EXTRACTION OF ASPIRIN FROM AN ANALGESIC

EXPERIMENTAL TECHNIQUES REQUIRED

Extraction (T 6), filtration (T 3), and melting point determination (T 4)

INTRODUCTION

In this online experiment, we will be exploring the use liquid-liquid extraction using a separatory funnel to separate the analgesic acetyl salicylic acid (also known as ASA or aspirin) from the other components of a commercial mixture. In a normal pre-COVID scenario, you would have synthesized aspirin in the laboratory in one experiment and performed a separation by the extraction of caffeine from tea using the type of procedure introduced at the end of the solubility activity.

Liquid-liquid extraction using a separatory funnel is a very important and common technique for practical organic chemistry. It is typically used as part of the post reaction process during "work-up" to help isolate the desired reaction product from reagents and other by-products. Liquid-liquid extraction relies on solubility differences between immiscible layers to extract compounds or wash out other materials to selectively increase the purity of the desired material, typically the reaction product.

Aspirin is probably the best-known analgesic, but several other chemically similar compounds are also used as analgesics. Among these are acetaminophen, and ibuprofen. In addition to the active ingredients, the medications of these drugs contain starch, lactose and other substances that act as binders and permit rapid solution, and sometimes also inorganic bases. Aspirin has a long history and it tracks back over 2400 years to the use of willow leaves which contain a precursor of aspirin. Aspirin itself was first synthesised in 1853 by the reaction of sodium salicylate with ethanoyl chloride and in 1897 Bayer studying aspirin and started sales in 1899.

The objective of the online experiment is to learn about the techniques involved in the extraction of aspirin from the analgesic mixture. The initial analgesic mixture contains three active ingredients. The mixture is first dissolved in an organic solvent, diethyl ether, to dissolve the active ingredients and remove binding agents or other additives. The organic solution of the active ingredients is then treated with an aqueous solution of a weak base to make the aspirin water soluble so that the aspirin transfers to the aqueous layer. Separation of the organic and aqueous layers then allows the aspirin to be precipitated from the aqueous layer and recovered by filtration. After drying in an oven, the melting point of the aspirin is measured to check against literature values to help confirm the identity.

In order to complete your preparations for this online laboratory activity you should make sure to work through the documents that describe the techniques that are involved: extraction (T 6), filtration (T 3), and melting point determination (T 4). These documents contain information about each of these techniques and there also videos you can watch of them being performed.

There is no experimental procedure provided in advance for this activity. During the activity you will be led through the procedure and you will need make notes and observations that you will then need to use to help you write your report (this will be the "primary graded activity" for this experiment.

REPORT

Before writing any Chem 353 laboratory report, we strongly recommend that you review section 9 p. 14 in the introductory section of the <u>student laboratory manual</u> that discusses how to write reports and/or from "<u>writing reports</u>" on the course website. Students often don't get the grades they would like because they make errors that are addressed in that section of the manual. These are avoidable errors.

The report for this experiment is a formal report. Remember that more it not necessarily better. It is important to be accurate and concise rather than verbose and vague. Proper English should be used and it should be written in your own words. A **guide** to the sections/information you should include is shown below.

REPORT SECTIONS FOR THE EXTRACTION OF ASPIRIN

MAXIMUM 4 PAGES including the following content (line spacing 1.5, Times (font size 12) or Arial (font size 10)).

- 1. Title, date of the experiment.
- 2. Introduction.
- 3. Experimental procedure.
- 4. Results (for example: weight of aspirin obtained, appearance, yields, melting point etc.).
- 5. Discussion of results.
- 6. Conclusions.
- 7. References.