Learn Chemistry

Quanticorp R&D: New Products for the Future

Resource Overview

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HOW TO USE THE RESOURCE

This problem based learning resource is intended for level 6 undergraduate students on BSc Chemistry or related programmes of study. The resource can be adapted to suit the subject knowledge of the students.

The resource is divided into a number of parts which can be used in whole or in part. Some parts give detailed guidance to students whereas others are less prescriptive. However, the resource can be easily adapted to make it more open-ended by omitting some of the student guidance.

The activities contained within the resource require that students study and use material from journal articles. In particular, it is necessary to secure access to the following articles:

1. M. Gerard, A. Chaubey and B.D. Malhotra. Application of conducting polymers to biosensors. *Biosensors & Bioelectronics,* 2002, 17, 345–359.
2. Scherman, O. A., Rutenberg, I.M., and Grubbs, R.H.Direct Synthesis of Soluble, End-Functionalized Polyenes and Polyacetylene Block Copolymers. *J. Am. Chem. Soc*, 2003, **125**, 8515.
3. Moorhead and Wenzel. Two Undergraduate Experiments in Organic Polymers: The Preparation of Polyacetylene and Telechelic Polyacetylene via Ring-Opening Metathesis Polymerization. *J. Chem. Ed*., 2009, **86**, 973.
4. Knoll, K., and Schrock, R., *J. Am. Chem. Soc*., 1989, **111**, 7989.

The resource contains one part which contains laboratory work, however, sample results are provided so the tasks can be undertaken without access to lab facilities.

It is assumed that the tutor has a good grasp of undergraduate chemistry subject knowledge, however, detailed tutor notes are provided which should be used judiciously to support student learning.

The following narrative summarises the rationale behind the operation of each part of the resource.

Part 0: New Products for the Future

An e-mail is sent to students to set the scene. The scenario is that the students are employed as development chemists in the R&D department of QuantiCorp, a company which makes immunoassay test kits but wishes to diversify, and they must do some research into prospective new products based on conducting polymer sensors. Students are divided into teams and directed to commence the tasks for Part 1.

The aim and objectives are contained in the e-mail but can be re-iterated after students have had a chance to digest it, as follows:

Aim
To undertake a feasibility study on the possibility of developing a glucose sensor based on conducting polymers.

Objectives

- Prepare a presentation for the Chief Executive on the feasibility of developing a glucose sensor based on a conducting polymer
- Complete an in-depth feasibility study by undertaking background research and laboratory investigations
- Prepare and present a technical summary of the feasibility study for the Head of R&D

Part 1 Getting Started

In this part, students are directed to complete two tasks. Task 1.1 is to make a simple electronic humidity sensor in order so that they can understand the basic principle of sensor operation, with which they may not be familiar; however, this task can be omitted if the resources are not available. Task 1.2 is a literature based activity wherein students must research and summarise a review article on conducting polymer sensors which should give them a basic overview of the field.

Part 2: Presentation

Using the information they have gathered in Part 1, students should now prepare an ‘elevator pitch’ style presentation for the Chief Executive. In order to facilitate this it is suggested to use the *Message Box* tool which gives a simple framework for structuring the presentation. There are two objectives here: to focus on the important issues using the *Message Box*; and to get students to think about how they should pitch their presentation in a commercial, rather than a purely science oriented, way for the benefit of the Chief Executive.

Part 3: Conducting Polymers

At this point it is necessary to direct the students towards a specific line of inquiry, namely the development of a glucose sensor based on the conducting polymer polyacetylene. It is possible to use other sensor/polymer combinations and adapt the resource to suit, however, the laboratory work is based around synthesis of polyacetylene.

A **problem solving strategy** based on the creative problem solving approach is introduced wherein the students should be encouraged to plan their approach (Task 3.1) and gather the necessary information before attempting to solve the problem. Tasks 3.2 and 3.3 are designed to direct the students to use Wikipedia in the right way - as a quick way to gain an overview and identify key concepts which can then be used as search terms for the primary literature. At the end of this process, the students should now have a method for synthesis of end functionalised polyacetylene, using the Grubb’s second generation catalyst, from cyclooctatetraene and a chain transfer agent.

Part 4: Synthesis of Polyacetylene

In this part students are asked to consider how to investigate the synthesis of en-functionalised polyacetylene using the ROMP of COT reaction with Grubb’s second generation catalyst. They should be able to easily summarise a suitable procedure using the two papers provided but they are also asked to consider how to investigate the reaction conditions, stoichiometry, structure and conductivity of the product. The students should be allowed a free hand in this because Part 5 gives the opportunity for the tutor to intervene as much or as little as he/she wishes in the experimental design.

Part 5: Synthesis of Polyacetylene

Here, students are asked to design the experiment. Comprehensive tutor notes are provided to enable the tutor to give advice on the various aspects of the design as follows:

(a) Overall reaction scheme **-** this should not present a problem.
(b) Investigate the yield – the tutor can give as little or as much guidance on this as he/she feels appropriate, however, the two papers give in Part 4 should provide sufficient information.
(c) Apparatus and special requirements – the use of the equipment to maintain an inert gas atmosphere will probably require advice from the tutor.
(d) Reagents and special requirements – this should not pose a problem except that it may be necessary to specify **dry** reagents. **NB: One of the reagents must be synthesised beforehand.** A method is described in the tutor notes for this part.
(e) COSHH – this should not pose a problem; a blank COSHH form is provided but tutors will probably have their own version.

An **optional handout** *STUDENT NOTES FOR TASK 5.1* is provided if the tutor does not wish to allow students to design their own experiment.

Part 6: Synthesis of Polyacetylene

In this part the actual laboratory synthesis is performed using the procedure designed by the students. Some of the practical elements may require tutor guidance and tutor notes are provided for this purpose.

Part 7: Synthesis of Polyacetylene

Having prepared the polymer students must now perform the analysis using UV-vis, NMR and FT-IT spectroscopies. This part of the lab activity can be tailored to suit the instrumental facilities available, however, the end-functionalised polyacetylene should be soluble in dichloromethane so can be analysed using instrumentation in most chemistry laboratories.

If either Part 6 and/or Part 7 cannot be preformed then the sample data given in Resource 7.1 can be used.

Part 8: Reporting

Students should produce a report of their synthesis and subsequent analytical investigations. The report is intended to be an assessed element which requires students to interpret their results, critically evaluate them, compare them with data from several related papers, retrieve and summarise information from the primary literature and the internet.

The report could be used to satisfy the following assessed learning outcomes with suggested assessment criteria:

Learning Outcome		Assessment Criterion
Knowledge and Understanding		
	Inorganic Chemistry	Overall quality of discussion and extent of additional reading
Laboratory Skills		
	Observation and record keeping	Quality and presentation of NMR, UV-vis and IR spectra
Problem Solving		
	Working on problems	Molar ratios of reactants
	Working on problems	Discussion of effect on yield
	Working on problems	Calculation of theoretical yield
	Working on problems	Calculation of double bonds by: NMR Woodward-Fieser rules HOMO-LUMO transition
	Working on problems	Calculation of cis/trans ratio from IR
Communication		
	Report writing	Correct structure with tables and figures correctly captioned and references correctly cited.
	Information search, retrieval, assessment and use	Use and reference to papers by: Schermann & Grubbs (2001) Scherman et al. (2003) Knoll & Schrock (1989)
		Summary of the effect of iodine doping on conduction

<u>A comprehensive model answer is provided in the tutor notes for assessment and feedback purposes</u>.

Part 9: Conjugation

This part of the project requires the students to investigate how to make the conducting polymer selective for a particular analyte.
In Task 9.1 they are asked to reconsider their initial research from Task 1.2 and also the requirements for selectivity in a sensor by considering the IUPAC definition and some examples of how this can be achieved. The last activity is a link to an article on the enzyme glucose oxidase, however, this can initially omitted to allow a discussion about the ways in which selectivity can be introduced (enzymes being an effective means), and the article introduced subsequently.

Task 9.2 is meant to be a revision of knowledge of functional group chemistry which forms the basis for understanding conjugation reactions, and which is most suitable for conjugation of the enzyme glucose oxidase. The simple answer required here is that it can form a peptide bond at the C- or N- terminus; in reality this is not such a simple reaction to perform under these conditions and requires the use of an activating agent, however, this level of detail is not required to achieve understanding of the basic principle.

Part 10: Conjugation

This part addresses how the polyacetylene can be synthesised to contain appropriate functional groups to allow conjugation of the glucose oxidase enzyme.
Task 10.1 requires students to research conjugation reactions on the internet. The objective is to find a conjugation reaction which will allow conjugation of the C- or N-terminus functional group on the enzyme with an appropriate end-functional group on the polymer. The polymer which they have synthesised contains *tert*-butyldimethylsiloxane functional groups, which will not conjugate with either the C-C or N- terminus on the enzyme. *tert*-butyldimethylsiloxane is an alcohol protecting group, so it may be possible to modify the polymer synthesis to include the trimethylsilyl chloride instead, which is a protecting group for –COOH. This can then be de-protected and conjugated with the N- terminus of the enzyme.

Part 11: Report to the R&D Team Leader

This is the final task, and can form the basis for a second assessment. This can be in the form of a 10-15 minute presentation or a final report.
A powerpoint presentation framework has been provided which can be adapted by the tutor for a plenary session.

Suggested Timetable

Part	Description	Format	Miniumum duration (min)
		Part 0: New Products for The Future	
0	Student Scenario	E-mail sent to students divided into groups	
		Part 1: Getting Started	
1.1	Building a humidity sensor	Student centered practical activity	60
1.2	Using the literature	Student centered library activity	90
		Part 2: Presentation	
2.1	Prepare an elevator pitch	Tutor led activity	30
2.2	Three minute presentation	Tutor led activity with student and tutor feedback	60
		Part 3: Conducting Polymers	
3.1	Planning		30
3.2	Research using the internet	Student centred activity but may require some tutor supervision	30
3.3	Research using the primary literauture	Student centered activity with tutor feedback	30
		Part 4&5: Synthesis of Polyacetylene	
4.1	Using the literature to develop a synthetic method	Tutor led but student centered with tutor feedback	120
5.1	Design an experiment	Student centred with tutor feedback	120
		Part 6: Synthesis of Polyacetylene	
6.1	Laboratory synthesis of polyacetylene	Tutor supervised but student led	180
		Part: Synthesis of Polyacetylene	
7.1	Laboratory investigation of product	Tutor supervised but student led	180
		Part 8: Synthesis of Polyacetylene	
8.1	Reporting	Student centred activity. Tutor guided and assessed.	180

	Part 9: Conjugation		
9.1	Research into selectivity of the sensor using the internet	Student centered activity with tutor feedback	120
9.2	Revision of functional group chemistry and conjugation reactions	Student centered activity with tutor feedback	60
	Part 10: Conjugation		
10.1	Modify synthetic procedure to allow conjugation of the sensing molecule.	Student centered activity with tutor feedback	60
	Part 11: Report to the R&D Team Leader & Plenary		
11.1	Technical summary of investigation	Student oral or written report	120
	Plenary session	Feedback from tutor	30