A FEW TIPS ABOUT LAB REPORTS (CHEM 30121)

Do not forget to have the cover page attached to your report with your class ID# on it but **NOT** YOUR NAME. **No ID# or incorrect ID#: no credit!**

Reports must be typed (except for the drawings of setups or chemical structures) based on what was recorded in your notebook.

GENERAL (adapted from http://www.professorkshow.com/sample-organic.html):

- Never use the words "I" "we" "our" "my" "the student" "the experimenter" or any other word referring to you directly (this includes the use of "one" which should ALSO never be used). It makes your paper appear unprofessional. Instead of writing "I added 5.0g NaCl to 50mL water" just write "5.0g NaCl was added to 50mL water."
- Avoid the use of the word "it"- your goal is to be clear and concise.
- Samples are "prepared" and graphs are "generated" or "produced" in the lab- nothing is ever "created."
- Chemicals are weighed or their mass determined. Words like "massed" and "weighted" should never be used.
- "Mole(s)" is like "dozen(s)" in that it is a number. Thus, "the number of moles of chloride" or "the amount of chloride" can be determined, but statements such as "the moles of chloride" make no sense grammatically or chemically.
- Tests are not "done" and samples are not "determined."
- No contractions or colloquialisms in formal writing.
- Do not attempt to "B.S." your way through a lab report...if you do not understand the material, seek help!
- The confusing misuse of big words is not eloquent.
- Expressing yourself so that the reader "kinda gets what you mean" is not sufficient.
- NEVER use the phrase "in this experiment" or any equivalent thereof ANYWHERE in your report.
- Spelling and grammar errors are not acceptable.
- Verbs should not be used as adjectives in an effort to economize your writing. It just makes for awkward writing. For example, "the samples were obtained" is correct, while "the obtained samples" is incorrect.
- Are the words "previously prepared" or "aforementioned" in your report? Chances are they are unnecessary. Remove them.
- Sample calculations must use real data from the lab. If the units of the answer do not match the calculation, the math can not possibly be correct.
- Express numbers in correct scientific notation with superscripts (3.0×10^{-6} NOT 3.0E-6 or 3.0x10^-6).
- Justify everything. If you say one method is better than another due to cost, the dollars involved had better be cited. All error must be explained.
- Be specific.
- Redundancy is not good. This includes repetition both within and between sections of your report.
- "Within" is correctly used above. Samples are not "within" a solution. Precise and accurate scientific writing is required.
- Watch Out For Unnecessary Capitalization… Chemicals are not people and should not be capitalized. Be sure abbreviations are correct, too: mL, mol, etc.
- Assume the reader is an organic chemist, but knows nothing about your experiment. For example, write your report as if you are explaining your results to an organic chemistry student

at a different university. Do not make assumptions that they know what the melting point is supposed to be, or why you used the techniques you did. Explain everything.

- PROOF READ your work

LABORATORY REPORT

1) Introduction (write in advance). A few paragraph to half a page in length. This must be written in a general way. It is more about the general concept than the particular experiment you will be conducting. For example, in extraction/recrystallization the names of the chemicals you will be using should not be mentioned. Instead it is about the generality of the technique. What will the experiment be illustrating? The introduction is very general and more about the theory. You should definitely not be using "I" or "We" in this section.
2) Reaction, mechanism, side reactions (write in advance). This is also theoretical and the lab lecture and main lecture should be what helps you.
3) Reactants and Products table (write in advance). In every lab, you should have a table ("get to know the chemicals you will be working with"), which contains all the information you might need: name and structures of the chemicals, molecular weight, physical properties (bp, mp, density, concentration, etc.), **AS WELL AS SAFETY INFORMATION** (from MSDS = Material Safety Data Sheet). For instance, this is where you need to make a note that bromine should not be mixed with acetone, and also how to handle an accident if you come into contact with bromine. This table is also what you will need to calculate yields, and to expect a certain physical property (such as melting point). Nowadays, this might take 10 min of your time, while it used to take at least 60 min before. That does not mean Wikipedia should be your source of information – instead chemical manufacturers sites like Sigma-Aldrich, Alfa, etc. will have more reliable data and will also have MSDS files. You should also consult the CHEMICAL COMPATIBILITY CHART. Partial example of this section:

Chemicals	Formula	MW (g/mol)	Mp (^{0}C)	Bp (^{0}C)	Density (g/mL)	Solubility at 25 ^{0}C	Notes, Safety, (M)SDS
4-nitroaniline	$O_2NC_6H_4NH_2$	138.12	146-149	n/a	n/a	Slightly soluble in water	Yellow color Toxic Harmful to aquatic life. Dispose in special container. LD50 Oral - Rat - 750 mg/kg
dichloromethane (methylene chloride)	CH_2Cl_2	84.93	-97	40	1.325	Insol. in water	Heavier than water. Organic phase will be at the bottom. Waste disposal: halogenated solvent waste Not flammable. Skin irritant: if spilled on skin wash with plenty of water and soap
benzoic acid	C_6H_5COOH	Fill out this information!					
sodium hydroxide	NaOH	40.00			Corrosive. Can cause severe burns. If spilled on skin wash with plenty of water/shower. Alternatively, first wash with mild acid (aq. ammonium chloride, or dilute aq. acetic acid = vinegar) then wash with plenty of water		
hydrochloric acid	HCl	36.46			Corrosive. Can cause severe burns. If spilled on skin wash with plenty of water/shower. Alternatively immediately wash with mild base (aq. sodium bicarbonate) then wash with plenty of water		

4) Experimental Procedure (Synopsis/Outline of the experiment). This is where you must write all the information necessary to run the experiment (write in advance). Most of this is in the handout. But you can refer to a setup from a previous lab too, as long as the report you turn in contains a copy of the relevant page. (For example; simple distillation setup and thermometer placement.) It is also okay to re-draw the setups. Although it is not required, this is also where you can draw a flow chart for the experiment. Often having all the information on a single page as a scheme is very helpful.
5) Experimental procedure as conducted (write during the lab). This is where you write things while running the experiments. This can be pretty much anything, including controller setting, colors, spills, whatever. Here is the only place where you can use the first person "I". In some cases, some of it can be crossed-out later. However, none of this should be written after lab.
   Purification is when you have a step to purify. That can be folded into the experimental procedure as conducted.
6) Data yield and observed properties of product(s) are self-explanatory. This is where you calculate yield (you must show the calculations) and record the observed mp or bp, etc. Also consult "Tips and Techniques" about how to report yields and physical constants. You can write some of this section after the lab (calculations), as long as you wrote down all the necessary data during the lab.
7) Side reactions when relevant. This will be for preparative experiments (the ones where you are making a new chemical from a different starting material). This section does not apply to extraction/recrystallization.
8) Interpretations/Deviations/Conclusions. Write this after the lab. Half a page to one page in length. This is a very important part of your report, and it needs to be written AFTER you have conducted the experiment. Here again, any sentence that has "I" or "WE" in it is wrong. Saying that your results are not good because of human error is not acceptable. Instead, you must explain what went wrong and why something else should have been better. No feelings, only science. This is the section, which mirrors or answers the introduction. It needs to be general. The conclusion needs to prove that you learned something. I do not want to see things like "my yield was low because of human error" or whatever. Chemistry is a science, not a feeling nor magic. If your yield is low or whatever, you need to explain that in a scientific way. You also need to explain what parameter was important in the experiment, what could be done differently to get better results. No matter, NO "I or we"!
9) Technique grade. See also the Syllabus for a representative list of point deductions. Breaking things, wrong thermometer placement, constant questions to your neighbor, consistently late arrival/departure from lab, safety, etc. is where you are evaluated. We are hoping we will not have to penalize students for lateness, but all labs can be completed by 5 pm with appropriate preparation/planning.
10) **Your notebook must be self-sufficient**. It is up to you to record everything you might need, not only for this semester, but also for the second semester lab. You should have drawings of all the important setups so you can refer to them later on. No pasting, stapling, loose leaf, etc. If you like, you can have a separate section of your notebook where you collect drawings of all the setups so you can quickly refer to them at any time (and in the 2nd semester). For a particular experiment, you still need to have the setups drawn in the lab report you will turn in.

CLASS ID #: _007____ Date:__10/01/1998__ Lab Section: Thursday

EXPERIMENTS: _Diels-Alder and Isomerization of Dimethyl Maleate to Dimethyl Fumarate (LAB 3)_____	Points Awarded	Points Possible
1. Lab Write-Up	**26**	40
Heading (date, title, etc.)	**2**	2
Introduction/Purpose	**4**	4
Reactions, Side Reactions (if any), Mechanisms	**8**	8
Tables/Calculations: reactants, reagents, product, safety, etc.	**4**	6
Experimental Procedure, including purification (if any)	**5**	5
Interpretation/Deviations/Conclusions	**3**	15
2. Results	**13**	20
Yield 1 (Diels-Alder adduct)	**5**	5
Melting Point 1 (Diels-Alder adduct)	**5**	5
Yield 2 (dimethyl fumarate)	**1**	5
Melting Point 2 (dimethyl fumarate)	**2**	5
Sub total	**39**	60
Deductions (time, safety, etc)	**0**	-
Technique Grade	**18**	20
Lab Lecture Quiz (Tuesday 09/29/98)	**17**	20
TOTAL	**74**	100

Comments: *Generally good technique but a bit slow. Good understanding of the experiments but conclusion section was poor for the Diels-Alder experiment. 2nd melting point was off + low yield. Methanol needed to be included in the Diels-Alder table.*

DIELS-ALDER REACTION

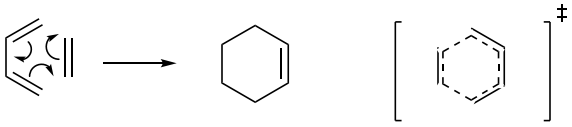
Reference: Experimental Organic Chemistry: A Miniscale and Microscale Approach, Gilbert and Martin,2nd Ed., pp. 176-178, 297-299, 344-348, 651-654.

INTRODUCTION: A cyclohexene derivative is prepared through the reaction between and a 1,3-diene and alkene (also called dienophile). This is an example of a cycloaddition and specifically a [4 + 2] cycloaddition: the Diels-Alder reaction. A reorganization of the pi-electrons occurs so that two new sigma bonds are formed in a concerted process. Because it is a concerted process (6-membered transition-state), Diels-Alder reactions are highly stereoselective.

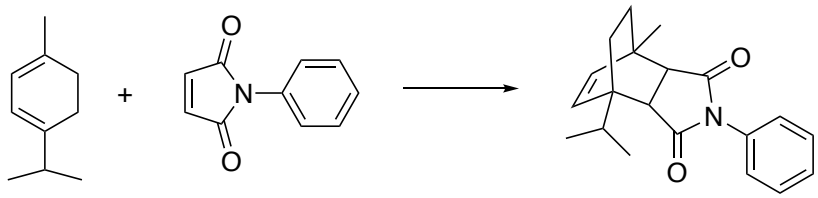
The Diels-Alder reaction is an example of a more general class of reactions called cycloadditions, themselves a subset of pericyclic reactions. Pericyclic reactions are defined as reactions which proceeds by concerted reorganization of electron pairs within a closed loop of interacting orbitals. In

cycloadditions, two or more molecules condense to form a ring by transferring electrons from pi-bonds to newly formed sigma-bonds.

REACTIONS:



4 + 2 cycloaddition



α-Terpinene (diene) | *N*-Phenylmaleimide (dienophile) | Diels-Alder Product

Side reactions should be very limited or inexistent, although the diene could potentially react with itself. But the dienophile is electron-poor and electronically matched with the electron-rich diene.

TABLE & CALCULATIONS:

Chemicals	Formula	MW (g/mol)	Mp (^{0}C)	Bp (^{0}C)	Density (g/mL)	Solubility at 25 ^{0}C	Notes, Safety, (M)SDS
alpha-terpinene	$C_{10}H_{16}$	136.10		173-175	0.837	Insol. in water	Flam. Liq. 3: H226 Acute Tox.(O) 4: H302 Skin Sens. 1B: H317 Eye Damage 2: H319 Aquatic (C) 2: H411
N-phenylmaleimide	$C_{10}H_7NO_2$	173.08	85-87			Insol. in water	poison CHEMWATCH HAZARD RATINGS Min Max Flammability: 1 Toxicity: 3 Body Contact: 2 Reactivity: 1 Chronic: 2 Min/Nil=0 Low=1 Moderate=2 High=3 Extreme=4  Oral, rat: LD50 = 58 mg/kg
Diels-Alder adduct	$C_{20}H_{23}NO_2$	308.18	not found (mp 134-136 ^{o}C given by Dr. M)				not found

Amounts used: terpinene 1 mL, N-phenylmaleimide, 0.40 g
(1 mL x 0.837 g/mL) / 136.1 g/mol = 6.1 mmol terpinene
0.40 g / 173.1 g/mol = 2.3 mmol *N*-phenylmaleimide
***N*-phenylmaleimide is the limiting reagent**
theoretical yield = moles of limiting reagent x MW of product
= 2.3 x 10^{-3} x 308.2 = 0.71 g of adduct
empty flask: 29.69 g
flask + product: 30.25 g
total: 0.56 g

Percent yield: 0.56 g/0.71 g = **79 %**

EXPERIMENTAL PROCEDURE:

Note: this section should NOT be in your reports. It is only included here to show you the differences between this (the procedure given to you before the lab) and what you need to write (which is exactly what you did in the lab, including deviations, observations, etc.)

I. Reaction. Place 0.4 g of N-phenylmaleimide in a stoppered 25 mL Erlenmeyer flask. Measure 1 mL of α-terpinene into the flask, and replace the stopper. Heat the flask gently for 25-30 min (power controller on 3) in the sand bath. The bright yellow color of N-phenylmaleimide may or may not disappear completely as the colorless product forms. After the heating period, remove the flask from the sand and allow it to cool to room temperature. (You can leave the heating mantle on.) White, powdery crystals should begin to form fairly quickly. If necessary, the flask can be cooled in an ice bath to induce crystallization.

II. Recrystallization. To purify the product by recrystallization, add 8 mL of methanol (CH3OH) to the erlenmeyer flask and heat the mixture on the heating mantle (power controller still on 3, and prepare the hot plate, as in lab 3), until all the crystals have dissolved. If some solid impurities remain, proceed with a hot filtration. If necessary, more methanol may be added, but the total volume should not exceed 10 mL. DO NOT STOPPER THE FLASK DURING THE HEATING PERIOD. Turn off the heat, stopper the flask, and allow the solution to cool slowly to room temperature. Better crystals are obtained when the flask is allowed to cool very slowly. Finally, cool the product in an ice bath and isolate the needles by vacuum filtration. The crystals may be washed with a very small amount of ice cold methanol. Transfer the product to a watch glass and spread the needles to facilitate drying. Weigh the purified product, calculate the yield, obtain a melting point, and turn in the crystals to your laboratory instructor.

ACTUAL PROCEDURE & OBSERVATIONS:

I. Reaction. 0.4 g of N-phenylmaleimide and 1 mL of α-terpinene were placed in a stoppered 25 mL Erlenmeyer flask. The flask was heated for 23 min (power controller on 2) in the sand bath. The bright yellow color of N-phenylmaleimide did not start to disappear until 15 minutes into the heating process. The color changed from deep yellow (*N*-phenylmealeimide) to very light yellow, indicating the majority of reactants converted to product.

White crystals precipitated out upon cooling to room temperature. Cooling the flask in an ice bath to induce crystallization was not necessary.

II. Recrystallization. To purify the product by recrystallization, 9.5 mL of methanol were added while heating to dissolve all the white solid. The solution was then cooled to room temperature slowly (by leaving the flask on the edge of the sand bath before placing it on the hood's floor. Then it was cooled in an ice bath. **Vacuum filtration yielded 0.56 g of white needle-like crystals.**

The melting point was first measured on the thermal ruler at 126 ^{o}C, after 15 min of air drying. When measured carefully on the MelTemp, the **melting point was 134-136 ^{o}C.**

INTERPRETATION/DEVIATIONS/CONCLUSIONS

The production of cyclohexene derivative was fairly easily attained. The reaction proceeded with very little difficulty. I did lose product during filtration and transfer to the flask. Overall, my percent yield was 79 %. There must be a way to decrease the loss of product during these transfers.

This conclusion is unsatisfactory. Never use the words "I" "we" "our" "my" "the student" "the experimenter" or any other word referring to you directly.

Saying that your results are not good because of human error is not acceptable. Instead, you must explain what went wrong and why something else should have been better.
You also need to explain what parameter was important in the experiment, what could be done differently to get better results.
Saying the yield was lower because of loss during transfer is wrong, unless it was a spill.
Possible conclusion points:
The Diels-Alder reaction is operationally simple only requiring heating.
In the particular example four stereocenters were formed in a single operation and the yield of product was good. The limiting reagent was the dienophile because it is a solid, whereas the diene was used in excess as a solvent. Perhaps using a different solvent (toluene?) could allow to use only stoichiometric amounts of reagent
Possibly a better yield could have been obtained if less solvent had been used in the recrystallization step, since the solvent will still dissolve some product even when cold. The product had a good melting point (the students were told mp 134-136 ^{o}C) and therefore must have been quite pure.

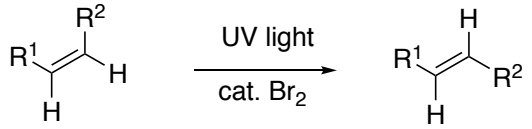
ISOMERIZATION OF DIMETHYL MALEATE TO DIMETHYL FUMARATE

Reference: Experimental Organic Chemistry: A Miniscale and Microscale Approach, Gilbert and Martin, 2nd Ed., pp. 181-183.

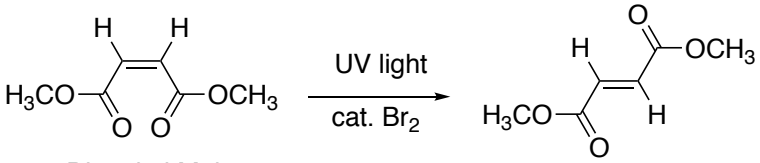
INTRODUCTION:

Alkene isomerization from the less stable cis (or *Z*) isomer to the more stable trans (or *E*) isomer can be achieved because it is thermodynamically downhill. One way to break the pi-bond is through a free radical mechanism. The free radical can be generated using UV-light and bromine.

REACTIONS:

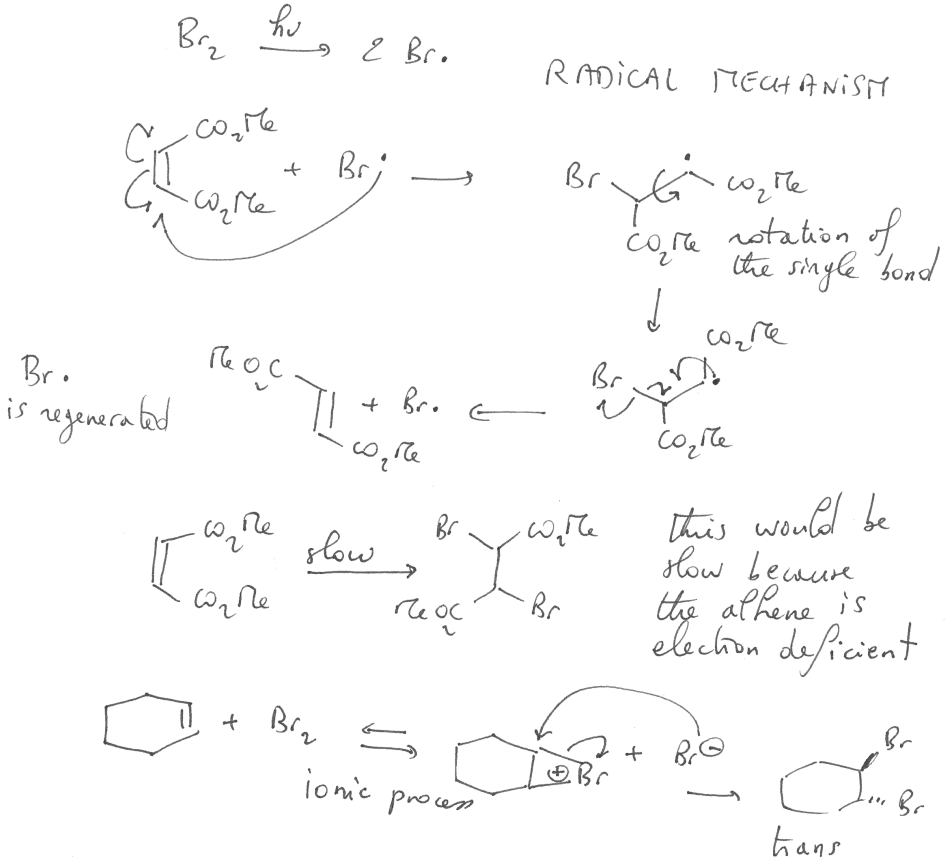


less stable cis — more stable trans



Dimethyl Maleate — Dimethyl Fumarate

MECHANISM:



Good, but it would be better to show the radical mechanism with equilibrium arrows, even if the backward steps can be uphill. Also, could show some termination steps (any two radicals combining).

TABLE & CALCULATIONS:

Chemicals	Formula	MW (g/mol)	Mp (^{0}C)	Bp (^{0}C)	Density (g/mL)	Solubility at 25 ^{0}C	Notes, Safety, (M)SDS
bromine	Br_2	159.82		59	3.119	soluble in water and in CH_2Cl_2	**Very hazardous.** **Incompatible with acetone**  LD50 Oral - Rat - 2,600 mg/kg
dimethyl maleate	$C_6H_8O_4$	144.14		204-205	1.152	soluble in water 77.9 g/L at 20 ^{o}C	Acute toxicity, Dermal (Category 3) Acute toxicity, Oral (Category 4) Skin irritation (Category 2) Eye irritation (Category 2A) Skin sensitization (Category 1) Specific target organ toxicity - single exposure (Category 3) LD50 Oral - rat - 1,410 mg/kg LD50 Dermal - rabbit - 610 mg/kg
dimethyl fumarate	$C_6H_8O_4$	144.14	102-106	192-193		not very soluble in water: 1.6 g/L at 20 ^{o}C in ethanol 10 g/L at 25 ^{o}C	Acute toxicity, Oral (Category 5) Acute toxicity, Dermal (Category 4) Skin irritation (Category 2) Eye irritation (Category 2A) Skin sensitisation (Category 1) Acute aquatic toxicity (Category 2) LD50 Oral - rat - 2,240 mg/kg LD50 Dermal - rabbit - 1,250 mg/kg
cyclohexene	C_6H_{10}	82.15	-104	83	0.811	insoluble in water	Highly flammable LD50 Oral - Rat - 1,940 mg/kg LD50 Dermal - Rabbit - > 200 mg/kg

Amounts used: bromine, a few drops of 0.6 M in dichloromethane. One drop is about 0.05 mL (6 x 0.05 mL) = 0.3 mL. Approximately 0.18 mmol of bromine was used.

Dimethyl maleate: 0.5 mL x 1.15 g/mL = 0.57 g
Since dimethyl maleate and dimethyl fumarate are isomers, they have the same molecular weight, so the **theoretical yield is also 0.57 g**

weight of dimethyl fumarate: 0.13 g

Percent yield: 0.13 g/0.57 g = **23 %**

EXPERIMENTAL PROCEDURE:
Note: this section should NOT be in your reports. It is only included here to show you the differences between this (the procedure given to you before the lab) and what you need to write (which is exactly what you did in the lab, including deviations, observations, etc.)

NOTE: REMEMBER THE SAFETY PRECAUTIONS ABOUT BROMINE FROM LAB 2! Bromine and acetone are incompatible.

I. Reaction. Prepare 3 test tubes and add 0.5 mL of dimethyl maleate into each tube. AS ALWAYS, working in your hoods – and wearing GLOVES – add enough of a 0.6 M bromine solution in dichloromethane dropwise with a pipet to TWO of the test tubes, until the orange color persists. To the third tube, add an equivalent amount of dichloromethane. Loosely cork all the test tubes. Place one of the tubes containing bromine in the dark (in your drawer or with the other tubes but wrapped in aluminum foil), and expose the other two test tubes to light. Place the tubes far enough from the light (about 20 cm) so that the solvent does not boil causing the popping of the corks. If discoloration of a solution containing bromine occurs, add an additional portion of the bromine solution to restore the orange color. After about 30 min, record in which test tube(s) crystals have appeared. [If none of the tubes contain crystals, continue the experiment for 30 more minutes.] Add 1-2 drops of cyclohexene to the tubes containing bromine to destroy the excess bromine.

II. Recrystallization. Add 2-3 mL of ethanol (95%) to any tube containing solid, and heat to dissolve the solid. [During the recrystallization, the power controller on the sand bath should be set to about 5]. Allow the solution(s) to cool slowly to room temperature and isolate the precipitate by vacuum filtration. Transfer the product to a watch glass and spread the needles to facilitate drying. Weigh the purified product, calculate the yield, obtain a melting point, and turn in the crystals to your laboratory instructor.

ACTUAL PROCEDURE & OBSERVATIONS:

1. Place one test tube with bromine (in dichloromethane) and dimethyl maleate in drawer (dark). Place another tube with bromine and dimethyl maleate in drawer in the light (a regular incandescent light bulb was used and the tube was in a test tube holder approximately 10 inches away). A third tube containing dimethyl maleate and dichloromethane but no bromine was exposed to light. Reactions were conducted for 30 minutes.

No precipitate was observed in the test tube not containing any bromine. In the light with bromine, a precipitate formed. No precipitate formed in the dark.

Two drops of cyclohexene were added to the tube with the precipitate and the tube that was in the dark in order to quench the bromine.

3 mL of ethanol were added to the tube containing the precipitate. Upon heating the precipitate dissolved. The tube was then allowed to cool down to room temperature and the precipitate was collected by vacuum filtration. But because a lot of the precipitate was stuck to the bottom of the test tube, more ethanol had to be used and this caused some loss of crystals.

The weight of crystals after 30 minutes of air-drying was 0.13 g. But when transferring the solid to turn it in, more was lost so only 0.07 g was turned in.

The melting point was 99 - 102 ^{o}C

INTERPRETATION/DEVIATIONS/CONCLUSIONS

Olefin isomerization has a limited scope since it requires that the desired isomer is the most stable. Also, in general, the isolation/purification of the product may not be as straightforward as in the experiment conducted, where the desired isomer precipitates. The reaction is simple to run but requires light and bromine in small amount to generate free radicals. The reactions conducted in the dark or in the absence of bromine did not proceed, supporting a free radical mechanism. Since the light used was an incandescent light, it is low energy and a UV light would have been better. Also, the reaction time may have been a bit short. The yield was relatively low for these reasons and also because too much recrystallization solvent was used. The melting point of the product was also much lower than expected, indicating it is impure.

OTHER THINGS LOOKED AT FOR THE GRADING

1) The introduction and conclusion should be general: the name of the exact chemical(s) employed should not be mentioned or should be only used as an illustration to show that this preparative experiment could apply to MANY organic compounds: Diels-Alder is representative of the [4+2] cycloaddition between a diene and a dienophile, the isomerization is less general but illustrates how the mechanism of a reaction can be studied. In both cases, purification by recrystallization is employed.

*2) **Reactions and mechanisms**. The reaction should summarize the experiment and be balanced: the structures should be drawn correctly (they were on the handout). The mechanism involving Diels-Alder should show either partial bonds or 3 arrows representing a cyclic movement of electrons: there should be mention somewhere of the concerted nature of the reaction. Isomerization: (a) arrows clearly shown and representing the proper electron flow, (b) reversible steps when relevant (ie. essentially all steps), c) for a radical mechanism, single headed arrows should be used, and d) must show the catalytic nature of the reaction (the bromine radical is regenerated). The equation for the reaction between bromine and cyclohexene should also be shown. In the case of the isomerization of electron-poor alkenes (as in the maleate to fumarate isomerization) it should be possible to use an ionic mechanism too, for example using an amine (reversible conjugate addition). In fact, this has been done using piperidine: Nozaki, K. J. Am. Chem. Soc. **1941**, 63, 2681-2683.*

***Side reactions** (Diels-Alder, isomerization): In this case think about those side reactions. For Diels-Alder, any sort of dimerization, and the exo product are okay. But the phenyl ring should NOT be employed as a diene! For isomerization, if addition of bromine to the double bond is shown, it can be considered a side reaction.*

*3) **Reagent Table**. Should be as complete as possible. Students should have discovered that the Diels-Alder adduct does not have a melting point reported in the literature. Bromine and cyclohexene should also be shown, as well as methanol and ethanol. They should have realized that MW for Diels-Alder adduct is the sum of the diene and dienophile. Similarly, the MW of the isomerized product is clearly the same as the starting material.*

*4) **Data Yield**. Make sure YIELD is spelled correctly and not "yeild". Calculations should be clear and accurate. A lot of the students seem to have some problems with that, so make sure this is done right. Mostly the points assigned should be given for actual yield. The average is around 70 %. For isomerization, yields above 50% are good. The reported yields should be rounded to the nearest integer because decimal places are meaningless (considering the accuracy of the weight or volume measurements).*

*5) **Synopsis.** The synopsis should be good since handouts were provided.*

*6) **Experimental Procedure as Conducted.** Mostly, look for something about how the setting on the power controller was changed (and why), something about the bromine – students seemed to have problems with that. What did you do when dimethyl fumarate did not precipitate spontaneously. Ideally, there should be a comment on how you organized the lab to be most efficient: you were told to do that, but you should explain why. For Diels-Alder, the complete or partial loss of color should be mentioned. Also you should say why they decided to hot filter or not.*
You should always report what you measure experimentally, not what you think the value should be!!! In fact, there could be reasons for why something is not as you expect it, and this is often something that should be discussed in your report.

*7) **Observed Properties of Products**. There should be two melting points reported (actually melting ranges: a single number should not be acceptable). Each compound should be a white crystalline solid. Each compound*

was collected in a properly labeled tube. The Diels-Alder compound is usually the best yield and looks like short needles. The fumarate should be long needles. Dimethyl fumarate: mp 103-104 °C (observed is good within 2-3 °C, okay ~ 5 °C, poor below that, Diels-Alder adduct: mp 134-136 °C (observed is good within 1-2 °C, okay within 5 °C, poor below that: for example 120-128 °C is zero point).

Yield and purity are always competing parameters.

*8) **Interpretation, Discussion**. A few things (by no means complete):*
There should be 2 parts, one for Diels-Alder, one for isomerization.
*High yield and wide melting range, or low yield with good melting point maybe most common. **Yield and purity are always competing factors.** In those situations, there should be a discussion: high yield may indicate a wet product, low yield may indicate a pure product. (Of course, other scenarios exist too!). Since the mp for the Diels-Alder adduct is not reported in the literature, there should be a discussion: narrow melting range should indicate a good/pure product. In other words, the interpretation of the melting points should include a discussion of what it means in terms of purity.*
There should be a comment that the recrystallization solvents are "good" solvents. If there were problems with the isomerization, there should attempt to explain what may be wrong (it is not a random approach!): it could be that the tubes were not clean (acetone will kill Br2, water will promote ionic reactions) or that exact amounts were not used, etc. Students should also realize that regular light is inefficient to promote the radical reactions, and that the low yield of product is not due to side reactions, but rather because of low conversion (a lot of starting material is unreacted), and that reaction time is also important. In terms of mechanism, the role of the control must be understood: the three tubes are designed to see the influence of bromine (they can even say that another control with no Br2 in the dark could have been useful). The mechanism is a radical mechanism (presented it in lab lecture), and Br2 does not add to the double bond to any significant extent. (it is not expected students explain why, but of course it is due to the fact that the double bond of maleate is electron poor, and therefore unreactive in electrophilic reactions). The purpose of cyclohexene to destroy (quench) the excess bromine must be discussed.
Also, the role of capping the tubes and when to do it or not should probably be explained. (In the Diels-Alder, one does not want to lose the diene by evaporation, but if a solvent is present the pressure would build up). As usual, possible loss in yields can also be attributed to using too much solvent, etc.
In your reports do not say "the results are not what I expected because I made a mistake", "because of experimental error" (unless you actually EXPLAIN what that error might be), etc. Unless you spilled something, there might not be an obvious mistake. If an explanation is: "my boiling range was wrong because my thermometer was too high" that's already much better, and better yet if you say your boiling range was LOW because the thermometer was too high.