

# Example Lab Report for Organic Labs

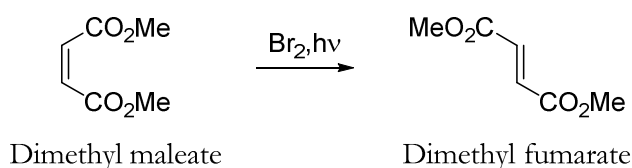
Actual lab report is in black; notes about how to write reports are in red.

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1/1/2000

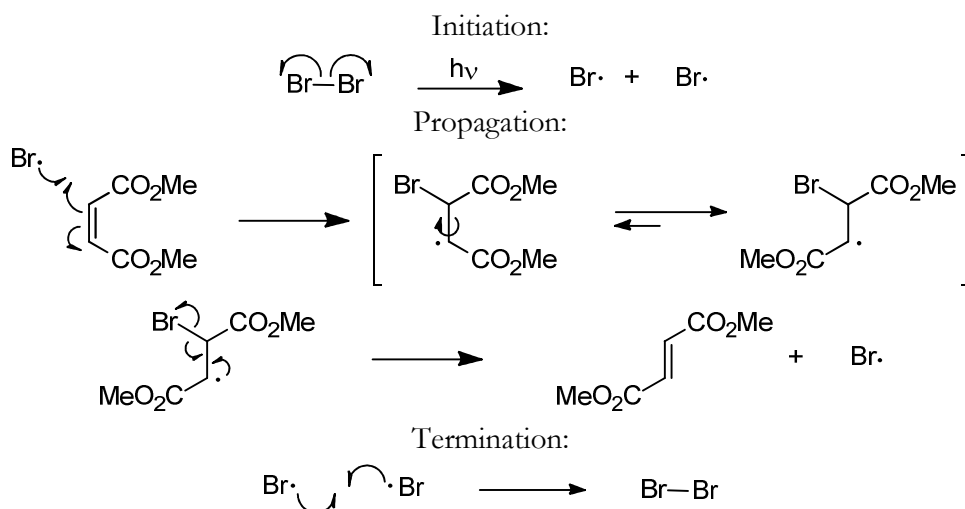
The introduction, physical data, and procedure make up the prelab, which you need to type up (not handwrite) before the experiment and bring with you. You won't turn it in yet, but your TA will initial it during the lab.

## Experiment 1 Prelab: TLC of Reactions – Isomerization of Dimethyl Maleate

**Introduction:** The purpose of this experiment is to convert dimethyl maleate to dimethyl fumarate, through a reaction with bromine and UV light. The final product will be purified by recrystallization and characterized by melting point and TLC. A secondary purpose for this experiment is to learn to use TLC to monitor reaction progress, and to select a good solvent system to use for this process. This is both a technique and preparative lab. If you are writing up only a technique lab, you don't need a mechanism but you do still need to show the chemical structures you will use.



Like other free-radical reactions, the mechanism consists of initiation, propagation and termination steps.



**Physical Data:**

All info is from CRC Handbook, except Hazards, which are from MSDS sheet for each compound. Note that information in superscript or parentheses (for instance, in density column) is not copied over – this just gives info about conditions when these quantities were measured. Solubility info is explained in CRC, but msc = miscible, s = soluble, sl = slightly soluble, i = insoluble.

Name	MW	MP (°C)	BP (°C)	Density (g/mL)	Solubility	Hazards
Dimethyl maleate	144.13	-19	202	1.1606	sl H <sub>2</sub> O, lig; s eth, etc	Hazardous in case of skin contact (irritant), of eye contact (irritant), of ingestion.
Dimethyl fumarate	144.13	101.7	193	1.37	i H <sub>2</sub> O; s ace, chl	Harmful in contact with skin. Causes skin irritation. Causes serious eye irritation
Bromine	159.808	-7.2	58.8	3.1028	sl H <sub>2</sub> O	Very hazardous in case of skin contact (corrosive). Hazardous in case of skin contact (irritant, permeator), of eye contact (irritant), of ingestion, of inhalation.
Dichloromethane	84.933	-95	39.8	1.3266	sl H <sub>2</sub> O; msc EtOH, eth; s etc	Causes eye and skin irritation. Causes respiratory tract irritation. Harmful if swallowed. May be harmful if inhaled.
Hexane	86.175	-95.27	68.72	0.6606	i H <sub>2</sub> O; vs EtOH; s eth, chl	Hazardous in case of skin contact (permeator), of ingestion, of inhalation. Flammable.
Ethyl acetate	88.106	-83.8	77.1	0.9003	s H <sub>2</sub> O; msc EtOH, eth; vs ace, bz	Hazardous in case of ingestion, of inhalation.

Theoretical yield:

Using abbreviations DM = dimethyl maleate, DF = dimethyl fumarate. Procedure calls for 15 drops of DM. Standard assumption is 20 drops = 1 mL, so procedure uses 0.75 mL DM.

$$(0.75 \text{ mL DM}) \left( \frac{1.1606 \text{ g DM}}{1 \text{ mL DM}} \right) \left( \frac{1 \text{ mol DM}}{144.13 \text{ g DM}} \right) \left( \frac{1 \text{ mol DF}}{1 \text{ mol DM}} \right) \left( \frac{144.13 \text{ g DF}}{1 \text{ mol DF}} \right) = 0.87 \text{ g DF}$$

Use the equation editor in Word (or equivalent program) to set up this calculation.

**Procedure:**

1. Make TLC standard of 1 drop dimethyl maleate in 1 mL DCM. Make another TLC standard of 10 mg dimethyl fumarate in 1 mL DCM.

2. Make up solvent mixtures of hexanes and ethyl acetate: 25:75, 50:50, and 75:25.
3. Run TLCs of the two standards in each solvent mixture, plus pure hexanes and pure ethyl acetate. Calculate  $R_f$  values for DM and DF in each solvent, plus  $\Delta R_f$  (difference in  $R_f$ s). Select solvent with highest  $\Delta R_f$  to use for next steps.
4. Make up TLC plates to use during reaction, in increments of 5 minutes (0 min, 5 min, 10 min, and 15 min). Each plate should have labels for DM, DF, and RXN (reaction mixture). Spot DM and DF standards onto each plate.
5. In 25 RBF, put stirbar, 4 mL DCM, and 15 drops DM. Spot rxn mixture onto "0 min" plate and develop in chosen solvent. Visualize with UV lamp.
6. Add 10 drops of bromine/DCM solution, stopper flask, expose to light, and start timing. Spot the reaction mixture onto a new plate every 5 minutes and develop the plate. Might need to make up more plates past 15 minutes if reaction is slow.
7. Keep spotting until relative darkness of spots in RXN column remains constant.
8. Add boiling chip and reduce reaction volume to about half.
9. Chill flask in ice bath, add 6 mL of hexanes, and wait for crystals to form.
10. Vacuum filter to collect crystals. Rinse with hexanes, then air dry.
11. Make up TLC spotter of final product and compare to DM and DF.
12. Weigh product and find yield and melting point.

This is the end of the prelab. The next part, Data and Observations, is handwritten in your notebook during the experiment and also initialed by your TA before you leave the lab. This part is not included in the example lab writeup.

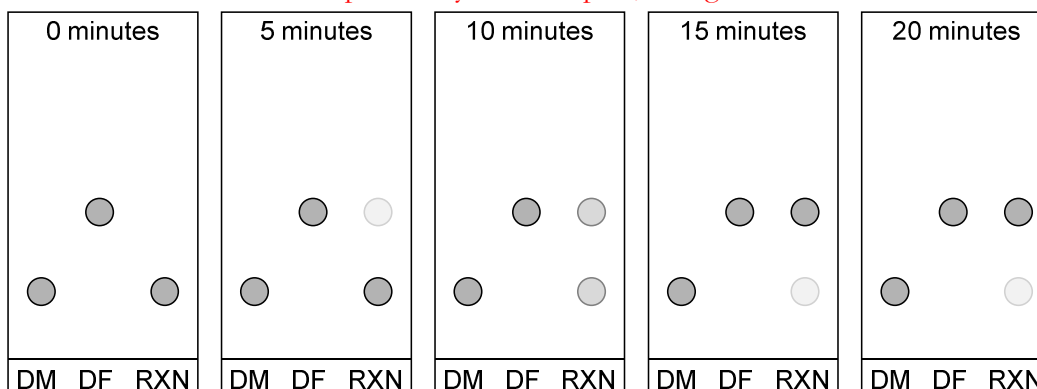
The discussion of results makes up the postlab, which is due the week after you perform the experiment. Note that this entire section should be written in third-person past tense.

### Experiment 1 Postlab: TLC of Reactions – Isomerization of Dimethyl Maleate

**Discussion of Results:** In this experiment, the goal was to isomerize dimethyl maleate to dimethyl fumarate. TLC was used to monitor the reaction as it proceeded, so an appropriate solvent was needed to maximize the readability and usefulness of the TLC plates. Standard spotters of the starting material (DM) and product (DF) were made in DCM, and plates were developed in 5 solvents: pure hexanes, 75:25 hexanes:ethyl acetate, 50:50 hexanes:ethyl acetate, 25:75 hexanes:ethyl acetate, and pure ethyl acetate. The  $R_f$  values for these solvent mixtures are shown below.

Solvent	DM $R_f$	DF $R_f$	$\Delta R_f$
Hexanes	0.075	0.175	0.10
75:25 Hex:EA	0.275	0.40	0.125
50:50 Hex:EA	0.55	0.65	0.10
25:75 Hex:EA	0.625	0.675	0.05
Ethyl acetate	0.725	0.75	0.025

The solvent with the largest  $\Delta R_f$  was 75:25 hexanes:ethyl acetate, so this was chosen to develop the plates during the reaction. 17 drops of dimethyl maleate were added to a round-bottom flask along with 4 mL DCM, and then bromine was added and the reaction timer was started. At 5-minute intervals, the reaction was spotted onto TLC plates, which are shown below. You can draw TLC plates like this by using the Drawing Elements toolbar in ChemDraw, or by using any other drawing/image program you prefer. If nothing else, you can print out the lab report and hand-draw the plates. Do not attach real TLC plates to your lab report, though!



At first the only spot visible in the reaction was dimethyl maleate, but over time this spot faded and the dimethyl fumarate spot became darker. The relative darkness of the spots did not change between 15 and 20 minutes, so the reaction was determined to be complete at this point.

The dimethyl fumarate product was purified by recrystallization. The reaction mixture was reduced by about half, and then the reaction was cooled and hexanes were added. This was done to reduce the solubility of the product, so that it formed crystals instead of remaining in solution. The starting material did not recrystallize due to its being a liquid at room temperature, so it was effectively separated from the product. The dimethyl fumarate product formed as long white crystals over the course of 15 minutes.

The product was isolated by vacuum filtration – during this time, a large amount of product was spilled on the floor and could not be recovered. The remaining product was washed with cold hexanes and allowed to air dry for several minutes, then weighed. The mass of product was 0.2234 g.

Since 17 drops of dimethyl maleate were used, the actual volume of DM was

$$(17 \text{ drops DM}) \left( \frac{1 \text{ mL DM}}{20 \text{ drops DM}} \right) = 0.85 \text{ mL DM}$$

This gives an updated theoretical yield:

$$(0.85 \text{ mL DM}) \left( \frac{1.1606 \text{ g DM}}{1 \text{ mL DM}} \right) \left( \frac{1 \text{ mol DM}}{144.13 \text{ g DM}} \right) \left( \frac{1 \text{ mol DF}}{1 \text{ mol DM}} \right) \left( \frac{144.13 \text{ g DF}}{1 \text{ mol DF}} \right) = 0.99 \text{ g DF}$$

The percent yield was calculated to be 23%.

$$\left( \frac{\text{actual yield}}{\text{theoretical yield}} \right) = \left( \frac{0.2234 \text{ g}}{0.99 \text{ g}} \right) = 23\% \text{ yield}$$

This is not very high, but since most of the product was spilled before weighing, the actual yield would probably have been much higher otherwise.

The product's melting point was recorded as 100.6°C. This is 1.1°C lower than the literature value, so the product is reasonably but not extremely pure. If higher purity were needed, the product could be recrystallized a second time to remove more starting material.

Overall, this experiment mostly worked well, but it would have worked better if the product had not been spilled. TLC was a useful technique for monitoring the reaction, once a good solvent was chosen, and all of the plates developed with no questionable spots or streaking. The product was fairly pure.