

Experiments are to be done *individually*.

• **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 Monday, January 22:** “Purpose,” and “Materials and methods”.

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

Reserve the next few pages for “Procedure” and “Data” and “Results”.

• **Post-lab:**

Complete “Results” section.

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

**Photocopy** the lab, all parts.

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 following should be included in your “Conclusions” section.

**Parts A and B:** Do points 1 ~~and 2~~ (p. 31). Treat each method separately; that is, there should be two sets of answers for points 1 ~~and 2~~. We did not do the optional exercise.

For point 1, give **three** sources of loss.

**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?

**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).