

Facile and promising method for Michael addition of indole and pyrrole to electron deficient trans- β -nitroolefins catalyzed by a hydrogen bond donor catalyst Feist's acid; and Preliminary study of anti-microbial activity

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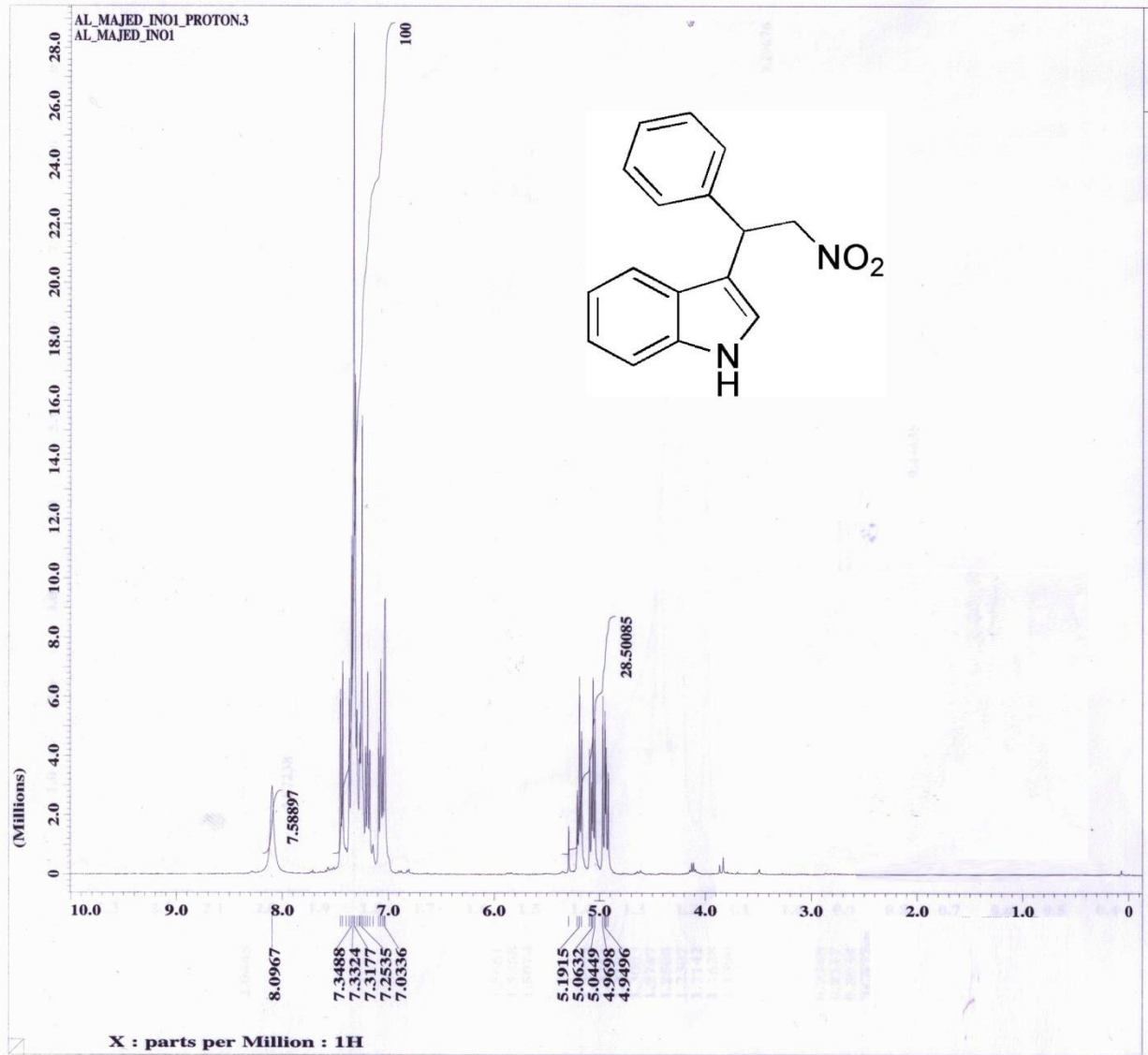
Generals:

Glassware was oven-dried overnight at 120 °C before use. Reactions were performed under an inert atmosphere using an argon filled glove box and standard Schlenk-line techniques. All the reactions were monitored by TLC analysis using Merck Silica Gel 60 F-254 thin layer plates. Column chromatography was performed on silica gel 100-200 mesh. **Materials:** Petroleum ether (PE), hexane and ethyl acetate for column chromatography were distilled prior to use. CH_2Cl_2 , EtOH were distilled from P_2O_5 and Mg respectively and stored on 4Å molecular sieves. Tetrahydrofuran, benzene, toluene were distilled from sodium benzophenone ketyl. Acetonitrile and dimethylformamide were dried by distillation over calcium hydride. Nitroolefins **2a – i** were prepared according to procedures reported in literature.¹ **Instrumentation:** NMR spectra were recorded with a Jeol spectrometer at 400 MHz (^1H -NMR) and 100 MHz (^{13}C -NMR.). The chemical shifts (δ in ppm) were reported down field from tetramethylsilane (TMS, δ scale) with the deuterated solvent resonance referenced as internal standard. Elemental analyses were performed on a Perkin Elmer 2400 Elemental Analyzer. IR spectra were obtained using FTIR-800 Model. Mass spectrometric analysis was conducted by using ESI mode on AGILENT Technologies 6410-triple quad LC/MS instrument.

Reference:

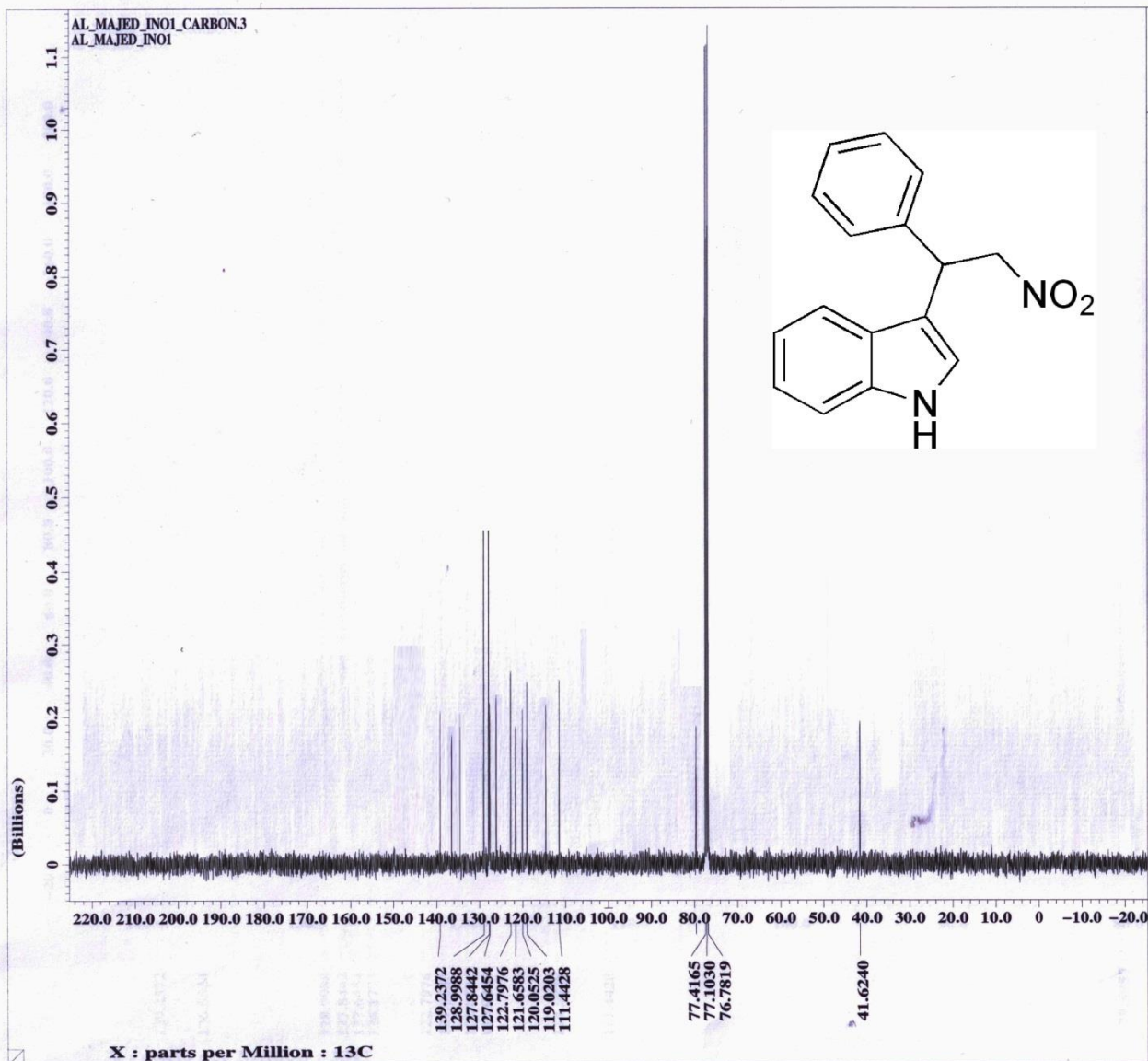
1. N. A. Lange and W. E. Hamburger, "Condensation of aromatic aldehydes with nitromethane in the presence of alcoholic sodium hydroxide," *Journal of the American Chemical Society*, vol. 53, no. 10, pp. 3865–3867, 1931.

Compound 7a: ¹H-NMR



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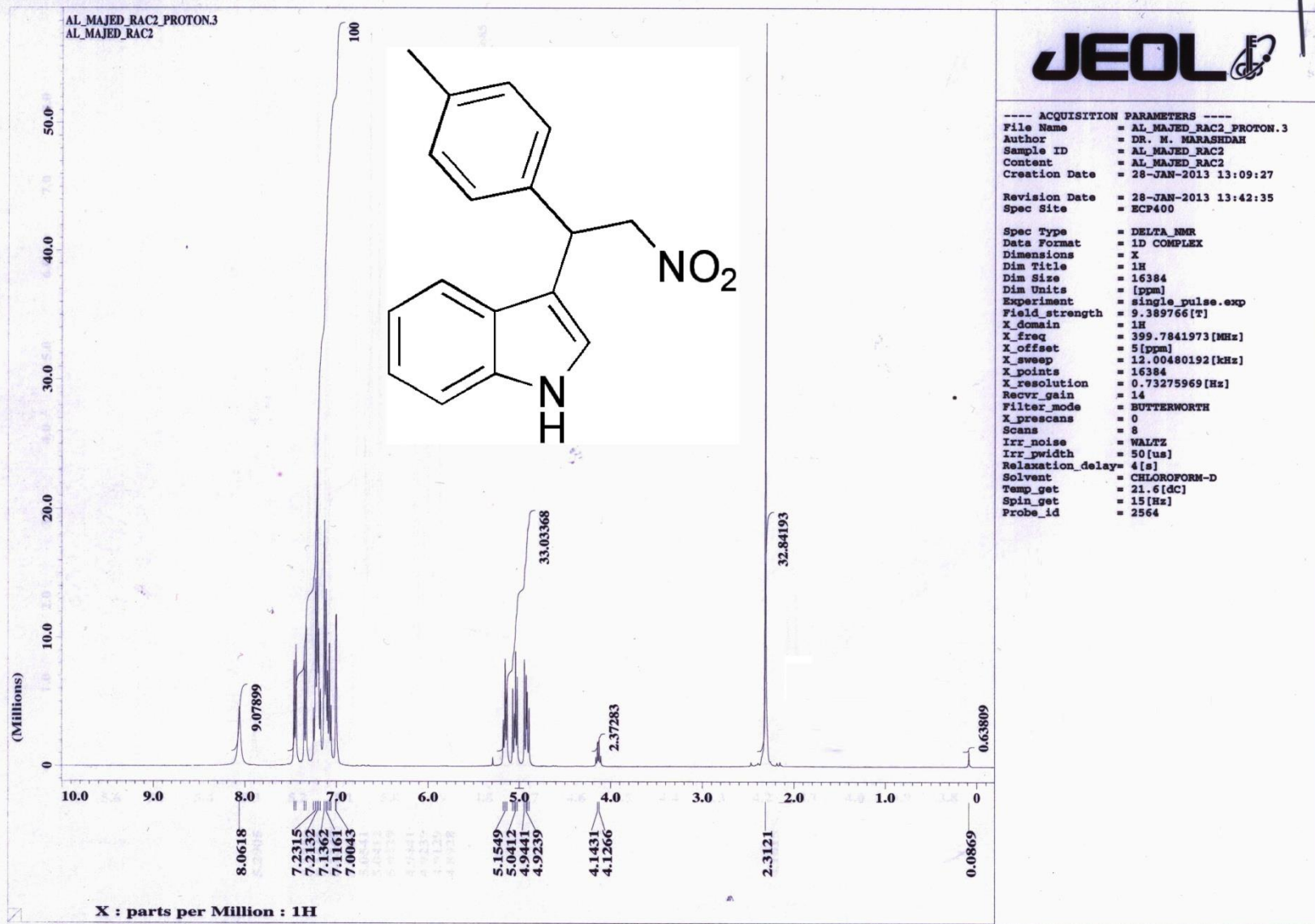
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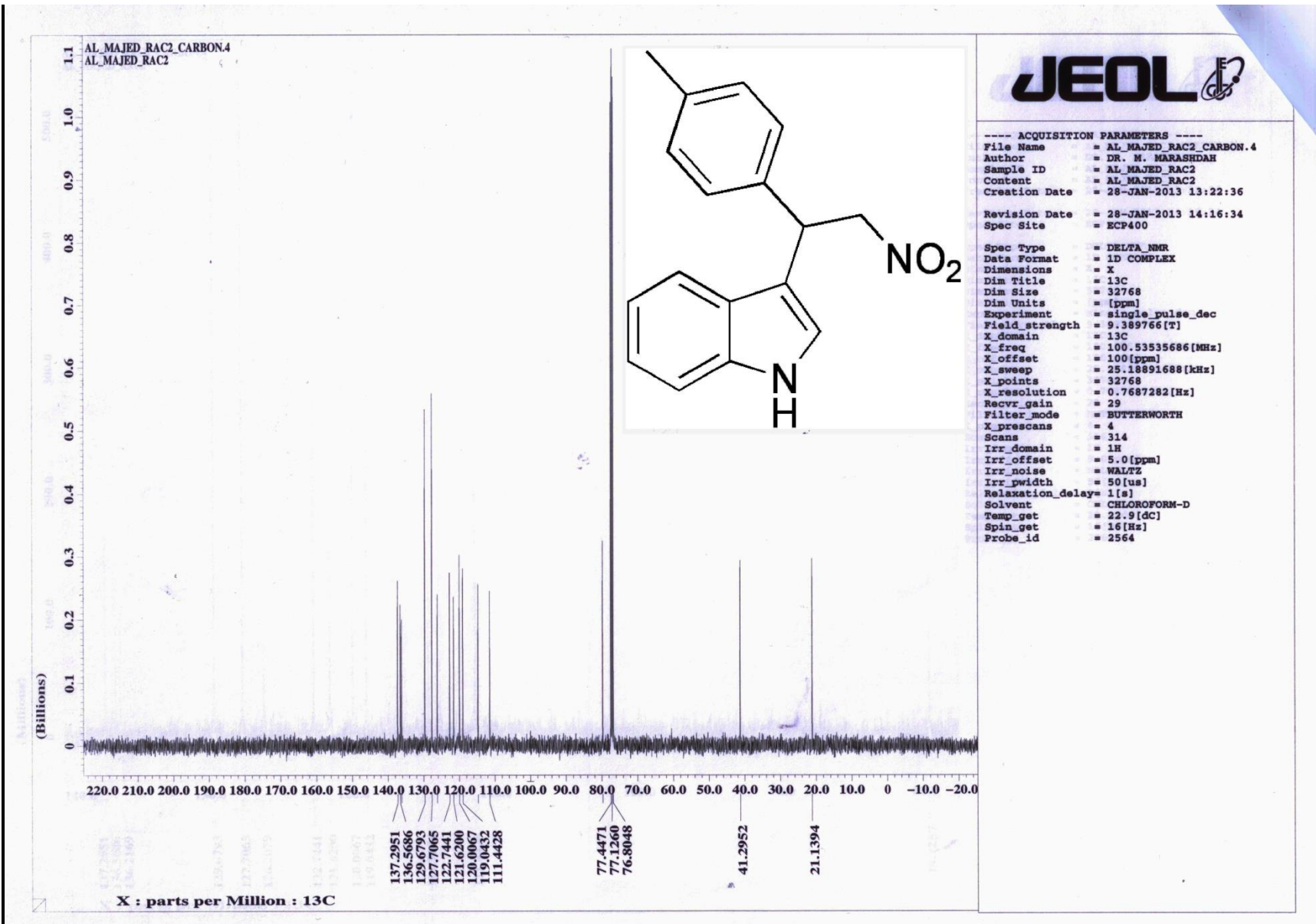
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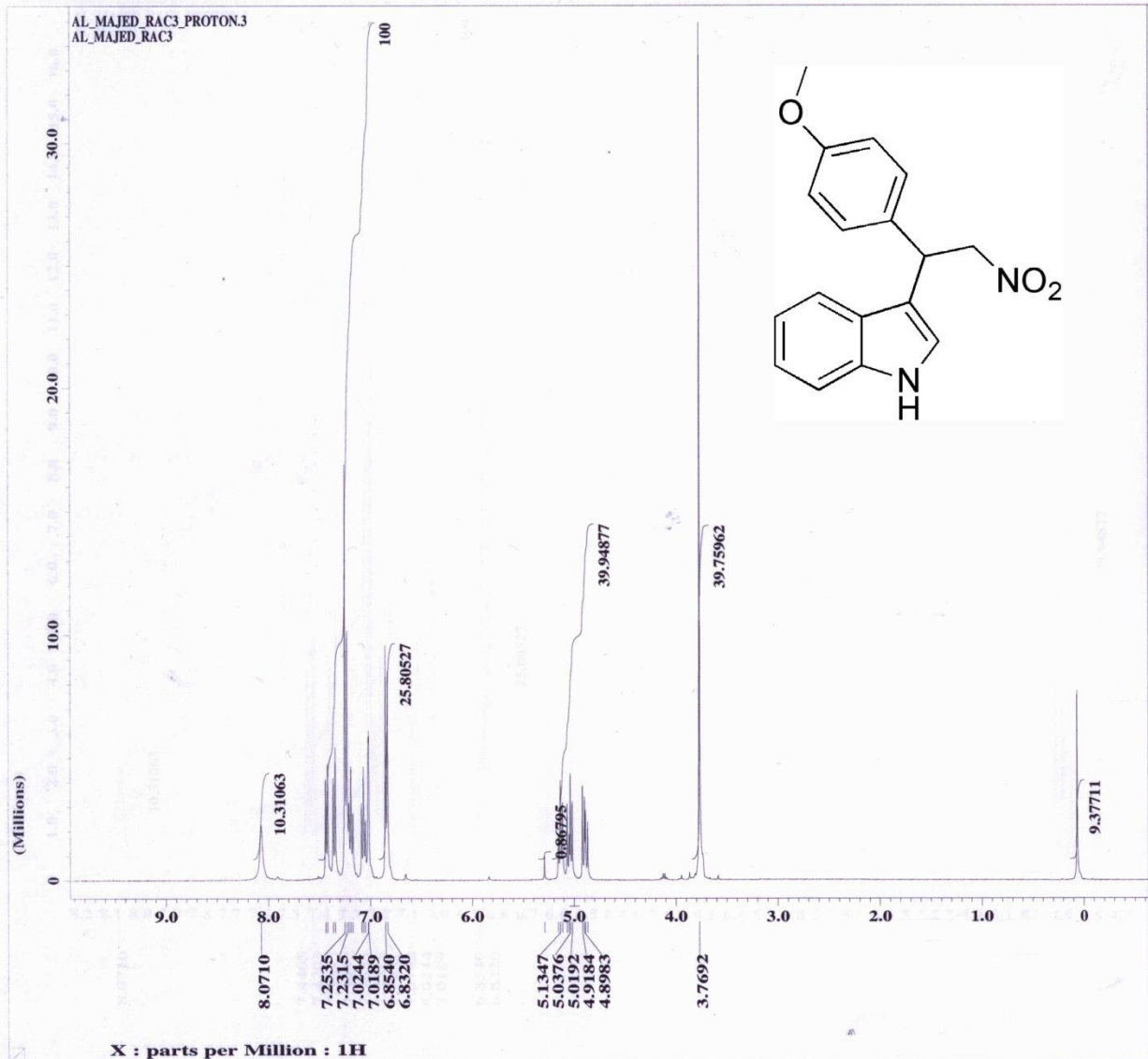
Compound 7b: ¹H-NMR



Compound 7b: ¹³C-NMR

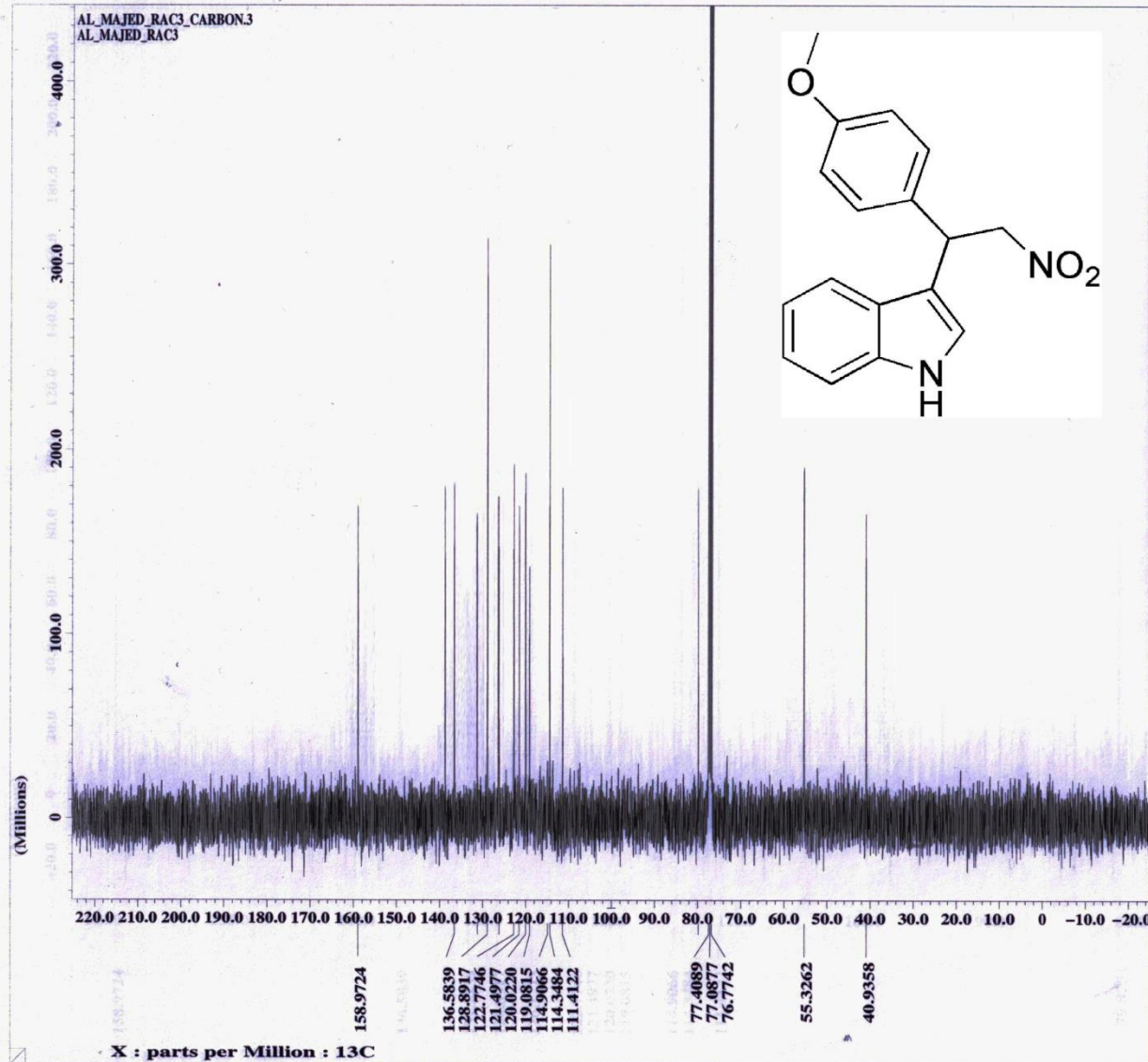


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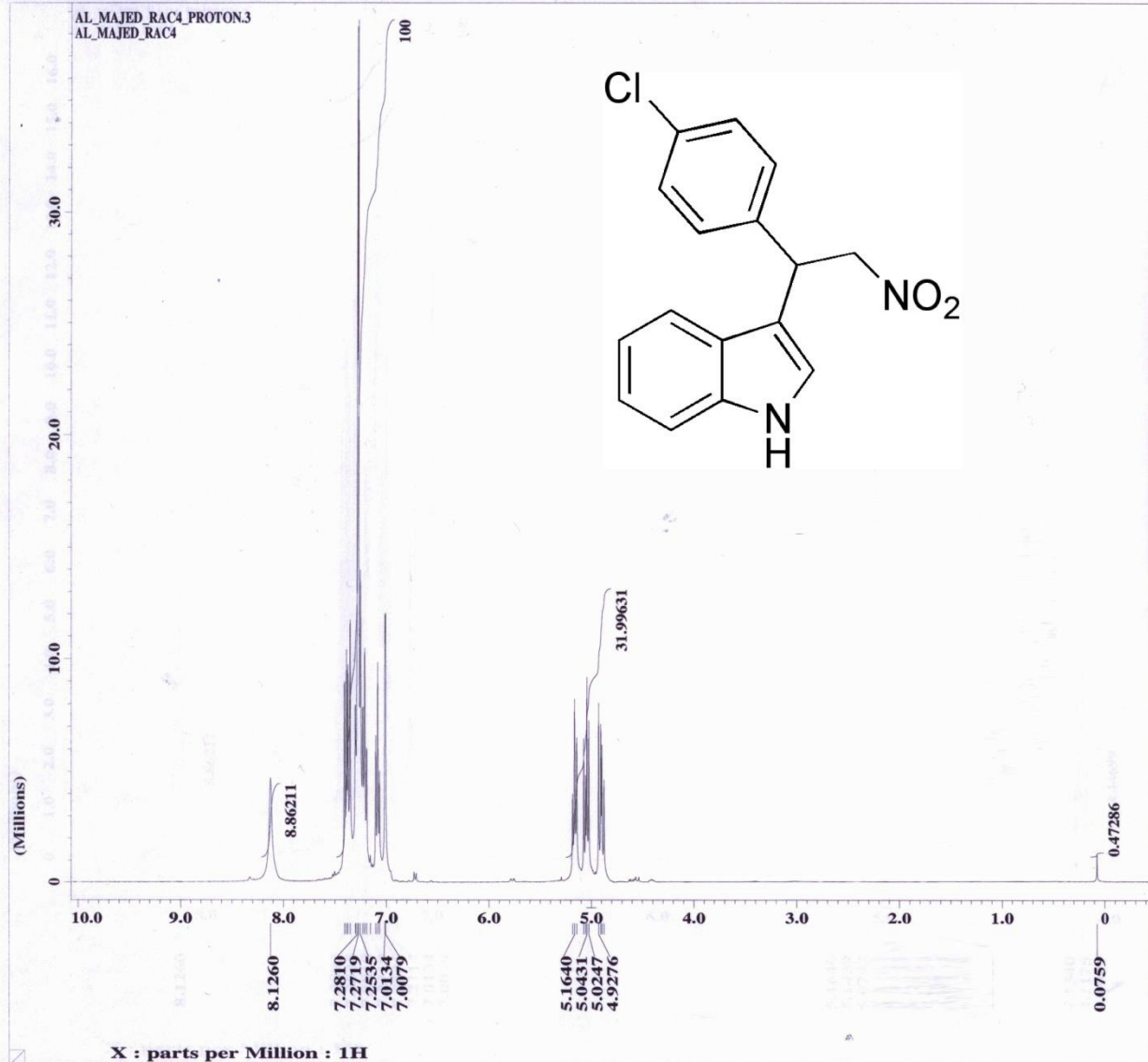
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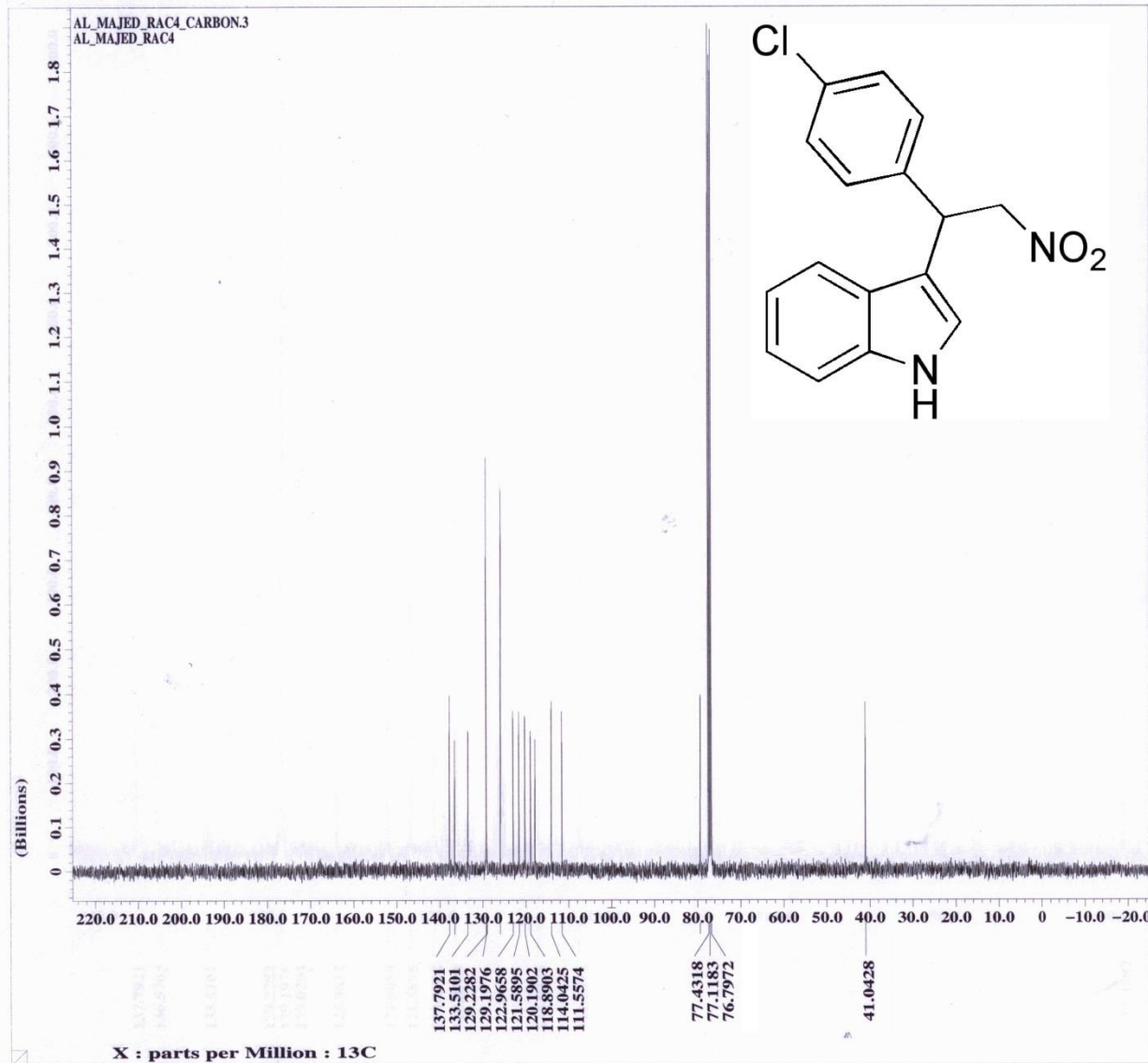
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Compound 7d: ¹³C-NMR

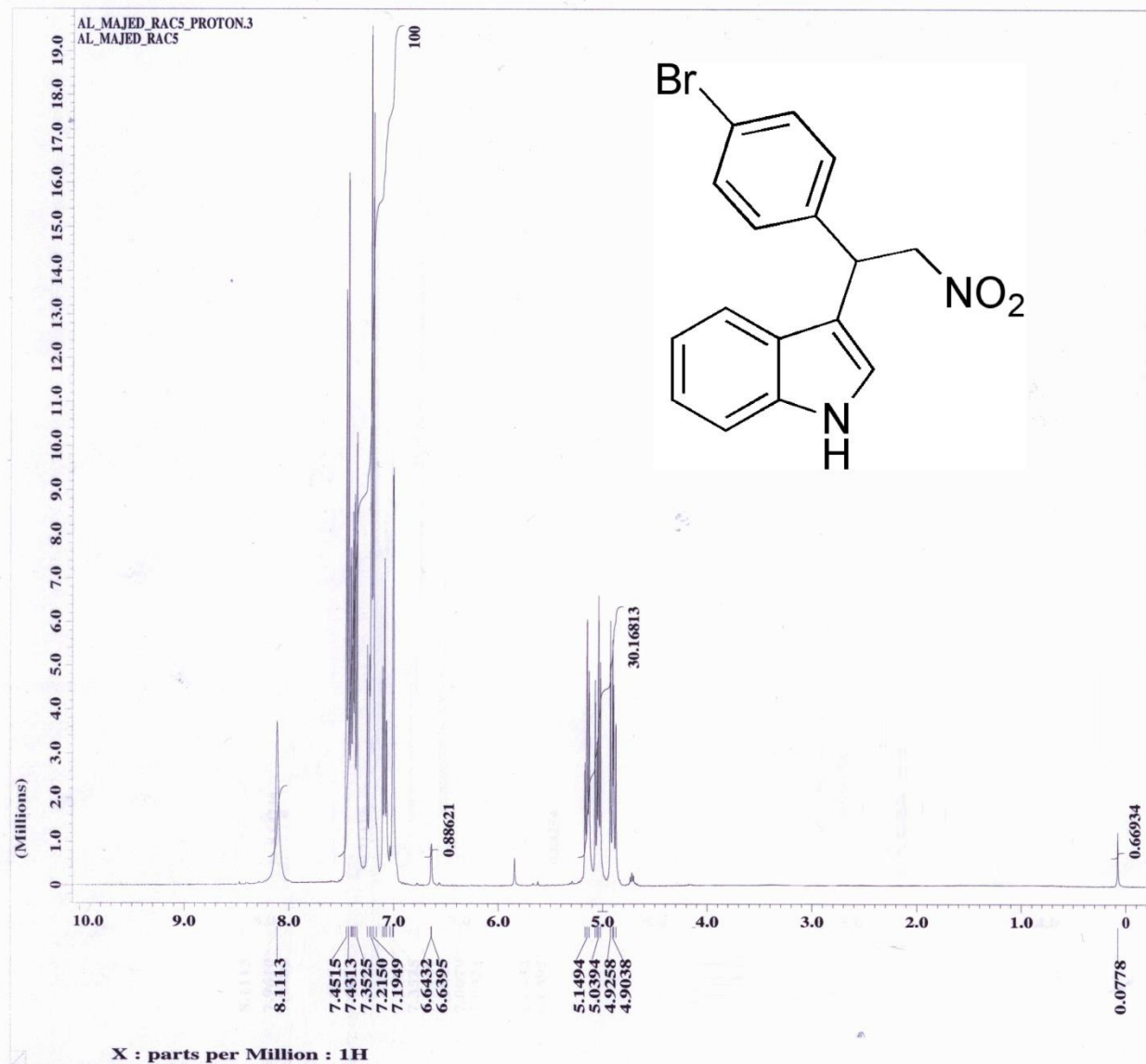


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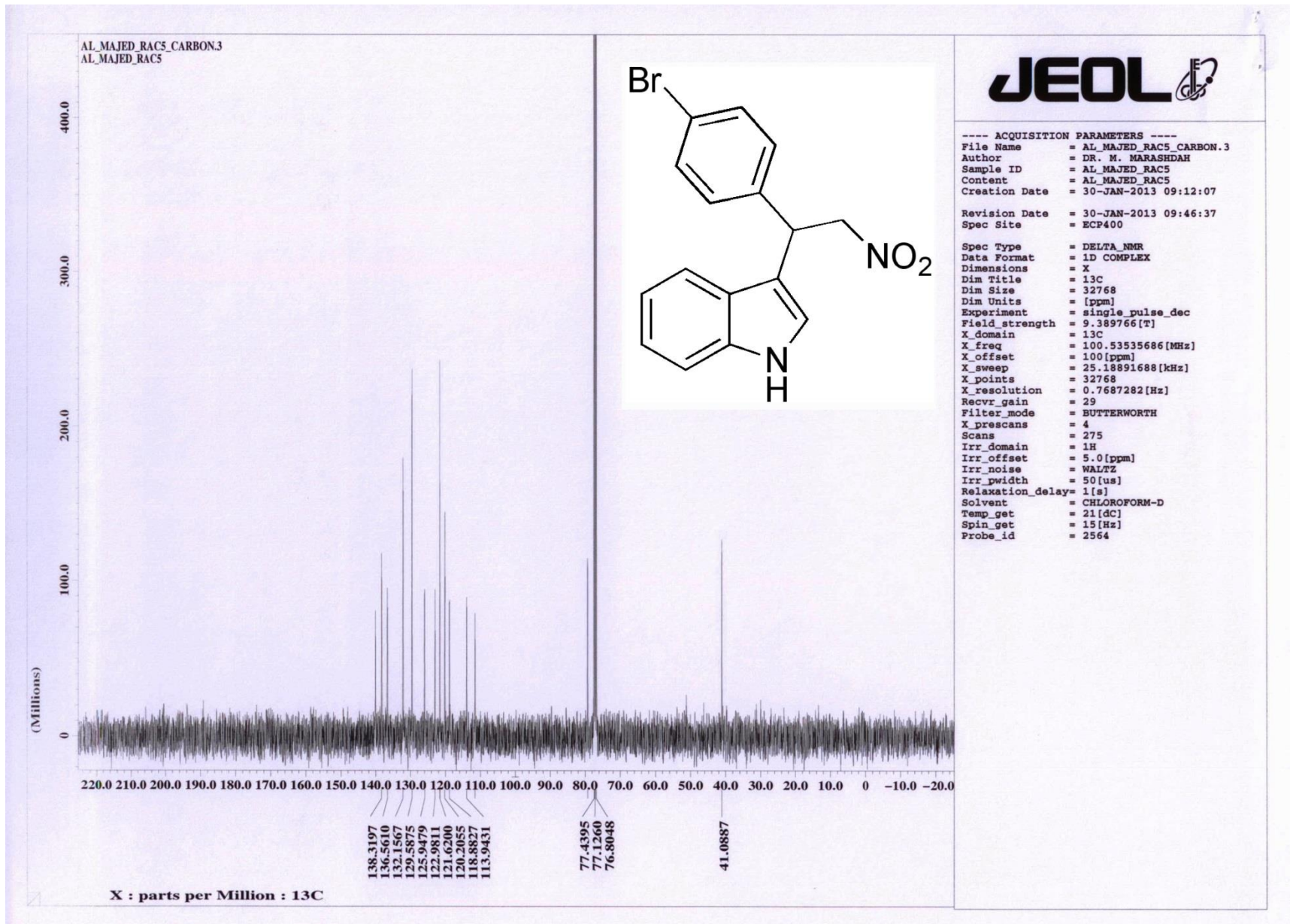
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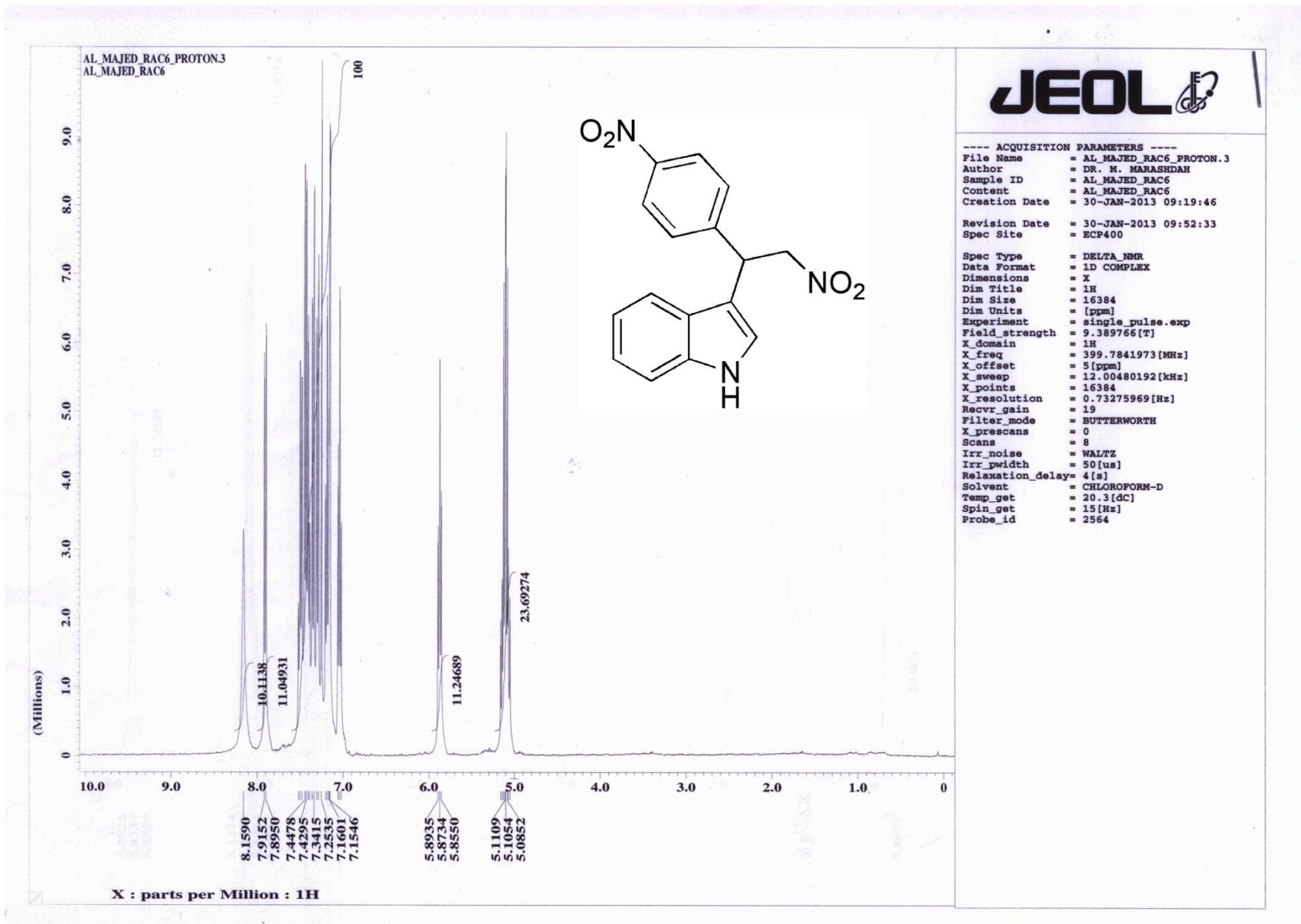
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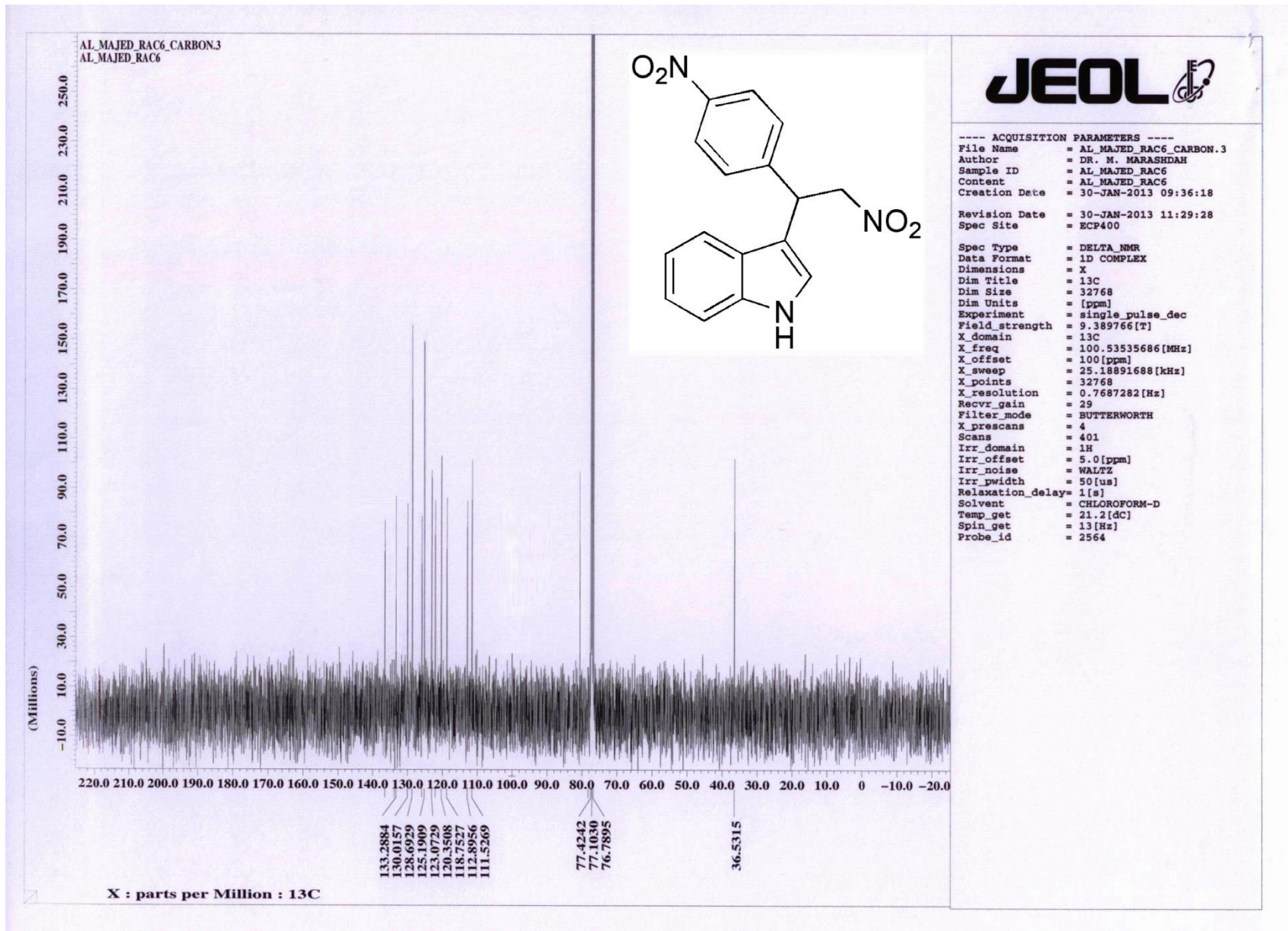
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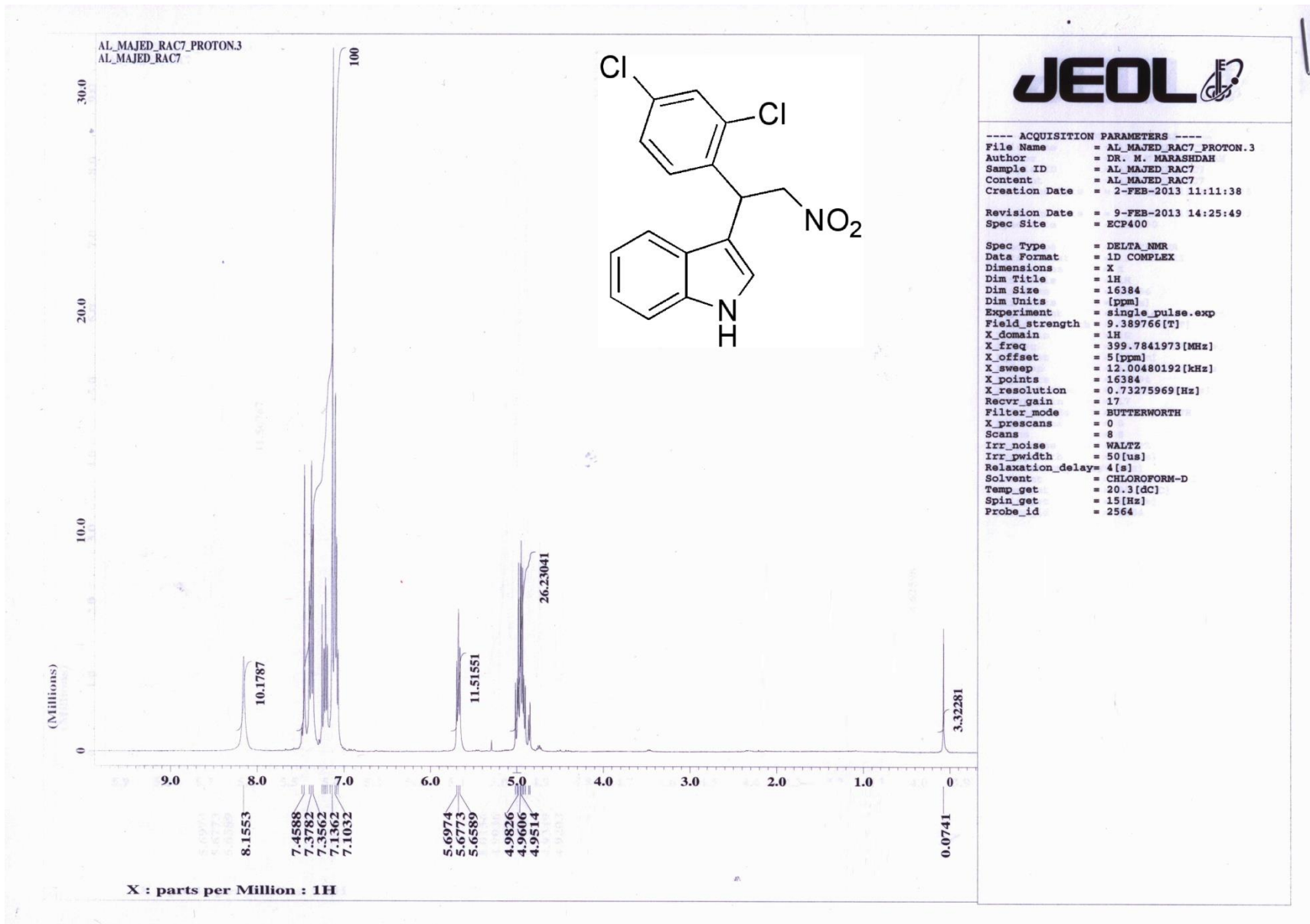
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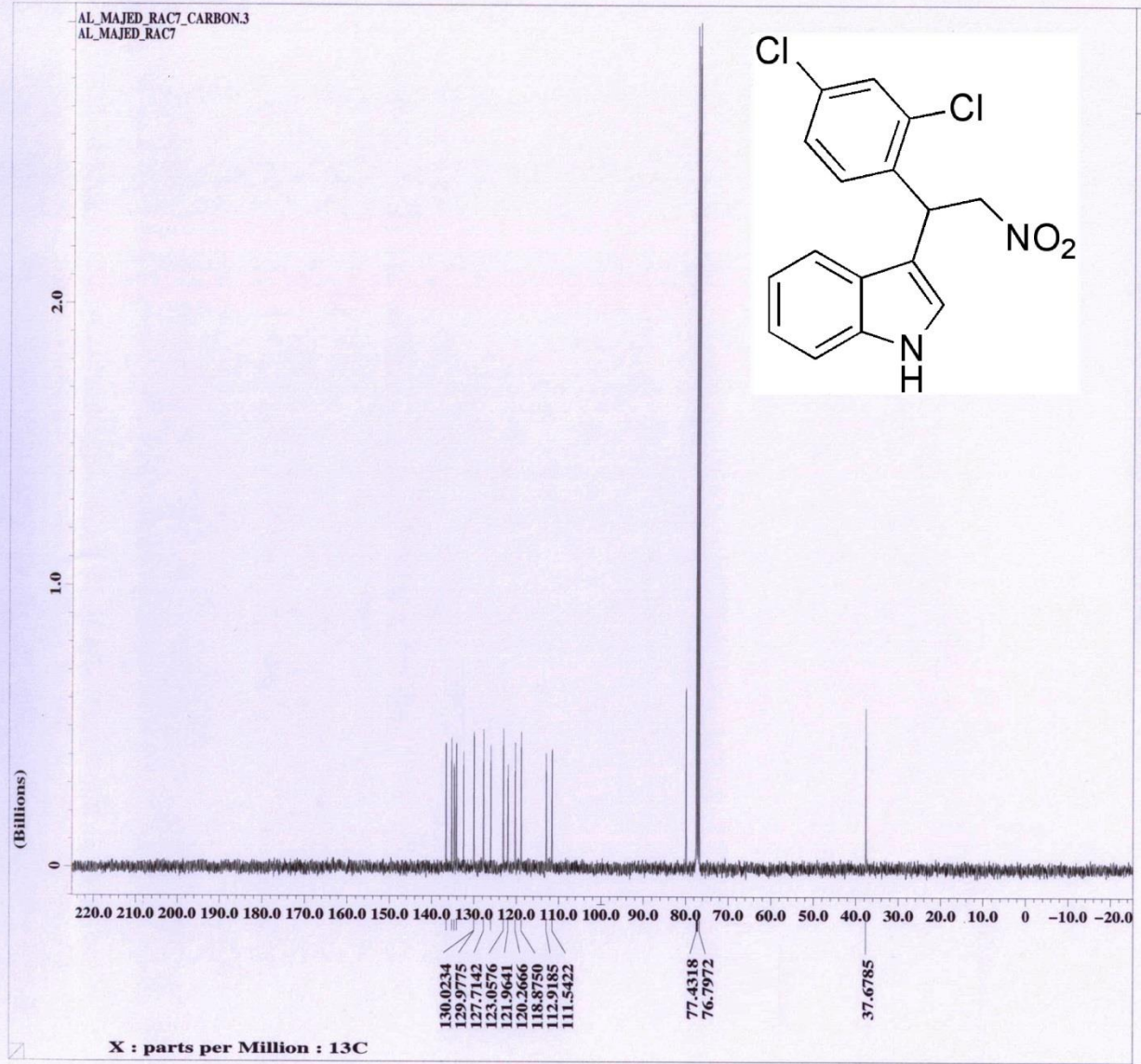
Compound 7f: ¹³C-NMR



Compound 7g: ¹H-NMR



Compound 7g: ¹³C-NMR



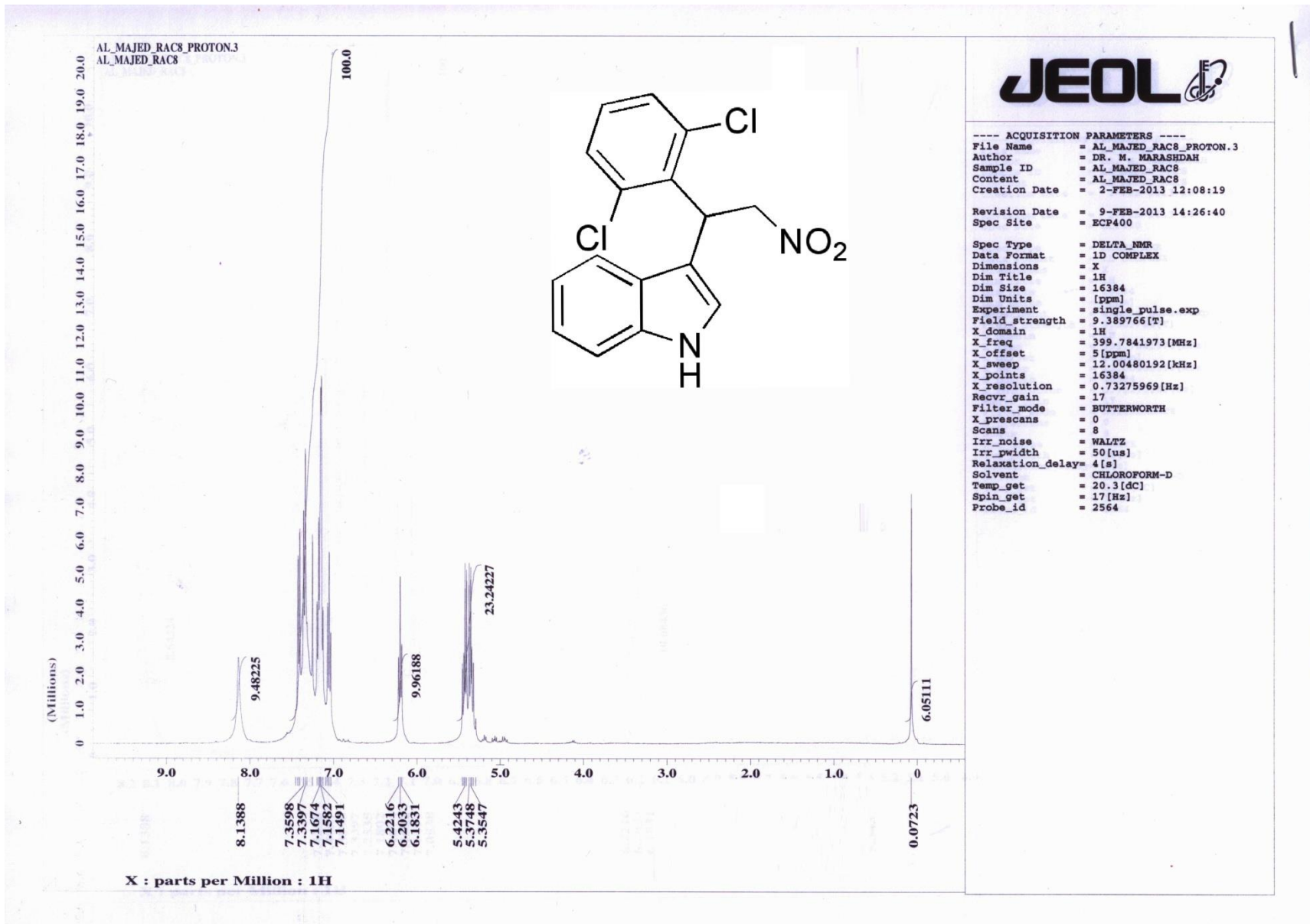
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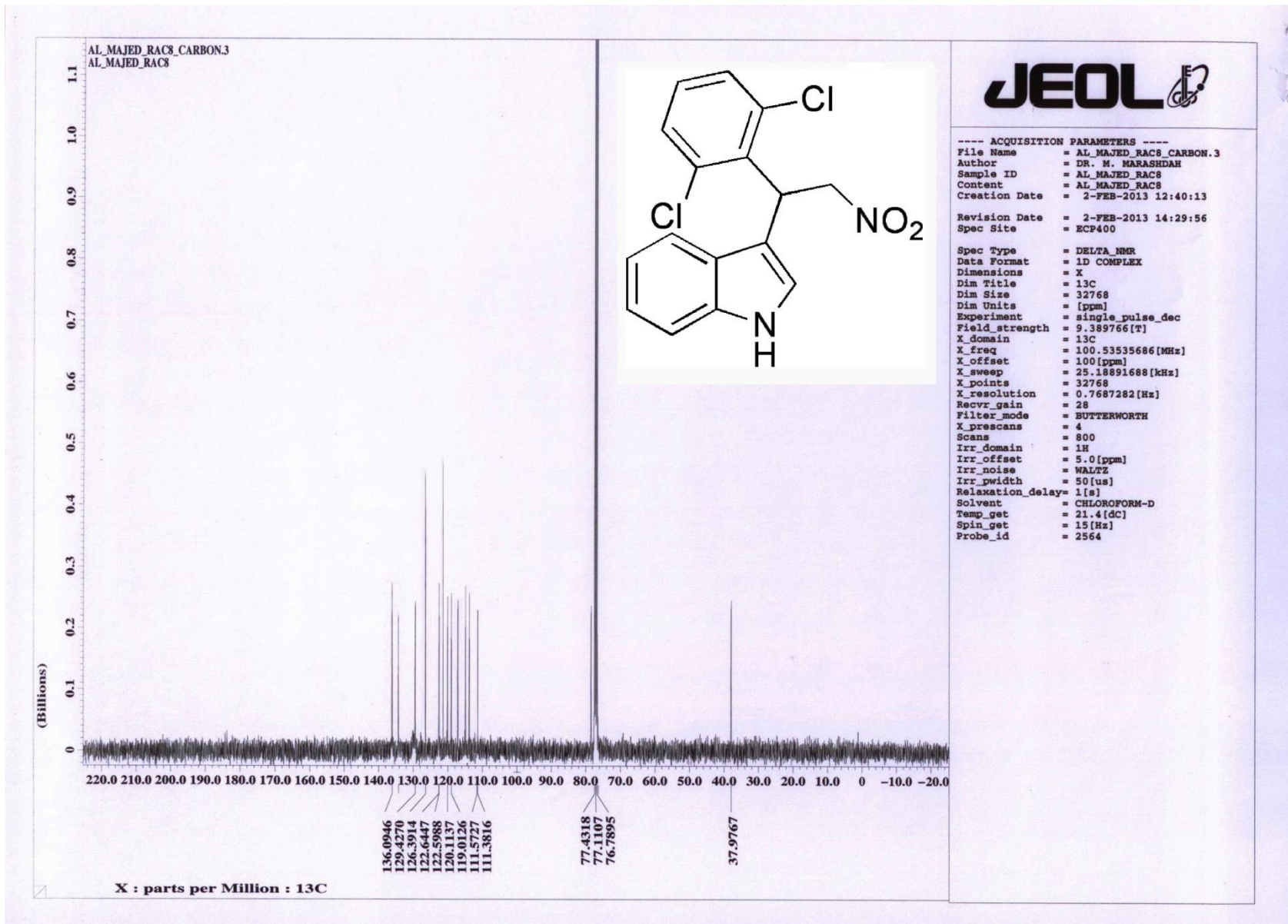
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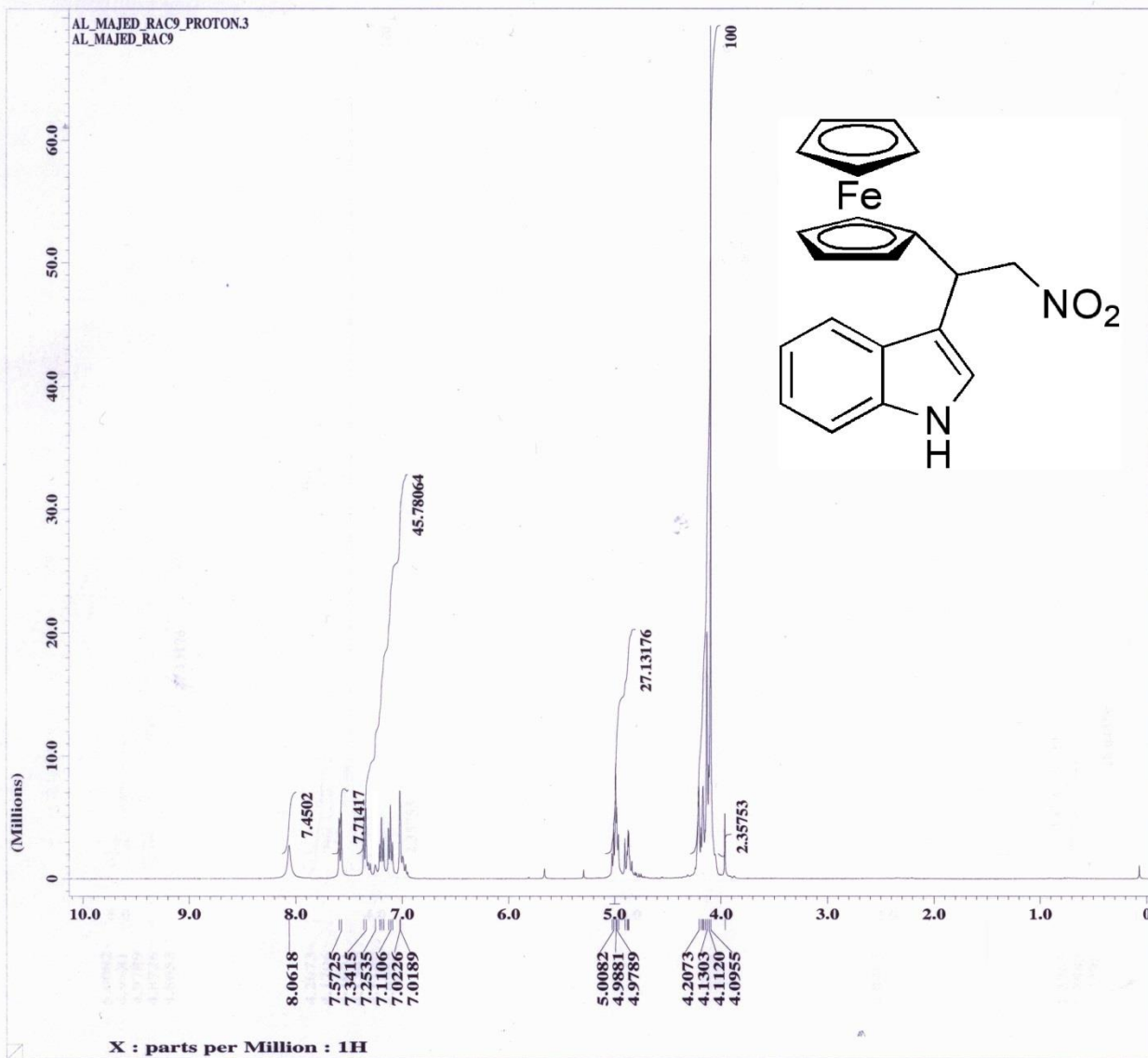
Compound 7h: ¹H-NMR



Compound 7h: ¹³C-NMR



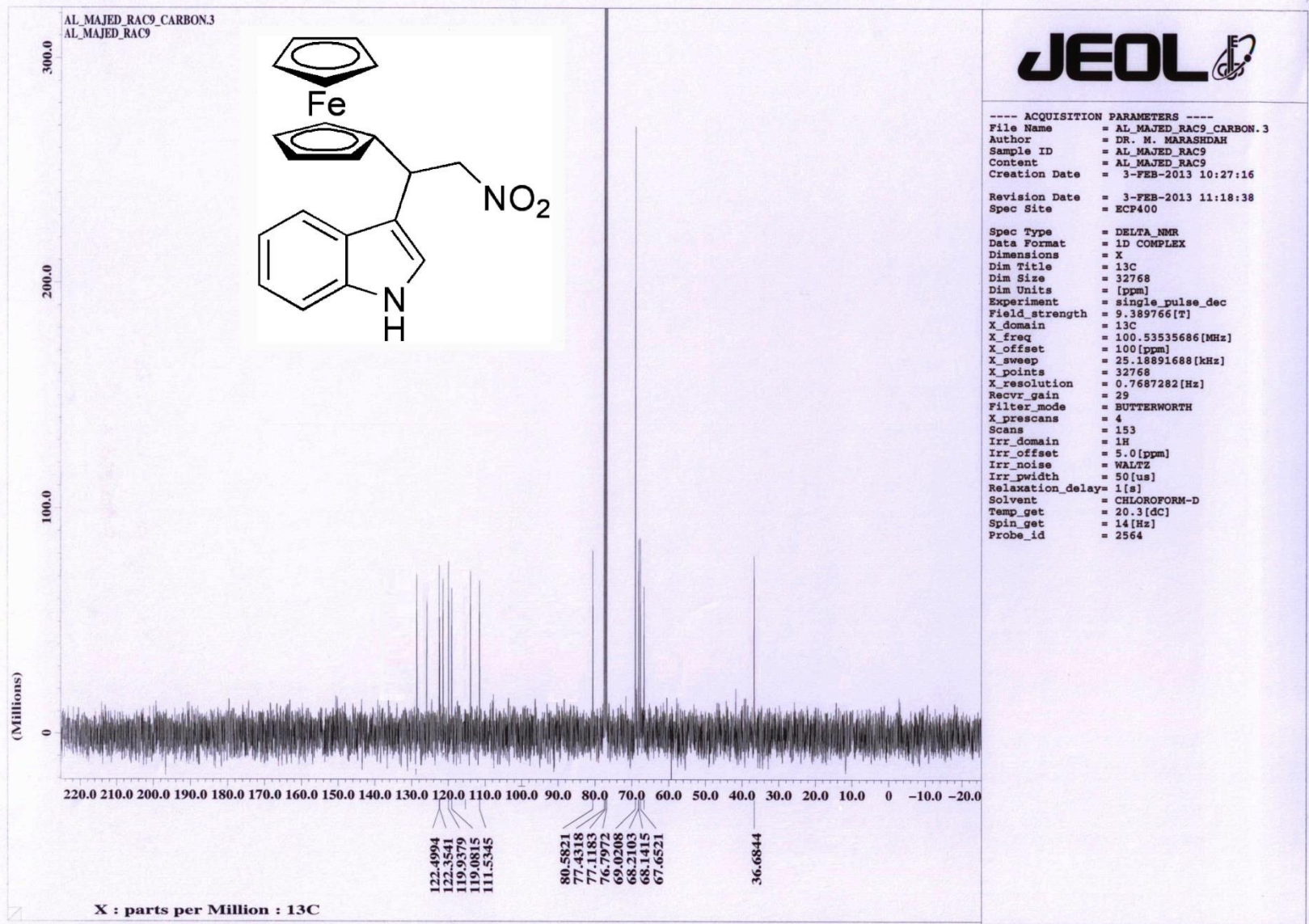
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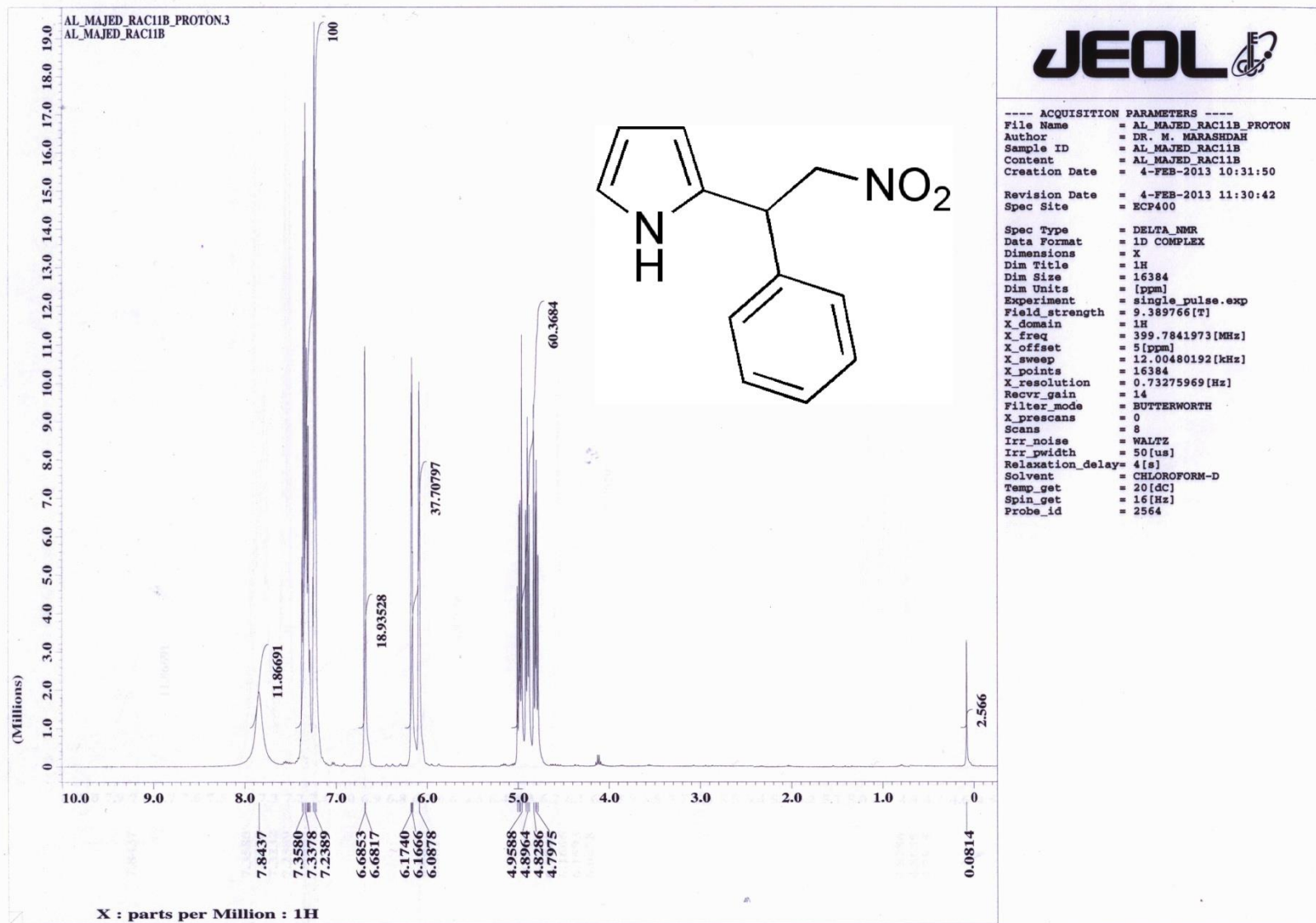
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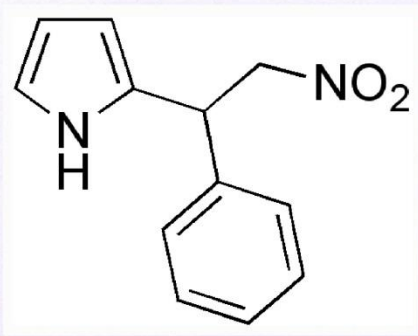
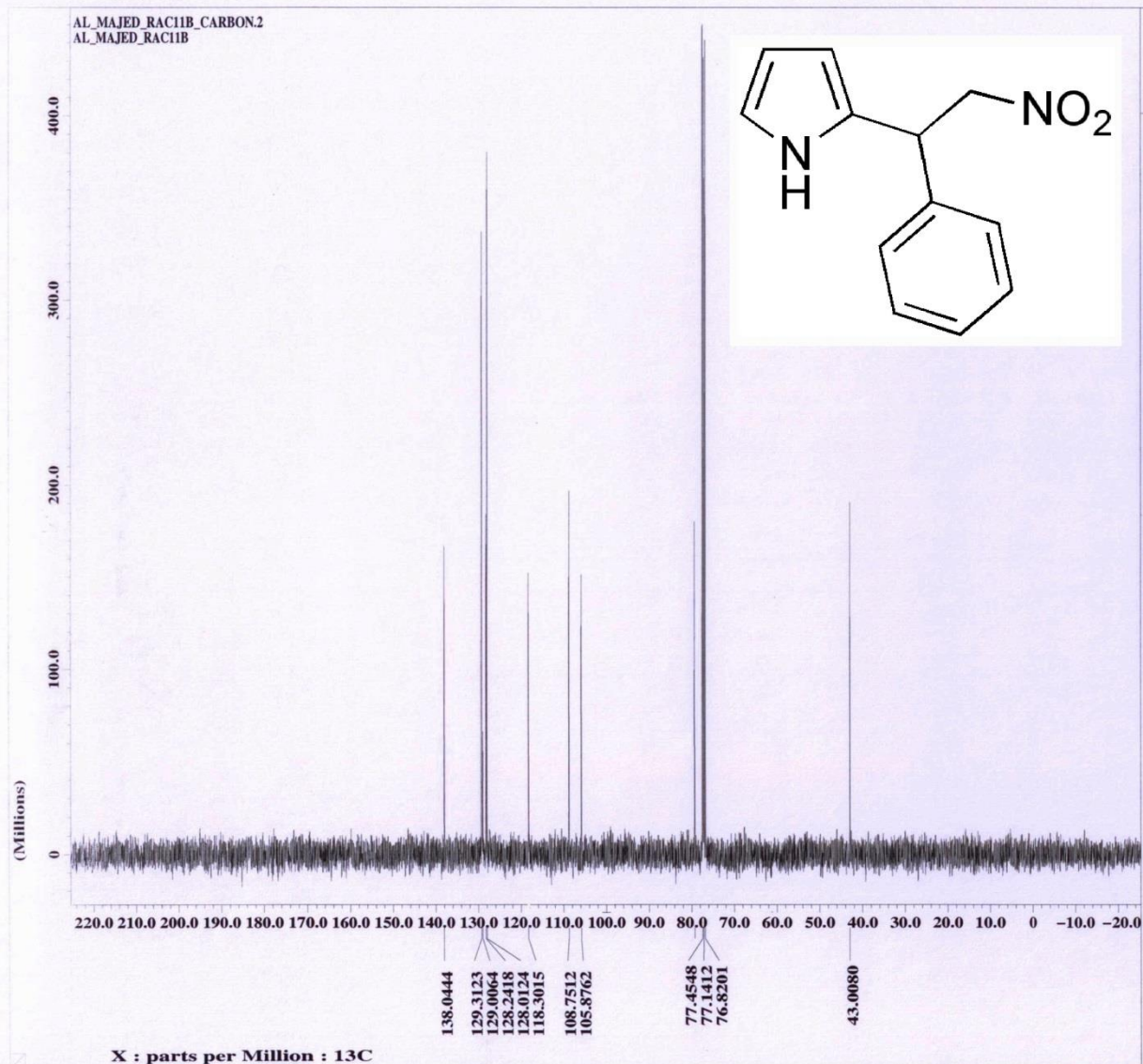
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Compound 8a: ¹H-NMR



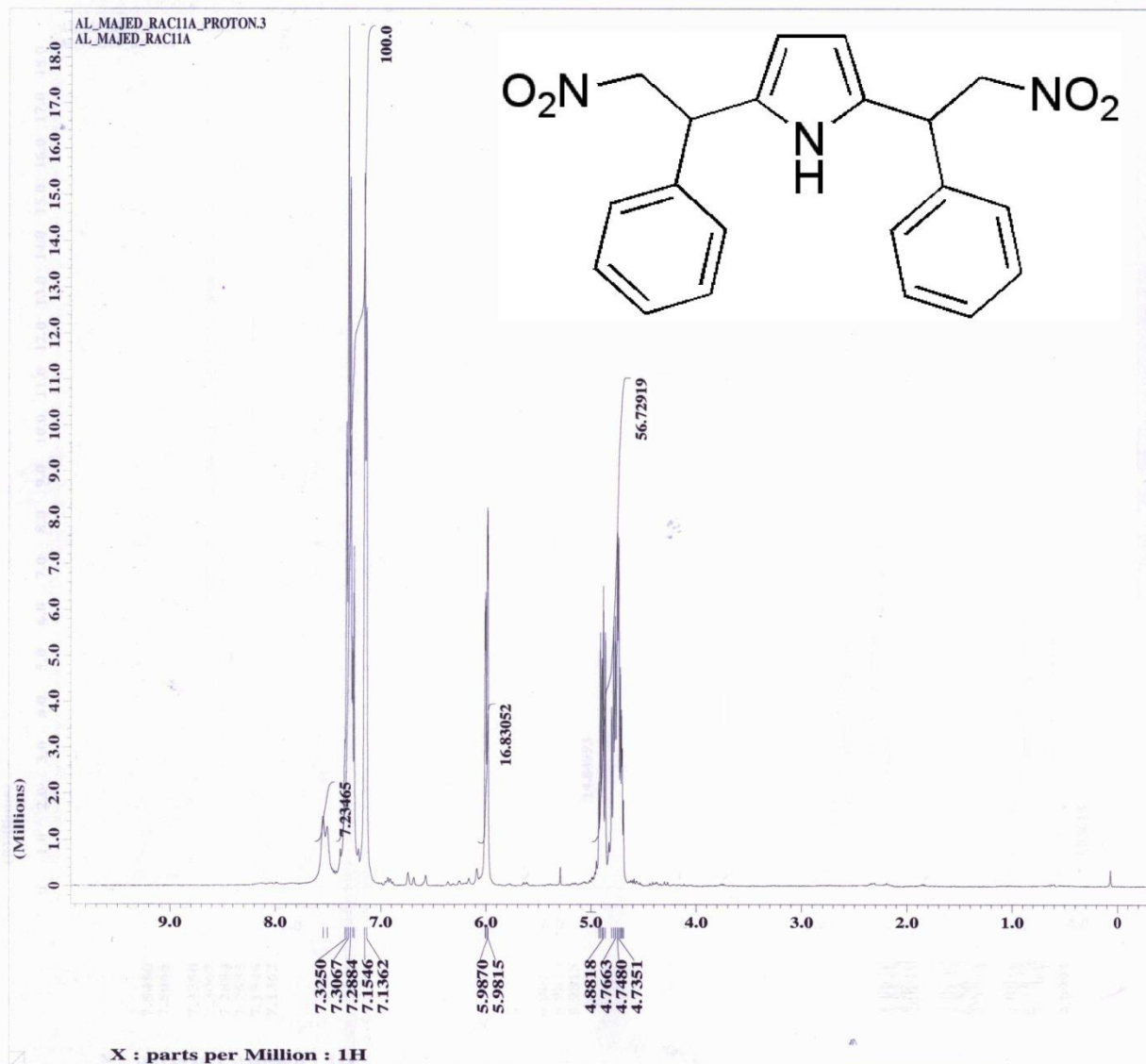
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Compound 9a: ¹H-NMR



JEOL

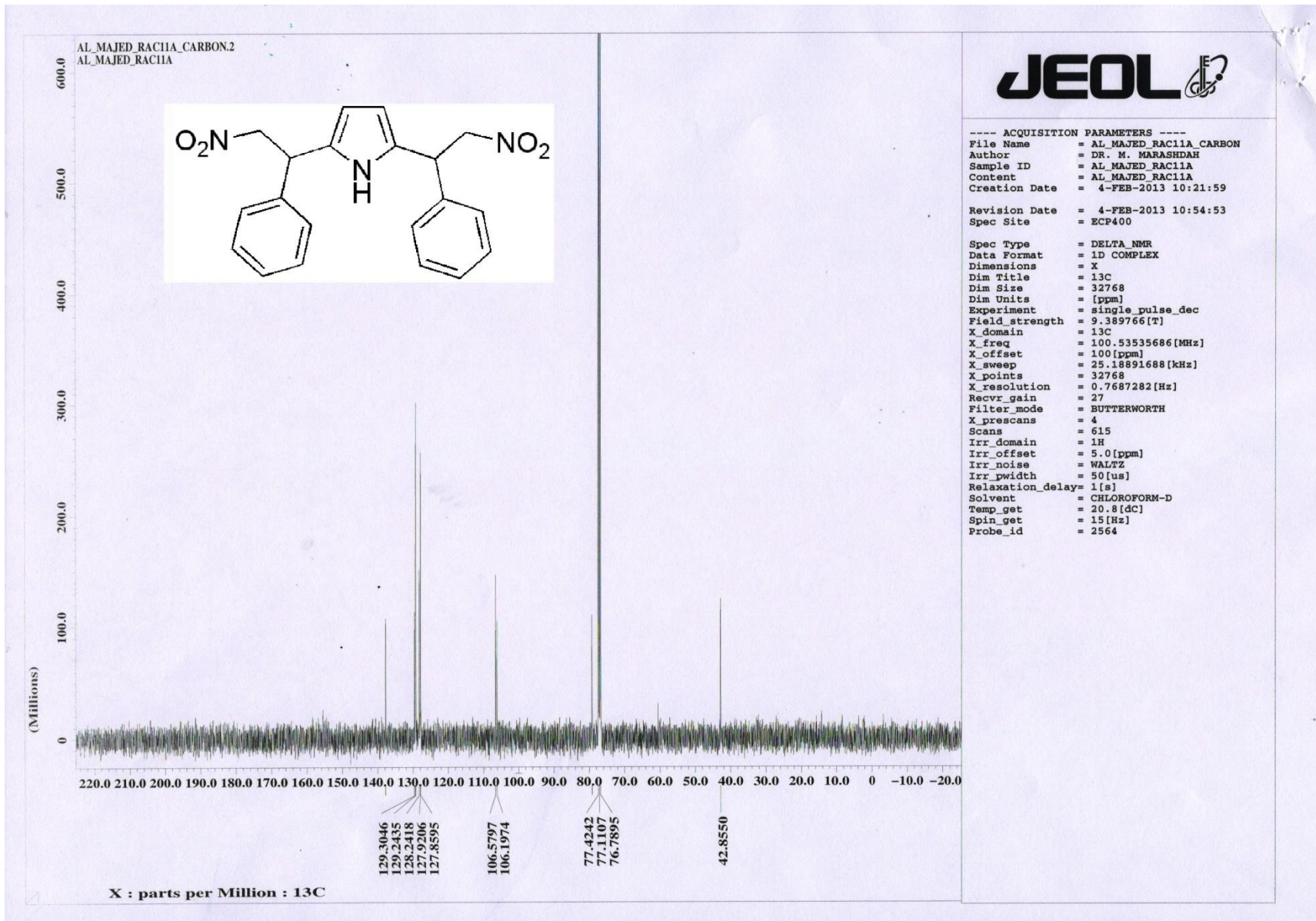
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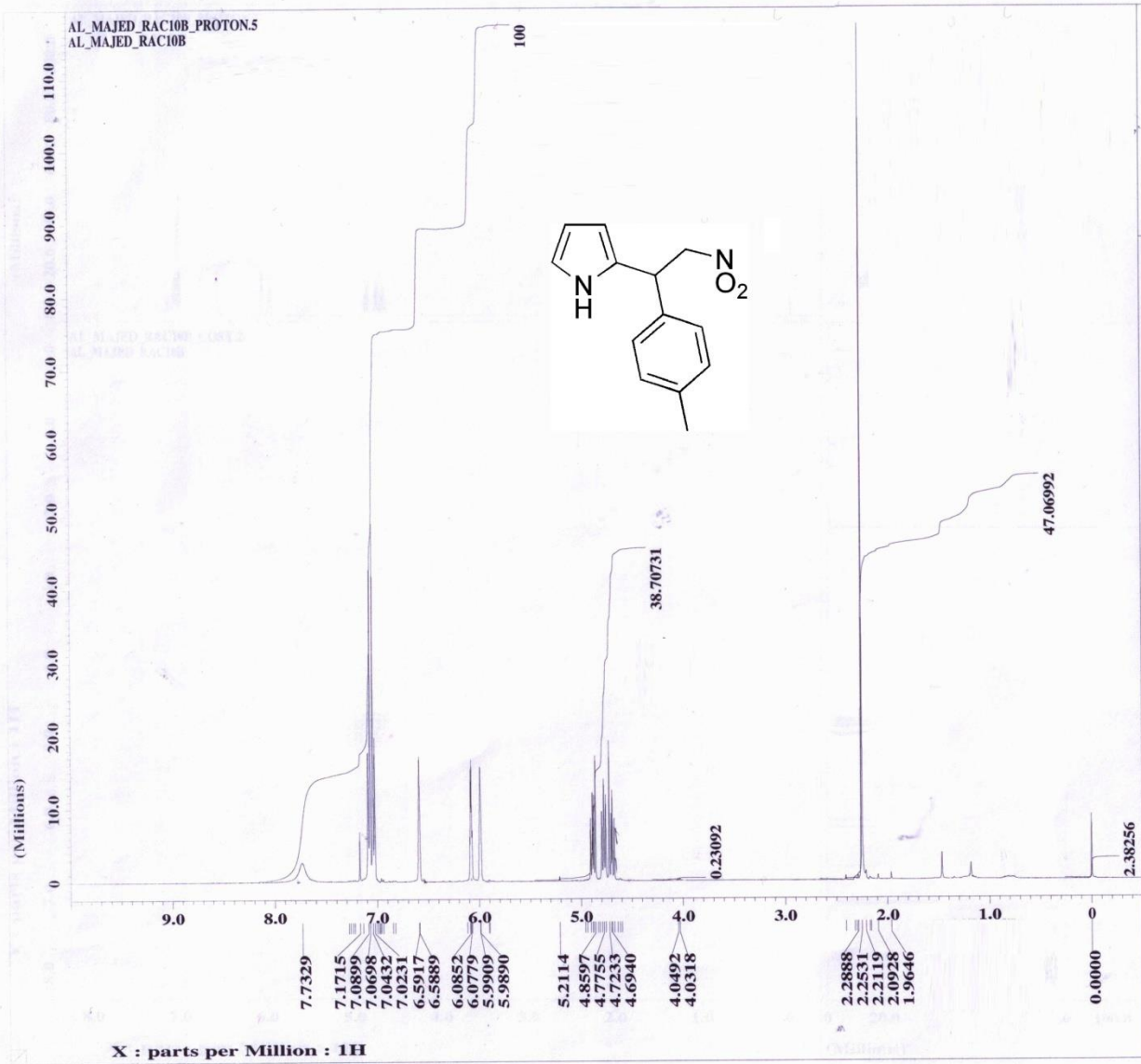
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Compound 9a: ¹³C-NMR



Compound 8b: ¹H-NMR

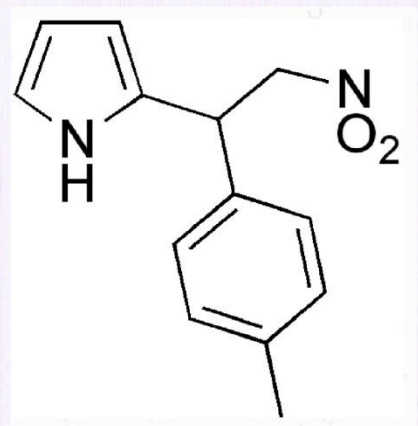
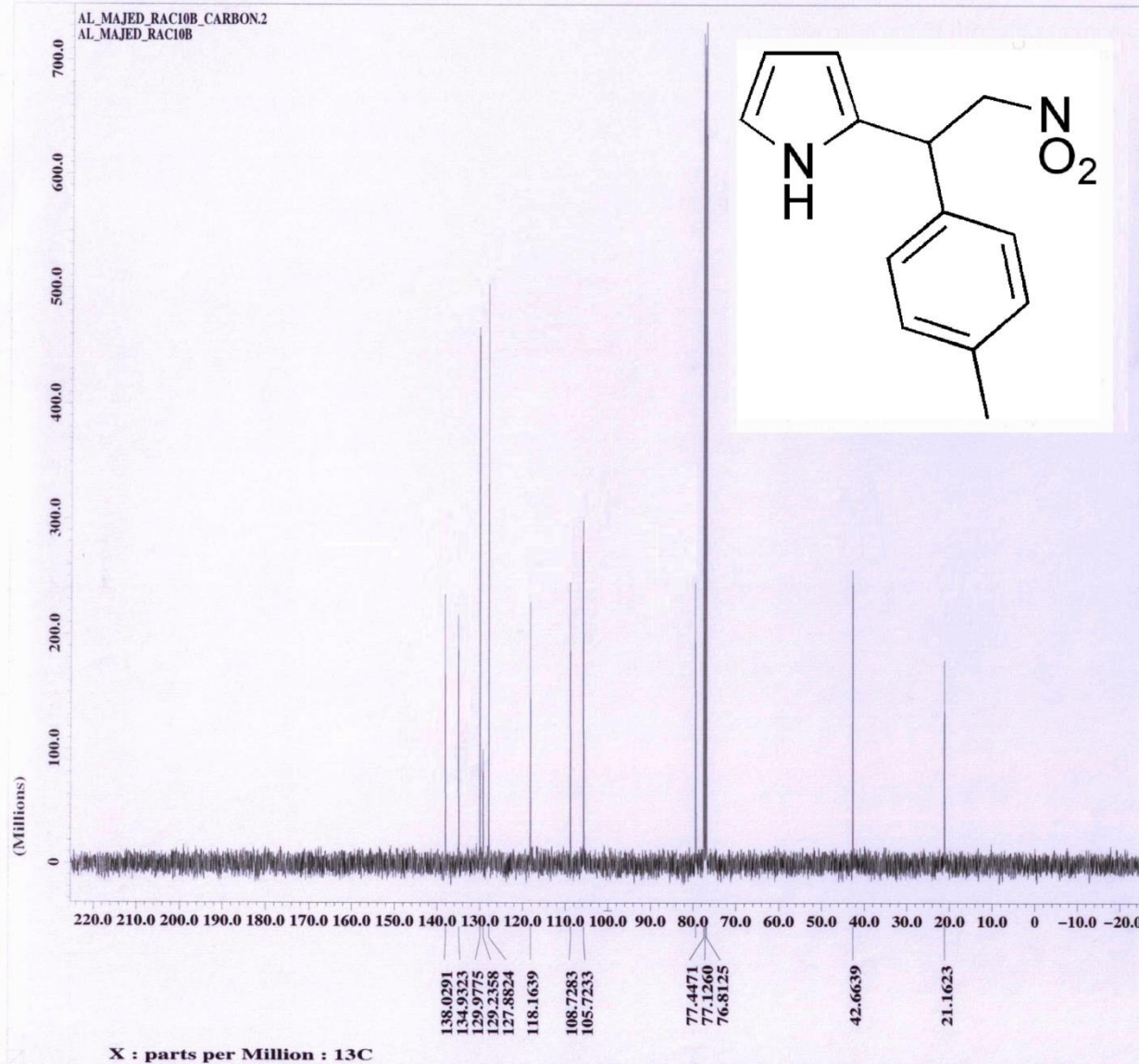


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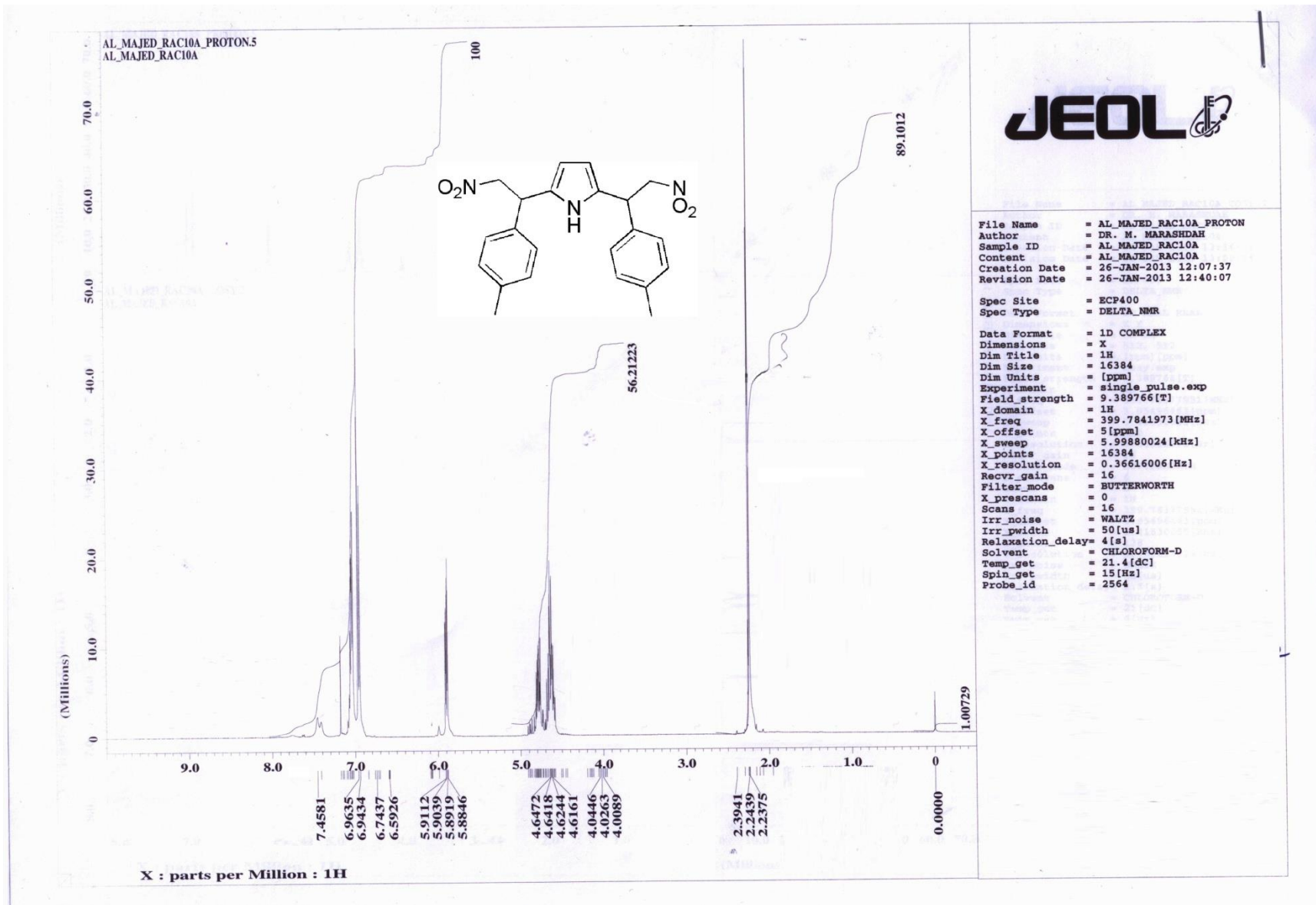
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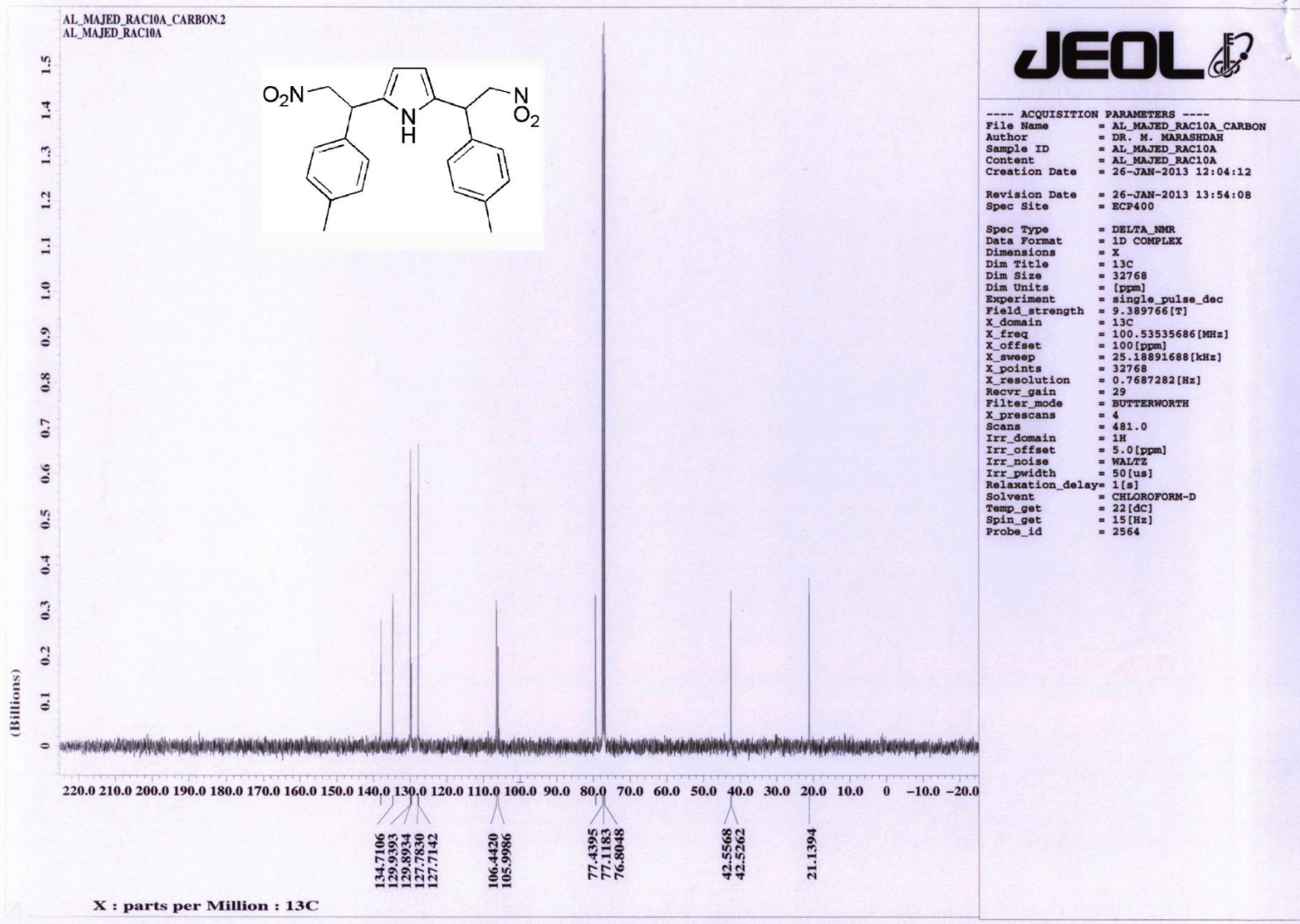
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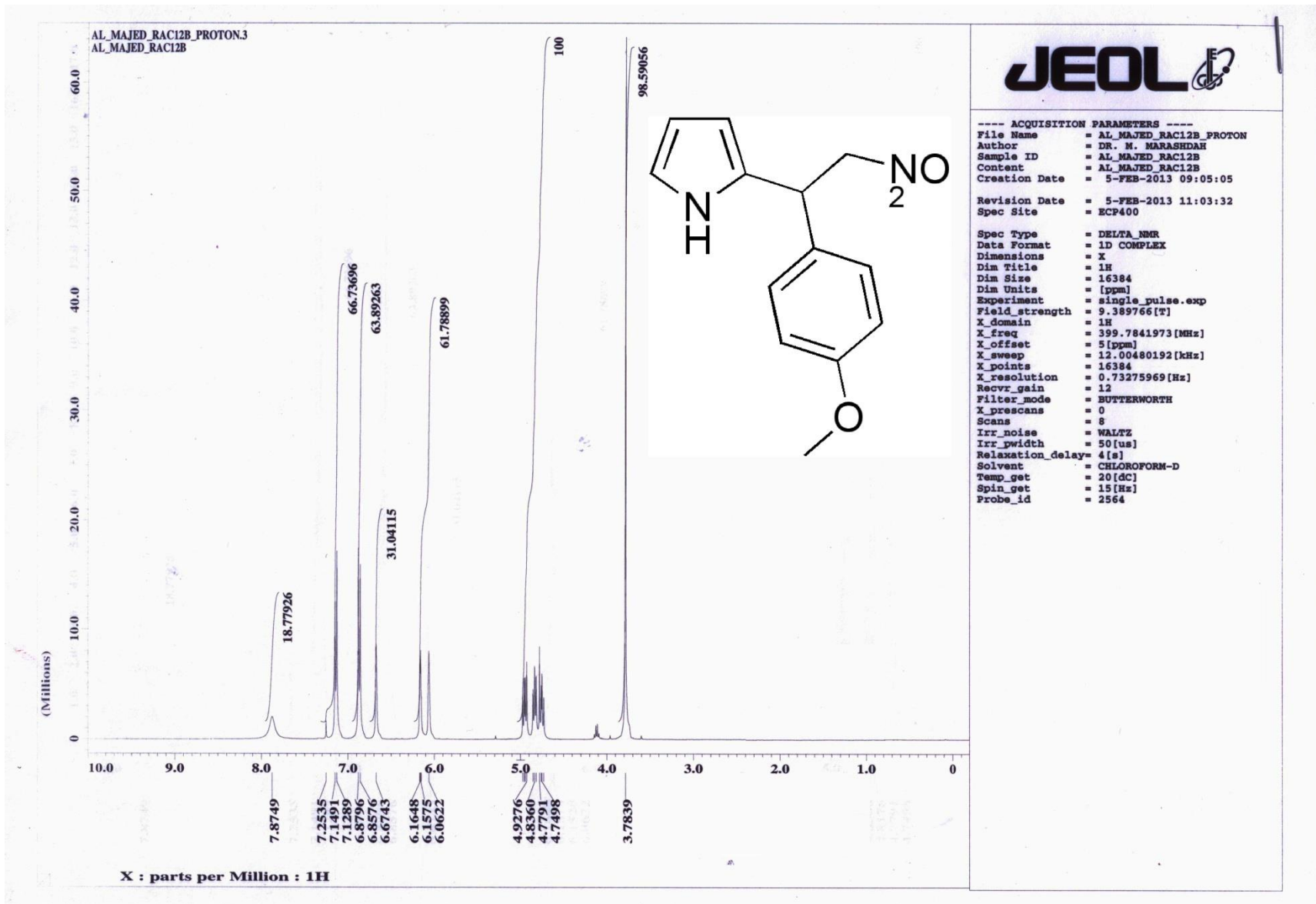
Compound 9b: ¹H-NMR



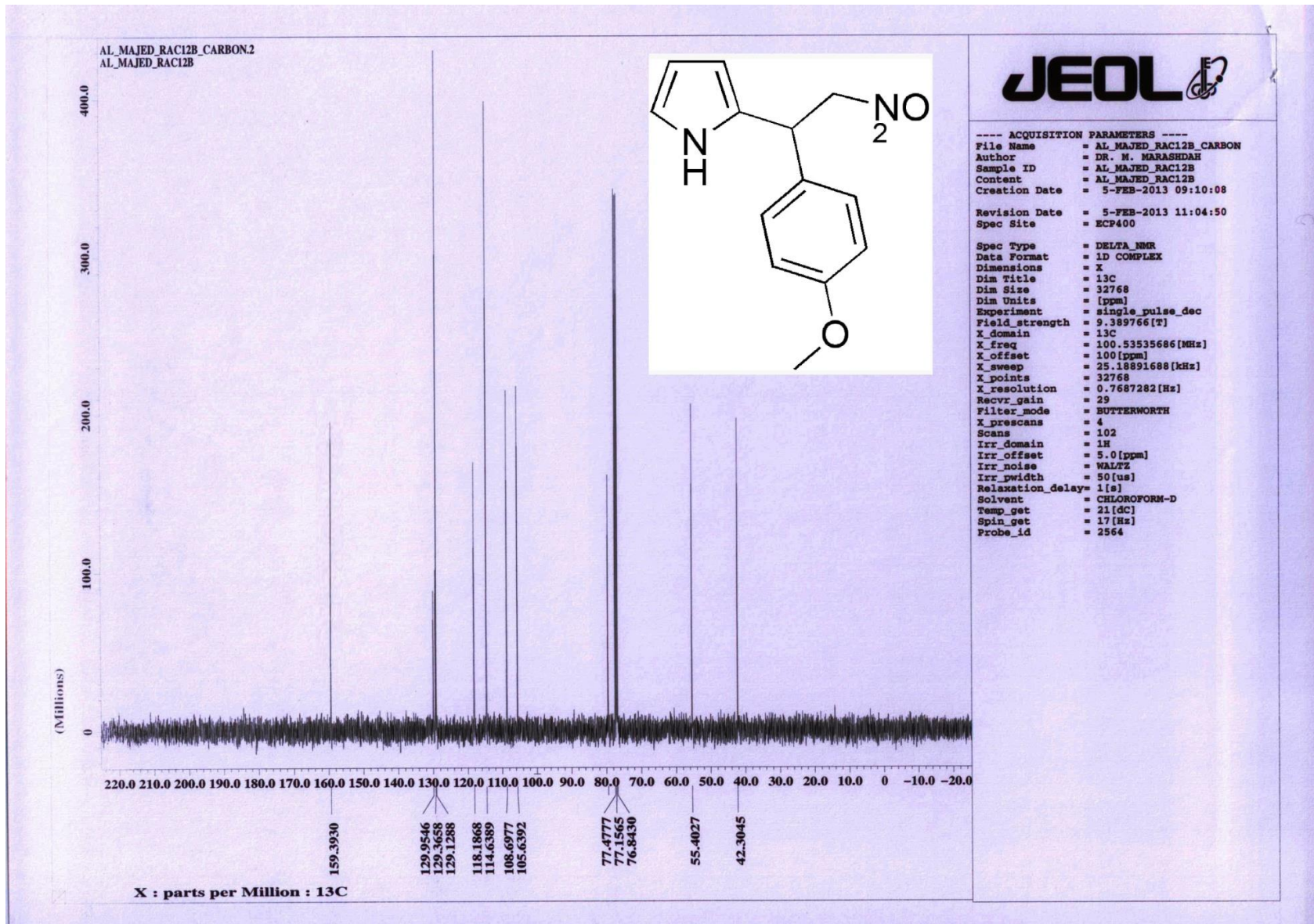
Compound 9b: ¹³C-NMR



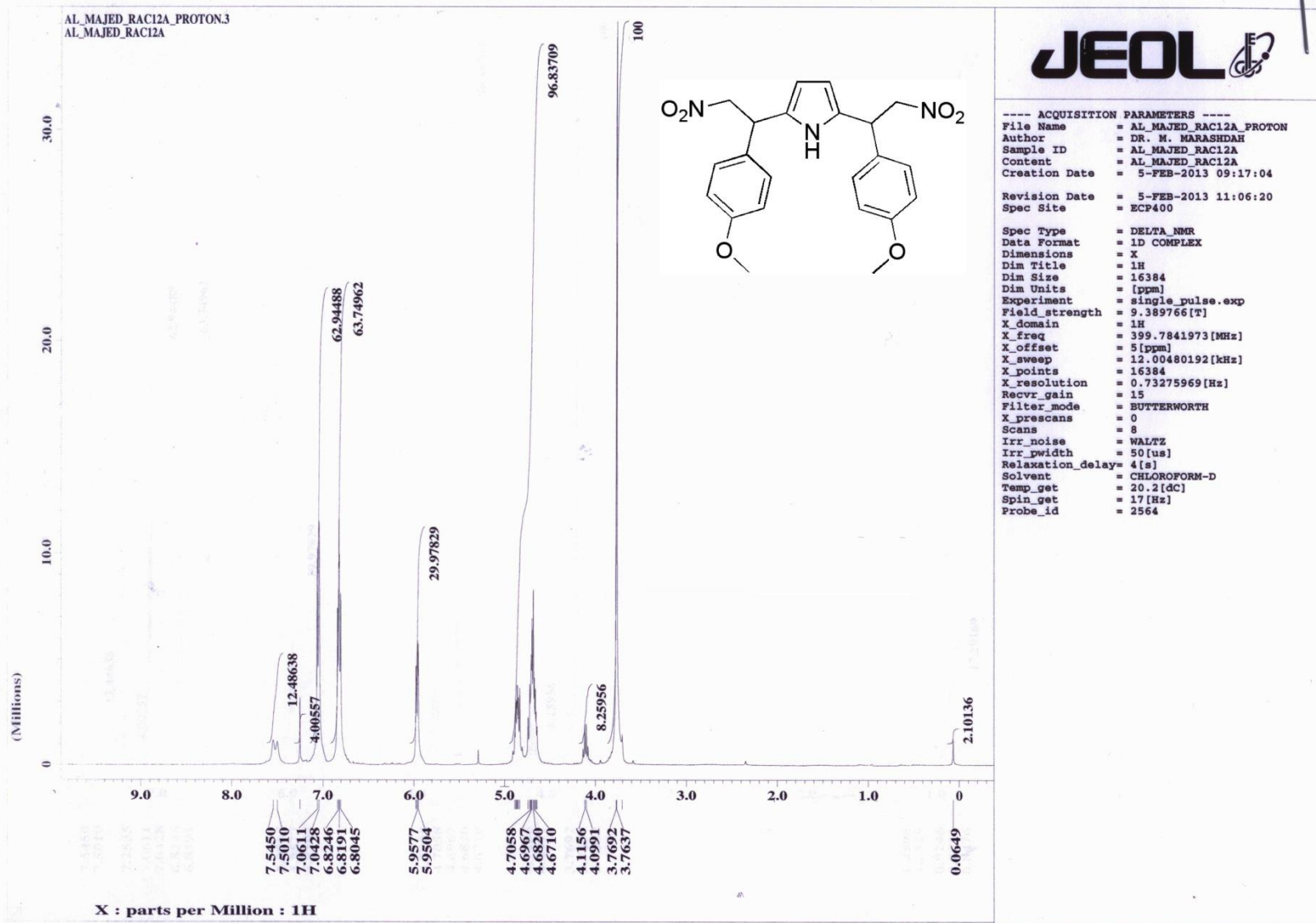
Compound 8c: ¹H-NMR



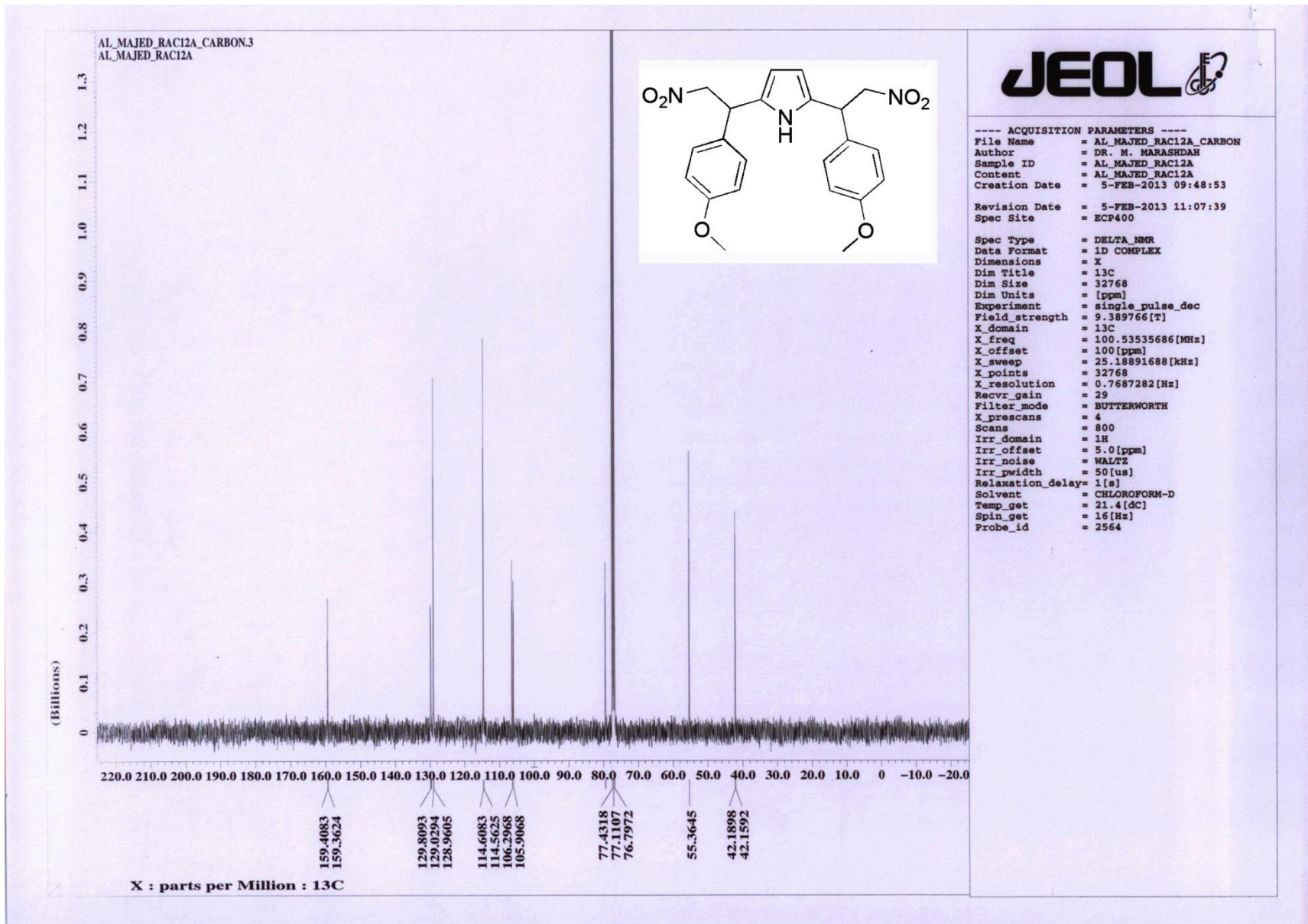
Compound 8c: ¹³C-NMR



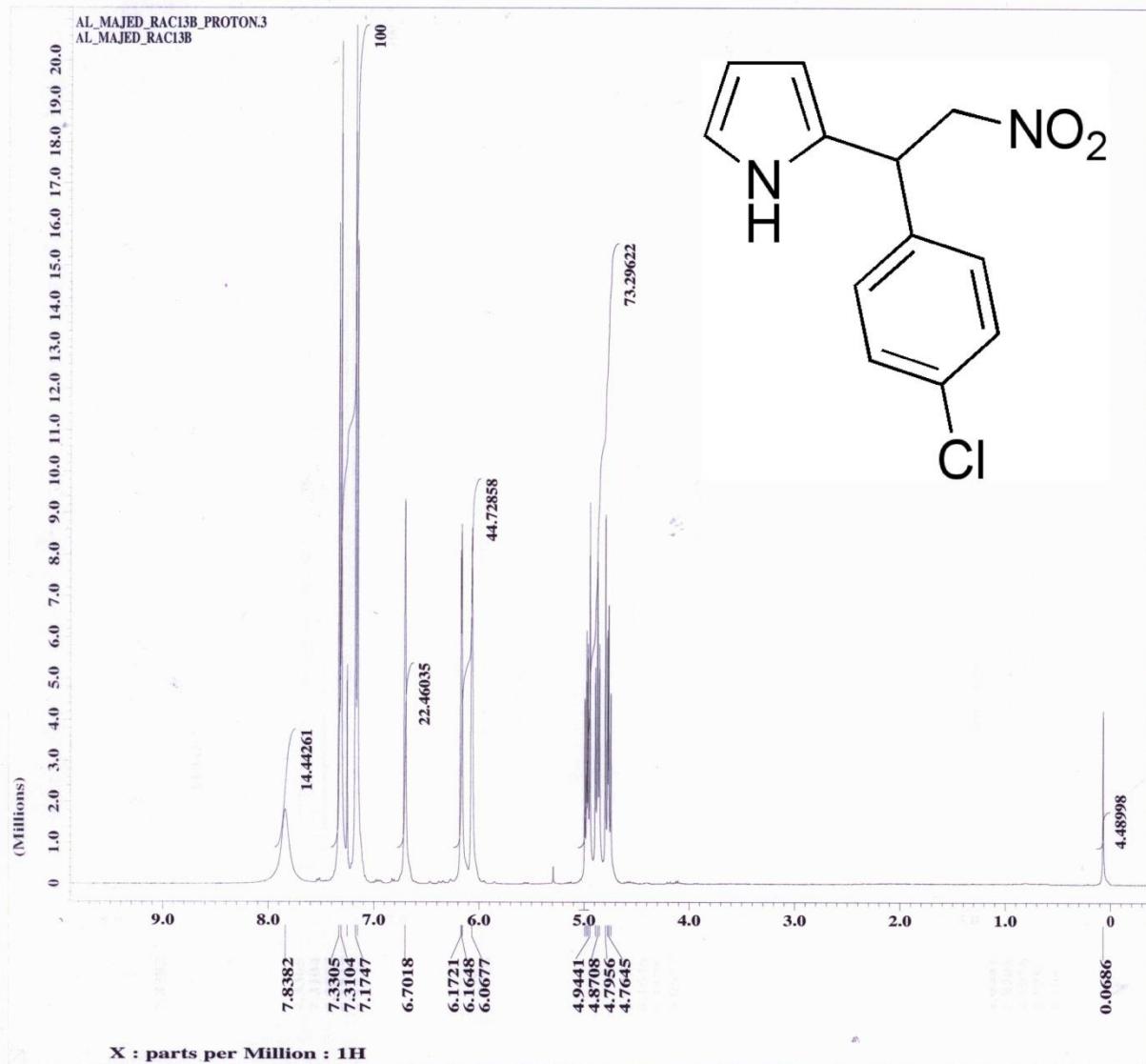
Compound 9c: ¹H-NMR



Compound 9c: ¹³C-NMR



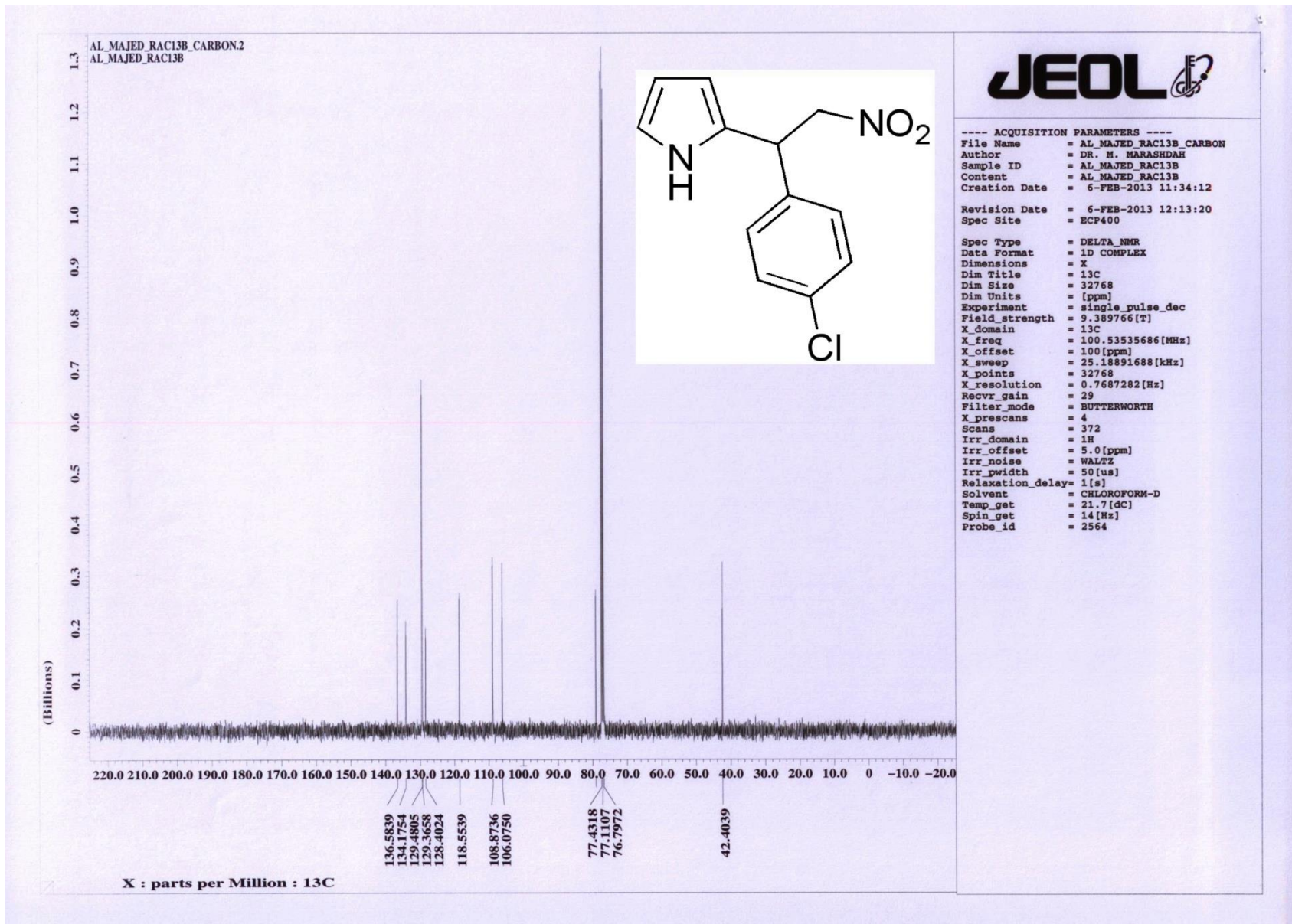
Compound 8d: ¹H-NMR



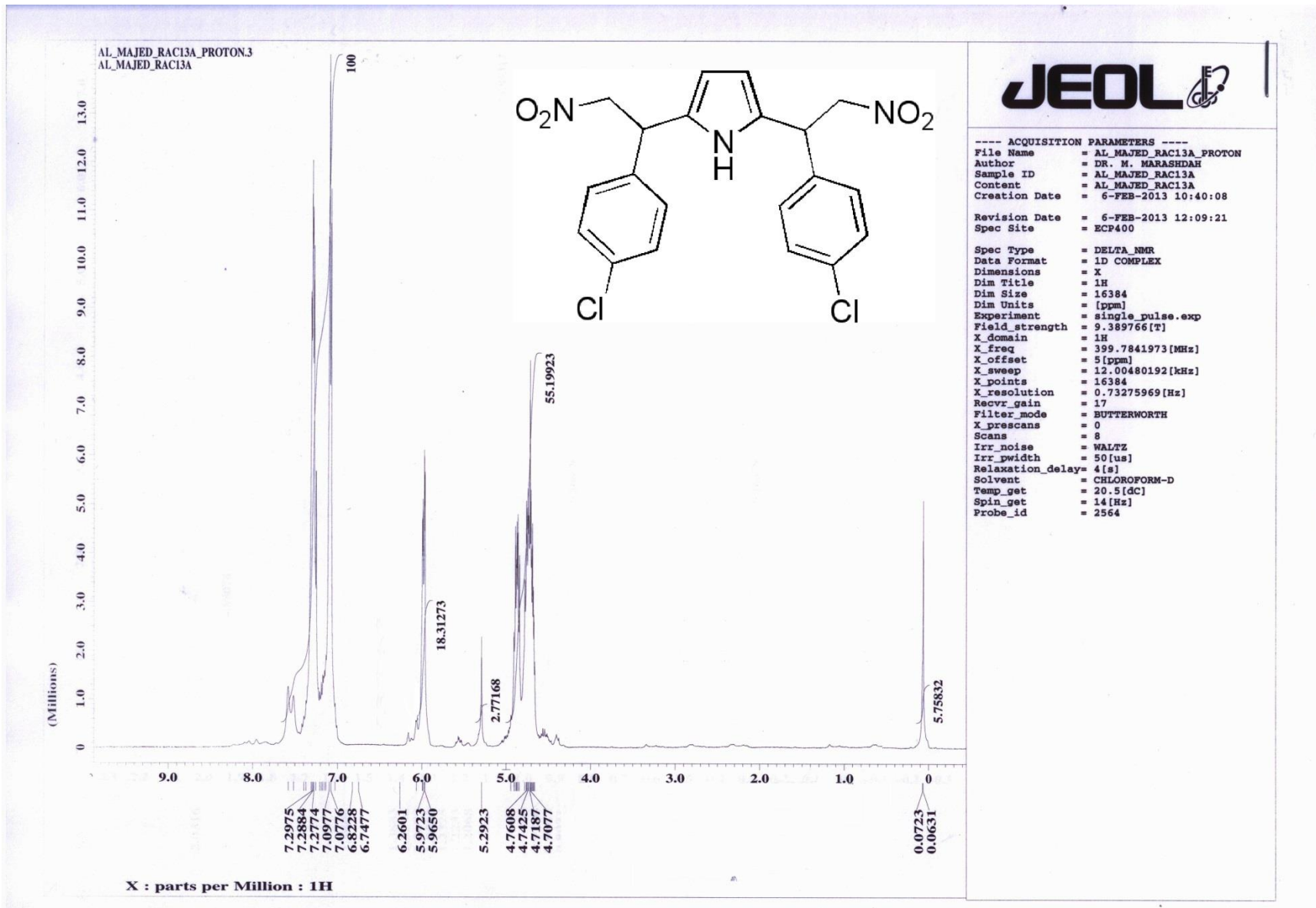
JEOL

---- ACQUISITION PARAMETERS ----
 File Name = AL_MAJED_RAC13B_PROTON
 Author = DR. M. MARASHDAH
 Sample ID = AL_MAJED_RAC13B
 Content = AL_MAJED_RAC13B
 Creation Date = 6-FEB-2013 11:18:49
 Revision Date = 6-FEB-2013 12:12:33
 Spec Site = ECF400
 Spec Type = DELTA_NMR
 Data Format = 1D_COMPLEX
 Dimensions = X
 Dim Title = 1H
 Dim Size = 16384
 Dim Units = [ppm]
 Experiment = single_pulse.exp
 Field_strength = 9.389766[T]
 X_domain = 1H
 X_freq = 399.7841973[MHz]
 X_offset = 5[ppm]
 X_sweep = 12.00480192[kHz]
 X_points = 16384
 X_resolution = 0.73275969[Hz]
 Recvr_gain = 15
 Filter_mode = BUTTERWORTH
 X_prescans = 0
 Scans = 8
 Irr_noise = WALTZ
 Irr_width = 50[us]
 Relaxation_delay = 4[s]
 Solvent = CHLOROFORM-D
 Temp_get = 20.5[DC]
 Spin_get = 14[Hz]
 Probe_id = 2564

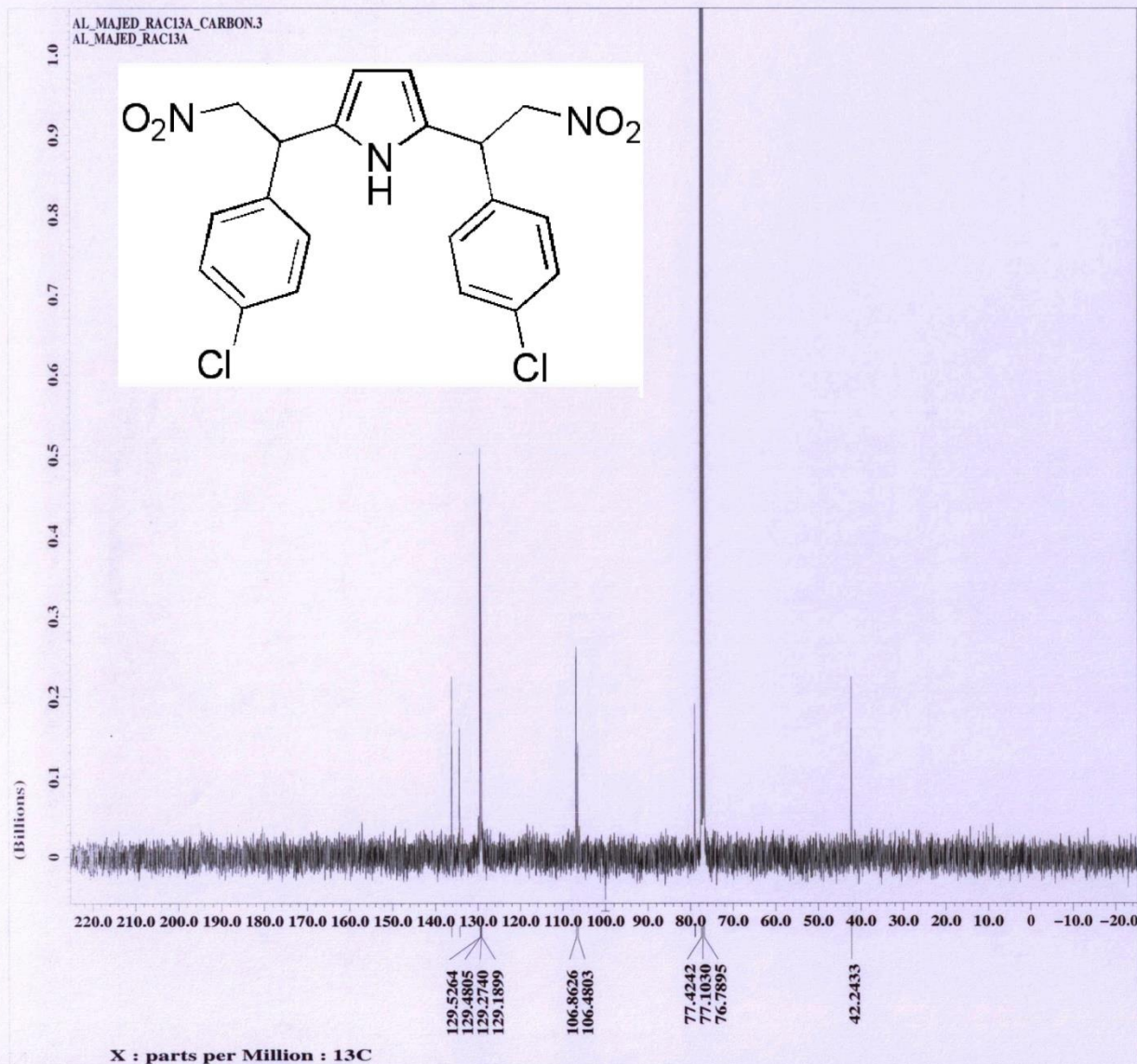
Compound 8d: ¹³C-NMR



Compound 9d: ¹H-NMR



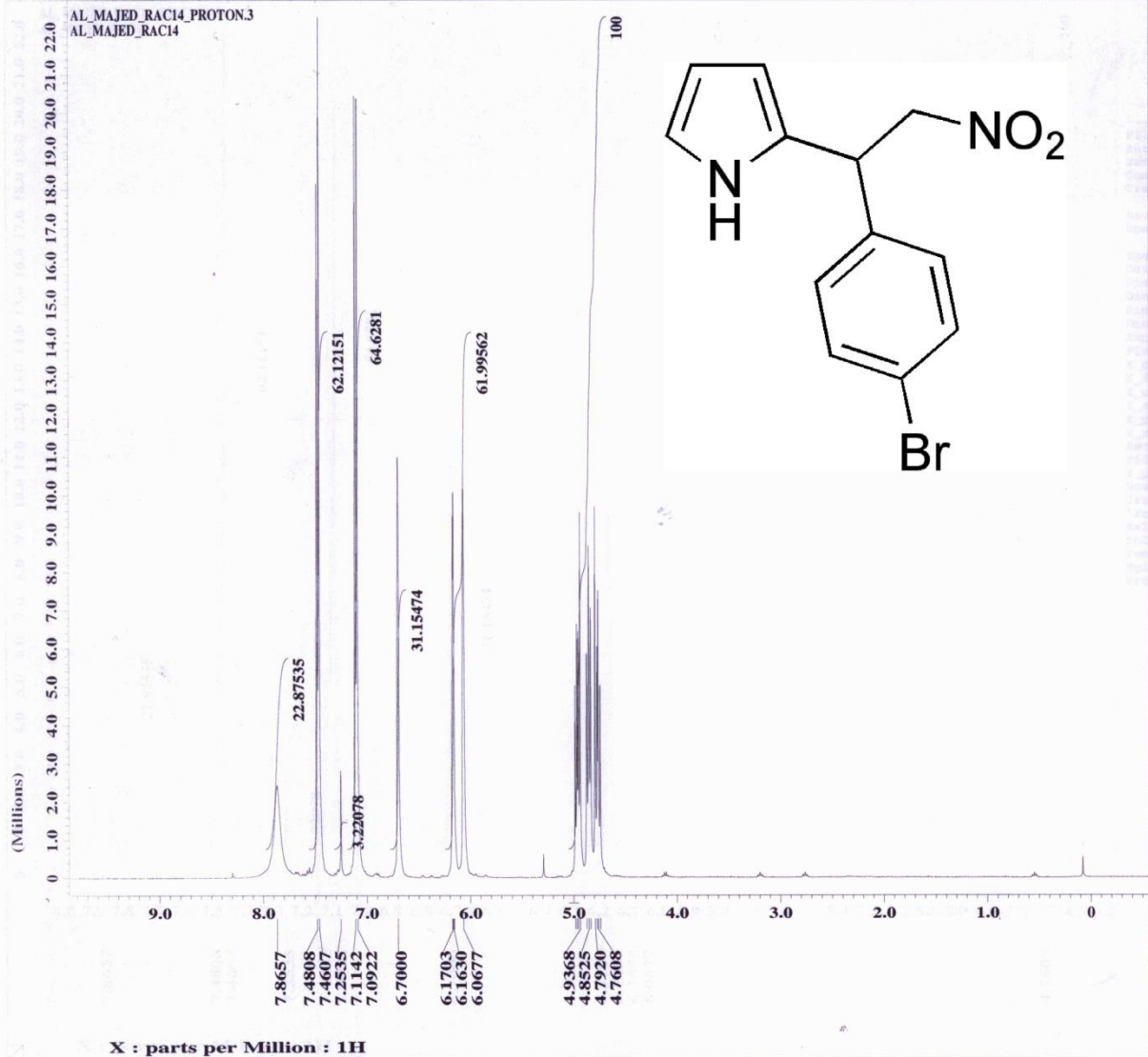
Compound 9d: ¹³C-NMR



JEOL

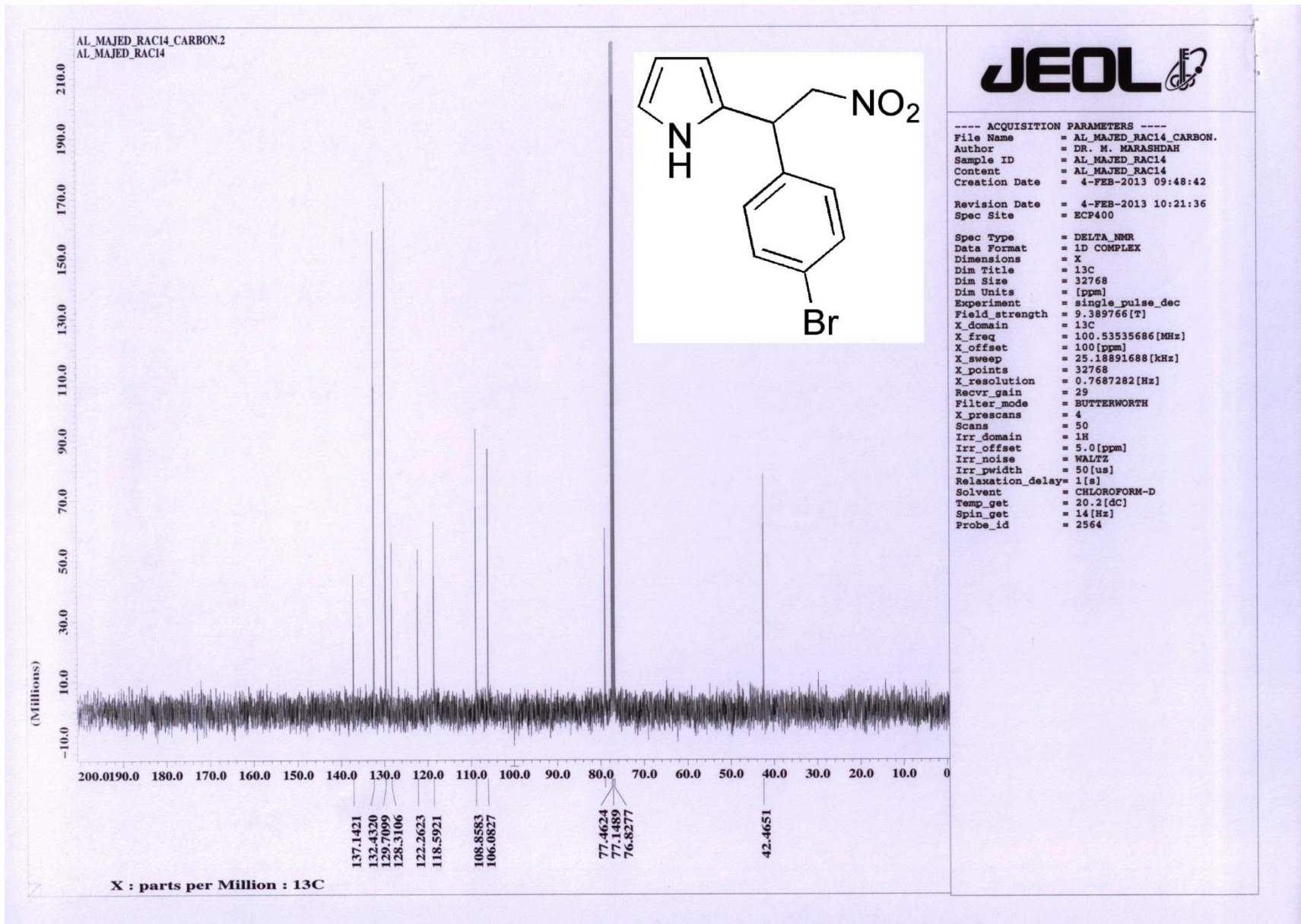
---- ACQUISITION PARAMETERS ----
 File Name = AL_MAJED_RAC13A_CARBON
 Author = DR. M. MARASHDAH
 Sample ID = AL_MAJED_RAC13A
 Content = AL_MAJED_RAC13A
 Creation Date = 6-FEB-2013 11:11:57
 Revision Date = 6-FEB-2013 12:11:12
 Spec Site = ECP400
 Spec Type = DELTA_NMR
 Data Format = 1D_COMPLEX
 Dimensions = X
 Dim Title = 13C
 Dim Size = 32768
 Dim Units = [ppm]
 Experiment = single_pulse_dec
 Field_strength = 9.389766[T]
 X_domain = 13C
 X_freq = 100.53535686[MHz]
 X_offset = 100[ppm]
 X_sweep = 25.18891688[kHz]
 X_points = 32768
 X_resolution = 0.7687282[Hz]
 Recvr_gain = 29
 Filter_mode = BUTTERWORTH
 X_prescans = 4
 Scans = 800
 Irr_domain = 1H
 Irr_offset = 5.0[ppm]
 Irr_noise = WALTZ
 Irr_pwidth = 50[us]
 Relaxation_delay = 1[s]
 Solvent = CHLOROFORM-D
 Temp_get = 21.5[dC]
 Spin_get = 14[Hz]
 Probe_id = 2564

Compound 8e: ¹H-NMR

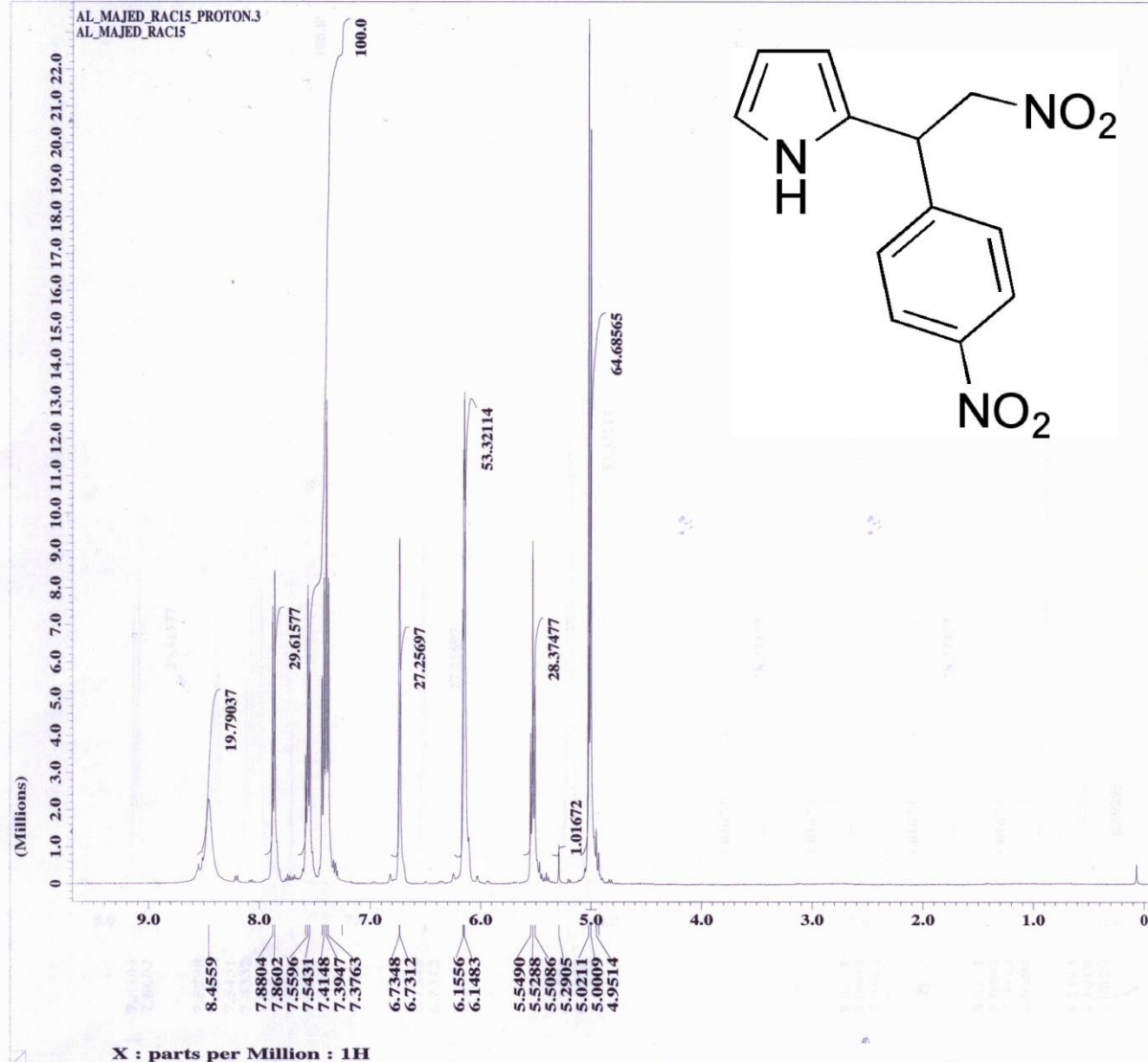


---- ACQUISITION PARAMETERS ----
 File Name = AL_MAJED_RAC14_PROTON.
 Author = DR. M. MARASHDAH
 Sample ID = AL_MAJED_RAC14
 Content = AL_MAJED_RAC14
 Creation Date = 4-FEB-2013 09:45:33
 Revision Date = 4-FEB-2013 10:18:57
 Spec Site = ECP400
 Spec Type = DELTA_NMR
 Data Format = 1D_COMPLEX
 Dimensions = X
 Dim Title = 1H
 Dim Size = 16384
 Dim Units = [ppm]
 Experiment = single_pulse.exp
 Field_strength = 9.389766[T]
 X_domain = 1H
 X_freq = 399.7841973[MHz]
 X_offset = 5[ppm]
 X_sweep = 12.00480192[kHz]
 X_points = 16384
 X_resolution = 0.73275969[Hz]
 Recvr_gain = 14
 Filter_mode = BUTTERWORTH
 X_prescans = 0
 Scans = 8
 Irr_noise = WALTZ
 Irr_pwidth = 50[us]
 Relaxation_delay = 4[s]
 Solvent = CHLOROFORM-D
 Temp_get = 19.6[dc]
 Spin_get = 14[Hz]
 Probe_id = 2564

Compound 8e: ¹³C-NMR



Compound 8f: ¹H-NMR



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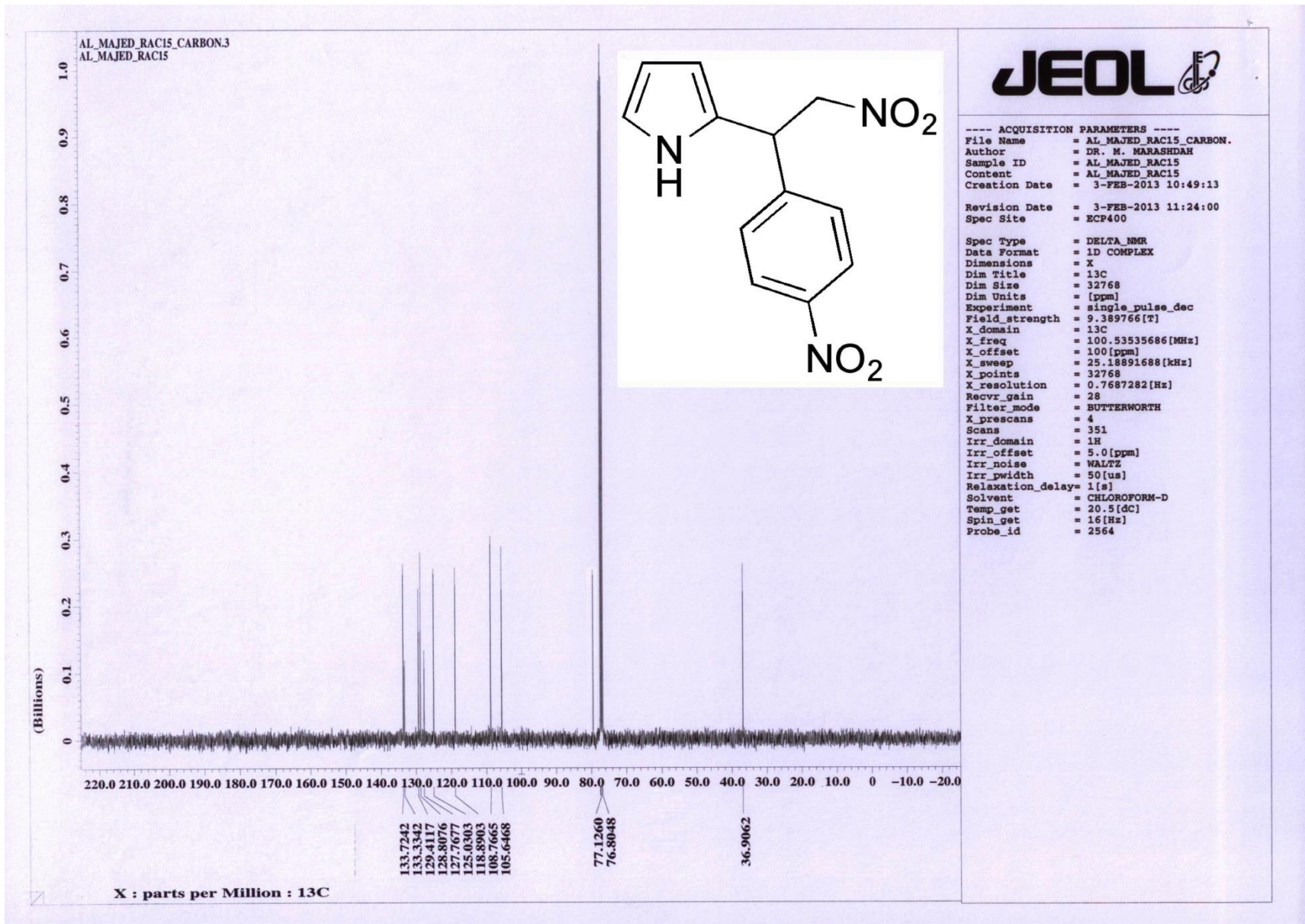
---- ACQUISITION PARAMETERS ----

File Name = AL_MAJED_RAC15_PROTON.
 Author = DR. M. MARASHDAH
 Sample ID = AL_MAJED_RAC15
 Content = AL_MAJED_RAC15
 Creation Date = 3-FEB-2013 10:34:30

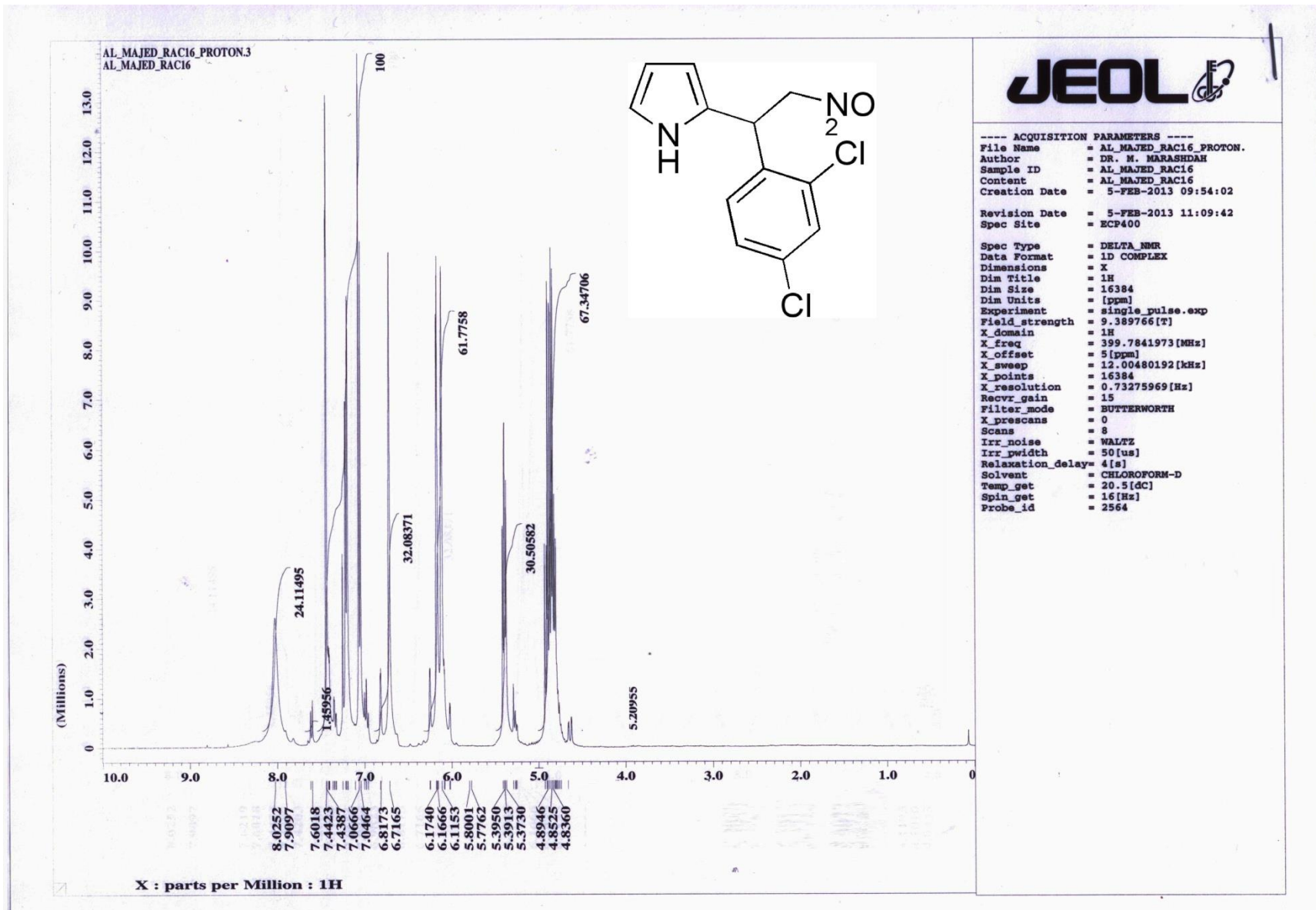
Revision Date = 3-FEB-2013 11:22:24
 Spec Site = ECP400

Spec Type = DELTA_NMR
 Data Format = 1D_COMPLEX
 Dimensions = X
 Dim Title = 1H
 Dim Size = 16384
 Dim Units = [ppm]
 Experiment = single_pulse.exp
 Field_strength = 9.389766[T]
 X_domain = 1H
 X_freq = 399.7841973[MHz]
 X_offset = 5[ppm]
 X_sweep = 12.00480192[kHz]
 X_points = 16384
 X_resolution = 0.73275969[Hz]
 Recvr_gain = 15
 Filter_mode = BUTTERWORTH
 X_prescans = 0
 Scans = 8
 Irr_noise = WALTZ
 Irr_pwidth = 50[us]
 Relaxation_delay = 4[s]
 Solvent = CHLOROFORM-D
 Temp_get = 19.5[dc]
 Spin_get = 16[Hz]
 Probe_id = 2564

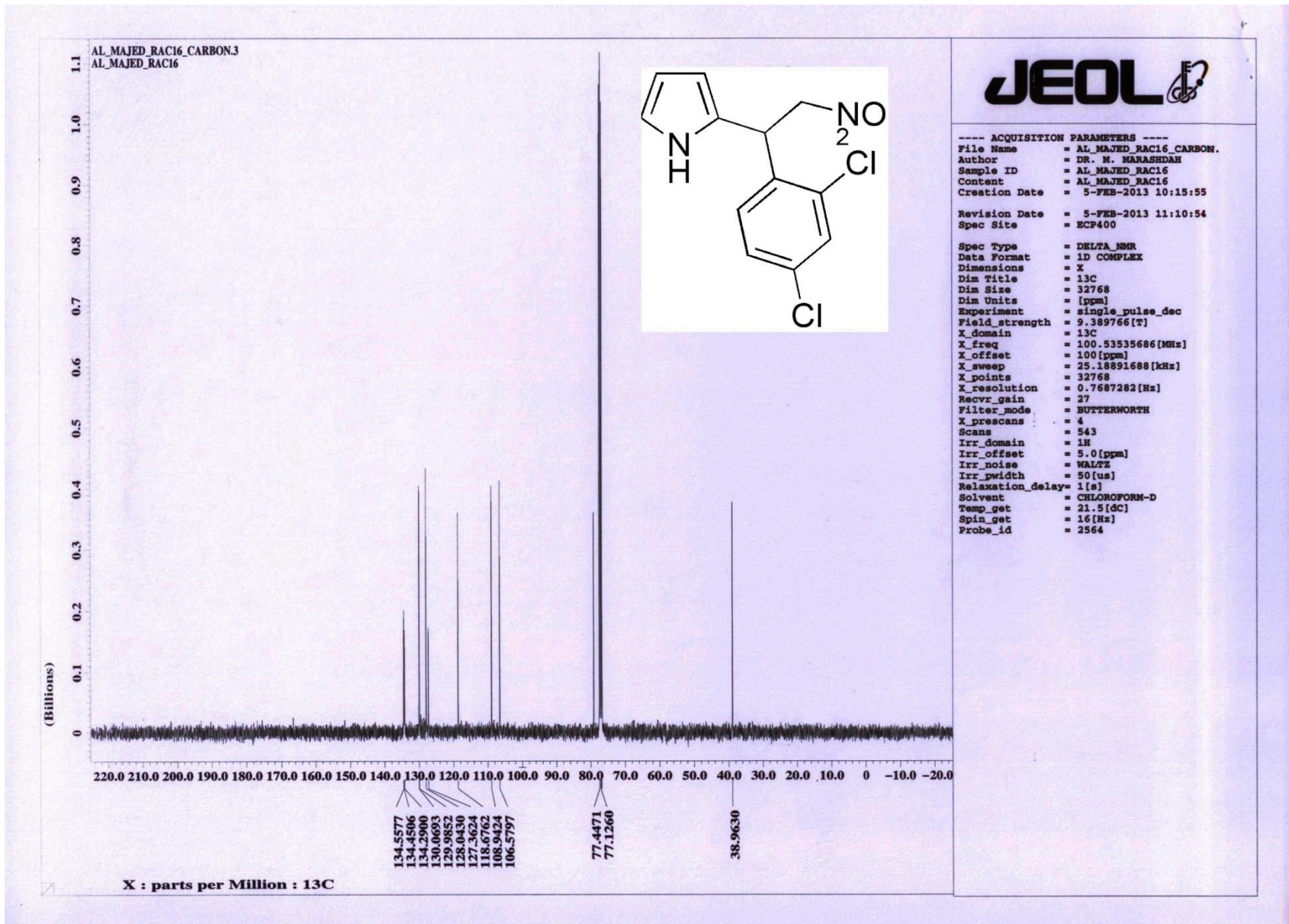
Compound 8f: ¹³C-NMR



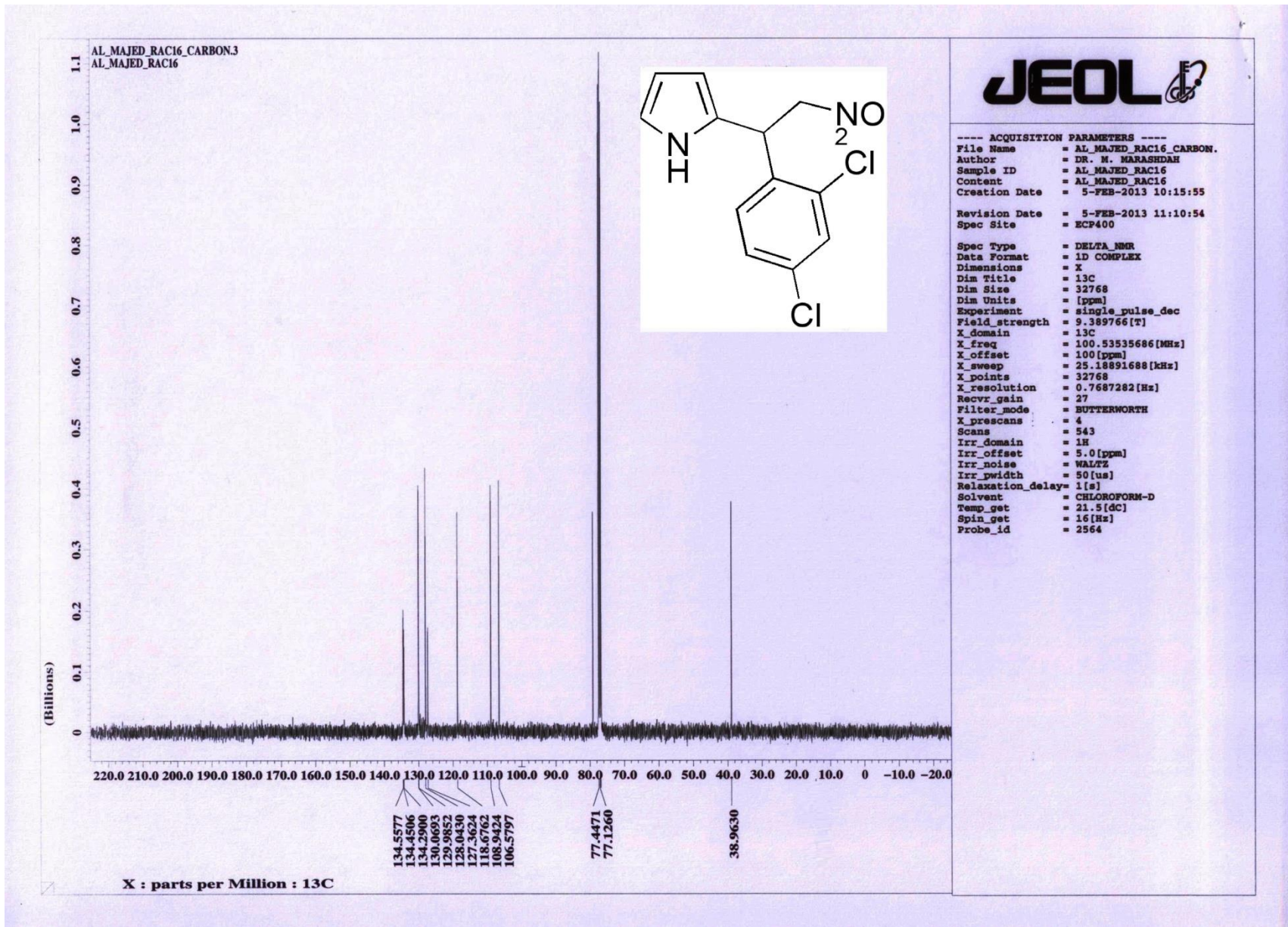
Compound 8g: ¹H-NMR



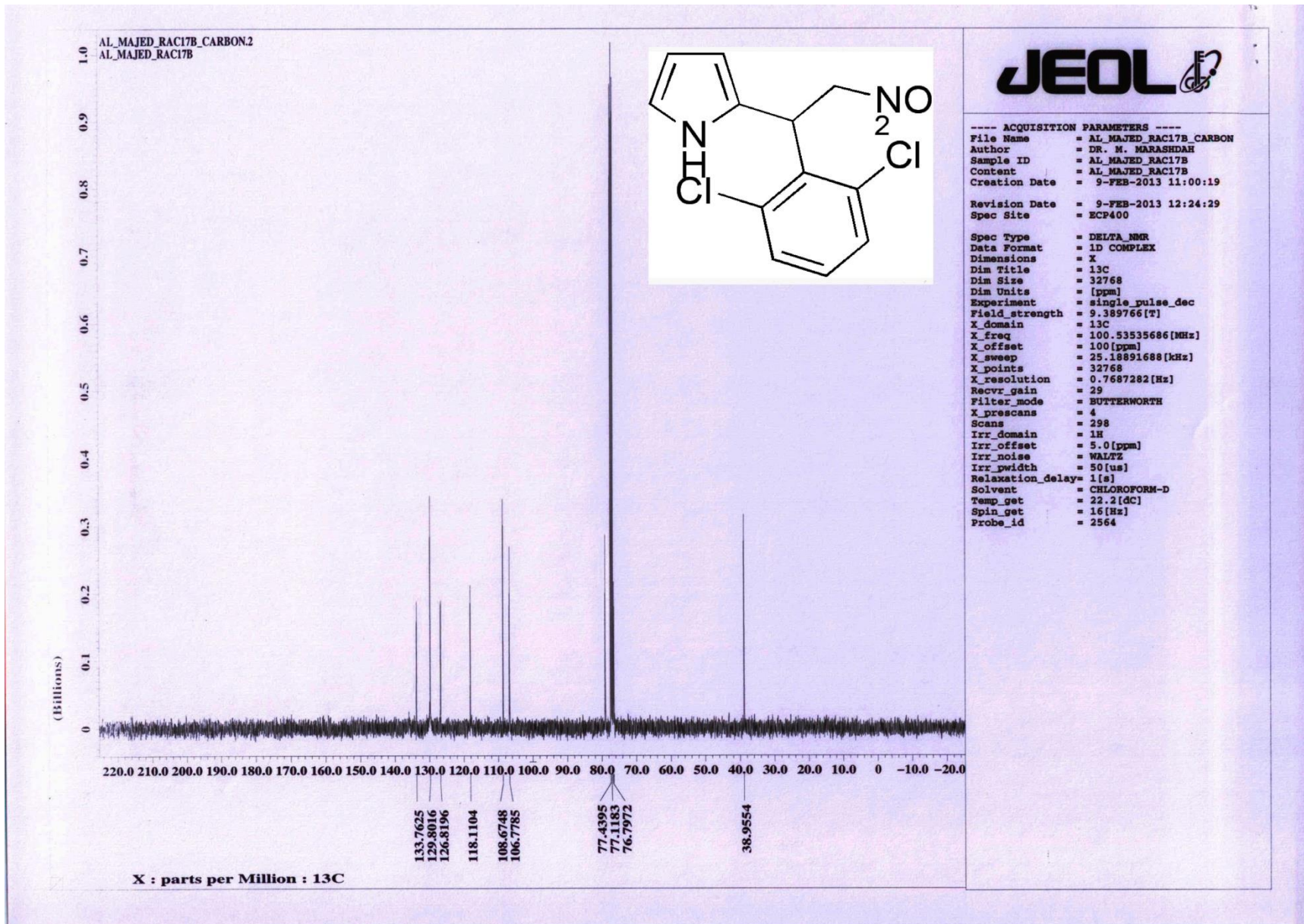
Compound 8g: ¹³C-NMR



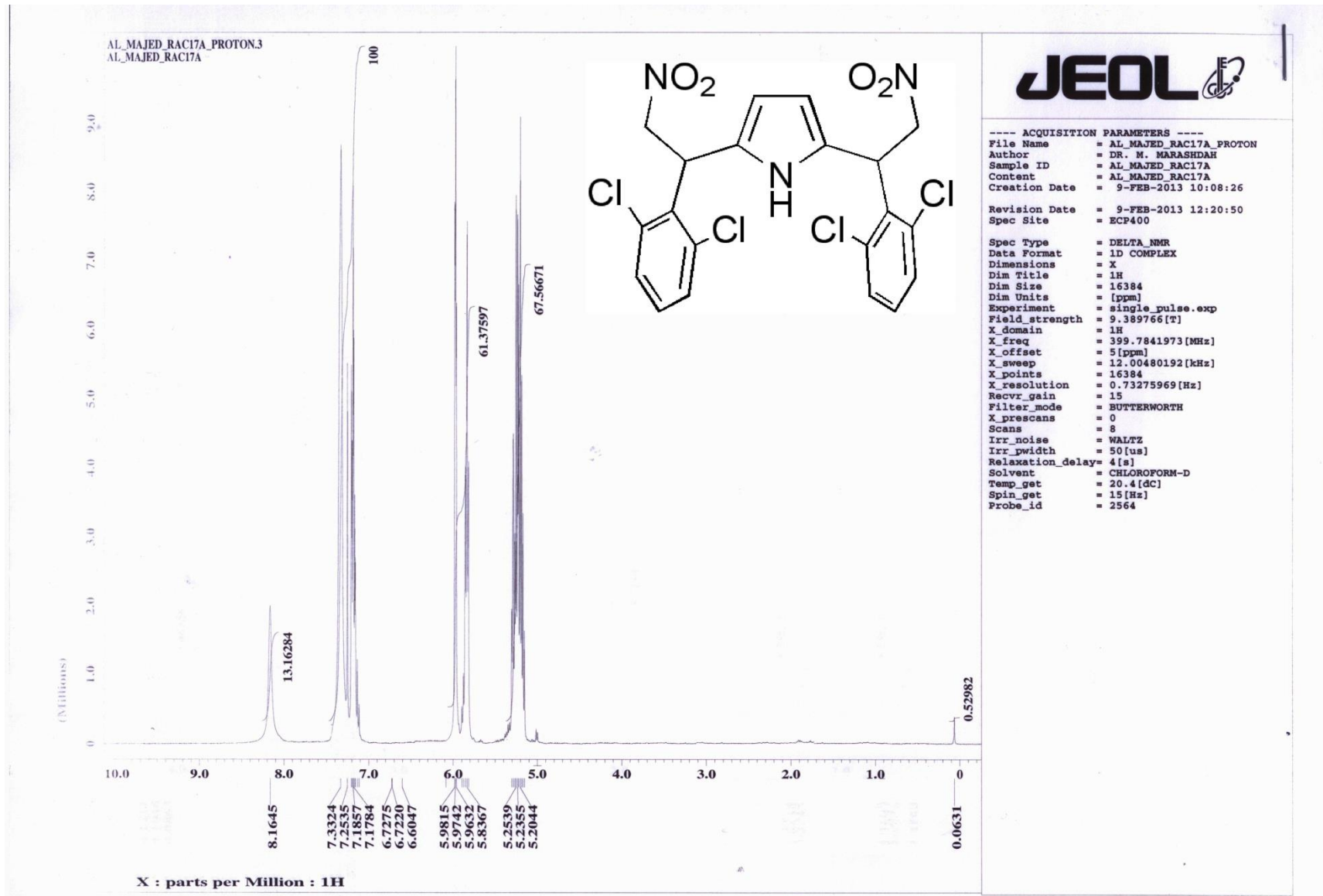
Compound 8h: ¹H-NMR



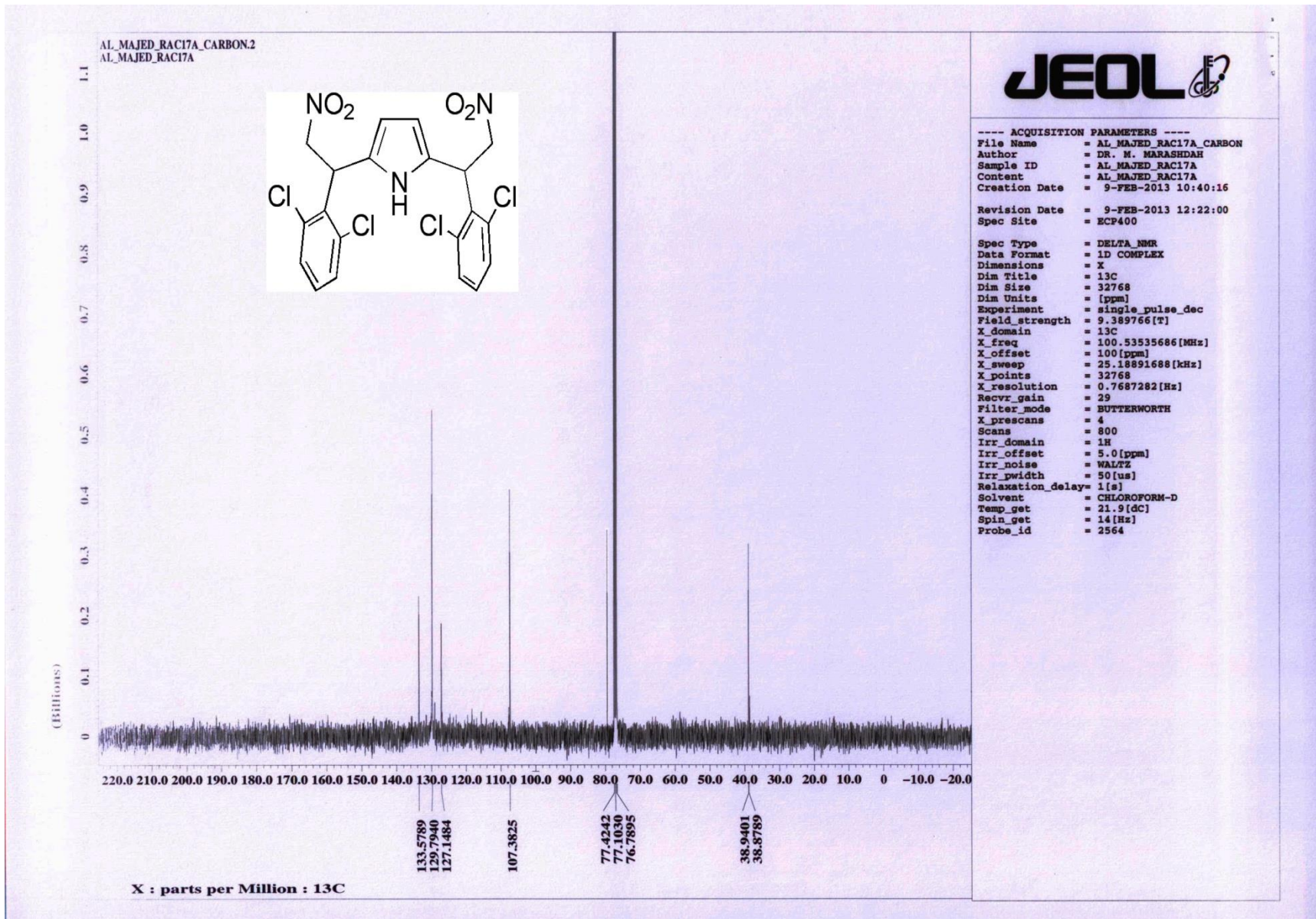
Compound 8h: ¹³C-NMR



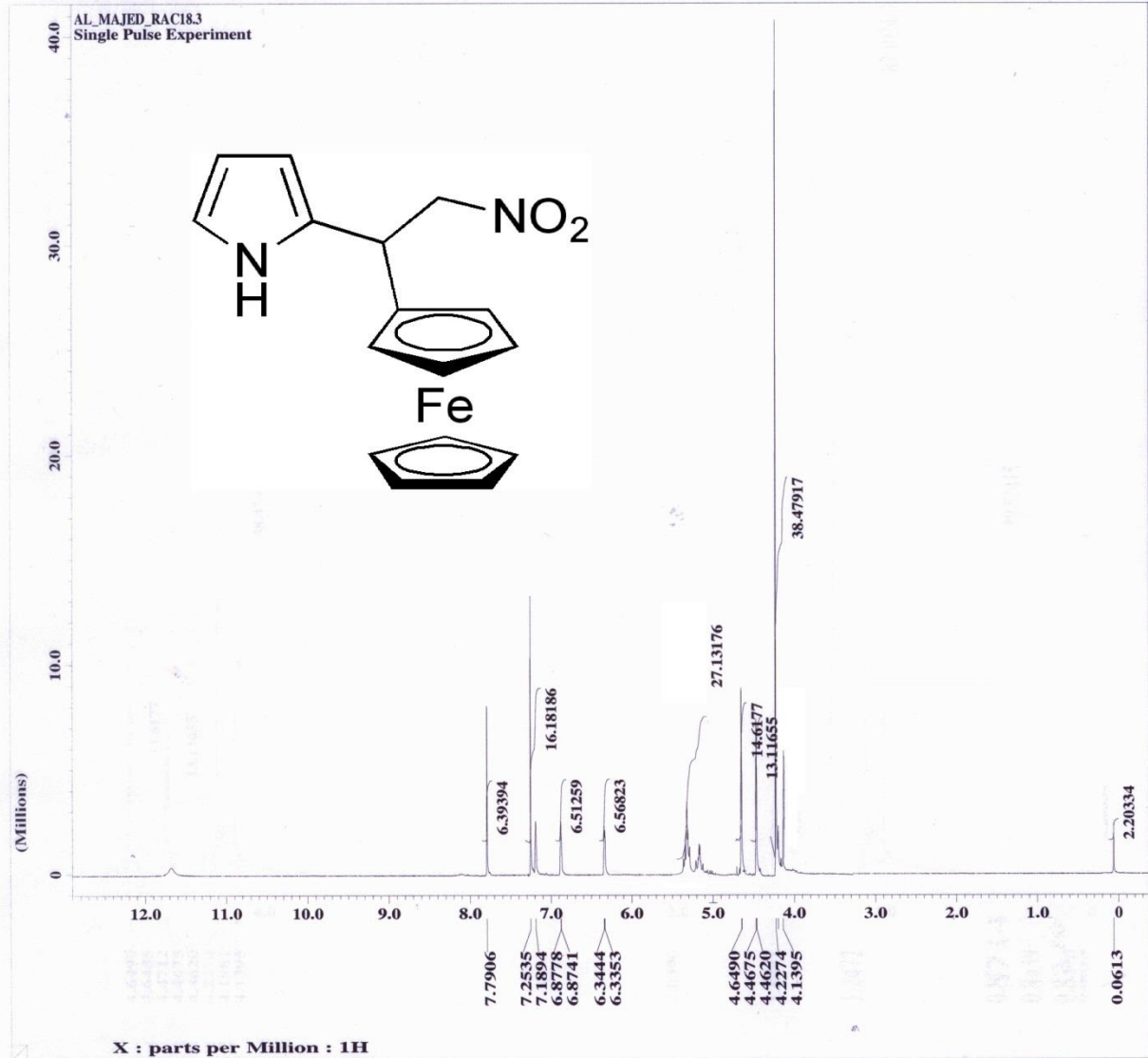
Compound 9h: ¹H-NMR



Compound 9h: ¹³C-NMR



Compound 8i: ¹H-NMR



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---- ACQUISITION PARAMETERS ----

File Name	= AL_MAJED_RAC18.3
Author	= DR. M. MARASHDAH
Sample ID	= AL_MAJED_RAC18
Content	= Single Pulse Experimen
Creation Date	= 6-FEB-2013 11:52:31
Revision Date	= 6-FEB-2013 12:25:35
Spec Site	= ECP400
Spec Type	= DELTA_NMR
Data Format	= 1D COMPLEX
Dimensions	= X
Dim Title	= 1H
Dim Size	= 16384
Dim Units	= [ppm]
Experiment	= single_pulse.exp
Field_strength	= 9.389766[T]
X_domain	= 1H
X_freq	= 399.7841973 [MHz]
X_offset	= 5 [ppm]
X_sweep	= 12.00480192 [kHz]
X_points	= 16384
X_resolution	= 0.73275969 [Hz]
Recvr_gain	= 17
Filter_mode	= BUTTERWORTH
X_prescans	= 0
Scans	= 8
Irr_noise	= WALTZ
Irr_pwidth	= 50 [us]
Relaxation_delay	= 4 [s]
Solvent	= CHLOROFORM-D
Temp_get	= 20.5 [dC]
Spin_get	= 12 [Hz]
Probe_id	= 2564

Compound 8i: ¹³C-NMR

