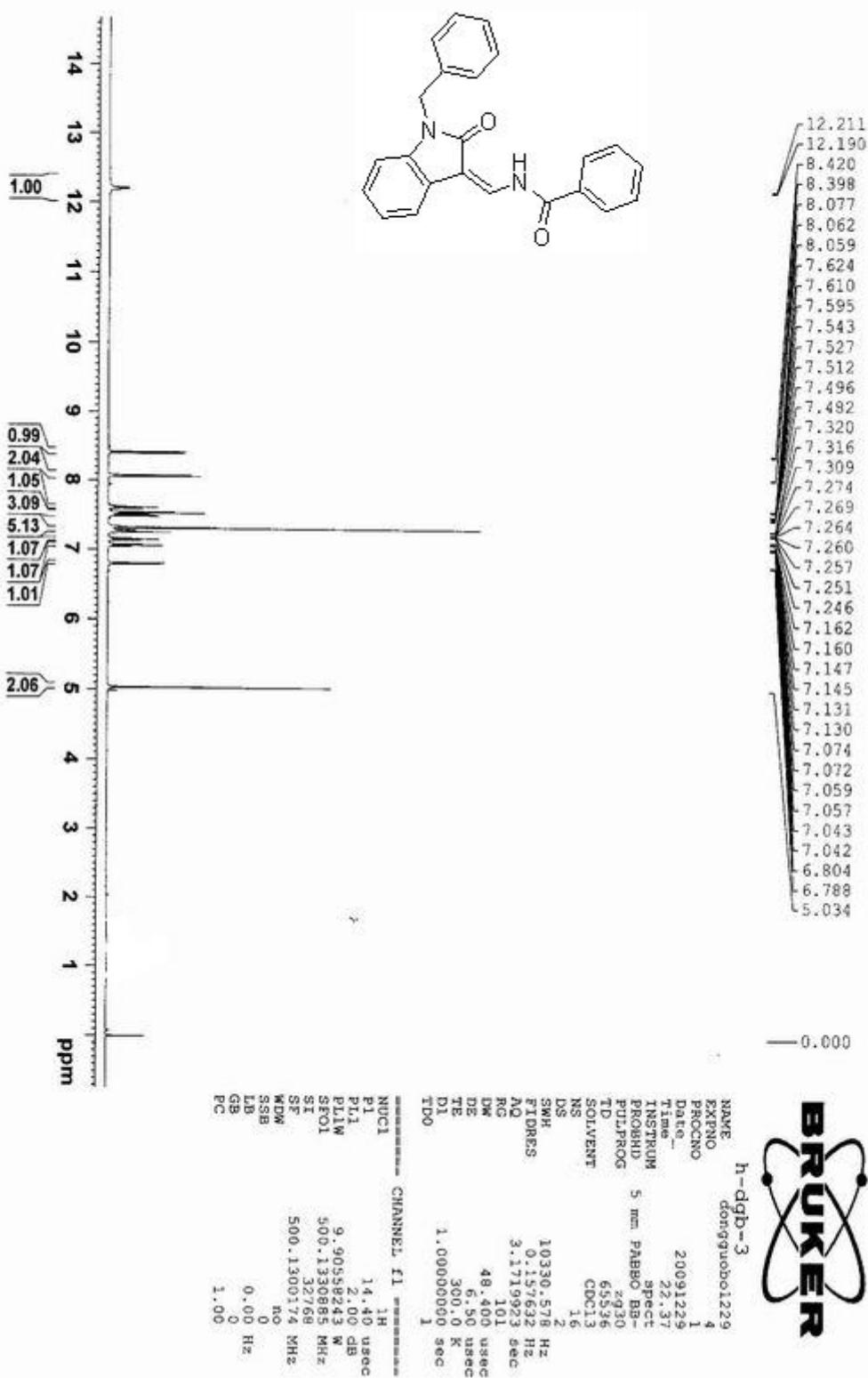

(Z)-N-((1-Methyl-2-oxoindolin-3-ylidene)methyl)benzamide (3)



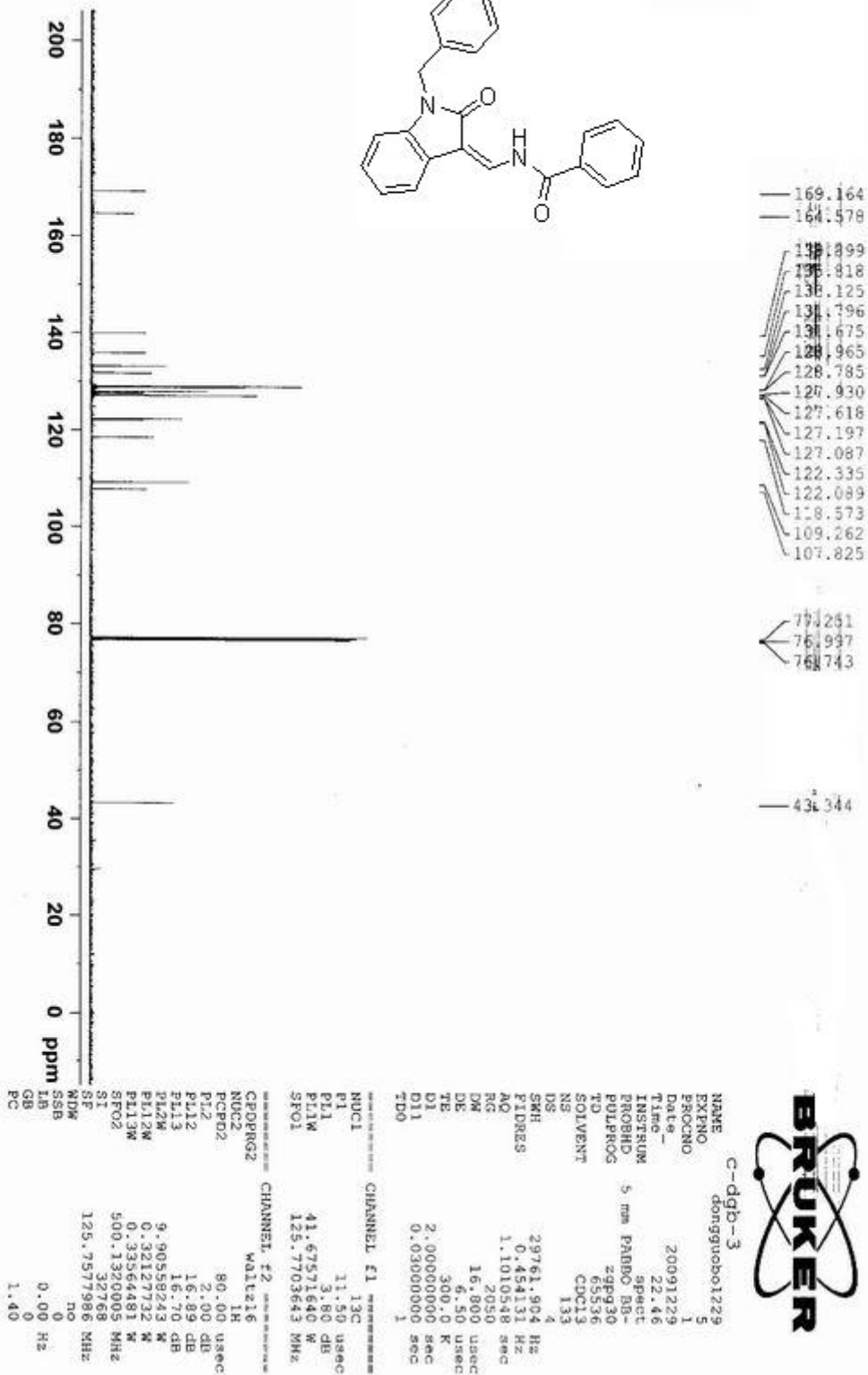
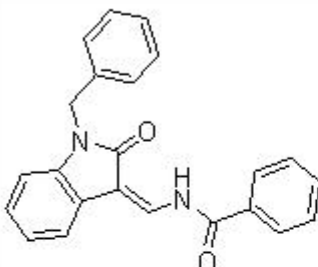
(Z)-N-((1-Methyl-2-oxindolin-3-ylidene)methyl)benzamide (3)



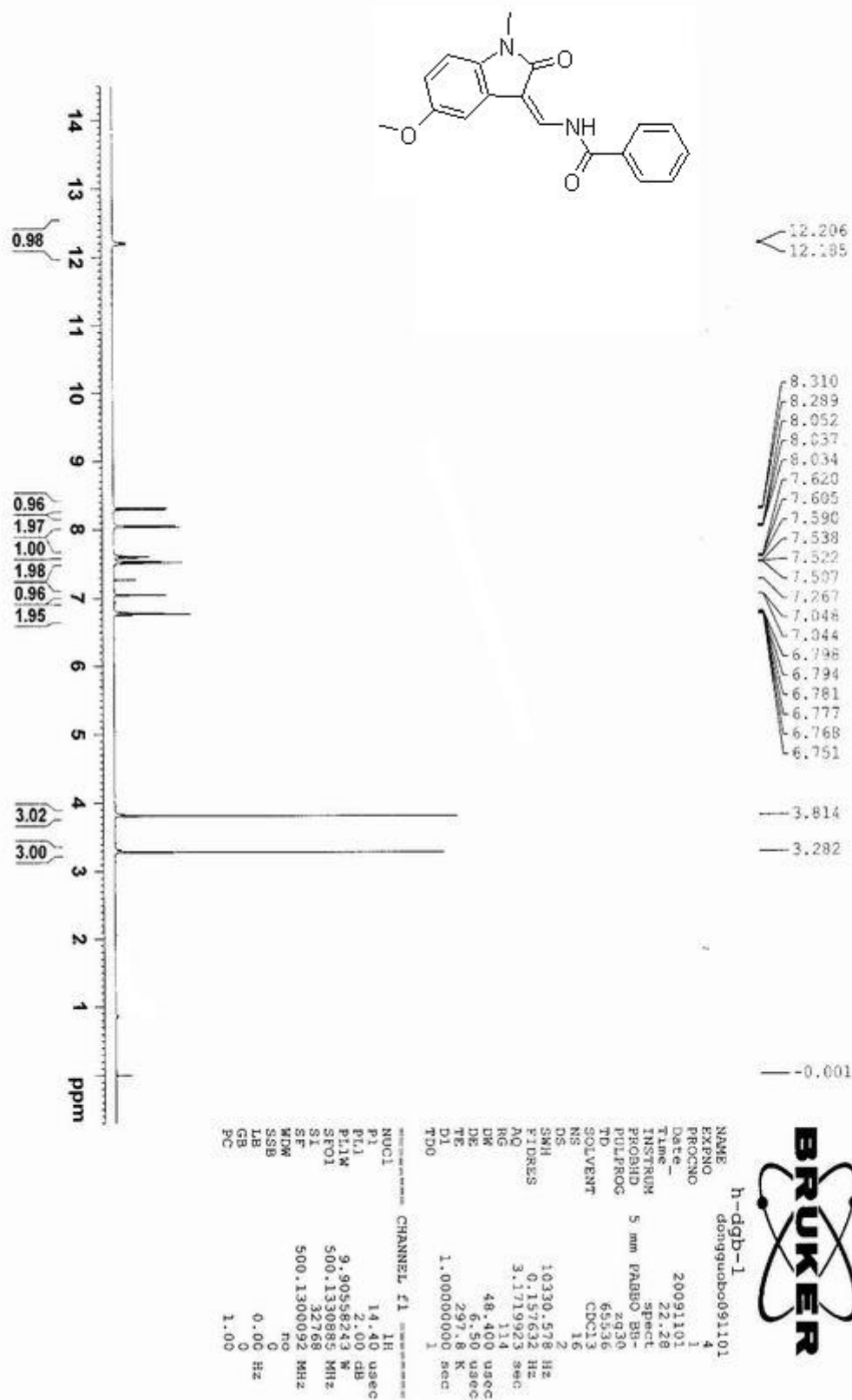
(Z)-N-((1-Benzyl-2-oxindolin-3-ylidene)methyl)benzamide (4)



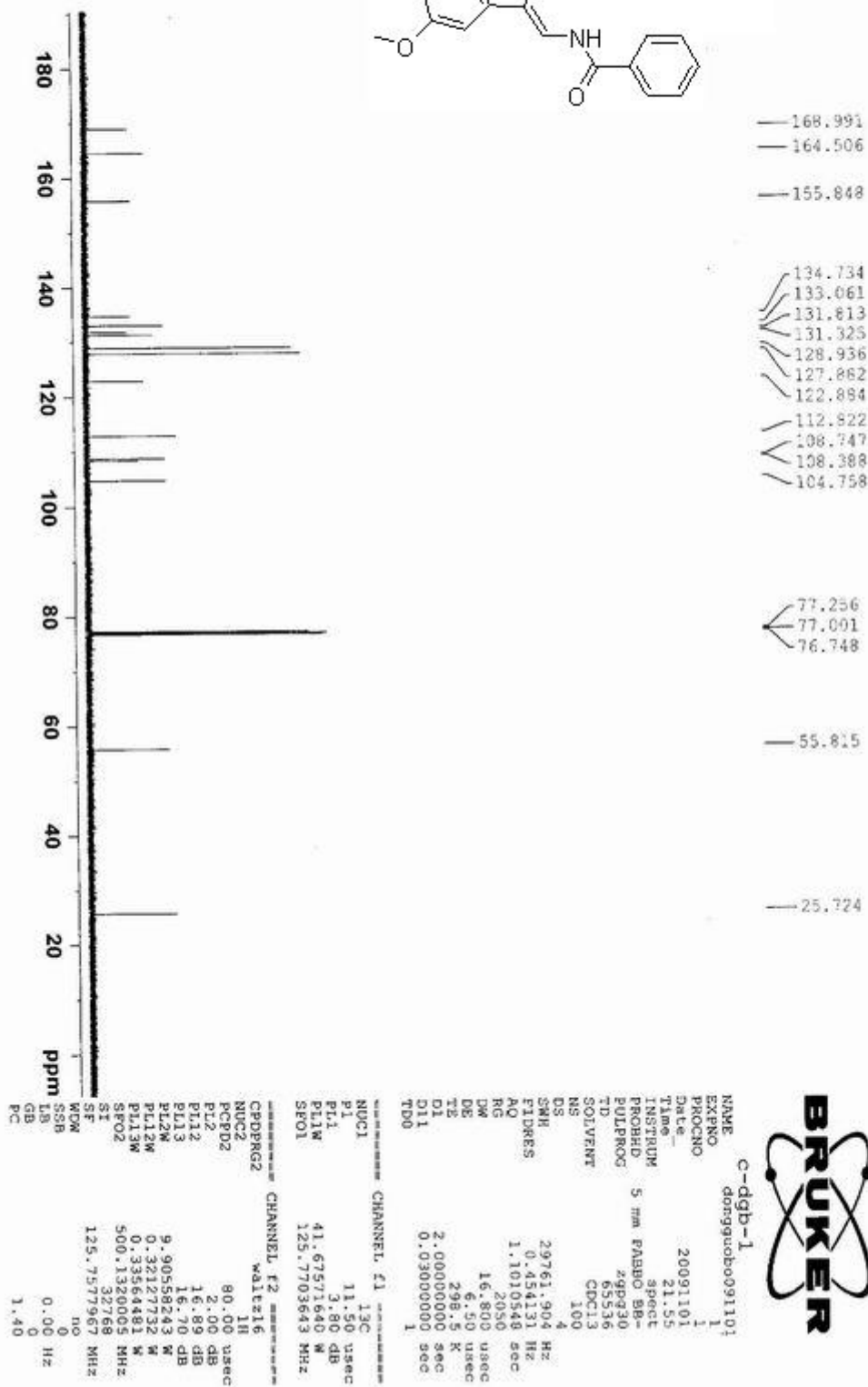
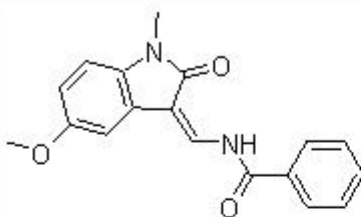
(Z)-N-((1-Benzyl-2-oxindolin-3-ylidene)methyl)benzamide (4)



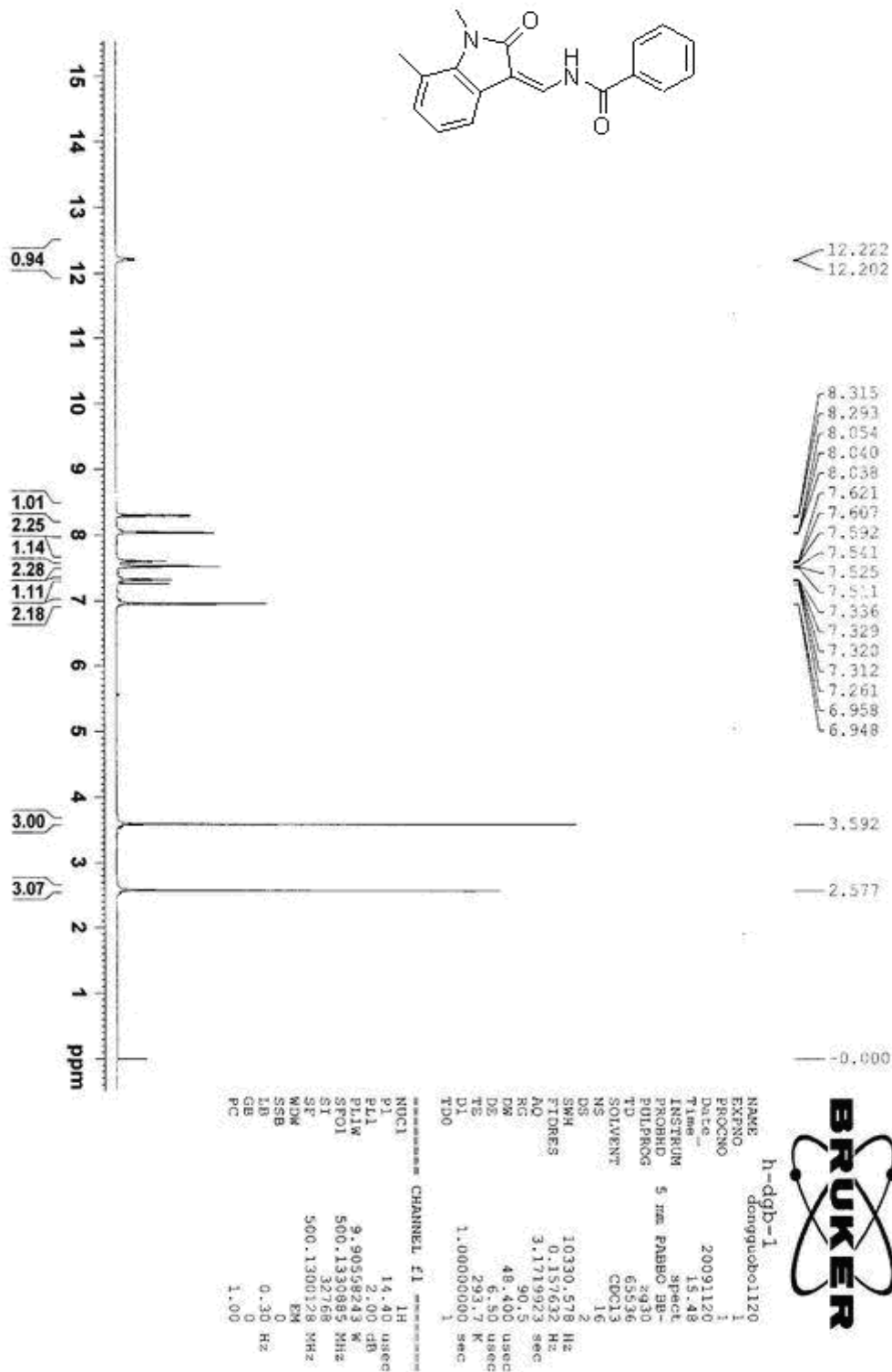
(Z)-N-((5-Methoxy-1-methyl-2-oxindolin-3-ylidene)methyl)benzamide (5)



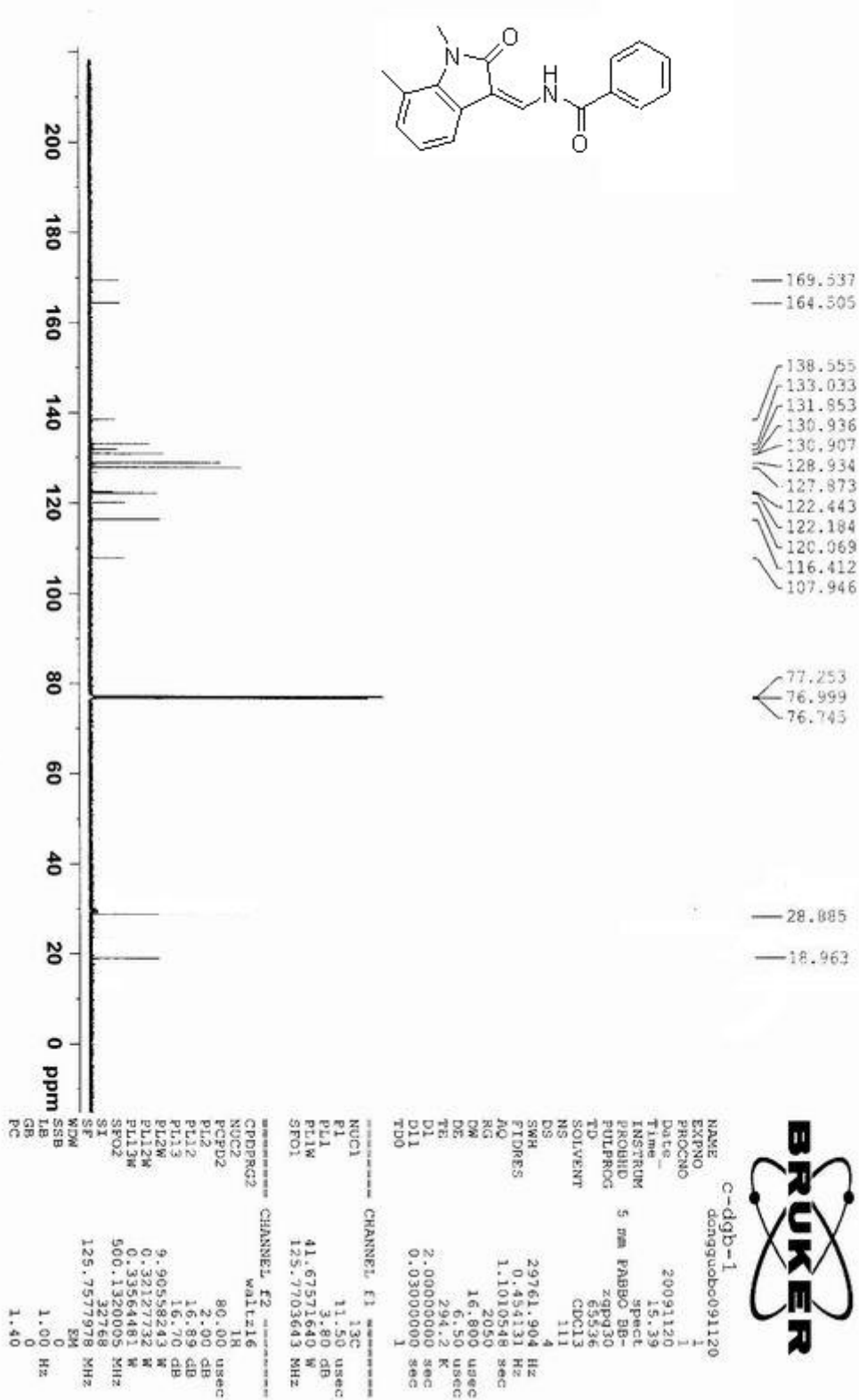
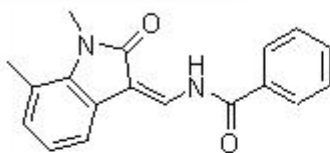
(Z)-N-((5-Methoxy-1-methyl-2-oxindolin-3-ylidene)methyl)benzamide (5)



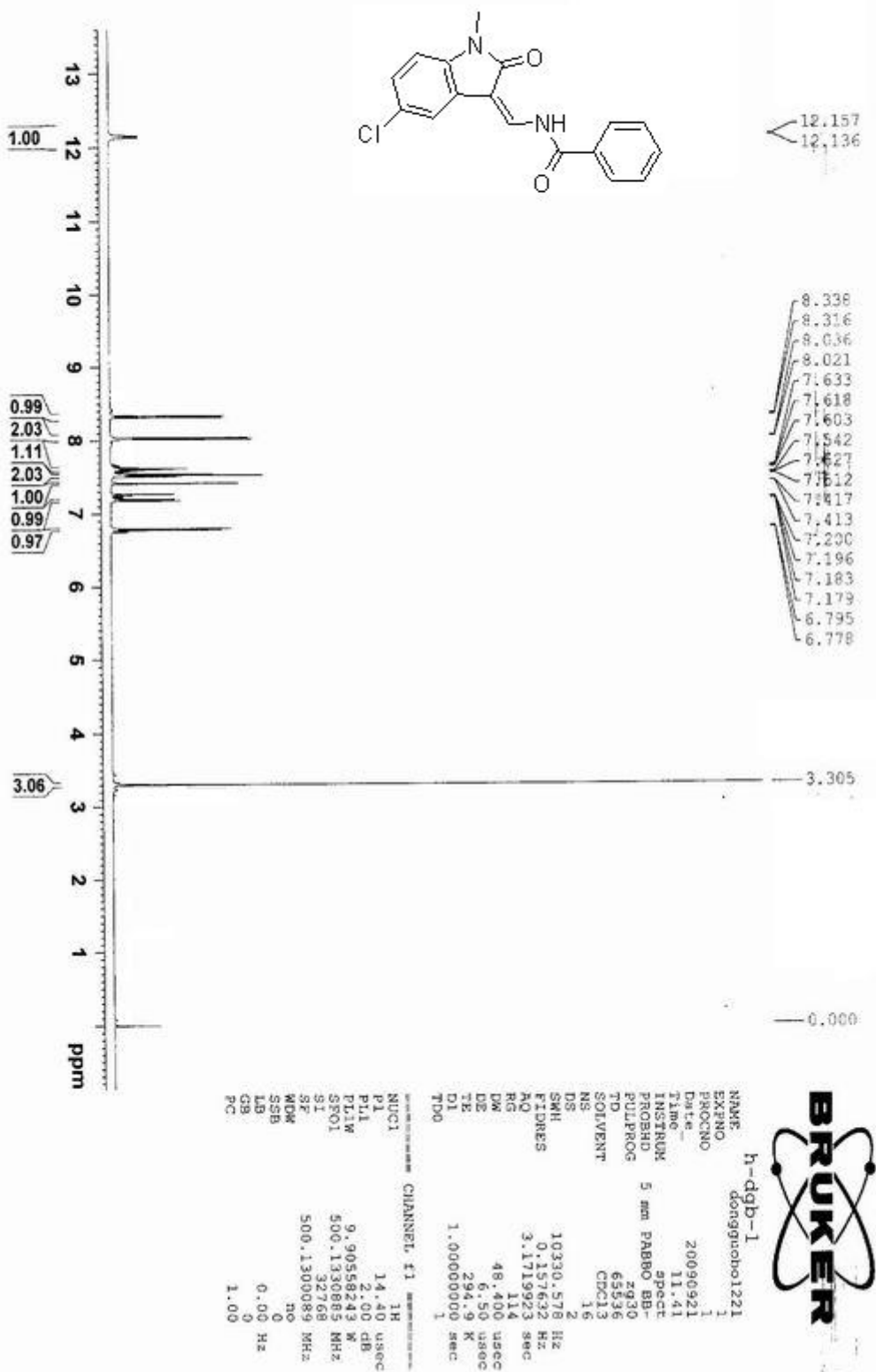
(Z)-N-((1,7-Dimethyl-2-oxoindolin-3-ylidene)methyl)benzamide (6)



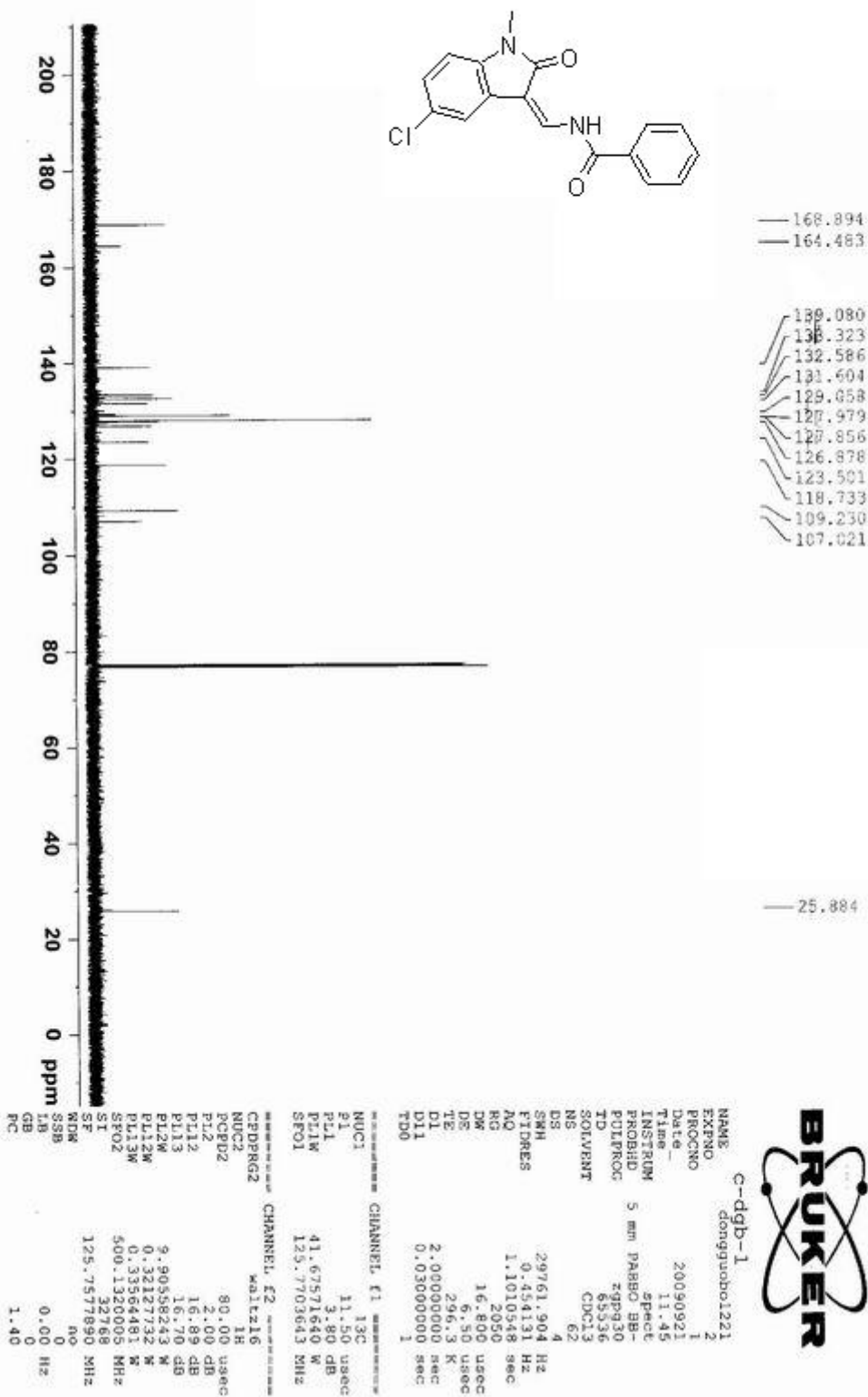
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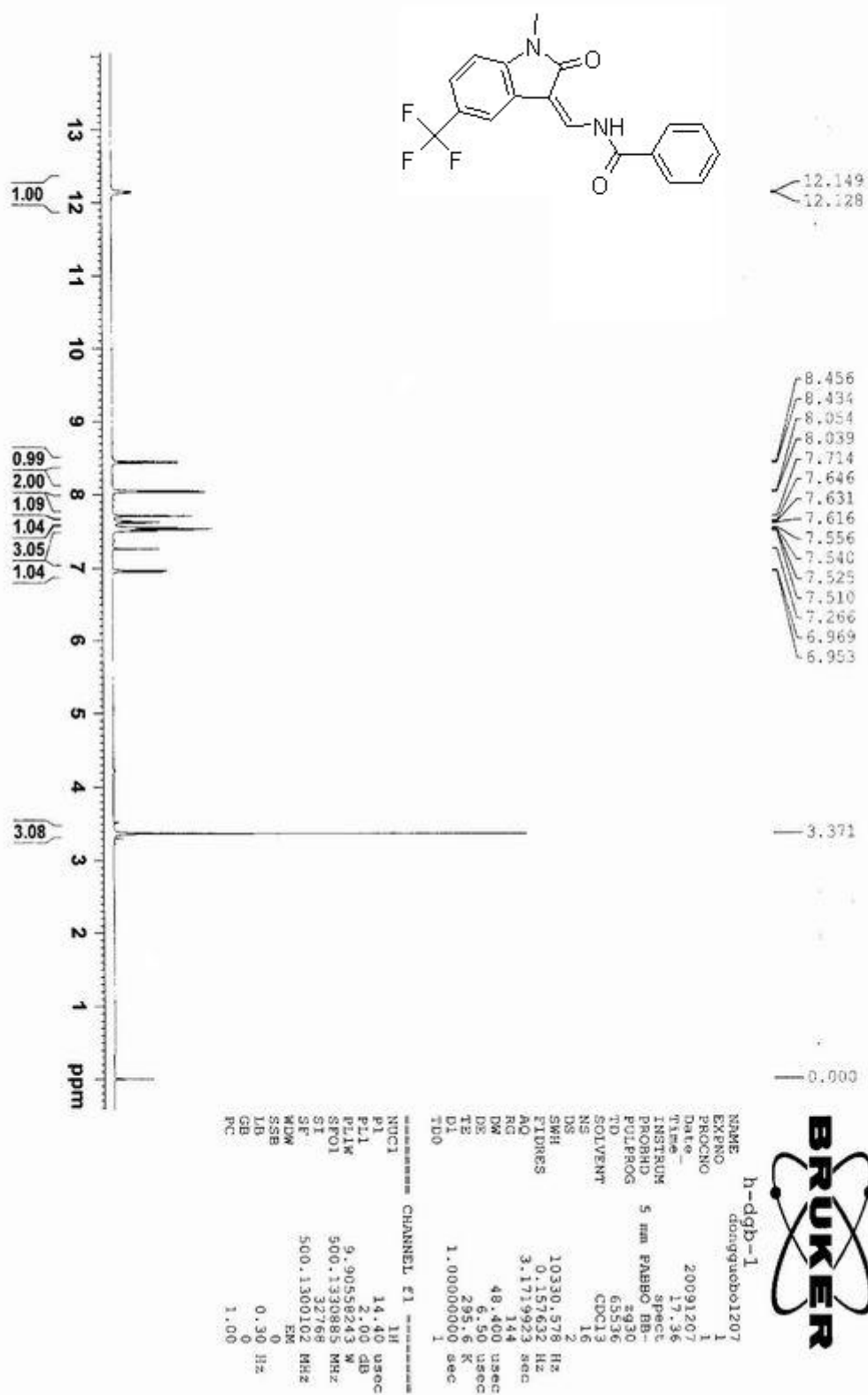
(Z)-N-((5-Chloro-1-methyl-2-oxoindolin-3-ylidene)methyl)benzamide (7)



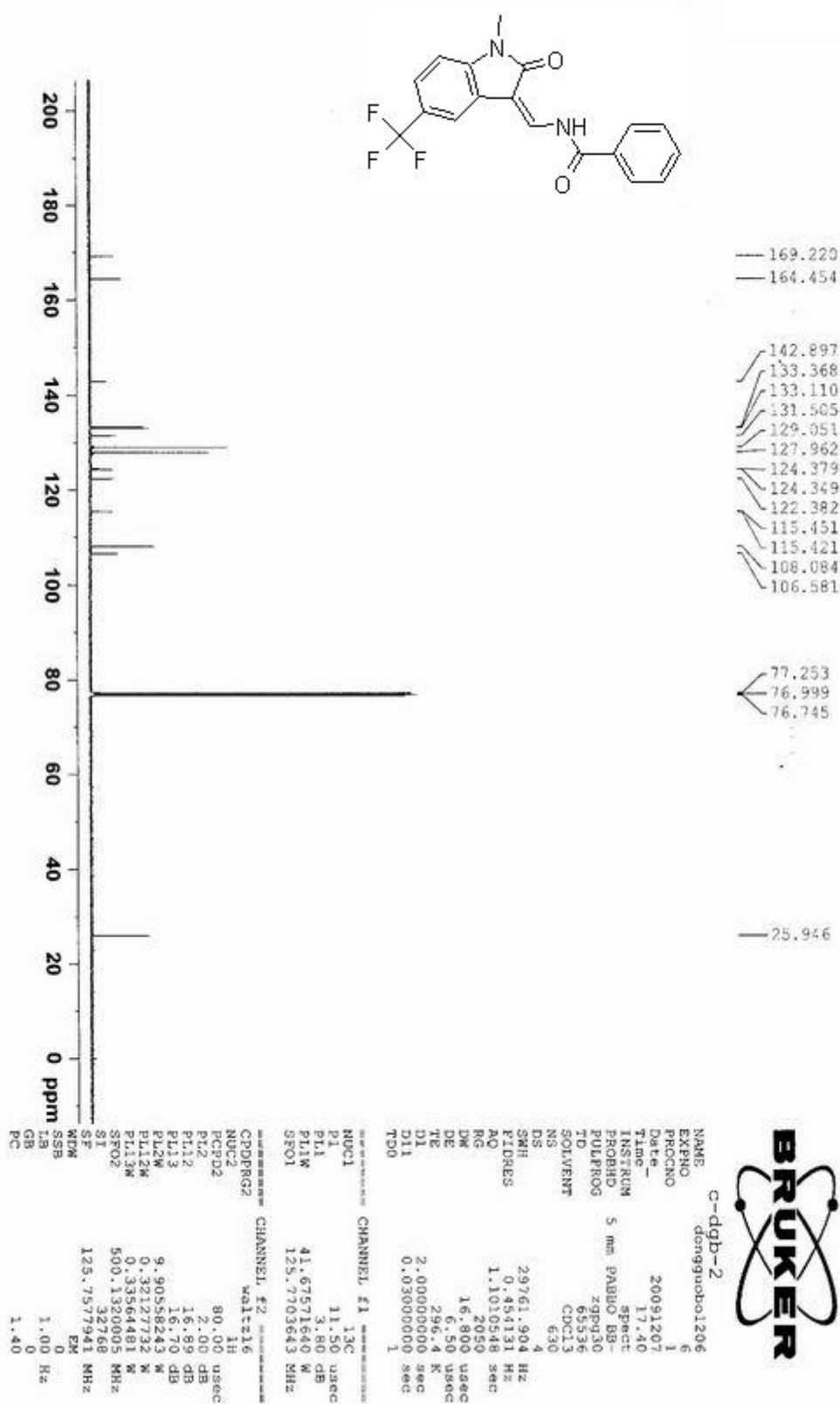
(Z)-N-((5-Chloro-1-methyl-2-oxindolin-3-ylidene)methyl)benzamide (7)



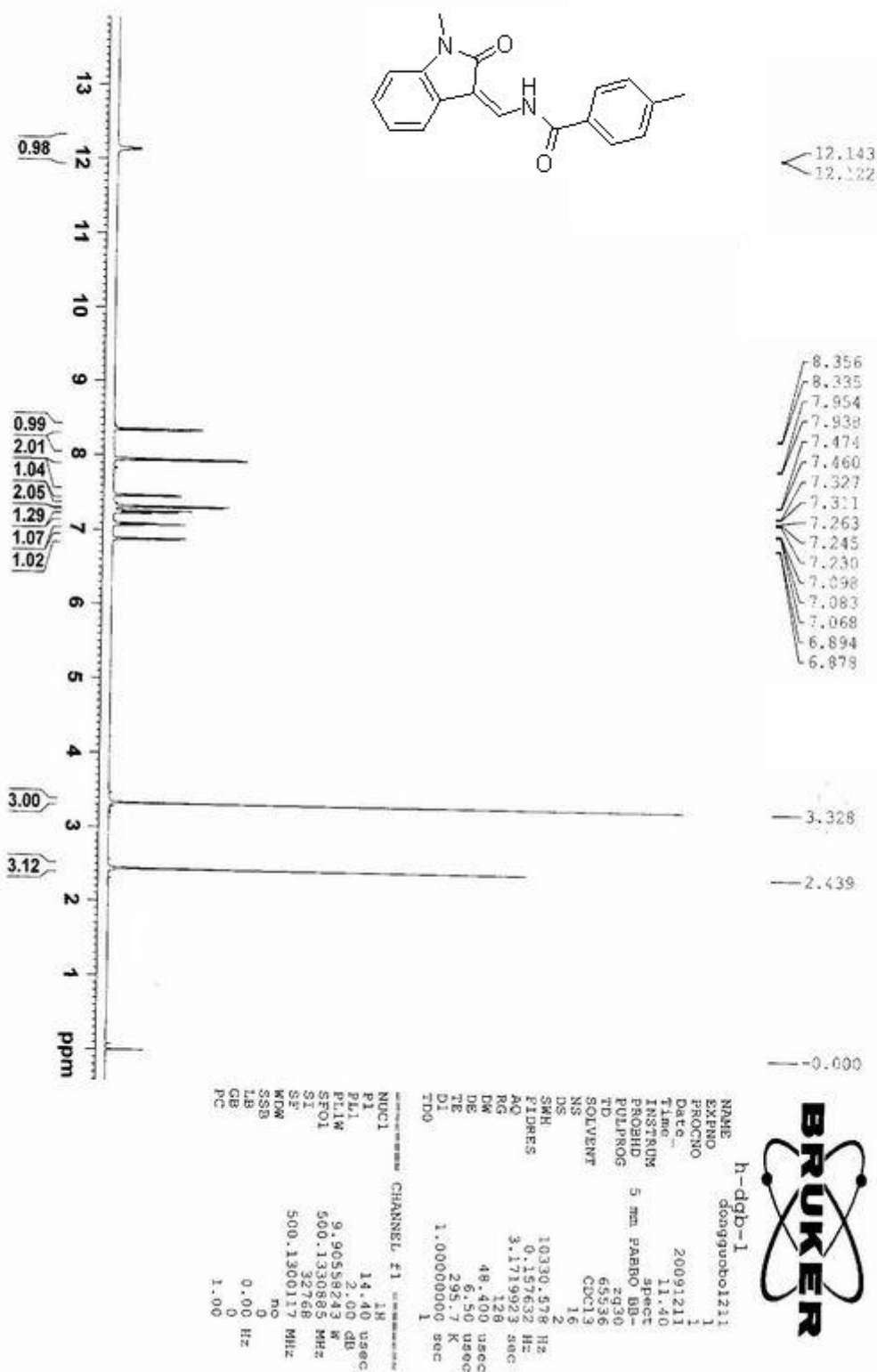
(Z)-N-((1-Methyl-2-oxo-5-(trifluoromethyl)indolin-3-ylidene)methyl)benzamide (8)



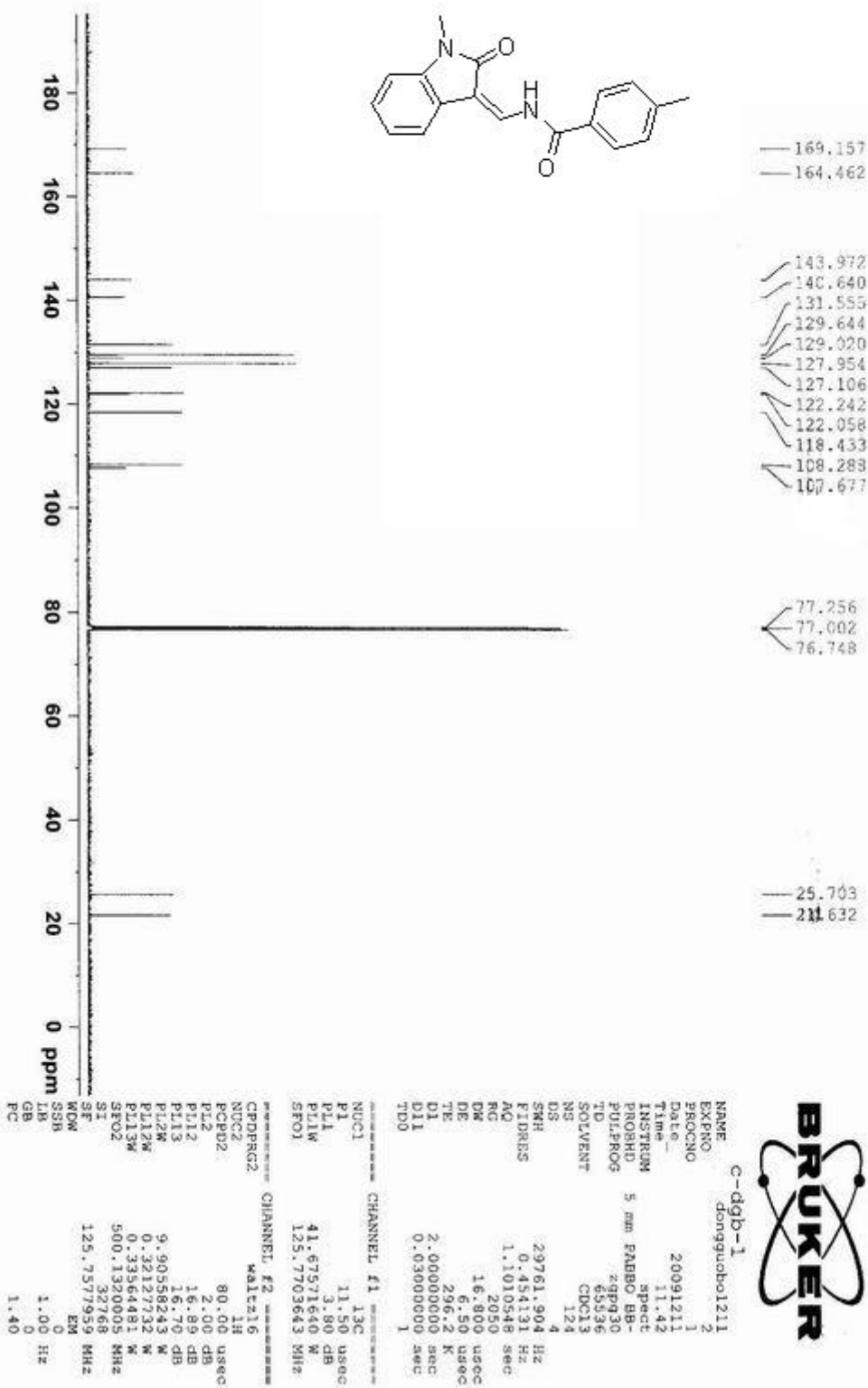
(Z)-N-((1-Methyl-2-oxo-5-(trifluoromethyl)indolin-3-ylidene)methyl)benzamide (8)



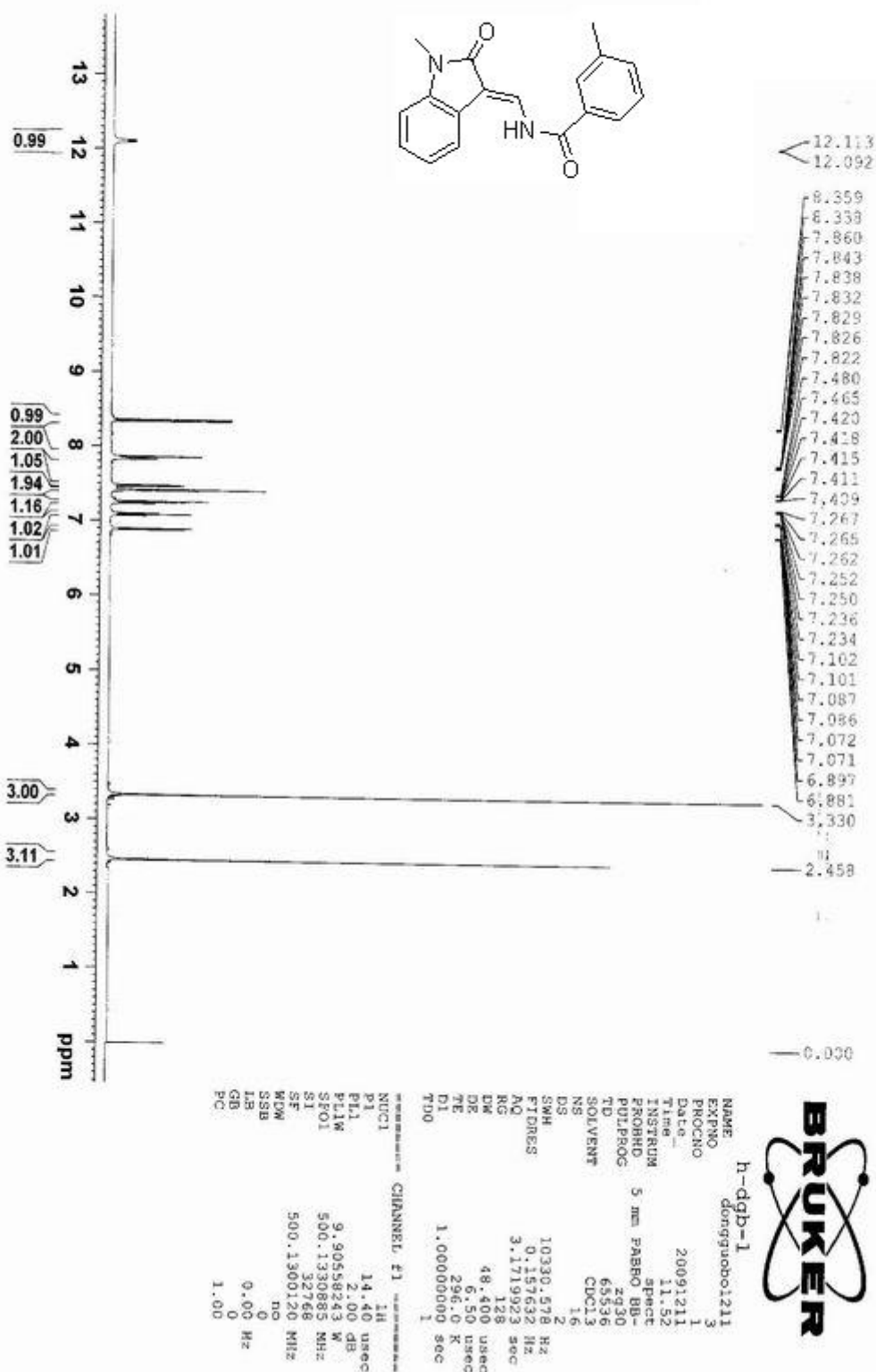
(Z)-4-Methyl-N-((1-methyl-2-oxoindolin-3-ylidene)methyl)benzamide (9)



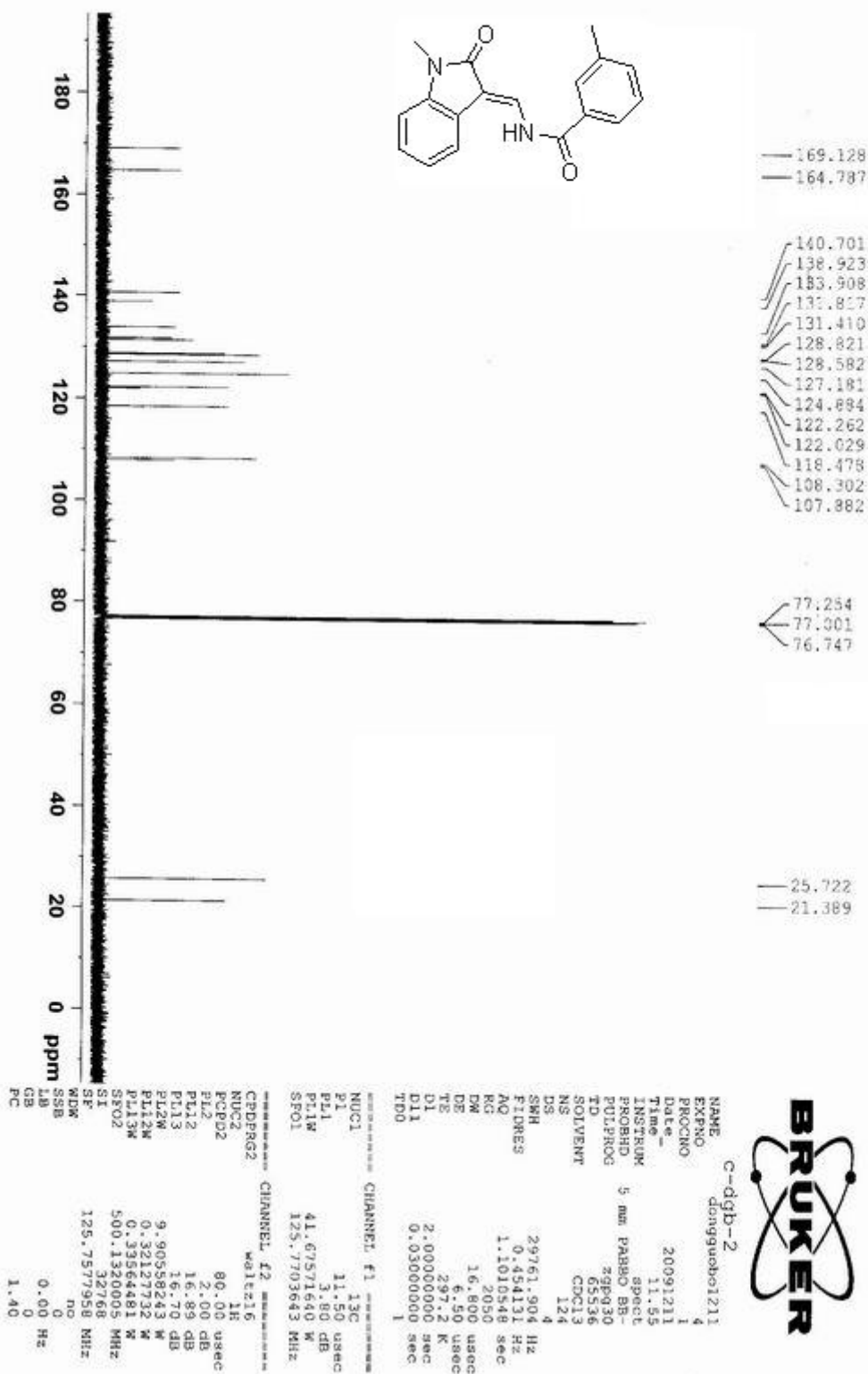
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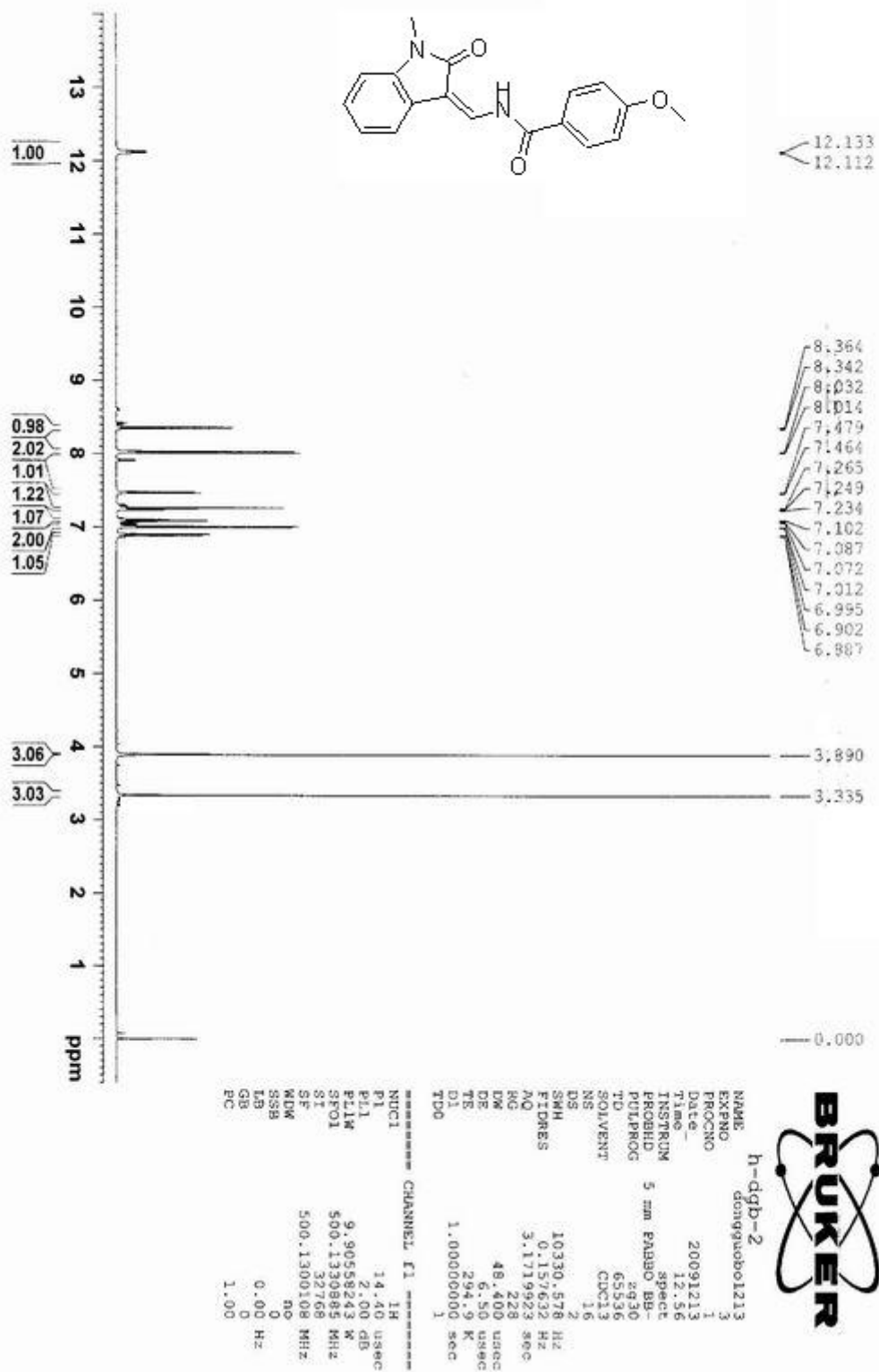
(Z)-3-Methyl-N-((1-methyl-2-oxindolin-3-ylidene)methyl)benzamide (10)



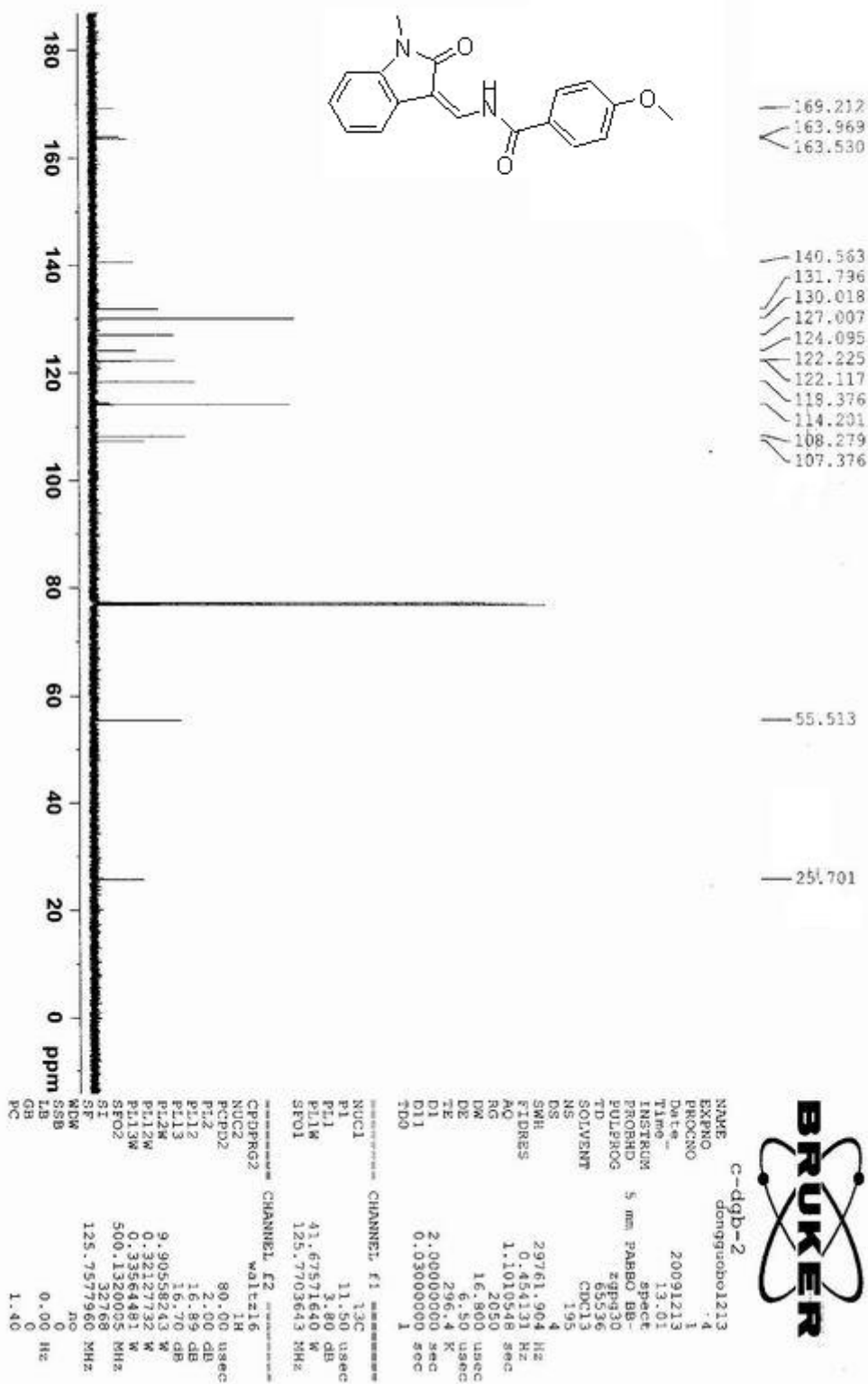
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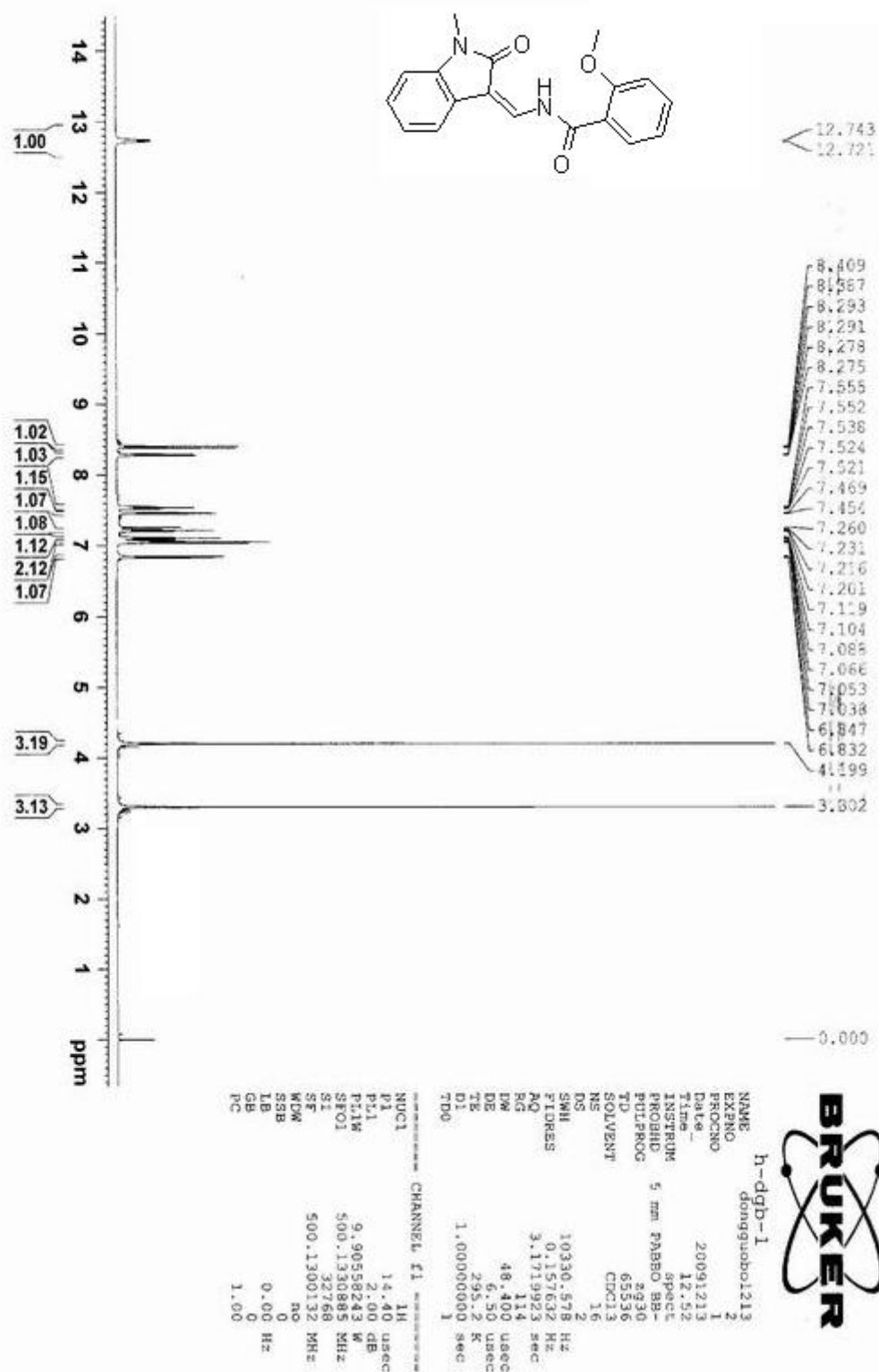
(Z)-4-Methoxy-N-((1-methyl-2-oxoindolin-3-ylidene)methyl)benzamide (11)



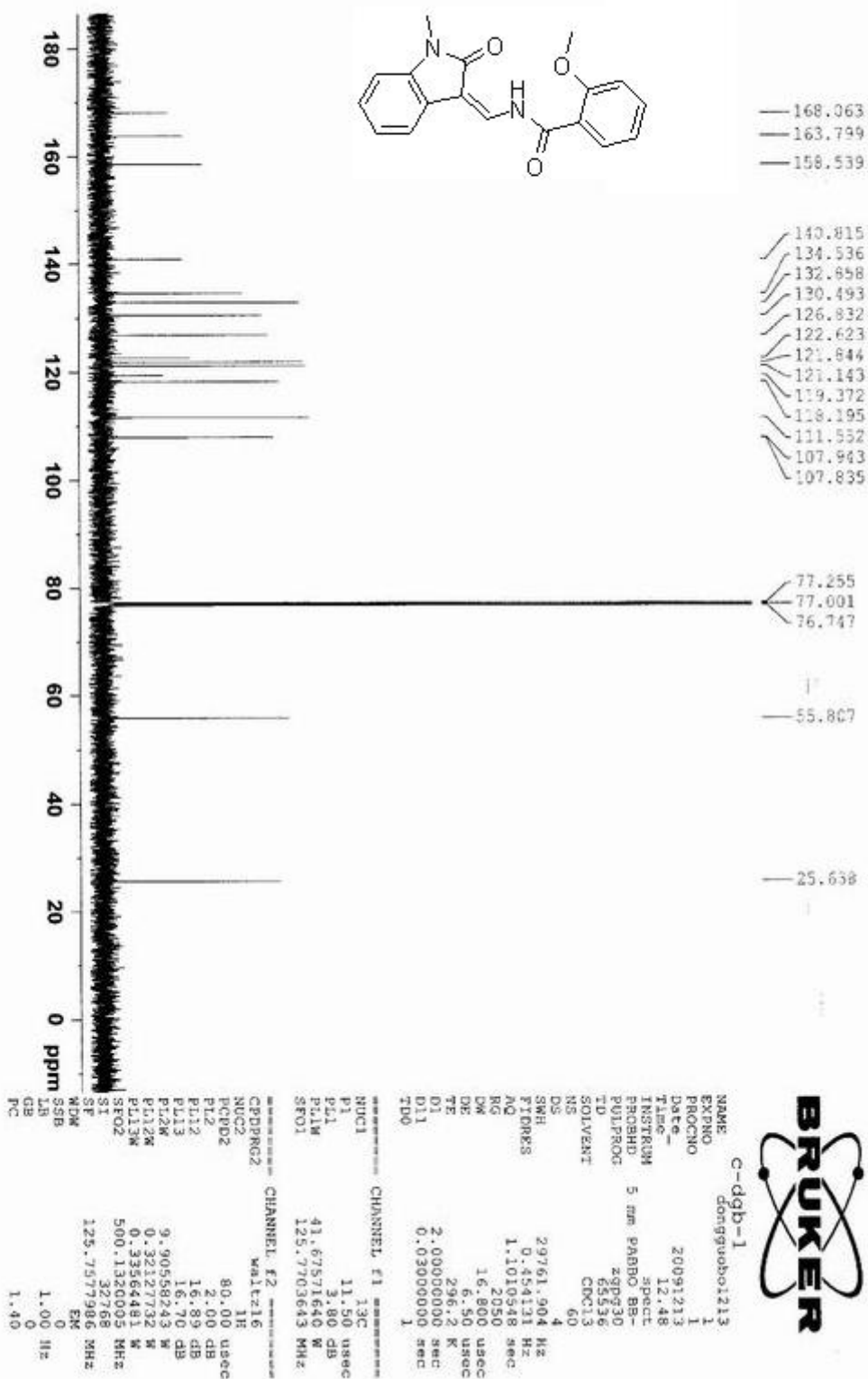
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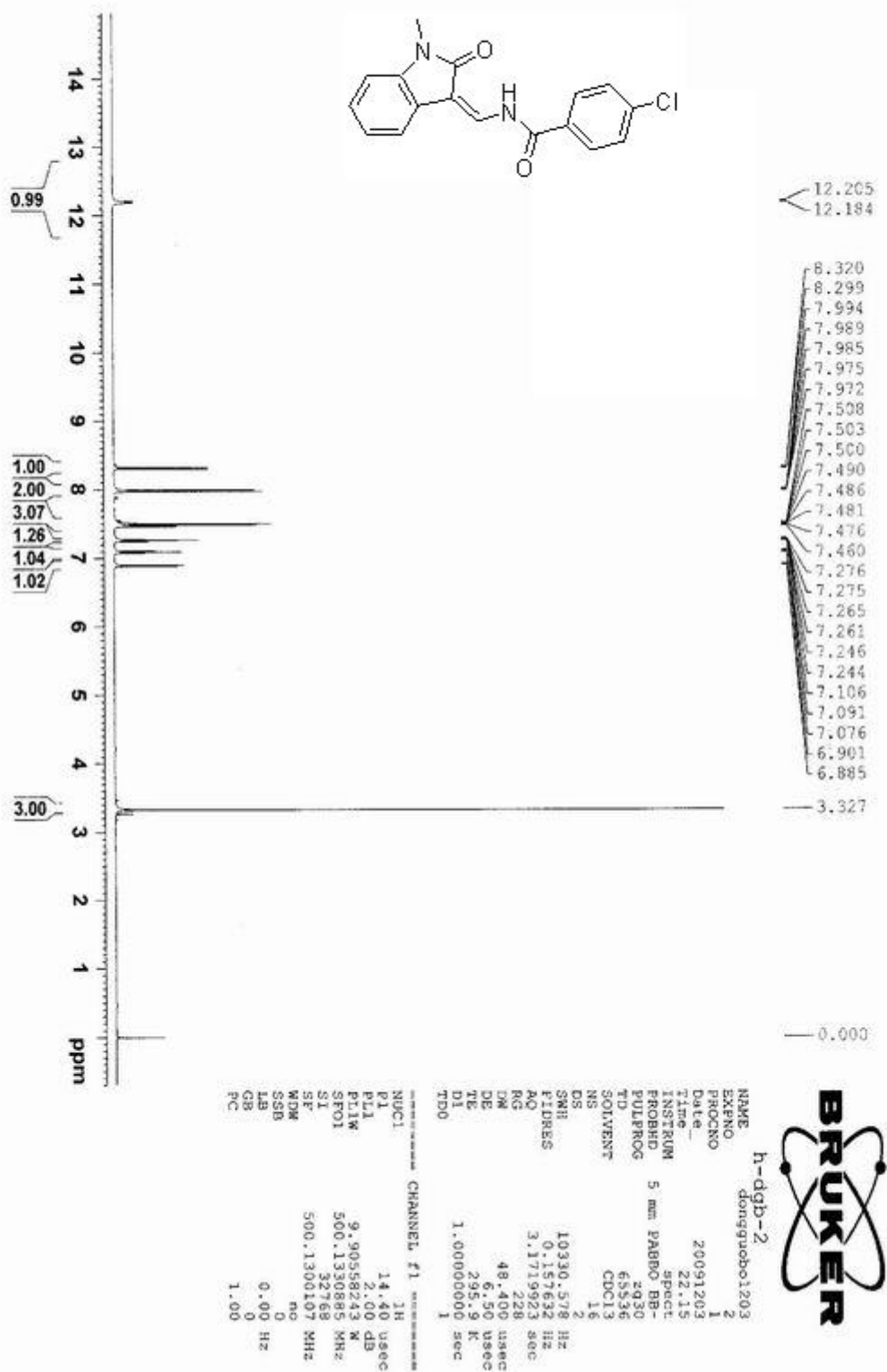
(Z)-2-Methoxy-N-((1-methyl-2-oxoindolin-3-ylidene)methyl)benzamide (12)



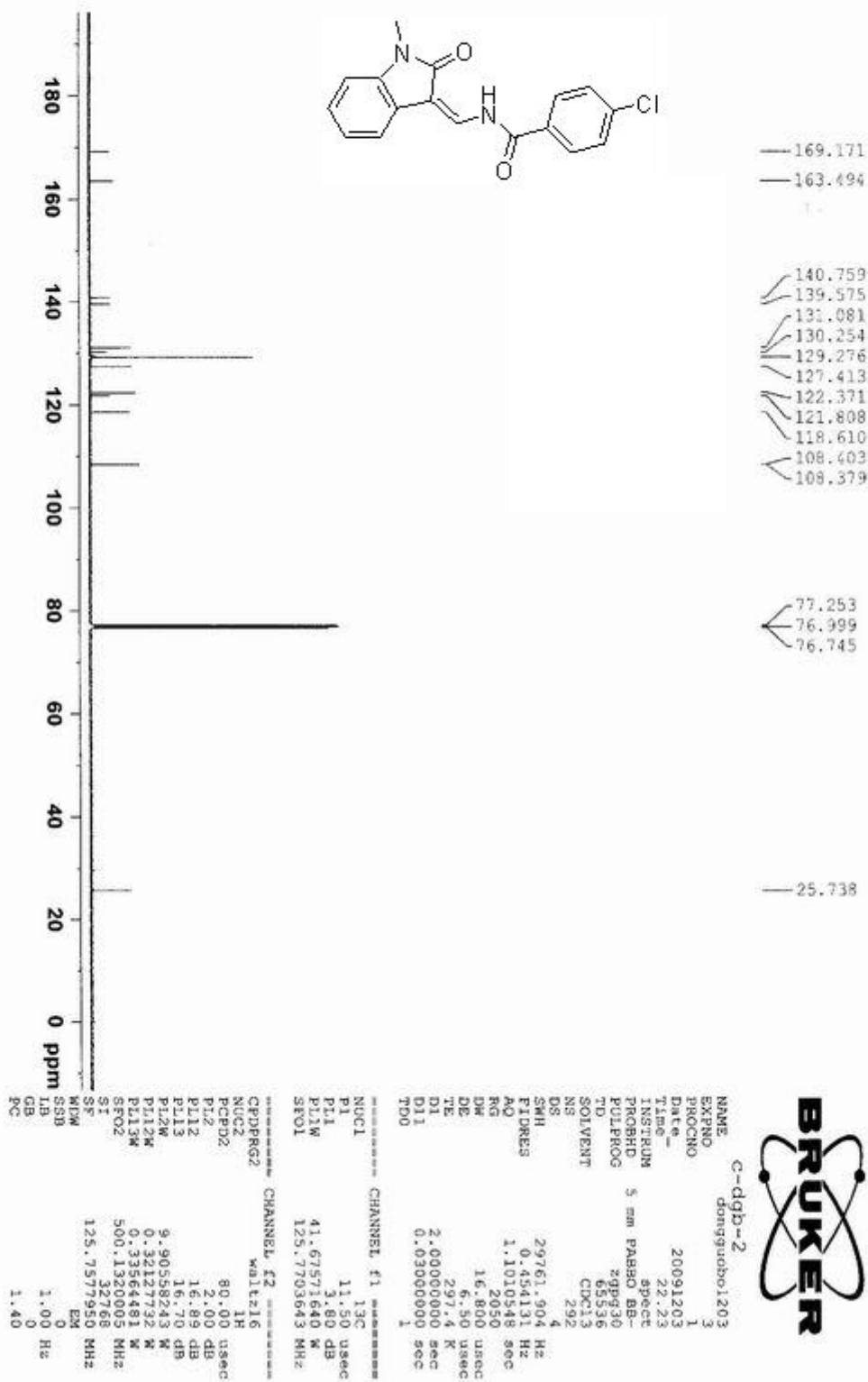
(Z)-2-Methoxy-N-((1-methyl-2-oxoindolin-3-ylidene)methyl)benzamide (12)



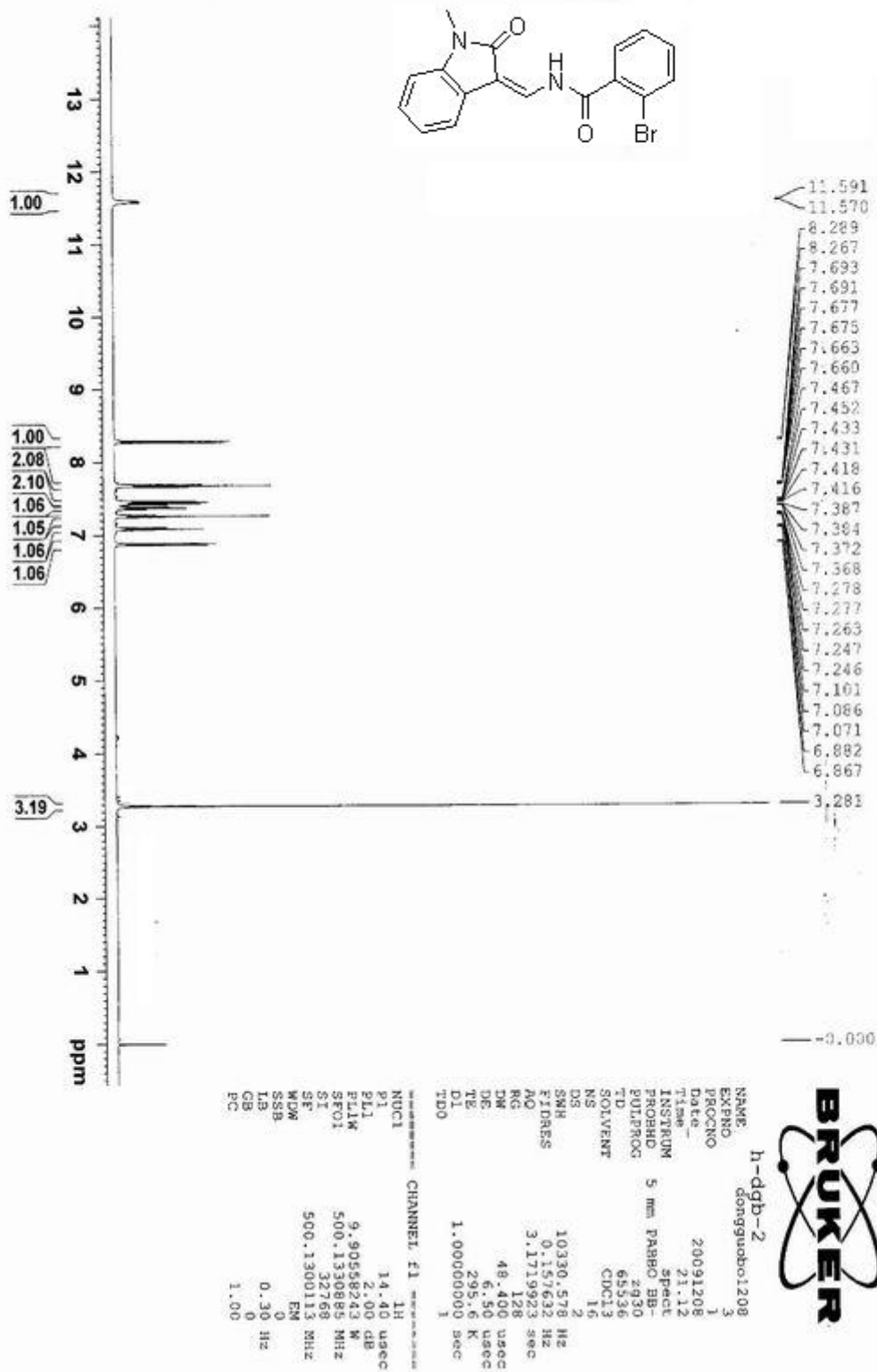
(Z)-4-Chloro-N-((1-methyl-2-oxindolin-3-ylidene)methyl)benzamide (13)



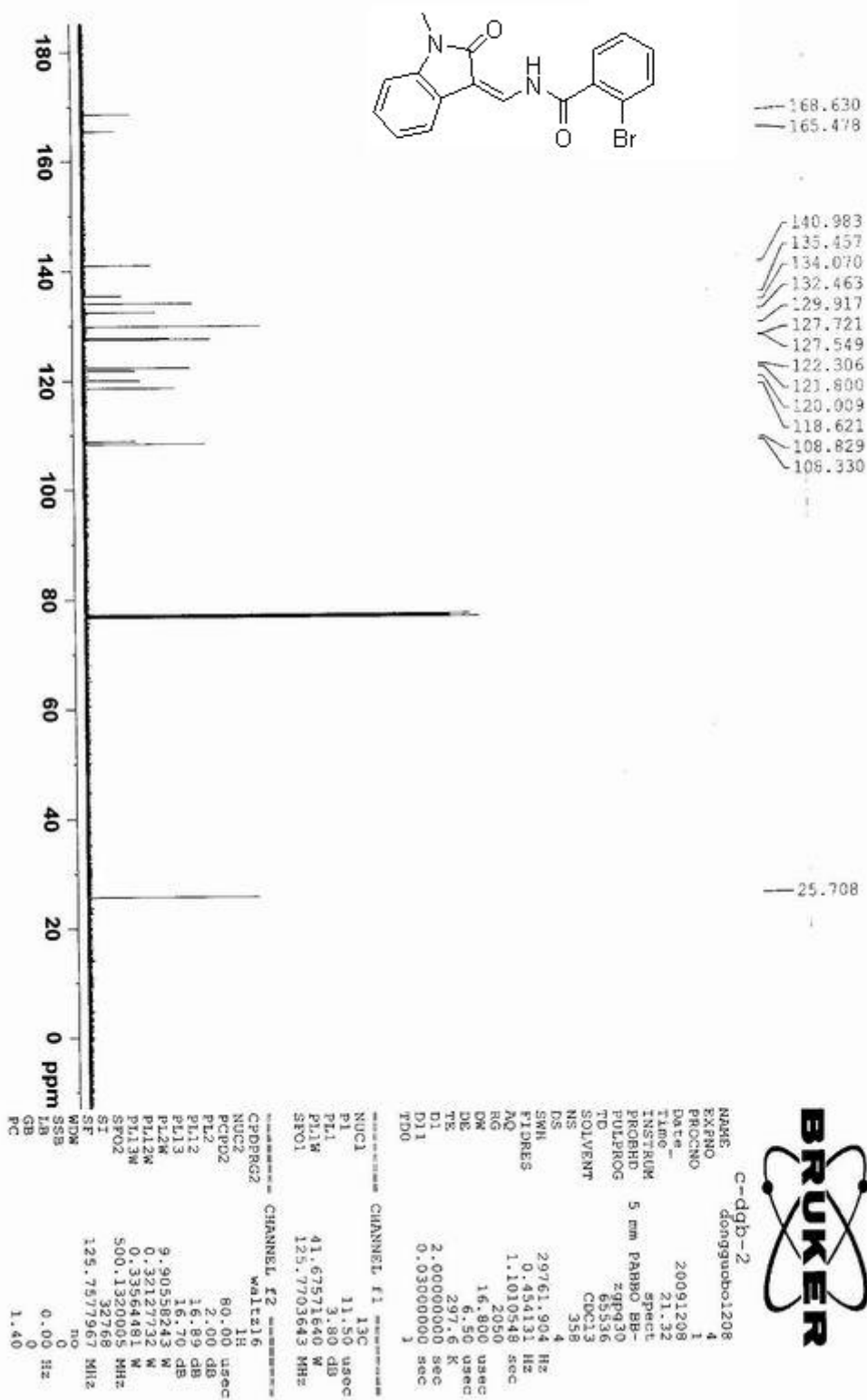
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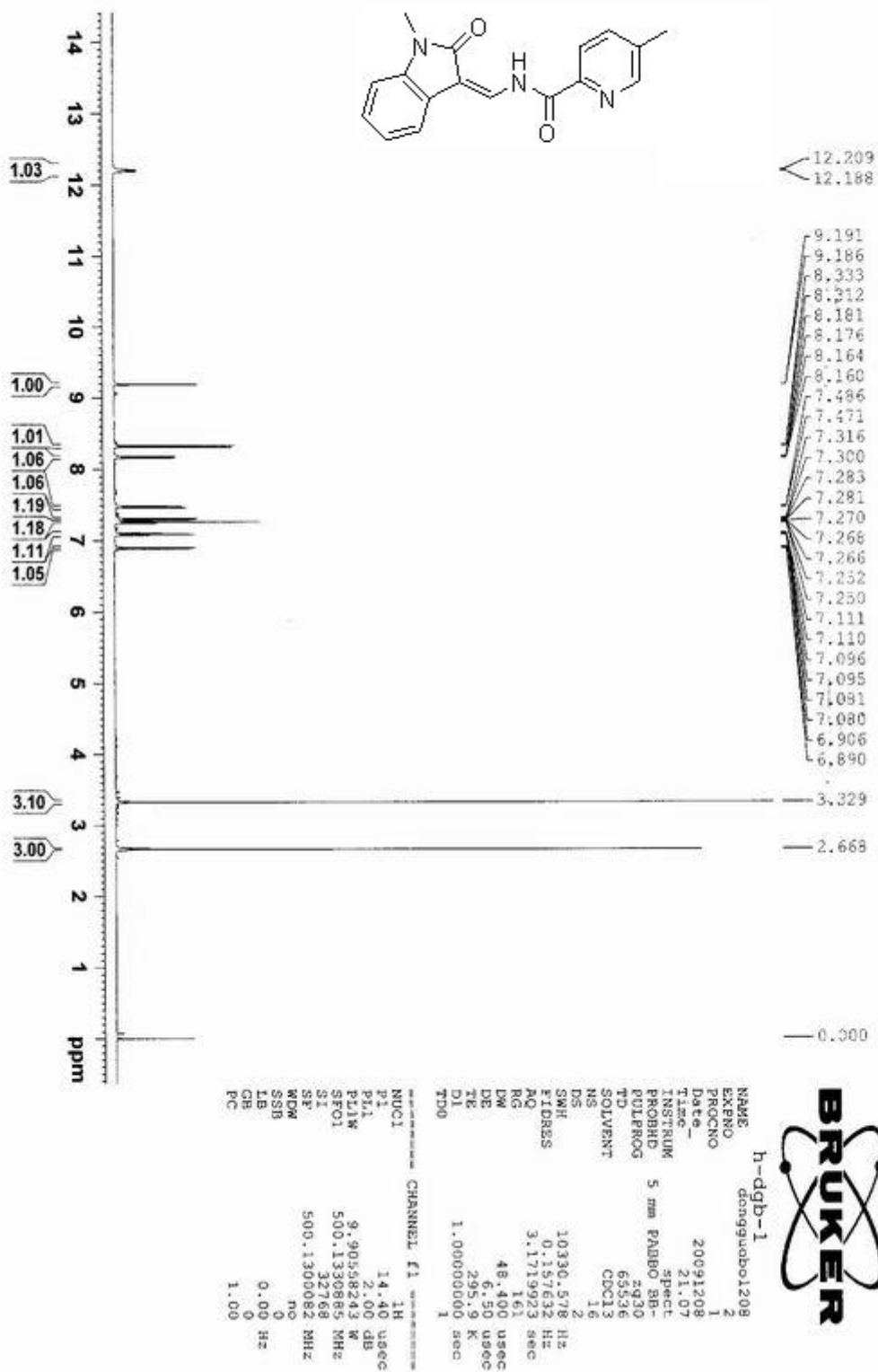
(Z)-2-Bromo-N-((1-methyl-2-oxindolin-3-ylidene)methyl)benzamide (14)



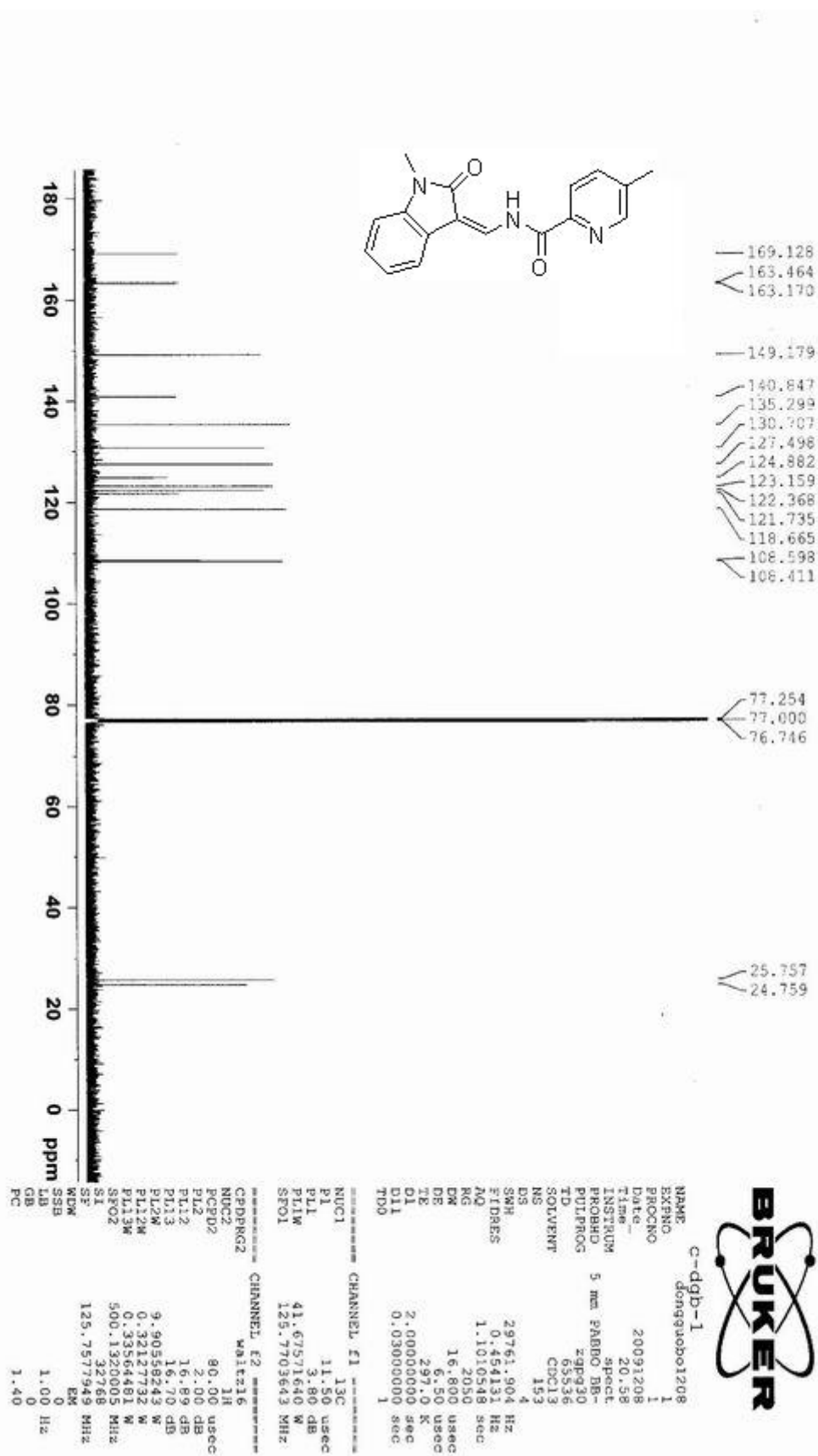
(Z)-2-Bromo-N-((1-methyl-2-oxindolin-3-ylidene)methyl)benzamide (14)



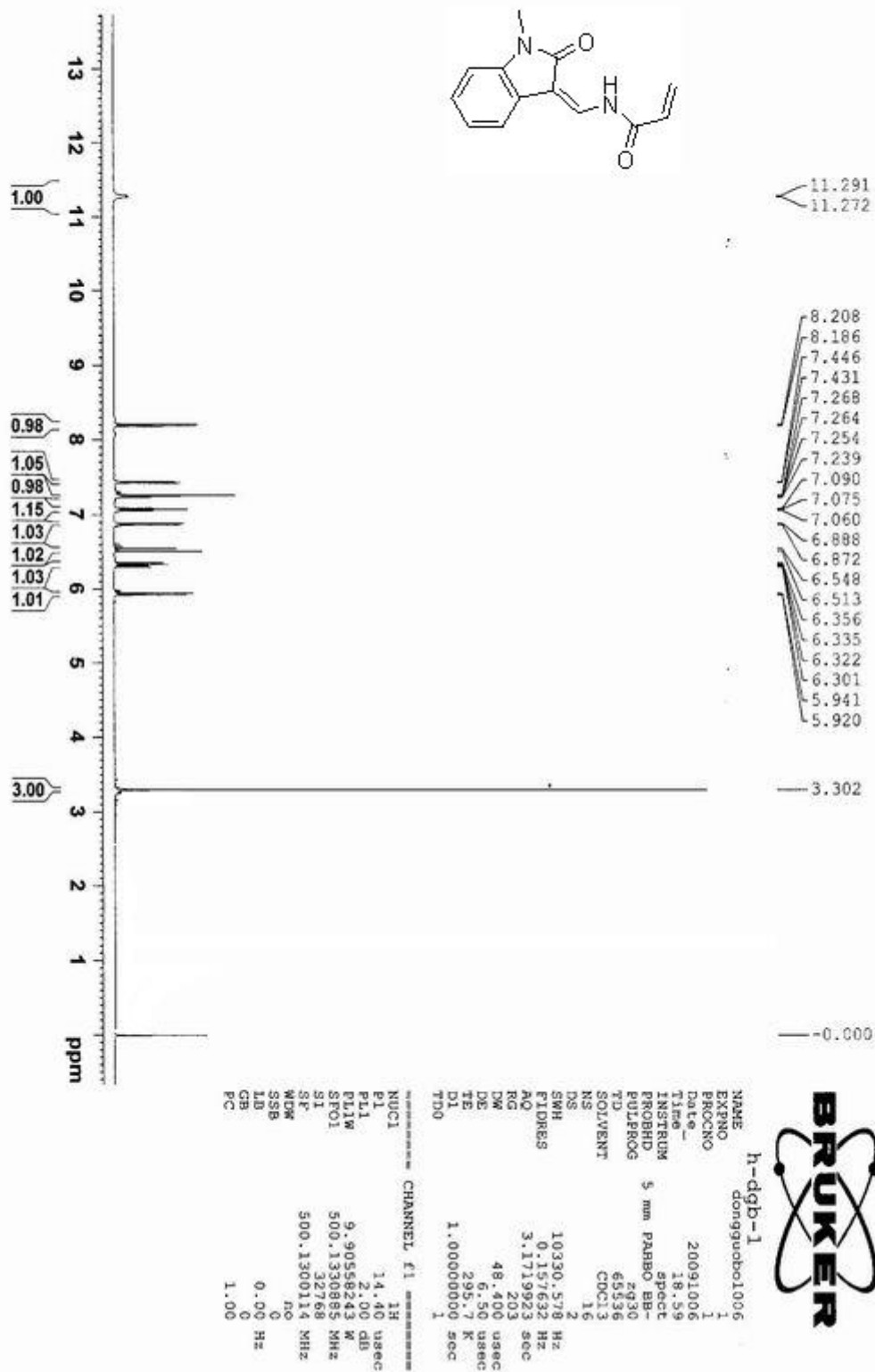
(Z)-5-Methyl-N-((1-methyl-2-oxindolin-3-ylidene)methyl)picolinamide (15)



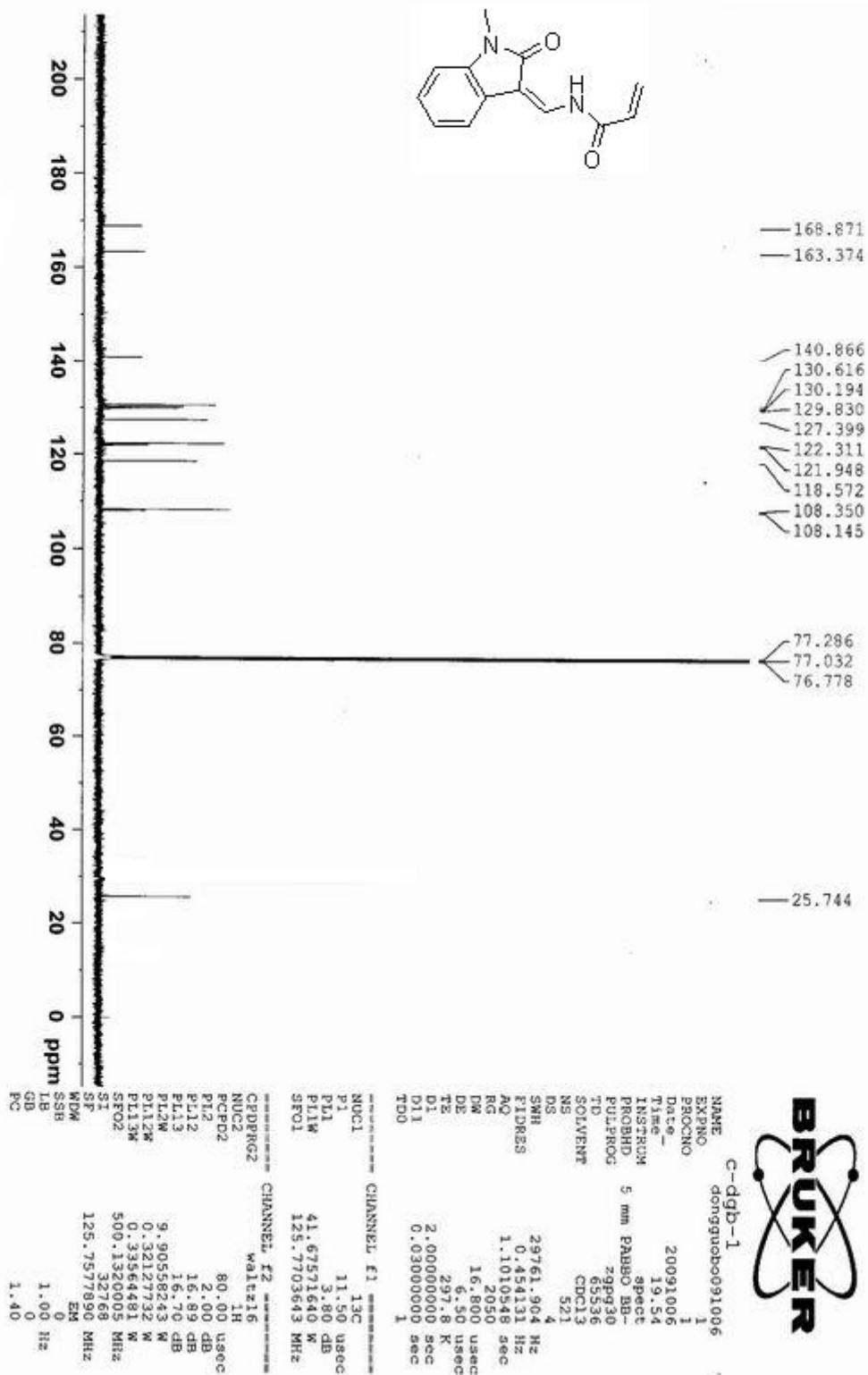
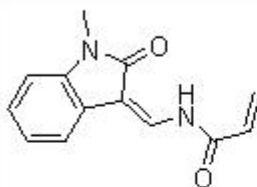
(Z)-5-Methyl-N-((1-methyl-2-oxindolin-3-ylidene)methyl)picolinamide (15)



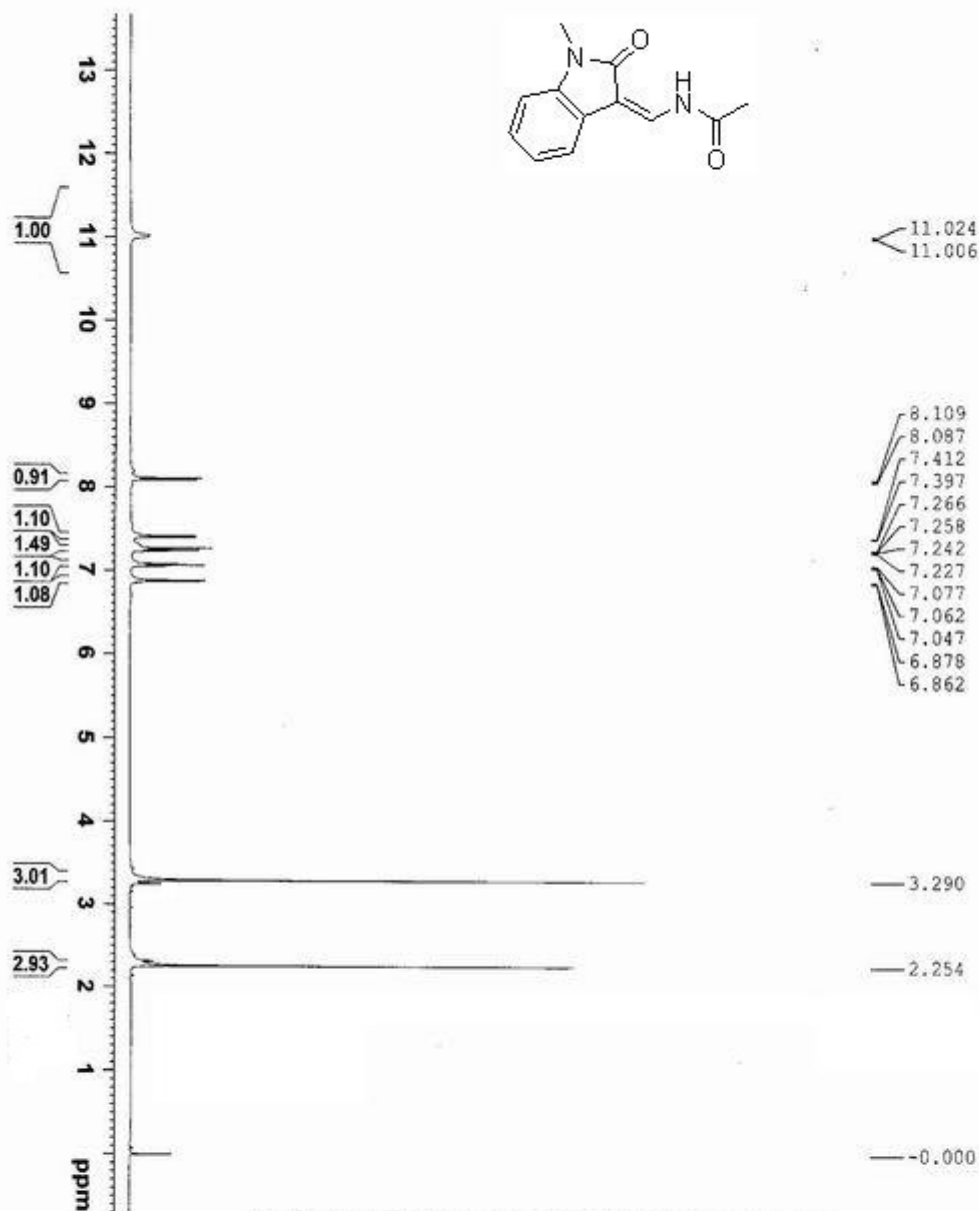
(Z)-N-((1-Methyl-2-oxindolin-3-ylidene)methyl)acrylamide (16)



(Z)-N-((1-Methyl-2-oxindolin-3-ylidene)methyl)acrylamide (16)



(Z)-N-((1-Methyl-2-oxoindolin-3-ylidene)methyl)acetamide (17)

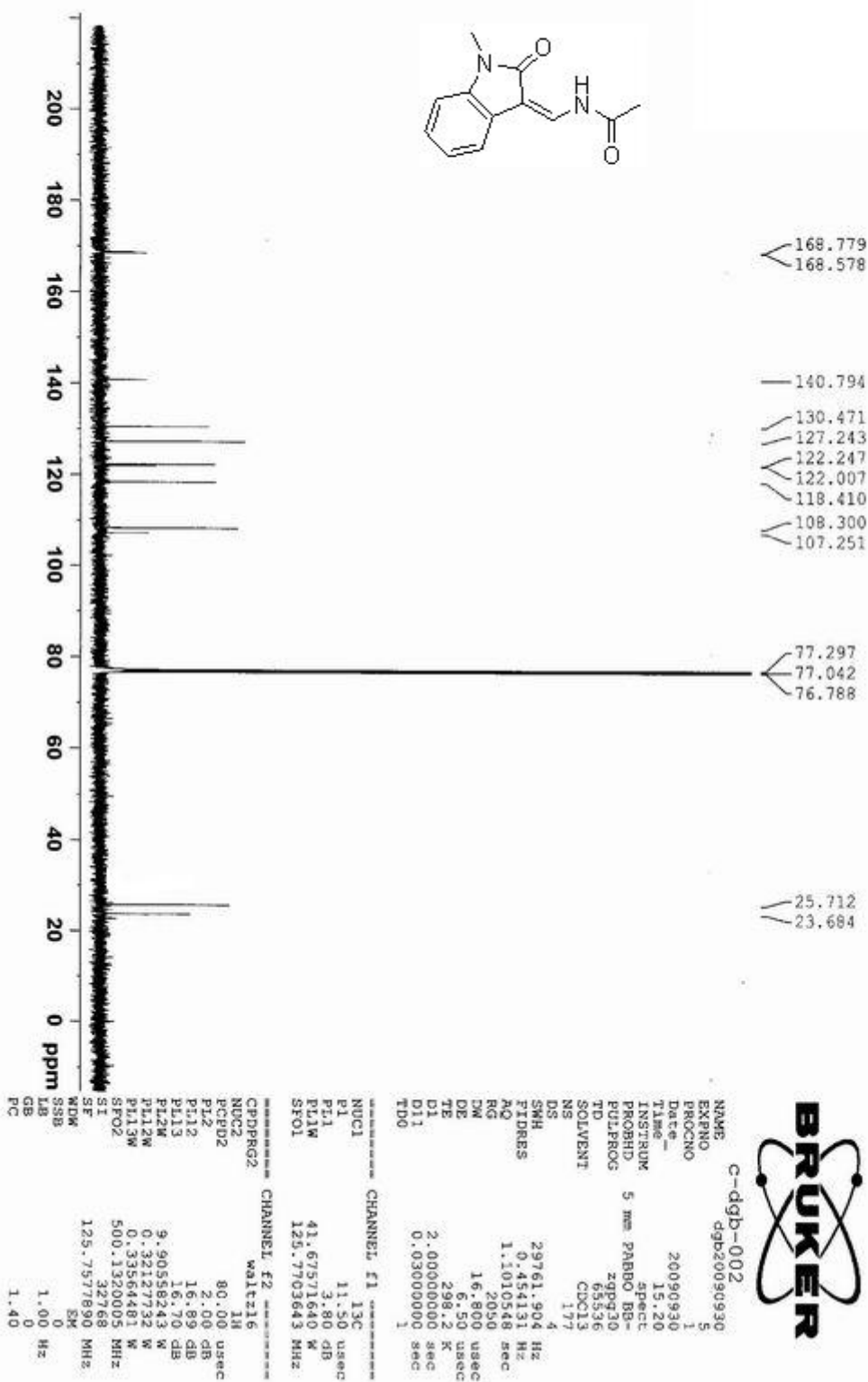


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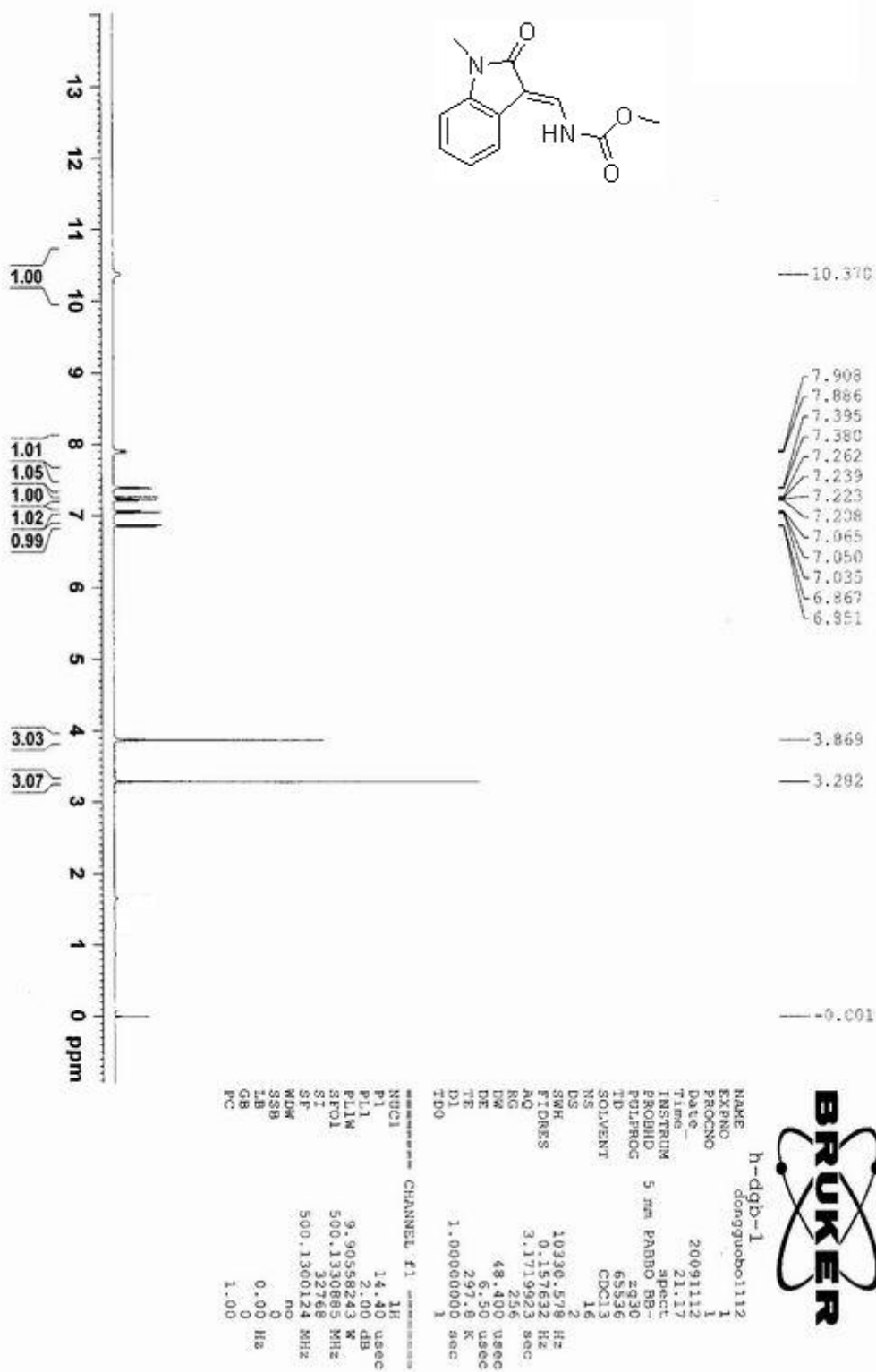
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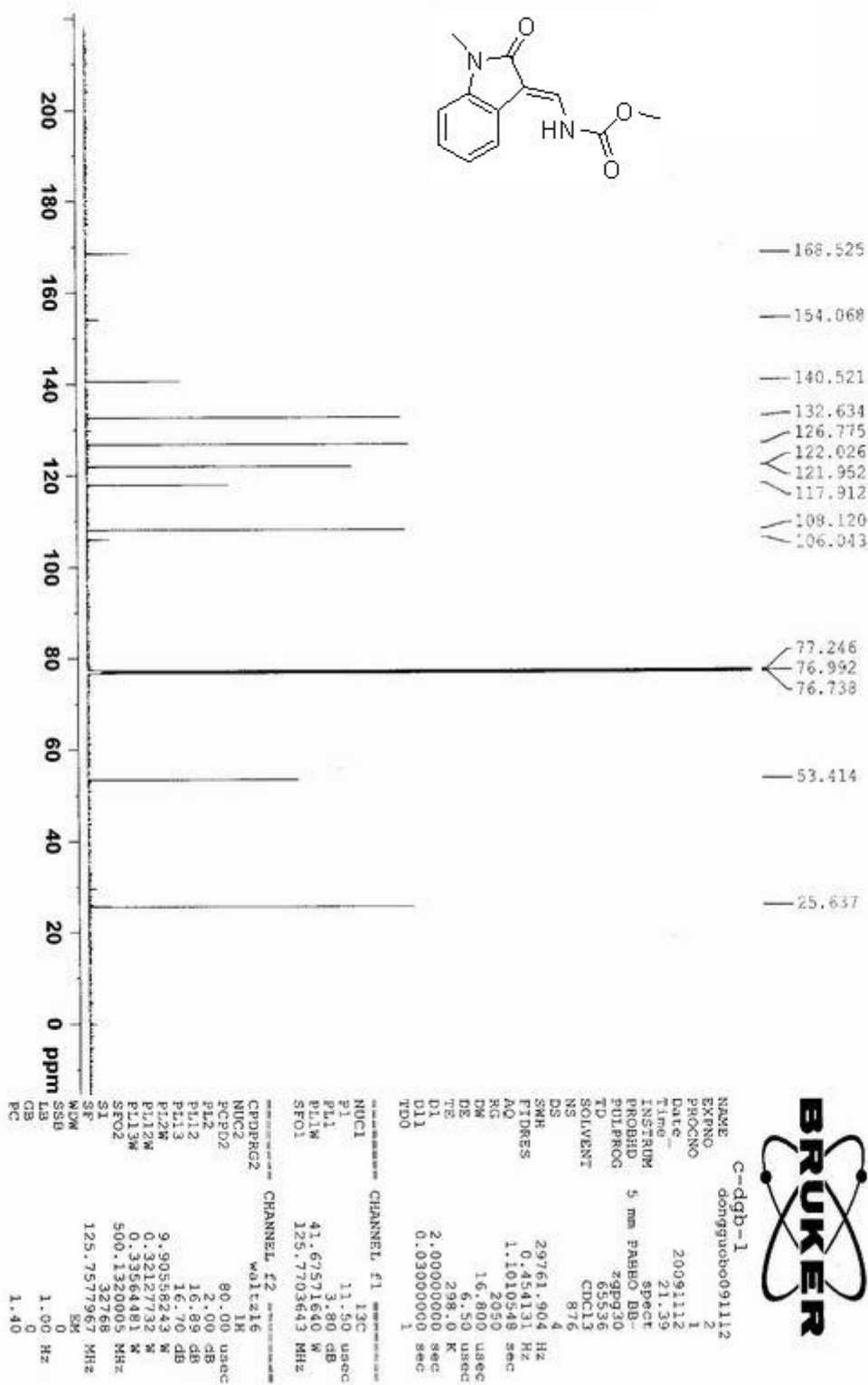
(Z)-N-((1-Methyl-2-oxoindolin-3-ylidene)methyl)acetamide (17)



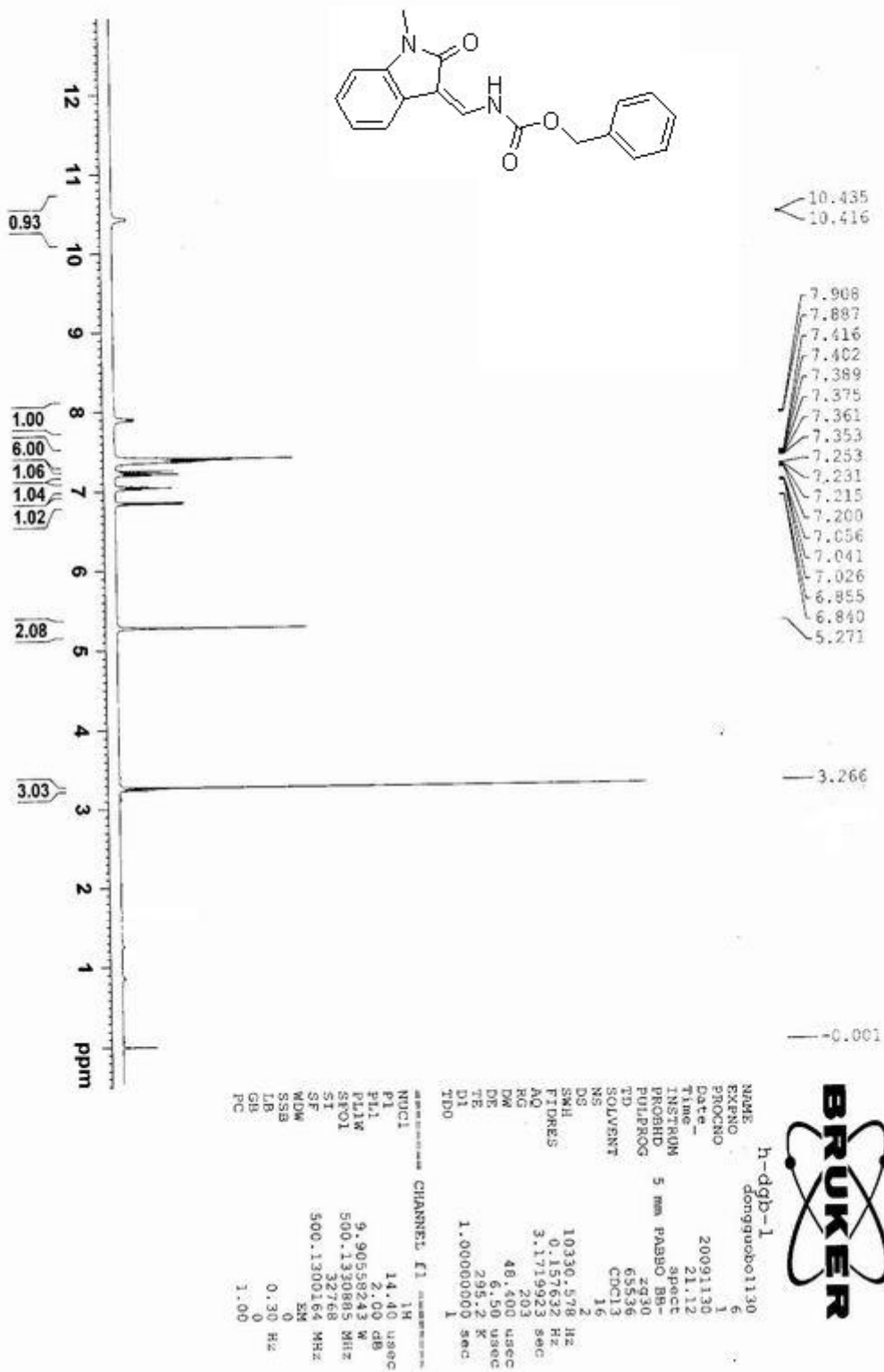
(Z)-Methyl-N-(1-methyl-2-oxindolin-3-ylidene)methylcarbamate (18)



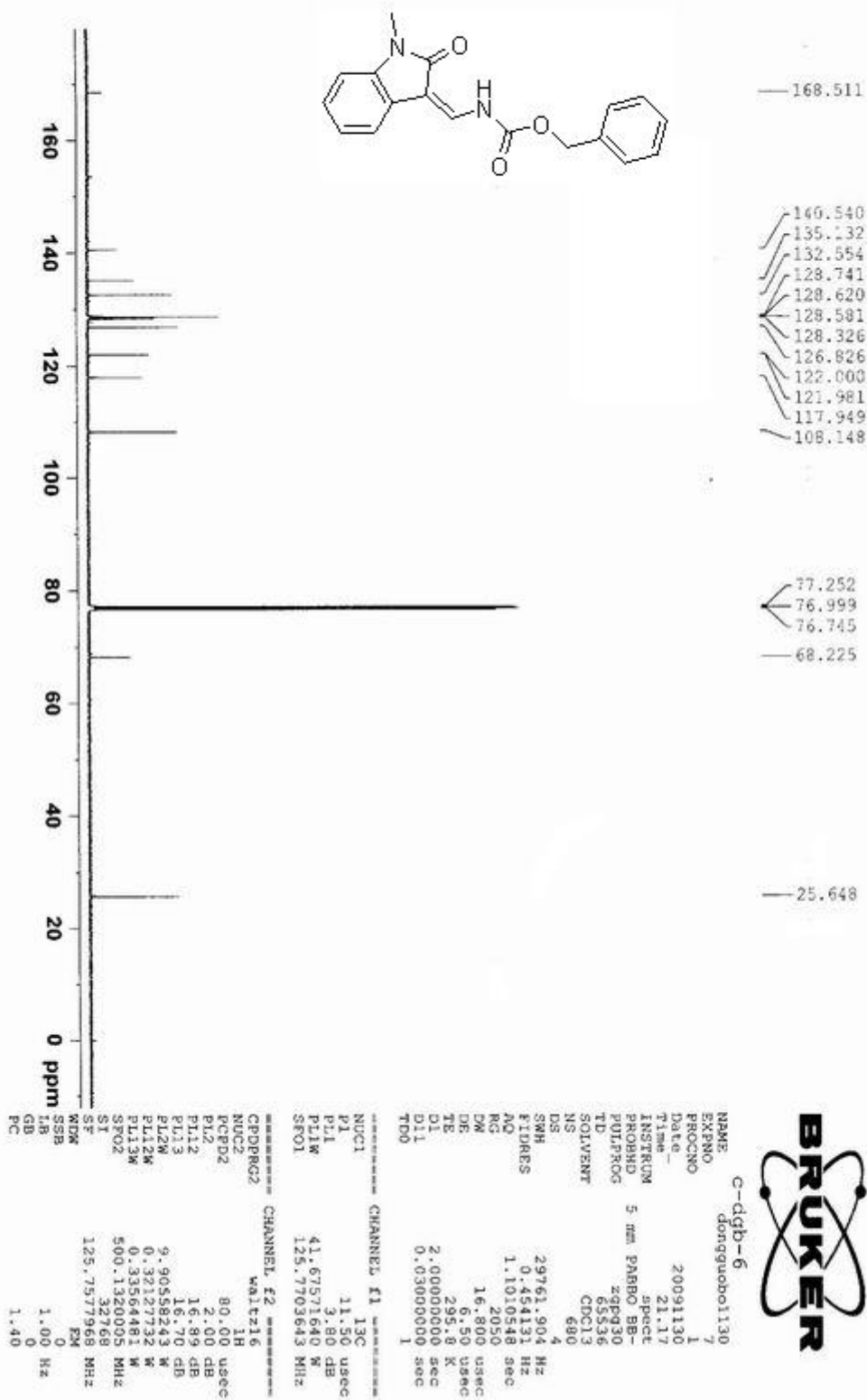
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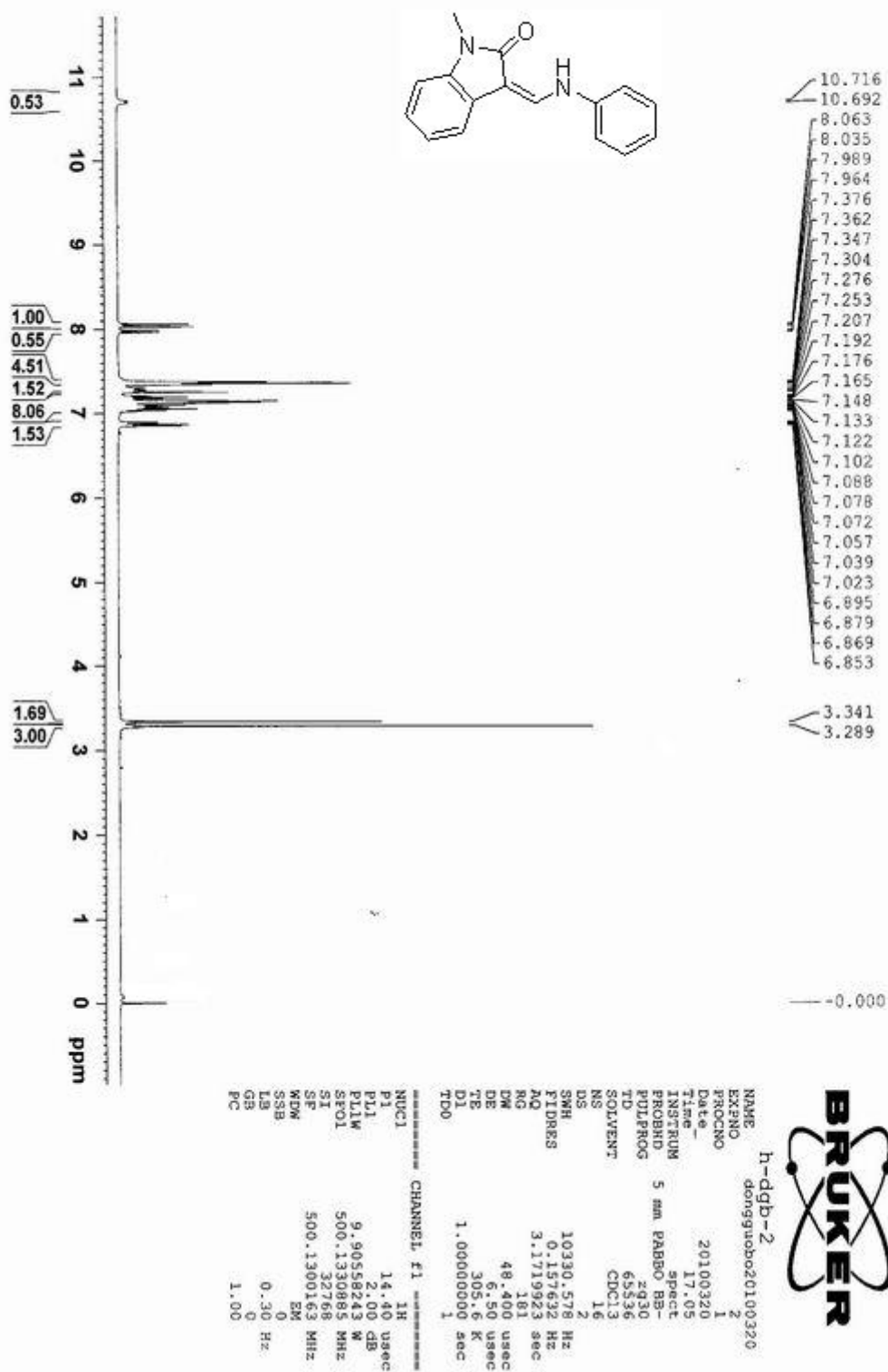
(Z)-Benzyl-N-(1-methyl-2-oxindolin-3-ylidene)methylcarbamate (19)



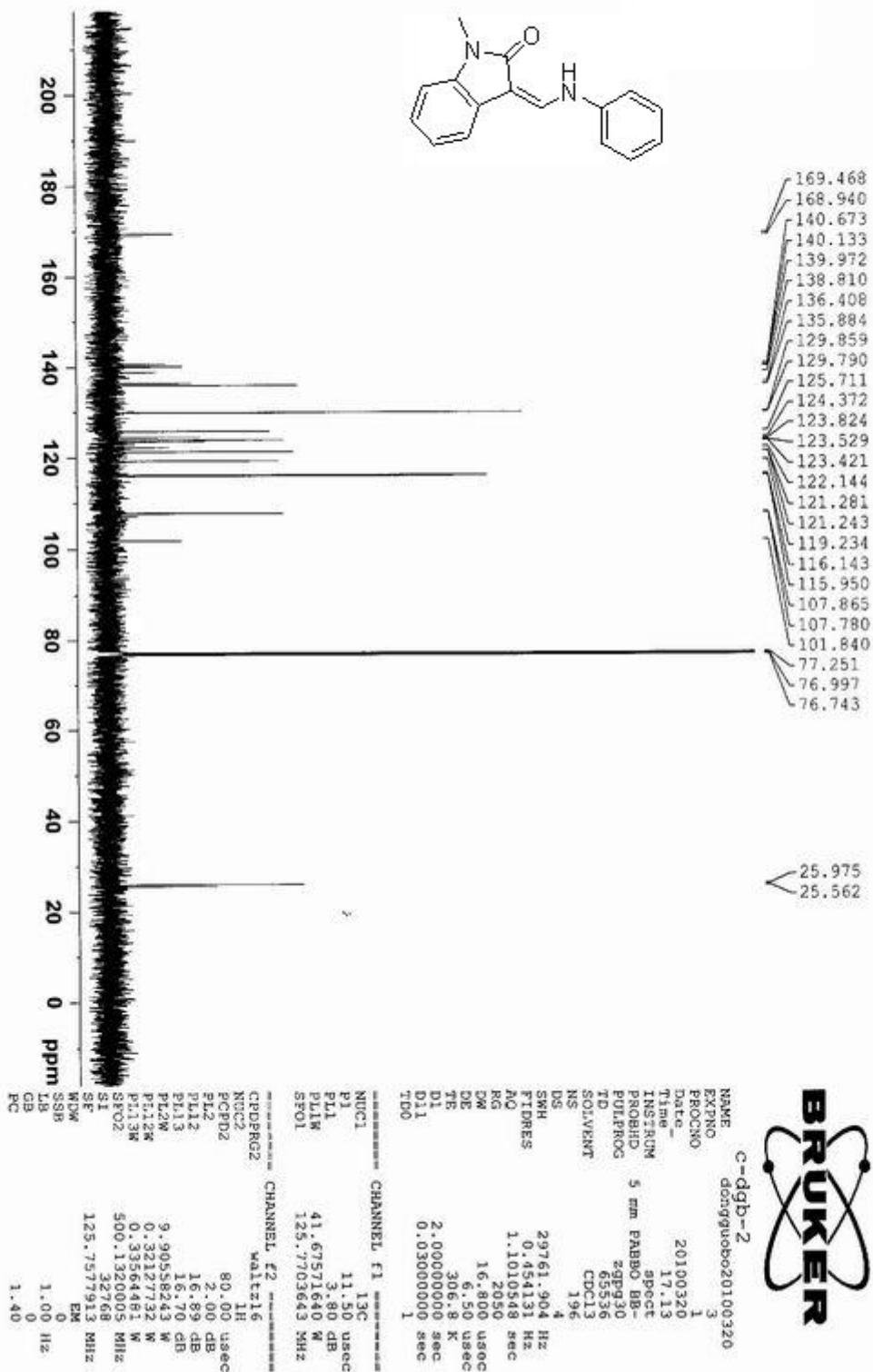
(Z)-Benzyl-N-(1-methyl-2-oxindolin-3-ylidene)methylcarbamate (19)



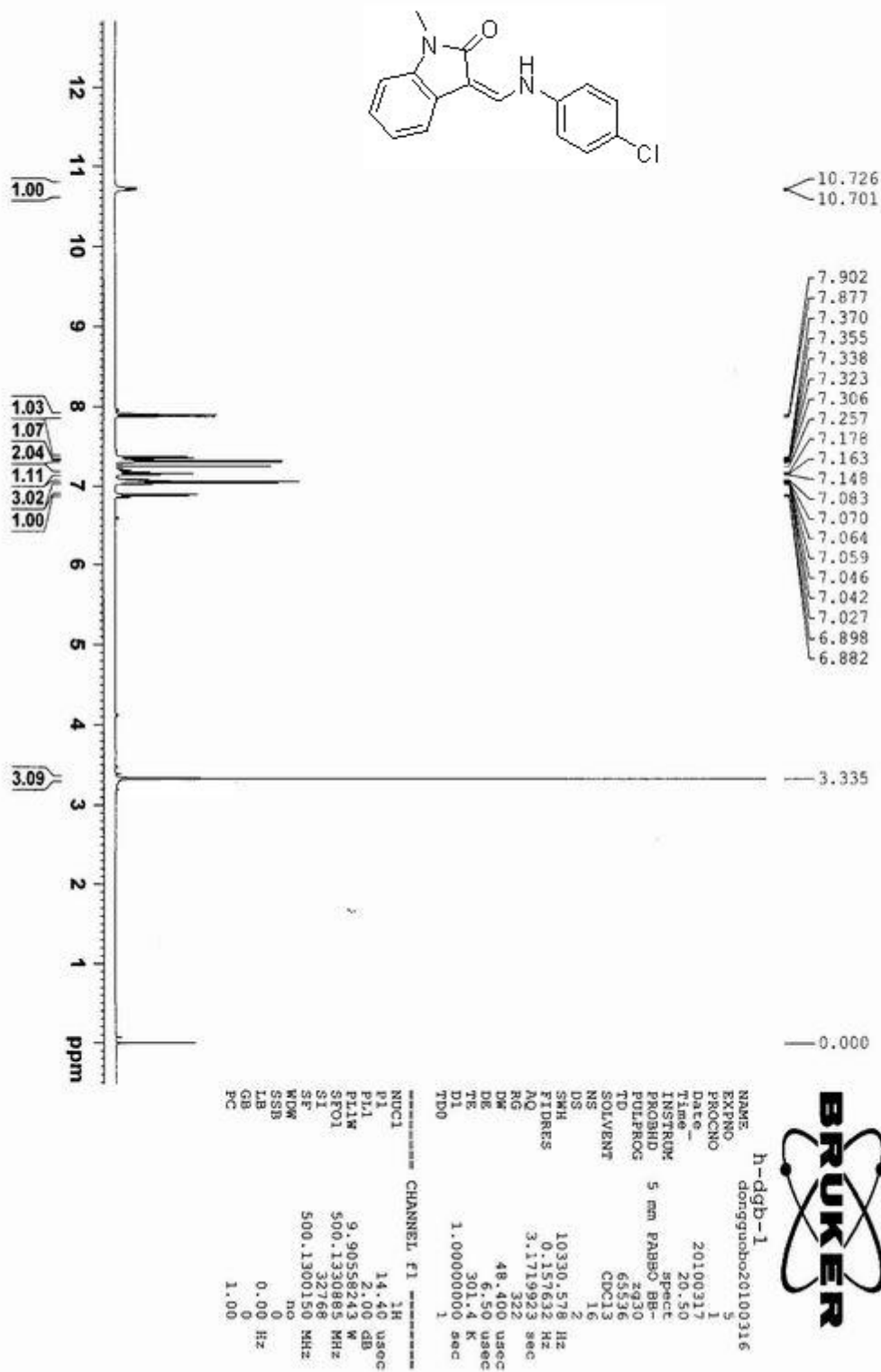
(Z) and (E)-1-Methyl-3-((phenylamino)methylene)indolin-2-one (20)



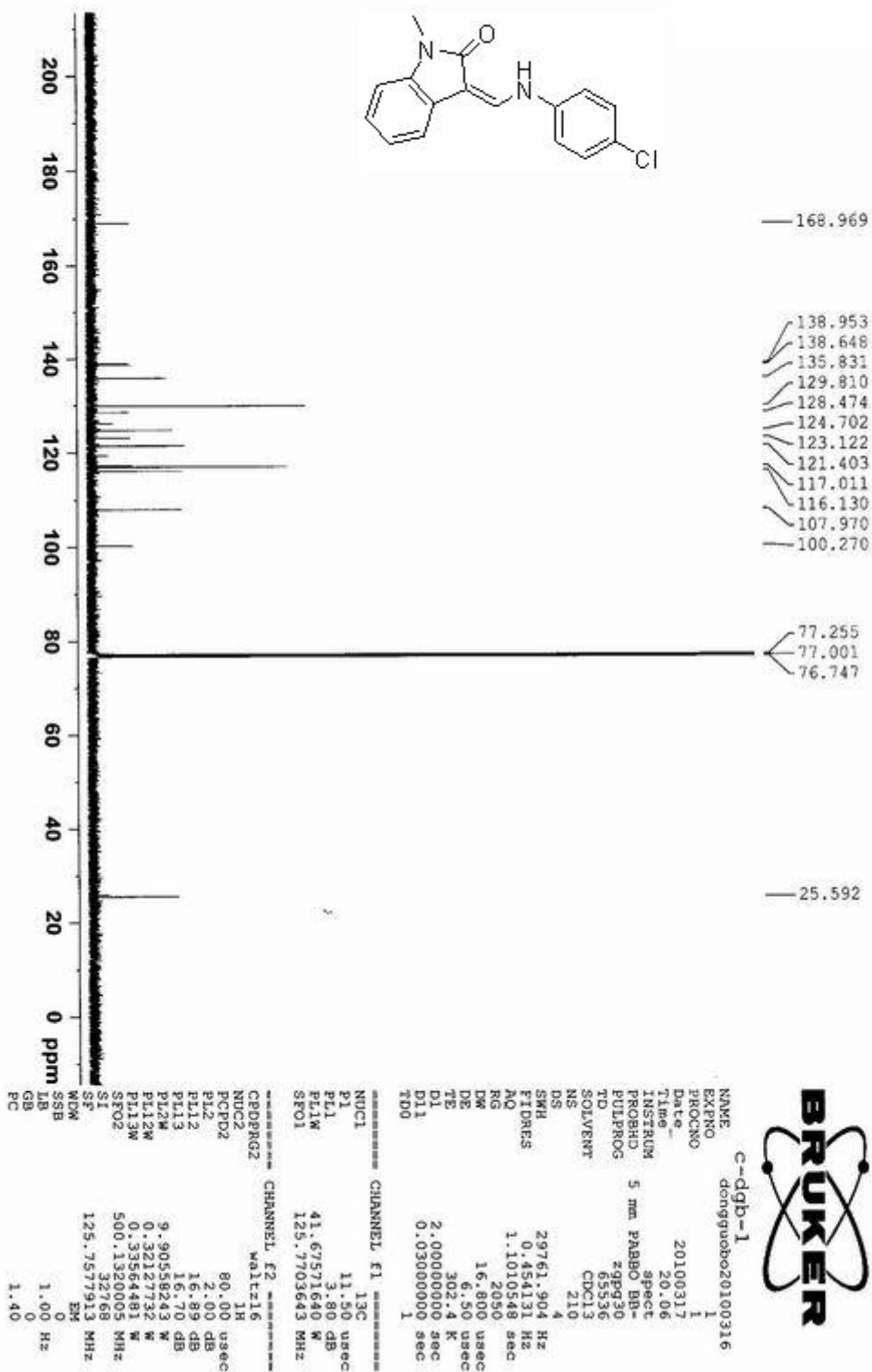
(Z) and (E)-1-Methyl-3-((phenylamino)methylene)indolin-2-one (20)



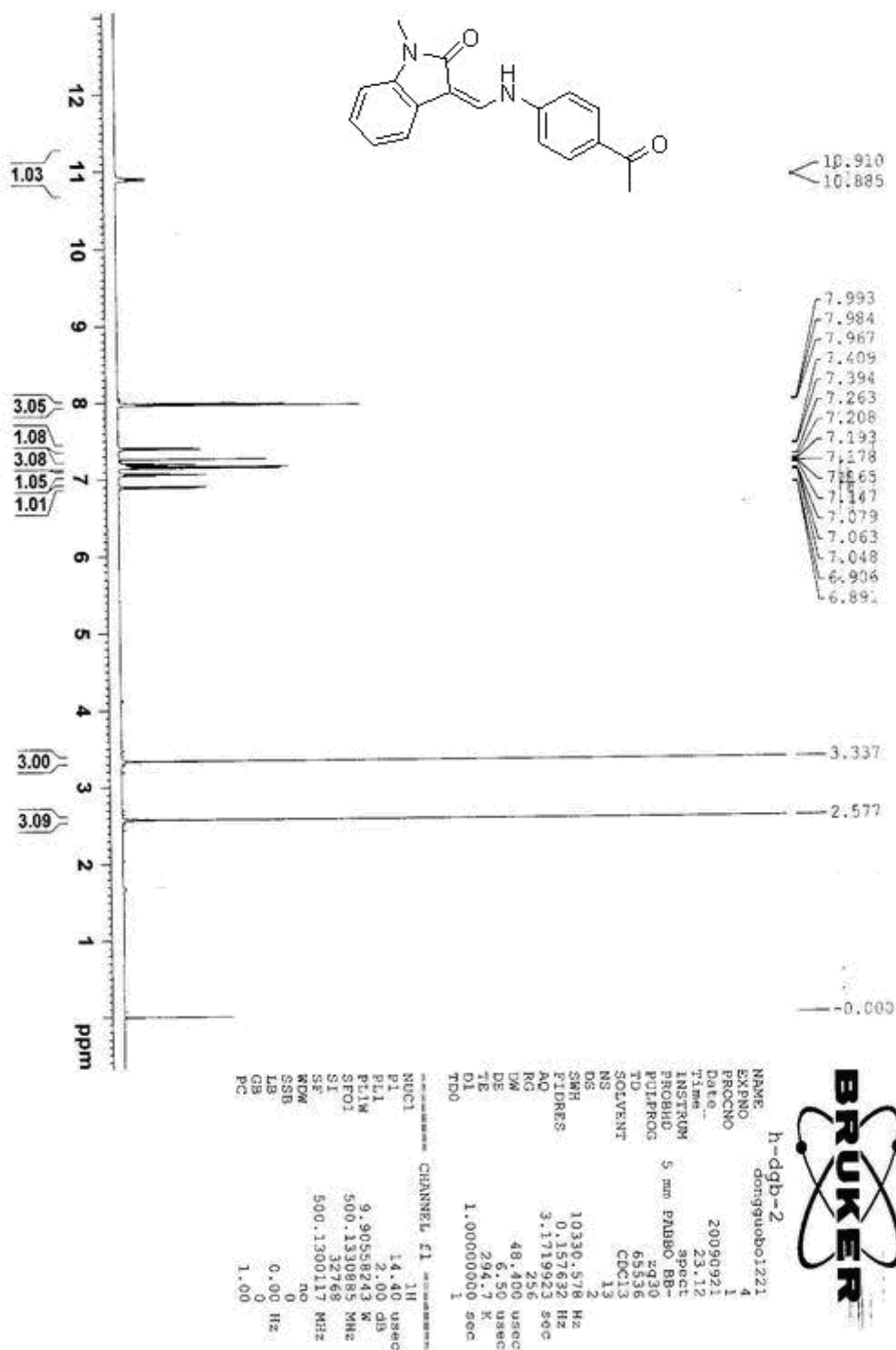
(Z)-3-((4-Chlorophenylamino)methylene)-1-methylindolin-2-one (21)



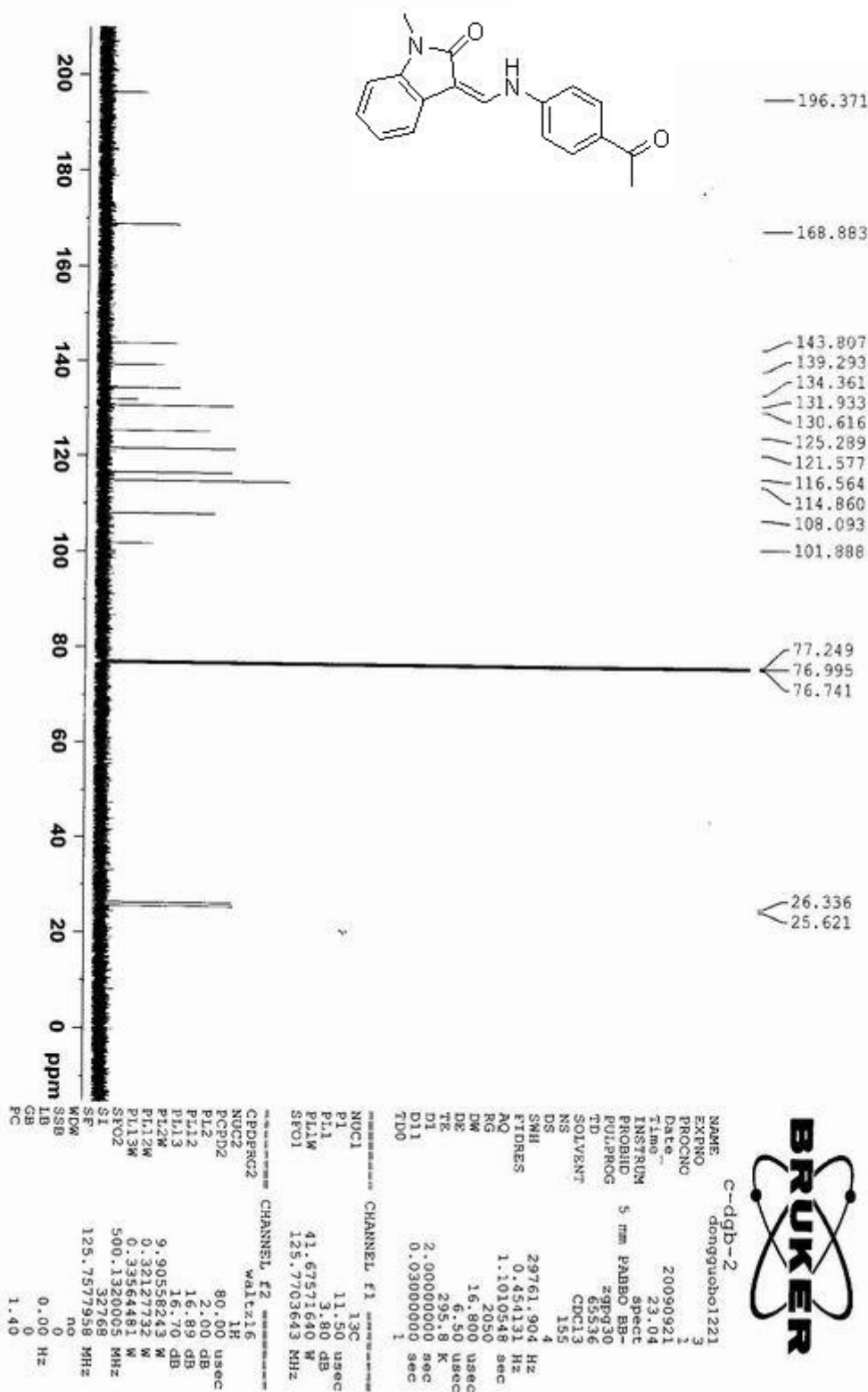
(Z)-3-((4-Chlorophenylamino)methylene)-1-methylindolin-2-one (21)



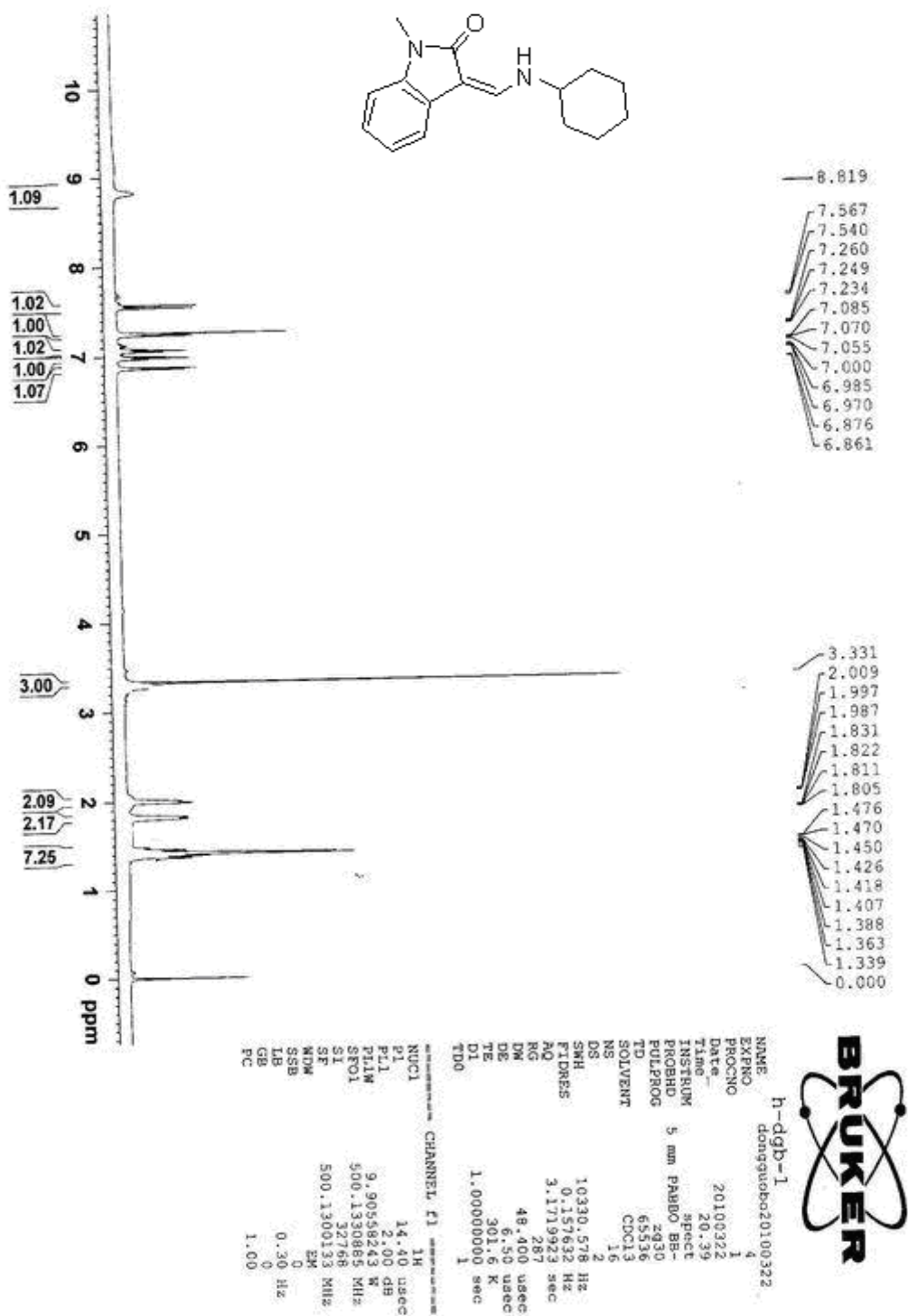
(Z)-3-((4-Acetylphenylamino)methylene)-1-methylindolin-2-one (22)



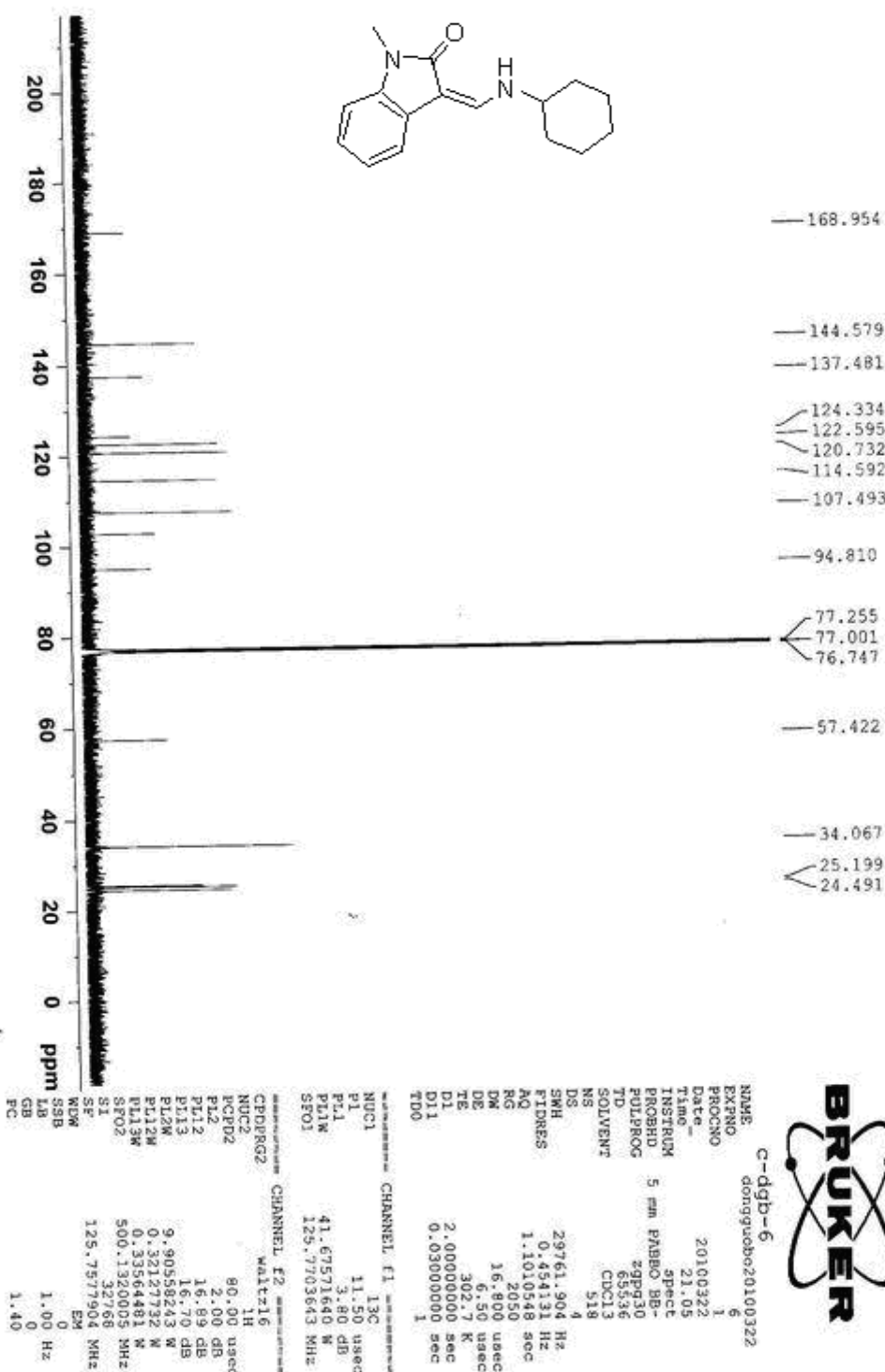
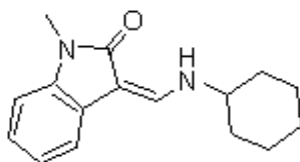
(Z)-3-((4-Acetylphenylamino)methylene)-1-methylindolin-2-one (22)



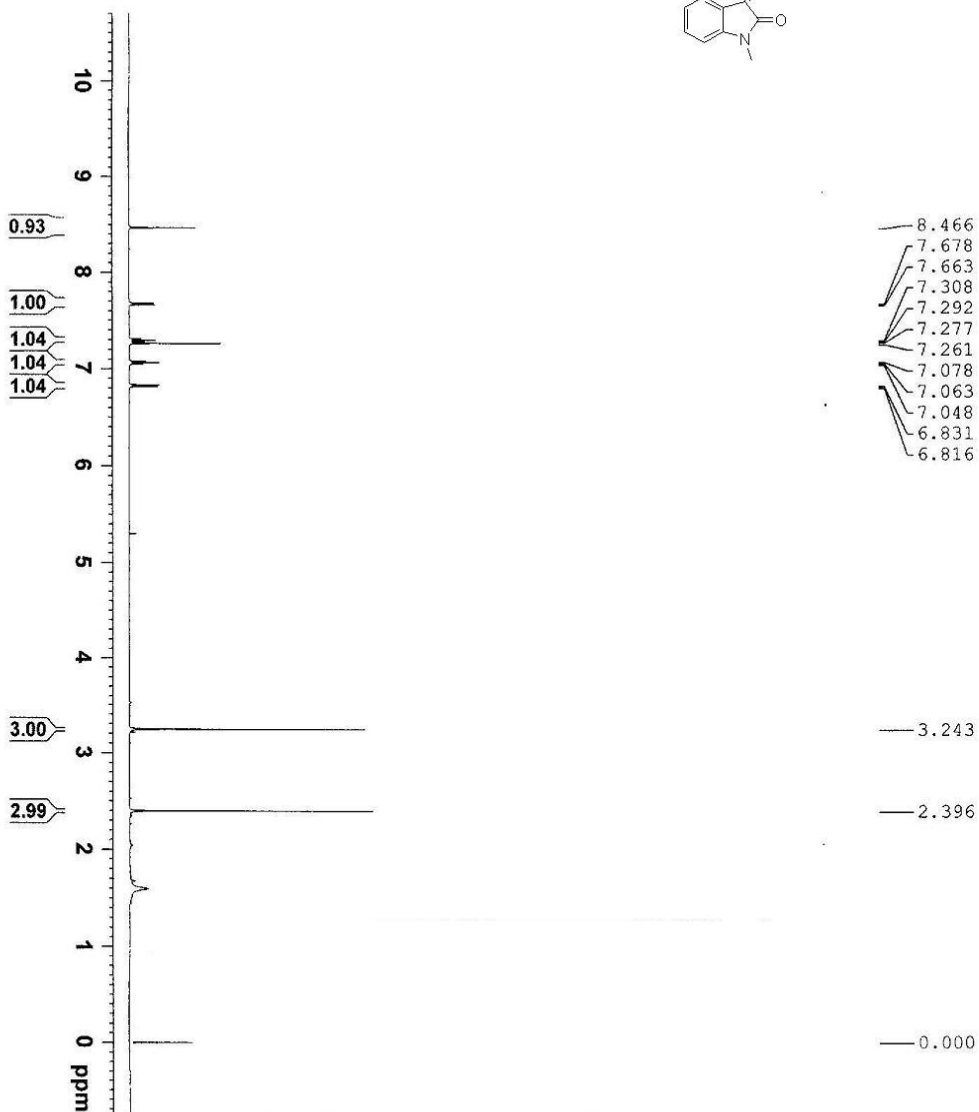
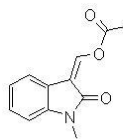
(Z)-3-((Cyclohexylamino)methylene)-1-methylindolin-2-one (23)



(Z)-3-((Cyclohexylamino)methylene)-1-methylindolin-2-one (23)



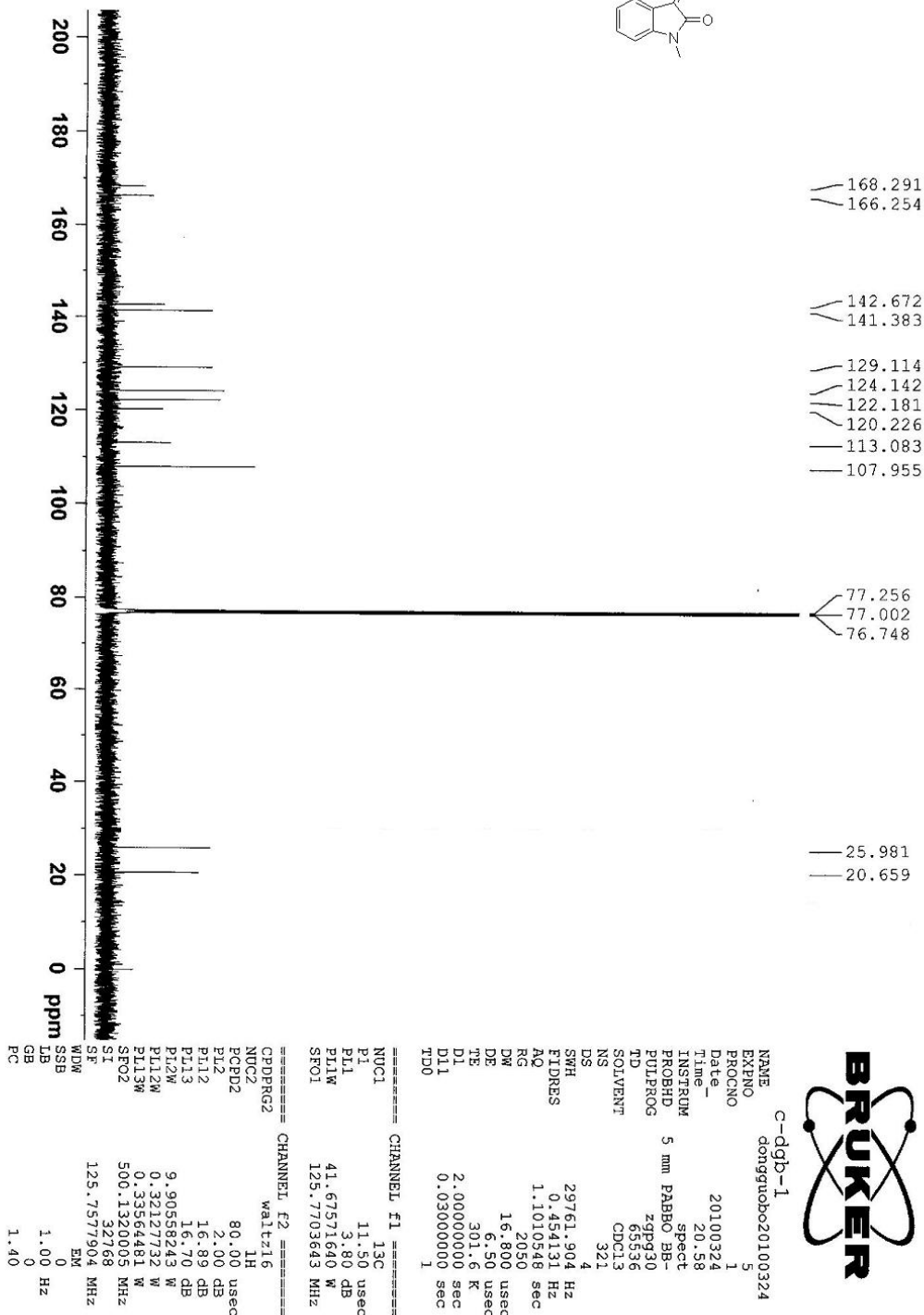
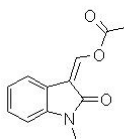
(Z)-(1-methyl-2-oxindolin-3-ylidene)methyl acetate (24)



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 PROCNO 1
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 TIME_ 20.47
 INSTRUM spect
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 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 10330.578 Hz
 FIDRES 0.157632 Hz
 AQ 3.1719923 sec
 RG 287
 DW 48.400 usec
 DE 6.50 usec
 TE 300.2 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 14.40 usec
 PL1 2.00 dB
 PL1W 9.90558243 W
 SFO1 500.1330885 MHz
 SI 32768
 SF 500.1300129 MHz
 WDW EM
 SSB 0
 TB 0
 GB 0
 PC 1.00

(Z)-(1-methyl-2-oxoindolin-3-ylidene)methyl acetate (24)



(E) The X-ray single-crystal diffraction analysis of product 3

