

Experiments are to be done *individually*., but you will work with a partner.

• **Pre-lab:**

Read: Experiment 3 Parts A and B (pp. 21 – 27), Technique 11 (pp. 647 – 668)

Skim: Technique 8 and Technique 9 (pp. 616 – 636)

Prepare for class on Tuesday, January 15: “Purpose” and “Materials”.

After the “Purpose” section, don’t forget to draw the **structure of sulfanilamide**.

In addition, within the “Materials” section, perform the two sets of “Pre-lab Calculations” on page 23 and on pages 25-26.

Sketch the apparatus/glassware you will be using in both experiments.

Reserve the next few pages for “Procedure” and “Data” and “Results”, but do not write any procedures or data tables yet.

• **During lab:**

If your desk is to the left of the fume hood you work in, do experiment 3A.

If your desk is to the right of the fume hood you work in, do experiment 3B.

Using your textbook, perform the experiment.

Write the procedure for the experiment as you perform it. Generate data tables (for instance, for impure and purified masses, melting point, etc.) as you need them.

• **For Thursday:**

Make a photocopy of the procedure you wrote. Turn in this photocopy first thing on Thursday. Clearly identify whether you did experiment 3A or 3B.

Take an NMR spectrum of your purified sulfanilamide (this will most likely be done on Thursday, January 24).

• **Post-lab:**

In the “Results” section, summarize your percent recovery of sulfanilamide from the impure mixture.

Include the NMR of your pure sulfanilamide. Make peak assignments (in order of chemical shifts) of all peaks (note that some peaks may be unrelated to the sulfanilamide).

The table below represents one way to report NMR results; you may also use the “lettered protons on the structure” representation, but make sure all the information is included.

Chemical shift (δ)	Functional group (identity of protons)	Splitting (singlet, doublet, etc.)	Integration value

Justify your peak assignments, based on chemical shift, splitting and integration.

The following should be included in your “Conclusions” section.

Parts A and B: Do point 1 (p. 31). Treat each method separately; that is, there should be two sets of answers for point 1. For point 1, give **three** sources of loss.

Percent recovery: Compare your percent recovery with the actual percent of impurity in the original sample and comment on your efficiency.

Melting point: One sentence on what the purified melting points told you about the purity of your sulfanilamide, compared to the impure sample.

NMR: Two sentences on what the NMR told you about the purity of your sulfanilamide. Just because your melting point range is tight, does this necessarily mean that all of the impurity is gone?

- **Lab Result Report: (Due Monday, January 29 at the beginning of lab)**

Photocopy the lab, all parts.

Answer questions:

End of Experiment 3 (page 31): 1, 2

End of Technique 11 (pages 667 and 668): 1 (all parts), 2, 7a, 7b, 8

For 7a and 7b, your description should include the actual (minimum) volume of hot solvent required to perform the crystallization.

Abstract:

- attach this to the **front** of your report
- must be **word-processed** on a **separate sheet of paper**

Please supply the missing information in the following abstract:

Your name, North Seattle Community College
PURIFICATION OF SULFANILAMIDE BY RECRYSTALLIZATION

An impure sample of sulfanilamide was purified by crystallization in (what solvent?) using both semi-microscale and microscale methods. The percent recovery for each method was _____ % and _____ %, respectively. This indicates that the (which method?) was more efficient, primarily because (give one or two good reasons why the method with the higher % was better, or why the method with the lower % was worse).