# Isolation of Shikimic Acid from Star Aniseed

by Michael Edmonds

## Experiment Overview

This laboratory experiment was designed as a practical component in the Natural Products Chemistry course (3rd year undergraduate) at Christchurch Polytechnic Institute of Technology, Christchurch, New Zealand. This experiment uses Soxhlet extraction followed by purification using ion exchange (Amberlite 1RA-400 resin), decolorization using activated charcoal and recrystallisation – all useful techniques in natural product isolation. Subsequent charcoal decolourisation and recrystallisation afford pure shikimic acid in 2-7% yield. This experiment would also be relevant to organic chemistry courses.

Most of the equipment used in this experiment should be available in tertiary institutes and even well equipped school laboratories. One key requirement is a high vacuum pump that is capable of removing acetic acid from the extracts. Most chemicals used in this experiment should be readily available with the exception of the Amberlite IRA 400 ion exchange resin. However this resin is relatively inexpensive and may be regenerated for reuse.

## Aims and Objectives

Concepts that students will learn:

- Soxhlet extraction – this type of extraction provides a continuous, recycled supply of pure solvent it allows complete extraction of the desired compound. This can be contrasted to extraction in a separating funnel which is never (theoretically) 100% extraction.
- An understanding of solubility and polarity is also necessary. Students can be asked to justify why methanol is used as a solvent (polar enough to extract shikimic acid) and also to suggest what other compounds it might remove (its organic character actually means it removes a large number of compounds, even some of the relatively nonpolar essential oil components).
- Subsequent suspension in water – hot water dissolves the shikimic acid but less polar compounds form an oil on the surface of the water (again reinforcing concepts of polarity/solubility).
- Ion exchange column – shikimic acid displaces acetate ions and binds with the column. The acid will stay bound to the column at neutral pH allowing impurities to be removed. However once the pH is dropped the shikimic acid is protonated and is released from the column. This is a good example of how altering the pH can alter whether a compound is neutral or has a charge.
- Compound is then decolourised with activated charcoal (an often underestimated purification technique). At this point the product is reasonably pure (determined by $^{1}$H NMR) and on removal of the solvent an offwhite/orange solid forms. Subsequent recrystallization affords the desired compound as a bright white solid.

- Verification of the purity/identity of the compound can be carried out in a number of ways depending of the level of the students involved and the equipment available. Analyses we have utilised included melting point and NMR ($^{1}H$ and $^{13}C$).

## Level of Experiment

Second or third year undergraduate

## Keyword Descriptions of the Experiment

**Domain**
organic chemistry, biological chemistry

**Specific Descriptors**
isolation, natural products, shikimic acid

## Course Context

This experiment is one of six practical experiments used in the 3rd Year Natural Products course at Christchurch Polytechnic Institute of Technology. It also has potential application in organic chemistry courses.

## Prerequisite Knowledge and Skills

An understanding of isolation and purification techniques used in natural product/organic chemistry is advisable before attempting this experiment.

## Time Required to Complete

**Prior to Lab:** 1-3 h preparation time for staff
**In Laboratory:** One or two 3 h sessions - the experiment is usually completed in two sessions, but this can be reduced to one session by having the Soxhlet extraction carried out before the students arrive in class.
**After Laboratory:** 1-2 h for write up

## Experiment History

Experiment was developed in 2001 as a teaching experiment in the Natural Products 7 course at CPIT (this course is part of the Bachelor of Applied Science which is offered at CPIT on behalf of Auckland University of Technology).

The experiment was developed by Michael Edmonds (CPIT staff member) in collaboration with Richard Payne, who in 2001 was a Ph.D. student at the University of Canterbury. The experiment is based on work by Adams *et al.* (1996) as well as references within the aforementioned paper, and has been published (Edmonds & Payne, 2005 - a PDF version of this paper is available in Related Documents.

## References

Adams, H., Bailey, N. A., Brettle, R., Cross, R., Frederickson, M., Haslam, E., MacBeath, F. S., & Davies, G. M. (1996). The shikimate pathway. Part 8. Synthesis of (−)-3(*R*)-amino-4(*R*),5(*R*)-dihydroxy-1-cyclohexene-1-carboxylic acid: The 3(*R*)-amino analogue of (−)-shikimic acid. *Tetrahedron,* **52**(*25*), 8565-8580.

Edmonds, M. & Payne, R. (2005). Isolation of shikimic acid from star aniseed. *Journal of Chemical Education,* **82,** 599-600.