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PRIFYSGOL
BANGOR
UNIVERSITY

**Royal Society of Chemistry
Analytical Division North West Region**

Schools Analyst Competition

March 2012

Experimental Handbook

RS•C
ROYAL SOCIETY OF CHEMISTRY



SCHOOLS ANALYST COMPETITION 2012

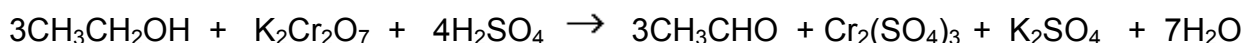
In this year's challenge your task is to measure the concentration of ethanol in a well known beer sample. You will be given a sample of a drink, the necessary laboratory equipment, and a method which you must use to measure the alcohol content.

At the end of the experiment your results will be assessed by the judges and they will select the winner on the basis of accuracy, precision and also the standard of your laboratory technique and data presentation. The 'true' value for the alcohol content has been worked out using the same method by department's staff, and your aim is to get as close as possible to this value. Please note that the method used may give a different value to that quoted on the bottle as this is based on a batch average

THEORY

This analysis is based upon the oxidation of ethanol by potassium dichromate (orange) under acidic conditions to produce acetaldehyde, (see reaction 1 below). The dichromate then oxidizes the acetaldehyde to acetic acid, (see reaction 2). As the dichromate oxidizes the various reactants, it is in turn reduced to Cr^{3+} which is green. The green colour is the result of a characteristic absorbance of light at a wavelength in the region of 600 nm.

Reaction 1



Reaction 2



The intensity of the green colour is measured using a spectrophotometer. The absorbance is directly proportional to the concentration of Cr^{3+} in the solution (the Beer-Lambert law). The concentration of Cr^{3+} is also proportional to the concentration of ethanol in the original sample. Measurements are made for a series of standard solutions containing a known concentration of ethanol and a graph is plotted of absorption against concentration. This graph, known as a

calibration curve, can then be used to calculate the concentration of ethanol in an unknown sample.

Because the beer sample will also contain other organic material, such as sugars, it is necessary to separate the ethanol by distillation prior to carrying out the oxidation. If this were not done the result would be higher than it should due to oxidation of these 'interfering' compounds.

Dr Tom Parry Jones OBE, an eminent Welsh entrepreneur and inventor, developed the world's first Electronic Breathalyser based on dichromate in 1974 and established Lion Laboratories to manufacture and market the products worldwide. It has since been replaced by more sophisticated electrochemical devices.

HEALTH AND SAFETY

It is essential that you read the following risk assessment, and are fully aware of the hazards associated with the materials you are using.

Hazard	Risk	Control Measures	First aid in case of accident
Acidified Potassium Dichromate solution	Highly corrosive, Oxidizing agent, Causes burns to skin and eyes. Toxic by ingestion.	Very small quantities (1 ml) issued in cuvettes. Substance must not be removed from the cuvette. Gloves, apron, and Safety glasses to be worn at all times	In case of contact with skin or eyes Rinse the affected area with cold water immediately for at least 5 minutes. Report the incident to a member of staff. Obtain medical advice
0.25% Ethanol solution	Harmful by ingestion, may cause dizziness and disorientation in excessive quantities. Serious long term chronic effects target organs Liver,	Small quantities issued (250 ml) to each group.	In case of contact with skin or eyes Rinse the affected area with cold water Report the incident to a member of staff.

	Kidneys, Heart and Brain.		
Beer and Wine samples	Harmful by ingestion, may cause dizziness and disorientation in excessive quantities. Serious long term chronic effects target organs Liver, Kidneys, Heart and Brain	Issue of small quantities closely supervised by staff.	In case of contact with skin or eyes Rinse the affected area with cold water Report the incident to a member of staff

Boiling water	Burns caused by spillage, splashes or contact with liquid or steam	Eye protection worn and tongs used to handle hot items. supervision and assistance of trained staff at all times. Solutions should be allowed to cool before handling.	Wash affected area under cold water and seek first aid assistance immediately
Glassware	Glassware breakage causing cuts	All glassware is pre-checked by technical staff and any damaged equipment should be returned. Care to be exercised especially when pushing rubber pipette fillers on to glass.	Contact a member of staff and obtain first aid assistance immediately in the case of an accident

GENERAL LABORATORY SAFETY

Pay careful attention to the safety advice that will be given to you prior to working in the lab. Familiarize yourself with the location of emergency exits. Eating, drinking or smoking is not permitted in the lab and mobile telephones must be switched off. Whilst working in the lab you must only touch chemicals and equipment that are used in your experiment.

Do not run or fool around. You must report any accidents or incidents, however small, immediately to a member of staff.

Please ask one of the demonstrators or technicians if you have any questions.

CHEMICALS AND EQUIPMENT

Each team will be provided with;

EQUIPMENT

- 25 ml bulb pipette and pipette fillers
- Beakers (100ml x 2, 600 ml x 1)
- Glass stirring rod
- Graduated pipettes (10 ml, 5 ml)
- Measuring cylinder (50 ml)
- 100 ml round bottom flask and distillation apparatus (still head, condenser etc)
- Heating mantle or hotplate
- Small cork ring to support round bottom flask
- Stopper for 100 ml round bottom flask
- 100 ml volumetric flask with stopper
- Test tubes (x 9) and rack
- Small funnel
- Pasteur Pipettes and teat
- Graph paper, ruler, pencil
- Tongs
- Plastic 1 cm cuvettes (x 9) in a rack
- Marker pen (medium)
- Boiling chips
- Glass wool
- Foil

CHEMICALS

- Beer sample (labeled A,B,C etc)
- Ethanol solution (0.25 wt%)
- Acidified potassium dichromate solution **CARE!**
- Deionized water (in wash bottle)

EXPERIMENTAL PROCEDURE

1 – Distillation

Note down which sample your group has been issued with (A,B,C etc) and transfer approximately 25 ml of the beer sample into a 100 ml beaker. Stir the beer until most of the effervescence has subsided and any froth has disappeared. Using a graduated pipette transfer 5 ml of your beer sample into a 100 ml round bottom flask and add 40 ml water and a boiling chip. Place the flask in a heating mantle and assemble a distillation apparatus (Please ask the technical staff for help – you will not lose any points). Ensure that the water is flowing within the condenser and heat the flask. Place an insulating layer of glass wool followed by foil around the top of the round bottom flask. This is to help speed up the reaction. Collect approximately 25 ml of distillate in a graduated beaker. Do not allow the heated flask to dry out. (**Tip:** Whilst waiting for the distillation to finish you may wish to prepare the standards as detailed in section 2, also the water bath (see section 3) can be prepared to save time later)

Transfer the distillate to a 100 ml volumetric flask and rinse the beaker several times with small amounts of deionised water, pouring the rinses into the volumetric flask. Make the volume up to the 100 ml mark with deionised water. Stopper the flask and invert it several times to mix the contents

2 - Preparation of the standards and sample

Label nine test tubes as described in the table below. You are provided with a standard 0.25 wt% solution of ethanol. In each test tube prepare the following solutions using a graduated pipette.

Test tube	ml of deionized water	ml of 0.25 wt% ethanol	Resulting wt% ethanol
blank	2.50	0.00	0.00
Standard 1	2.00	0.50	0.05
Standard 2	1.50	1.00	0.10
Standard 3	1.00	1.50	0.15
Standard 4	0.50	2.00	0.20
Standard 5	0.00	2.50	0.25
Sample (reading 1)	Each of these three tubes will contain 2.5ml of your sample distillate only. Three replicate measurements can then be made		
Sample (reading 2)			
Sample (reading 3)			

3 – Oxidation of the sample and standards

A waterbath will be provided for you by the technical staff, take care when using as the hotplate and beaker will be approximately 80°C

Warning: Acid dichromate solution is highly corrosive and an oxidizing agent. Gloves and eye protection must be worn and the following operation must be performed in a fume cupboard using extreme caution.

Add 2.5 ml of acid dichromate solution to each of your nine test tubes. Place the test tubes into the water bath and once boiling re-commences heat the test tubes for 15 minutes. Remove the test tubes using tongs and allow them to cool to room temperature. Using a Pasteur Pipette carefully transfer enough solution from each tube to two thirds fill a disposable plastic cuvette. Note the position in the rack of each cuvette to avoid confusion. **DO NOT WRITE ON THE CUVETTES!**

4 – Measurement of absorbance

Take your cuvettes containing the standards and samples to the spectrophotometer and with the assistance of a demonstrator measure the absorbance of each cuvette at a wavelength of 590 nm. Record your results. The blank should be placed in the reference beam of the spectrophotometer. After recording your absorbances, pour the dichromate solutions into the labeled waste bottle. **Do NOT pour down the sink!**

5 – Treatment of results

You may now use the data that you have obtained to plot a graph of the absorbance against the concentration of each standard. You can then use the graph to read the concentration of ethanol in your sample. Remember that you initially took 5 ml of the beer sample and diluted the ethanol obtained to a total volume of 100 ml. You will need take this into account when calculating the ethanol concentration in the beer by multiplying the result by 20.

RESULTS SHEET
(to be handed in with your graph)

NAME OF SCHOOL _____

SAMPLE NAME _____

SAMPLE	ABSORBANCE	CONCENTRATION (%)
Standard 1		0.05
Standard 2		0.10
Standard 3		0.15
Standard 4		0.20
Standard 5		0.25
Sample (reading 1)		
Sample (reading 2)		
Sample (reading 3)		

Average absorbance for three replicates _____

Concentration of ethanol in unknown _____%

Concentration of ethanol in original drink* _____%

** Remember to multiply your answer by twenty to account for the dilution factor*

When you have completed your results sheet please hand it to the judges with your calibration graph

FOR JUDGES USE ONLY	
<u>Accuracy</u> (Actual value – Measured mean value) / Actual Value	
<u>Precision</u> (spread of replicate values)	
Graph presentation	

Laboratory technique (Observed during experiment)	
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