

Acetylation of Ferrocene

by Jonathan Morris

Experiment Overview

This experiment describes the Friedel-Crafts acylation of ferrocene with acetic anhydride, using phosphoric acid as the catalyst, to form acetylferrocene. It provides a means of introducing the chemistry of electrophilic aromatic substitution and the metallocenes. This material is part of the Level II lecture material at the University of Adelaide and this experiment provides a means of exploring this chemistry. As the reaction does not go to completion, chromatography is required to separate the product from the starting material. This experiment introduces how to carry out thin-layer and column chromatography. The students are expected to develop an experimental design, based on column chromatography, that will allow them to isolate pure material.

Aims and Objectives

The aims of the practical are:

- ☐ To illustrate that the chemistry of ferrocene is similar to that of benzene.
- ☐ To develop competency in analysis of reaction mixtures by thin-layer chromatography.
- ☐ To show the student how to develop an experimental design for the separation of mixtures by column chromatography.
- ☐ To develop competency in separation of compounds by column chromatography.

Level of Experiment

The experiment is designed to be run in one afternoon (4-6 hours) for Level II undergraduates.

Keyword Descriptions of the Experiment

Domain

organic chemistry, inorganic chemistry

Specific Descriptors

synthesis, chromatography, electrophilic aromatic substitution

Course Context

The experiment develops practical skills related to the underlying principles of synthetic chemistry that is presented in most second lecture courses. At the University of Adelaide, the students doing this experiment would have had lectures on aromatic chemistry at first year, as well as lectures on aromatic chemistry and spectroscopy at the second year level. The lectures in second year would cover the principles of electrophilic aromatic substitution and have included examples of mono-substituted aromatics and metallocenes

Prerequisite Knowledge and Skills

Ideally, students should also have an understanding of chromatography and separation of compounds. However, this is not considered essential as students will learn these concepts as part of the experiment. However, if they haven't had much exposure to the principles of chromatography this may mean that they will require more help in developing their method for purifying acetylferrocene.

Time Required to Complete

Prior to Lab: 1 h

In Laboratory: 4-6 h

After Laboratory: 0-1 h, depending on the extent of write up required (see comments, below)

Experiment History

This is a well-known experiment that has been utilised in many undergraduate laboratories. The author is aware of it having been used as a Level II or Level III experiment, and that it is often part of a longer experiment, wherein the ferrocene is first synthesised and then, the reactivity is investigated. In addition, the use of boron trifluoride and aluminium chloride as alternate catalysts have often also been explored. The chromatography section is often provided in full detail. To the author's knowledge the experiment with phosphoric acid is also used at the University of Canterbury and the University of Otago, NZ.

The original experiment has been published in: Fieser and Williamson, Organic Experiments, 7th ed. D.C. Heath and Co.

Submission Details

The University of Adelaide has used the experiment as presented here for the Synthetic Chemistry section of the second year Chemistry course for many years. It has been refined over the years by several people. Whilst the listed author is responsible for the educational analysis of this experiment, his submission of it to ACELL is done on behalf of all academic staff.

Comments

Time Required:

The practical involves background reading in order to answer some preliminary questions. Also, it is expected that the student would have to prepare a sheet that details the experiment in a flow-chart design. This is required for all Synthetic Chemistry experiments at the University of Adelaide.

Report Writing:

Students are required to write up their results into a laboratory notebook during the formal laboratory session. Rather than a formal report, the emphasis is on teaching students how scientists record their data and their observations. A series of questions are used to guide the students to

address all aspects of the practical. The notebook would be handed in for marking. It is expected that the report would take the form of:

- Title
- Diagram of reaction
- Reagent table
- The experimental procedure
- Analysis of the data / spectra
- The mechanism of the reaction
- Answers to any questions

Safety aspects:

As with the majority of synthetic chemistry experiments, this experiment should be run in the fume hood. It is expected that the students would have read the MSDS sheets for all the chemicals used in the experiment. To aid in this, the pre-lab questions would address the safety issues.

References

Fieser, L. F. & Williamson, X. X. (XXXX). *Organic Experiments*. (7th ed.) Place: D. C. Heath and Co. Chapter 38.

Shriver, D. F., Atkins, P. W., & Langford, C. H. (1994). *Inorganic Chemistry*. (2nd ed.) New York: Freeman. pp. 660 and 687-693.

McMurry, J. (2000). *Organic Chemistry*. (5th ed.) London: Brooks / Cole. pp. 600-605.