

## **Marder Group Policies on Lab Operations**

*This document was updated on May 20, 2008.*

This document contains some policies for general operations for the Marder group. The key principle is to assume responsibility for the efficient operation of the group. Please do not try to cut corners and leave things for other people. It undermines a sense of cooperation, which is essential to a functioning and friendly lab.

Please be courteous of your lab mates and treat them with respect.  
Please be generous with your time if someone needs help.

You are required to read this document and sign the last sheet of this document, returning it to Marsha within a week of receiving this document, or before you begin working laboratory work if you are new to the lab.

If you have any questions about anything in the document, ask Seth, Steve, or Mariacristina.

### **Chemical Inventory (Chematix)**

When you begin working in the lab, you should learn how to use the Chematix chemical inventory. It is found at:

<https://www.chematix.gatech.edu/Chematix/>

The inventory is a record of all chemicals in the laboratories. No chemical is exempt. Each chemical container is identified by a unique barcode, and its contents, size and storage location are stored in the database. Every time a chemical is ordered it must be entered into the database, and every time a chemical is used up or moved to a different location this must be recorded.

It is a departmental requirement that the inventory be kept up to date, but also it is necessary for the smooth running of the laboratory that we know what chemicals we have and where they are.

### **Ordering**

When placing an order, a p-card should be obtained from Marsha. Even if you have a record of a group p-card number, which you should not be keeping anyway, Marsha should know about the order before you are placing it to make sure that there is enough money in the account and so she can decide which account you should use.

When placing an order for a chemical, you should first check the group inventory to make sure we do not have the chemical, as we don't want or need a large number of identical chemical containers taking up space in the laboratory. You should get a member of the group to countersign your order form to confirm that the order is necessary. If we have some of the chemical you need, but you feel it has decomposed or is otherwise not good enough for the reaction you want to do, it is your responsibility to dispose of it before ordering new material.

### **Use of Instruments**

All instruments should have a logbook. Users should sign the logbook when they begin using an instrument, not waiting until they are finished. This is necessary because if you are not next to the instrument and someone wants to know who is currently using it, they can find out from the logbook.

If someone from another group uses the instrument, the person should include their advisor's name as well as their own.

When someone uses an instrument, one of the people in charge of the instrument should train the person how to use the instrument, even if the person has used a similar instrument. This is necessary so that the person in charge of the instrument can know that the person using the instrument has received appropriate instruction on how to use the instrument.

You should also read the short instrument manual compiled by the group member in charge.

Any problems with the instrument should be immediately noted in the logbook and the person assigned to maintain that instrument should be informed.

The instrument should be left in a condition so that the next person can use it. Thus, you should clean up around the instrument and return setting to the "normal" setting if they exist.

### **Vacuum Pumps and Rotary Evaporation Units**

When an oil vacuum pump is used regularly, the oil should be changed approximately every 2 months. Consult Simon and Steve for instruction on using the pumps and schlenk line safely.

When you want to remove residual solvent from a material, your vacuum line should be used rather than a rotary evaporator pump. In addition to being a more effective vacuum than the rotary evaporator pumps, the oil vacuum pumps are less expensive and are not used by the entire group. Using rotary evaporator pumps for removing residual solvent

overuses the rotary evaporator pumps in a less effective manner than the oil vacuum pumps. Also, when removing a solvent with an oil vacuum pump, it is necessary to collect the solvent through a liquid nitrogen trap so that the solvent does not get into the vacuum pump, which destroys the seals over time. If you are unsure of how to use liquid nitrogen in a trap, ask Simon or Steve.

When you are finished using a rotary evaporator, the collection vessel should be emptied of solvent, and the bump trap should be cleaned and promptly returned to the rotary evaporation area.

### **Specific Guide to properly maintaining and using the rotovaps and house vacuums.**

#### Rotovaps:

Before you begin, check:

- 1) Is the chiller turned on, and at a suitably low temperature to begin? (around 0 degrees Celsius). If not, then wait until chiller temperature is low enough.
- 2) Are the collection flasks (both on the rotovap and on the vacuum) empty? If not, empty them into the halogenated waste. These should always be empty anyway, as you should always empty them upon finishing.
- 3) Is there enough water in the rotovap bath? If not, then put some in. The accuracy of the temperature on the rotovap baths is dependent upon there being a suitable amount of water in them (about half full). If the water bath is still hot from a previous user, make sure the temperature is not too high for your low boiling point solvent, like ether.

#### Rotovapping:

- 1) A bump trap should always be used, in case your solvent bumps.
- 2) Different solvents require different amounts of vacuum and heat to ensure effective evaporation. Do not pump down to 80 torr for dichloromethane – this will result in bumping of the solvent. Likewise, if you try to evaporate toluene at 400 torr, it will not work. (Try to start at low vacuum level (large P value on the screen), then adjust the pressure or heat as needed)
- 3) Although it is understandable that you want a fast rate of evaporation of your solvent into the rotovap collection flask, it should not be so fast that solvent is making its way through the vacuum and into the vacuum collection flask. If you see this happening, please adjust the pressure and/or heat accordingly to stop this. Passing solvents through the pump will decrease the lifetime of the diaphragms.
- 4) If you want to pump down as low as you can go, to perhaps drive off the last bit of solvent before you take an NMR or put your compound onto hi-vacuum, please empty the rotovap and vacuum collection flasks FIRST. If there is solvent in the rotovap collection flask, and you attempt to pump down to low, it will result in solvent being pulled through the pump.

When you are done removing solvent:

- 1) Press VENT on the vacuum pump and open the valve on the rotovap to allow air to enter. Once the pressure is ambient, stop spinning your flask and remove your round bottom flask.
- 2) Empty both the collection flask on the rotovap and the collection flask on the vacuum pump into the appropriate waste containers.
- 3) Turn off the heating on the water bath if you used it.
- 4) Turn off the pump if you are done rotovapping and no one is using the rotovap adjacent.
- 5) Make sure ALL the pumps, water bath heaters, and chillers are off if you are the last one to leave.

#### GENERAL TIPS AND ADVICE

- 1) DO NOT attempt to evaporate strong acids ( $\text{HCl}$ ,  $\text{H}_2\text{SO}_4$ ) or caustic and corrosive chemicals (i.e.  $\text{Br}_2$ ) on the rotovap. This is non-negotiable. If you are unsure about what you are trying to evaporate, then please ask whoever is in charge of the rotovaps.
- 2) If you are attempting to rotovap using a 1 liter or 2 liter flask: This can be done successfully, but please follow these guidelines:
  - a. Be careful about the weight. The rotovaps can handle these sizes of flask, but the round bottom flask MUST be appropriately buoyed in the water bath (even if you do not want to heat it). This will relieve the weight on the vapor tube on the rotovap. If the round bottom is not appropriately buoyed, you run the risk of cracking or snapping the vapor tube as the weight will be too much. **Some of these vapor tubes are irreplaceable!!**
  - b. Watch the collection flask carefully. Most, if not all, of the rotovap collection flasks can hold 750 mL of solvent. If you are rotovapping a lot of solvent, please check the collection flask from time to time and empty it when necessary.
- 3) Most of the pumps are rated to a pressure of 9 torr. This means that theoretically they can pull down this low. In reality, due to the number of connections, the age of some of the rotovaps, etc., this is not achievable. However, most of the rotovaps should pull down to ~30-40 torr. If you put on a flask and can only get down to ~50-60 torr, this does not mean there is something wrong! Depending on the fits of the bump trap, round bottom you are using, and size of the round bottom you are using, it may not be always possible to achieve the operational pressure. HOWEVER, if you are consistently, with various flasks, only are able to achieve pressures of 80-90 torr, then there probably is a problem, and you should notify someone in charge.
- 4) Do not touch anything on the chillers except for the on/off button. If they are not getting as cold as you are used to seeing them get, do not attempt to adjust the settings or fix them yourself. Inform one of the people in charge.
- 5) If you see something wrong with the rotovap setup (i.e., vacuum louder than usual, vapor tube is cracked/broken, coolant is leaking from the cooling lines) DO

NOT USE THE ROTOVAP/VACUUM. Inform one of the people in charge ASAP. If you can not find anyone who is in charge, then take the responsibility and put up a note telling people not to use that specific rotovap/pump.

- 6) Do not attempt to fix anything yourself.

House vacuums:

Use:

- 1) When you want to use the house vacuum, go over to the cabinet it is stored in and flip the switch on.
- 2) Attach what you want to pull a vacuum on the appropriate hose in your hood and turn the valve to use the vacuum.
- 3) Do not allow the vacuum to act upon standing amounts of low-boiling / volatile solvents!!! What do I mean by this? For example, what if you are recrystallizing a compound in dichloromethane? You then set up the filter flask, turn on the vacuum and pour your solution through the filter paper. In most cases the dichloromethane is going to be pulled through rather quickly. Do not just leave and come back in an hour! Once most of the solvent is pulled through into the filter flask, take the vacuum hose off and empty the flask. Then reattach it and allow it to pump further on your compound. This takes very little time to do and will save the pump from pumping too much solvent through it.  
If you really need to filter some volatile solvent which goes through slowly, try to use a preliminary trap in a cold bath (ice bath or dry ice / IPA bath) to try to catch most of the solvent before it reaches the pump.
- 4) The pumps have an outlet to a pipe that provides suction so as the solvents that do get pulled through to not get emptied into the air. You can see these white pipes at the end of the bench. You will see a piece of tubing going from the outlet on the pump to one of these pipes. If you see solvent getting sucked up through this hose then this probably means that somebody did not follow guideline #3. Do not just walk away and think it is not your problem. You use these pumps too. See who is using the pump and inform them of this.

General guidelines to house vacuums:

- 1) When you are done with the pump, turn it off. If you plan on using it in the next 15 minutes, it is ok to leave it on. However, under no circumstances should the pump be left on over night!! Make sure the last person who leaves double checks to verify this.

## Computers and Offices

If a computer was purchased by group funds, it is a **group** computer, even if it is sitting on your desk, and you are expected to share the computer with other group members if they need to use it.

No personal software or illegal software should be put on the computers.

Files should be backed up frequently, and you can do this using the group server.

All group computers should have a password required for login and should have a user login for other members than yourself.

It is recommended that laptop computers be secured to your desk by a lock to prevent theft.

When no one is in an office, the office should be closed **and locked at all times**. Thus, if you are the last person out, lock the door always.

### **Group Meetings and Monthly Reports**

Everyone is expected to attend group meeting unless they are sick or are out of town. If you are going to miss a group meeting, you must have permission from Seth.

When you are presenting at group meetings, bring your lab notebook and relevant characterization data (NMR, MS, EA, etc.) in case there are questions about your procedure or characterization data.

A Powerpoint and a pdf version of your group meeting (as well as your monthly reports) should be put on the group server after you have given a group presentation.

All Chemdraw files should also be included separately of the report files.

The group has software for Adobe Acrobat Reader 6.0 if your computer does not already have the ability to convert Powerpoint and Word files.

A hard copy of your monthly report should be turned in to Marsha along with photocopies of your lab notebook for the time frame covered by your report. Marsha can tell you when your report's due date is.

You should be starting your semiannual reports on a timely basis, and they should include a clear plan of the work you expect to accomplish over the next six months.

### **Short-term Plan for Literature Meetings**

For your responsibility in literature meetings, it is your job to read the most recent issue of the journals you have been assigned before the first meeting. For every article that you deem relevant to research in the group (acknowledging that the judgement will be subjective from person to person), Seth suggests that you should present the article in 3

slides, trying to keep the presentation of your articles to 20 minutes in length without questions.

For the second set of meetings, you are responsible for reading any issues of the journal that have been published since your last presentation. Keep in mind that some journals will not have issues every month, while others will have multiple issues per month (i.e. JACS). Also, it is possible that you will see no articles that you deem relevant to our group's research or multiple articles within the same journal.

We suggest that you pick a few articles to present, but the choice is up to you. If you have so many articles that you think you cannot keep your presentation to 20 minutes, add a slide at the end with a list of citations with titles that you think the group might find interesting.

When you have presented, you should put your presentation on the group server in a folder yet to be created, in powerpoint and pdf. We will also try to have some sort of endnote setup so that you can add your files using endnote.

Graduate students are expected to participate in literature meetings, held on Fridays at 1:00 p.m. Postdocs are invited but not required to attend.

## **Marderizations**

1) When you come into Seth's office for Marderizations, be prepared to show your data for all experiments that work and DON'T work.

If things don't work, ask yourself:

- a) did I check the literature to see if the compounds are known, have I followed a literature prep?
- b) did I ensure that **all** materials (reagents, solvents) are pure (dry, oxygen free) if needed?
- c) if a reaction did not give the expected result did I attempt to figure out why the reaction did not work-- what did happen, was starting materials consumed, did I work it up correctly, did I form a lot of something unexpected, if so what is it? Please don't just say the reaction didn't work, without given explicit observations.

YOU KNOW that Steve, Simon and Seth WILL ask you these things so you may as well expect that each time and be prepared to answer these questions.

When you explain things, be clear in describing the reaction on which you are working. If you are doing something new, expect to explain why you have chosen to do it.

2) For all new people in the in the group and when you start a new project, you should present to Seth a folder, with at least 25 of the most relevant article to your work, with for each article, a one page summary of its the key points

3) For each person in the group, you should identify at least one article per week that is pertinent to your work and we will create folders on the server by topic. Put a pdf file of that article on the server and give Seth a copy of the article with a hand written summary of the key points.

### **Traveling to Meetings (grad students & postdocs)**

Each member of the group will have access to a travel budget of \$1300/yr for attending a conference. Students who have complete their oral exam, can "borrow" against the following years travel allowance if a meeting costs more than \$1300. Student and postdocs can also bank up to 1 year of travel money starting now.

Before traveling (either on vacation or official university business), please fill out a Travel Info. Sheet, available from the group website, and give it to Marsha so that she can fill out a Travel Authority Request before you travel.

### **Travel to Other Institutions**

Travel grants are available for traveling within STC institutions. Talk to Seth for request for permission and funds for traveling. Travel bookings should be taken care of at least ten days prior to traveling.

### **Common Equipment**

All drawers and cabinets should be labeled as to their contents, whether they are personal or common drawers.

All personal drawers should include a label with the person's initials so it is known that the drawer is personal. All public drawers should be labeled as public by having a letter that corresponds to the bench (A – R) and a number (1,2,3, etc.). There is a list of drawers and their contents posted outside of each lab.

Certain equipment should be kept in common drawers for the group to use. Common equipment includes that listed below but applies to any glassware that you don't use on a regular basis:

- thermometers
- distillation equipment
- soxhlet extractors
- addition funnels
- mortars and pestles
- volumetric flasks

- UV-Vis and fluorescence cells
- column adaptors (you should have only one of each type at most)
- bump traps
- schlenk flasks that you don't use **weekly** at least
- solvent storage schlenks
- Dean-Stark traps
- pressure vessels
- stoppers (you should have one of each type at most)
- Glass syringes
- scintered glass funnels (also called fritted funnels, you can have one of each type at most)
- buchner funnels (you can have one of each type at most)
- Needles and cannulas
- Burettes
- Columns
- Flashlights
- Condensers (you should have two at most)
- Tools
- Dark glassware
- Weird glassware that you don't know how to use
- pH paper (we don't keep enough around for everyone to keep it in their own drawers)
- filter paper – keep in a communal area in the labs so we don't have duplicates!
- Glass and Teflon taps (unless it is in a piece of glassware)

Any common equipment that is found in personal drawers will be removed from the personal drawers.

When you are finished using glassware such as columns, round bottom flasks, reflux condensers, distillation apparatus, the glassware should be cleaned immediately so that other group members can use it. This action helps minimize the amount of glassware needed for the group to function.

Only two heating stir plates should be kept in each hood unless you are actively using more than two heating stir plates for heating AND stirring. Otherwise, extra heating stir plates should be kept in a common area.

### **Group Cleaning Up**

1. Group cleanup is from 2 to 4 p.m. on Wednesday afternoon (see the schedule).  
Make sure to schedule your time to be there for it.
2. People working in each lab room work as a team, and everybody takes a turn to be the team leader. The leader has the authority to organize and supervise the group

cleanup, especially the common area and the instrumental rooms. (See the schedule, feel free to exchange if needed, and inform us.)

3. All broken glassware is supposed to put at Xuan's bench, and all broken electronics at Yadong's bench. We will send them to the glass shop and electrical shop.
4. Inspection starts at 4pm by Xuan and Yadong. The inspection report is sent out to all group members by email on Thursday morning. Open discussions are welcome in the group meeting.
5. The inspection standard is the same as last time as following, which is subject to adjust. All suggestions are welcome.

For the personal area, we expect people to clean glassware, and put them in their homes based on the directory made by Xuan and Yanrong; clean your own bench and hood area; only keep maximum five commercial starting materials, and return the rest to the group storage; store and label properly all your home-made chemicals (i.e. remove them from round-bottom flasks and put into vials where feasible); keep no more than two heating stir plates per hood unless actively using them; keep the common draws under your bench organized. For the common area, we expect to see the clean balance area, the organized supply shelf, and clean evaporator bench.

## **Ordering**

When you finish a bottle of common solvent, it is your responsibility to replace the solvent so that it is available for others to use. Each main side of the lab has a method of organization that should be posted on the solvent cabinets. Please follow these instructions for solvent use and replacement.

When placing an order, a p-card should be obtained from Marsha. Even if you have a record of a group p-card number, which you should not be keeping anyway, Marsha should know about the order before you are placing it to make sure that there is enough money in the account and so she can decide which account you should use.

When placing an order for a chemical, you should first check the group inventory to make sure we do not have the chemical.

When a chemical arrives, it should be put in the inventory, which is located on a website at [www.chematix.gatech.edu](http://www.chematix.gatech.edu). As of March 3, 2006, Reddy is the only one who can add and subtract people from this system. Reddy and Xuan will show you how to use this system. The Chematix system should accurately reflect the location of chemicals in the lab. If you are going to use a chemical for more than 20 minutes, you should change the

location of the chemical in the Chematix system. Each chemical bottle has a unique barcode and should be up to date in its status.

## **The Laboratory Notebook**

*It is:*

A legal document that:

- Establishes date of inventions
- Establishes that you actually did the work; if it is not in the notebook it didn't happen

The primary place people in the future can go to reproduce your work

It is written for someone else to read and understand.

- That person could be a student, or in fact in patent litigation cases an attorney, judge or even jury.

*Basics*

It should be:

- Bound
- Consecutively numbered
- Have high quality paper
- Have your name on the front.
- Have entries that are written in pen that will not smear or dissolve in solvents
  - ▶ Never erase or "white out" things in a lab notebook
  - ▶ Errors should have line drawn through them and the correct entry should be shown below it

It is the property of your institution and you should not keep lab notebooks at home.

*Procedures*

In general, it should not leave the building (copies can)

It should provide a summary of what the experiment is trying to achieve

It should be signed, read and witnessed every day by another person, not directly involved in your project

- Remember it is a legal document
  - ▶ Establishes date of inventions
  - ▶ Establishes that you actually did the work; if it is not in the notebook it didn't happen

It should not have slang, jargon, or short-hand that is not easily recognized by someone completely unfamiliar to your work

It should be complete

### Repeated Procedures

If you follow a literature procedure give the complete reference.

Regardless of whether it is in the literature or not, the first time you do it write down everything you have done.

- Be sure to explicitly point out deviations from the published procedure

If you repeat the procedure, just refer to the name of the procedure and the appropriate page(s) of your notebook: however, note all amounts, yields, and changes in procedure.

### *Important "Do's"*

First thing every day write in the date

- If you have multiple experiments over multiple days always give the date.

Use continuation pages

- give the page number things are continued on and the page number they were continued from

Void all blank pages clearly

Always record the information directly into the notebook, not on loose paper, not into a computer

Include calculations as appropriate

Keep a photocopy of notebook pages outside the lab

When in doubt write it down- it may not seem important now but could later

Create table of contents

Include all observations,

Limit opinion and conclusions

- For example avoid phrases such as --- the reaction failed, the experiment didn't work.

### *Inventorship*

In the US if two sets of people claim inventorship, the patent is awarded to the first to invent, which is determined in complex legal proceedings

If in examining a patent a reference is cited against a patent application by the patent examiner, an applicant can swear back to the date of invention to demonstrate it preceded the reference

- Your notebook is the key piece of evidence to support this date.
- For it to have it full legal value it should be read and by another person not directly involved in the research. This person should indicated that they have read and understood the entries and should sign and date it

### *Data*

The notebook should show a person where to go to find primary data

Data should be kept in a separate folder or notebook, with location noted in the book

All loose data should be clearly labeled with a notebook ID# and a date

Keep all data whether you think it is good, bad, important or unimportant

### *Further Reading:*

Writing the Laboratory Notebook: by Howard M. Kanare, published by the American Chemical, Society, Washington DC

**Appendix 1: Example of a Laboratory Notebook page.**

February, 28, 2003



	amine	K <sub>2</sub> CO <sub>3</sub>	4-bromobutyrate	DMF	Theo.product
equivalents	1	1.1	1.1		1
grams/mol	193.25	138.21	195.06		307.38
grams	5.35	4.15	5.85		8.50
mmol	27.68	30	30		27.68
mL			4.29	50	
Density			1.363		

*Risks:* starting amine) unknown, treat as toxic

Potassium carbonate) harmful if swallowed, irritating to eyes, respiratory system, and skin

4-bromobutyric acid ethyl ester) irritant to eyes, respiratory system, and skin; combustible

dimethylformamide) toxic, may be toxic to fetus, harmful by inhalation and skin contact, irritant to eyes and skin, combustible, readily adsorbed through skin

*Plan:* Dissolve the starting material in DMF under N<sub>2</sub>, then add bromide and base. Heat to 100 °C overnight. After cooling, if the reaction appears complete by TLC, pour the reaction into ice water, then extract the product with ether three times, washing with water. Dry with MgSO<sub>4</sub>, filter, and concentrate. Possibly further purify by chromatography and / or recrystallization.

*Procedure:*

February 28, 2003

3:05 PM Dissolved the phenol in 50 mL of DMF sparged with N<sub>2</sub>.3:14 PM Added K<sub>2</sub>CO<sub>3</sub> and the bromide and let stir.

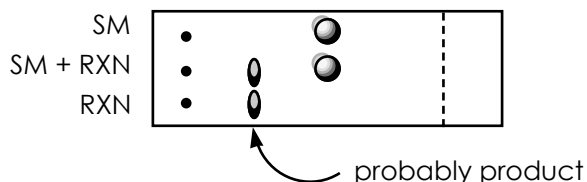
3:30 PM Submerged the reaction flask in an oil bath and began heating to 100 °C

4:13 PM: The reaction was at 100 °C.

### March 1, 2003

6:20 AM Took the reaction out of the bath.

8:16 AM: TLC of the reaction (silica, eluent = 20 % EtOAc in hexanes):



8:30 AM Poured the reaction into a sep funnel with 100 mL of ether and 50 mL of ice H<sub>2</sub>O. The reaction flask was washed with 25 mL of ether 3 times and 50 mL of H<sub>2</sub>O once. The ethereal layer was extracted with ice H<sub>2</sub>O (50 mL x 6), dried over MgSO<sub>4</sub> and filtered. The solvent was rotovapped off to give a yellow oil.

4:00 PM Let the oil stand overnight JD-1-1A

### March 2, 2000

12:00 PM The crude material was chromatographed (silica gel, eluent = 20 % ethyl acetate in hexanes to 40% EtOAc in hexanes in 10% increments of EtOAc). When TLC indicated the material was coming off the fractions were collected (4 x 150 mL). The solvent was removed and the liquid was put under vacuum ( $5 \times 10^2$  torr) overnight. JD-1-1B

### March 3, 2003

4:30 PM The flask was taken off the line, capped, and put in the back of the hood.

### March 7, 2003

<sup>1</sup>H NMR labeled JD-1-2A taken on sample JD-I-1B, consistent with product.

An example of an experimental section:

**4-(*N,N*-diethylamino)-2-(ethyl butanoyloxy)benzaldehyde (5):** A mixture of 5.35 g (27.7 mmol) of 4-*N,N*-diethylamino-2-hydroxybenzaldehyde, 5.85 g (40.0 mmol) of ethyl 4-bromobutyrate, and 4.15 g (30.0 mmol) of K<sub>2</sub>CO<sub>3</sub> in 50 mL of DMF was stirred for 18 h at 100° C. The reaction was allowed to cool to room temperature and

poured into a separatory funnel containing 100 mL of ether and 50 mL of iced H<sub>2</sub>O. The organic phase was washed with iced H<sub>2</sub>O (50 mL × 5), dried over MgSO<sub>4</sub>, and filtered. Evaporation of the solvent from the filtrate gave a yellow oil that was chromatographed (silica gel; eluent = 20% EtOAc/hexanes to 40% EtOAc/hexanes) to give 7.31 g (86%) of **5** as a yellow oil. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ (ppm) 10.14 (s, 1H), 7.68 (d, *J* = 9 Hz, 1H), 6.25 (dd, *J* = 9 Hz, *J* = 2 Hz, 1H), 6.00 (d, *J* = 2 Hz, 1H), 4.12 (q, *J* = 7 Hz, 3H), 4.07 (t, *J* = 6 Hz, 2H), 3.40 (q, *J* = 8 Hz, 2H), 2.53 (t, *J* = 8 Hz, 2H), 2.15 (qn, *J* = 8 Hz, 2H), 1.24 (t, *J* = 7 Hz, 3H), 1.19 (t, *J* = 7 Hz, 3H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ (ppm) 186.61, 172.94, 163.39, 153.74, 130.04, 114.04, 104.26, 93.01, 66.69, 60.34, 44.65 (2), 30.50, 24.35, 14.10, 12.48 (2); Anal. Calcd for C<sub>17</sub>H<sub>25</sub>NO<sub>4</sub>: C, 66.43; H, 8.20; N, 4.56. Found C, 66.49; H, 8.12; N, 4.50.

Examples of additional characterization:

IR (KBr, thin film)  $\nu_{\max}$ : 2017, 2953 (s, OH), 2855 (s), 2192, 1512, 1360, 1082, 887(w) cm<sup>-1</sup>.

GC-MS *m/z* (% relative intensity, ion): 415 (9, M + 4), 413 (32, M + 2), 411 (23, M<sup>+</sup>), 311 (35), 301 (95), 215 (20).

HRMS-FAB (*m/z*): [M + H]<sup>+</sup> calcd for C<sub>21</sub>H<sub>38</sub>N<sub>4</sub>O<sub>6</sub>S, 475.259; found, 475.256.

MS-MALDI (*m/z*): [M + 2H]<sup>+</sup> calcd for C<sub>204</sub>H<sub>210</sub>N<sub>6</sub>O<sub>18</sub>: 3035.90; found, 3035.2.

EIMS (70 eV) *m/z*: M<sup>+</sup> 420 (15), 241 (15), 201 (59), 135 (14), 69 (23).

mp 175.5 °C (lit.<sup>25</sup> mp 175-176 °C)

bp 127 °C

UV (hexanes)  $\lambda_{\max}$ , nm ( $\epsilon$ ) 250 (1070), 224 (1900).

GPC (solvent): Mw = x, Mn = x, PDI = x.

### Compound Characterization Form

For ease in organization of your new compounds, please organize all compounds using the following data sheet, which is separately on the group website in Word and pdf file. Having this data in one place will help know what data is needed for complete characterization for publishing compounds.

**Compound:**

**Literature References:**

**Notebook page numbers:**

**Yields:**

**Appearance:**

**Formula:**

**Molecular Mass:**

**Mass Spec:**

**<sup>1</sup>H NMR:** (solvent:     ;     MHz )

**File Name:**

**<sup>13</sup>C NMR:** (solvent:     ;     MHz )

**File Name**

**UV:** (solvent:     )

**File Name:**

**IR:**

**Melting Point/Boiling Point:**

**R<sub>f</sub>:** (solvent) R<sub>f</sub>

**Elemental Analysis:**

*Calculated*

*Found*

$\Delta$

C:

H:

N:

I have read and understood the Marder Group Policy on Lab Operations document.

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Printed Name	Signed Name	Date
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