Preparation of Aspirin (Acetylsalicylic Acid) and Thin-Layer Chromatography of Analgesic Drugs

by Corry Decker, Adrian George, Siggi Schmid

Experiment Overview

This experiment consists of two distinct sections:

Synthesis of acetylsalicylic acid: Students add acetic anhydride, salicylic acid and sulfuric acid and heat the mixture on a water bath. The crude product is recrystallised and dried.

Thin-layer Chromatography: Students compare their prepared product with a commercial one. Students also run a separate TLC with four standards and one unknown. By comparison with R_F values and colours in UV light as well as in an iodine chamber they identify the components of analgesic tablets. The given standards are: acetaminophen, acetylsalicylic acid, salicylamide and caffeine. The unknowns are: Paracetamol (acetaminophen), Aspirin (acetylsalicylic acid) and Panadol Extra (acetaminophen & caffeine).

Level of Experiment

First year undergraduate

Keyword Descriptions of the Experiment

Domain
organic chemistry

Specific Descriptors
synthesis, acetylsalicylic acid, recrystallisation, thin-layer chromatography, analgesic drugs

Course Context

The experiment is carried out as part of the 1st year organic course, which teaches the basic principles of organic chemistry, such as classes of compounds, types of bonds, types of reactions etc. The experiment demonstrates one particular type of organic reaction (esterification) so the lecturer should have covered the theory of esterification as well as the basic principles of chromatography before the experiment is carried out.

Prerequisite Knowledge and Skills

The experiment is straightforward, easy to carry out and due to the importance to pain-killing drugs in our society is very much related to the real world and has high student interest. There are no prior practical skills required in the lab, which makes it an ideal candidate for a 1st year undergraduate experiment.

Time Required to Complete

Prior to Lab: 30 min
In Laboratory: 3 h
After Laboratory: 30 min

Submission Details

For Preparation of Acetylsalicylic Acid:

Whilst the authors listed are responsible for the educational analysis of this experiment, its submission to ACELL is done on behalf of all academic staff of the School of Chemistry at the University of Sydney. This experiment is quite commonly performed in first year university teaching laboratories and there are several sources, such as Olmsted (1988), Pavia, Lampman and Kris (1988), or Osborne (1998). Any modifications thereof were done by: Drs R. W. Baker, A.V. George, M. M. Harding, S. W. Lazer, all of the School. of Chemistry at the University of Sydney.

For TLC:

Whilst the author listed is responsible for the educational analysis of this experiment, its submission to ACELL is done on behalf of all academic staff of the Department of Chemistry at the University of Waikato. The original source is Cormier, Hudson, and Siegel (1979). Any modifications thereof was done by Dr Merilyn Manley-Harris, of the Department of Chemistry at the University of Waikato, email: manleyha@waikato.ac.nz.

Comments

This experiment is a combination of two experiments (preparation of aspirin and thin layer chromatography) and each may easily be carried out separately if a shorter experiment is required.

References

http://firstyear.chem.usyd.edu.au/LabManual/E29.pdf.

Cormier, R. A., Hudson, W. B., & Siegel, J. A. (1979). Thin layer chromatographic separation of common analgesics – a consumer experiment. *Journal of Chemical Education,* **56,** 180.

Olmsted, J. (1988). Synthesis of aspirin, a general chemistry experiment. *Journal of Chemical Education,* **75,** 1261.

Osborne, C. (Ed.). (1998). *Aspirin: A Curriculum Resource for post-16 Chemistry Courses.* London: Royal Society of Chemistry. Available from http://www.chemsoc.org/PDF/LearnNet/rsc/Aspirin_full.pdf.

Pavia, D. L., Lampman, G. M., & Kris, G. S. (1988). *Introduction to Organic Laboratory Techniques: A Contemporary Approach.* (3rd edition). Philadelphia: Saunders College Publishing.