

Protocol for Training and Authorization

Please complete all the steps in order. You are not permitted to use the spectrometers alone until you receive authorization

Step 1, Pretraining: You need to know how to prepare a high quality NMR sample. Read pps 75-81 in “High-Resolution NMR Techniques in Organic Chemistry” (T. D. W. Claridge, Pergamon, 1999).

Step 2, Training: Contact the Director to arrange for training. Arrange for a series of practice sessions with Macduff. You need to learn how to shim and how to collect and process data for basic ^1H , ^{13}C and DEPT NMR experiments. Take the self-test when you and your trainer feel confident that you have learned the needed skills.

Step 3, Self test – ^1H NMR spectrum:

- Prepare a sample of 5% ethyl crotonate in CDCl_3
- Collect data from 8 scans under the conditions of the standard ^1H NMR experiment, process the data in the standard fashion;
- Print spectrum in the required format (see below).

Step 4, Self test – ^{13}C and DEPT135 NMR spectrum:

- Prepare a sample of 20% ethyl crotonate in CDCl_3
- Collect data from 32 scans under the conditions of the standard ^{13}C and DEPT135 NMR experiment, process the data in the standard fashion;
- Print spectrum in the required format (see below).

Step 5, Review: Review the quality of your spectra with your trainer and then your advisor. Sample spectra are appended to this document. The quality of your spectra should be as good as or better than these samples. Is your baseline even? Are your peak shapes narrow and symmetrical? Can you see the small splittings in the ^1H NMR spectrum? Etcetera.

Step 6, Request Authorization: Complete the authorization request form and submit the form along with your spectra to the Director. You will be notified shortly whether you are authorized.

PITTCHEM NMR facility

SAFETY ISSUES

1. The magnetic field can erase information from credit cards, electronic access/ID cards and magnetic storage media. Set aside these items; do not take them near the magnet. Analog watches can also be significantly slowed down once they are magnetized.
2. Samples containing radioactive nuclides are not allowed in the NMR facility.
3. Toxic or compounds with offensive smell should be taken utmost care responsibly according to their nature. In case of any spill please report immediately.
4. Gloves should never be worn while operating the computer / spectrometer.
5. For VT control, users are responsible to know the temperature limitations of their NMR samples and to know how to use the spectrometer safely within these limitations.
6. The Bruker 500 and 600 MHz spectrometer is equipped with pneumatic anti-vibration legs. While this improves data quality, it makes the magnet very vulnerable to damage due to rocking. Never lean against the magnet.

DOs & DON'Ts RUNNING NMR EXPERIMENTS

1. No one is to use the NMRs without training by Damodaran or Macduff. This includes changing nuclei, probe tuning, temperature experiments, etc. Carefully follow all written instructions. If in doubt ask!
2. Do not change pulse power levels or the lock power level.
3. Follow the signup rules posted on FACES website.
4. Never stop or remove the sample when another person's experiment is running.
5. Use only good quality NMR tubes. Use 507-PP and higher for the 300. Use 528-PP or 535-PP tubes for 500/600. If you seal an NMR tube, make sure it is symmetric and doesn't wobble.
6. Make absolutely sure that the outside of the NMR tube is clean with no dirt, oil, crud, etc. Make sure you wipe the lower part of the tube with a Kimwipe before inserting in the magnet.
7. Promptly report any instrument problems, including warning messages, to Damodaran, or Macduff. Do not attempt any repairs on your own and do not contact the instrument shop for repairs.

8. Immediately report any accidents, such as breaking a tube in the magnet, or spills to Damodaran or Macduff. Accidents happen and will usually be excused; however, failure to report accidents or repeated careless accidents will result in loss of NMR privileges.
9. Place the standard sample back into the magnet when you have finished your experiments. After observing an X nucleus other than ^{13}C , change back to ^{13}C .
10. Accurately record your instrument use including date(s), “am” or “pm”, total time used, type of experiment, and comments in the logbook.
11. Keep the lab clean. Throw out used Kimwipes, chemical gloves, scraps of paper, and labels into the trash can. Remove your samples and any glassware.

Requisition for authorization to use the PittChem NMR Facility

I request authorization to become a basic user of the PittChem 300 MHz NMR Spectrometers. I read and I agree to follow the “Safety issues” and “Dos and Don’ts running NMR experiments”. I have attached my ^1H , ^{13}C and DEPT135 spectra plotted as required.

Time spent Training and Practicing \approx _____ hours

Spectrometer Used:

Pitt Email:

Phone:

Room #:

Date:

User Name

Trainer Name

Advisor Name

Signature

Signature

Signature

1H NMR of 5% Ethyl Crotonate in CDCl3

6.87
6.85
6.83
6.82
6.81
6.80
6.78
6.75
5.72
5.71
5.71
5.70
5.67
5.66
5.65
5.65
4.05
4.05
4.03
4.01
3.98
1.75
1.73
1.73
1.72
1.71
1.70
1.15
1.12
1.11
1.10
1.09

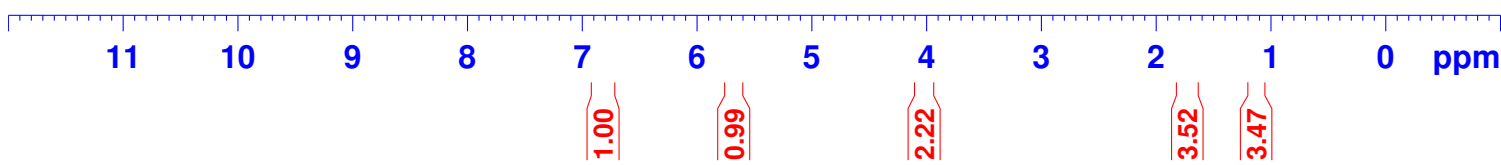
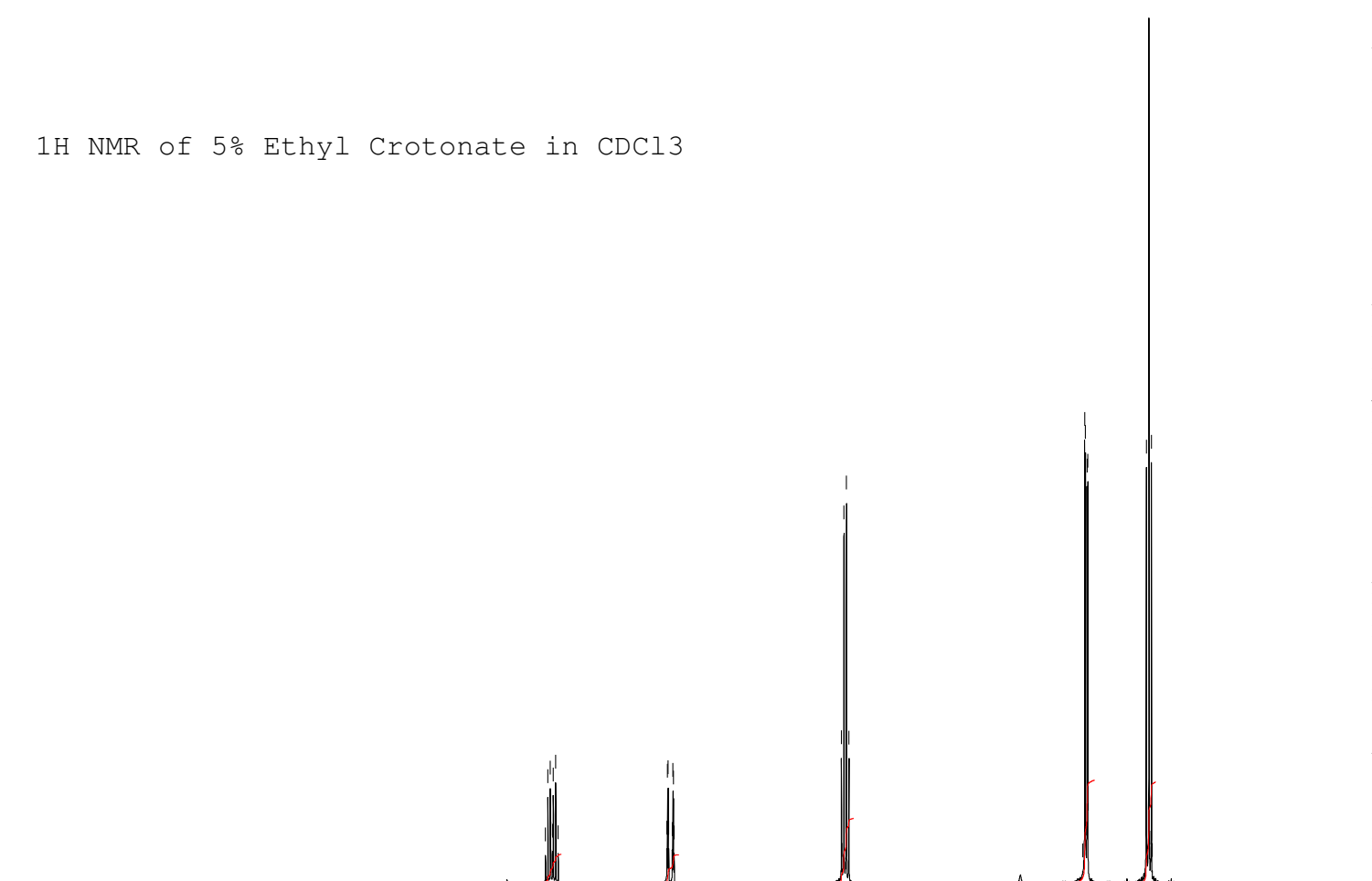
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PROCNO    1

F2 - Acquisition Parameters
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PULPROG   zg
TD         65536
SOLVENT   CDCl3
NS         8
DS         2
SWH        6218.905 Hz
FIDRES     0.094893 Hz
AQ         5.2691445 sec
RG         12.7
DW         80.400 usec
DE         6.00 usec
TE         300.0 K
D1         2.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         9.00 usec
PL1        1.00 dB
SFO1       300.3818550 MHz

F2 - Processing parameters
SI         32768
SF         300.3800079 MHz
WDW        no
SSB        0
LB         0.00 Hz
GB         0
PC         1.00
    
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11 10 9 8 7 6 5 4 3 2 1 0 ppm

1.00 0.99 2.22 3.52 3.47

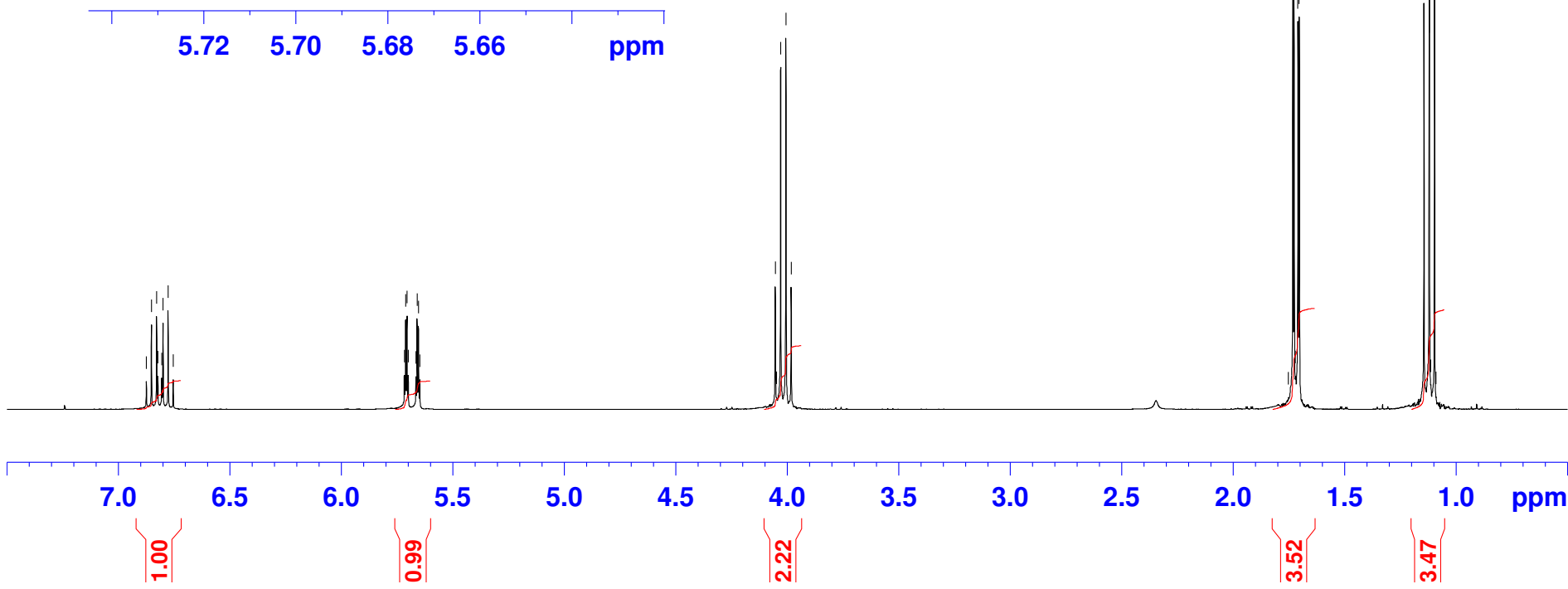
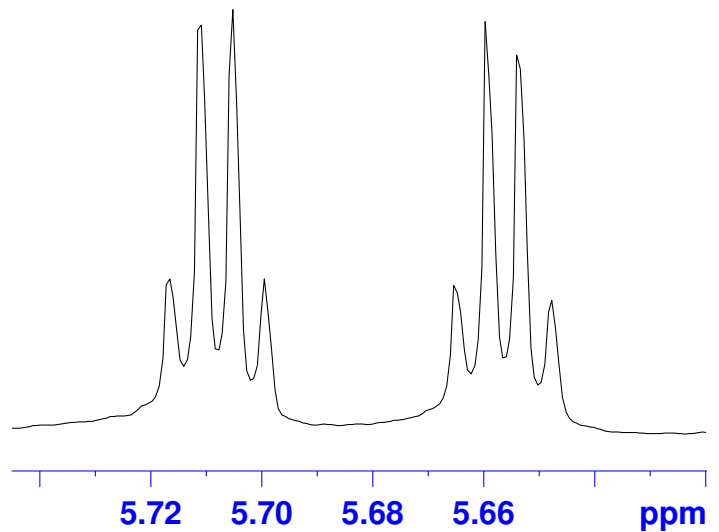
6.87
6.85
6.83
6.82
6.81
6.80
6.78
6.75

5.72
5.71
5.71
5.70
5.67
5.66
5.65
5.65

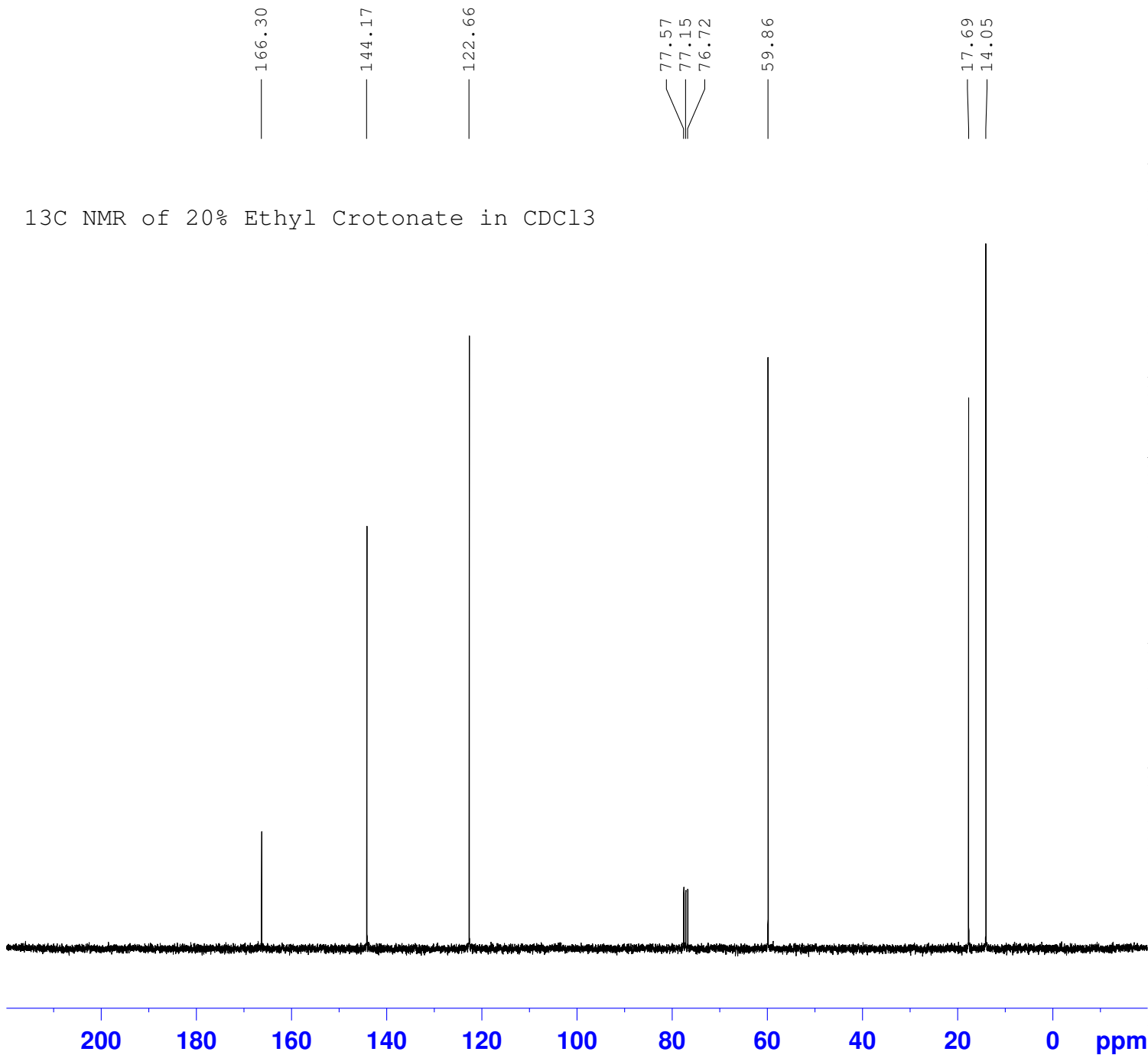
4.05
4.05
4.03
4.01
3.98

1.75
1.73
1.73
1.72
1.71
1.70
1.15
1.12
1.11
1.10
1.09

¹H NMR of 5% Ethyl Crotonate in CDCl₃



13C NMR of 20% Ethyl Crotonate in CDCl3



Current Data Parameters
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EXPNO 2
PROCNO 1

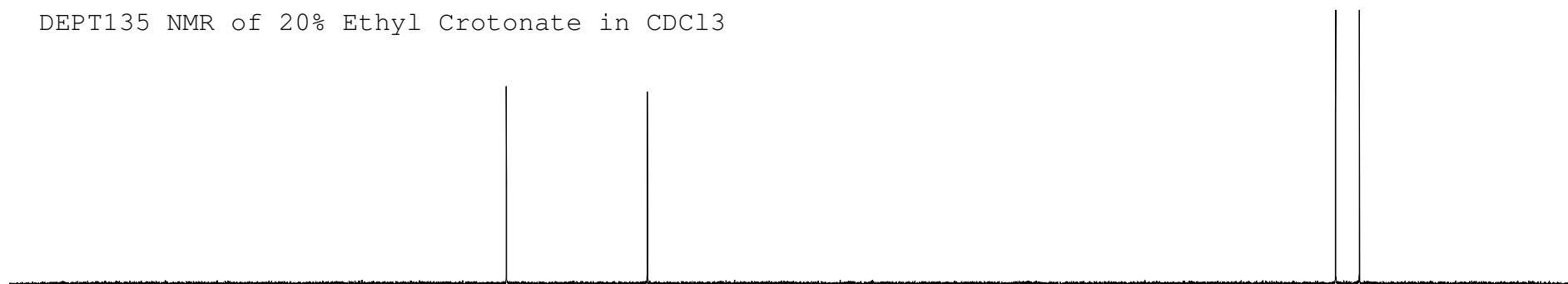
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TD 65536
SOLVENT CDCl3
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DS 4
SWH 18115.941 Hz
FIDRES 0.276427 Hz
AQ 1.8088436 sec
RG 32768
DW 27.600 usec
DE 6.00 usec
TE 300.0 K
D1 10.00000000 sec
d11 0.03000000 sec
DELTA 9.89999962 sec
TD0 1

==== CHANNEL f1 =====
NUC1 13C
P1 9.00 usec
PL1 1.80 dB
SFO1 75.5381641 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 92.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 300.3812015 MHz

F2 - Processing parameters
SI 65536
SF 75.5306110 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

DEPT135 NMR of 20% Ethyl Crotonate in CDCl3



¹³C NMR of 20% Ethyl Crotonate in CDCl3

