Nickel-Catalyzed Suzuki–Miyaura Cross-Coupling in a Green Alcohol Solvent for an Undergraduate Organic Chemistry Laboratory

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Supporting Information – Table of Contents

Materials and Methods

Unless stated otherwise, commercially obtained reagents were used as received. Bis(tricyclohexylphosphine)nickel(II) dichloride, 97%, (CAS:19999-87-2) was obtained from Sigma-Aldrich. *Tert*-amyl alcohol, 99%, pure, (CAS: 75-85-4) and potassium phosphate, tribasic, 97%, pure, anhydrous, (CAS: 7778-53-2) were obtained from Acros Organics. 5-Bromopyrimidine, 98%, (CAS: 4595-59-9), furan-3-boronic acid, 98%, (CAS: 55552-70-0), and 2-methoxypyridine-3-boronic acid, 97%, (CAS: 163105-90-6) were obtained from Combi-Blocks, Inc. Thin-layer chromatography (TLC) was conducted with EMD gel 60 F254 pre-coated plates (0.25 mm) and visualized using a combination of UV, anisaldehyde, and vanillin staining techniques. Silicycle Siliaflash P60 (particle size 0.040–0.063 mm) was used for flash column chromatography. ^{1}H NMR spectra were recorded on a Bruker spectrometer (at 500 MHz) and are reported relative to deuterated solvent signals. Data for ^{1}H NMR spectra are reported as follows: chemical shift (δ ppm), multiplicity, coupling constant (Hz) and integration. ^{13}C NMR spectra were recorded on a Bruker Spectrometer (at 125 MHz). Data for ^{13}C NMR spectra are reported in terms of chemical shift.

Equipment and Supplies

(VWR®) Borosilicate glass vials, 1 dram, 15 X 45 mm, short type

Solid green Melamine caps with PTFE liner (size 13-425)

Teflon tape and Parafilm M

Beaker (50 mL)

Erlenmeyer flask (125 and 250 mL)

Graduated cylinders (10 and 100 mL)

Filter funnel, paper, and flask

Separatory funnel

Stirring hotplates

Heating block (alternatively, oil bath can be used)

Magnetic stirring bar

Pasteur pipettes and pipette bulbs

Spatulas

Weighing papers

Test tubes (13 X 100 mm)

Test tube rack

NMR tubes (1 per student)

NMR caps (1 per student)

$CDCl_3$, 99.8% (CAS: 865-49-6); obtained from Cambridge Isotope Laboratories, Inc.

Pre-Lab Handout

1. Provide a brief description of the Suzuki–Miyaura cross-coupling reaction.

2. Suzuki–Miyaura cross-couplings use organoboron reagents as coupling partners, as opposed to Stille couplings, which use organotin reagents. List two desirable characteristics of organoboron reagents that render Suzuki–Miyaura cross-couplings especially attractive.

3. Define the term ‘green chemistry’.

4. Anastas and Warner popularized the concept ‘12 principles of green chemistry’. List 6 of these key ideas.

5. For the experiment you will perform, you will be given one of two possible boronic acids as the nucleophilic coupling partner. The other reagent ingredients are consistent regardless of which boronic acid is used. Draw chemical structures of the electrophile, catalyst, base, and solvent you will be using.

Electrophile	**Catalyst**	**Base**	**Solvent**

6. Briefly explain the role of the base in the reaction.

7. Safety is a paramount concern when conducting any laboratory experiment. Describe three safety concerns you have about performing this particular experiment and any precautions you will take to meet these concerns.

8. Considering the '12 Principles of Green Chemistry', briefly describe two ways in which the reaction you are performing is considered 'green'.

Student Handout

A. Objectives

This experiment is an introduction to cross-coupling reactions and features a modern twist. Specifically, this experiment involves the Ni-catalyzed Suzuki–Miyaura reaction to link aromatic fragments that contain heteroatoms, or 'heterocycles' (Figure S1). Additionally, the importance of 'green' and sustainable chemistry will be introduced in this laboratory experiment. NMR spectroscopy will be used to determine the outcome of the reaction, which should provide analytical training to the experimenter.

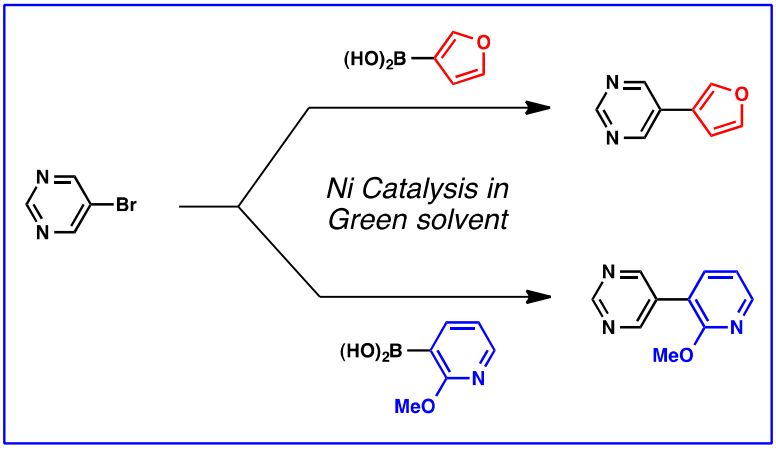

Figure S1. The nickel-catalyzed Suzuki–Miyaura cross-coupling reactions performed in this undergraduate laboratory experiment.

B. Introduction

Cross Coupling Reactions

Cross-coupling reactions have become one of the most utilized tools to assemble carbon–carbon (C–C) and carbon–heteroatom bonds in chemistry.[1] In fact, the 2010 Nobel Prize in Chemistry was awarded to Richard Heck, Ei-ichi Negishi, and Akira Suzuki for their pioneering work in Pd-catalyzed cross-coupling chemistry that is now used in a host of academic and industrial applications. Among these powerful transformations, the Suzuki–Miyaura cross-

coupling, generally defined as the transition-metal-catalyzed cross-coupling between an organic halide and an organoboron compound, has become one of the most attractive approaches for the assembly of C–C bonds.[2] Other types of important cross-coupling reactions include Stille couplings, Sonogashira Couplings, and Heck couplings to build C–C bonds, in addition to Buchwald–Hartwig couplings to build carbon–heteroatom bonds.

Nickel Catalysis, Heterocycles, and Green Chemistry

Although the use of palladium catalysis is most common in cross-coupling reactions, complementary approaches to achieve such couplings are highly sought after. One attractive alternative involves the use of nickel catalysts in place of palladium catalysts (Figure S2). Several aspects of nickel render it attractive: (a): nickel is readily available, as opposed to palladium, which is considered a 'precious metal'; (b) nickel metal is considerably cheaper compared to palladium metal, as shown in Figure S2, although the overall price for a given chemical process will depend on a variety of factors; (c) nickel is considered less toxic compared to palladium; for example, greater amounts of trace nickel (roughly 2x) can remain in a final orally administered drug substance compared to palladium; (d) although not the focus of this laboratory experiment, nickel catalysis can be used to react several unconventional electrophiles in place of commonly used aryl halides;[3] (e) nickel catalysis can be used to form linkages between two heterocyclic coupling fragments;[4] heterocycle–heterocycle couplings are generally challenging for catalytic reactions because heteroatoms such as oxygen and nitrogen can bind to metals and 'poison' the catalysts. Heterocycles, or organic molecules that possess heteroatoms such as nitrogen and oxygen, are prevalent in polymers, materials, ligands, bioactive compounds, and natural products. Methods to link heterocycles together are important, as the resulting products called bis(heterocycles) are also important classes of compounds (see Figure S3).

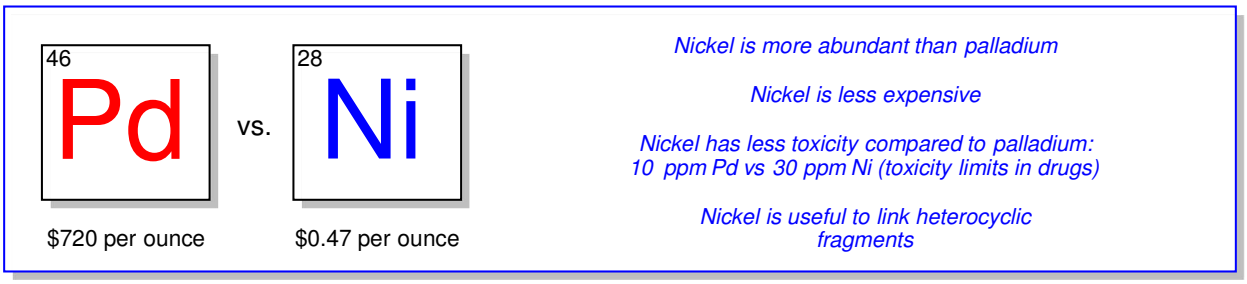

Figure S2. Nickel as an attractive alternative to palladium.

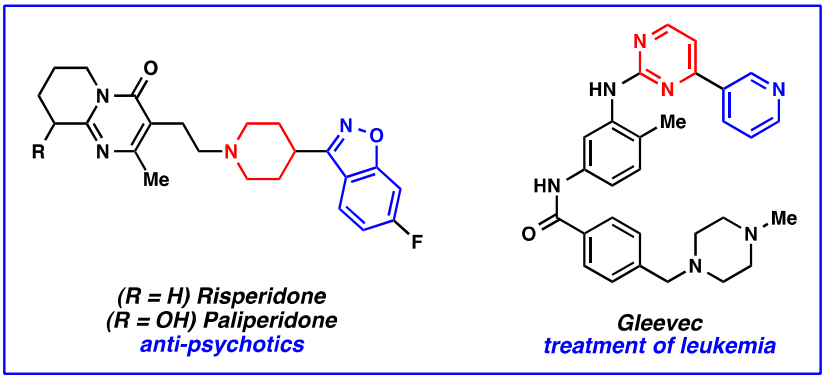

Figure S3. Examples of top selling drugs which contain bis(heterocycle) motifs.

By shifting from palladium to nickel, there are also opportunities in the realm of 'green chemistry'. 'Green chemistry', or the design of chemical processes that reduce or eliminate the use of hazardous and environmentally unfriendly substances, is one of the most important modern initiatives in the chemical sciences.[5] For instance, the pharmaceutical sector is readily embracing more 'green' processes to circumvent many unfavorable aspects of drug production, including the use of unrecoverable starting materials, excessive use of environmentally unfriendly organic solvents for separation and purification, and use of toxic reagents. It is estimated that 25–100 kg of waste is produced per kg of drug produced in a 6–8 step industrial drug manufacturing process.[6] Of this waste, 85% consists of organic solvent.[6] Therefore, the ability to efficiently carry out cross-coupling reactions in more environmentally friendly

solvents,[7,8] such as alcohols (e.g., *tert*-amyl alcohol) remains a challenge and important goal of green chemistry research.

Mechanism of the Suzuki–Miyaura Coupling

The general mechanism of the Suzuki–Miyaura cross-coupling reaction, catalyzed by the nickel catalyst used in this laboratory, is outlined in Figure S4.[2d] The first step of the proposed mechanism is to generate an active Ni(0) catalyst. This is achieved by the boronic acid, which reduces the starting Ni(II) pre-catalyst to a related Ni(0) complex. The complex then inserts into the carbon–bromide bond through a process called ***oxidative addition***, where the nickel changes to the +2 oxidation state. The oxidative addition intermediate then undergoes ***transmetallation***, where the organic fragment attached to boron is transferred to the nickel center. This is facilitated by the presence of base.[9] In the final step, ***reductive elimination*** forms the key C–C bond and regenerates the active Ni(0) catalyst, which, in turn, can re-enter the catalyst cycle.

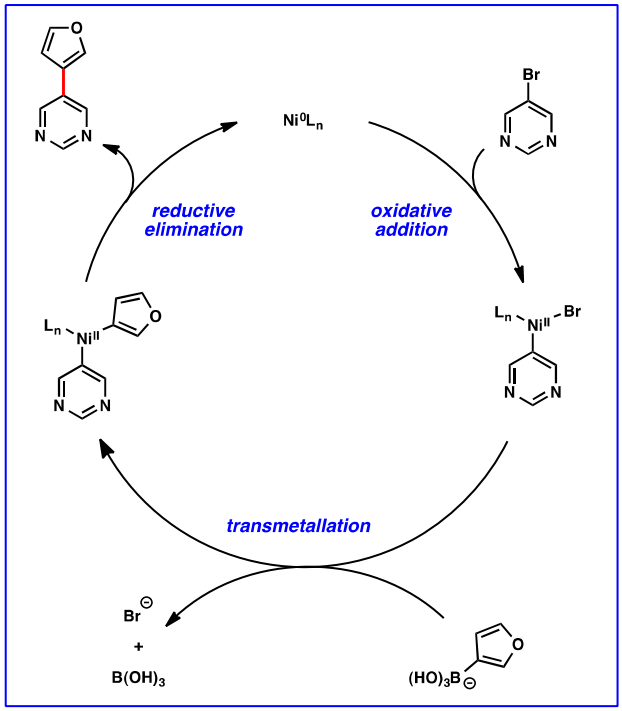

Figure S4. Proposed reasonable mechanism of the nickel-catalyzed Suzuki–Miyaura coupling.

C. Experimental

Safety Hazards and Considerations

Closed-toed shoes, long pants, safety glasses, gloves, and flame-resistant laboratory coats should be worn at all times. All hazardous materials should be handled and disposed of in accordance with the recommendation of the materials' safety data sheet and EH&S. Bis(tricyclohexylphosphine)nickel(II) dichloride ($NiCl_2(PCy_3)_2$) and heterocyclic boronic acids may be harmful if inhaled, swallowed, or absorbed through skin. 5-Bromopyrimidine and potassium phosphate are irritants and may be harmful if inhaled, swallowed, or absorbed through skin. *Tert*-amyl alcohol is an irritant and may therefore cause skin and eye irritation; it is also flammable and may be harmful if inhaled, swallowed, or absorbed through skin. Hydrochloric acid and sodium hydroxide are corrosive and can cause burns to skin, eyes, and respiratory tract. Stock solutions of 1 M aqueous hydrochloric acid (HCl) and sodium hydroxide (NaOH) should be used and prepared in a fume hood. Ethyl acetate and hexanes are flammable and volatile organic solvents. The *n*-hexane in hexanes is a neurotoxin. $CDCl_3$ is toxic and a cancer suspect agent. The products of the coupling are not considered harmful, but care should be taken to avoid inhalation or contact with skin. Stirring-hotplates should be used inside the fume hood and kept away from flammable solvents. When the reaction is quenched with HCl, the reaction mixture should be at room temperature (see page S11).

Experimental Procedures

Note: students will be randomly assigned one of two heterocyclic boronic acids (see Figure S5). After completing the experiment, purification, and NMR analysis, students will determine which boronic acid they were given and the structure of the cross-coupled product. Students must support their structural assignment based on their collected NMR data.

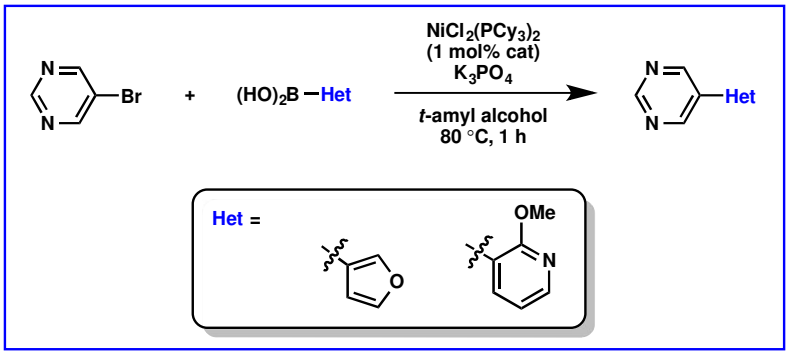

Figure S5. Cross-coupling reaction between 5-bromopyrimidine and a heterocyclic boronic acid.

Add 5-bromopyrimidine (100.00 mg, 0.63 mmol, 1.00 equiv), $NiCl_2(PCy_3)_2$ (4.40 mg, 0.0063 mmol, 0.01 equiv), unknown heterocyclic boronic acid (1.58 mmol, 2.50 equiv), and K_3PO_4 (603.00 mg, 2.84 mmol, 4.50 equiv) to a 4 mL glass vial (1 dram).

For unknown boronic acid # 1, add 176 mg.
For unknown boronic acid # 2, add 240 mg.

After adding all of the solids, add a stir bar and *t*-amyl alcohol (2.10 mL). Cap the vial tightly with a green thermoset screw cap. Seal the green cap with Teflon tape and then wrap the vial with parafilm to prevent the risk of solvent leaking. Please ask your instructor or teaching assistant if you need help with this or any other part of the experimental protocol. Allow the heterogeneous mixture to stir at 23 °C for 30 min, and then heat the mixture to 80 °C for 1 h. Remove the reaction vessel from the heat source and set the vessel aside in the fume hood for 5-10 min for cooling. Alternatively, upon completion of the reaction, the temperature of the heat source can be adjusted to 23 °C and the vessel can be removed once the temperature reaches 23 °C. Quench the reaction by adding 1 M aqueous HCl (3 mL) dropwise.

Separate the organic and aqueous layers using a separatory funnel. Extract the aqueous layer with EtOAc (3 x 4 mL). Wash the combined organic layers with 1 M aqueous NaOH (4

mL), brine (4 mL), dry the organic layer over magnesium sulfate ($MgSO_4$), and filter the mixture to remove $MgSO_4$. Perform thin layer chromatography (TLC) (recommended solvent mixture is 3:1 Hexanes:EtOAc) and record your observation. Remove the solvent using a rotary evaporator.

Purify the crude residue by silica gel flash chromatography (recommended solvent mixture is 3:1 Hexanes:EtOAc). Collect the fractions in test tubes and then transfer the product-containing fractions to a round bottom flask. Wash the test tubes with a small quantity of EtOAc and combine the washing solvent to the round bottom flask. Remove the solvent using a rotary evaporator to yield the desired cross-coupled product.

Weigh the coupling product and calculate the isolated and percent yield. Prepare a sample for NMR analysis: measure about 50 mg of the product into a test tube and dissolve the product in roughly 1 mL of $CDCl_3$ and then transfer the resulting solution to an NMR tube by pipette. Acquire ^{1}H and ^{13}C NMR spectra. Analyze your NMR data, along with the attached NMR data for 5-bromopyrimidine, to identify the unknown boronic acid and the cross-coupled product.

Postlab Worksheet

1. Provide a sketch of your TLC plate and indicate how you visualized compounds on the plate. Calculate the R_f value of the coupling product. Be sure to indicate the solvent system you used for TLC.

2. Describe the physical appearance of the coupling product you obtained after chromatography and isolation.

3. Attach ^{1}H and ^{13}C NMR spectra of the coupling product and answer questions (a) and (b).

(a) Consider the ^{1}HNMR spectrum. For all peaks that correlate to the boronic acid fragment in the product, list the chemical shift, multiplicity, and integration.

(b) Consider the ^{13}CNMR spectrum. For all peaks that correlate to the boronic acid fragment in the product, list the chemical shift.

4. Based on inspection of your NMR data, the ^{1}HNMR and ^{13}CNMR data for the bromopyrimidine substrate provided, and the two possible structures shown in the laboratory handout, provide chemical structures for the 'unknown' boronic acid and the resulting product you isolated.

Unknown # ____	**Coupling product**

5. Provide the mass of isolated product you obtained and calculate the percent yield for the reaction.

6. You can measure catalyst efficiency by calculating the catalyst turnover number (TON), which is defined as the amount of reactant (moles) divided by the amount of catalyst (moles) times the % yield of product. A large TON (typically 10^3 or greater) indicates a stable, long-lived catalyst. Based on the resulting experimental yield, calculate the corresponding TON for the nickel catalyst used.

7. Do you observe any impurities by ^{1}H NMR analysis? _____. If impurities are observed, speculate as to what the impurities may be. You may choose among the options listed below. If you observe residual solvent(s), provide the structures for the solvent(s) and speculate as to the source of the solvent(s) in the NMR spectrum.

a. Bromide substrate
b. Boronic acid substrate
c. Nickel catalyst
d. Solvent

8. Suggest two modifications of this experimental protocol that would make it 'greener'.

Notes for Instructors

K_3PO_4 is hygroscopic and excess water could be deleterious to the reaction. The base is dried by flame-drying the powder under vacuum and stored in an airtight glass vial (preferably inside a desiccator).

The identities of the unknown boronic acids are furan-3-boronic acid and 2-methoxypyridine-3-boronic acid for unknown #1 and #2, respectively. Students' yields ranged from 28–95% for those who used the furanyl boronic acid, whereas yields were in the 39–100% range for those who employed the pyridyl boronic acid after purification of the crude product by column chromatography. Average yields were 61% and 76%, respectively.

A typical chromatographic purification on silica gel for this experiment requires 75–150 mL of the eluent (3:1 Hexanes:EtOAc). The students can perform the purification using a 2.5 cm X 15 cm column, loaded with 60 mL silica gel. When loading the compound on the column, if the crude product does not completely dissolve in the eluent, the addition of a few drops of EtOAc is recommended.

Reaction Set-Up Using a Round Bottom Flask

An alternative way to set up the reaction is by using round bottom flask, reflux condenser, and oil bath (Figure S6). Add 5-bromopyrimidine (100.00 mg, 0.63 mmol, 1.00 equiv), $NiCl_2(PCy_3)_2$ (4.40 mg, 0.0063 mmol, 0.01 equiv), unknown heterocyclic boronic acid (1.58 mmol, 2.50 equiv), and K_3PO_4 (603.00 mg, 2.84 mmol, 4.50 equiv) to a dry and clean 10 mL round bottom-flask.

For unknown boronic acid # 1, add 176 mg.
For unknown boronic acid # 2, add 240 mg.

After adding all of the solids, add a stir bar and *t*-amyl alcohol (2.10 mL). Grease the ground glass joints to prevent the risk of solvent leaking. *The reaction mixture could also be diluted to 0.6 M if preferred.* The round bottom flask is then equipped with a reflux condenser. Allow the heterogeneous mixture to stir at 23 °C for 30 min, and then heat the mixture to 80 °C for 1 h. Remove the reaction vessel from the heat source and set the vessel aside in the fume hood for 5-10 min for cooling. Alternatively, upon completion of the reaction, the temperature of the heat source can be adjusted to 23 °C and the vessel can be removed once the temperature reaches 23 °C. Quench the reaction by adding 1 M aqueous HCl (3 mL) dropwise.

Refer to S12 for the detailed work-up and purification procedure.

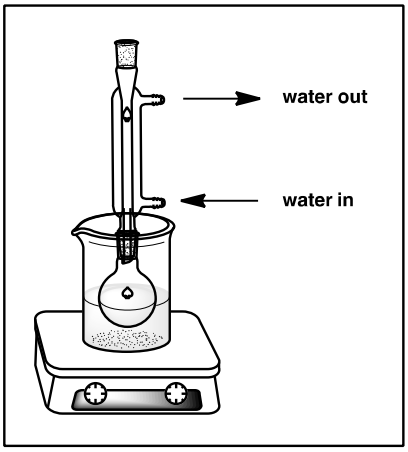

Figure S6. Reaction set-up using a round-bottom flask.

Reaction Set-Up Using a Microwave Reactor

Add 5-bromopyrimidine (103.3 mg, 0.65 mmol, 1.00 equiv), $NiCl_2(PCy_3)_2$ (4.4 mg, 0.0063 mmol, 0.01 equiv), the unknown boronic acid (1.58 mmol, 2.50 equiv), anhydrous powdered K_3PO_4 (603.00 mg, 2.84 mmol, 4.50 equiv) to a 10 mL microwave reaction vessel. After adding all of the solids, add a stir bar and *t*-amyl alcohol (2.10 mL).

For unknown boronic acid # 1, add 176 mg.
For unknown boronic acid # 2, add 240 mg.

Place the microwave reaction vessel in the (CEM Discover) microwave cavity. Set the temperature, pressure, and power to 150 °C, 200 psi, and 300 W, respectively. Irradiate the mixture at 150 °C for 10 min (ramp time 20 min). Remove the reaction vessel from the heat source and set the vessel aside in the fume hood for 5-10 min. Alternatively, upon completion of the reaction, the temperature of the heat source can be adjusted to 23 °C and the vessel can then be removed once the temperature reaches 23 °C. Quench the reaction slowly by adding 1 M aqueous HCl (3 mL).

See pages S11 and S12 for the detailed work-up and purification procedure.

Student Outcomes

As noted in the manuscript, 29/30 students who performed this experiment were able to obtain their desired products. In the case of low yields, students generally explained this outcome by suggesting their products may have been lost in the purification steps. In rare cases, students also observed the recovery of starting material. We also conducted anonymous student evaluations of this experiment. Students were asked what concepts they had learned and the replies included the following: “Green Chemistry”, “Green solvents”, “cross-couplings”, “catalytic cycles”, “Suzuki–Miyaura couplings”, “Nickel catalysis”. In addition, some of the comments provided by students are as follows:

- “Clean and simple reaction. Loved the experiment.”
- “Interesting lab! It made me think more about green chemistry, which is not discussed often.”

- "Overall, this experiment is a fun experience and I have a better understanding of cross-coupling after this experiment."
- "Using catalysis to create C–C bonds is so easy and efficient, compared to other methods such as air sensitive Grignard reagent, etc."
- I was surprised to learn the many possible ways to perform green chemistry. This gave me the option to seek out a job related to research in green chemistry.

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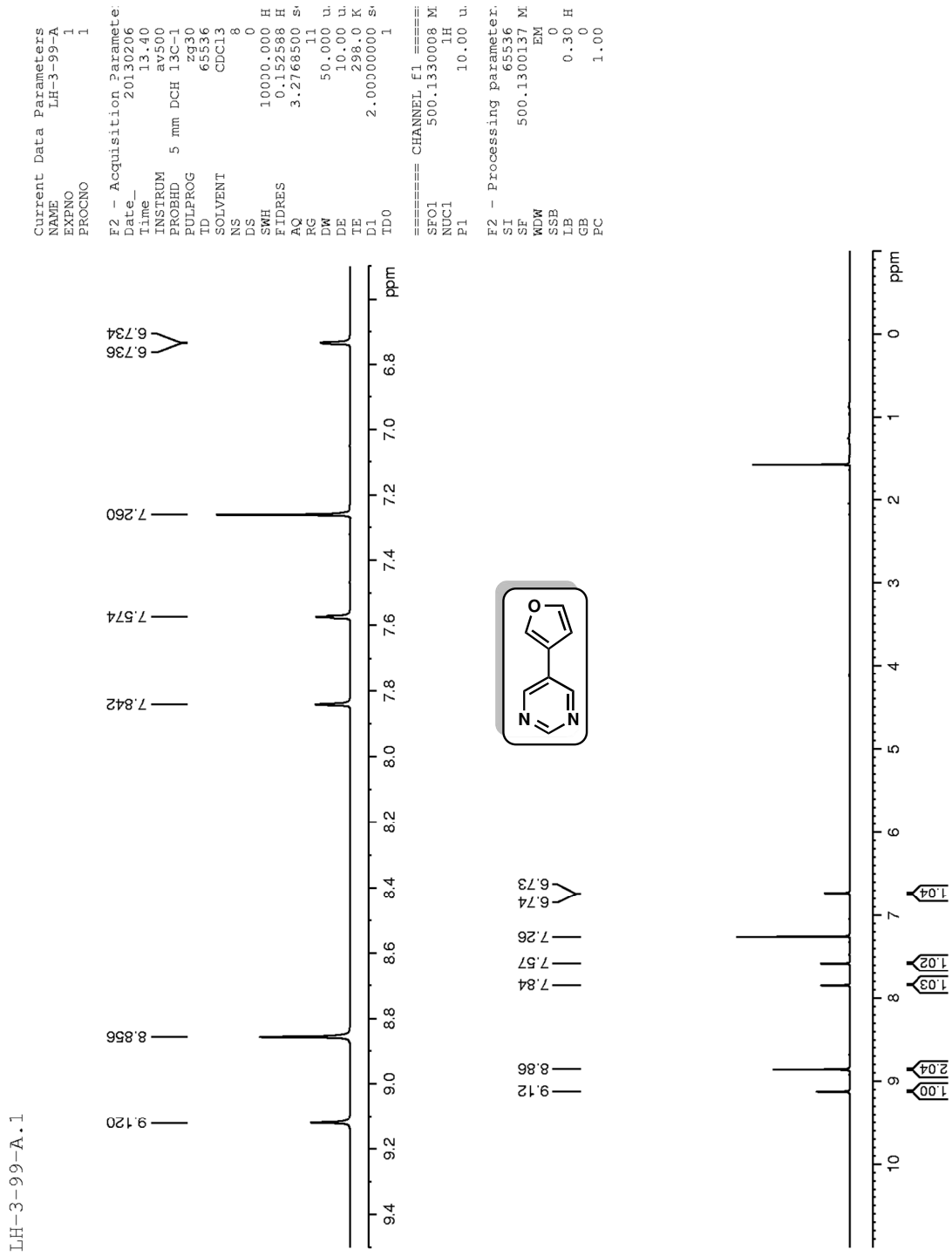
NMR Spectra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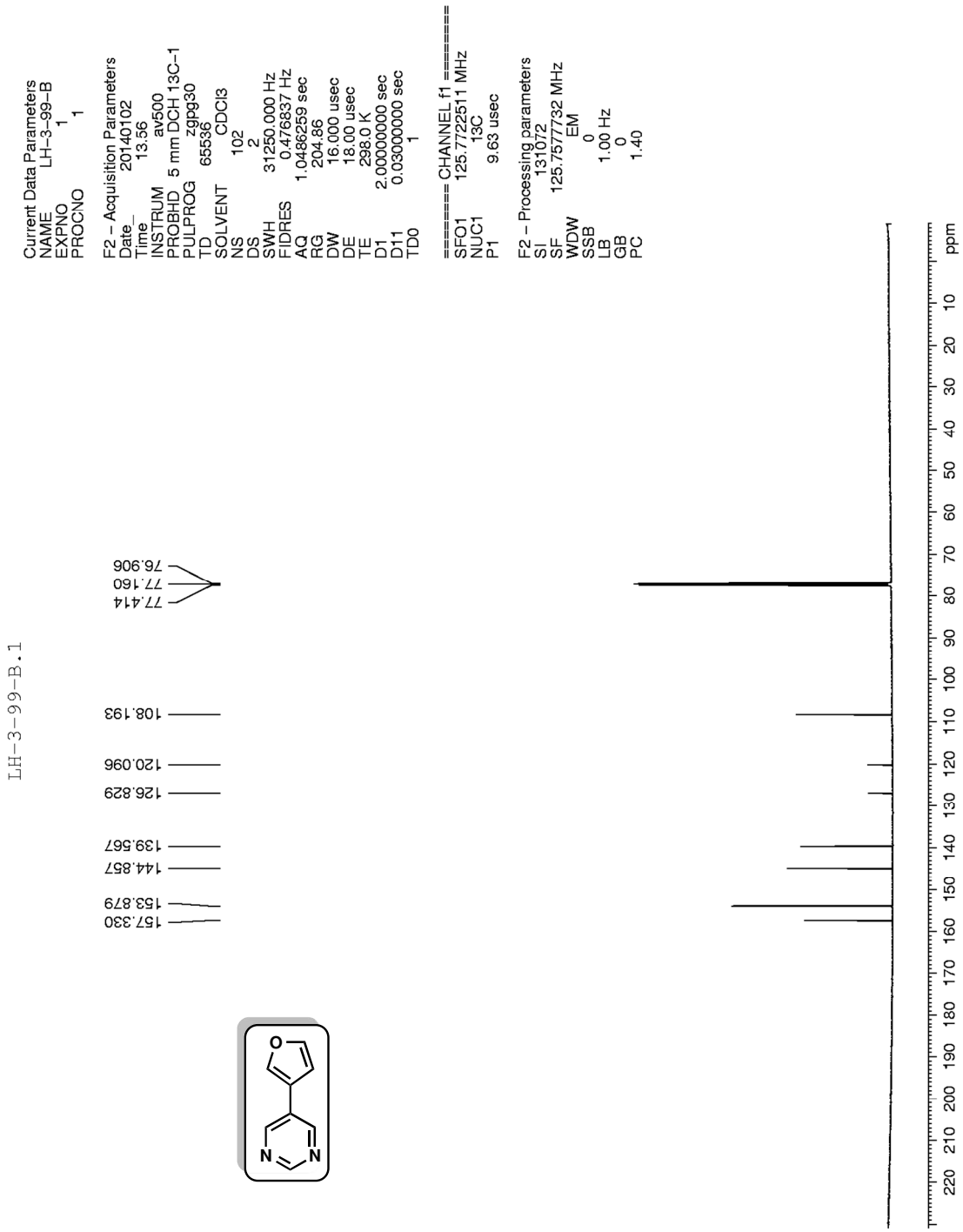
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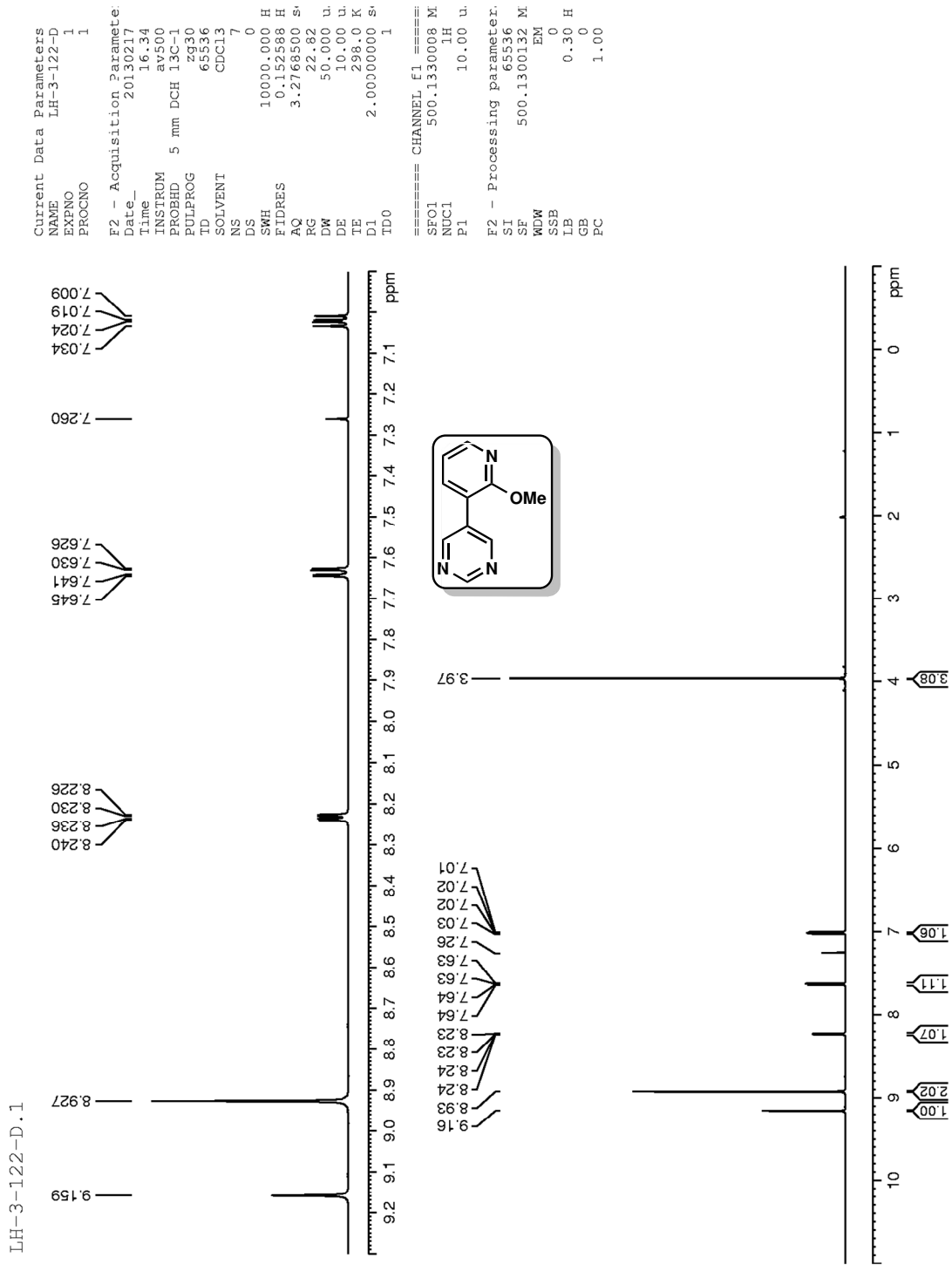
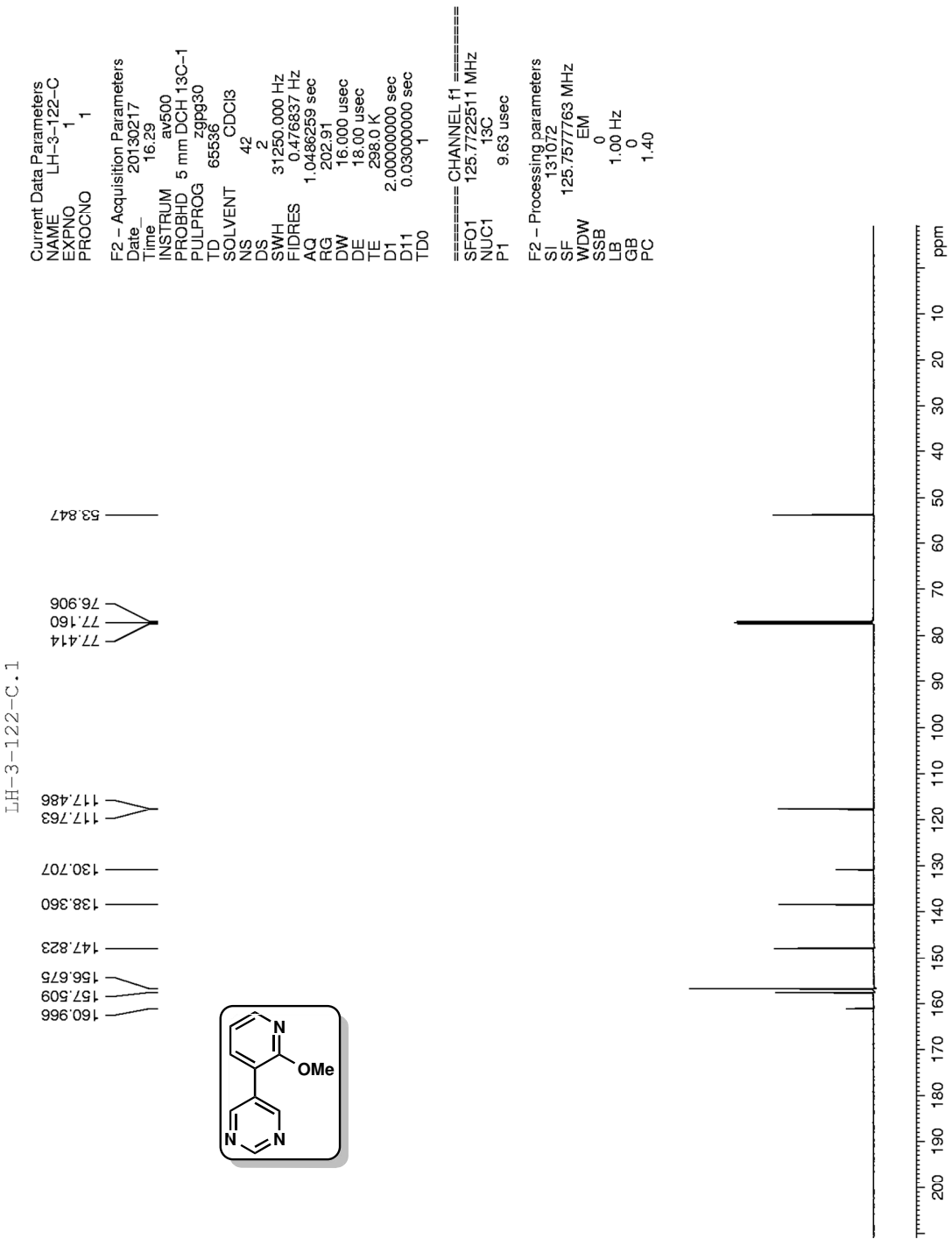
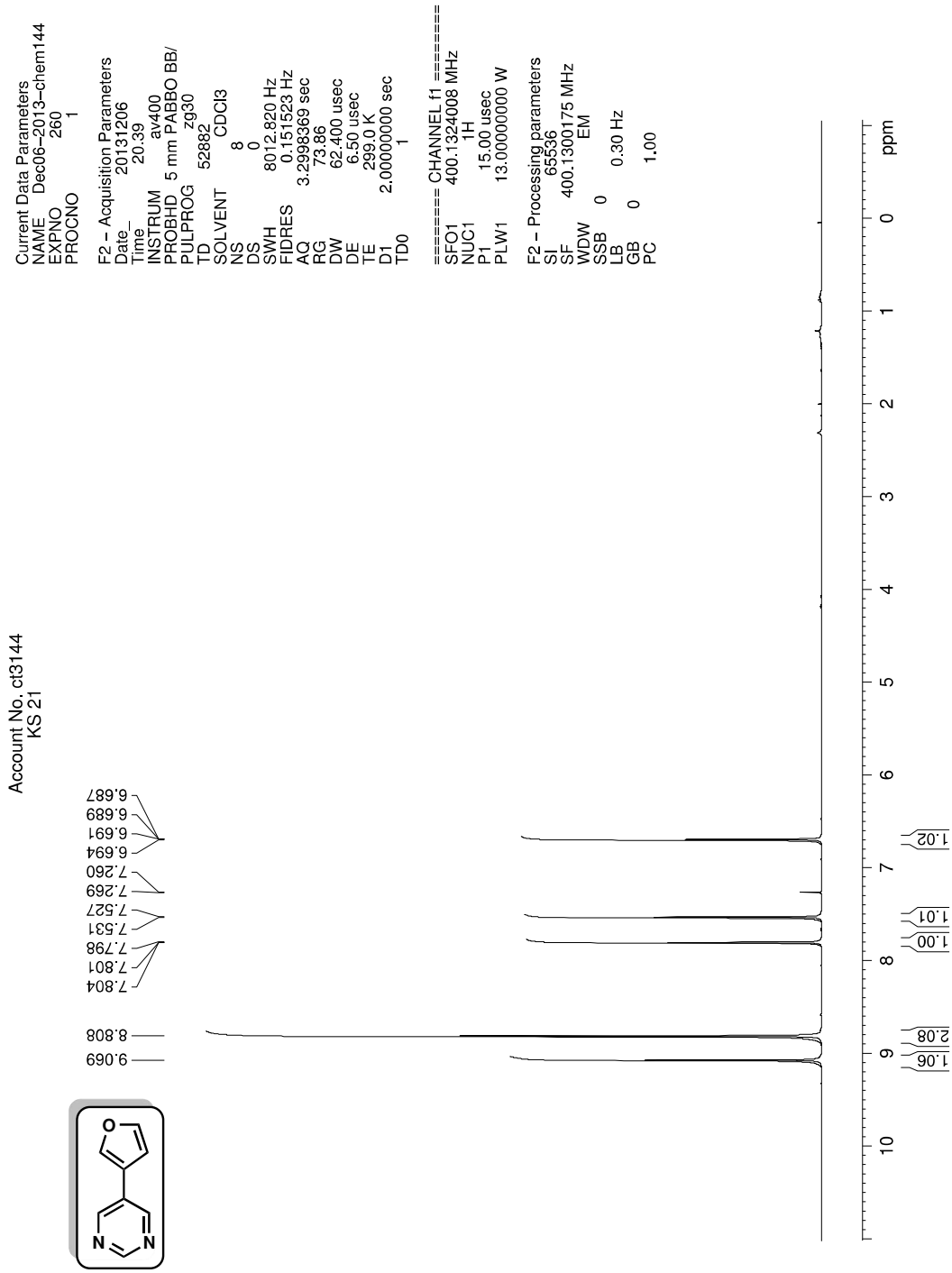
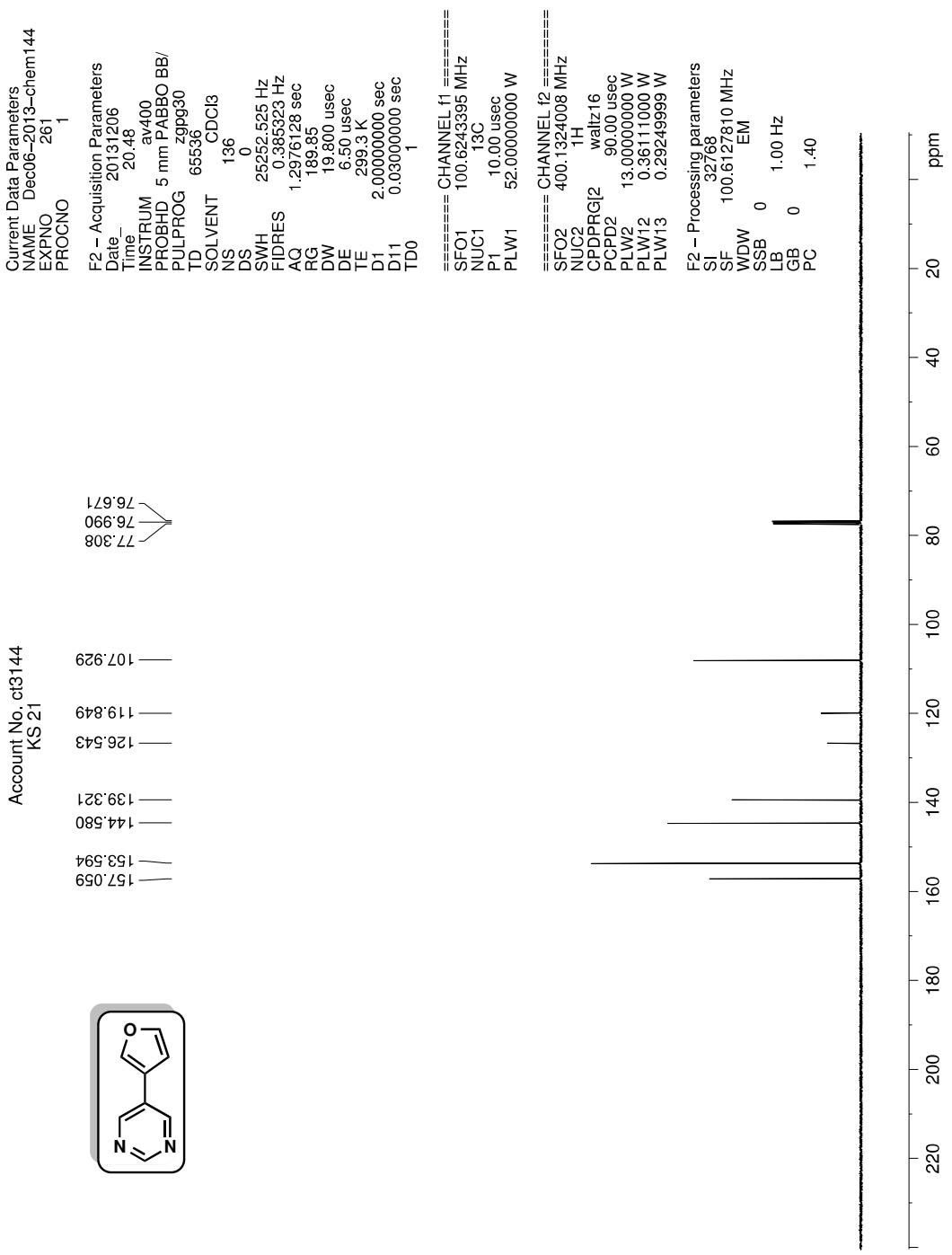
153.879
144.857
139.567
126.829
120.096
108.193
77.414
77.160
76.906
O
N
N
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

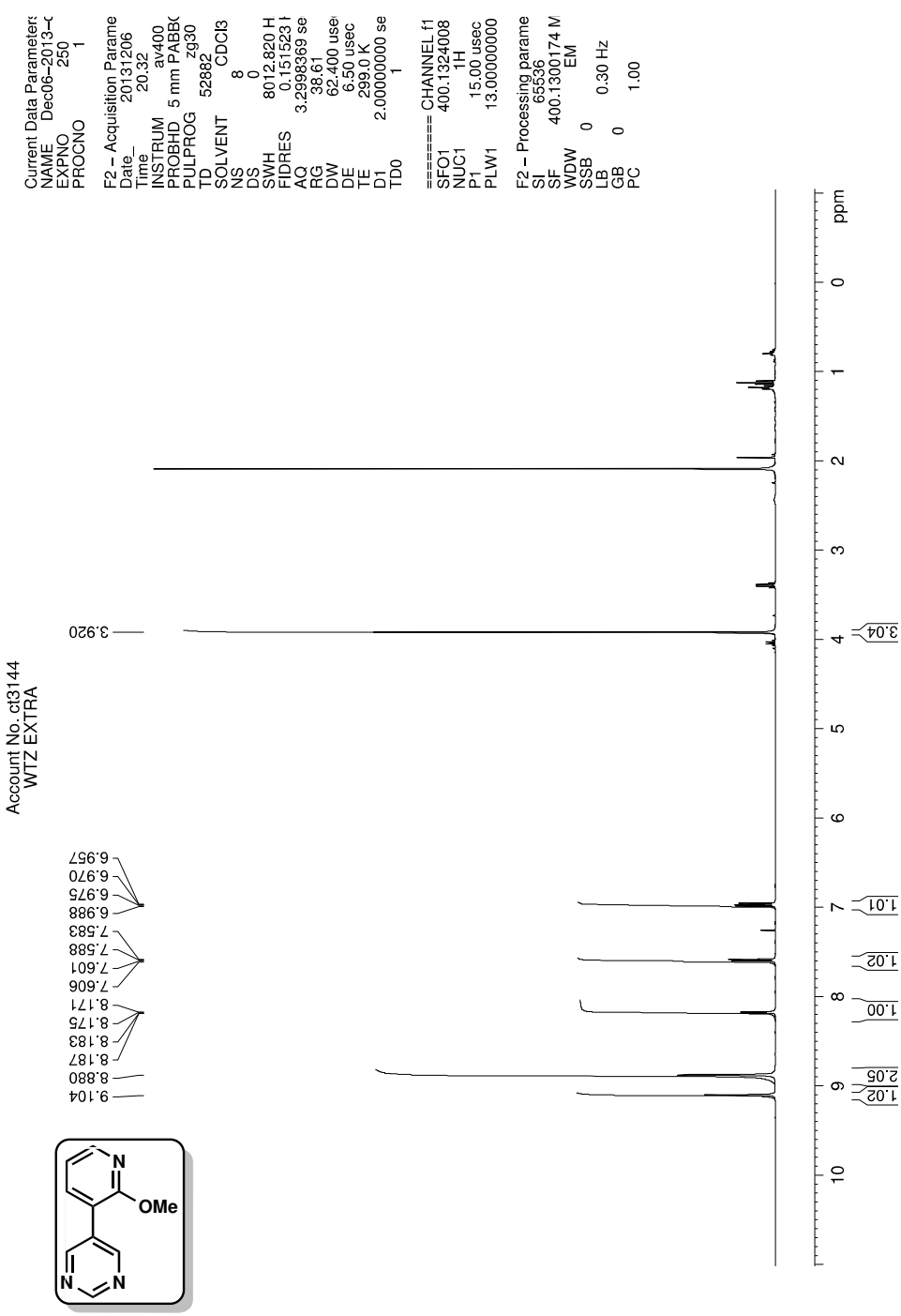

LH-3-122-D.1
Current Data Parameters
NAME LH-3-122-D
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130217
Time 16.34
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 7
DS 0
SWH 10000.000 H
FIDRES 0.152588 H
AQ 3.2768500 s
RG 22.82
DW 50.000 u
DE 10.00 u
TE 298.0 K
D1 2.00000000 s
TD0 1
======== CHANNEL f1 ======
SFO1 500.1330008 M
NUC1 1H
P1 10.00 u
F2 - Processing parameter
SI 65536
SF 500.1300132 M
WDW EM
SSB 0
LB 0.30 H
GB 0
PC 1.00
9.159
8.927
8.240
8.236
8.230
8.226
7.645
7.641
7.630
7.626
7.260
7.034
7.024
7.019
7.009
ppm
9.16
8.93
8.24
8.24
8.23
8.23
7.64
7.64
7.63
7.63
7.26
7.03
7.02
7.02
7.01
3.97
OMe
N
N
N
3.08
1.06
1.11
1.07
2.02
1.00
ppm

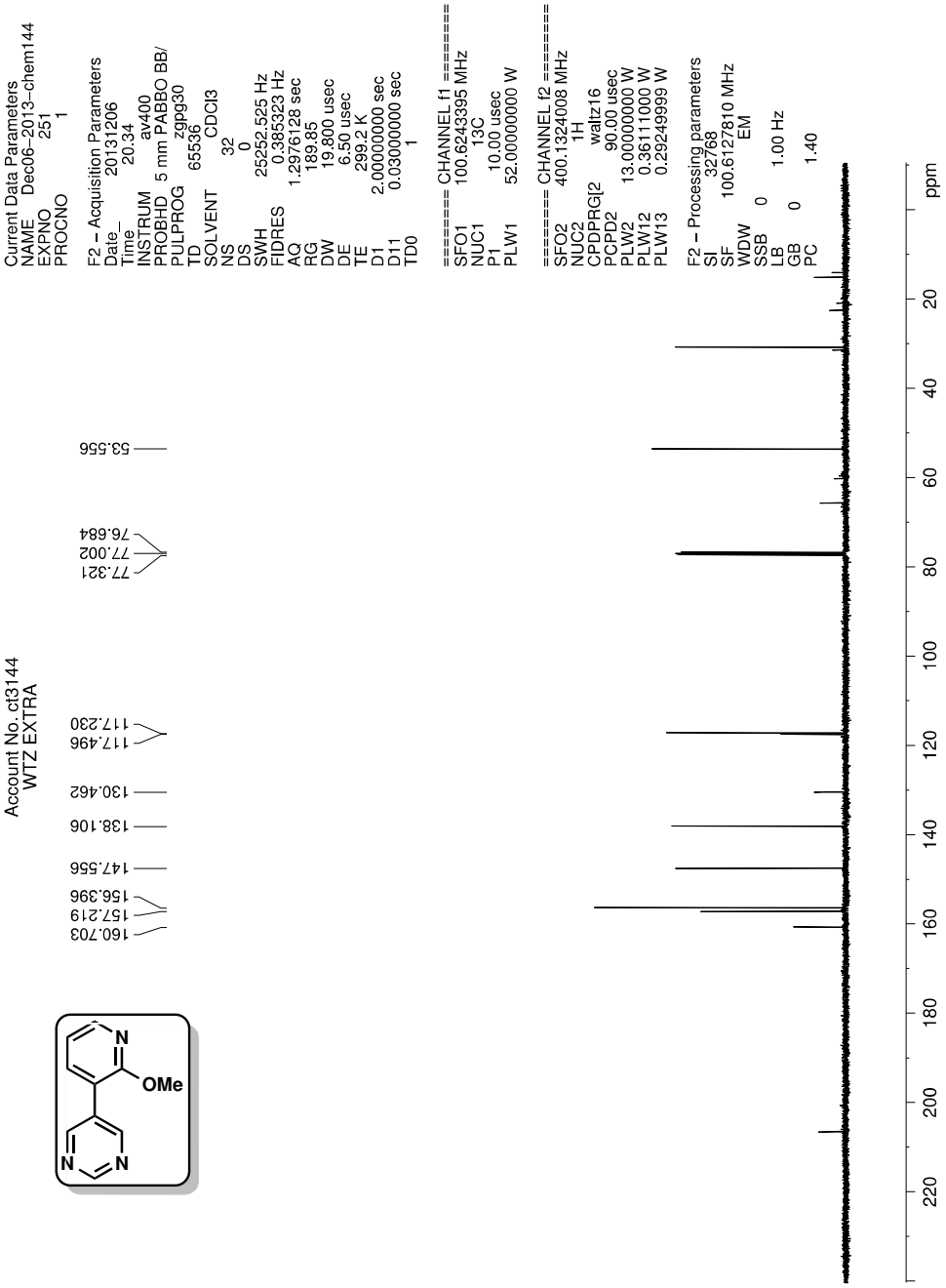

Current Data Parameters
NAME LH–3–122–C
EXPNO 1
PROCNO 1
F2 – Acquisition Parameters
Date_ 20130217
Time 16.29
INSTRUM av500
PROBHD 5 mm DCH 13C–1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 42
DS 2
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0486259 sec
RG 202.91
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7722511 MHz
NUC1 13C
P1 9.63 usec
F2 – Processing parameters
SI 131072
SF 125.7577763 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
LH-3-122-C.1
160.966
157.509
156.675
147.823
138.360
130.707
117.763
117.486
77.414
77.160
76.906
53.847
OMe
N
N
N
ppm

Representative NMR Spectra from Students


Account No. ct3144
KS 21
Current Data Parameters
NAME Dec06--2013--chem144
EXPNO 260
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131206
Time 20.39
INSTRUM av400
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 52882
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.151523 Hz
AQ 3.2998369 sec
RG 73.86
DW 62.400 usec
DE 6.50 usec
TE 299.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324008 MHz
NUC1 1H
P1 15.00 usec
PLW1 13.00000000 W
F2 - Processing parameters
SI 65536
SF 400.1300175 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.069
8.808
7.804
7.801
7.798
7.531
7.527
7.269
7.260
6.694
6.691
6.689
6.687
1.06
2.08
1.00
1.01
1.02
ppm


Account No. ct3144
KS 21
157.059
153.594
144.580
139.321
126.543
119.849
107.929
77.308
76.990
76.671
O
N
N
Current Data Parameters
NAME Dec06–2013–chem144
EXPNO 261
PROCNO 1
F2 – Acquisition Parameters
Date_ 20131206
Time_ 20.48
INSTRUM av400
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 136
DS 0
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976128 sec
RG 189.85
DW 19.800 usec
DE 6.50 usec
TE 299.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6243395 MHz
NUC1 13C
P1 10.00 usec
PLW1 52.00000000 W
======== CHANNEL f2 ========
SFO2 400.1324008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 13.00000000 W
PLW12 0.36111000 W
PLW13 0.29249999 W
F2 – Processing parameters
SI 32768
SF 100.6127810 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
220
200
180
160
140
120
100
80
60
40
20
ppm


Current Data Parameters
NAME Dec06-2013-c
EXPNO 250
PROCNO 1
F2 - Acquisition Parame
Date_ 20131206
Time 20.32
INSTRUM av400
PROBHD 5 mm PABB(
PULPROG zg30
TD 52882
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 H
FIDRES 0.151523 I
AQ 3.2998369 se
RG 38.61
DW 62.400 use
DE 6.50 usec
TE 299.0 K
D1 2.00000000 se
TD0 1
======== CHANNEL f1
SFO1 400.1324008
NUC1 1H
P1 15.00 usec
PLW1 13.00000000
F2 - Processing parame
SI 65536
SF 400.1300174 M
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Account No. ct3144
WTZ EXTRA
9.104
8.880
8.187
8.183
8.175
8.171
7.606
7.601
7.588
7.583
6.988
6.975
6.970
6.957
3.920
OMe
N
N
N
N
1.02
2.05
1.02
1.00
1.02
1.01
3.04
ppm


Account No. cl3144
WTZ EXTRA
160.703
157.219
156.396
147.556
138.106
130.462
117.496
117.230
77.321
77.002
76.684
53.556
N
OMe
N
N
Current Data Parameters
NAME Dec06–2013–chem144
EXPNO 251
PROCNO 1
F2 – Acquisition Parameters
Date_ 20131206
Time 20.34
INSTRUM av400
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 32
DS 0
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976128 sec
RG 189.85
DW 19.800 usec
DE 6.50 usec
TE 299.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6243395 MHz
NUC1 13C
P1 10.00 usec
PLW1 52.00000000 W
======== CHANNEL f2 ========
SFO2 400.1324008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 13.00000000 W
PLW12 0.36111000 W
PLW13 0.29249999 W
F2 – Processing parameters
SI 32768
SF 100.6127810 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
220
200
180
160
140
120
100
80
60
40
20
ppm