Guidelines for Research
Tour Group 2003
Rice University

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I. Group Organization and General Information

Welcome to James M. Tour’s group at Rice. This information is provided to help you get acquainted with the group’s procedures so that you can get quickly started with your research.

General: The group's labs are in Dell Butcher Hall (DBH) rooms 260 (phone x6249) and 300B (x6251). The group computers are in DBH 300A. See Jake Ciszek, the group's IT person, to get logged onto the group computers. The computer programmers occupy room 243 (x2454). The Tour Group Lab Manager is Dr. Dustin James (DBH 255, x6247). The Group’s Safety Officer is Austen Flatt (DBH 300B, x6251). The Chemistry Department Safety Coordinator is Rose Gray-Dye, (Space Science (SS) Room 216, x6236). You may bring your own computer to the lab and obtain web access by getting an IP address assigned to you through Dustin or by contacting Dr. Problem (problem@rice.edu) or by using the Rice IT web page request form.

The Chemistry Department Coordinator is Diana Gomez (SS 203, x3277), in the Chemistry Office. Diana does the paper work so that you can get paid each month. Diana will also have the different keys that you will need to get into the labs. Diana also maintains petty cash for reimbursement of expenses when you buy materials (with prior approval) for the lab. Leah Benard-Boggs is the Sr. Department Administrator (SS 207, x5850).

The following is a list of basic operating principles that are important. Please follow them to the best of your ability. Gross disregard for these guidelines are cause for dismissal from the group (see dismissal policy in section II).

A. Maintain security. Please close and lock all doors when you are the last to leave.

B. Please conserve our resources. Turn off nitrogen valves when they are not being used, and when they are; please use the lowest flow rate possible. When you leave at night, turn off your lights and pull the hood sash down.

C. Supplies can be purchased from the Chemistry Stock room on the second floor of the Space Science Building. Keep track of how much you spend. You will be asked to
periodically review spending reports. Be frugal with glassware purchases; all glassware purchases should be pre-approved by Dr. Tour. Catalogs are available in the office, Room 255. Tour Group purchase order forms are available from the Group's Office Assistant (JR Valdez, Room 255, x6248). Fill out a form with a short explanation of why you want the material. If ordering a chemical have Francisco (Pancho) Maya, the group's inventory control person, initial the appropriate spaces. Place the request in Dr. Tour's in-box. After approval from Dr. Tour, the JR or Dustin will place the order.

D. Experiments must be thoroughly documented in a hardbound laboratory notebook, in ink, at the time of completion. Each notebook page should be dated. More information is provided in section IV on lab notebooks and data collection. Office supplies such as lab notebooks, pads, paper, pens, and the like are available in the office. Computer diskettes and zip disks are also available. The chemists will need to start a file system with a manila folder for each compound made or experiment run. Your initials followed by the notebook number and the page number designate compounds and experiments. The first notebook you use is #I. So "JMT-I-1" means the first page of James M. Tour's first notebook. Each manila folder will contain all the spectra recorded for that compound. If more than one compound is made on each page, each compound can be indicated by a letter, such as "JMT-I-1A".

E. Computer programmers should use commonly agreed-upon conventions for naming variables in their programs, and should insert comments into their code to indicate the conventions used. Programs written should have a level of documentation and comments such that projects can be taken up and understood by new group members without having to re-write the entire set of code. Back-ups should be made of source code and compiled programs as appropriate.

F. All programs placed on computers should be licensed and paid for appropriately. Group computers should never be used for computer games. Never!

G. All group members write six-month reports that contain written summaries of their work, plus experimentals for the chemists. The experimentals will contain all spectral information, as if you were writing for J. Am. Chem. Soc. See part J below for information about less formal weekly reports for the subgroup meetings. The six-month reports can be written using the software on the group computers.

H. Spectral data are to be cross-referenced to the corresponding notebook and labeled with appropriate structural and data collection information. All spectra for an experiment should be in one file folder with the same JMT-I-1 number.

I. Products from reactions, homemade reagents, and intermediates should be stored in vials, bottles, or flasks (if storage is temporary), which are labeled with an appropriate structure and notebook cross-reference. Anything that will be "on the shelf” overnight must be labeled. No storage in round bottom flasks or NMR tubes is permitted.

J. The chemists' group meeting is each Tuesday from 3:00 to 5:30 p.m. The labs are divided into halves, and each half presents their work on the board every fourth week. At
the end of the meeting, one person presents a problem set from the recent literature, with responsibility rotating through the group. That person is also responsible for bringing refreshments to the meeting (sodas, cookies, chips, etc.). The rotation list is posted in each of the labs and is maintained by Stephanie Chanteau.

There are subgroup meetings every week. Each group member submits a hand-written or word-processed summary of the work they have done in the last week, on a clean sheet of paper. You should list every reaction, with the notebook number and yield that you carried out that past week. Dr. Tour gets a copy for the group files. The computer programmers meet every Thursday from 3:00 to 5:00 p.m. The chemists meet each Friday at about 3:00 p.m., with each sub-group, such as molecular electronics, nanotrucks, nanotubes, etc. meeting separately with Dr. Tour. The secretary posts a schedule of subgroup meeting order on the bulletin board outside the office.

K. Your attendance at all departmental seminars in the field of Organic Chemistry is mandatory and attendance at seminars in other fields is highly encouraged.

L. You should plan to spend a minimum of three uninterrupted hours per week reading the current chemical literature.

M. The group's Safety Officer is Austen Flatt. He will give you forms to read and sign. Wear safety glasses in the labs at all times. Lab coats are also required. All water lines for cooling must be secured with a double-wrap of metal wire to ensure steadfastness. Keep the flow at a moderately low rate so as not to burst a line.

**Lab Sign Out Procedure**

If you are the last person out of the lab at night, before leaving the laboratory make sure that—

- All N₂ Bubblers have a flow rate of 1 bubble per second or less
- All of the labs hood sashes are totally closed
- All water lines are secured with wires
- All faucets are turned off (except for reaction in progress)
- All variacs are on cork rings to prevent shorts if a water leak arises
- All stills are turned off (THF is turned to low)
- All lights are turned off
- All doors are locked (including instrument and computer rooms)

Blast shields are available. Always use them for potentially exothermic reactions. Always keep your hood sash down.

**Nitrations are a special case. The Tour Lab policy is:**

1. The first time you nitrate a new compound, do it on less than 1g.
2. Run multiple times at 1g scale before scaling up.
3. 10g is the absolute upper limit.
4. Use blast shield(!), safety glasses and safety mask, and green apron when running nitrations and working them up.
5. When drying a nitrated aromatic, ensure that this takes place in a hood behind a bast shield. Often, explosions occur on drying. Use them as “wet” compounds if at all possible.

N. Group Responsibilities: the senior members of the group will assign you a group job. The procedures are completing the different jobs that are posted on the web page http://www.ruf.rice.edu/~kekule/grouppage1.htm. Please be responsible and help the group achieve what it needs to achieve by completing your job as needed. The laboratory must be kept clean. It is difficult to do good work on surfaces that are filthy and cluttered. A thorough laboratory clean up will take place three times a year.

O. Office hours: Dr. Tour has an open door policy. Generally, you are welcome to talk to Dr. Tour at any time, just knock on his door if it is closed. If he is available, he will ask you to come in.

P. Come to the laboratory to do chemistry. If you want to read in the laboratory, pick up a textbook or scientific journal and learn something new. If you want to read newspapers or novels, do this on your lunch break in a designated eating area. There is no eating or drinking in the lab.

Q. External activities (athletic activities, laundry, shopping) should not interfere with your research.

II. Work Ethic

Graduate Students: Organic synthesis, computer science, and other forms of scientific research and study require a strong work ethic, which means spending much time and effort reading and working in the lab or at the computer. As you proceed through your career you will find that most good scientists are diligent workers with tenacity, fortitude, curiosity, and common sense. Some people have a bit of luck thrown to them, but they have prepared themselves for luck by becoming knowledgeable and observant. It is called “serendipity.”

You would not be here if you did not have success in your field as your personal goal. Since it is my personal goal, and the overall goal of Rice University, to produce high quality scientists who can make significant contributions to their fields as well as humanity, I expect the students training in my group to work long hours and to show considerable progress in attaining the targets set before them. In the pages following are some expectations and guidelines for the day-to-day conduct of research that I believe, based on my experience, will help you achieve all of our goals.

Failure to adhere to the three guidelines listed immediately below may result in written notification of my dissatisfaction. Accumulation of such notices can result in permanent dismissal from the group. Repeated infractions of other guidelines, or failure to make
corrections to inappropriate behavior or technique may also result in written notifications.

1. Sixty (60) laboratory/library hours minimum per week are required. This requirement should be met, as much as possible, during the normal daytime hours of the department.

2. At the very minimum, an average productivity level of two complete reactions per workday should be maintained. Complete means the isolation and initial characterization of all reaction products.

3. Sexual Harassment—See the web-posted Rice Policy No. 830-01 that we will follow precisely.

III. Vacations

1. I don't monitor vacation days, and I don't expect that it should be necessary. But send me an email prior to your departure, which notes the days you will be gone, and an emergency contact phone number where you can be reached.

2. You may spend one day per week outside the laboratory. It is my hope that you will spend this time resting and being refreshed.

3. In terms of extended vacation, you are permitted 14 days plus Christmas and Thanksgiving per year. Prior to your departure you should notify me or Dustin of your plans and give emergency phone numbers to JR.

4. Personal emergencies and illness are understandable and a completely different matter.

IV. Laboratory Notebooks and Data Collection for Chemists

Listed below are the most important guidelines to follow in documenting your research in a laboratory notebook. An example experimental procedure is found in the Appendix.

1. Use only the hardbound notebooks from the stockroom or the office.

2. Leave sufficient room at the beginning of the notebook to include an index of experiments done. When you complete a notebook, fill in the index before you start another notebook. Ideally, you should be filling in the index as you go.

3. Write only in ink and date each page. If you mark something out, initial and date the mark-out. Never completely obscure or remove such mistakes. When an experiment or page is complete, sign your name and note the date at the bottom of the page. Such information can be useful when applying for patents or for proving priority in publications. For new reactions or potential inventions, have another person who is capable of understanding the work sign as a witness.
4. Do not leave blank pages in your lab notebook. Use the pages in order, and if you have to continue an experiment on a non-sequential page, note “continued on page xx” and “continued from xx” on the appropriate pages. If you inadvertently leave a blank page in your notebook, make a large “X” across the page, and sign and date it. If you leave half a page blank, it is also useful to cross it out so no further additions can be made. These are good habits to develop in graduate school because industry, especially the pharmaceutical industry, requires this type of diligence (and more!) in maintaining a notebook. Your future job could depend on how well you keep your notebook.

5. At the top of the page of each new experiment write an equation with starting materials on the left, expected product(s) on the right, and reagents and experimental conditions over and under the forward arrow in the middle. It is usually best to write structures of organic materials since names can be long and difficult to write. Under each chemical write the molecular formula, formula weight, density, molarity, molality, concentration, mmol, equivalents, and/or other useful data that will enable you or someone else to know what is going on at a glance.

6. It is important to note color changes, formation of precipitants, gas evolution, exotherms or endotherms, or any other observations, and write them down in your notebook. This information can be critical both in scaling up a reaction, and in figuring out what happened if something goes wrong. The information can also be very helpful if you want to repeat an experiment that produced unusual results (sometimes good results!).

7. In your description of the experiment, you should note at what time you added reagents and reactants, and how fast, i.e. drop-wise, portion-wise, all-at-once, via syringe pump, or whatever. Were gases added under the surface of the liquid or to the headspace? Were liquids added under the surface of the liquid or allowed to drip into the mixture? Was the mixture stirred by a magnetic stir bar or by a mechanical stirrer? Write this information in your notebook.

8. Record the quantity of solvent and calculated molarity of the reaction. If the solvent was distilled prior to use note this and the drying method, if any, used in the distillation. The source of all starting materials should be clearly noted with either a manufacturer’s name or a notebook cross-reference. When using a commercially produced chemical, it is useful to record the lot number. There have been many cases in which one lot number of a particular chemical performed differently than a second lot number of the same chemical.

9. Measure and record the temperature of both the heating or cooling bath and the interior of the reaction mixture if possible. Note whether you carried out the reaction under an inert atmosphere. Did you use oven-dried glassware?

10. When possible, all reactions are to be monitored by GC or TLC from time zero until the reaction is complete.

11. If you quench a reaction, note how you did it, i.e. poured onto ice, carefully added acid, sparged with nitrogen, etc. If there was foaming, exothermic activity, or other reaction...
on quenching, write it down in your notebook. How long did the foaming last? Was it
difficult to handle? Did anything spill out that will affect yield later?

12. When doing an extractive work-up, count the number of extractions and write down the
names and quantities of solvents used. It is more efficient to use small quantities of the
extraction solvent three times rather than a large quantity one or two times. This also
conserves expensive solvents.

13. Make note of the drying methods employed after work-up (e.g., brine wash, MgSO₄,
Na₂CO₃, Na₂SO₄ etc.) and how many cycles you went through. Did you filter using a
Buchner funnel, a fluted filter paper, through cotton (filtering through a wad of cotton
will often catch the last few droplets of water since they will gloom onto the very polar
cotton fibers), or what? Was the filtrate cloudy? It may not have been dried well
enough. Did you combine all the organic extracts into one, and then dry the whole, or did
you dry some extracts separately? What color were the extracts? What color was the
aqueous layer, if any? Was the aqueous layer cloudy or clear? A cloudy aqueous layer
could mean that separation of the layers after extractions was not as efficient as it could
have been.

14. If you purify by flash column or other method, record the type of chromatography used,
the eluant system, the column or prep plate size, and the quantity of silica gel or other
solid phase.

15. If you purify by crystallization, did you dissolve the solid product in hot solvent (at what
temperature?) and allow to cool, or did you precipitate the solid by trituration? When
you filtered the crystals did you wash them with clean solvent? Did you recover any
product from the mother liquor? Write it down.

16. If a compound is distilled or sublimed at reduced pressure, both the temperature and the
pressure should be noted. Also note the literature values and references if they are
known. Did you do a short path distillation, use a Vigreux or packed column, take
fractions, experience bumping, or set the apparatus such that you used a specific reflux
ratio? Write it down.

17. For each reaction, ensure that you have one spectral file folder that has the corresponding
reaction number on it and all the spectra therein.

18. Calculate a crude weight and obtain a ¹H NMR of the crude reaction mixture.

19. Following purification, calculate the yield and carry out initial characterization of all
products.

20. For repeat experiments you may list only the reagent/substrate data and pertinent
procedural changes. Cross-reference this notebook page to the most recent full
experimental that you are following. When changing conditions note the changes and
your reasons for making the changes.
21. When following a literature procedure, whether it is the exact procedure or simply a representative example, list the reference at the top of the page under the equation. Distinguish exact vs. representative procedures.

22. Keep representative HPLC and GC traces in the spectral file. For HPLC, take note of flow rate, solvent system, column type, column size, eluant system, type of elution (gradient or isocratic), and amount of compound introduced per injection. For GC, note injection volume and sample concentration, column type, and oven, injector and detector temperatures.

23. Each new compound that will appear in print (publication/thesis) must be fully characterized. The characterization data should be stored in a manila folder with a structural representation and checklist of acquired analytical data attached or written on the outside. For a compound to be completely characterized the folder must contain:

   a) Chemical Abstracts name and Chemical Abstracts registry number (if available).

   b) Experimental procedure (JACS format). See the attached representative experimental procedures. Follow this format very carefully. Pay special attention to the bracketing method for enclosing the moles and volumes of reagents, the protocol for reporting spectra, the use of General Experimental Procedures if you use the same reaction three times or more, the use of citation methods, etc. If a compound is known in the literature, you must provide a reference for that compound, and you need only obtain the proton NMR. If the specific compound is not known but you used another’s protocol, you must cite their protocol. If the compound is not known, you must minimally obtain FTIR, proton and carbon NMR, HRMS or combustion analysis.

   c) Tabulated data and a hard copy of $^1$H NMR, $^{13}$C NMR, IR, and MS spectral data, and C/H combustion analysis (if not possible, then HRMS data).

   d) Physical Properties: determine the optical rotation of chiral compounds and melting point of solids and the boiling point of distilled liquids or oils.

   e) X-ray structure (if obtained). This should include all data in hard copy form as well as a floppy disk containing the structural information in Chem 3D format.

V. Laboratory Safety

Police/Emergency Phone: x6000

See the Tour Group’s web page for the Chemical Hygiene Plan for more information on Lab Safety.
VI. Departure From The Group

Upon completion of your stay please do the following:

1. Be sure all spectral files are labeled with a notebook cross-reference and a structure. Put them in banker-type boxes and coordinate with Dustin to have them properly filed in our storage facility on South Main Street. Your notebooks will be kept in Dr. Tour’s office.

2. If you have not already done so, prepare characterization folders for all compounds *(vide supra)*.

3. Clean out your freezer space saving samples of all useful characterized intermediates. Place these in the plastic storage boxes, labeled with your name. Insert a chemical inventory list of the box’s contents, with the number of grams or mg for each compound, into the box itself. Give a copy of the complete inventory list of your compounds to Dr. Tour.

4. Large quantities of potentially useful intermediates should be put in brown bottles, labeled accordingly, stored in the appropriate freezer or shelf location, and added to the chemical inventory.

5. Put all computer files in one folder on the computer with your name, and provide them on a zip disk to Dr. Tour.

6. Return all keys to Dustin or Diana Gomez, and arrange for mail forwarding with the Human Resources office through paperwork you will fill out with Diana.

7. Wash and return all glassware to the proper storage location.

8. Return all chemicals to the proper shelf or freezer location and update the chemical inventory as needed.

9. Give electronic copy of thesis and reports to Dr. Tour.

10. Ensure a thesis copy is sent to Dr. Tour.

VII. Some Advice from Dr. Tour

1. **Technique:** Good laboratory skills are a very important part of the chemist’s career development. Even if you don’t end up working in the lab after you graduate, you may be directing others who are. It is easier to teach others when you’ve done it yourself. Organic synthesis involves complex tasks that must usually be done in a certain order. Plan each experiment carefully, especially if it is one you’ve never done before. When you plan carefully, and make as many observations as possible, the reason for a negative result can be more readily ascertained. **Plan your day properly.** Before leaving after a day’s work, make a list of reactions to be run on the following day, and get as far as
possible in preparing to run them. Then, when you enter the next morning, you can begin your work without delay.

2. **Non-research** time: Get out of the chemistry building in your free time. Learn something about art, literature, sports, religion or music. Get to know other people in the Rice community by participating in various campus activities. If you have a significant other, pay attention to them, let them know you are thankful for their support. A breath of fresh air and relaxation in the sunshine can often clear the way for a break-through in your thought process.

3. **Personal Hygiene:** Although not customary in all countries, Americans generally bathe at least several times per week. As a result, many Americans are offended by the infrequent bathing habits of others (whether Americans or internationals). Thus, you may be leaving a negative impression of yourself without ever knowing it. Unfortunately, bad impressions are often difficult to overcome. Likewise, be sure to use an underarm deodorant since most Americans find body odor to be most offensive. I have seen people causing themselves to be ostracized by others simply because of poor personal hygiene habits.

4. **Goals:** People are visual by nature. Set high, obtainable goals for yourself and visualize yourself achieving those goals. Athletes are commonly known for doing this, but it can work for scientists also. By visualization, you are forced to form a plan, and by planning, you ready yourself for each step in the process.

5. **Peers:** Gossip reflects poorly on you, and consumes time needlessly. Saying bad things about others to build yourself up yields negative results in the end, and people will tend to steer away from you. It is a surprisingly small world out there and you never know when you will run into a former lab partner or graduate schoolmate. You may be relying on them for your next job.

6. **Recommendations:** When you request that I write a recommendation letter on your behalf, the quality of my recommendation will be directly related to the quality and effort of your research. Minimal effort will result in a recommendation with minimal support. I won’t risk my reputation, and the reputation of other group members, by writing letters that do not accurately reflect a person’s skills, effort, knowledge, and results.

7. **These Guidelines:** These guidelines are designed to ensure that everyone knows what is expected and that the same things are expected of everyone. This is not a comprehensive outline of how to conduct oneself in graduate school. Always use common sense and remember, “do unto others as you would have them do unto you.” A corollary is “work hard for me and I will work hard for you.”

8. Parts of this information have been extracted from the group handbook produced by Prof. John L. Wood of Yale University.