CHEMISTRY
THE NEXT GENERATION

Murder in the lab

1
ACTIVITY

14
4 HRS
100 STUDENTS
35 STUDENTS PER GROUP
14 SUPERVISORS
2740

background

This event gives students a flavour of state of the art analytical techniques that are used in an area of chemistry, which many students know something about from television – **forensic science**.

scenario

A body is found in the laboratory. Death by poisoning is suspected.

- **Suspect A** is a researcher working on the extraction of pharmaceutically active compounds from plants, grown at the university.
- **Suspect B** is a synthetic chemist working on the synthesis of medicines for treating cardiac problems, with a particular interest in synthesising analogues of phytochemicals.

pre-planning required

weeks before

- Ensure all chemicals are available and the equipment is working.
- Ensure all samples are prepared and in appropriately labelled vials: artificial saliva, coffee and soil.
- Contact your community police officer to ask if they can come and give a talk.

days before

- Brief your demonstrators – point out that they may be asked certain questions – these are highlighted with prompts on the student sheets.
- Trial all separations on each instrument used in the activity.

facilities required

Analytical laboratories, gas chromatography-mass spectrometry (GC-MS), high performance liquid chromatography (HPLC), inductively coupled plasma-atomic emission spectroscopy (ICP-AES), capillary electrophoresis (CE), an incident room and a lecture theatre for registration.

materials required

scene of crime

- 'body' – a postgraduate volunteer
- police tape
- soil
- mug (half filled with coffee)
- sample set per group: 1 x saliva (PVA-polyvinyl alcohol), 2 x coffee (from the coffee cup and control sample), 1 x extract of unknown, and 3 x soil (from laboratory floor and suspect's shoes)
- clipboard
- scenes of crime officers (SOCO)
- fingerprint kit

laboratory

- instrument hand-outs

incident room

- white/blackboard – with: photographs of victims and suspects, a list of useful telephone numbers (victim's family, forensic laboratory etc) and a results table with reference numbers for sample bottles etc and space for results to be added.

SAFETY

A risk assessment must be done for this activity.

Answers

1. HPLC shows an unusual peak, not expected in coffee. We saw caffeine as well
2. HPLC shows the same peak is in the saliva, we even saw a trace of caffeine there too!
3. GC-MS identifies it as brucine
4. CE shows that the compound is natural because of the enantiomeric ratio
5. ICP-AES shows it has extra minerals that are not in the soil found outside in the main campus
6. ICP-AES shows that they are plant nutrients, maybe from a growing medium – a glass house
7. Suspect A the natural product chemist! Plus, it matches some soil from suspect A's shoes
8. No, but it is useful evidence that will contribute to the case

This activity is based on an event developed by Dr Eileen Buckley-Dhoot, and delivered by Dr Neil Williams and Dr Gillian Lambe from Kingston University.

continued overleaf

Murder in the lab

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NOTE

Throughout the demonstration, in addition to technical information, the demonstrators should stress the importance of sampling, labelling and good laboratory practice.

SAFETY

Brucine is very harmful and it is advisable to obtain factual chromatograms and electrophoretograms for handing out, but alternatives can be used on the day.

Suggested timings for the day

Four groups of 20-25 students can be staggered throughout the day

09.30 Reception and welcome.

10.00 Introduction to the crime scene. Advise students to read the worksheets thoroughly before starting the activity.

10.15 Analytical laboratories

Sub-group 1
Activity/Demonstration
HPLC separation of the saliva sample and coffee sample (identify caffeine and an unknown).

Sub-group 2
Activity/Demonstration
GC-MS identification of unknown as brucine. Demonstration given and supplied with a chromatogram.

Sub-group 3
Activity/Demonstration
CE identification of unknown is run and separated into its enantiomers to establish if its source is natural or synthetic.

Sub-group 4
Activity/Demonstration (incident room)
ICP-AES analysis of soil from the laboratory floor and each of the suspects shoes.

11.15 Each sub-group reports back findings (incident room).

11.30 Sub-groups report back findings to the main group. Students are asked to make comments and suggest the identity of the murderer. The police officer summarises the evidence and arrests the suspect (on suspicion).

11.45 Fingerprinting by community police officer. Talk on role and career opportunities for chemistry graduates in forensic science.

12.45 Talks on career opportunities given by an industry representative. Alternatively a talk on the importance of a university education by a student ambassador.

analytical techniques

The following is a guide to what samples can be used and what to expect from the techniques used. The demonstrations should be done by suitably qualified demonstrators.

HPLC – samples run: coffee and saliva. This is used to establish if the coffee is contaminated. An unknown peak is detected. The same peak is also detected in the victim's saliva.

GC-MS – sample run: extract of unknown. The GC-MS is of the compound brucine. Brucine is an alkaloid closely related to strychnine and is found in some plant species.

CE – sample run: extract of unknown. For safety reasons it is best not to use brucine, so an analogue can be used for the chiral capillary electrophoresis demonstration. Natural and synthetic brucine are compared and the enantiomeric ratio shows that the form in the victim's coffee is natural.

ICP-AES – samples run: soil from the laboratory floor and soil from the victim's shoes. The demonstrator should also discuss the mineral content of the local soil. The soil on the laboratory floor has unusually high levels of plant nutrients present – eg P, K, Mg etc.

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Murder in the lab

introduction

Many of you may have seen forensic scientists portrayed on the TV – *eg Waking the Dead* and *CSI*, but what exactly does a forensic scientist do? Basically, they apply science to the law. Forensic scientists are involved in a wide range of activities for examining physical evidence. **Scenes of Crime Officers**, known as **SOCOs** are involved in searching for evidence at crime scenes. This normally includes blood and other body fluids, textile fibres, paint, hair and even footwear. Some specialise in firearms, explosives and examining documents. Most forensic scientists are normally based in the laboratory but some are required to visit scenes of crime initially to look for evidence to take back to the laboratory.

the activity

In this activity you are forensic chemists in charge of the investigation into the suspicious death of a professor.

Professor Brown is found slumped across his desk in his office, he had been working on a research paper and drinking coffee, there is a small pool of saliva next to his face. When it is established he is dead the SOCO takes samples. She takes a sample of the coffee from the professor's cup, a saliva sample, and also finds some soil next to the professor's desk. She labels the samples carefully and seals them. She also looks for fibres, hair etc, she takes measurements and photographs. Enough information is gathered to be able to recreate the scene after it has been cleaned up.

Two suspects are later identified. Professor Green is a natural product chemist. She isolates pharmaceutically active molecules from plants she grows on campus, or collects in South America. Professor Black is a synthetic organic chemist and he is interested in synthesising phytochemicals. Both professors are questioned and both supply samples, including soil from their shoes.

Back at the laboratory you are given the samples. You need to record what they are and their serial numbers, then you need to analyse them.

Samples

- coffee
- one of saliva (artificial); and
- one of soil

You will use four analytical techniques in the laboratory. You will be assisted by analytical technicians and they will explain how the instruments work. It is your job to note down the results and any other observations that you think might be important to the investigation. You will then all report back to the commanding officer.

Analytical techniques

High pressure liquid chromatography (HPLC)

1. Run your coffee sample.

2. Compare it to a sample of coffee made using the same coffee machine with the same brand of coffee, this is known as a standard. Is there anything unusual about the professor's coffee?

3. Run the saliva sample. Are there any similarities between the two samples? Why do you think this is?

Gas chromatography – mass spectrometry (GC-MS)

1. Run the coffee sample and isolate any unknown peaks.

2. Use the mass spectrometer to identify any contaminants. The technician will also tell you if the saliva sample contained the same compound.

3. What have you found in the coffee? Was it in the saliva as well?

4. Ask the technician to extract any unknown compounds and pass them onto the capillary electrophoresis technician.

Capillary electrophoresis (CE)

The unknown compound (X) is extracted and the technician shows you the sample. The sample is run on the CE and separated into its enantiomers.

NOTE

Enantiomeric ratios may be different between synthesised and natural versions of a molecule.

Inductively coupled plasma – atomic emission spectroscopy (ICP-AES)

Do you remember that there was soil beside the professor's desk?

1. Run an extract of the soil on the ICP, the technician will explain how he/she prepared the sample. Is there anything unusual about the soil? How does it differ from ordinary soil found on a typical suburban campus? Where do you think it might have come from?

2. The technician will also show you spectra from the victim's and the suspects' shoes. Record any observations.

The theory of each of these analytical techniques is available on a separate sheet.

The students assemble in the incident room and report back their findings, the coordinator helps by asking leading questions:

1. Was there anything unusual about the coffee?
2. How did you find out if the victim actually drank the coffee?
3. What is in the coffee?
4. Was it natural or man-made?
5. What is unusual about the soil on the laboratory floor?
6. Where might it have come from?
7. Who had access to that facility?
8. Is the above enough for a conviction?

Further study

Study topics that could be based on the event.

Analytical Science
There were separation methods used in this event – *ie* chromatography and electrophoresis. Distinguish between these two methods by outlining the main differences between them.

MS
How was mass spectrometry used to identify the unknown peak?

ICP-AES
This is a way of detecting and quantifying the elements in the soil, it is based on atomic emission spectroscopy, find out what similar analytical techniques are available in analytical laboratory.

Organic chemistry
Natural product synthesis is an art as well as a science, find out about the work of the Nobel Laureates in chemistry Sir Robert Robinson 1947, Robert Burns Woodward 1965 and Elias James Corey 1990.

Forensic Science
When samples are collected from a crime scene, describe how you think they should be treated. For example, recording, storing, tracking etc.

SAFETY

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further information

More information on forensic science can be found in the following websites and books:

www.forensic-science-society.org.uk
www.forensic.gov.uk

Crime Scene to Court: The Essentials of Forensic Science published by the Royal Society of Chemistry 2004. ISBN 0854046569
Traces of Guilt, BBC publication 1998. ISBN 0563369647

Further information on forensic science courses in higher education is available in *Forensic science: Implications for higher education 2004*. SEMTA, 2004. SEMTA is the Sector Skills Council (SSC) for Science, Engineering and Manufacturing Technologies.

More information on the analytical techniques used in this activity are available in the Royal Society of Chemistry publications, *Modern chemical techniques* and *More modern chemical techniques.*
www.chemsoc.org/networks/learnnet/ModernChemTech.htm
www.chemsoc.org/networks/learnnet/more-chemtech.htm

Extension questions

1. What is a chiral compound? Draw an example and highlight the chiral centre.

2. How do synthetic chemists approach the synthesis of natural compounds? You could read about the Nobel Laureates: Sir Robert Robinson 1947, Robert Burns Woodward 1965 and Elias James Corey 1990.

3. How do the techniques chromatography and electrophoresis differ? How are they the same?

4. What is a buffer and why are they used?

5. Do you think it would be useful to interface a mass spectrometer to liquid chromatography or a CE? Explain why or why not.

6. Inductively coupled plasma spectroscopy is capable of exciting lots of elements at the same time and sorting out the characteristic radiation. Atomic emission spectroscopy is a sophisticated version of the flame test you may have done using a Bunsen burner. Describe the characteristic radiations you experienced when you looked at flame tests in school. Did you notice the more solution you had in the flame the more intense the colour?

7. Could you carry out any of the analysis in this event using an alternative analytical technique?

8. Why is sample labelling and record keeping so important? What could happen to the case if the scientist's records are incomplete or ambiguous?

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