

Supporting Information

© Copyright Wiley-VCH Verlag GmbH & Co. KGaA, 69451 Weinheim, 2013

Supporting Information

Copper(II)-Catalyzed Aerobic Oxidative Coupling between Chalcone and 2-Aminopyridine *via* C-H Amination: An Expedient Synthesis of 3-Aroylimidazo[1,2-*a*]pyridines

Kamarul Monir,^a Avik Kumar Bagdi,^a Subhajit Mishra,^a Adinath Majee,^a and Alakananda Hajra*^a

^a Department of Chemistry, Visva-Bharati (A Central University), Santiniketan-731235, India

Table of contents:

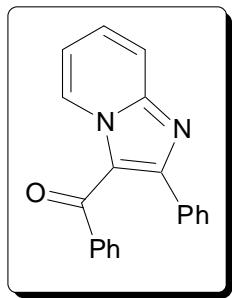
1.	Experimental Section	S2
	A. General	S2
	B. General Experimental Procedure	S2-S4
	C. Spectroscopic Data	S4
2.	References	S10
3.	¹H and ¹³C NMR spectra of Compounds	S11-S47

1. Experimental Section

A. General:

¹H NMR spectra were determined on a Bruker 400 (400 MHz) and Bruker 300 (300 MHz) spectrometer as solutions in CDCl₃. Chemical shifts are expressed in parts per million (δ) and the signals were reported as s (singlet), d (doublet), t (triplet), m (multiplet) and coupling constants J were given in Hz. ¹³C NMR spectra were recorded at 100 MHz and 75 MHz in CDCl₃ solution. Chemical shifts are expressed in parts per million (δ) and are referenced to CDCl₃ ($\delta = 77.16$) as internal standard. TLC was done on silica gel coated glass slide (Merck, Silica gel G for TLC). All solvents were dried and distilled before use. Commercially available substrates were freshly distilled before the reaction. Solvents, reagents and chemicals were purchased from Aldrich, Fluka, Merck, SRL, Spectrochem and Process Chemicals. All reactions involving moisture sensitive reactants were executed using oven dried glassware. The synthesis of chalcone was carried out according to the reported method.¹

B. General procedure for synthesis of imidazo[1,2-a]pyridine derivatives:

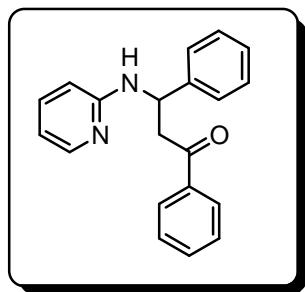


Phenyl-(2-phenyl-imidazo[1,2-a]pyridin-3-yl)-methanone (3a):

A mixture of 2-aminopyridine (**1a**, 1.2 mmol), chalcone(**2a**, 1 mmol), Cu(OAc)₂.H₂O (0.1 mmol), and 1,10-Phenanthroline (0.1 mmol) in 1,2-dichlorobenzene (2 mL) was stirred in a reaction tube at 120 °C for 12 hours using O₂ balloon. The reaction was monitored by TLC. After completion of the reaction, it was cooled to room temperature. Then it was filtered and washed with dichloromethane. The filtrate was concentrated and the crude product was purified by column chromatography on silica gel using petroleum ether/ethyl acetate (2:1) as eluent. Yield:

84%; ^1H NMR (CDCl_3 , 400 MHz) δ : 9.54 (d, J = 6.8 Hz, 1H), 7.80 (d, J = 9.2 Hz, 1H), 7.51 (d, J = 8.8 Hz, 2H), 7.3 (d, J = 9.6 Hz, 2H), 7.28-7.26 (m, 1H), 7.18-7.01 (m, 7H); ^{13}C NMR (CDCl_3 , 100 MHz) δ : 187.2, 154.8, 147.2, 138.4, 133.8, 131.6, 130.0, 129.4, 129.1, 128.1, 127.6, 127.4 (2C), 119.8, 117.3, 114.5. Anal. Calcd. For (%) $\text{C}_{20}\text{H}_{14}\text{N}_2\text{O}$: C, 80.52; H, 4.73; N, 9.39. Found C, 80.51; H, 4.63; N, 9.41.

Typical procedure for synthesis of Michael adduct 1,3-diphenyl-3-(pyridin-2-ylamino)-propan-1-one (A):



1,3-Diphenyl-3-(pyridin-2-ylamino)-propan-1-one (A):

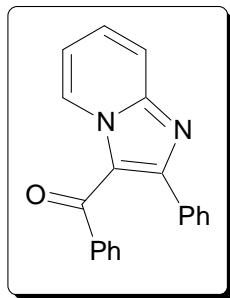
A mixture of 2-aminopyridine (**1a**, 1.2mmol), chalcone (**2a**, 1mmol) in 1,2 dichlorobenzene (2mL) was stirred in a reaction tube under ambient air at 120 °C for 18 hours. The reaction was monitored by TLC. After completion of the reaction, it was cooled to room temperature. Then it was filtered and washed with dichloromethane. The filtrate was concentrated and the crude product was purified by column chromatography on silica gel using petroleum ether/ethyl acetate (2:1) as eluent. Yield: 48%. ^1H NMR (CDCl_3 , 400 MHz) δ : 7.96 (d, J = 6.0 Hz, 1H), 7.84-7.81 (m, 2H), 7.48-7.41 (m, 1H), 7.39-7.32 (m, 4H), 7.29-7.20 (m, 3H), 7.19-7.17 (m, 1H), 6.47-6.44 (m, 1H), 6.27 (d, J = 8.4 Hz, 1H), 5.34-5.33 (m, 2H), 3.59-3.53 (m, 1H), 3.41-3.36 (m, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ : 197.9, 157.7, 148.0, 142.4, 137.4, 136.7, 133.2, 128.6, 128.5, 128.1, 127.2, 126.4, 113.3, 107.4, 52.5, 45.6. Anal. Calcd. For (%) $\text{C}_{20}\text{H}_{18}\text{N}_2\text{O}$: C, 79.44; H, 6.00; N, 9.26. Found C, 79.53; H, 6.12; N, 9.13.

Typical procedure for synthesis of phenyl-(2-phenyl-imidazo[1,2-a]pyridin-3-yl)-methanone (3a**) from 1,3-diphenyl-3-(pyridin-2-ylamino)-propan-1-one (A):**

A mixture of 1,3-diphenyl-3-(pyridin-2-ylamino)-propan-1-one (**A**, 0.25mmol), Cu(OAc)₂.H₂O (0.025 mmol) and 1,10-Phenanthroline (0.025 mmol) in 1,2-dichlorobenzene (1 mL) was stirred in a reaction tube with O₂ balloon at 120 °C for 12 hours. The reaction was monitored by TLC. After completion of the reaction, it was cooled to room temperature. Then it was filtered and washed with dichloromethane. The filtrate was concentrated and the crude product was purified by short column chromatography on silica gel to afford the compound **3a**.

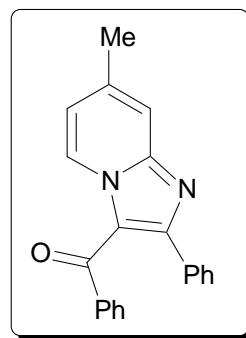
C. Spectroscopic Data:

Phenyl-(2-phenyl-imidazo[1,2-a]pyridin-3-yl)-methanone (Table 2, 3a):



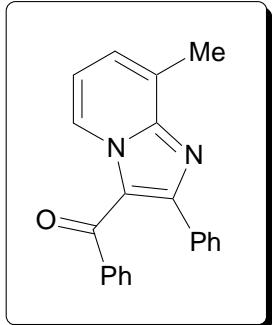
Yield: 250 mg, 84%; Gummy mass; ¹H NMR (CDCl₃, 400 MHz) δ : 9.54 (d, *J* = 6.8 Hz, 1H), 7.80 (d, *J* = 9.2 Hz, 1H), 7.51 (d, *J* = 8.8 Hz, 2H), 7.3 (d, *J* = 9.6 Hz, 2H), 7.28-7.26 (m, 1H), 7.18-7.01 (m, 7H) ; ¹³C NMR (CDCl₃, 100 MHz) δ : 187.2, 154.8, 147.2, 138.4, 133.8, 131.6, 130.0, 129.4, 129.1, 128.1, 127.6, 127.4 (2C), 119.8, 117.3, 114.5 ; Anal. Calcd. For (%) C₂₀H₁₄N₂O: C, 80.52; H, 4.73; N, 9.39. Found C, 80.51; H, 4.63; N, 9.41.

(7-Methyl-2-phenyl-imidazo[1,2-a]pyridin-3-yl)-phenyl-methanone (Table 2, 3b):



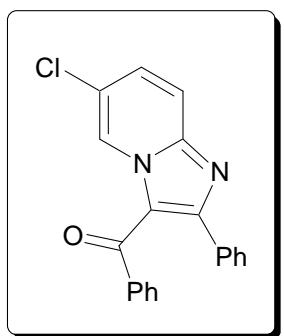
Yield: 249 mg, 80%; Gummy mass; ¹H NMR (CDCl₃, 400 MHz) δ : 9.35 (d, *J* = 7.2, 1H), 7.53 (s, 1H), 7.40 (d, *J* = 8.8 Hz, 2H), 7.22-7.14 (m, 3H), 7.05-6.96 (m, 5H), 6.86 (d, *J* = 6.8 Hz, 1H), 2.43 (s, 3H) ; ¹³C NMR (CDCl₃, 100 MHz) δ : 187.0, 154.9, 141.0, 138.6, 133.7, 131.5, 130.1, 129.4, 128.1, 127.6 (2C), 127.4, 119.6, 117.2, 115.9, 21.5; Anal. Calcd. For (%) C₂₁H₁₆N₂O: C, 80.75; H, 5.16; N, 8.97. Found C, 80.65; H, 5.14; N, 8.99.

(8-Methyl-2-phenyl-imidazo[1,2-a]pyridin-3-yl)-phenyl-methanone (Table 2, 3c):



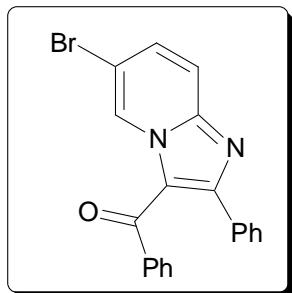
Yield: 243 mg, 78%; Gummy mass; ^1H NMR (CDCl_3 , 400 MHz) δ : 9.29 (d, $J = 6.8$ Hz, 1H), 7.38 (d, $J = 8.4$ Hz, 2H), 7.22 (d, $J = 7.6$ Hz, 2H), 7.18-7.09 (m, 2H), 7.00-6.92 (m, 5H), 6.86 (t, $J = 6.8$ Hz, 1H), 2.61 (s, 3H); ^{13}C NMR (CDCl_3 , 100 MHz) δ : 187.2, 154.3, 147.4, 138.6, 134.1, 131.4, 130.1, 129.3, 127.9, 127.5, 127.5, 127.2, 125.7, 120.9, 114.4, 16.9; Anal. Calcd. For (%) $\text{C}_{21}\text{H}_{16}\text{N}_2\text{O}$: C, 80.75; H, 5.16; N, 8.97. Found C, 80.68; H, 5.26; N, 8.80.

(6-Chloro-2-phenyl-imidazo[1,2-a]pyridin-3-yl)-phenyl-methanone (Table 2, 3d):



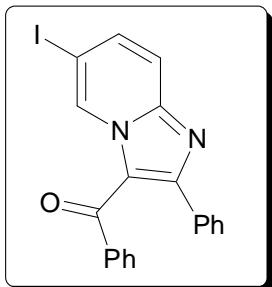
Yield: 269 mg, 81%; Gummy mass; ^1H NMR (CDCl_3 , 400 MHz) δ : 9.54 (s, 1H), 7.67 (d, $J = 9.6$ Hz, 1H), 7.44-7.41 (m, 3H), 7.25-7.19 (m, 3H), 7.06-7.00 (m, 5H); ^{13}C NMR (CDCl_3 , 100 MHz) δ : 187.4, 155.2, 145.7, 138.3, 133.6, 132.1, 130.5, 130.2, 129.8, 128.6, 127.9, 126.3, 123.0, 120.3, 117.8; Anal. Calcd. For (%) $\text{C}_{20}\text{H}_{13}\text{ClN}_2\text{O}$: C, 72.18; H, 3.94; N, 8.42. Found C, 72.23; H, 4.01; N, 8.31.

(6-Bromo-2-phenyl-imidazo[1,2-a]pyridin-3-yl)-phenyl-methanone (Table 2, 3e):



Yield: 286 mg, 76%; Gummy mass; ^1H NMR (CDCl_3 , 400 MHz) δ : 9.70 (s, 1H), 7.71 (d, $J = 9.6$ Hz, 1H), 7.58 (d, $J = 9.2$ Hz, 1H), 7.50 (d, $J = 8.0$ Hz, 2H), 7.32-7.25 (m, 3H), 7.15-7.06 (m, 5H); ^{13}C NMR (CDCl_3 , 100 MHz) δ : 187.3, 154.8, 145.7, 141.2, 138.1, 133.4, 132.6, 132.1, 130.2, 129.6, 128.5, m128.3, 127.8, 117.9, 115.5, 109.5; Anal. Calcd. For (%) $\text{C}_{20}\text{H}_{13}\text{BrN}_2\text{O}$: C, 63.68; H, 3.47; N, 7.43. Found C, 63.80; H, 3.45; N, 7.47.

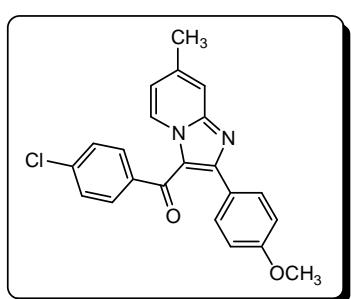
(6-Iodo-2-phenyl-imidazo[1,2-a]pyridin-3-yl)-phenyl-methanone (Table 2, 3f):



6.75

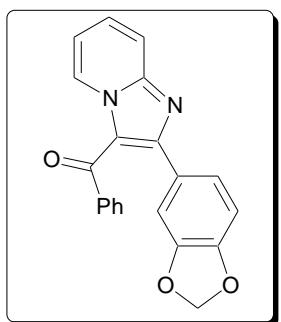
Yield: 335 mg, 79%; Gummy mass; ^1H NMR (CDCl_3 , 400 MHz) δ : 9.78 (s, 1H), 8.09 (d, J = 6.4 Hz, 1H), 7.70-7.61 (m, 2H), 7.50-7.45 (m, 2H), 7.32-7.27 (m, 2H), 7.14-7.08 (m, 5H); ^{13}C NMR (CDCl_3 , 100 MHz) δ : 187.3, 154.3, 145.8, 138.1, 137.2, 133.0, 132.1, 130.2, 129.9, 129.6, 128.6, 128.3, 127.8, 119.7, 118.3, 78.0; Anal. Calcd. For (%) $\text{C}_{20}\text{H}_{13}\text{IN}_2\text{O}$: C, 56.62; H, 3.09; N, 6.60. Found C, 56.57; H, 3.29; N,

(4-Chloro-phenyl)-[2-(4-methoxy-phenyl)-7-methyl-imidazo[1,2-a]pyridin-3-yl]-methanone (Table 2, 3g):



Yield: 287 mg, 76%; Gummy mass; ^1H NMR (CDCl_3 , 400 MHz) δ : 9.31 (d, J = 6.8 Hz, 1H), 7.51 (s, 1H), 7.32 (d, J = 6.8 Hz, 2H), 7.11 (d, J = 6.8 Hz, 2H), 6.99 (d, J = 6.8 Hz, 2H), 6.85 (d, J = 7.2 Hz, 1H), 6.53 (d, J = 8.8 Hz, 2H), 3.66 (s, 3H), 2.43 (s, 3H); ^{13}C NMR (CDCl_3 , 100 MHz) δ : 185.5, 159.9, 154.8, 147.7, 141.3, 137.6, 137.1, 131.4, 130.8, 127.9, 127.3, 125.9, 119.2, 117.2, 115.9, 113.3, 55.2, 21.5; Anal. Calcd. For (%) $\text{C}_{22}\text{H}_{17}\text{ClN}_2\text{O}_2$: C, 70.12; H, 4.55; N, 7.43. Found C, 70.20; H, 4.67; N, 7.49.

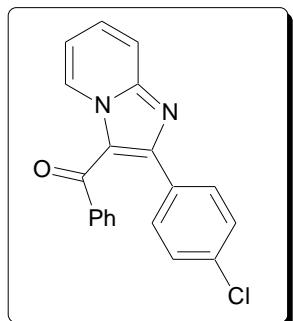
(2-Benzodioxol-5-yl-imidazo[1,2-a]pyridin-3-yl)-phenyl-methanone (Table 2, 3h):



Yield: 284 mg, 83%; Gummy mass; ^1H NMR (CDCl_3 , 400 MHz) δ : 9.49 (d, J = 6.8 Hz, 1H), 7.78 (d, J = 8.4 Hz, 1H), 7.52-7.49 (m, 3H), 7.29 (t, J = 7.6 Hz, 1H), 7.14 (t, J = 7.6 Hz, 2H), 7.05 (t, J = 7.6 Hz, 1H), 6.85 (s, 1H), 6.75 (d, J = 8.0 Hz, 1H), 6.48 (d, J = 8.0 Hz, 1H), 5.83 (s, 2H); ^{13}C NMR (CDCl_3 , 100 MHz) δ : 187.2, 154.3, 147.7, 147.2, 147.1, 138.7, 131.7, 129.4, 129.3, 128.1, 127.8, 127.6, 124.9, 119.6, 117.1,

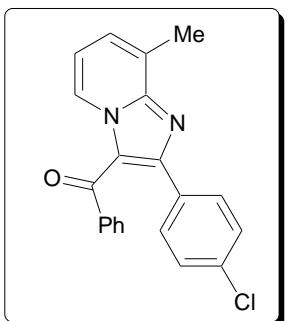
114.6, 110.1, 107.7, 101.0, 21.3; Anal. Calcd. For (%) C₂₁H₁₄N₂O₃: C, 73.68; H, 4.12; N, 8.18. Found C, 73.77; H, 4.25; N, 8.23.

[2-(4-Chloro-phenyl)-imidazo[1,2-a]pyridin-3-yl]-phenyl-methanone (Table 2, 3i):



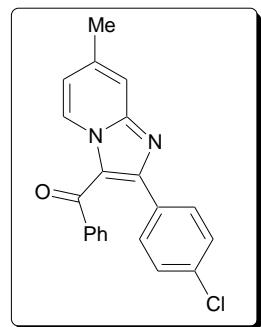
Yield: 269 mg, 81%; Gummy mass; ¹H NMR (CDCl₃, 400 MHz) δ : 9.44 (d, *J* = 6.8 Hz, 1H), 7.73 (d, *J* = 8.8 Hz, 1H), 7.46 (d, *J* = 8.4 Hz, 1H), 7.42 (d, *J* = 8.0 Hz, 2H), 7.24 (t, *J* = 7.6 Hz, 1H), 7.17 (d, *J* = 8.8 Hz, 2H), 7.07-7.02 (m, 3H), 6.98 (d, *J* = 8.4 Hz, 2H); ¹³C NMR (CDCl₃, 100 MHz) δ : 187.0, 153.3, 147.3, 138.4, 134.4, 132.4, 131.9, 131.3, 129.4, 129.3, 128.1, 127.8, 119.9, 117.3, 114.7; Anal. Calcd. For (%) C₂₀H₁₃ClN₂O: C, 72.18; H, 3.94; N, 8.42. Found C, 72.24; H, 4.13; N, 8.46.

[2-(4-Chlorophenyl)-8-methyl-imidazo[1,2-a]pyridin-3-yl]-phenyl-methanone (Table 2, 3j):



Yield: 263 mg, 76%; Gummy mass; ¹H NMR (CDCl₃, 400 MHz) δ : 9.41 (d, *J* = 6.4 Hz, 1H), 7.50 (d, *J* = 7.2 Hz, 2H), 7.36-7.27 (m, 4H), 7.14 (t, *J* = 7.6 Hz, 2H), 7.08-7.02 (m, 3H), 2.74 (s, 3H); ¹³C NMR (CDCl₃, 100 MHz) δ : 187.2, 153.1, 138.6, 134.2, 132.8, 131.8, 131.4, 129.4, 128.2, 127.9, 127.8, 127.5, 125.9, 114.8, 17.0; Anal. Calcd. For (%) C₂₁H₁₅ClN₂O: C, 72.73; H, 4.36; N, 8.08. Found C, 72.81; H, 4.51; N, 8.13.

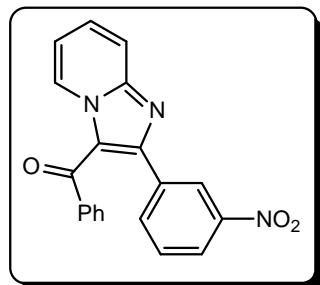
[2-(4-Chlorophenyl)-7-methyl-imidazo[1,2-a]pyridin-3-yl]-phenyl-methanone (Table 2, 3k):



Yield: 270 mg, 78%; Gummy mass; ¹H NMR (CDCl₃, 400 MHz) δ : 9.43 (d, *J* = 7.2 Hz, 1H), 7.56 (s, 1H), 7.48 (d, *J* = 8.0 Hz, 2H), 7.32 (t, *J* = 7.6 Hz, 1H), 7.24 (d, *J* = 8.4 Hz, 2H), 7.13 (t, *J* = 8.0 Hz, 2H), 7.06 (d, *J* = 8.4 Hz, 2H), 6.94 (d, *J* = 7.2 Hz, 1H), 2.52 (s, 3H); ¹³C NMR (CDCl₃, 100 MHz) δ : 186.8, 153.7, 147.8, 141.0, 138.6, 134.3, 132.6, 131.7, 131.3,

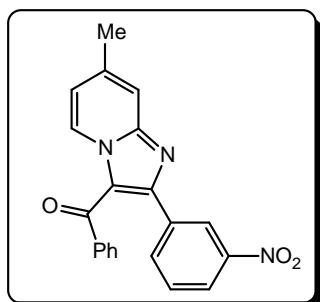
129.4, 127.8, 127.4, 119.7, 117.2, 116.0, 21.5; Anal. Calcd. For (%) C₂₁H₁₅ClN₂O: C, 72.73; H, 4.36; N, 8.08. Found C, 72.85; H, 4.51; N, 8.13.

[2-(3-Nitro-phenyl)-imidazo[1,2-a]pyridin-3-yl]-phenyl-methanone (Table 2, 3l):



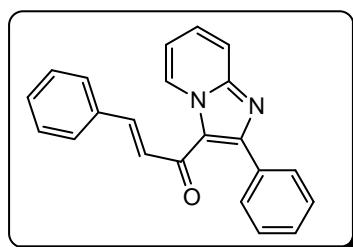
Yield: 277 mg, 81%; Gummy mass; ¹H NMR (CDCl₃, 400 MHz) δ : 9.47 (d, *J* = 7.2 Hz, 1H), 8.02 (s, *J* = 9.0 Hz, 1H), 7.90 (d, *J* = 8.8 Hz, 1H), 7.75 (d, *J* = 8.8 Hz, 1H), 7.62 (d, *J* = 8.0 Hz, 1H), 7.50 (t, *J* = 8.4 Hz, 1H), 7.40 (d, *J* = 8.4 Hz, 2H), 7.23-7.14 (m, 2H), 7.08 (t, *J* = 7.6 Hz, 1H), 7.0 (t, *J* = 8.0 Hz, 2H); ¹³C NMR (CDCl₃, 100 MHz) δ : 186.9, 152.0, 147.5, 147.4, 138.4, 135.7, 132.1, 129.9, 129.4, 128.8, 128.4, 128.0, 125.3, 122.9, 120.4, 117.6, 115.3; Anal. Calcd. For (%) C₂₀H₁₃N₃O₃: C, 69.96; H, 3.82; N, 12.24. Found C, 70.13; H, 3.95; N, 12.29.

[7-Methyl-2-(3-nitro-phenyl)-imidazo[1,2-a]pyridin-3-yl]-phenyl-methanone (Table 2, 3m):



Yield: 285 mg, 80%; Gummy mass; ¹H NMR (CDCl₃, 400 MHz) δ : 9.42 (d, *J* = 7.2 Hz, 1H), 8.05 (s, 1H), 7.94 (d, *J* = 7.2 Hz, 1H), 7.67 (d, *J* = 7.6 Hz, 1H), 7.55 (s, 1H), 7.43 (d, *J* = 7.2 Hz, 2H), 7.28-7.18 (m, 2H), 7.05 (t, *J* = 7.6 Hz, 2H), 6.96 (d, *J* = 7.2 Hz, 1H), 2.50 (s, 3H); ¹³C NMR (CDCl₃, 100 MHz) δ : 186.6, 152.2, 147.8, 147.3, 141.6, 138.6, 135.8, 135.6, 131.8, 129.3, 128.7, 127.9, 127.5, 125.2, 122.8, 120.1, 117.8, 116.1, 21.6; Anal. Calcd. For (%) C₂₁H₁₅N₃O₃: C, 70.58; H, 4.23; N, 11.76. Found C, 70.63; H, 4.33; N, 11.86.

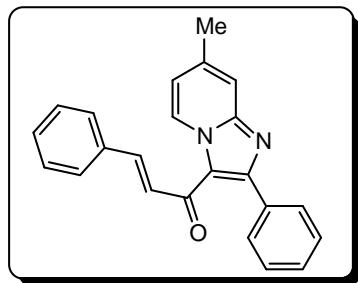
3-Phenyl-1-(2-phenyl-imidazo[1,2-a]pyridin-3-yl)-propenone (Table 2, 3n):



Yield: 269 mg, 83%; Gummy mass; ¹H NMR (CDCl₃, 400 MHz) δ : 9.81 (d, *J* = 7.2 Hz, 1H), 7.79 (d, *J* = 8.8 Hz, 1H), 7.70-7.64 (m, 3H), 7.52-7.45 (m, 4H), 7.25-7.20 (m, 3H), 7.09 (d, *J* = 6.8 Hz, 2H), 7.05 (t, *J* = 6.8 Hz, 1H), 6.83 (d, *J* = 15.6 Hz, 1H); ¹³C NMR (CDCl₃, 100 MHz) δ : 180.5, 154.2, 146.9, 141.0, 134.8,

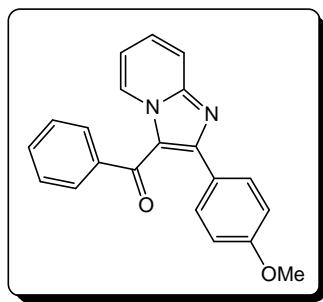
130.5, 129.9, 129.5, 129.2, 128.9, 128.6, 128.4, 128.0, 125.3, 122.0, 117.1, 114.8; Anal. Calcd. For (%) C₂₂H₁₆N₂O: C, 81.46; H, 4.97; N, 8.64. Found C, 81.55; H, 5.134; N, 8.77.

1-(7-Methyl-2-phenyl-imidazo[1,2-a]pyridin-3-yl)-3-phenyl-propenone (Table 2, 3o):



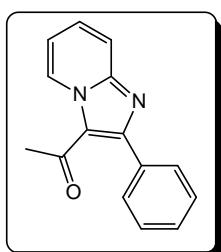
Yield: 270 mg, 80%; Gummy mass; ¹H NMR (CDCl₃, 400 MHz) δ : 9.62 (d, *J* = 7.2 Hz, 1H), 7.61-7.54 (m, 3H), 7.44-7.39 (m, 4H), 7.18-7.12 (m, 3H), 7.01 (d, *J* = 6.8 Hz, 2H), 6.81 (d, *J* = 7.2 Hz, 1H), 6.75 (d, *J* = 15.6Hz, 1H), 2.37 (s, 3H) ; ¹³C NMR (CDCl₃, 100 MHz) δ : 180.2, 154.8, 147.6, 141.2, 140.6, 135.2, 134.9, 130.5, 129.9, 129.2, 128.7, 128.4, 128.2, 128.1, 125.4, 121.9, 117.3, 115.9, 21.5; Anal. Calcd. For (%) C₂₃H₁₈N₂O: C, 81.63; H, 5.36; N, 8.28. Found C, 81.71; H, 5.47; N, 8.32.

[2-(4-Methoxy-phenyl)-imidazo[1,2-a]pyridin-3-yl]-phenyl-methanone (Table 2, 3p):



Yield: 242 mg, 74%; Gummy mass; ¹H NMR (CDCl₃, 300 MHz) δ : 9.48 (d, *J* = 6.0 Hz, 1H), 7.79 (d, *J* = 7.2 Hz, 1H), 7.50-7.44 (m, 3H), 7.26-7.22 (m, 3H), 7.11-7.00 (m, 3H), 6.58 (d, *J* = 6.9 Hz, 2H), 3.67 (s, 3H) ; ¹³C NMR (CDCl₃, 75 MHz) δ : 187.4, 159.7, 154.6, 147.3, 146.3, 139.2, 138.6, 131.7, 131.5, 128.2, 127.8, 126.2, 119.6, 117.2, 114.5, 113.3, 55.2; Anal. Calcd. For (%) C₂₁H₁₆N₂O₂: C, 76.81; H, 4.91; N, 8.53. Found C, 76.79; H, 4.88; N, 8.50.

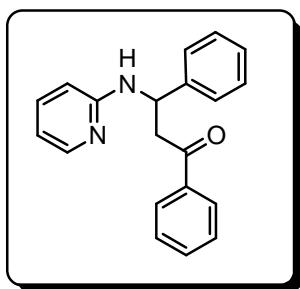
1-(2-Phenyl-imidazo[1,2-a]pyridin-3-yl)-ethanone (Table 2, 3q):



Yield: 61 mg, 26%; Gummy mass; ¹H NMR (CDCl₃, 400 MHz) δ : 9.80 (d, *J* = 7.2 Hz, 1H), 7.72 (d, *J* = 8.8 Hz, 1H), 7.47-7.45 (m, 3H), 7.19-7.17 (m, 2H), 7.07-7.05 (m, 2H), 2.13 (s, 3H) ; ¹³C NMR (CDCl₃, 100 MHz) δ : 192.0, 157.3, 145.9, 140.1, 139.0, 133.7, 133.0, 128.9, 128.5, 127.9, 123.2, 111.6, 21.8; Anal. Calcd. For (%) C₁₅H₁₂N₂O: C, 76.25; H, 5.12; N, 11.86. Found C,

76.21; H, 5.10; N, 11.83.

1,3-Diphenyl-3-(pyridin-2-ylamino)-propan-1-one (A):



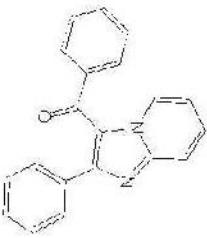
Yield: 145 mg, 48%; Gummy mass; ^1H NMR (CDCl_3 , 400 MHz) δ : 7.96 (d, $J = 6.0$ Hz, 1H), 7.84-7.81 (m, 2H), 7.48-7.41 (m, 1H), 7.39-7.32 (m, 4H), 7.29-7.20 (m, 3H), 7.19-7.17 (m, 1H), 6.47-6.44 (m, 1H), 6.27 (d, $J = 8.4$ Hz, 1H), 5.34-5.33 (m, 2H), 3.59-3.53 (m, 1H), 3.41-3.36 (m, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ : 197.9, 157.7, 148.0, 142.4, 137.4, 136.7, 133.2, 128.6, 128.5, 128.1, 127.2, 126.4, 113.3, 107.4, 52.5, 45.6; Anal.

Calcd. For (%) $\text{C}_{20}\text{H}_{18}\text{N}_2\text{O}$: C, 79.44; H, 6.00; N, 9.26. Found C, 79.53; H, 6.12; N, 9.13.

2. References:

1. *Vogel's Textbook of Practical Organic Chemistry* (5th Edition) [A.I. Vogel, A.R. Tatchell, B.S. Furnis, A.J. Hannaford, P.W.G. Smith], Page No: 1034.

7.541
 7.538
 7.520
 7.502
 7.498
 7.470
 7.452
 7.448
 7.380
 7.364
 7.360
 7.333
 7.330
 7.327
 7.319
 7.314
 7.310
 7.303
 7.282
 7.273
 7.270
 7.267
 7.264
 7.260
 7.252
 7.236
 7.233
 7.181
 7.153
 7.149
 7.146
 7.134
 7.130
 7.124
 7.117
 7.113
 7.105
 7.099
 7.085
 7.080
 7.067
 7.064
 7.059
 7.056
 7.050
 7.035
 7.031
 7.017



Current Data Parameters
 Dr. A MJE
 EXPNO 171
 PROTON

E2 - Acquisition Parameters

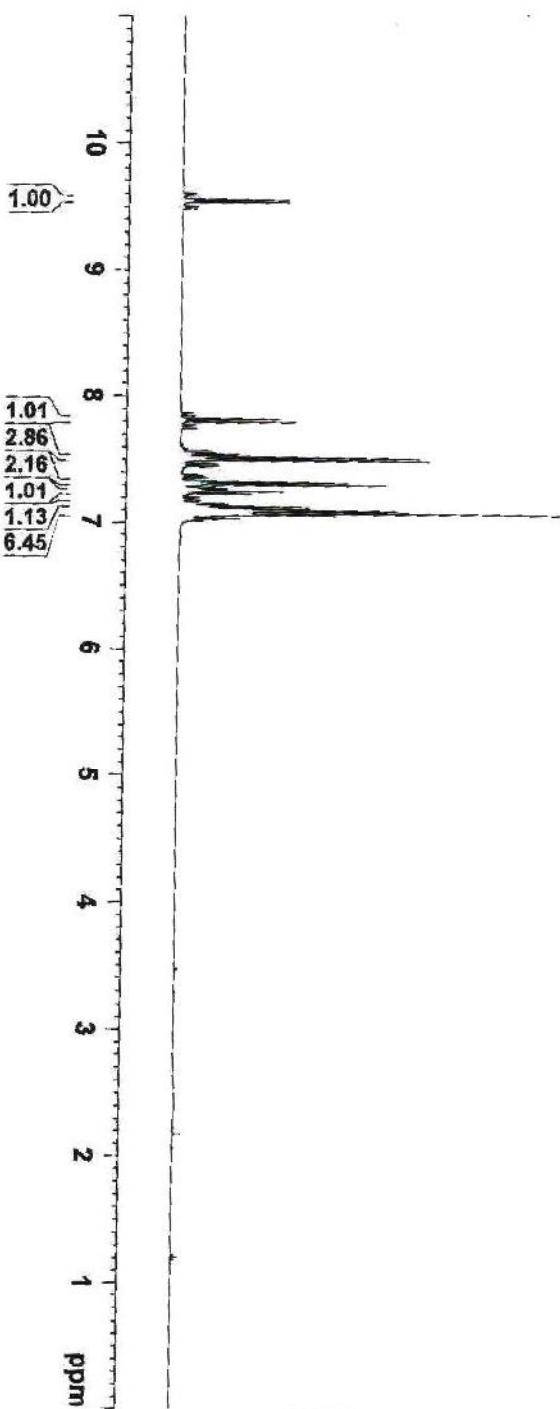
Date 2010217
 Time 16:51
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG z330
 TD 32768
 SCVLENT CDCl3
 NS 24
 DS 2
 SWH 8223.685 Hz
 FIDRES 0.250967 Hz
 AQ 1.923444 sec
 RG 37.83
 DR 60.00 usec
 DE 6.00 usec
 TE 291.1 K
 D1 1.000000 sec
 TDO

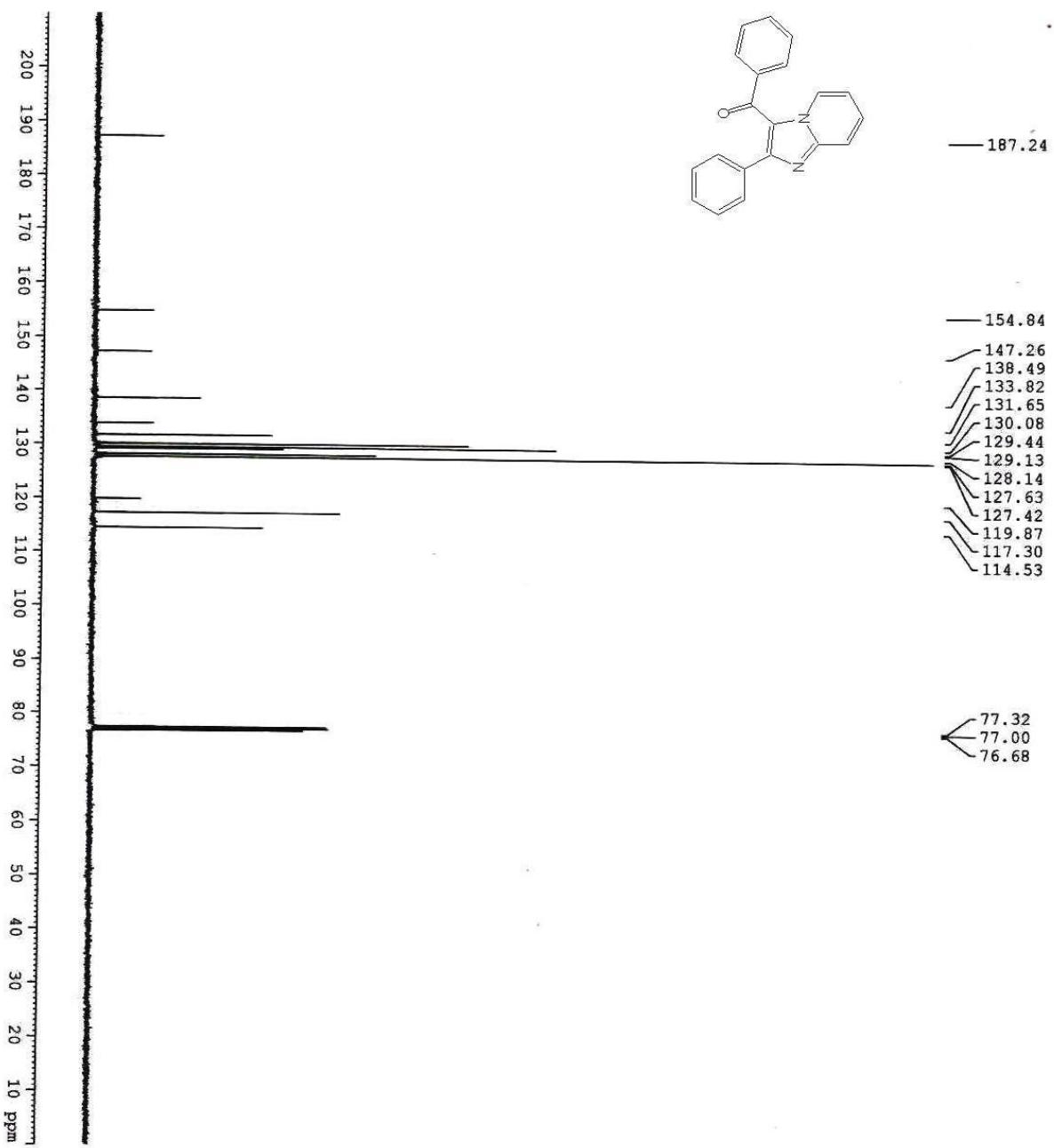
===== CHANNEL f1 =====

NUC1 1H
 PL 14.75 usec
 FWHM 11.9999989 W
 SPOL 400.1524711 MHz

F2 - Processing parameters

SI 16384
 SF 400.150000 MHz
 PDW EM
 SS3 0
 LB 0
 GA 0
 PC 1.00





Current Data Parameters
NAME Dr. A. Majee
EXPNO 112
PROCNO 1

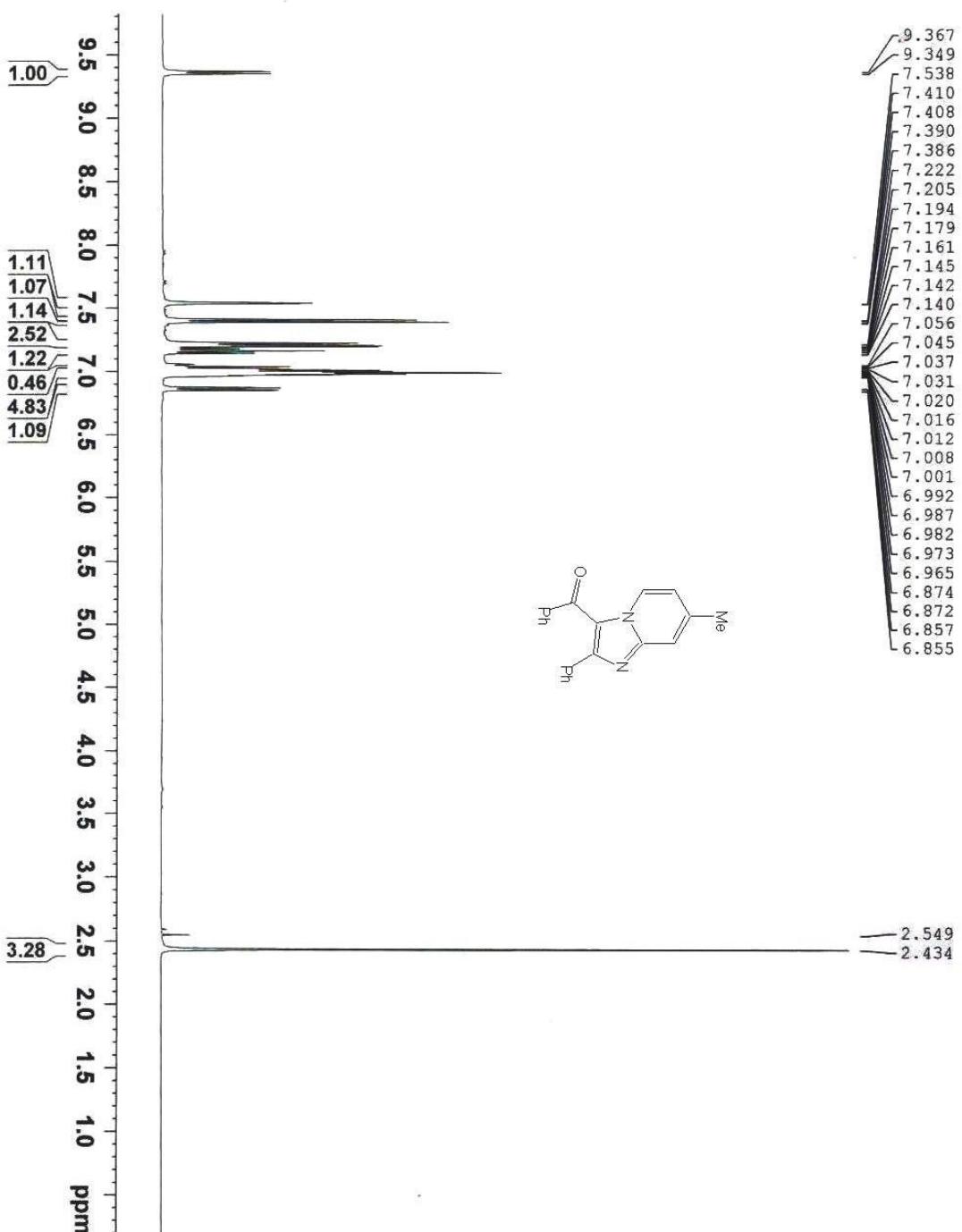
F2 - Acquisition Parameters
Date 2010217
Time 17.02
INSTRUM spect
PROBOD 5 mm PABBO BB/
EULFROG
TD 32768
SOLVENT CDCl₃
NS 160
DS 2
SWH 24038.461 Hz
ETRINES 0.733586 Hz
AQ 0.601624 sec
RG 37.83
DW 20.800 usec
DE 6.50 usec
TE 292.3 K
D1 2.0000000 sec
D11 0.03000000 sec
TDD0 1

NUC1 CHANNEL f1 13C
P1W1 8.90 usec
PFW1 54.0000000 W
SP01 10.6278588 MHz

CPDPRG2 CHANNEL f2 walt16
NUC2 1H
PCP02 80.00 usec
PFW2 12.0000000 W
PFW12 0.4079299 W
PFW13 0.26107001 W
SP02 400.1516006 MHz

F2 - Processing parameters
SI 16384
SF 100.6178100 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

BRUKER

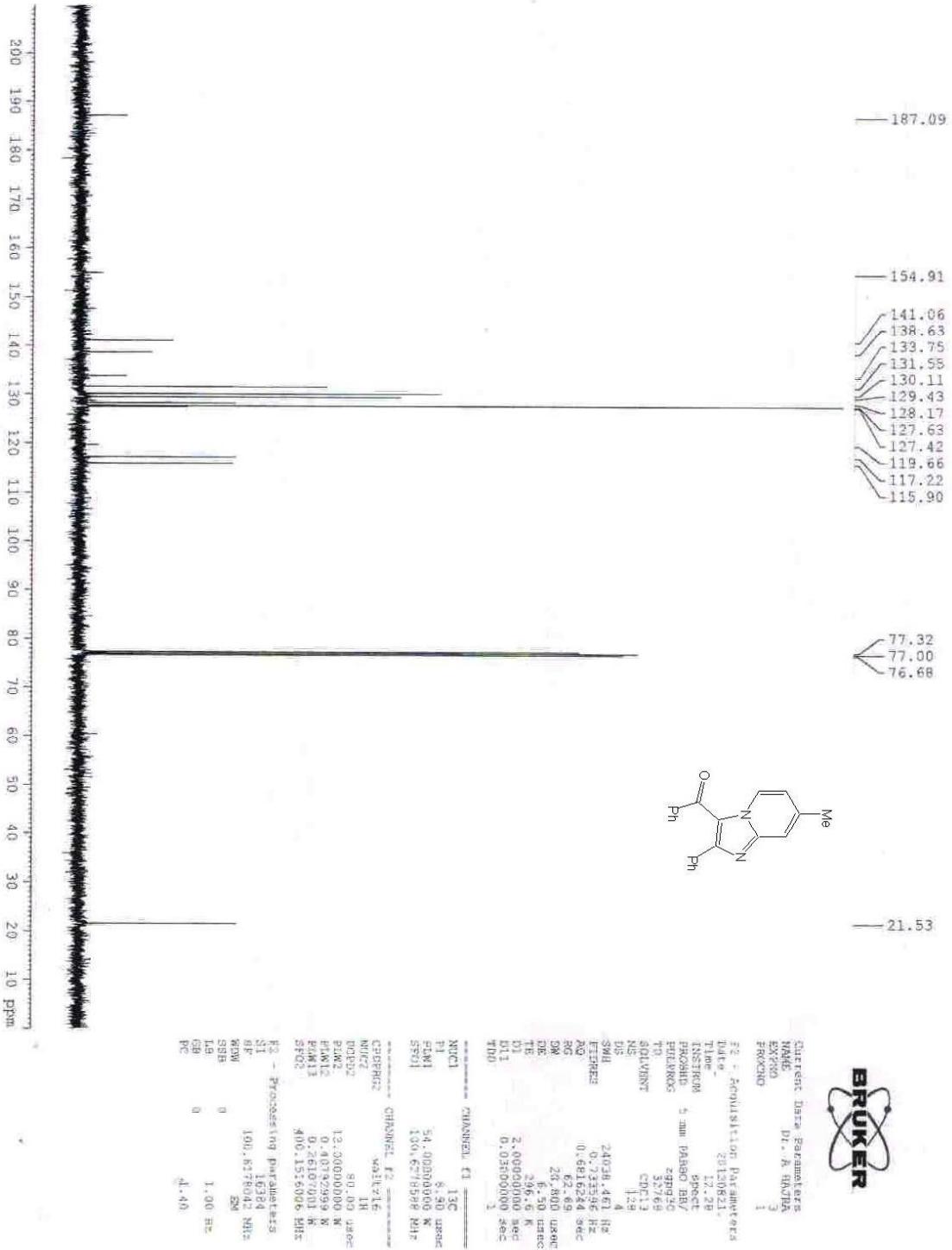


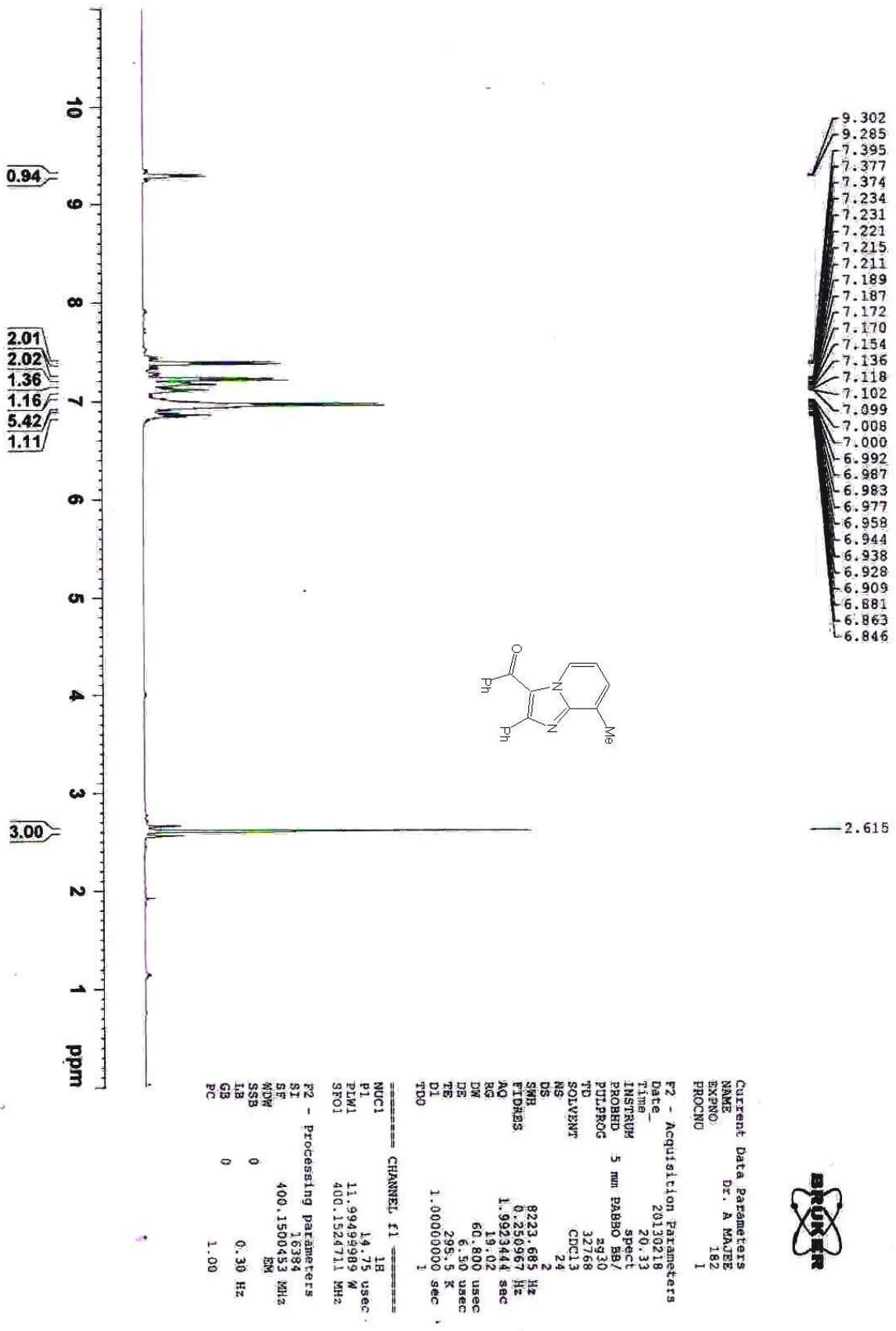
Current Data Parameters
 NAME HAJRA
 EXPNO 2
 PROCHIO 1
 F2 - Acquisition Parameters
 Date 20120821
 Time 12.12
 INSTRUM spect
 PROBID 5 mm PABBO BB/
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 32
 DS 2
 SWH 8223.685 Hz
 FIDRES 0.250567 Hz
 AQ 1.992344 sec
 RG 67.81
 DW 6.800 usec
 DE 6.50 usec
 TE 295.7 K
 T1 1.000000 sec
 D1 1
 TDO 1

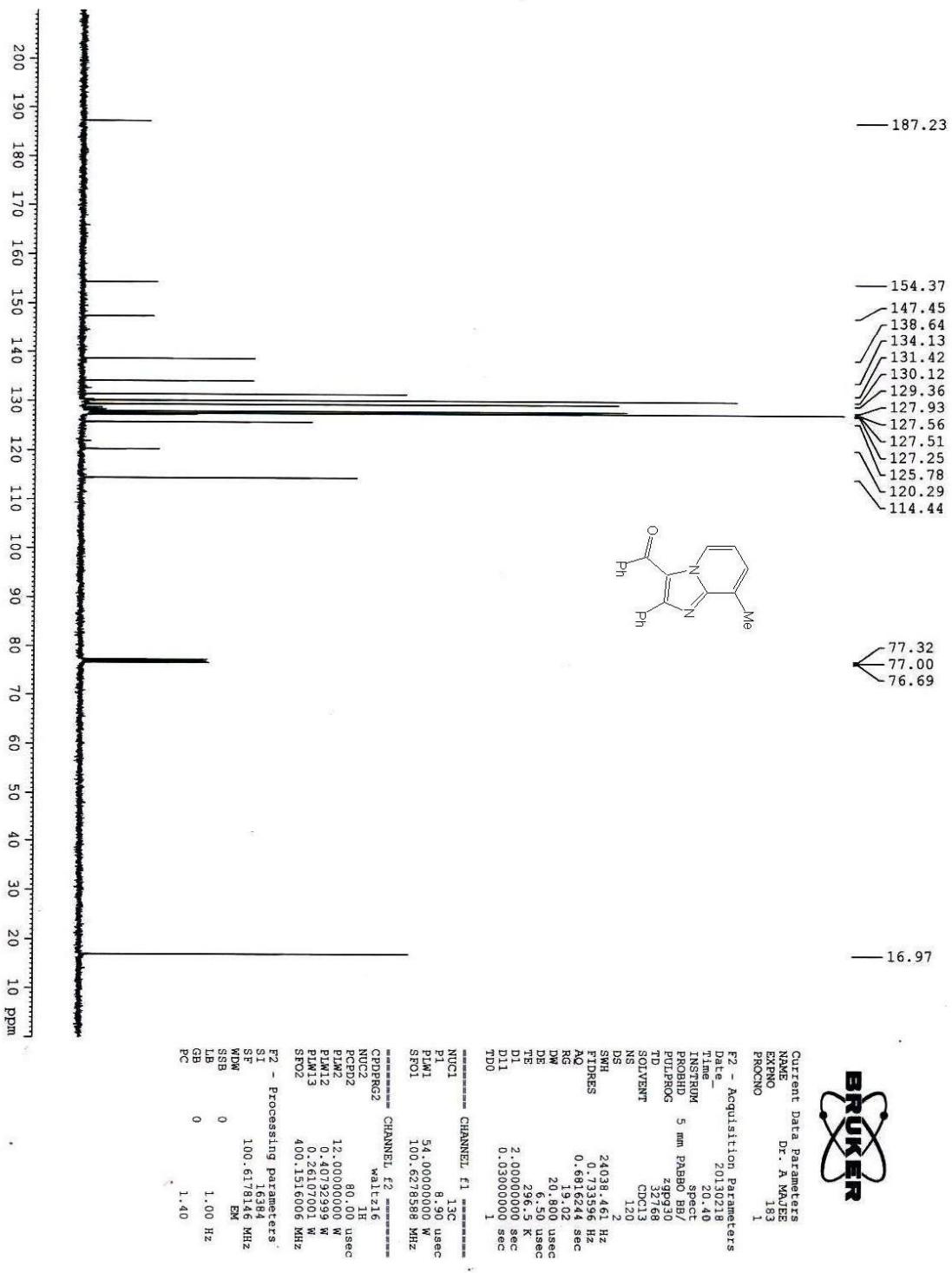
===== CHANNEL f1 =====
 NUC1 1H
 P1 14.75 usec
 PLW1 11.9949989 W
 SF01 400.1524711 MHz

F2 - Processing Parameters
 SI 16384
 SF 400.1500533 MHz
 MDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

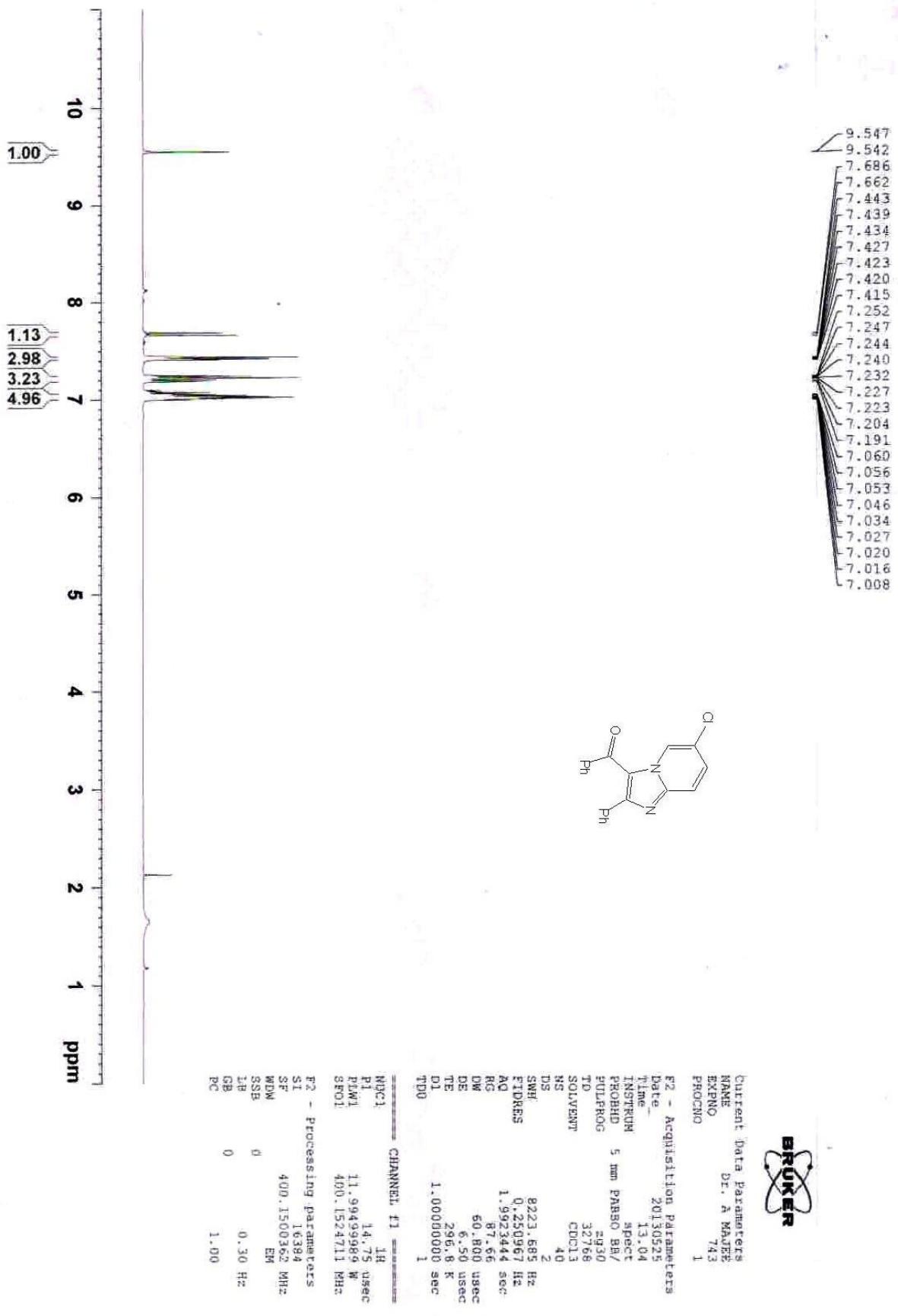
BRUKER

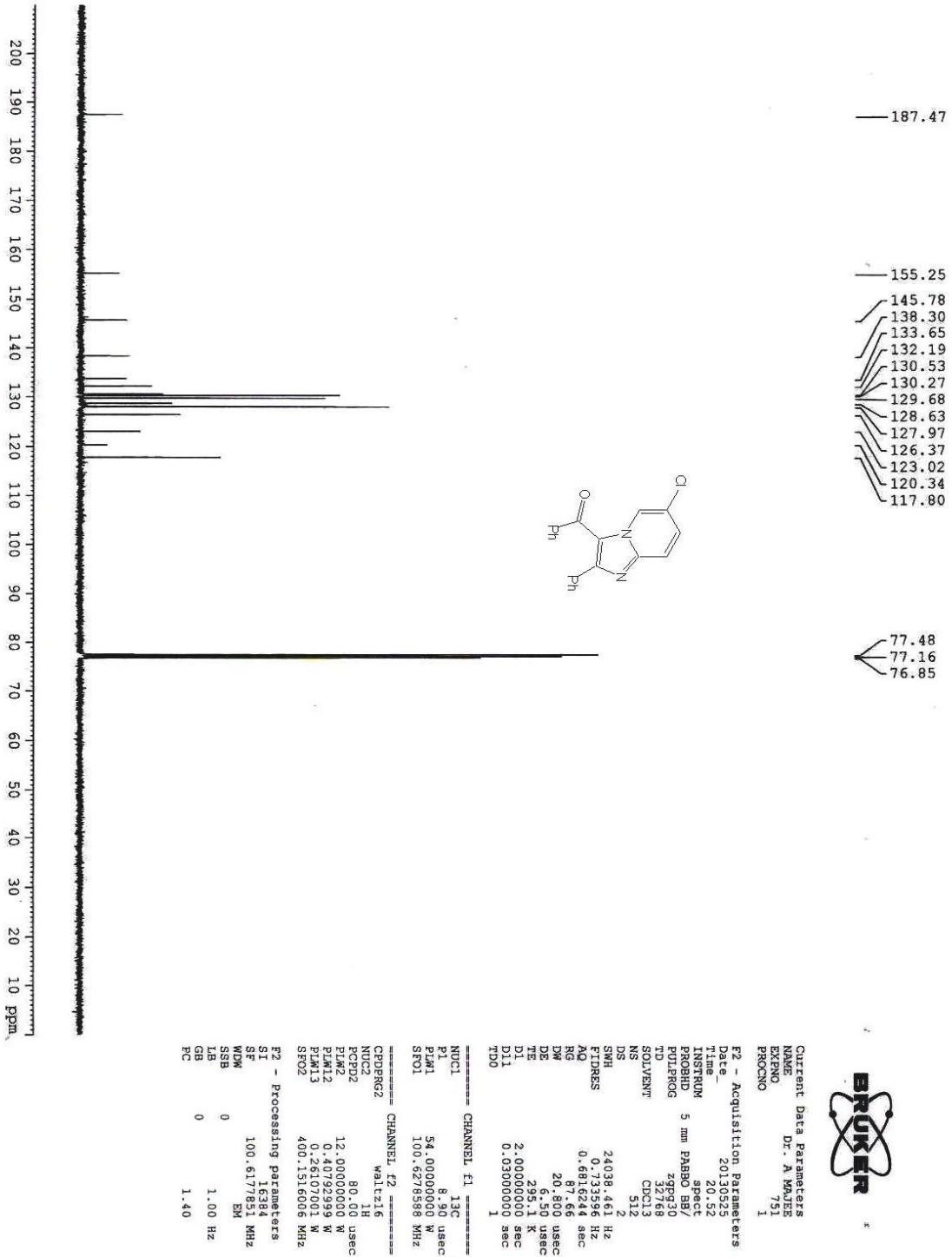




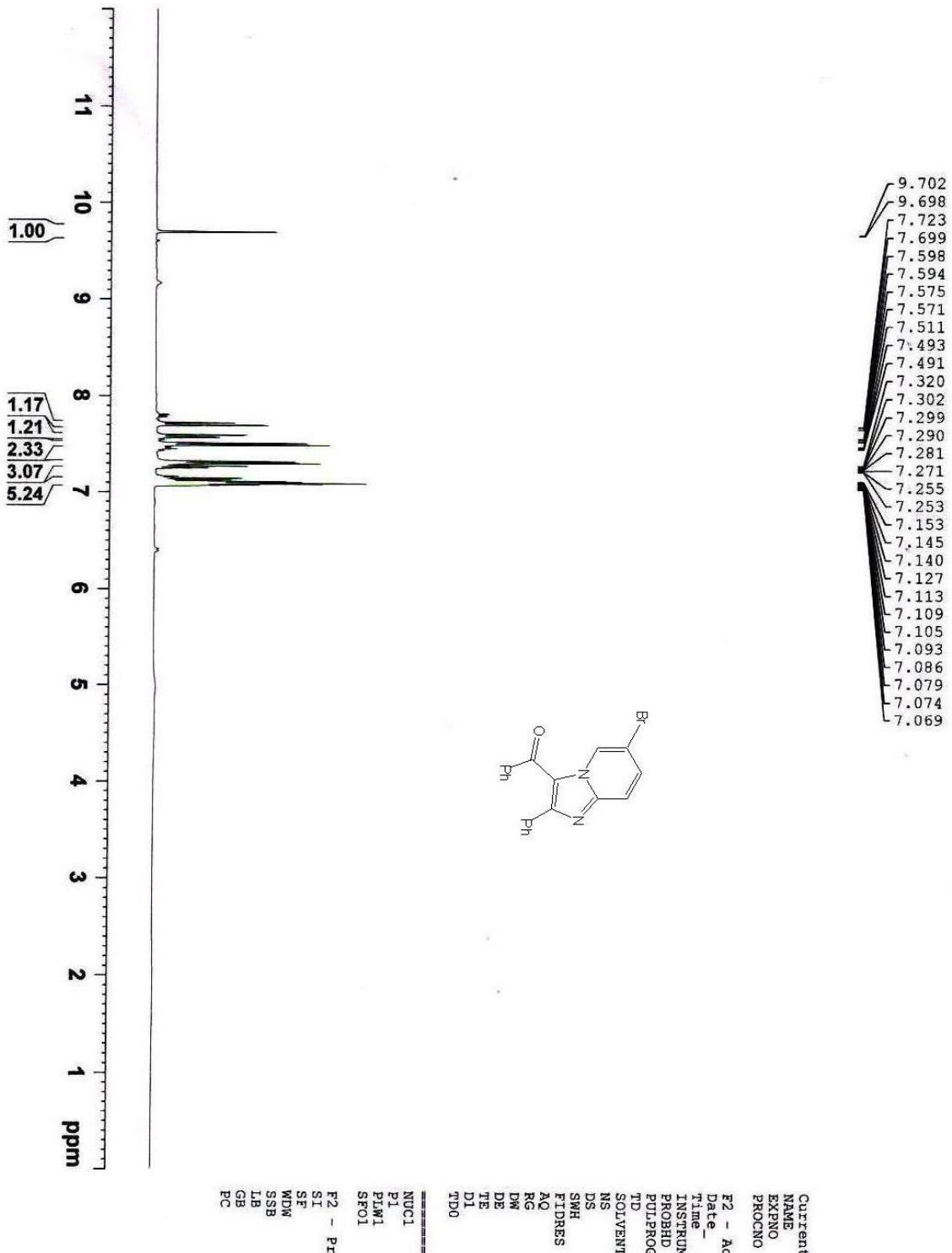


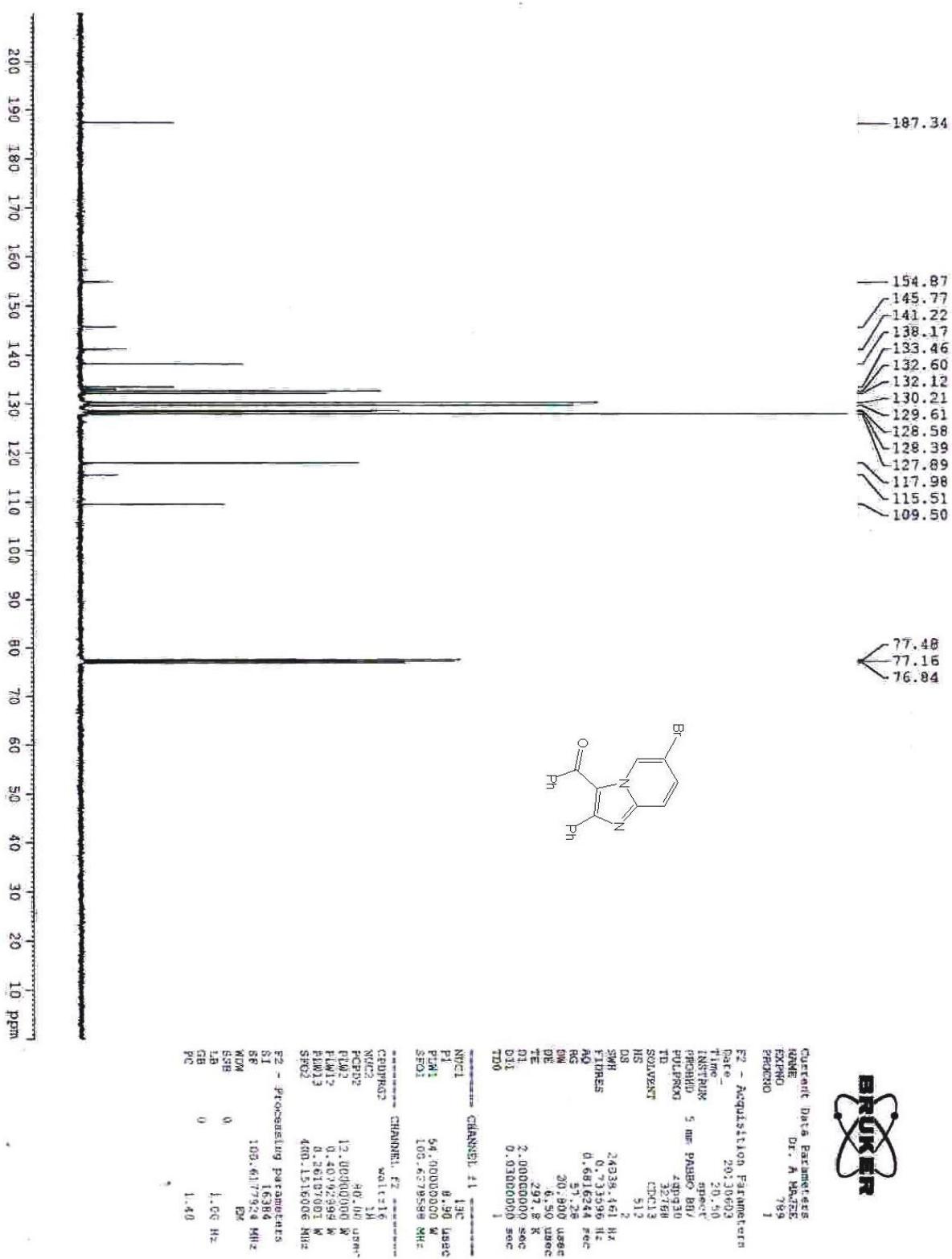
BRUKER



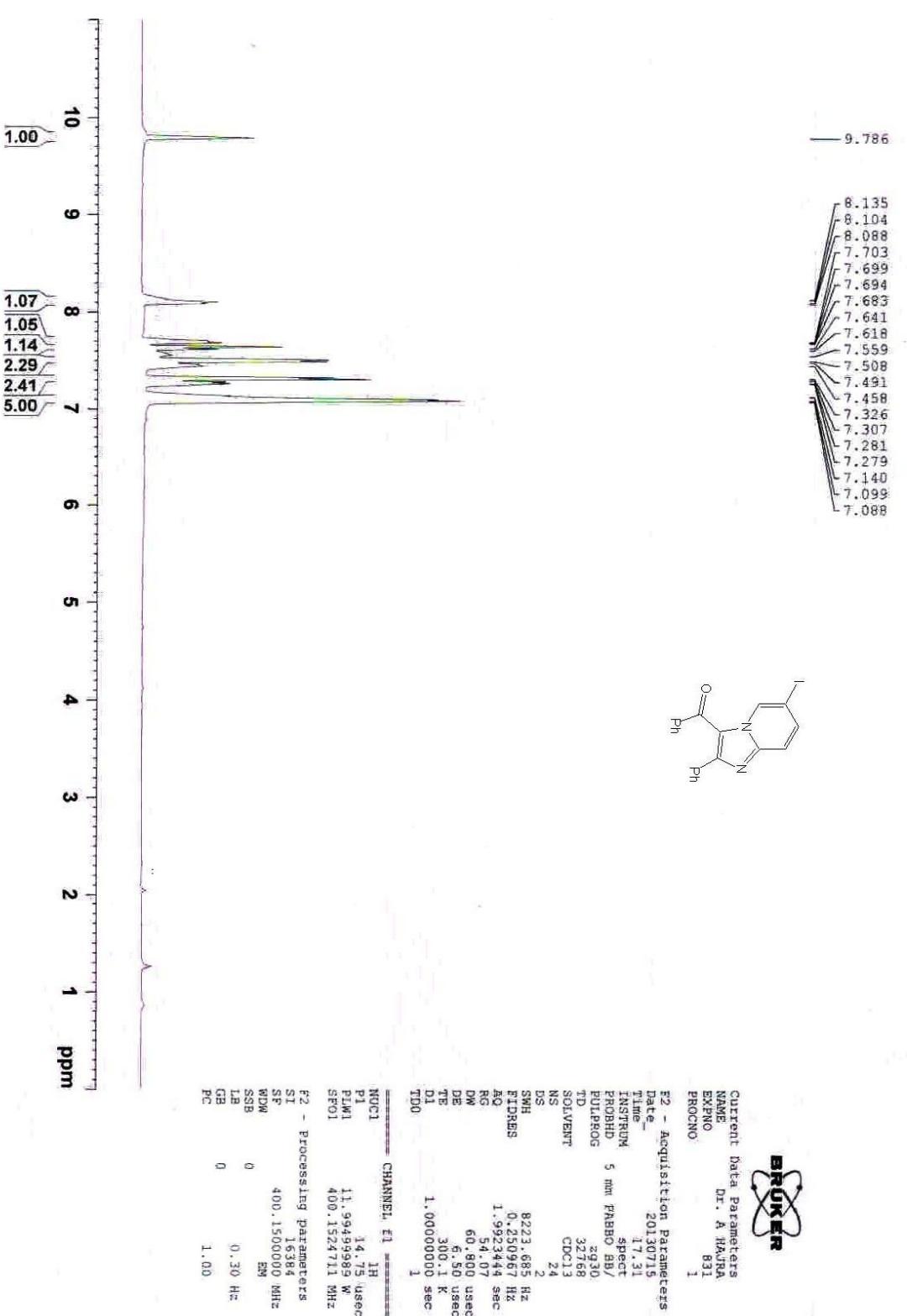


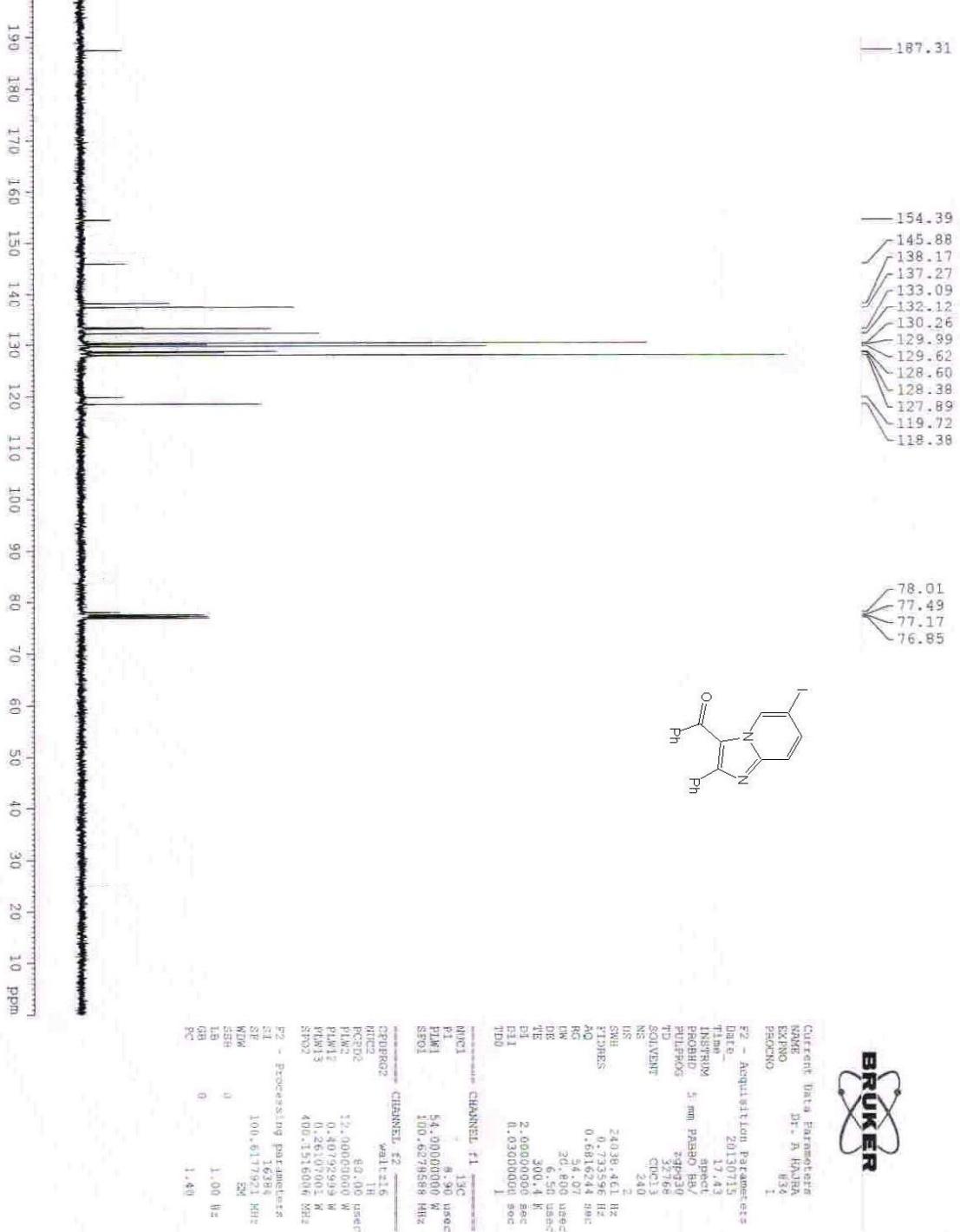
BRUKER



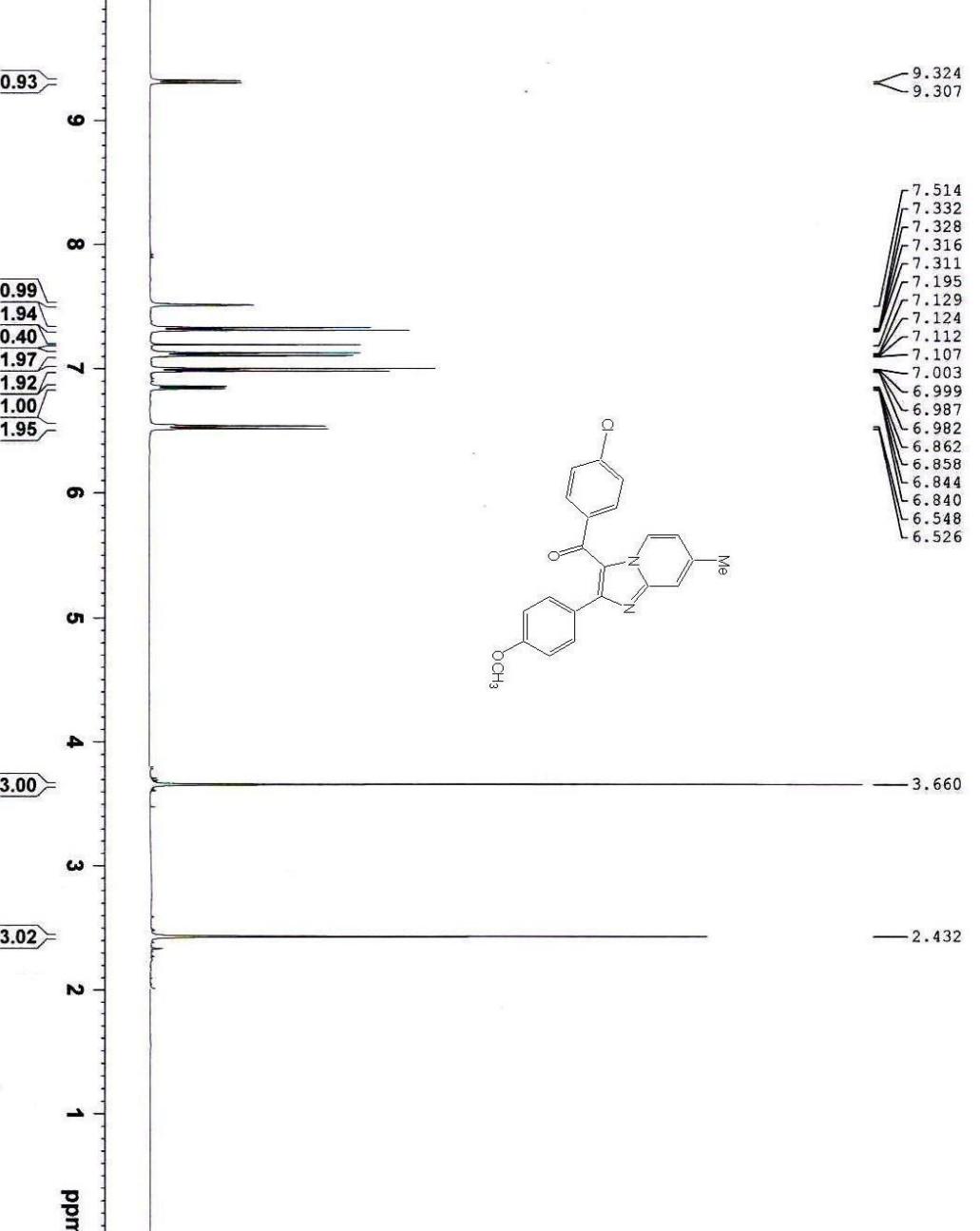


BRUKER





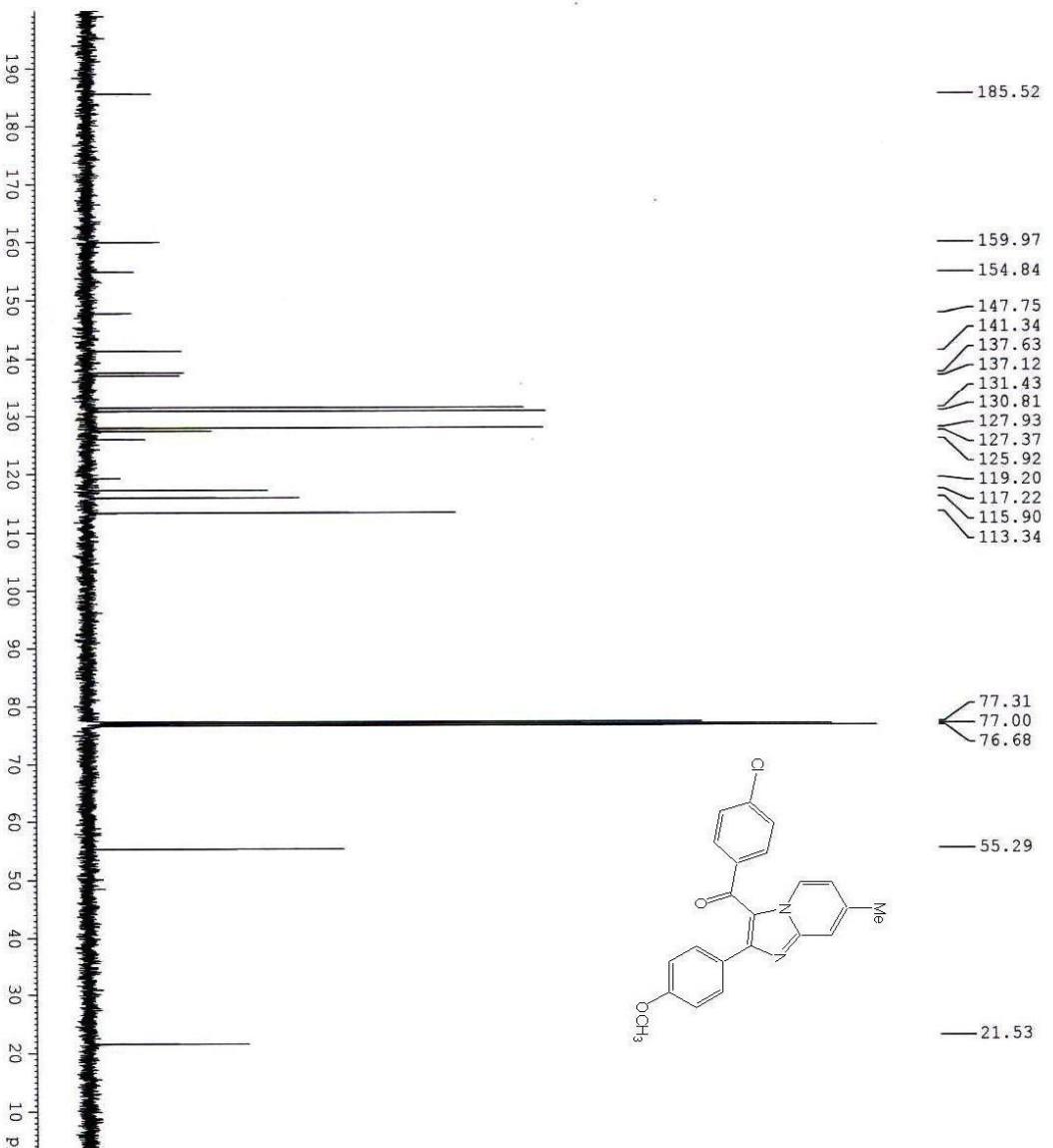
BRUKER



Current Data Parameter
NAME Dr. A HAJ
EXNNO
PROCNO
F2 - Acquisition Para
Date_ 20108
Time_ 21.
INSTRUM 5 mm PABBO B
PROBHD PUPROB
PULPROG zg
TD 327
SOVENT CDC
NS
DS
SWH 8223.6
FIRES 0.2209
AQ 1.9934
RG 87.
DW 60.8
DE 6.
TE 298
DI 1.000000
TDO

===== CHANNEL f1 =
NUC1 14.
P1 11.994999
P1W1 400.15247
SFO1
F2 - Processing param
SI 163
SF 400.15003
WDW
SSB 0
LB 0
GB 0
PC 1.





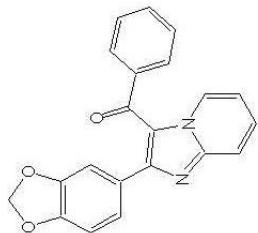
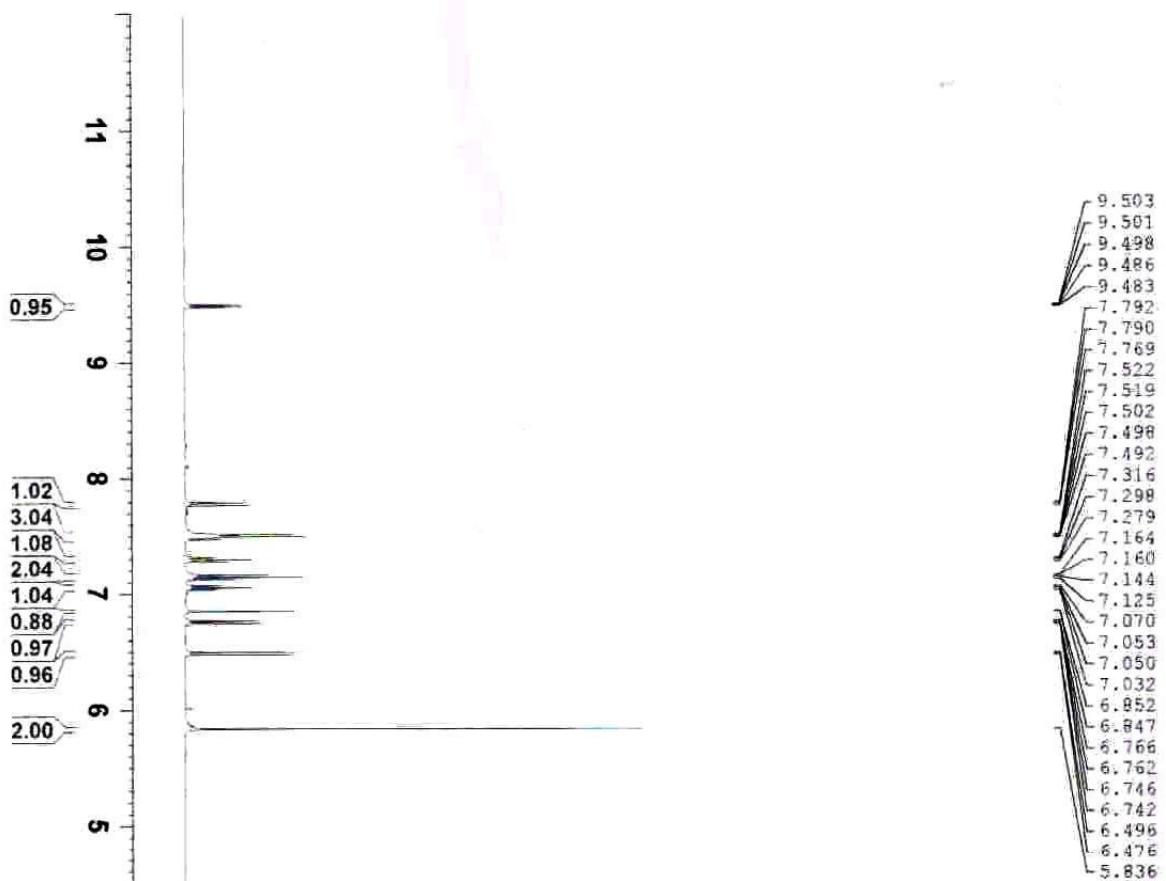
Current Data Parameters
NAME: A_HA1RA
EXPNO: 38
PROCNO: 1
F2 - Acquisition Parameters
Date: 20120227
Time: 22:08
INSTRUM: spect
PROBHD: 5 mm PABBO/PROB
PULPROG: zappr30
TD: 32768
SOLVENT: CDCl3
NS: 256
DS: 24038461 Hz
SWH: 0.73396 Hz
FIDRES: 0.6816144 sec
AQ: 0.02269 sec
DW: 204.00 usec
DE: 6.50 usec
TE: 29.1 K sec
D1: 2.000000 sec
D11: 0.0300000 sec
TDO: 1

===== CHANNEL F1 =====
NUC1: 1H
P1: 8.30 usec
P1M1: 54.0000000 W
P1M2: 100.0228588 MHz
SF01:

===== CHANNEL F2 =====
NUC2: 1H
PCPD2: 80.00 usec
P1W2: 12.0000000 W
P1W12: 0.40732399 W
P1W13: 0.26107001 W
SF02: 400.15166006 MHz

F2 - Processing parameters
SI: 16384
SF: 100.6178012 MHz
NMW: 0 EM
SSB: 0
LB: 1.00 Hz
GB: 0
PC: 1.40

BRUKER



Current Data Parameters
NAME Dr. R MAJEE
EXPNO 1005
PROCNO 1

F2 - Acquisition Parameters

DATE	20130712
TIME	20.54
INSTRUM	Spect
PROBHD	5 mm PABBO BB/
PULPROG	zg30
TD	32768
SOLVENT	CDCl ₃
NS	2
DS	2
SWH	8223.685 Hz
ETRIMES	0.250967 Hz
AQ	1.992344 sec
RG	40.87
DW	60.800 usec
DE	6.50 usec
TE	2.981 K
D1	1.0000000 sec
TBO	

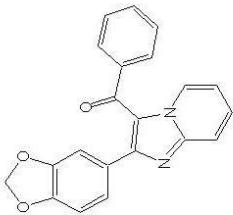
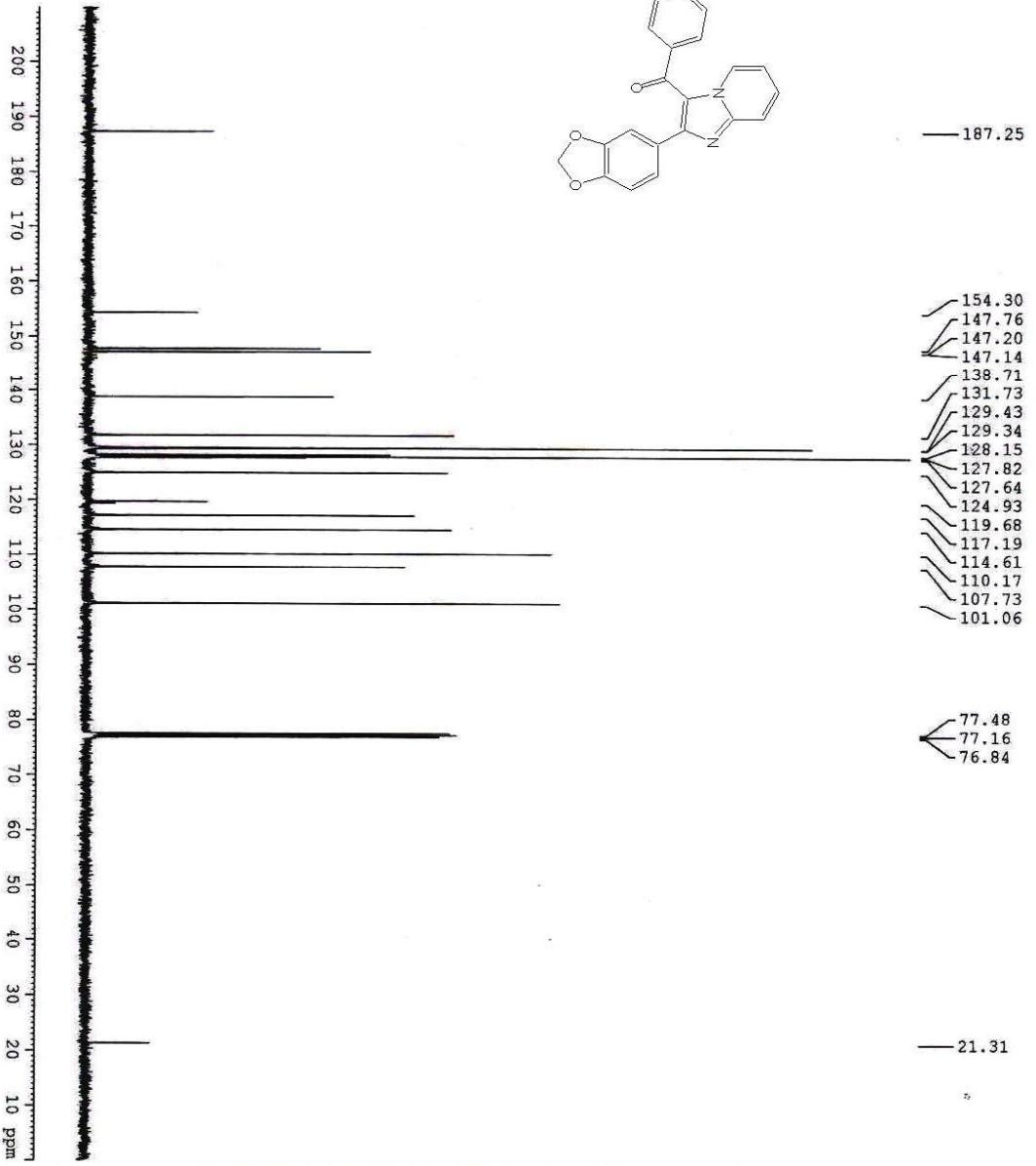
===== CHANNEL f1 =====

NUC1	¹ H
RL	14.75 usec
PW1	11.9949999 W
SP01	400.1524711 MHz

F2 - Processing parameters

SI	16384
SF	400.1500000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	1.00
PC	

BRUKER



BRUKER

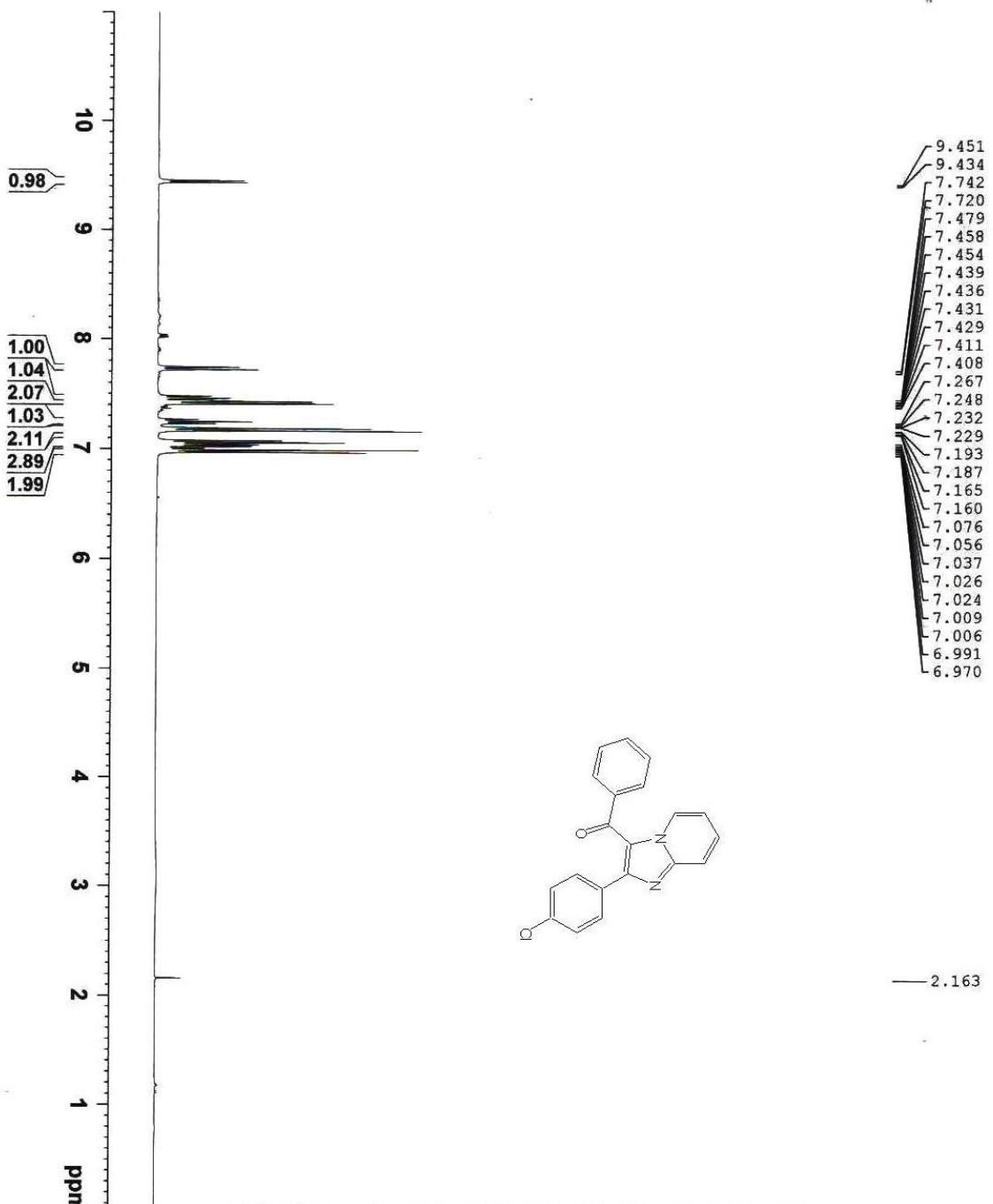
Current Data Parameters
NAME Dr. A MAJEE
EXPNO 1012
PROCNO 1

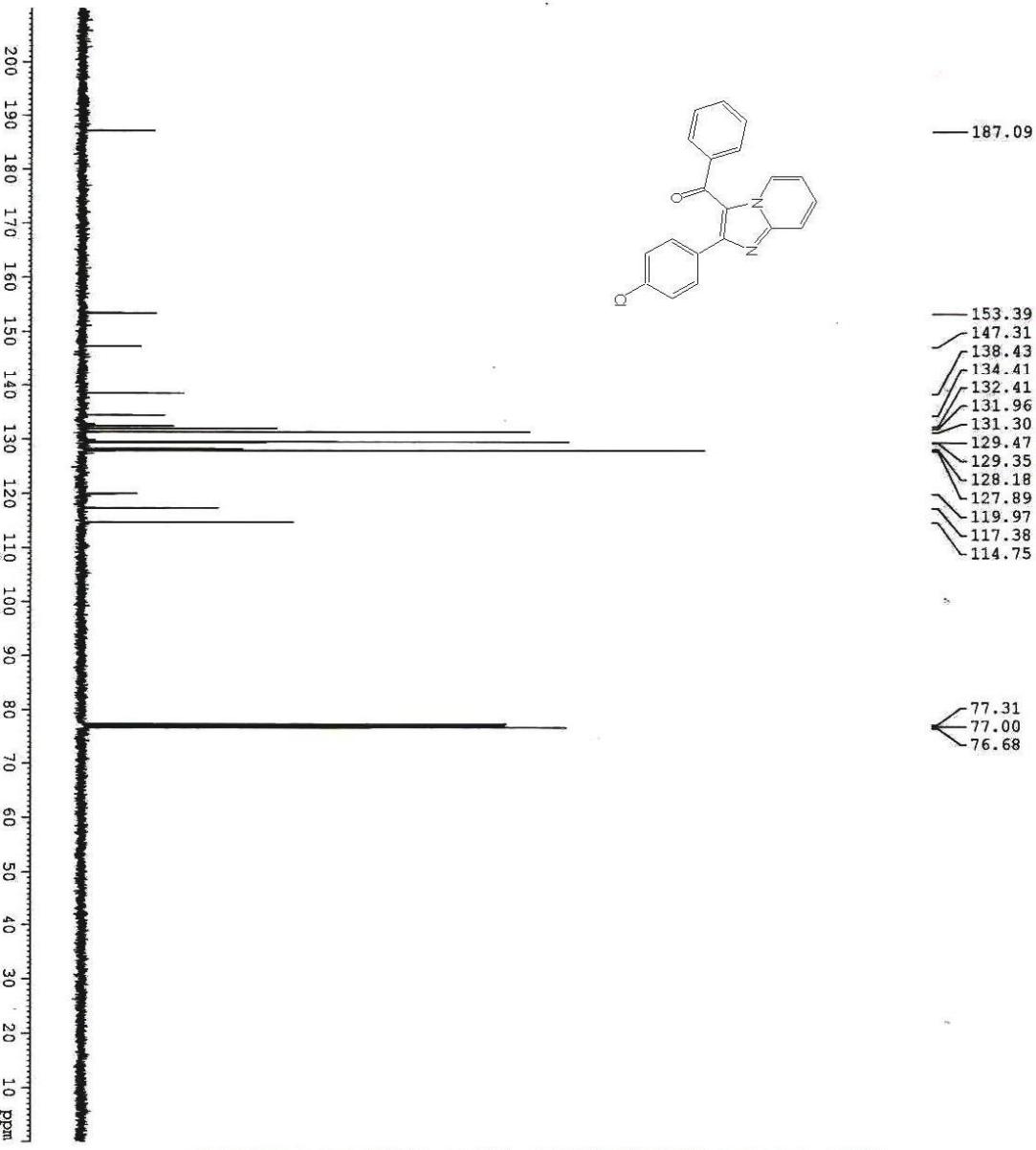
F2 - Acquisition Parameters
Date 20130714
Time 19.56
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 2
DS 2
SWH 24038.461 Hz
ETDESS 0.73356 Hz
AQ 0.6816244 sec
RG 54.07
DW 20.800 usec
DE 6.50 usec
TE 293.6 K
D1 2.0000000 sec
D11 0.0300000 sec
TDO 1

===== CHANNEL f1 =====
NUCL 13C
P1 8.90 usec
PLW1 54.000000 W
SF01 100.628588 MHz

===== CHANNEL f2 =====
NUCL 1H
PCP02 80.00 usec
PLW2 12.0000000 W
PLW1,2 0.0072959 W
PLW1,3 0.24117001 W
SFO2 400.1516006 MHz

F2 - Processing parameters
SI 16384
SF 100.6177983 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





Current Data Parameters

NAME	Dr. A. MAJEE
EXPT	539
PROCNO	1

F2 - Acquisition Parameters

Date	2013026
Time	20:37
INSTRUM	SPCT
PROBHD	5 mm PABD BB/
PULPROG	32P30
TD	32768
SOLVENT	CDCl3
NS	240
DS	2
SWH	24038.461 Hz
ETRATES	0.733396 Hz
AQ	0.616244 sec
RG	62.69
DW	20.00 usec
DE	6.50 usec
TE	297.9 K
D1	2.000000 sec
D11	0.0300000 sec
TDO	1

NUC1 CHANNEL F1 13C

PL1	8.90 usec
PLM1	54.0000000 W
PLF1	100.6278388 MHz

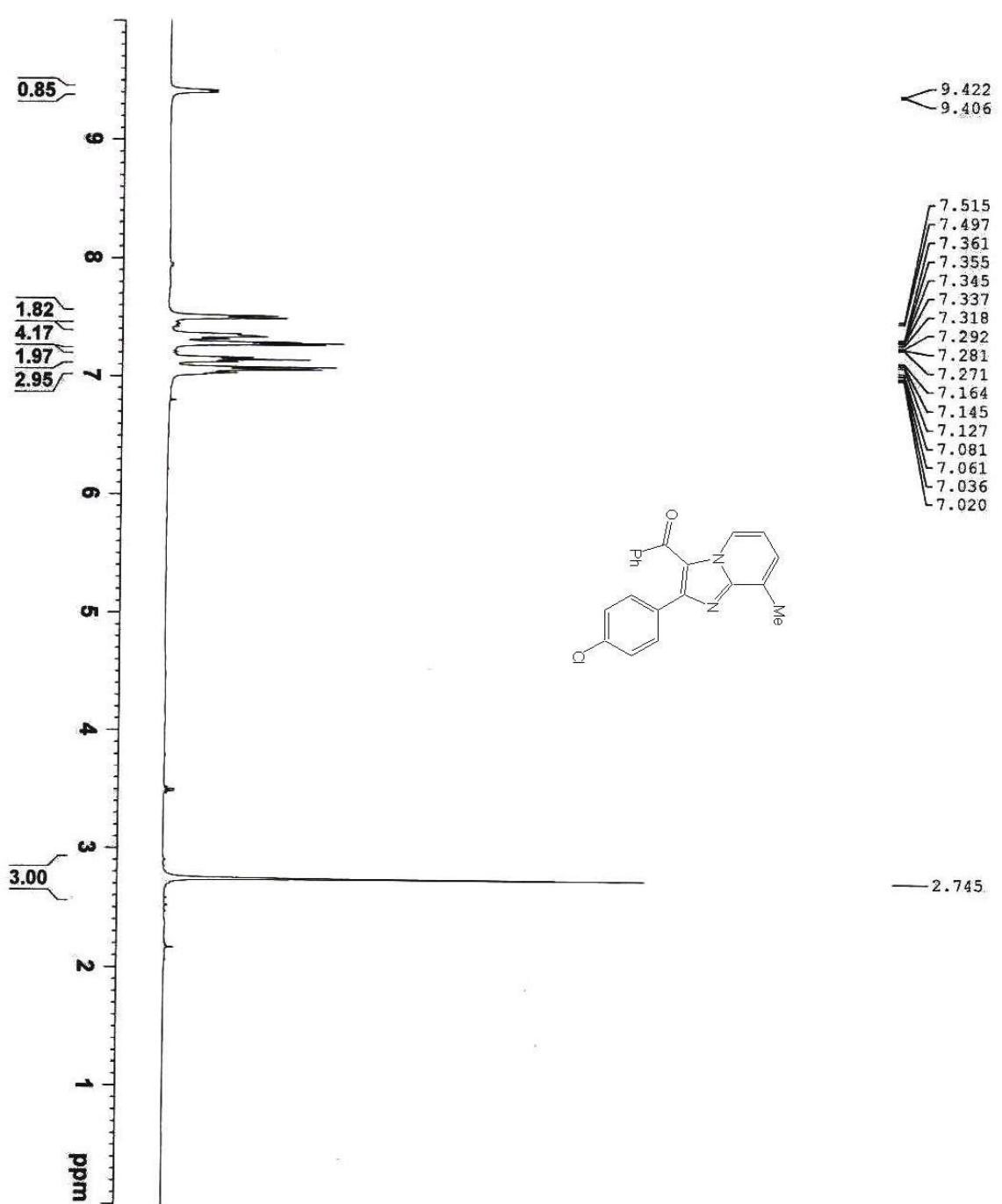
NUC2 CHANNEL F2

CPDP,RG2	1H
PLM2	80.00 usec
PLF2	12.0000000 W
PLW1,2	0.4039299 W
PLW1,3	0.6610701 W
SP02	400.1516006 MHz

F2 - Processing parameters

SI	16384
SF	100.6178041 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



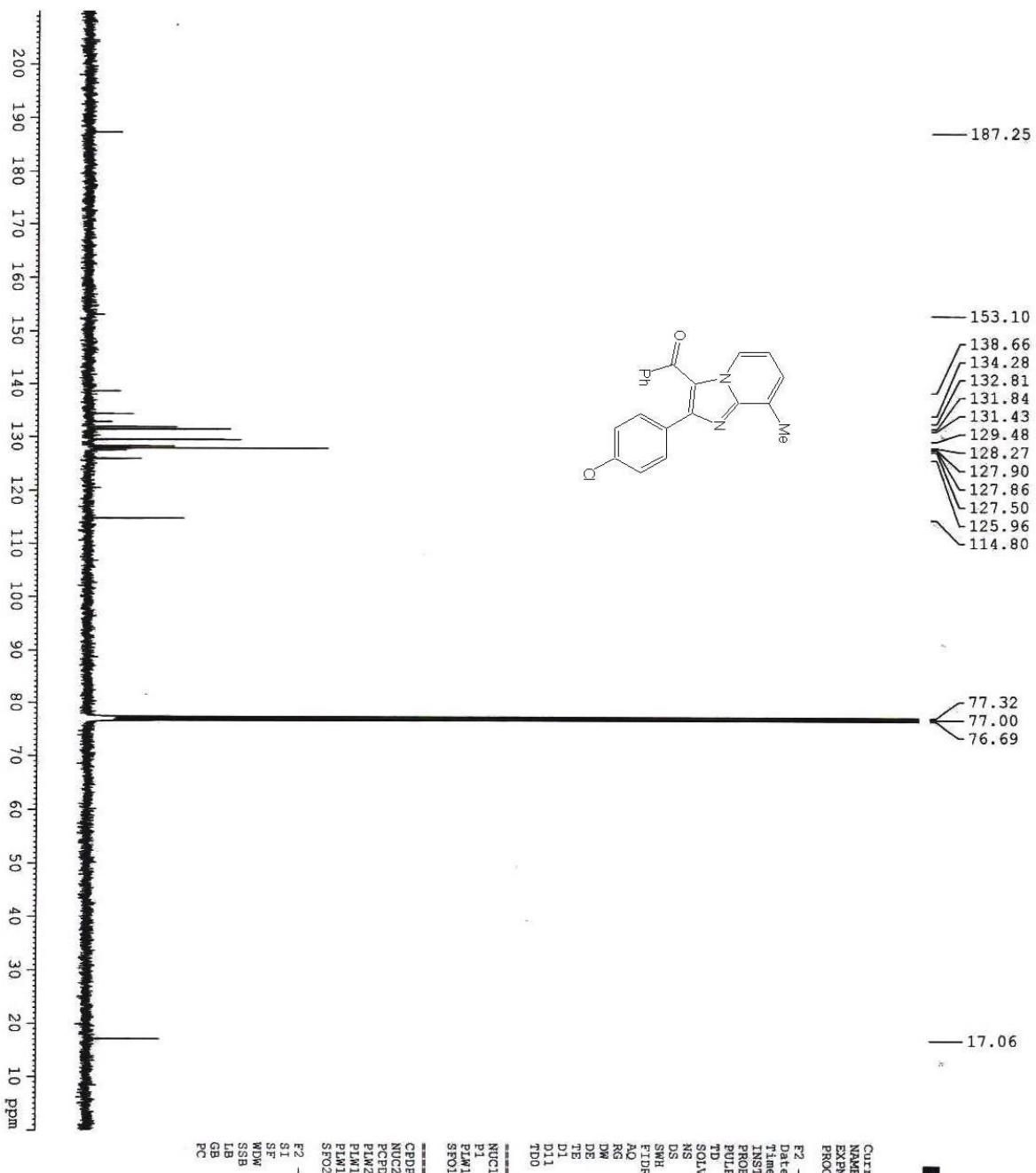


Current Data Parameters
 NAME Dr. A MAJEE
 EXPNO 552
 PROCN 1

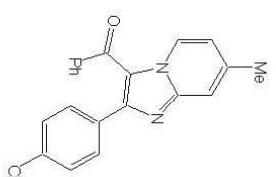
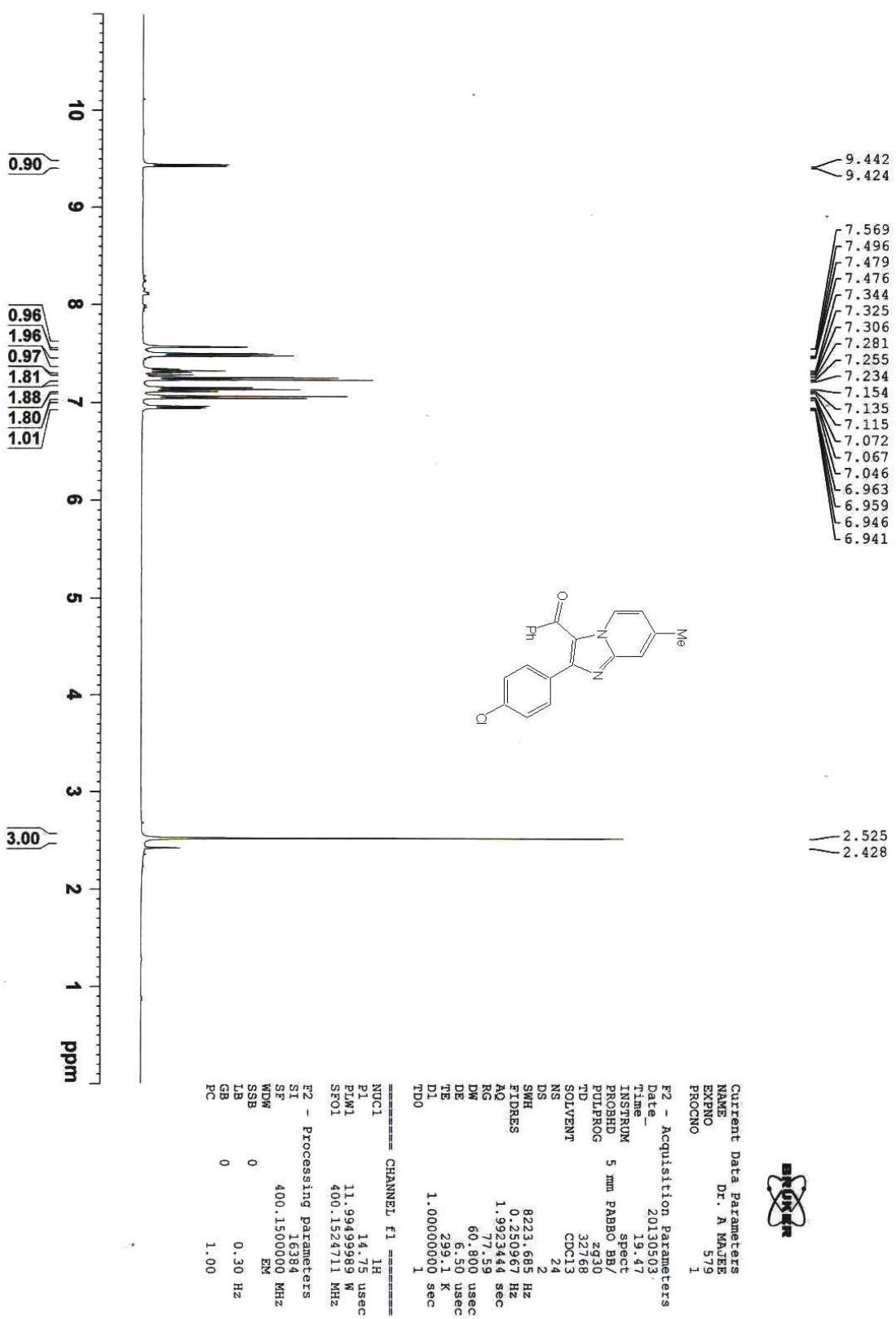
P2 - Acquisition Parameters
 Date 20130428
 Time 20.35
 INSTRUM spect
 PROBID 5 mm PABBO BB/
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 32
 DS 2
 SWH 8223.685 Hz
 FIDRES 0.250967 Hz
 AQ 1.992344 sec
 RG 106.66
 DW 60.800 usec
 DE 6.50 usec
 TE 297.5 K
 D1 1.0000000 sec
 TDO 1

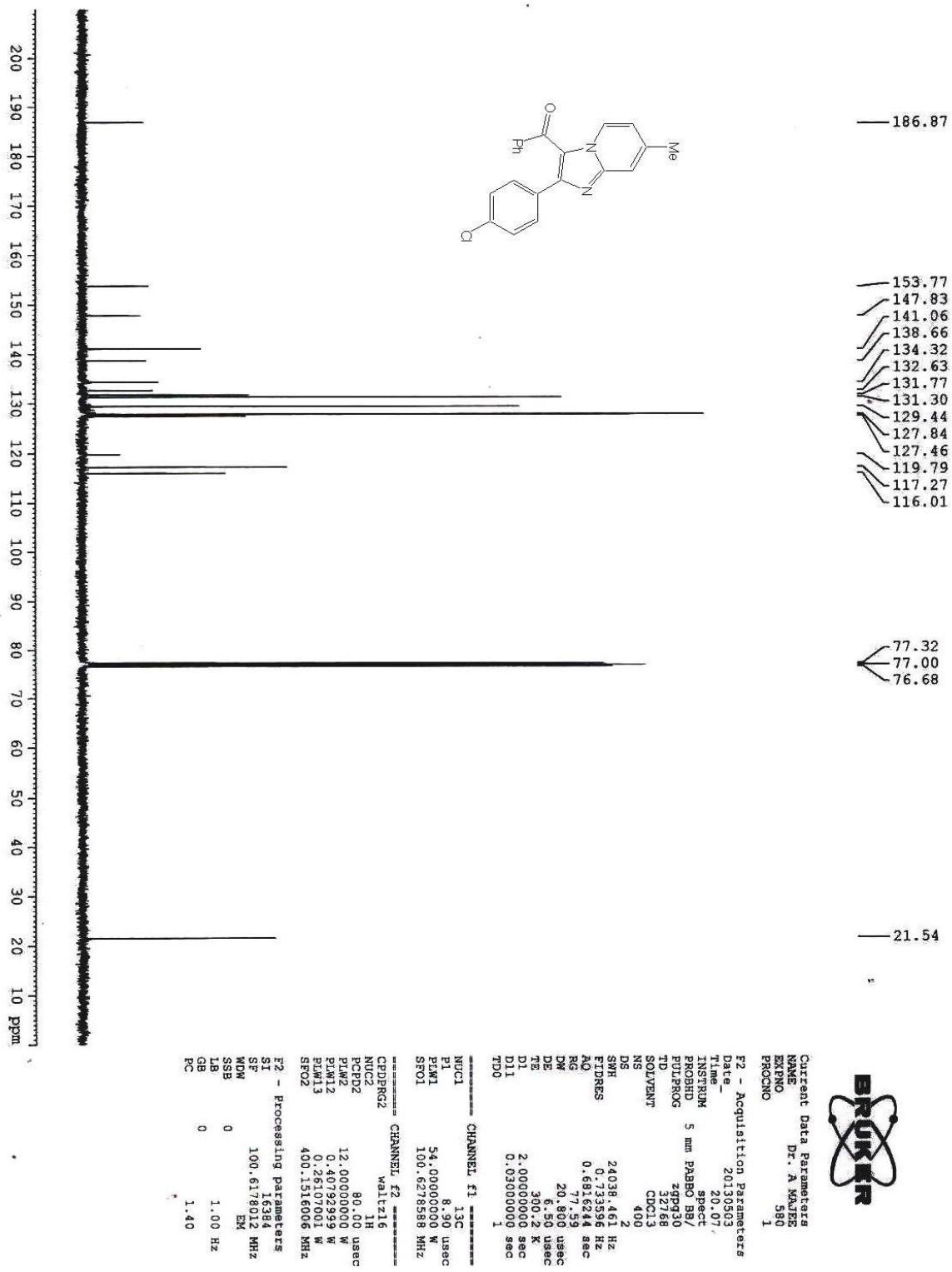
===== CHANNEL f1 =====
 NUC1 1H
 PI 14.75 usec
 PW1 11.99499989 W
 SF01 400.1524711 MHz

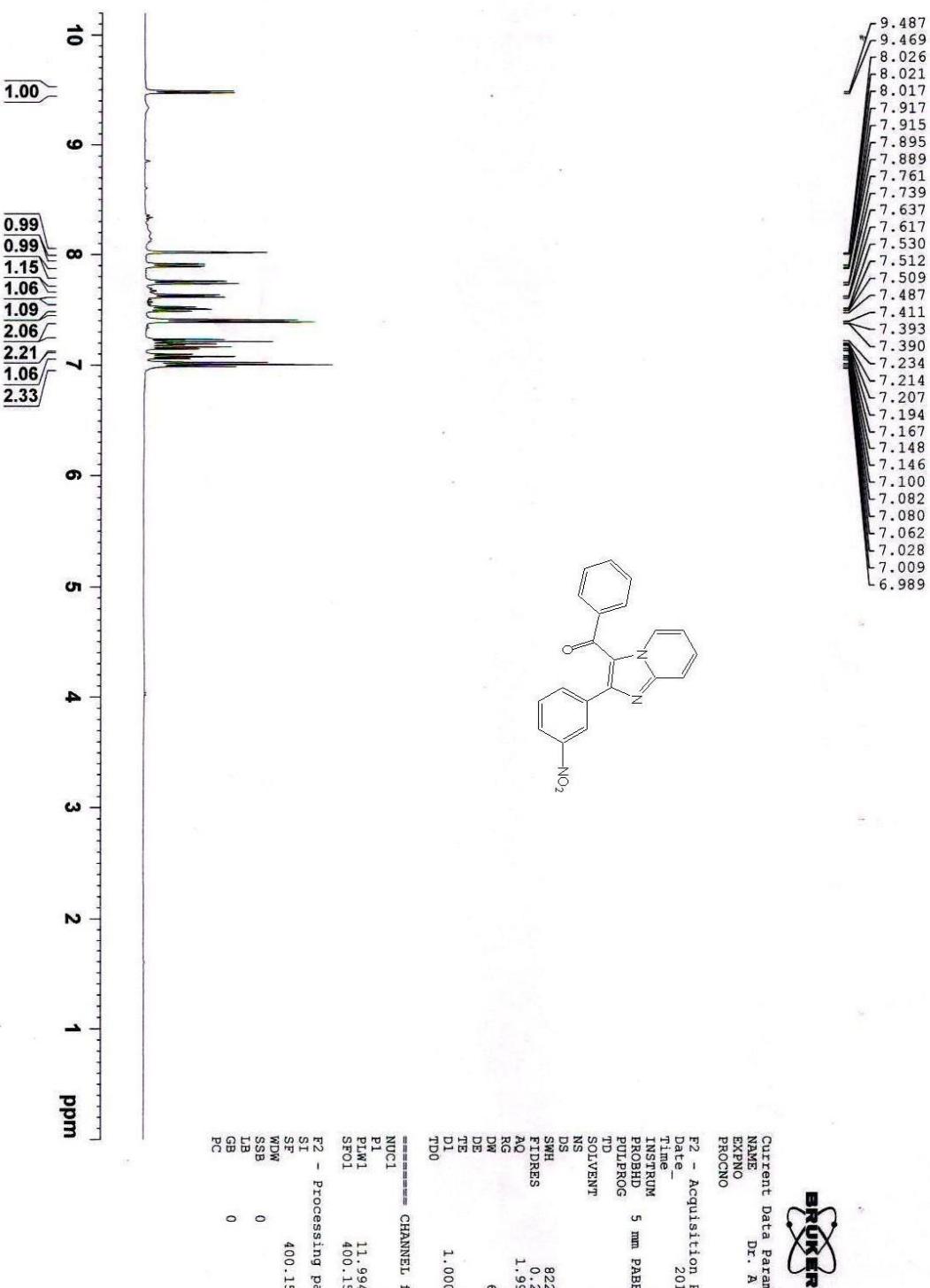
F2 - Processing parameters
 SI 16384
 SF 400.1500000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 1.00
 PC

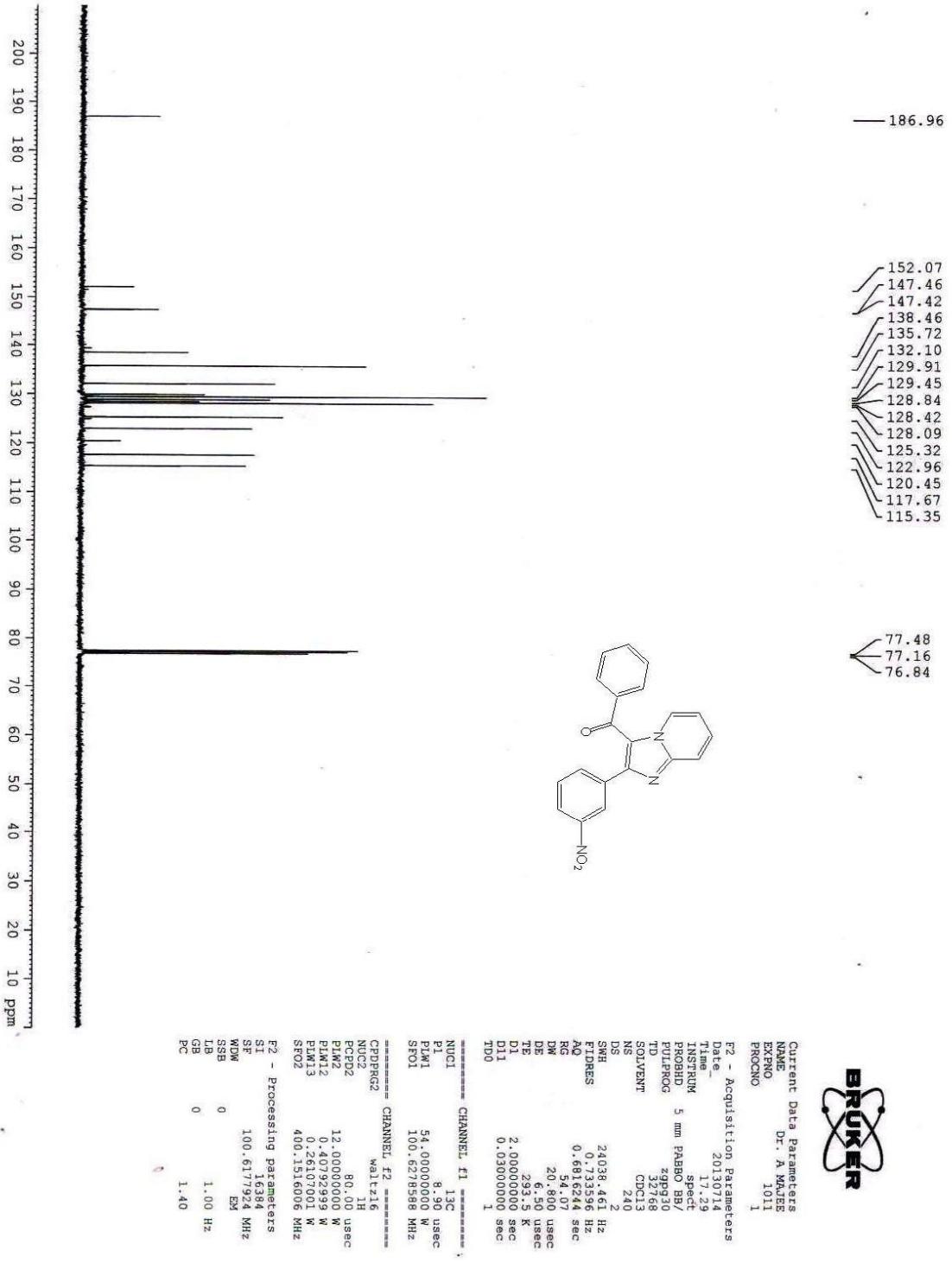


BRUKER

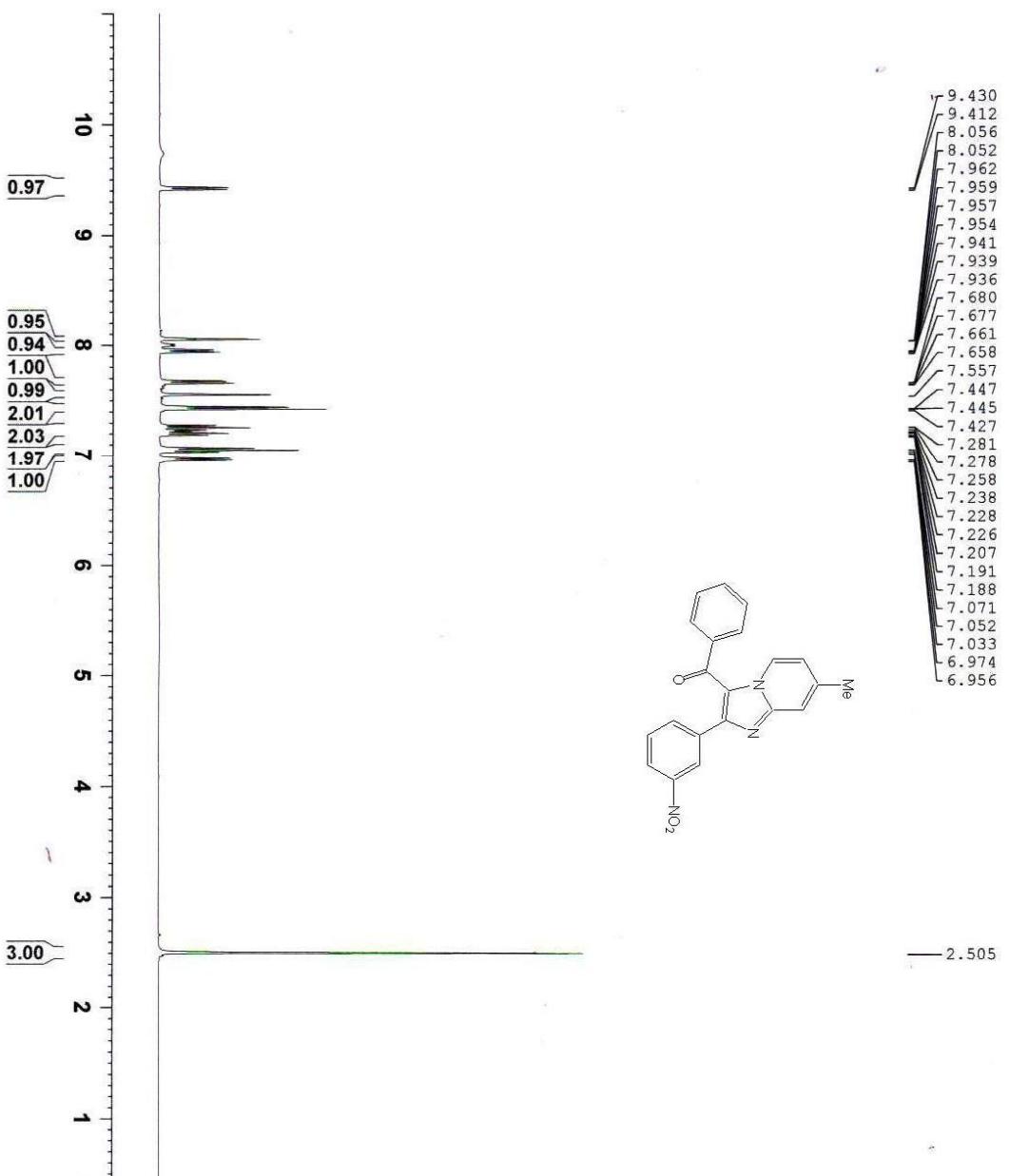




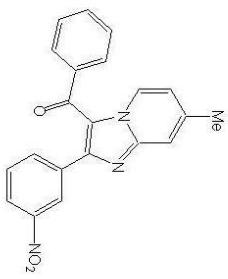


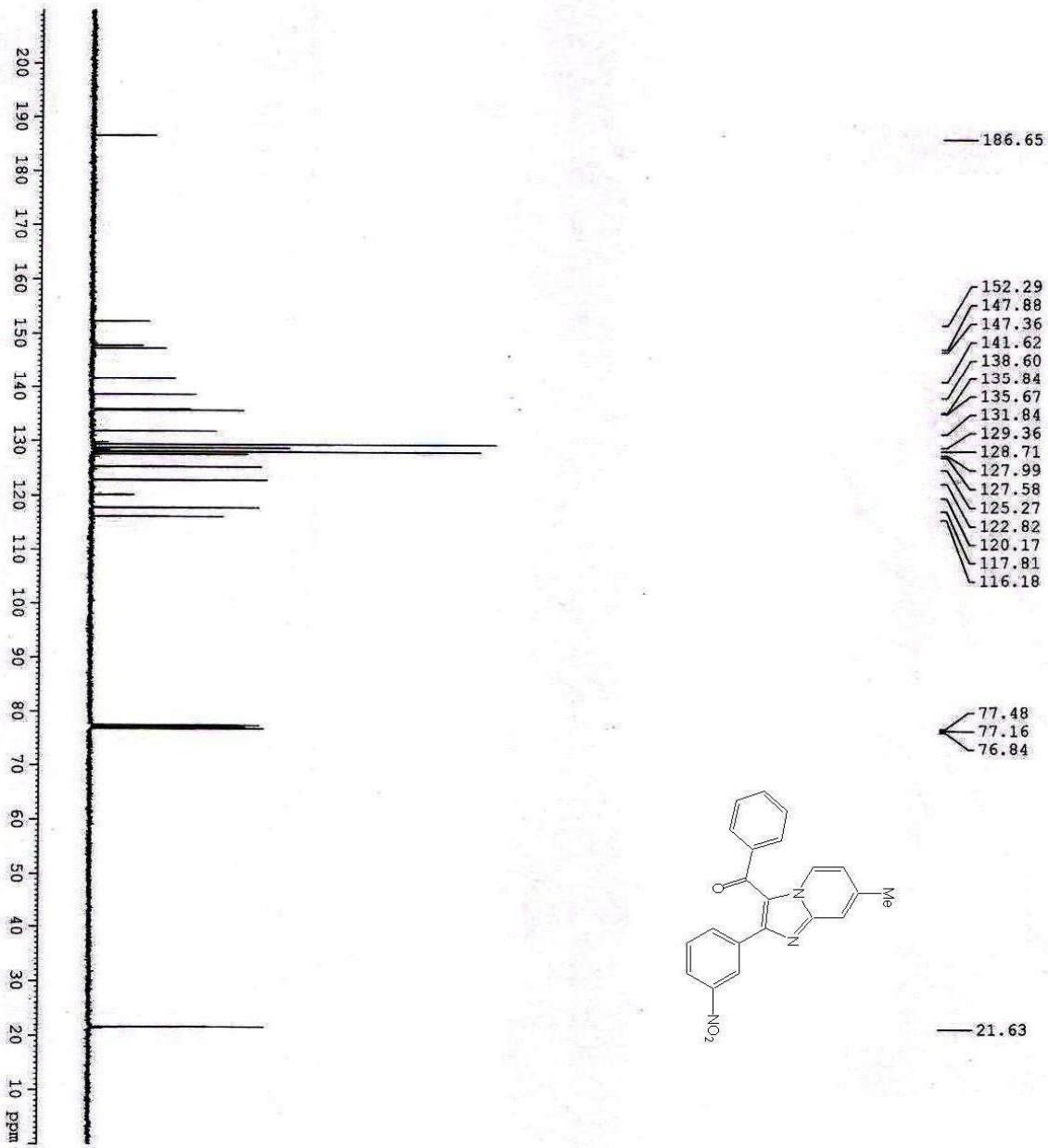


BRUKER



	Current	Data	Parameters
NAME	Dr.	A HAJRA	
EXPNO		850	
PROCNO			1





Current Data Parameters
 NAME Dr. A. HAJRA
 EXPNO 851
 PROCN 1

P2 - Acquisition Parameters
 Date_ 20130725
 Time_ 20:45
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zppg30
 TD 37768
 SOLVENT CDCl₃
 NS 160
 DS 2
 SWH 24038.461 Hz
 FIDRES 0.733596 Hz
 AQ 0.6816241 sec
 RG 37.83
 DW 20.800 usec
 DE 8.50 K
 T1 2.0000000 Sec
 D1 0.0300000 Sec
 TDO 1

CHANNEL f1: 13C

NUC1 13C
 PLW1 8.90 usec
 SF01 54.00000000 W
 CPDRG2 100.6270588 MHz

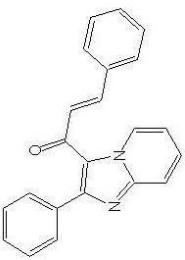
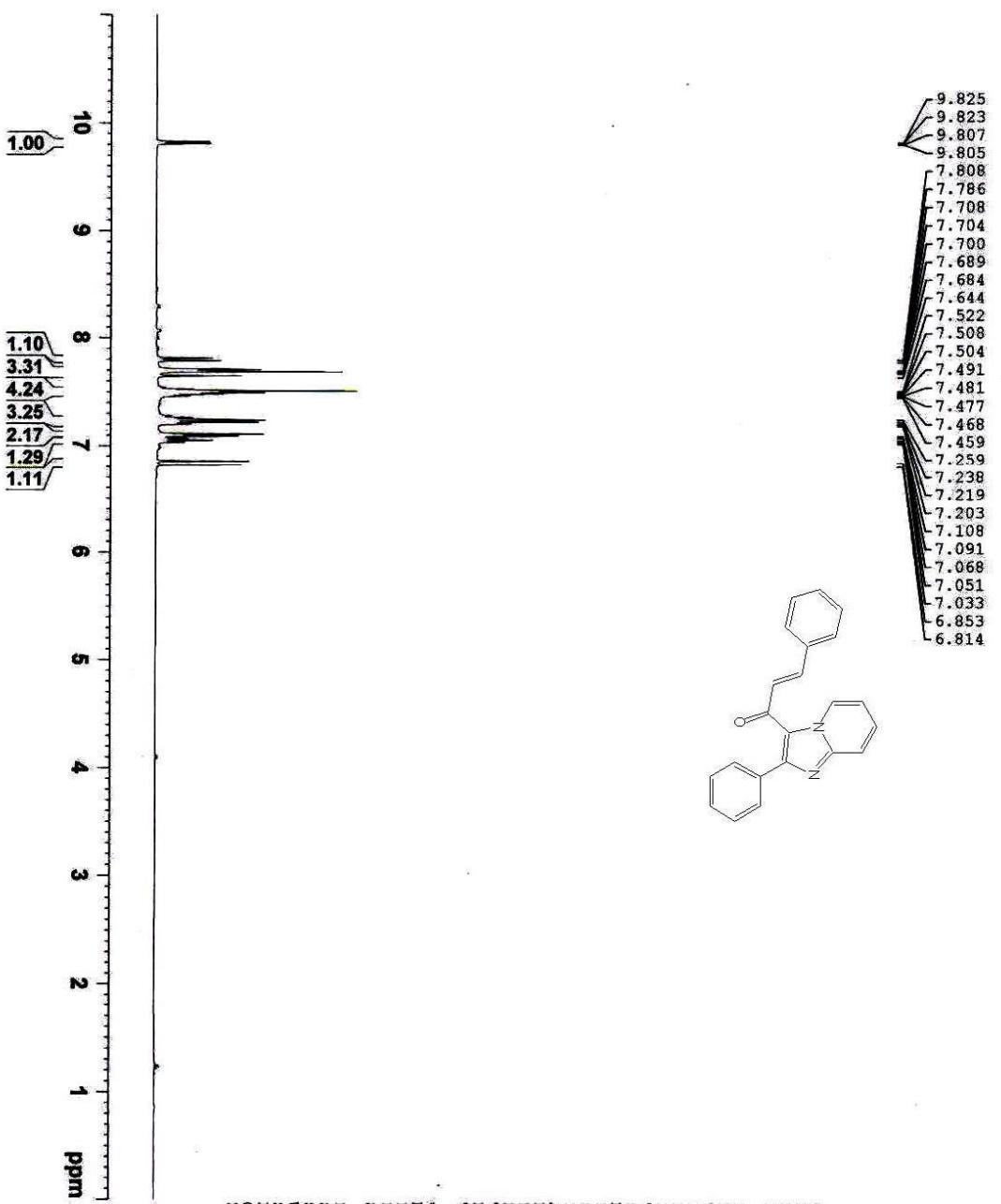
CHANNEL f2: 1H

NUC2 1H
 PCP02 80.00 usec
 PLW2 12.00000000 W
 PLW12 0.40793999 W
 PLW13 0.28107001 W
 SFO2 400.1516006 MHz

P2 - Processing parameters:

S1 100.6177946 MHz
 PDDW 0 ER
 SSB 0 1.00 Hz
 LB 0
 GB 1.40
 PC





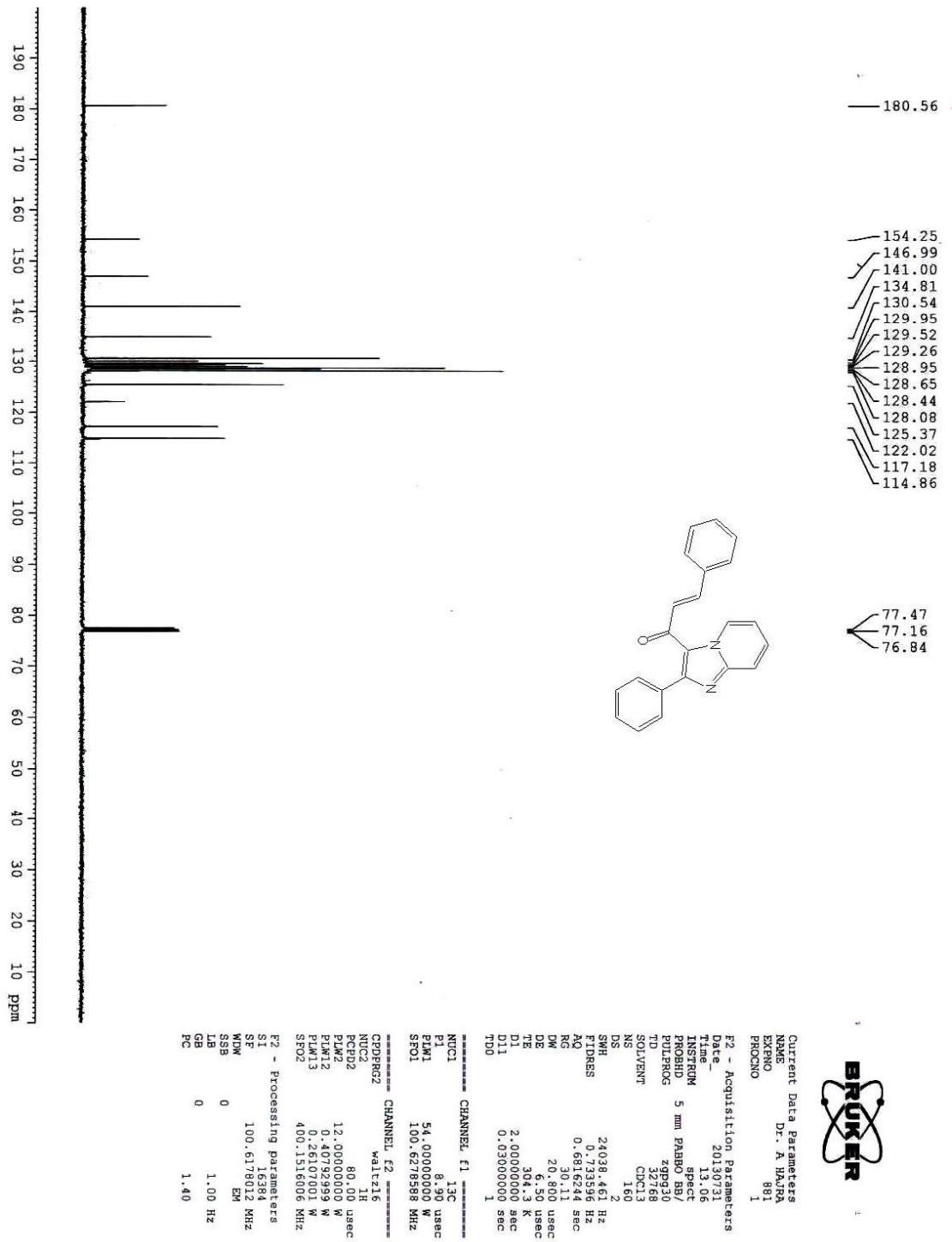
Current: Data Parameters
NAME DR. A HAJRA
EXPNO 880
PROCNO 1

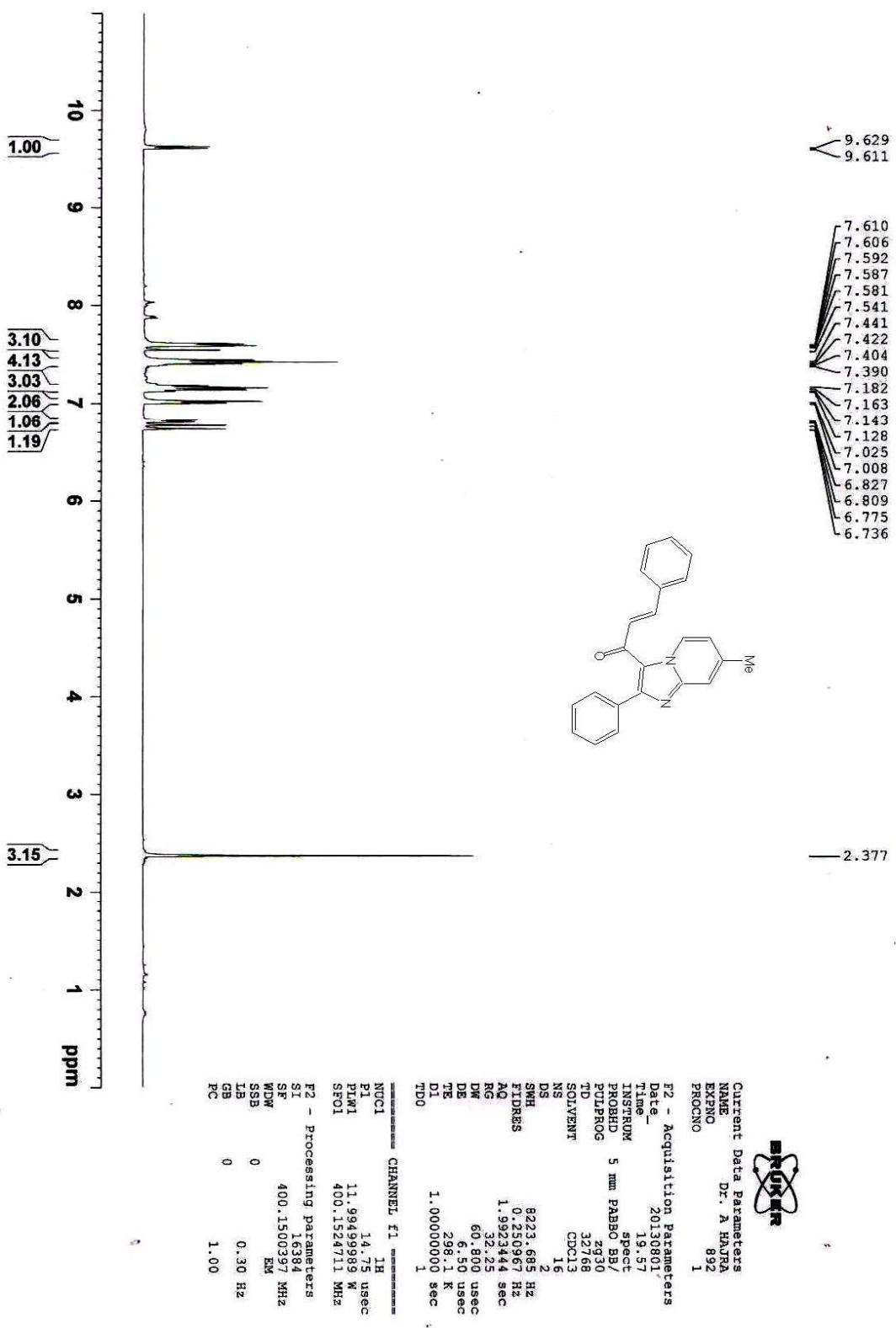
F2 - Acquisition Parameters
Date_ 20130731
Time_ 12:55
INSTRUM spect
PROBID PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.250867 Hz
AQ 1.992344 sec
RG 30.11
DW 60.800 usec
DE 6.50 usec
TE 305.7 K
D1 1.0000000 sec
TDO 1

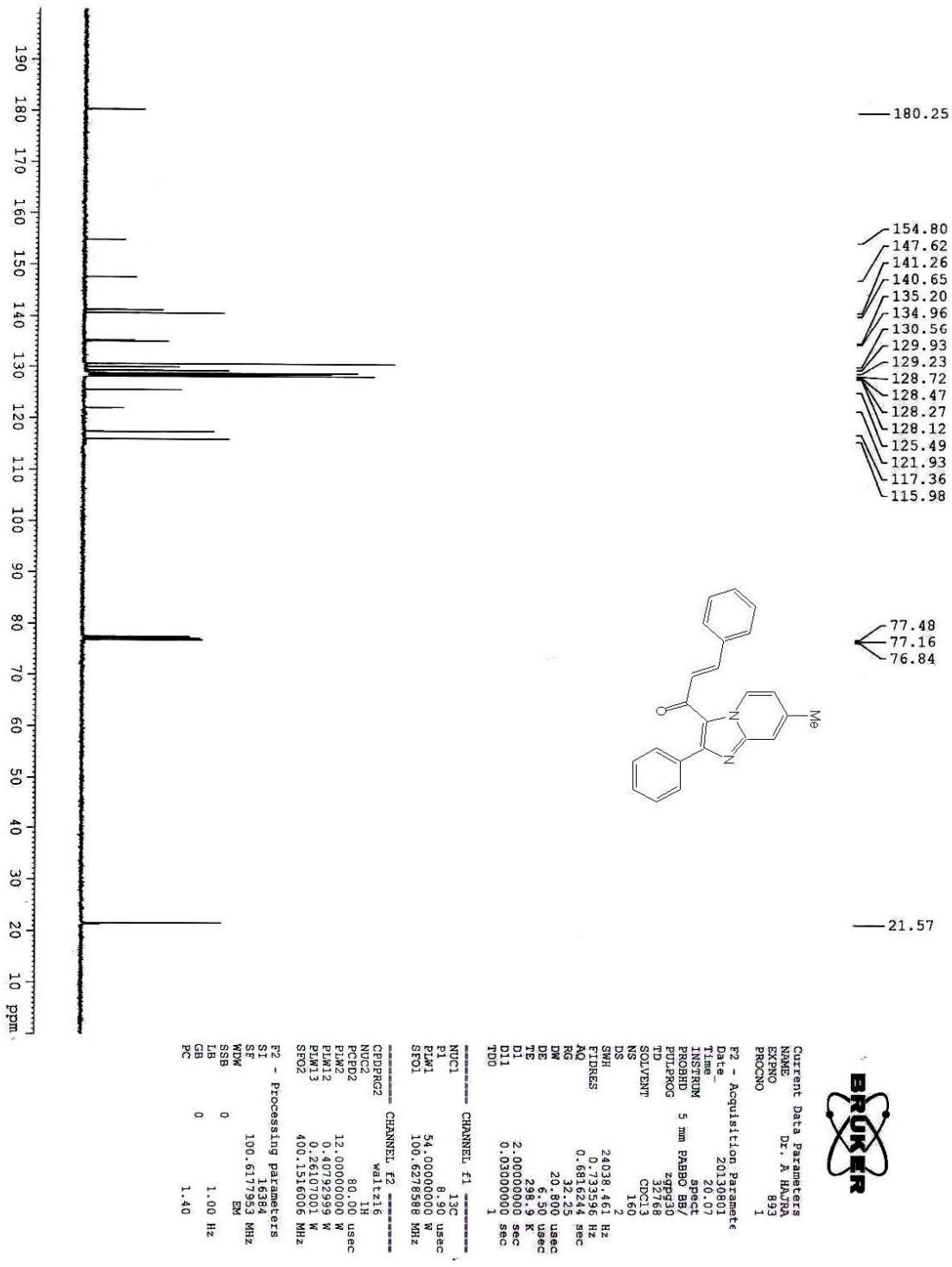
CHANNEL f1
NUC1 1H
P1 11.99414 1.75 usec
PR1 11.9949989 1.000 sec
SF01 400.1524711 MHz

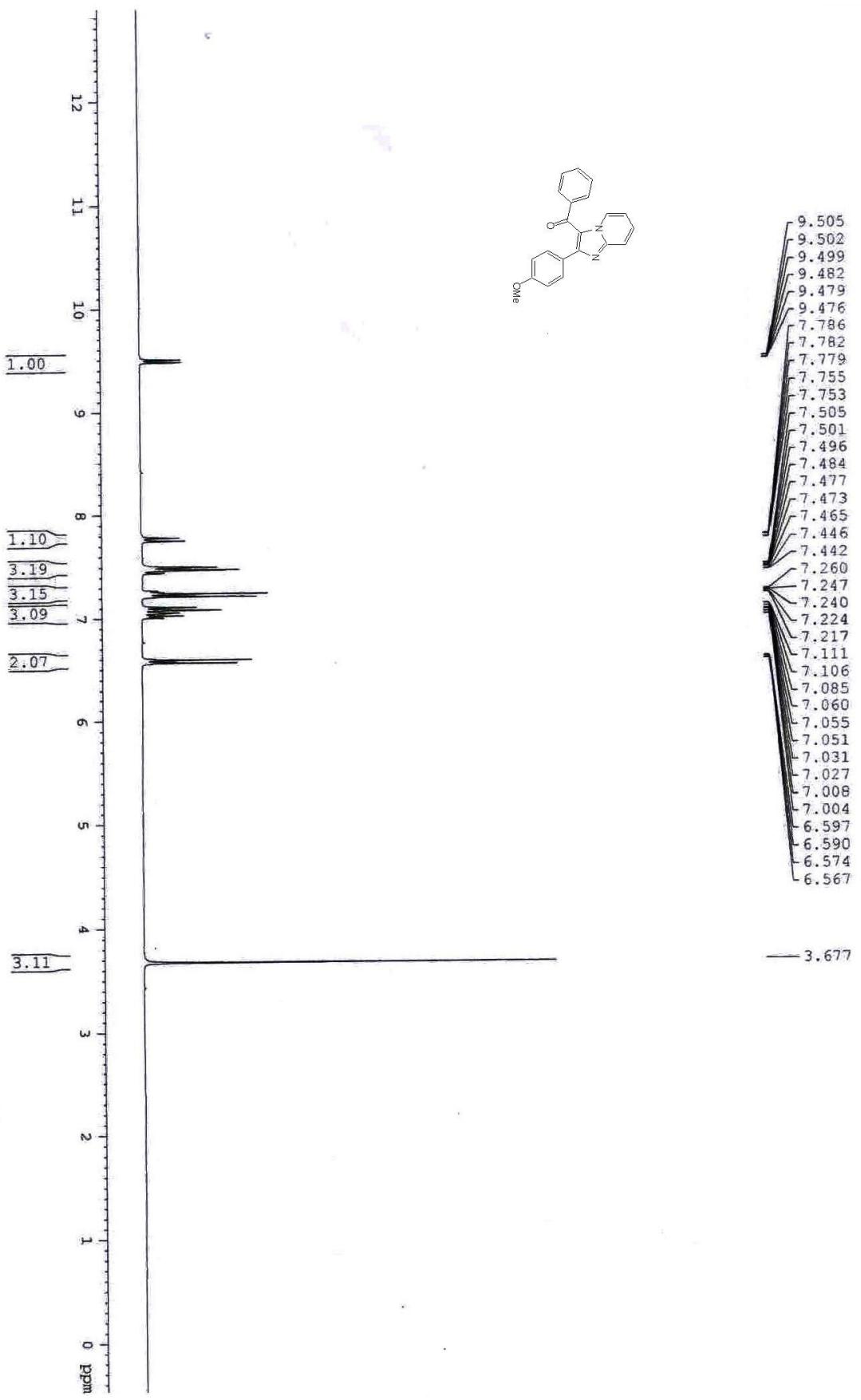
F2 - Processing Parameters
SI 16384
SF 400.150000 MHz
WDW EM
SSB 0
LB 0 0.30 Hz
GB 1.00
PC

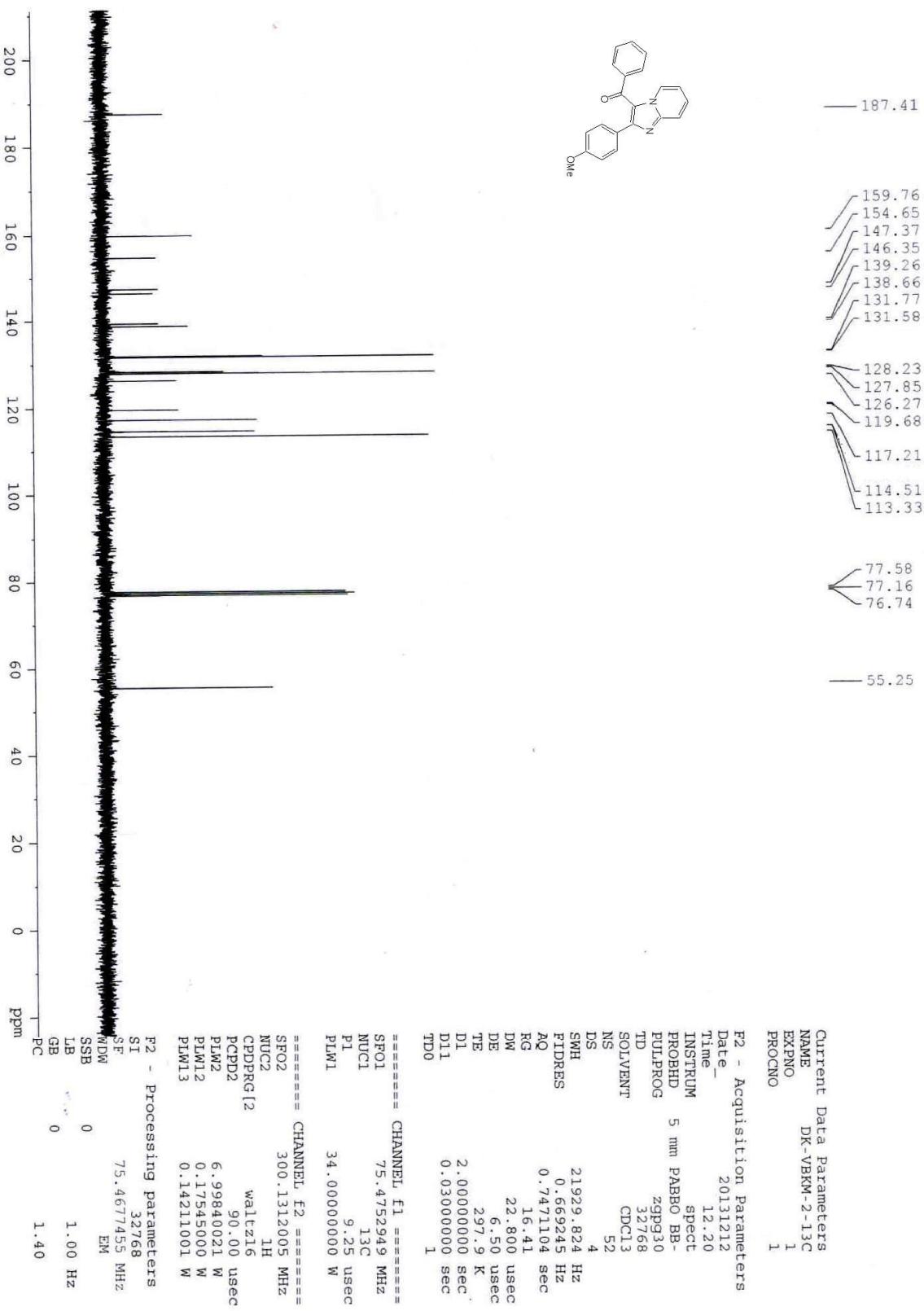


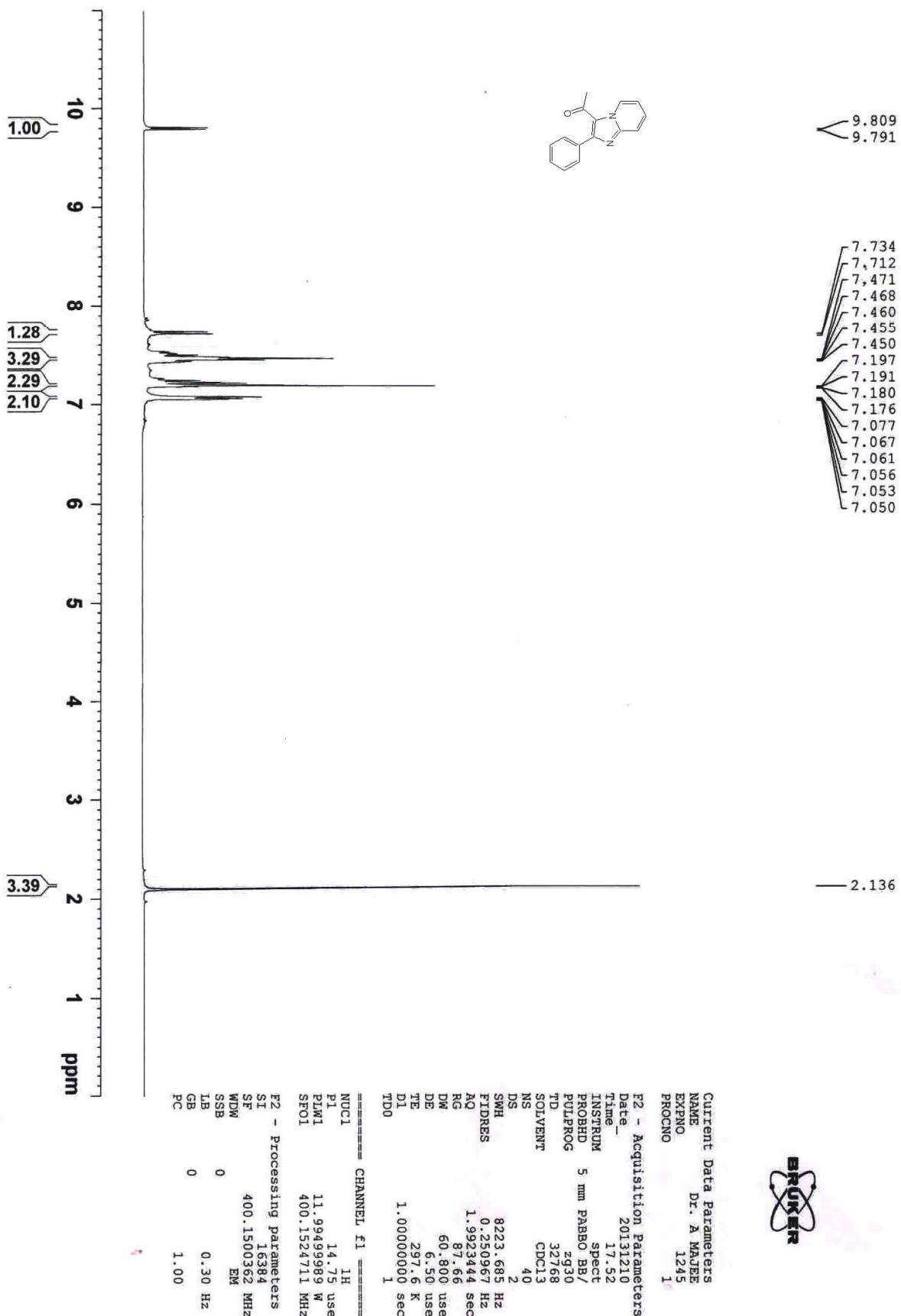


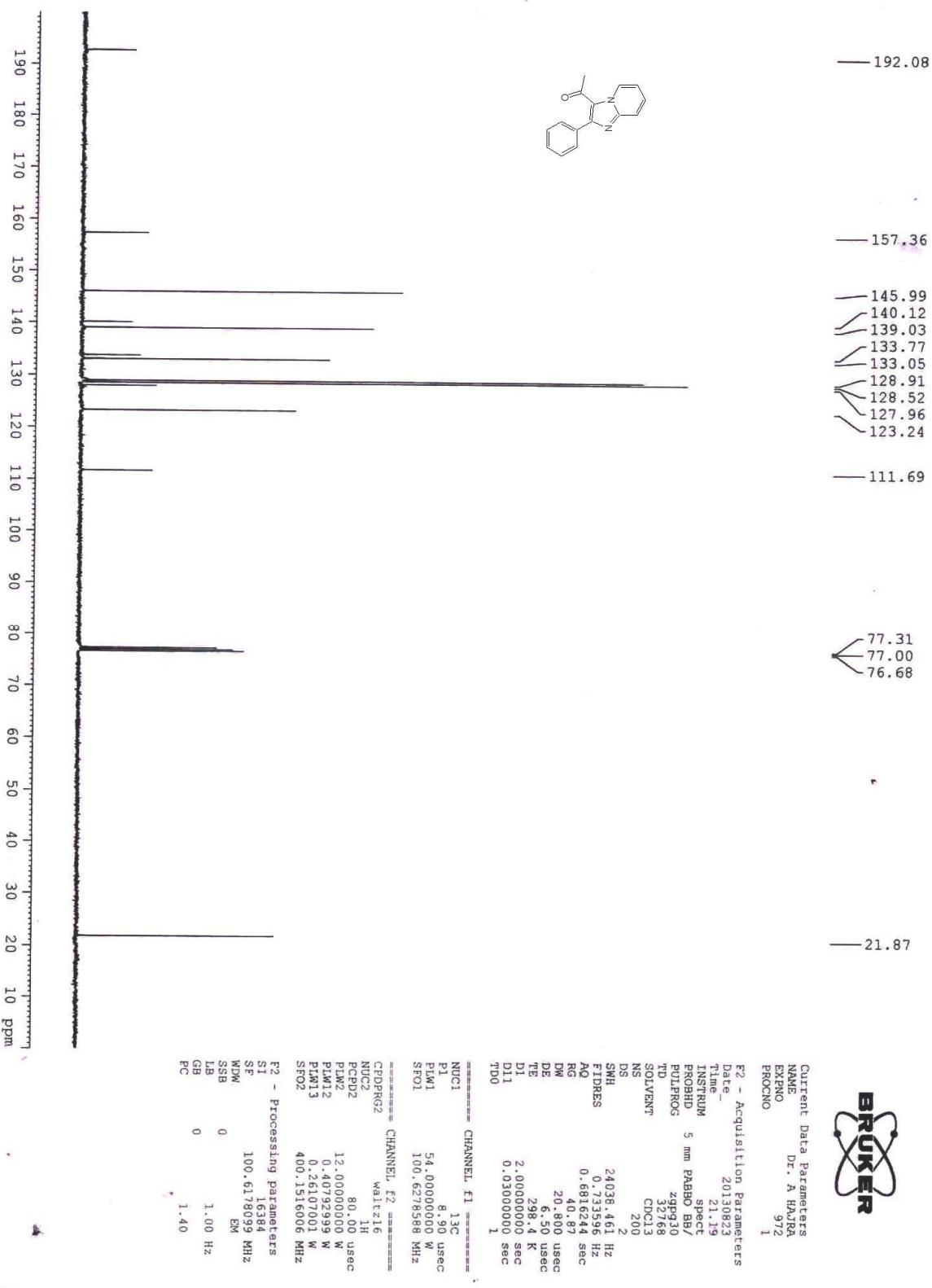




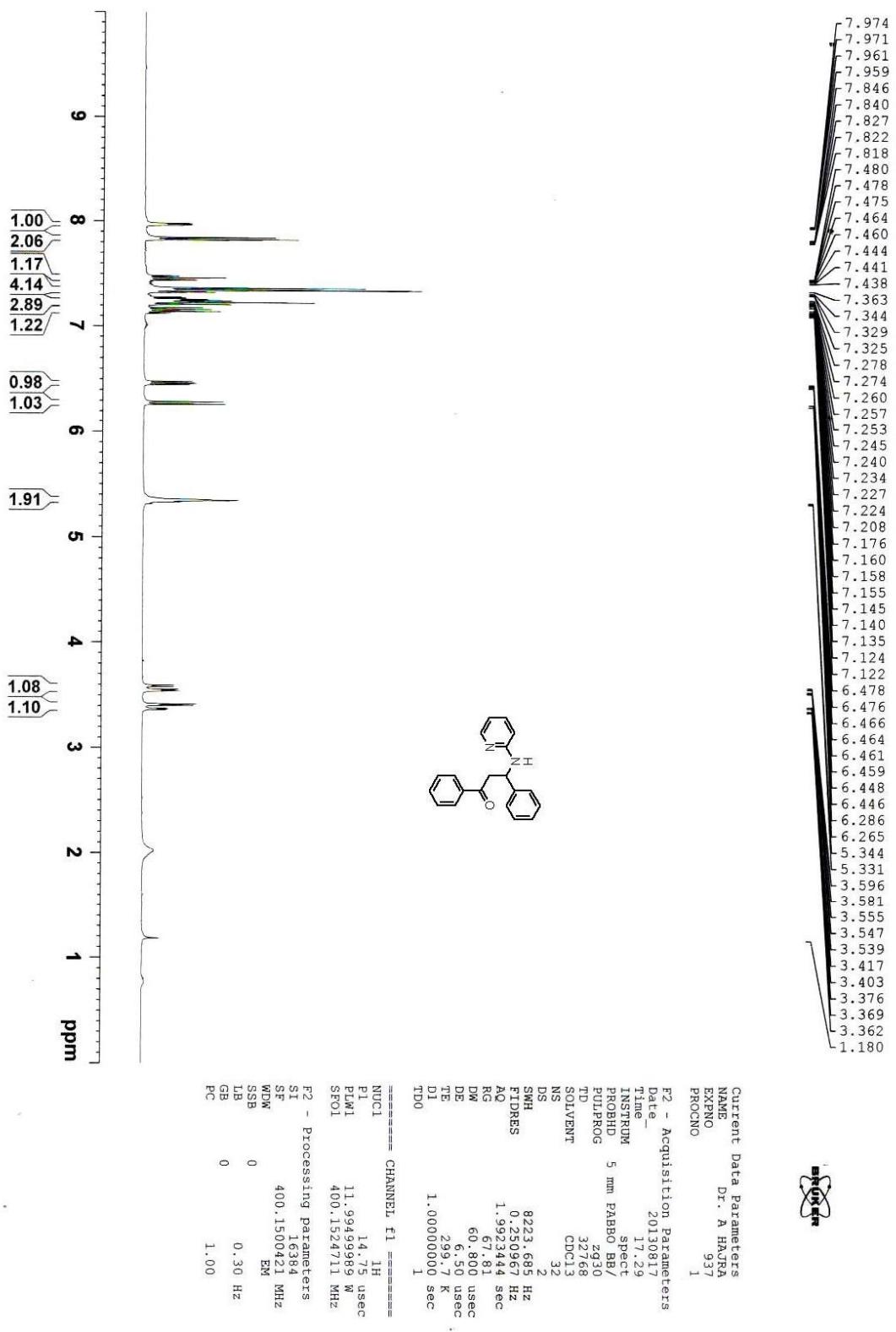


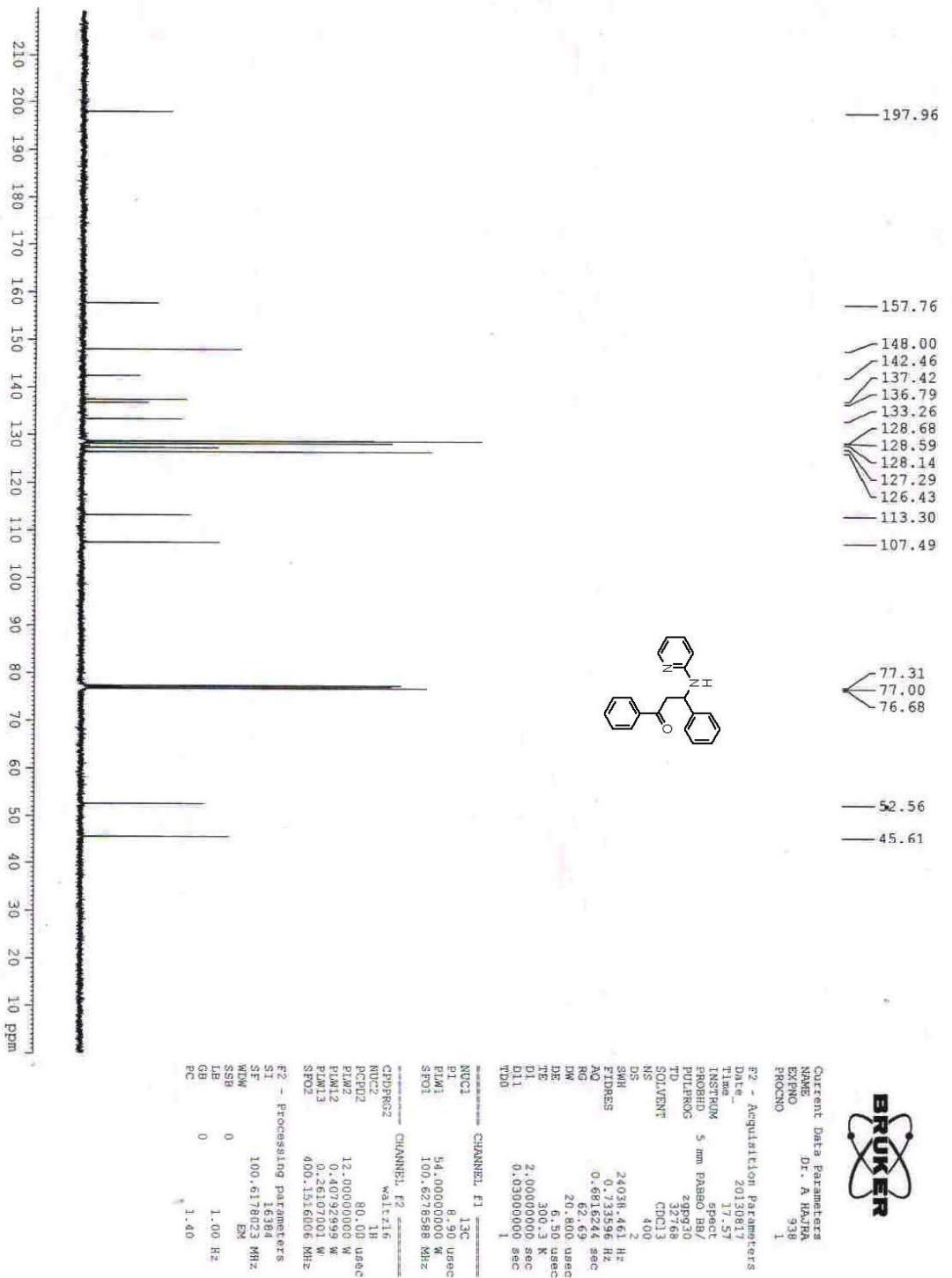






BRUKER





BRUKER

