Learn Chemistry

Quanticorp R&D: New Products for the Future

Student Guide

Developed by Dr. E Hywel Evans

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QUANTICORP R&D PART 0: NEW PRODUCTS FOR THE FUTURE

You are employed as development chemists in the R&D department of QuantiCorp, a high-tech company based in California which manufactures dipstick immunoassay pregnancy test kits. The Chief Executive has decided that it's time to diversify into new areas in order to grow the business and is asking the R&D department to come up with some possible areas for development. A scoping exercise has come up with the possibility of developing a glucose sensor based on conducting polymers. In order to get the best ideas the Chief Executive has decided to split you up into a number of competitive teams to investigate the feasibility of this idea. She wishes to hear your ideas upfront before allowing you undertake a feasibility study. After the feasibility study you will have to report back to the Head of R&D and the best study will be adopted and rewarded with a salary bonus.

QUANTICORP R&D PART 1 GETTING STARTED

You have just been sent a memo with instructions to present your idea to the Chief Executive for consideration by the end of the week. Panic sets in as you realise that you know nothing about electronic sensors, let alone conducting polymers. Luckily, however, the daughter of one of your team members has just completed a high school science project which looks like it might be of interest and she has agreed that you can use it for training purposes.

Task 1.1 – getting up to speed (60 min.)

Open **Resource 1.1**. Read the instructions for building a simple humidity sensor using the materials supplied. Assemble the sensor and perform the following tests:

Adjust the sensitivity until the LED alarm responds to the humidity of human breath

Wet your index finger and hold it near the sensor without touching so that the LED alarm responds.

Task 1.2 - using the literature (90 min.)

You had some fun with the science project and got the LED to light up in the face of your breath, but now you have to start considering the science in more depth. Open and read the paper by **Gerard *et al*.**[1], and answer the questions below.

1. How could you make this sensor respond to chemicals other than water?
2. How could you make the sensor more specific for the detection of a particular chemical?
3. What are the potential applications for these types of sensor?

When you have finished collate the information.

[1] M. Gerard, A. Chaubey and B.D. Malhotra. Application of conducting polymers to biosensors. *Biosensors & Bioelectronics,* 2002, 17, 345–359

RESOURCE 1.1: SCIENCE PROJECT: A SIMPLE WATER SENSOR

by
Amy V. Clever

(adapted from M. J. Sailor, Water Sensor Experiment, UCSD NanoLab, University of California, San Diego)

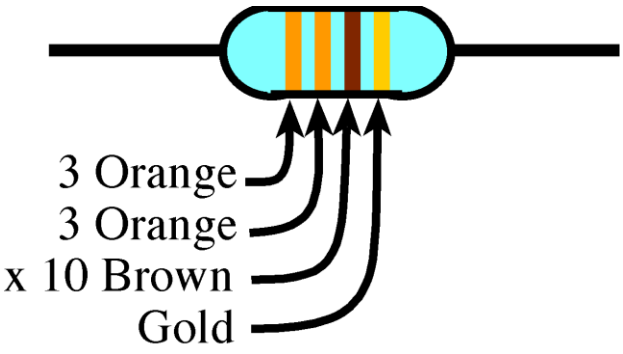
Equipment

9 V Battery
Battery clip
100 kΩ resistor (Brown)
330 Ω resistor (Blue)
npn transistor (2)
red LED
30 pF capacitor (water vapor sensor-see text)
100 kΩ variable resistor
plug-in board
Manual range digital multimeter
Assorted wires

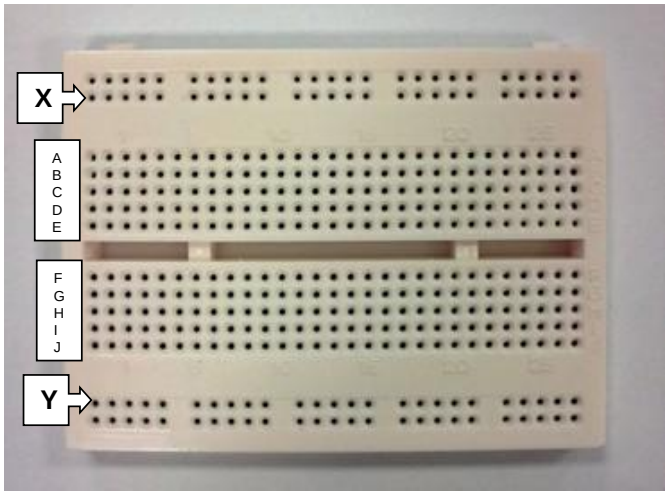
Measuring Resistance

Test the resistance of your skin with the multimeter set to '2000 k' on the **Ω** resitance setting). Now hold the the metal tip of the black probe between the thumb and forefinger of one hand and the metal tip of the red probe in the other. Your skin resistance should read around 500-1000 kΩ. Your skin resistance is therefore between 500,000 to 1,000,000 Ω. If your skin is dry it will register higher; if it is moist you will get a lower reading. This is the principle on which police 'lie detector' tests operate. The lie detector assumes that if you are lying you will sweat, causing the reading to change to a smaller value.

Now test the resistance of the resistors supplied in your kit. The resistors have a color code painted on them in a series of stripes so that you can easily identify their values without having to measure them. The first color band is the tens place, the second color band is the ones, and the third band is called the multiplier. The fourth color band is called the tolerance (it's like the quality of the device), which you can ignore. So the resistor pictured right is orange-orange-brown and should be 3 + (3 x 10) or 330 Ω. When you measure this resistor with your multimeter set to '2000 Ω' it should read pretty close to 330 Ω. The other resistor in your kit (the brown one) has a resistance of 100 kΩ. The color code is: brown = 1, black = 0, and yellow = 10,000.

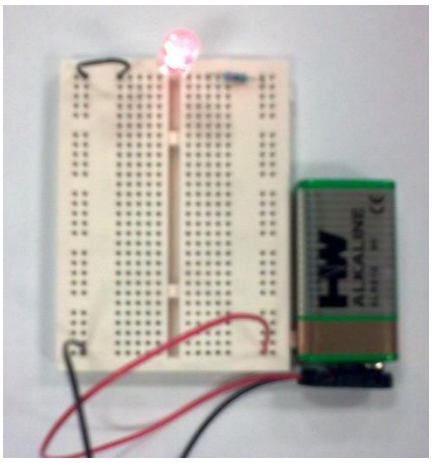


The plug-in board



The plug-in board allows you to connect all the components together. It is wired as follows: all holes in row X are connected together and all holes in row Y are connected together. Each individual column of five holes are connected together. Rows X and Y will be used to supply power from the battery. Each column consists of 5 holes, which are all connected to each other. If you plug something into column 1-D, you can connect it to something else by putting a wire into column 1-A, 1-B, 1-C, or 1-E.

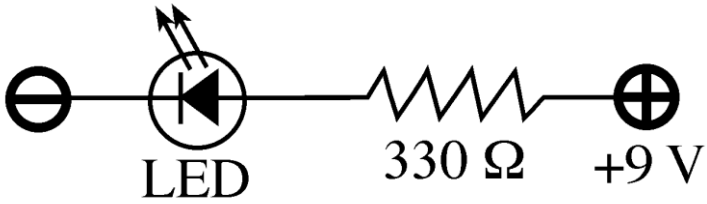
Experiment #1 Wiring up a Light-Emitting Diode (LED)



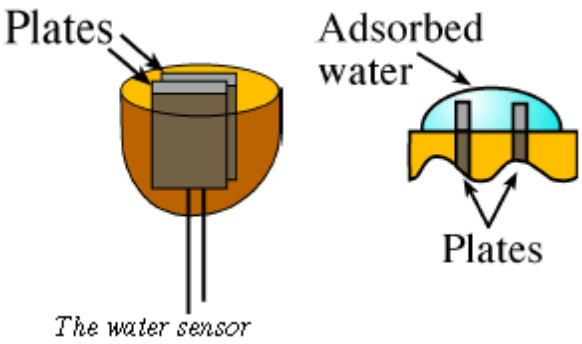
In this experiment you will test how the plug-in board works by lighting up the red lamp provided. It is referred to as a "light-emitting-diode" or LED.

1. Attach the battery clip to the 9V battery then plug the red wire into row X and the black wire into row Y.
2. Plug in the 330 Ω resistor to pinholes 1-X and 1-C.
3. Plug in the LED to holes 1-E and 1-F (longer of the 2 wires should be in 1-F)
4. Plug in the black jumper wire from 1-I to 1-Y

Does the red light come on? If not, you plugged in the LED backwards. The long wire from the LED should be in hole 1-F,not 1-E, which is because a Diode only lets electricity flow in one direction. We say it has a "polarity" because you have to pay attention to which side goes to (+) and which to (-) on the battery. In this last experiment, the resistor was needed to resist the flow of electricity. If you don't use it, too much electricity flows through the LED, causing it to burn out. The resistor is called a "current limiter" because it limits the flow of current, or electricity, in the circuit. The diagram for the circuit you just built is as follows:



Experiment #2 A Water Vapor Sensor

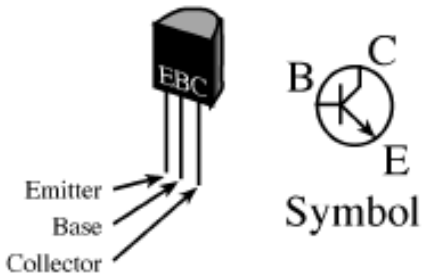


The water sensor

The water sensor. In this project you use a transistor to amplify the signal from the humidity (water) sensor. The humidity sensor works by electrical conductivity; when water vapor condenses on the plates it forms a thin film that conducts electricity between the two plates. In that sense the water sensor is like a resistor except that its resistance value changes depending on the amount of water in the air. It was made by clipping the top off of a 30 pF capacitor using a pair of wire clippers.

Transistor.

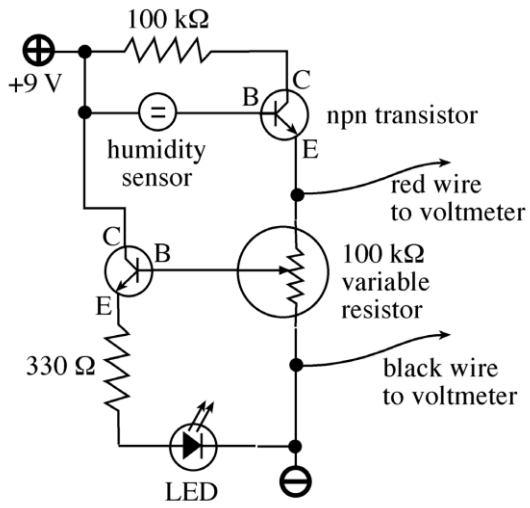
A transistor is an amplifier that will convert the very small change in resistance from the humidity sensor into a larger resistance change that you will measure with your voltmeter. It has three connections; the collector, the base (also called the gate), and the emitter. The collector and the emitter are like the connections to a pipe that carries water, and the base is like the faucet. Ordinarily the base is closed and the resistance between the collector and emitter is very large. When a small voltage is applied to the base, it reduces the resistance between the collector and the emitter, allowing current to flow. Your kit contains a transistor which is marked with the letters E, B, and C, which stand for emitter, base, and collector, respectively.

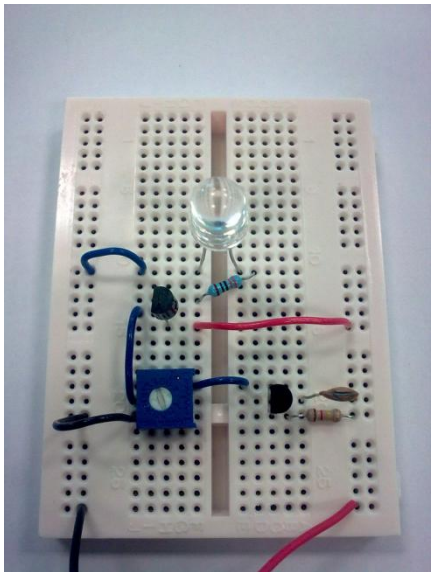


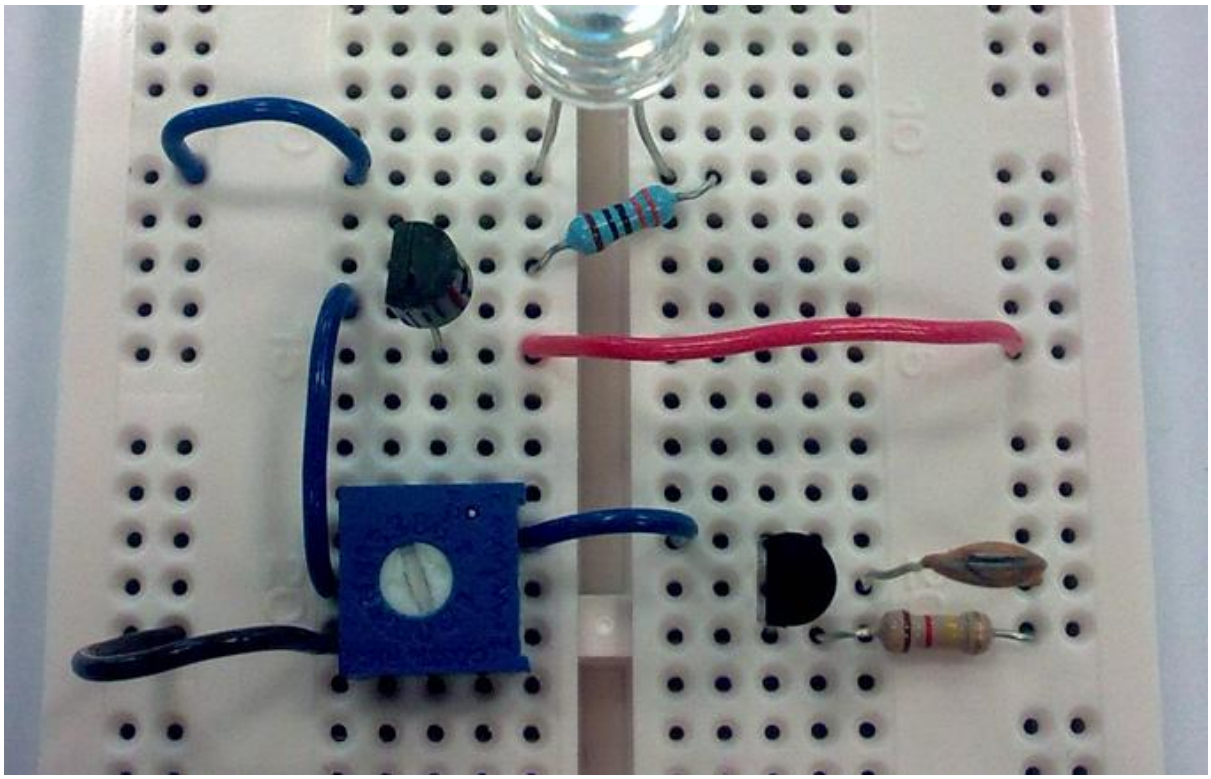
an npn transistor

Wiring up the water sensor

In this part of the experiment, you will create a water vapour sensor with a red LED in the circuit as an over-limit alarm. The LED will light up when the sensor detects a high amount of water vapor. The circuit diagram is shown below, along with a photograph of the circuit.

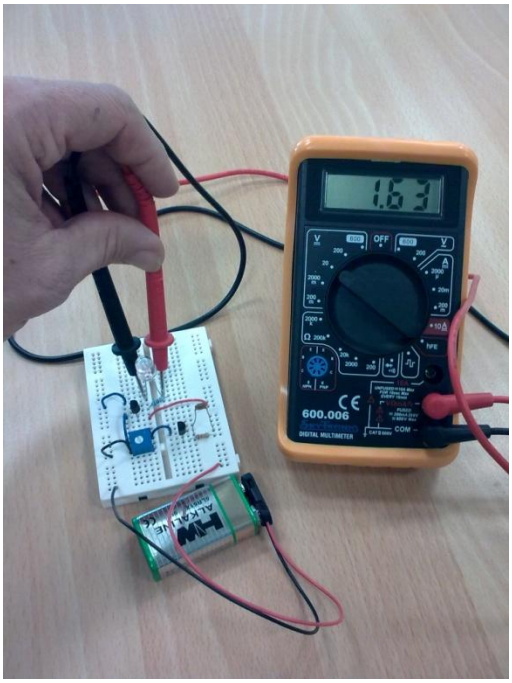






The voltage signal output from the first transistor is sent to the gate of the second transistor. The circuit also incorporates a 100 kΩ variable resistor which allows you to set the threshold level for the over-limit alarm. This circuit is sensitive enough to detect the water vapor on your breath, and can even detect the water evaporating from your skin. If you place your finger close to the sensor without touching it the red light should come on. You will be able to adjust the threshold level where the red light comes on by adjusting the dial on the variable resistor using the paperclip provided. If you find that the red light stays on gently rub the surface of the sensor with some tissue paper to get rid of excess moisture.

You can also measure the voltage reading by touching the probes of the multimeter to the base of the LED legs as shown. You should notice a small change in the voltage reading when you breathe on the sensor.



QUANTICORP R&D PART 2: PRESENTATION

Task 2.1 Preparing a presentation for the Chief Executive (30 min)

Your next task is to prepare a presentation for the Chief Executive (CE). She is a busy woman and you will be up against the other teams in the R&D department who want their ideas to be taken on, so you have only five minutes to convey the key information. This is called an 'elevator pitch', so you need to make sure that you get across key points of relevance to the CE. To do this you are going to use a tool called the 'Message Box' (**Resource 2.1**):

Issue

Write down the issue that you need to address, e.g. 'developing capability to produce a new sensor based on conducting polymers'.

Problems

What are the main problems that you expect to encounter when addressing the issue?

Solutions

What are the possible solutions to the problems?

Benefits

Assuming that you are successful what are the direct benefits of your idea, e.g. scientific and production outcomes.

Relevance to audience

In this case the audience will be the Chief Executive so you have to tell her what relevance this has the company's strategic vision and bottom line.

You now have the basic information for a short (**3 min**) presentation which you will present to the Chief Exec.

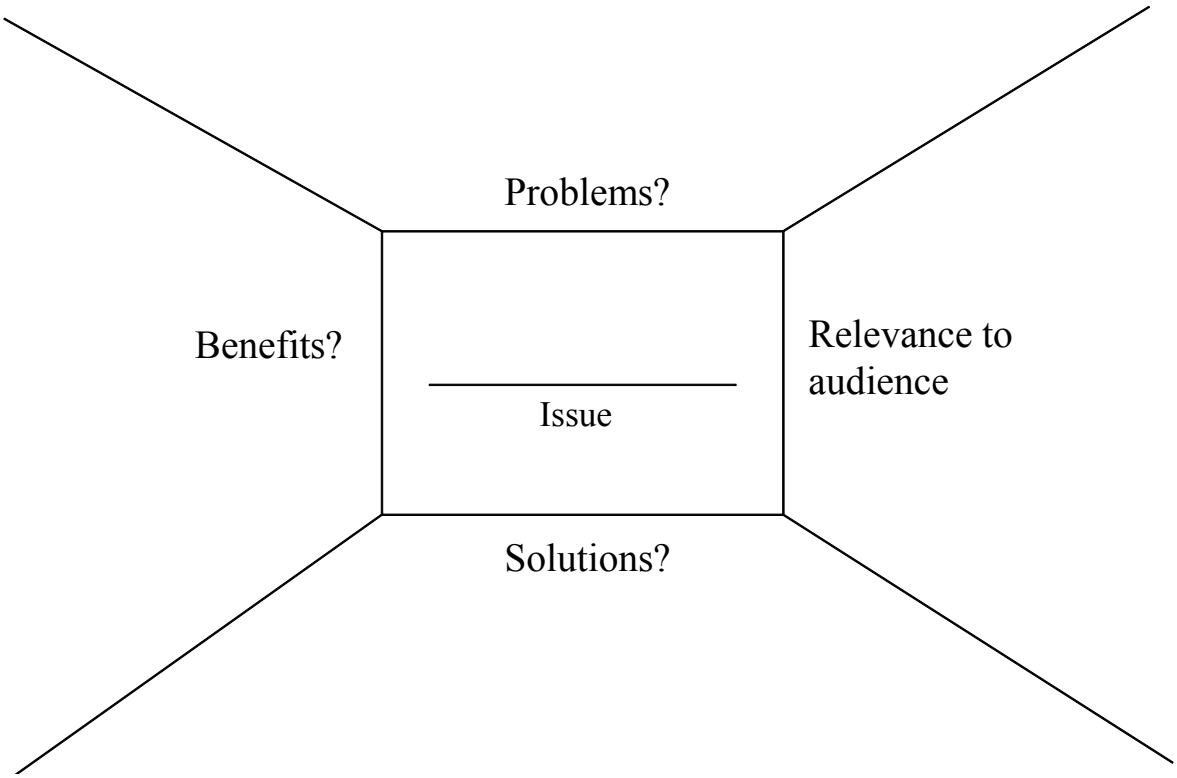
Task 2.2 – presentation to the Chief Executive (60 min)

You have 3 minutes to present your idea to the C.E. Each member of the team should address one of the points in the message box. Remember, your pitch should be clear, concise and coherent, and directly address your remit.

RESOURCE 2.1

Message Box:

Take a few minutes to fill in a one-sentence description of the issue and four supporting facts/arguments which answer the listed questions.

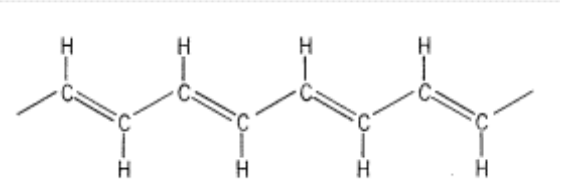


QUANTICORP R&D PART 3: CONDUCTING POLYMERS

Market research has identified the following area where there is a market niche:

- A glucose sensor

You initial research has identified the conducting polymer polyacetylene (1) as a possible basis for this type of sensor.



1

You now need a problem solving strategy to guide you in the development. A simple but powerful framework is given below.

1. Identify the problem and decide what you want to achieve:
    a. identify a conducting polymer for development into a sensor;
2. Plan how you want to proceed.
3. Establish what you already know, what you don't know and what you need to find out:
    a. research into synthetic methods;
    b. functionalization of the polymer;
    c. synthesis of the polymer;
    d. conjugation with a sensing molecule;
4. Come up with some solutions, alternative approaches and new ideas
5. Critically evaluate your solutions and decide whether any of them solve the problem
6. Repeat 3-5 as many times as necessary

Task 3.1 – Planning (30 min)

You have been given a specific problem but before going any further you should decide on a plan of action. In your plan you should decide:

- Who will do what
- How the team will communicate and share information
- How often and where you will meet
- A framework and process for solving the problem (it is suggested that you use the framework given above)

Task 3.2 – Research into synthetic methods (30 min)

Your laboratory is equipped with basic laboratory equipment for undertaking synthetic organic chemistry but you only have a limited budget and need an answer quickly . . . so you resort to Wikipedia http://en.wikipedia.org/wiki/Polyacetylene

Answer the following questions and copy the trail of Wikipedia links into the grid below (the first one has been done for you and make sure that you only copy words which are links to another page). You will need to read the whole text of the entries and click forward and back between some of them in order to get all the answers and links:

1. What is the name of the compound most commonly used to prepare polyacetylene?
2. What is the most common synthetic route?
3. What is the name of the catalyst used in this polymerisation method?
4. What will this catalyst tolerate?

polyacetylene

Now go to Google Scholar http://scholar.google.co.uk and enter the text of the links into the search bar.

Within the top ten hits there should be a relevant article by Scherman *et al.* published in 2003 in the *Journal of the American Chemical Society*. Download the full text.

Task 3.3 Functionalization of the polymer (30 min)

Refer to the article you downloaded in Task 3.1 and extract the following information:

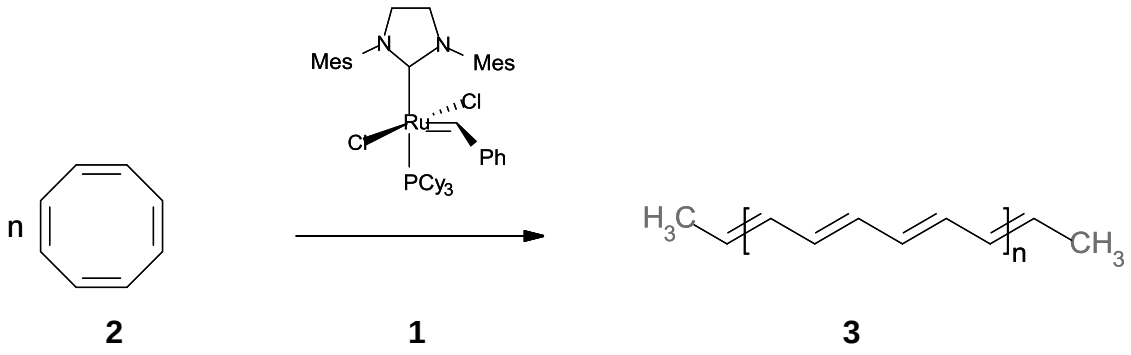
1. Outline the reaction scheme for the production of telechelic polyacetylene with functionalized ends by using a chain transfer agent;
2. State the main advantage of the particular catalyst used;
3. Give the structures of the different CTAs that were used.

QUANTICORP R&D PART 4: SYNTHESIS OF POLYACETYLENE

In order to test the feasibility of scaling up production of polyacetylene you first need to conduct a lab-scale synthesis. This is step 3c in your problem solving strategy:

1. Identify the problem and decide what you want to achieve:
    a. identify a conducting polymer for development into a sensor;
2. Plan how you want to proceed.
3. Establish what you already know, what you don't know and what you need to find out:
    a. research into synthetic methods;
    b. functionalization of the polymer;
    c. synthesis of the polymer;
    d. conjugation with a sensing molecule;
4. Come up with some solutions, alternative approaches and new ideas
5. Critically evaluate your solutions and decide whether any of them solve the problem
6. Repeat 3-5 as many times as necessary

The basic reaction yields solid polyacetylene (Scheme **1**), but the polymer can be end-functionalized using a chain transfer agent (CTA)



2 **1** **3**

Scheme 1 ROMP of COT reaction

Task 4.1 (120 min)

A survey of the literature has turned up some useful sources of information:

A paper by Moorhead and Wenzel[2] describes the preparation of polyacetylene using the Ring opening metathesis and the Grubb's second generation catalyst, and subsequent investigation of the structure of the polymer.

(a) What was the name and full structure of the CTA used for Experiment 2?
(b) Why was it used?

[2] 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.

Now read the paper by Scherman *et al*.[3]

(c) Summarise the experimental procedure used for the production of $TBSOCH_2$-capped polyacetylene (in solution).
    (i) Which reaction conditions, using CTA **5a**, gave the best and worst yields?
    (ii) How was the CTA reagent **5a** synthesised?

How would you investigate this reaction with respect to:

(d) Reaction conditions
(e) Stoichiometry
(f) Structure of the product
(g) Conductivity of the product

[3] 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.

QUANTICORP R&D PART 5: SYNTHESIS OF POLYACETYLENE

Task 5.1 (120 min)

Design an experiment to investigate the yield for the synthesis of $TBSOCH_2$-capped polyacetylene. You will need to apply your problem solving strategy to:

(a) decide an overall reaction scheme;
(b) investigate the yield;
(c) decide on the apparatus and any special requirements that you will need;
(d) decide on the reagents and any special preparation that they require;
(e) complete a COSHH evaluation of the chemicals.

STUDENT NOTES FOR TASK 5.1

Optional handout

Reagents

The experiment can be performed effectively with reagents prepared at the following concentrations:

- Grubb's 2nd generation catalyst (**1**) – a 7.5 mg cm^{-3} solution in CH_2Cl_2
- COT (**2**) – a yellow liquid
- TBS-protected *cis*-2-butene diol (**5**) – a clear liquid which has been previously synthesised.

Dry CH_2Cl_2 is also required for preparation of the catalyst solution and solubilising the polymer.

Investigating the stoichiometry of the reaction

The effect of reagent concentration on yield can be investigated by students working in six pairs with different ratios of reactants, and pooling the information afterwards. Prepare **six** different reaction mixtures as outlined in Table **1**.

Table 1 Reaction mixtures

Reaction	**Amount of reactant /** cm^3		
	X (5)	**Y (2)**	**Z (1)**
1	1.6	0.5	1.0
2	1.6	0.75	1.0
3	1.6	1.0	1.0
4	1.6	0.5	0.5
5	1.6	0.5	1.5
6	-	0.5	1.0

The molar masses and densities of the reagents are as follows:

COT (2)	M_r = 104.15 g mol^{-1}; ρ = 0.9250 g cm^{-3}
Grubbs 2nd generation catalyst (1)	M_r = 848.97 g mol^{-1}
CTA (4)	M_r = 316.57 g mol^{-1}; ρ = 0.8587 g cm^{-3}

Procedure

Pipette X cm^3 of (**5**) into a 15 cm^3 Quickfit test-tube, seal with a Suba-Seals® and flush with nitrogen. Add Y cm^3 of (**2**) using glass syringe fitted with a stainless steel (s/s) luer-lock needle. To this mixture add Z cm^{-3} of a 7.5 mg cm^{-3} solution of (**1**) in CH_2Cl_2 using a glass syringe fitted with a s/s luer-lock needle (**NB use separate syringes and needles for each of the reagents)**. The brownish yellow solution should become dark orange after a few minutes. Transfer to a heating block at 55 °C and allow to react for 24 h. Remove and pour the contents into a 150 cm^3 QF conical flask containing 100 cm^3 of methanol, with stirring. Filter the deep red suspension using a 1.8 cm diameter Hirsch funnel and a 250 cm^3 Buchner flask. Allow to dry

under vacuum overnight, record the mass of polymer produced and calculate the yield.

Performing the experiment in an inert atmosphere

The following equipment can be used to provide an inert atmosphere in the reaction vessel. Provide this on the bench and advise students on its use as appropriate:

- Balloons that can be filled with nitrogen
- Luer fittings (cut down plastic syringes) that can be attached to the balloons
- Nitrogen cylinder with an appropriate luer fitting for filling the balloons
- Gas-tight luer-tipped cannulas for gas transfer from balloon to reagent vessels
- Quick-fit test tubes (15 cm^3) with Suba-Seals®

Attach the inert gas balloon to a Luer fitting (cut down plastic syringe). It is a good idea to place a layer or two of Sellotape or blueroll between the balloon and retaining clip to prevent pinching the balloon rubber.

Slowly fill your balloon with the appropriate gas at the cylinder located at a fume cupboard. Either pinch the neck of the balloon with your fingers or cap the Luer fitting with a cork-stoppered needle to retain gas until you return to your apparatus.

Quickfit vessels are often capped with Suba-Seals® for this type of work. Before puncturing the seal it is advisable to make a marker pen dot at the location you're going insert a cannula or needle as this will make relocating the puncture point much easier and negate compromising the seal with many random puncture sites.

Remember that if you are purging a vessel you will need somewhere for gas to escape! When using a cannula as a vent, it is advisable to confirm that this is actually occurring by dipping the open end in a small beaker of solvent or water. Never cause a sharp bend or constriction in a cannula.

RESOURCE 5.1

COSHH Assessment

Faculty/Department:	School/Section:
Assessment No.	Assessor:
Description of procedure or experiment *(Include how long and how often this is carried out and the quantity of substance used)*	

Identify the persons at risk:	Staff ☐	Contractors ☐	Public/students ☐
Name the substance involved in the process, the supplier (if known) and the information source.			

Classification *(state the category of danger, tick all that apply)*

 ☐ Very Toxic	 ☐ Irritant	 ☐ Extremely Flammable
 ☐ Toxic	 ☐ Sensitising	 ☐ Highly Flammable
 ☐ Corrosive	 ☐ Biological	 ☐ Flammable
 ☐ Harmful	 ☐ Oxidising	 ☐ Environmental

Hazard Type (tick all that apply)

☐	☐	☐	☐	☐	☐	☐	☐
Gas	Vapour	Mist	Fume	Dust	Liquid	Solid	Other (State)

Route of Exposure (tick all that apply)

☐	☐	☐	☐	☐
Inhalation	Skin	Eyes	Ingestion	Other (State)

Workplace Exposure Limits (WELs) *please indicate n/a where not applicable*

Long-term exposure level (8hrTWA):	Short-term exposure level (15 mins):

State the Risks to Health from Identified Hazards (include Risk Phrases)

Control Measures: *(for example extraction, ventilation, training, supervision). Include special measures for vulnerable groups, such as disabled people and pregnant workers. Take account of those substances that are produced from activities undertaken by another employer's employees. (include Safety Phrases)*
Can a less hazardous substance be used? Yes ☐ No ☐ If so why is it not being used?
Is health surveillance or monitoring required? Yes ☐ No ☐

Personal Protective Equipment *(state type and standard)*			
 ☐ Dust mask		 ☐ Visor	
 ☐ Respirator		 ☐ Goggles	
 ☐ Gloves		 ☐ Overalls	
 ☐ Footwear		 ☐ Other	

First Aid Measures

Spillage/Uncontrolled Release Procedures

Storage

Disposal of Substances & Contaminated Items
Hazardous Waste ☐ Skip ☐ Run to Drain ☐ Return to Supplier ☐ Other ☐ (If Other Please State):.....

Name of Assessor:	Signed:	Date:
Name of Safety Manager/ HOS/ HOD:	Signed:	Date:
Review Date:	Signed:	Date:
Review Date:	Signed:	Date:

Review Date:	Signed:	Date:

QUANTICORP R&D PART 6: SYNTHESIS OF POLYACETYLENE

Task 6.1 (120 min)

Perform a synthesis of $TBSOCH_2$-capped polyacetylene.

QUANTICORP R&D PART 7: SYNTHESIS OF POLYACETYLENE

Task 7.1 (180 min)

Using the analytical techniques identified in Task 4.1 to investigate the structure of your product.

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STUDENT NOTES FOR TASK 7.1

Solubility

It is necessary to dissolve the product in an appropriate solvent in order to perform some of the spectroscopic investigations. Test the solubility of each of the products from reactions 1-6 in the following solvents:

- CH_2Cl_2
- Pentane
- Acetone
- Ether

UV-vis Spectroscopy

Add ~1 mg (a small speck) of your red solid to approximately 4 cm^3 of an appropriate solvent (discuss this with the tutor before proceeding) and record the UV-Vis spectrum between 250 and 600 nm.

IR Spectroscopy

Record the IR spectrum of ~ 2mg your red solid.

NMR Spectroscopy

Dissolve 10 mg of polymer in an appropriate solvent (discuss this with the tutor before proceeding) and acquire a proton NMR spectrum. Integrate the polyene (δ ~ 6-7 ppm) methylene (δ ~ 4.2 ppm) backbone peaks.

QUANTICORP R&D PART 8: REPORTING

Task 8.1 (180 min)

Prepare a report of your investigation. This should include a full experimental procedure and COSHH assessment. Address the following in your discussion:

Analysis of the Product

NMR

Using your spectrum for the polymer product, ratio the integrals for the polyene and methylene peaks and estimate the number of double bonds.[4]

UV-vis Spectroscopy

Conjugated compounds are those that contain alternating double and single bonds. Here the π orbital of the double bond overlaps with the σ orbital of the single bond. This allows for the π electrons to delocalise (become dissociated for one particular atom). As the amount of conjugation increases the π → π* transition becomes lower in energy, so absorption in the UV-vis spectrum occurs at longer wavelengths. It follows then, that the number of double bonds, and hence the length of the polyacetylene chain, can be estimated from the UV-vis spectrum.

A simple method for calculating λ_{max} for the π → π* transition is to follow the Woodward-Fieser rule, as follows:

1. A polyacetylene with two double bonds will have λ_{max} at 217 nm
2. For each additional conjugated double bond λ_{max} will increase by +30 nm increments

Use these rules to calculate the number of conjugated double bonds from the UV-vis spectrum of your sample of polyacetylene.

Refer to Tables V and VI, Figures 13 and 15, and the associated discussion on pages 7999-8000 in the paper by Knoll and Shrock.[5] Use this information to estimate the number of double bonds in your polyene.

Compare the three methods which you have used to calculate the number of double bonds and comment on their accuracy.

Now calculate the average number of monomer units per polymer chain

IR Spectroscopy

Compare your spectrum with that of Scherman *et al.*4 and identify the principal peaks in the FT-IR spectrum which correspond with the major features of the product polymer. Estimate the relative proportions of the *cis* and *trans* isomers.

Effect on Yield

Using your figure for the average number of monomer units per polymer chain, calculate the theoretical yield.

Calculate molar ratios of the reactants for each of the reactions 1-5. Discuss the effect on yield.

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4 Scherman, O. A., Rutenburg, I.A., and Grubbs, R.H., *J. Am. Chem. Soc*., 2003, **125**, 8515.

5 Knoll, K., and Schrock, R., *J. Am. Chem. Soc*., 1989, **111**, 7989

Conductivity of the Poylmer

Access the information on conducting polymers at the Nobel Prize site[6] by downloading the text file containing the advanced information [(advanced information) The Nobel Prize in Chemistry: Conductive Polymers]. Read the article and summarise, in your own words, how doping with iodine makes the polymer conducting (about *400 words*).

[6] The Nobel Prize in Chemistry 2000: Advanced Information http://www.nobelprize.org/nobel_prizes/chemistry/laureates/2000/advanced.html [accessed 25 Nov 2011].

QUANTICORP R&D PART 9: CONJUGATION

The final step in your problem solving strategy is to alter the polymer to make it sensitive to a specific analyte:

1. Identify the problem and decide what you want to achieve:
    a. identify a conducting polymer for development into a sensor;
2. Plan how you want to proceed.
3. Establish what you already know, what you don't know and what you need to find out:
    a. research into synthetic methods;
    b. functionalization of the polymer;
    c. synthesis of the polymer;
    d. **conjugation with a sensing molecule**;
4. Come up with some solutions, alternative approaches and new ideas
5. Critically evaluate your solutions and decide whether any of them solve the problem
6. Repeat 3-5 as many times as necessary

Before you can do this you first have to decide on the type of sensing molecule you will need to attach and the method of conjugation.

Task 9.1 Selectivity for glucose

Review Task 1.2 and list the ways which you identified to make the sensor more specific for the detection of a particular chemical.

The IUPAC recommendations on *Selectivity in Analytical Chemistry*[7].in particular the discussion of '*Useful interactions*' and '*Selectivities in methods*' gives examples of different types of selectivity shown in the matrix below:

Chemical reactions	Surface adsorption	Associate formation
Absorption of radiation	Electrochemical reactions	Enzyme reactions
Immunochemical reactions	Separation selectivity	Detection selectivity

In your teams, consider the following questions for 10-15 minutes:

1. Do any of these correspond with your findings?
2. Can you think of specific examples which demonstrate these different types of selectivity?

[7] IUPAC. Selectivity in analytical chemistry (IUPAC recommendations 2001). *Pure Appl. Chem*, 2001, **73**, 1381.

3. From your reading of the literature, which of these examples has the most obvious application for a glucose sensor?
4. Follow this link to the Protein Data Bank http://www.rcsb.org/pdb/101/motm.do?momID=77 and read the article on the 'Molecule of the Month'.

Task 9.2 Conjugation with the sensing molecule

A conjugation reaction is a synthetic reaction wherein a foreign molecule is covalently linked with an endogenous biological molecule to give a product known as a conjugate.

Biological molecules are like any other chemicals in that they have a variety of functional groups which can react with other molecules, so you can make use of these to carry out a variety of conjugation reactions.

1. Quickly brush up on your functional groups by matching the structures in Resource 9.1 to the names below:

Alcohol

Alkene

Amine

Amide

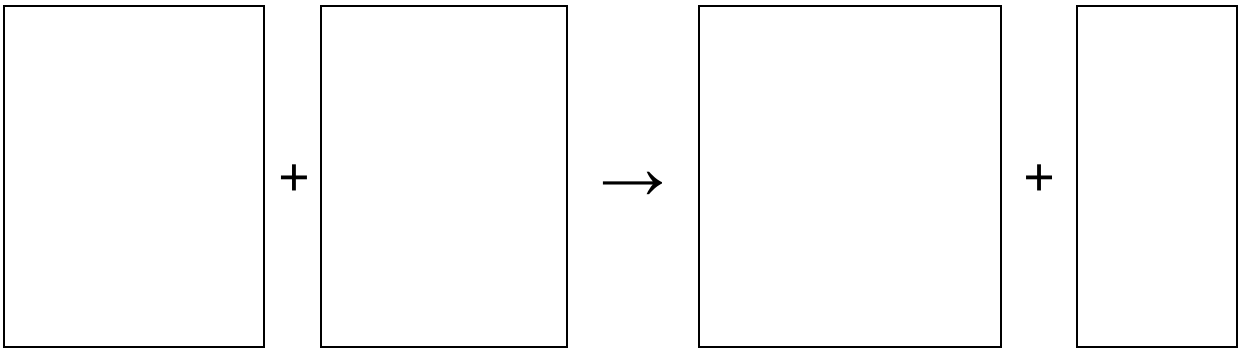
Carboxylic acid

Ester

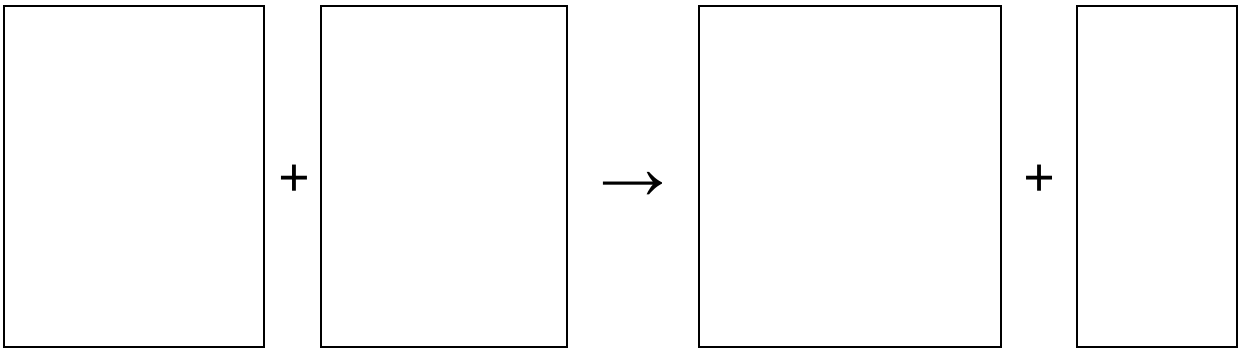
Hemiacetal

2. Now use Resource 9.2 to draw reaction schemes which match the following statements:

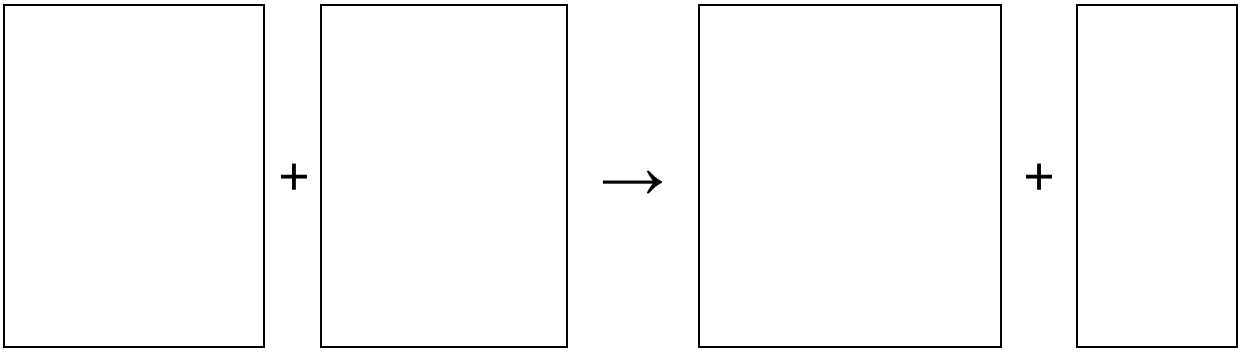
(a) Monosaccharides contain hemiacetal functional groups and undergo condensation reactions to form disaccharides.



(b) Amino acids contain carboxylic acid and amine functional groups and undergo condensation reactions to form peptides.



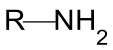
(c) Fatty acids and glycerol contain carboxylic acid and alcohol functional groups respectively and undergo condensation reactions to form triacylglycerols.

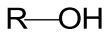


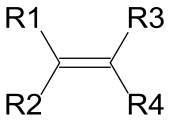
3. Follow the link to the Protein Database http://www.pdb.org/
   (a) search for *glucose oxidase*
   (b) scroll down to the entry for 1GPE GLUCOSE OXIDASE FROM PENICILLIUM AMAGASAKIENSE and click on the name;
   (c) Click on the *Sequence* tab at the top of the page and scroll down to view the protein chain sequence;
   (d) Note the first three letters in the chain - these are the 1-letter codes for the first three amino acids;
   (e) Look up the 1-letter symbols on the *Sigma-Aldrich Amino Acid Reference Chart* http://www.sigmaaldrich.com/life-science/metabolomics/learning-center/amino-acid-reference-chart.html and draw the structures;
   (f) Read the following extract from Wikipedia http://en.wikipedia.org/wiki/N-terminus and draw the structure of the tri-peptide formed from these three amino acids. Identify the N- and C- terminus;

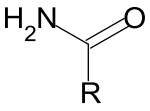
4. Suggest a possible reaction scheme for conjugation of glucose oxidase with another functional group.

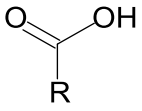
RESOURCE 9.1

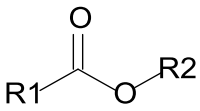


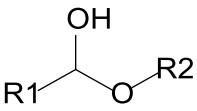




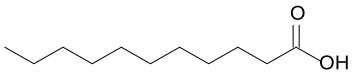


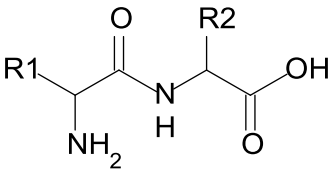


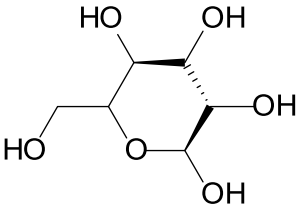


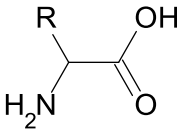


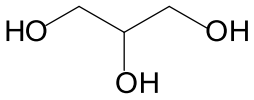
RESOURCE 9.2

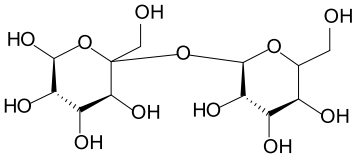

O
OH


O
R2
R1
N
H
OH
NH2
O


HO
OH
OH
HO
O
OH


R
OH
H2N
O


HO
OH
OH


OH
OH
HO
O
O
O
HO
OH
HO
OH
OH
OH

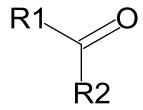
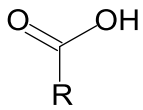
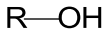
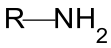
QUANTICORP R&D PART 10: CONJUGATION

Task 10.1 Review the reaction scheme for synthesis of your telechelic polymer (60 min)

- What is the definition of a telechelic polymer?
- What are the functional groups on the ends of the polymer?
- How were these introduced?
- Will this allow you to conjugate it with a protein?

Go to http://www.organic-reaction.com/organic-synthesis/protecting-groups/, find out the purpose of the functional group which you have used and complete the relevant row in the table below.

Find a similar reaction for the –COOH functional group and complete the row in the table.

		Reaction conditions	
Functional group	Structure after protection	Protection	De-protection
			
			
			
			

Propose a modified reaction scheme for synthesis of your telechelic polymer so that it can be conjugated with a protein.

QUANTICORP R&D PART 11: REPORT TO THE R&D TEAM LEADER

Task 11.1 Preparing a report for the R&D Team Leader (120 min)

Your final task is to prepare a technical summary for the R&D Team Leader describing the outcome of your investigation. You need to convince the Team Leader of the scientific and technical merit of your proposal so that it can be taken to the pilot stage, so the science must be convincing. You can use your problem solving strategy as a basis as follows:

1. Identify the problem and decide what you want to achieve:
2. Outline your plan
3. Present your solution
4. Summarise the scientific and technical basis for your solution
5. Critically evaluate your solution, including the benefits and potential risks of proceeding.

You will be advised whether this is to be in the form of a presentation or a written report.