CATCHING
THE DRUG CHEATS

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Science, Engineering, Technology and Mathematics Network, Registered in the UK No. 3236201 Registered as a Charity No. 1058056
There is a lot of material in this pack. Teachers might find it useful to first read the Teacher notes introduction, and the student Challenge brief.

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YOUR CHALLENGE

Doping control is extremely important to help look after the integrity of sport and the health of sportsmen and women. It makes sure that they are safe and that competition is fair. This way only the most talented, honest and hard-working people in sport will win.

Unfortunately, sometimes these people are denied success due to others who have taken drugs to help them win. The doping control process aims to prevent this by regularly testing sportsmen and women to find out if they have taken any banned substances.

Your challenge is to produce a presentation proving you have the skills and quality to provide a proficient anti-doping laboratory service.

The challenge has two stages:

STAGE 1

Prove that you can assemble and use apparatus to perform…
- a titration
- Thin Layer Chromatography
- light microscopy
- specific gravity measurement
- colorimetry

This may require learning new techniques with the focus on proving you have the ability to follow instructions and manipulate apparatus rather than being concerned with the quality of your results.

STAGE 2

Prove that you can identify and/or measure unknown substances using…
- Qualitative analysis (microscopy, Thin Layer Chromatography or colour/spot test), or
- Quantitative analysis (specific gravity, colorimetry or titration)

Through your analysis you must demonstrate repeatability and reproducibility.

Both stages require not only carrying out the tests but providing information about your laboratory, methods and research evidence. You should refer to the Challenge Checklist for more information on what you must include.

THINGS TO CONSIDER

Workload – Although you need to make sure you meet the challenge brief, you should not take on too much work. Work in teams of about six people and plan carefully.

Organisation – Allocation of different aspects to team members could be very useful. For example: Quality control (making sure the right things are being done!); Chain of custody records; Security; Confidentiality; Making sure samples can be tracked; Making sure you know who carries out each test.

Evidence – Written, photographic, video, drawing, graphic

Presentation – Use scientific language and terminology; Understand the challenge and be able to talk about every aspect of it.
Quality – Success is not dependent on quantity of work. The quality of your work is extremely important. Therefore, if you are unable to carry out all techniques and investigations, this can be balanced by the quality of your reports.

Some helpful information can be found on the Factsheets. The Test Procedure sheets will provide you with instructions to follow.
CATCHING
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CHALLENGE CHECKLIST

ACCREDITATION FOR ANTI-DOPING TESTING
This is what your laboratory team needs to show, to prove that you are able to do the work. How well can you use tests to analyse samples, identify and/or measure specified substances? You can use photos, documents, videos, drawings, letters – whatever you think best.

THE PROCEDURES
You will need to show that you can carry out these procedures:
- a titration
- thin layer chromatography
- specific gravity measurement
- colorimetry
- light microscopy

THE TESTS
For each sample:
- select the correct test technique/procedure
- decide what substance you must identify or measure

Part A: Use microscopy, Thin Layer Chromatography and a colour/spot test to perform Qualitative analysis.
Part B: Use specific gravity, colorimetry and titration to perform Quantitative analysis.

As well as carrying out the tests, you should give more information – see the checklist.

LABORATORY ACCREDITATION CHECKLIST

1. RECORDS OF STAGE ONE
Reports on each of the procedures you carried out showing:
- your apparatus and using the apparatus
- results sheets and calculations
- any uncertainty about the results
- how many people did the same test and whether they came up with the same result

2. RECORDS OF STAGE TWO
For each Sample:
- a record
- if there is an Adverse Analytical Finding, include the data that support your conclusions
- the trace to the scientists who performed the tests
Reports on each of the procedures you carried out showing:
- results sheets and calculations
- any uncertainty about the results
- how many people did the same test and whether they came up with the same results
- how you decided if a test result was positive or negative
3. THE PEOPLE
- Chart showing your names and jobs
- Evidence that everyone knows their responsibilities for:
  - keeping to the Code of Ethics
  - keeping results confidential
  - Laboratory Internal Chain of Custody

4. THE LABORATORY
- Description of the building and laboratory space
- Evidence of emergency procedures, and good security (such as a controlled zone – where only the laboratory scientists are allowed)
- Equipment List (the apparatus that you used and anything else you think you might use)
- Evidence about chemicals (waste disposal complies with the law; Environmental, Health and Safety policies, to protect people and the environment)

5. CODE OF ETHICS
- Show that confidentiality was kept.

6. SECURITY
Laboratory Internal Chain of Custody records:
- Descriptions of how you kept Samples securely
- Descriptions of how you kept records securely
- Description of how computers are protected so that no-one can copy data (e.g. passwords)

7. RESEARCH ACTIVITIES
- Proof that you have carried out research on Doping Control with a presentation on its importance.
CATCHING THE DRUG CHEATS

THE WORLD ANTI-DOPING AGENCY (WADA)

Established in 1999, WADA promotes, co-ordinates and monitors the fight against doping in sport in all its forms, in all countries.

It is independent and composed and funded equally by sport and governments across the world. Its work includes scientific research, education, developing anti-doping capacities, and monitoring the World Anti Doping Code.

WADA works towards a world that values and fosters a doping-free culture in sport. Athletes are the focal point of WADA’s efforts – it strives for a level playing field, so that athletes concentrate on excellence through their natural talent: “playing true”.

WADA ACCREDITED ANTI-DOPING LABORATORIES

Anti-doping laboratories carry out sports doping control tests. Laboratories that wish to perform these analyses under the World Anti-Doping Code must achieve and maintain accreditation from WADA.

The International Standard for Laboratories gives the standards that must be met.

There are currently 35 laboratories around the world accredited to conduct human doping control sample analyses.

UK ANTI-DOPING (UKAD)

UK Anti-Doping is responsible for the UK’s anti-doping policy and is accountable to the Department for Culture, Media and Sport. UKAD ensures that sports bodies in the UK comply with the World Anti-Doping Code and tests athletes in more than 40 sports.

KING’S COLLEGE LONDON

King’s College London is one of England’s oldest and most prestigious universities. It is in the heart of London with more than 19,700 students, including 6,200 postgraduates. It is ranked as one of the world’s top 25 universities.

The Drug Control Centre at King’s College London was established in 1978 to analyse samples collected from human sports competitors. The Centre assists UK Anti-Doping with its anti-doping programme which covers events and training in the UK.

ACCREDITATIONS

The Centre, as well as being accredited by the World Anti-Doping Agency (WADA), is also accredited by the UK Accreditation Service (UKAS) as a testing laboratory.

It was accredited to the ISO Guide 25 quality standard in February 1997. The Centre was the first International Olympic Committee/WADA accredited laboratory to achieve this distinction.
There are lots of drugs that enhance performance. Drugs might improve strength, stamina or reaction speed, or they may help an athlete to remain calm and relaxed. Some help athletes to control their weight or increase their pain threshold. Further drugs can be taken to try to hide the fact that athletes have been using banned substances!

**TYPES OF DRUG**

Perhaps the most well-known drugs used by athletes are **Anabolic Steroids**. Examples include nandrolone, testosterone and stanozolol. Steroids are used to build muscles, increasing strength. Other types of drugs include...

- **Peptide Hormones** e.g. erythropoietin (EPO) and human growth hormone (hGH). They are used to increase fitness and stamina, some with similar effects to anabolic steroids.
- **Stimulants** e.g. amphetamines, pseudoephedrine and cocaine. They are used to boost energy and increase alertness.
- **Relaxants** e.g. beta-blockers and cannabis. They are used to relax, stay calm under pressure, and provide a steady hand.
- **Diuretics** These are masking agents, used to lose weight, and hide the use of other drugs.
- **Painkillers** e.g. cortisone and local anaesthetics. They are used to mask pain, increasing threshold.

**ALCOHOL**

Alcohol is a relaxant. However, it is not banned in all sports. Any sport where being too relaxed may be unsafe prohibits alcohol. Examples include those that involve potentially dangerous equipment, such as car racing, archery, and shooting.

**CAFFEINE**

Caffeine is a stimulant. It can be found in tea, coffee, energy drinks and even chocolate. Some people take caffeine pills. Although it is not a banned substance in most sports, a close eye is being kept on its use.

**BE CAREFUL**

The majority of athletes are honest and ‘clean’ (do not take performance enhancing drugs), but they have to be very careful about what they eat, drink and use to keep fit and healthy. They have to make sure any herbal remedies, supplements and medicines they take are free of banned substances. This not only applies to medication prescribed by their doctor, but also those on supermarket shelves – known as over the counter (OTC) remedies. Many OTC cold, ‘flu and cough remedies contain pseudoephedrine, a stimulant that is banned during competition.

**THERAPEUTIC USE EXEMPTION**

Sometimes banned substances can be allowed. It may be essential for an athlete’s health. Athletes apply for therapeutic use exemption (TUE).

A good example is insulin. Although this is on WADA’s list of prohibited substances, it is used to manage diabetes. Five-time Olympic gold medallist, Sir Steve Redgrave, is diabetic.
WHO IS TESTED?

Any professional athlete, whatever sport they represent, can be tested at any time. Due to this, athletes always have to notify doping control authorities of their whereabouts.

The majority of sports people are ‘innocent’. This adds to the argument that anti-doping testing can invade people's personal lives. However, it is a fact of life, particularly for the top, most successful athletes. It’s not their fault that this is the case. Testing is necessary because of those who participate in doping. Because of them, the likes of Usain Bolt (the world's fastest man) will always have their doubters.

In 2008, the year of the Beijing Olympics, it was reported that Usain Bolt was tested at least 11 times.

In 2003, when tennis player Roger Federer won his first Wimbledon title, he was reportedly tested a staggering 21 times. That’s nearly once every fortnight!

Needless to say, both Federer and Bolt have never had results to suggest any wrong-doing.

Professional athletes trust that doping control is there to support and help them. It is for the integrity of sport and the health of athletes. In doing so, everyone can be much more confident that those who are successful are the most talented and dedicated.

TESTING POSITIVE

At the Seoul Olympics in 1988, Canadian sprinter Ben Johnson tested positive for the steroid, stanozolol. As a result he was stripped of the gold medal that he had won in a new world record time.

Other well known athletes who have tested positive for banned substances are Australian cricketer, Shane Warne (diuretics), ex-Manchester United footballer, Jaap Stam (nandrolone) and British athlete, Dwayne Chambers (anabolic steroid, THG).

WRONGLY ACCUSED

One of the difficulties is that some banned substances can occur naturally in the body. There are guidelines on the amount of a substance that can be allowed, but the levels which occur in the body varies between individuals. Illness can affect this more.

Further still, anti-doping laboratories have to be sure that they handle samples very carefully. If not, this can affect substance levels.

In 1994, British 800 m runner Diane Modahl, was given a four year ban for taking testosterone. However, it was eventually accepted that during a time when her urine sample was not refrigerated, bacterial activity could have increased the testosterone level. Modahl was cleared of doping and her ban from international competition lifted. That same year, she won a Commonwealth Games bronze medal.
Scientists at anti-doping laboratories, such as Christiaan Bartlett from the Drug Control Centre at King’s College London, use highly sophisticated methods to detect and measure banned substances. Christiaan says he also, “juggles several projects at once, works to meet strict time deadlines, and has to be meticulous with a fine eye for detail.”

The most common techniques used are:

- High Performance Liquid Chromatography (HPLC)
- High Resolution Gas Chromatography (HRGC)
- Mass Spectrometry (MS)

An explanation of these can be found in *Instrumental analysis* at [www.chemguide.co.uk](http://www.chemguide.co.uk)

More often than not, one type of chromatography (and sometimes both) is used with mass spectrometry. These techniques can detect anabolic steroids, diuretics, stimulants, relaxants such as cannabis, and painkillers such as morphine, in samples of urine.

Detecting peptide hormones (eg. EPO) needs different techniques, such as immunoassay and isoelectric focusing.

Testing blood samples requires flow cytometry.

The pH and specific gravity of urine are also measured. Because urine samples are small, suitable specific gravity meters have to be used, such as palette refractometers.

There is a full list of techniques, and what each tests and measures, on the following pages (from the Drug Control Centre, King’s College London).
# Schedule of Accreditation

**issued by**
United Kingdom Accreditation Service  
21 - 47 High Street, Feltham, Middlesex, TW13 4UN, UK

**Drug Control Centre**

<table>
<thead>
<tr>
<th>Issue No: 018</th>
<th>Issue date: 09 April 2010</th>
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**Accredited to**
ISO/IEC 17025:2005

**Testing performed at the above address only**

## Detail of Accreditation

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<thead>
<tr>
<th>Materials/Products tested</th>
<th>Type of test/Properties measured/Range of measurement</th>
<th>Standard specifications/Equipment/Techniques used</th>
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<tbody>
<tr>
<td><strong>HUMAN URINE SAMPLES</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Chemical Analysis</td>
<td></td>
<td>Documented In-House Methods:</td>
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<tr>
<td>pH</td>
<td></td>
<td>Sample integrity check using pH meter</td>
</tr>
<tr>
<td>Specific gravity</td>
<td></td>
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<td></td>
<td>using liquid and gas chromatography coupled to mass spectrometry (LC-MS/MS, GC-MS, GC-MS/MS)</td>
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<td>using radioimmunoassay</td>
</tr>
<tr>
<td>Beta-2 agonists (b2ag)</td>
<td></td>
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<tr>
<td>Hormone antagonists and modulators</td>
<td></td>
<td>using gas chromatography coupled to mass spectrometry (GC-MS and GC-MS/MS)</td>
</tr>
<tr>
<td>Diuretics and other masking agents</td>
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<td>Enhancement of oxygen transfer</td>
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<tr>
<td>Stimulants</td>
<td>using gas chromatography and liquid and gas chromatography coupled to mass spectrometry (GC-NPD, GC-MS, LC-MS/MS)</td>
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<td>Beta-blockers</td>
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### HUMAN SERUM SAMPLES

<table>
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<tr>
<th>Human Growth Hormone (hGH) Ratio</th>
<th>Using luminescence detection</th>
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<td>Testosterone/Epitestosterone ratio and Epitestosterone</td>
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<tr>
<td>19-norandrostenedione</td>
<td>using gas chromatography coupled to mass spectrometry (GC-MS, GC-MS/MS)</td>
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<td>Salbutamol</td>
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<td>Cathine</td>
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<td>Pseudoephedrine</td>
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<td>Morphine</td>
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<td>Using luminescence detection</td>
</tr>
<tr>
<td><strong>HUMAN BLOOD SAMPLES</strong></td>
<td>Blood parameters: Haemoglobin concentration Percentage of reticulocytes Number of red blood cells (erythrocytes), Number of platelets, Number of reticulocytes, Number of all white blood cells (leucocytes), Haematocrit (erythrocyte ratio of total blood volume) Mean cell (erythrocyte) volume in total sample, Mean haemoglobin volume per Number of all red blood cells, Mean cell (erythrocyte) haemoglobin concentration</td>
<td>Using Flow cytometry (Sysmex Haematology analyser)</td>
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**{FACTSHEET} ANTI-DOPING TECHNIQUES**

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**CATCHING THE DRUG CHEATS**

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OR CHAIN OF POSSESSION

Doping Control Officers (DCO) are responsible for athletes’ samples until they reach the laboratory. Before sending the samples to the laboratory, the DCO’s job involves chaperoning athletes from notification until the sample collection is complete, to ensure that there has been no opportunity for any sample manipulation. You will find a chain of custody protocol used in many situations – for example, forensic evidence that may be used in court, environmental monitoring and, of course, for anti-doping checks. Its purpose is to show that there has been no opportunity for a sample to be tampered with. Records must show that the sample has been with a responsible person at all times. Each time the sample goes from one person to another, it is recorded. The form is dated and signed.

WHEN THE SAMPLE IS COLLECTED

CONTAINERS
The Doping Control Officer offers the athlete a selection of containers; the athlete chooses which one they want for their samples. Containers are sealed with a tamperproof seal.

SAMPLE NUMBER
Each sample container has a unique number. This number stays with the sample through all the analyses to the final report and then in storage.

FORM
The DCO and the athlete both check that the number on the form and the number on the container are the same. They record where the sample was taken and the time. They may also record other things – perhaps the athlete was unwell or was unable to supply enough blood or urine. They both sign the form.

TRANSPORT TO THE LABORATORY
Whenever the sample goes from one person to another, this is recorded on a form. This shows who was in charge of the sample (has Custody) at any time. When the DCO gives the sample to the courier, they both note the time/date and sign the form. When the sample arrives at the laboratory, the courier and lab representative note the time/date and sign the form. At the laboratory, the same care is taken: every time the sample goes from one person to another, the form is signed, with a note of the date and time.
CATCH THE DRUG CHEATS

FACTSHEET CHAIN OF CUSTODY

LABORATORY INTERNAL CHAIN OF CUSTODY

LOG-IN
Samples are logged in at the laboratory. Immediately, the sample is checked: are tamperproof seals intact? are proper signatures present? especially important for blood, how long has it been in transit? and so on.

STORAGE
Every time a sample is put into storage, or taken out, the time and date is recorded on the form with a signature.

ANALYSIS
Every time a scientist takes a portion of the sample (aliquot) to analyse, they note it and sign the form.

DISPOSAL
When a scientist disposes of the aliquots, they must use a safe method. The frozen sample can be destroyed after three months. If there is a problem, the sample may be kept for eight years. Everything is noted on the form, with a date and signature.

KEEPING RECORDS
All records on negative Samples are kept in secure storage for at least two years. All records on Samples with an Adverse Analytical Finding are kept in secure storage for at least eight years.
The International Standard for Laboratories ensures that all laboratories, across the world, work to the same high standard and report their results in the same way. WADA inspects the laboratories to check them.

**ACREDITATION HAS SIX PARTS**
1. Questionnaire
2. Pre-probationary test
3. ISO/IEC 17025 accreditation
4. Probation tests
5. Final proficiency test
6. Staying accredited

**BECOMING ACCREDITED**

1. **QUESTIONNAIRE**
The candidate laboratory:
   - lists staff and their qualifications
   - describes the laboratory building
   - describes the security arrangements for Samples and records
   - lists all equipment and materials
   - describes why the methods they use are valid
   - gives a business plan that shows how they will analyse 3000 Samples every year
   - lists the laboratory’s sponsors

2. **PRE-PROBATIONARY TEST**
If the candidate laboratory has to take a pre-probationary test, they must:
   - find the amount of Prohibited Substances in ten samples
   - write a test report for each of the samples
   - do this within fifteen days
WADA usually visits the candidate laboratory, at the laboratory’s expense.

3. **ISO/IEC 17025 ACCREDITATION**
The laboratory has to meet the International Standards ISO/IEC 17025.
This will show that the laboratory has proper quality standards for how it conducts tests.
An independent assessor will visit and then report, in English or French, to WADA.
4. PROBATION TESTS
During the probationary period, the laboratory must analyse twenty samples sent by WADA.

5. FINAL PROFICIENCY TEST
In the final proficiency test, the laboratory must:
- analyse twenty more samples with WADA representatives present
- carry out tests within five days of opening the samples
- write a Test Report for each sample
- provide a Laboratory Documentation Package, if there is an Adverse Analytical Finding
The final test assesses both scientific competence and how well they could deal with many samples. Costs for the WADA visit are at the laboratory’s expense.

6. STAYING ACCREDITED
(See also Code of Ethics Factsheet.)
- The candidate laboratory must abide by the Code of Ethics.
- All employees must show that they understand and will abide by the Code of Ethics.
- The laboratory must send a letter to say they comply, signed by its Director.

OTHER ASPECTS

7. RESEARCH ACTIVITIES
The laboratory must have a plan and budget for research in the field of Doping Control.

8. SHARING KNOWLEDGE
The laboratory must show it is willing to share knowledge with other WADA accredited laboratories.

9. PROFESSIONAL LIABILITY INSURANCE
The laboratory must have an insurance policy for professional liability, for at least two million US$.

10. MORE DETAILS
See Appendix A for some of the details that a laboratory has to give.
STAYING ACCREDITED

1. BEFORE ANY SPORTING EVENT
At least one month before an Event, the laboratory must send a report to WADA showing:
- the contract between the laboratory and the Event organiser with the schedule of testing
- a chart for the staff with their job titles and responsibilities
- training plan for new scientists
- list of instruments and equipment, saying who owns them
- the criteria for deciding if results are positive or negative and what will happen to the results (Adverse Analytical Findings, Atypical Findings, etc.)
- how reports on tests will be kept secure

2. CONFIDENTIALITY
All reports must be kept confidential (as in the Code of Ethics).

3. DOCUMENTATION
- Each Sample must have its own record.
- If there is an Adverse Analytical Finding or Atypical Finding, the record must include the data to support the conclusions.
- Each step of testing must be traceable to the scientist who performed it.
- Each Sample report must be made within ten days (much less for some competitions).

4. SERVICES AND SUPPLIES
- Chemicals must be suitable for the analysis and be pure.
- Waste disposal complies with the law. This includes biohazard materials, chemicals and radioisotopes.
- There must be Environmental, Health and Safety policies, to protect staff, public and the environment.

5. CONTROL OF RECORDS
Results and reports on all Samples and the Laboratory Internal Chain of Custody records must be in secure storage.
APPENDIX A: ACCREDITED LABORATORIES – THE BASICS

1. STAFF
There should be a file for everyone, with their CV or qualifications, job description and training records. Everyone must know their responsibilities for laboratory security, confidentiality of results, Laboratory Internal Chain of Custody, and the standard procedures for any methods that they use.

The Laboratory Director must be qualified:
- PhD in science or medicine
- experience in the analysis of substances used in doping
- training in the forensics of Doping Control

The Certifying Scientist reviews all results and certifies the reports. Qualifications:
- BSc in Medical Technology, Chemistry or Biology [or 8 years’ experience in a Doping Control laboratory]
- experience in analysing biological fluids for doping materials
- experience in using techniques such as chromatography, immunoassay and mass spectrometry

Supervisors must understand all of the quality control procedures, including reporting test results and maintaining the Laboratory Internal Chain of Custody. Qualifications:
- BSc in Medical Technology, Chemistry or Biology [or 5 years’ experience in a Doping Control laboratory]
- experience in analytical testing including Prohibited Substances
- experience in using techniques such as chromatography, immunoassay and mass spectrometry

2. BUILDING
The laboratory building must have:
- air conditioning
- well-maintained electrical services and emergency power
- ways of keeping refrigerated or frozen samples safe, if there is a power cut
- good security, with controlled zones – authorised staff only with access and visitors recorded

3. TEST METHODS
The laboratory has to develop its own standard methods to detect substances on the Prohibited List.

Factors that show a method is fit-for-purpose:
- Specific – detects only the substance of interest
- Identification capability – detects and identifies a substance for many Samples
- Robust – the method gives similar results even when conditions are a little different
- Intermediate Precision – results are the same at different times and with different scientists
- Carryover – not carrying the substance from Sample to Sample
- Uncertainty – finding a Prohibited Substance is enough to report an Adverse Analytical Finding

4. DATA
The laboratory must prevent intrusion (hacking) and copying data from computers.
data (the plural of datum) – the facts and figures from practical tests and research
They provide the evidence for reports.

valid data – how true
Does a test measure what it is supposed to be measuring? If you are asked to find the strength of a magnet but you measure its length, then no matter how carefully you do it, you will not get valid data.

qualitative data – what
Observations are qualitative: ‘What colour?’

quantitative data – how much
Measurements are quantitative: ‘How long?’

accuracy – how close a measured value, such as mass or volume, is to the true value

precision – how close to each other are a number of measurements
You cannot describe a single measurement as precise!
Look at these four archery targets ...

sensitivity – the right size tool for the job
To measure the distance a long jumper jumps, it’s better to use a 10 metre tape than a 10 centimetre ruler. To measure somebody’s temperature, it’s better to use a clinical thermometer rather than a 0–360 °C thermometer.
uncertainty – the upper and lower values between which the true value is likely to be
No matter how carefully you measure something, there will always be a margin of doubt. You can say, “That loaf of bread weighs 1 kilogram, give or take a few grams”.
Look at the graduation marks on a measuring cylinder. In these diagrams, the graduations are 1 cm³ apart:

The volume of liquid is 24 cm³.

The volume is somewhere between 24 cm³ and 25 cm³ – perhaps about 24.6 cm³.
We cannot be sure and this is the uncertainty.

The greater the distance between the two graduations, the more confident you can be in estimating the volume.
It is why the graduation mark on a pipette is on its narrow neck.

repeatable data – a scientist repeats a measurement and gets the same results every time
reproducible data – another scientist repeats the same measurement and gets the same values
It is possible that one scientist gets the same results each time, but is making the same mistake every time. So, they compare their data with those that other scientists obtain. If they use the same procedures and get the same results, then the results are reproducible.

errors – these are a source of uncertainty
Some causes: a wrongly calibrated instrument, an unskilled operator, a problem with the method.

valid conclusion – it can be justified by the data and the methods you used to collect them
How confident are you in your results and conclusions?
ADAMS: Anti-Doping Administration and Management System maintained by WADA

ADVERSE ANALYTICAL FINDING: The laboratory report when a Prohibited Substance is found in a sample

ALIQUOT: A portion of a sample of urine or blood taken from an athlete

ANTI DOPING ORGANISATION: These include WADA and UK Anti-Doping

ATHLETE: Anyone who competes at any level in a sport controlled by its National Governing Body

ATYPICAL FINDING: An abnormal result that needs further tests (possibly leading to an Adverse Analytical Finding)

BLOOD COLLECTION OFFICER: A professionally trained officer, authorised to collect blood samples from athletes

CHAIN OF CUSTODY: The people and organisations responsible for a sample from the time it’s taken from the athlete until it is finally destroyed

CHAPERONE: Someone authorised to notify an athlete that they must submit to testing and to escort the athlete to the Doping Control Station

COMPETITION: A single race, match, game or other athletic contest

DCO: Doping Control Officer authorised to collect samples for Doping Control

DOPING CONTROL: All steps, from planning where, who and how athletes will be tested, through to the final outcome of any appeals. It includes organising athletes’ whereabouts information, sample collection and handling, laboratory analysis, TUEs, results management, hearings and appeals.

DOPING CONTROL STAFF: Doping Control Officer, Blood Collection Officer, Chaperone

DOPING CONTROL STATION: The place where the samples are collected

FAILURE TO COMPLY: When an athlete fails to submit to doping control

IN-COMPETITION: 12 hours before a competition until the end of sample collection for that competition

INDEPENDENT OBSERVER: A team who observe and advise on Doping Control

INTERNATIONAL-LEVEL ATHLETES: Athletes listed by a sports’ International Federation who must give up-to-date whereabouts information and make themselves available for testing when required

INTERNATIONAL STANDARD: The standard used by WADA showing how to carry out procedures properly
CATCHING THE DRUG CHEATS

{FACTSHEET} DEFINITIONS

LEAD DCO: The DCO who looks after the Sample Collection team, the Doping Control Station, sample collection and transferring the samples to the testing laboratory

MARKER: A chemical compound that shows up during tests if a Prohibited Substance or Prohibited Method has been used

NO ADVANCE NOTICE: Testing with no advance warning to the athlete

OUT-OF-COMPETITION: Testing which is not In-Competition

PROHIBITED LIST: List of prohibited substances and methods issued by WADA

PROHIBITED SUBSTANCE: Any substance on the Prohibited List

SAMPLE/SPECIMEN: Any biological material collected for Doping Control

SAMPLE COLLECTION EQUIPMENT: Apparatus used to collect or hold the athlete’s specimen

- To collect the urine sample as it leaves the athlete’s body – Sample bottle kits with sealable, tamper-evident bottles
- To collect a blood sample – tamper-evident Blood Sample Collection kit with needles and self seal

SAMPLE COLLECTION: All the activities from notifying the athlete until the athlete leaves the Doping Control Station

SAMPLE COLLECTION FORM: The form for recording information during sample collection

TAMPERING: Interfering with a sample to alter the results of tests

TESTING: All the activities from deciding where, who and how athletes will be tested, through to analyses at the laboratory

THERAPEUTIC USE EXEMPTION (TUE): Permission to use a Prohibited Substance for a legitimate medical condition

UK ANTI DOPING: United Kingdom Anti-Doping Limited, the anti-doping organisation for the UK

WADA: The World Anti-Doping Agency

WHEREABOUTS: Some athletes have to tell the authorities their whereabouts so that Out-of-Competition Tests can be carried out
EQUIPMENT

- measuring cylinder with a detachable plastic base
- cork to fit the measuring cylinder with a hole bored in it to fit the LED
- LED (light emitting diode)
- LDR (light dependent resistor)
- black electrical tape or black enamel paint
- 3 V battery
- 100 Ω resistor
- multimeter (set to read resistance)
- cardboard box to cover colorimeter (apart from the multimeter)
- glue

HEALTH AND SAFETY

If you need to do soldering, your teacher will tell you what to do and what precautions you need to take. Remember, a risk assessment must be carried out before you start.

METHOD

1. Take the detachable base off the measuring cylinder. If it has not already been done, drill two 1 mm holes in the base, 4 mm apart, one for each of the two LDR leads.
2. Put the LDR into the base, with its leads through the hole. Put a little glue around the holes to seal them.
3. Put the measuring cylinder back, pushing it down gently until it sandwiches the LDR. Cover the cylinder with black electrical tape. It’s important to make it light-tight.
4. Check the LED is working before it’s put into the black tape-covered measuring cylinder. If it doesn’t glow first time, try turning the battery round the other way.
5. Put the cork with the LED into the top of the measuring cylinder.
6. Connect the LDR to a multimeter set to read resistance.
1. CONFIDENTIALITY

No-one may comment to the media on individual results.

2. RESEARCH

Research must have proper ethical (e.g. human subjects) approval and follow the Helsinki accords.
Laboratories must comply with the laws on handling controlled (illegal) substances.

3. ANALYSIS

COMPETITIONS
Laboratories must only accept and analyse Samples if they know where they came from.
Laboratories should make sure that Samples were collected as in the World Anti-Doping Code.

OUT-OF-COMPETITION
Laboratories should accept Samples taken during training (Out-of-Competition) only if they have been collected and sealed properly as part of an official anti-doping programme.
Laboratories must not privately accept Samples from individual Athletes.
These rules apply to all sports.

CLINICAL OR FORENSIC
Occasionally the Laboratory may analyse a sample for a banned drug from an ill person, to help with diagnosis.
There must be a letter certifying that it is for medical diagnosis.
The Laboratory must take great care in work for forensic investigations. It should only give expert testimony if it does not question the science of the anti-doping programme.

OTHER ANALYTICAL ACTIVITIES
The Laboratory must not analyse products such as dietary supplements unless specifically asked to by an Anti-Doping Organisation.

SHARING INFORMATION
Laboratories must inform WADA immediately when they detect a new or suspicious doping agent.

4. CONDUCT DETRIMENTAL TO THE ANTI-DOPING PROGRAMME

- Laboratory staff should not have convictions such as fraud, embezzlement, perjury, etc.
- Laboratory staff must not advise Athletes on ways to mask detection of Prohibited Substances.
This remains valid for at least five years after a scientist leaves the Laboratory.
Scientists use microscopes to look at objects that are too small to see with the naked eye. At an anti-doping laboratory, they may examine suspicious powders and blood cells, for example.

You have probably used a hand lens or magnifying glass. It magnifies what you are looking at, so it looks bigger, but not by very much. A microscope has two lenses – one at the bottom, and another at the top. The bottom lens (the objective) magnifies the object you put underneath it. You look through the top lens (the eyepiece), which magnifies it again, so it looks even bigger. On some microscopes you can change the objective lens to alter the magnification.

You can use a microscope to examine two types of sample:
- transparent objects – those that light can shine through
- opaque objects – those where it can’t

You need to light these two types in different ways.
- For a transparent object you must shine the light through the sample from underneath, using the mirror.
- For an opaque object you must shine the light from above.

You focus the microscope by turning the focusing knobs. These move the lens closer to the object, or further away, until the object looks clear and sharp, not fuzzy.

**WARNING**

When focusing, it is best to start with the objective lens close to the sample slide, then gradually raise it. If you need to lower the lens again, be careful not to crush the slide.

**YOUR TASK**

You must show that you can:
- set up a microscope correctly for transparent and opaque objects
- position the object so that the correct part of it shows up
- focus the microscope to give a clear, sharp image
- describe in words and/or draw what you see
EQUIPMENT

- microscope
- prepared sample slide(s)
- transparent object
- opaque object
  
  (Your teacher will tell you what materials you are looking at.)
- lamp

PROCEDURE

If you are using a different type of microscope, you may need to change some of these instructions. Your teacher should tell you what to change.

1. Collect your equipment together near a window. Take care NEVER to point the mirror so that it could reflect the Sun directly – it could blind you.
2. Make sure that you can identify the parts shown on the diagram.
3. If the microscope has more than one objective lens, identify the lowest power. This is the shortest lens, marked with the lowest number (e.g. x4, rather than x10 or x40). Rotate this lens into position.
4. Turn the coarse focus knob to lower the objective lens as close to the stage as it will go without touching.
5. Place the sample slide under the clips on the stage.
6. Turn the mirror as shown in the diagram, so it reflects light up through the sample.
7. Look through the eyepiece. Slowly move the mirror backwards and forwards, left and right, until the light is bright and even. **(Note: If it's a dull day, you may need to shine the lamp onto the mirror instead of using daylight.)**
8. Move the slide around until you can see the sample. It will probably look very fuzzy. Slowly turn the coarse focus knob, to move the objective lens away from the slide, until the sample is in focus – that is, you can see the image clearly.
9. Slowly adjust the fine focus knob until the image is as sharp as possible.
10. Show your teacher that you have succeeded in setting up the microscope correctly.

You can move the slide around to examine different parts of the sample. You may need to readjust the fine focus.

Notice that when you move the slide, the image moves in the opposite direction. When you move the slide left, the image moves right. You need to remember this when looking for a particular part of the object.

To examine an opaque sample, leave out steps 6 and 7 (adjusting the mirror). Instead, shine the lamp onto the sample slide from above the stage.
Scientists measure **specific gravity** to compare liquids. The specific gravity of a liquid tells us how much heavier or lighter the liquid is, compared with the same volume of water.

\[
\text{specific gravity} = \frac{\text{mass of liquid}}{\text{mass of an equal volume of water}}
\]

Specific gravity (S.G.) has no units of measurement. It’s just a number, because it is one mass divided by another.

**QUESTION**

Why is the S.G. of water exactly 1.000?

Dissolving something in a liquid usually increases the liquid’s specific gravity. The higher the concentration of the solution, the higher the S.G. So measuring the S.G. gives us an idea of how concentrated the solution is.

Anti-doping tests include measuring the specific gravity of the athlete’s urine. They measure it as soon as the athlete gives the urine sample. The testing laboratory measures it again, and compares the two measurements.

**QUESTION**

Assuming both measurements are correct, why do they need to measure it again? What do you think they are checking for?

**METHODS OF MEASURING SPECIFIC GRAVITY**

We can measure specific gravity:
- by weighing an accurately measured volume of the liquid, or
- by using a hydrometer

A hydrometer is a weighted glass tube that floats in the liquid. The higher the liquid’s S.G. the higher the tube floats. So the scale increases downwards.

The scale can be confusing because:
- it is ‘upside down’
- it often shows only the last two figures
- some numbers appear twice, so you need to know what each one means

The hydrometer scale, right, reads from 0.980 to 1.120. The figures 80 and 00 near the top mean 0.980 and 1.000. The same figures near the bottom mean 1.080 and 1.100.

Using a hydrometer is a quick and easy method for you to measure specific gravity. However, the volume of urine taken from an athlete is too small for a hydrometer to float in. Instead, doping control officers will use hand-held refractometers – see the photo – which are easy to use outside the laboratory.
YOUR TASK
You must show that you can accurately determine the specific gravity (S.G.) of a liquid:
- by measuring its mass and volume, and calculating its S.G., and
- by using a hydrometer, if available

If a hydrometer is available, use both methods below to measure the same liquids. If you are working in a pair, use one method each, then swap over for the second sample. Compare your results.

EQUIPMENT
- balance
- 3 x 10 cm³ measuring cylinders
- 3 x droppers
- hydrometer, if available
- hydrometer tube (e.g., 250 cm³ measuring cylinder)
- sample liquids X and Y

PROCEDURE: MEASURING MASS AND VOLUME
For accurate results you need to measure volumes very carefully. You must use a dropper to give single drops (one at a time) and read the measuring cylinder correctly (at the bottom of the meniscus).

1. Collect your equipment together.
2. Draw up a results table like the one below. Use it for both methods.
3. Place a dry 10 cm³ measuring cylinder on the balance. Tare the balance, so it reads zero.
4. Carefully add just under 10 cm³ of water. Then add more, drop by drop, until you have exactly 10.0 cm³. The bottom of the meniscus must be level with the line on the cylinder.
5. Record the mass of 10.0 cm³ of water in the table.
6. Using another dry measuring cylinder and dropper each time, repeat steps 3–5 with samples X and Y.
7. Calculate the specific gravity of X and Y, as shown in the results table. Give your answers to three decimal places.

QUESTION
Why should you use a clean, dry measuring cylinder and dropper each time?
CATCHING THE DRUG CHEATS

{TEST PROCEDURE} DETERMINE SPECIFIC GRAVITY

PROCEDURE: USING A HYDROMETER

Read the notes on hydrometers above. Make sure you know how to turn the scale numbers on your particular hydrometer into specific gravity readings.

1. Collect your equipment.
2. Pour sample X into the hydrometer tube (or measuring cylinder) until about three-quarters full.
3. Carefully lower the hydrometer into the liquid. If it rests on the bottom, add more X until it floats.
4. Make sure your eye is level with the surface of the liquid.
5. Read the scale at the bottom of the meniscus (on the hydrometer, not on the measuring cylinder).
6. Turn the number into a specific gravity reading. (See below.)
7. Record the specific gravity of X in the table.

Example
Scale reading = 24
Specific gravity = 1.024

8. Wash and dry the hydrometer, and also the tube (or use another clean, dry tube).
9. Repeat the procedure for sample Y.

COMPARING RESULTS

Compare the specific gravities you obtained by each method.

QUESTIONS

1. Suggest reasons for any differences in the results for the same liquid using different methods.
2. Which method do you think is more accurate? Why?

RESULTS TABLE

<table>
<thead>
<tr>
<th>using weighing</th>
<th>using hydrometer</th>
</tr>
</thead>
<tbody>
<tr>
<td>mass of water (= m) g</td>
<td>specific gravity of X (=)</td>
</tr>
<tr>
<td>mass of X (= m) g</td>
<td>(m_{X} \div m_{water})</td>
</tr>
<tr>
<td>mass of Y (= m) g</td>
<td>specific gravity of Y (=)</td>
</tr>
<tr>
<td></td>
<td>(m_{Y} \div m_{water})</td>
</tr>
</tbody>
</table>
As its name suggests, a colorimeter measures colour – but note the different spelling!
When you dilute a coloured solution, such as Ribena®, its colour becomes weaker or paler. The higher the concentration, the stronger the colour; the lower the concentration, the paler the colour.
A colorimeter measures the strength of colour. We use this to work out the concentration of a solution.
Using a colorimeter is called colorimetry.

HOW A COLORIMETER WORKS
A light shines through the coloured sample solution. The solution absorbs some of the light – just like sunglasses do. The stronger the concentration and colour, the more light it absorbs.
The colorimeter detects the light passing through, and measures how much light the sample has absorbed. A higher meter reading means more light has been absorbed. Therefore the concentration must be higher.
To work out the actual concentration, you must calibrate the colorimeter. To do this you take readings with a series of solutions of known concentration.

WHAT IF THE SAMPLE IS COLOURLESS?
You can obviously use colorimetry to analyse coloured solutions, such as Ribena® or copper sulfate. However, the banned substances that anti-doping laboratories need to detect are usually colourless. Also, the concentration of a banned substance in urine will be very low. Even if it were coloured, the colour of the solution would be very pale.
However, we can still use colorimetry to analyse colourless or very pale substances. We add another chemical that reacts with the banned substance to give a coloured solution.

YOUR COLORIMETER
Proper colorimeters are expensive. This procedure uses a simple home-made version, made from:
- LED (Light Emitting Diode) – to act as the light source (instead of a bulb)
- measuring cylinder – to fill with the sample solution
- LDR (Light Dependent Resistor) – to detect and measure the light passing through the solution
- meter – to measure the resistance of the LDR
The resistance of the LDR decreases when light shines on it – the more light, the lower the resistance. When a coloured solution absorbs some of the light, less shines on the LDR. So its resistance increases, giving a higher meter reading.
If you use a commercial colorimeter, your teacher will show you how to operate it.
YOUR TASK

You must show that you can:

* set up a colorimeter
* calibrate it correctly
* use it to determine the concentration of a sample solution

You must first prepare a set of solutions of known concentration. You will use these to calibrate your colorimeter. You can then analyse the unknown sample.

EQUIPMENT

- colorimeter and multimeter
- 2 droppers
- rack of 6 test tubes labelled 10, 8, 6, 4, 2 and 0
- box to cover colorimeter
- unknown sample solution U
- 2 x 100 cm³ beakers
- 10 cm³ measuring cylinder
- rubber bung
- standard solution S

PROCEDURE

PART A: PREPARING THE KNOWN SOLUTIONS

1. Put 30–40 cm³ of coloured solution S into one beaker, and water into the other.
2. Pour about 9.5 cm³ of S into the measuring cylinder. Using the dropper, add more S to get exactly 10.0 cm³.
   Note: The bottom of the meniscus must be on the graduation line. If you go over the line, use the dropper to suck some out and try again.
3. Pour this into the test tube labelled ‘10’.
4. In the same way, start with about 7.5 cm³ of S, and top up to exactly 8.0 cm³.
5. Using the other beaker and dropper, carefully add 2.0 cm³ of water to make 10.0 cm³ of mixture. Pour this into tube ‘8’.
6. Put the bung in and invert the tube a few times to mix the solution well.
7. In the same way, make mixtures containing:
   * 6.0 cm³ of S + 4.0 cm³ of water for tube ‘6’
   * 4.0 cm³ of S + 6.0 cm³ of water for tube ‘4’
   * 2.0 cm³ of S + 8.0 cm³ of water for tube ‘2’
8. Measure out 10.0 cm³ of water only, and pour it into tube ‘0’.
PART B: CALIBRATING THE COLORIMETER

Note: If you are using a commercial colorimeter, ignore steps 2, 4 and 6. Your teacher will tell you how to alter the rest of the procedure if necessary.

1. Draw up a table like the one below.

<table>
<thead>
<tr>
<th>Tube</th>
<th>10</th>
<th>8</th>
<th>6</th>
<th>4</th>
<th>2</th>
<th>0</th>
<th>U</th>
</tr>
</thead>
<tbody>
<tr>
<td>Concentration (%)</td>
<td>100</td>
<td>80</td>
<td>60</td>
<td>40</td>
<td>20</td>
<td>0</td>
<td>U</td>
</tr>
<tr>
<td>Meter reading (Ω)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

2. Set the multimeter to whichever resistance (ohms, W) range your teacher tells you. Make sure you know which meter scale to read.

3. Pour solution ‘10’ into the colorimeter tube.

4. Fit the LED in the top. Then cover the colorimeter (but not the meter) with a box, to keep light out.

5. Switch on and wait for the meter needle to rise. Read the highest value reached, and record it in the table. Then switch off.

6. Lift the colorimeter tube out of the plastic base. Remove the LED.

7. Pour the solution back into tube ‘10’, in case you need to check it again later.

8. Rinse the colorimeter tube with a little water, empty it, and put it back in the base.

9. Repeat steps 3 to 8 with each of the other five solutions in turn.

10. Plot a graph of your meter readings against concentration. Draw a smooth curve through, or close to, all the points. This is your calibration curve.

   Note: If any graph point appears badly wrong (is not near the curve), check it. Put that solution back into the colorimeter and take the reading again.

PART C: ANALYSING UNKNOWN SAMPLE U

1. Measure out 10.0 cm³ of solution U into the colorimeter tube.

2. Take a meter reading as before. Record it in table column U.

3. Use your calibration curve to convert this reading into a concentration value. Record it in the table.

QUESTIONS

1. When preparing the known solutions, why must you measure the volumes of S and water as accurately as possible?

2. Why is it important to keep light out of the colorimeter by covering it with the box?

3. You always put 10 cm³ of each solution in the colorimeter. Suggest why you should always use the same volume. What would happen if the volumes differed?
**Titration** is a **quantitative** method of analysis – as opposed to a **qualitative** method such as microscopy or thin layer chromatography.

- **Qualitative** analysis tells you what substance(s) the sample contains.
- **Quantitative** analysis tells you how much of the substance it contains.

Titration is used to measure the quantity of a particular substance in a sample solution. We normally give this as a **concentration** – that is, the amount contained in 1 cubic decimetre (1 dm³) of the solution. [1 dm³ = 1000 cm³]

To perform a titration, you gradually add one solution to another until it changes colour. You must measure the volume of both solutions carefully and accurately. For this, you use two special pieces of glassware – a **pipette** and a **burette**.

- A **pipette** (see Fig.1) measures out an exact volume of solution at the start of a titration. The one you are using has only one graduation mark, so can measure only one fixed volume, such as 25.0 cm³.
- A **burette** (see Fig.2) measures the volume of solution you add during a titration. It has a continuous scale, usually 0 to 50 cm³, so can measure any volume in between.

As you gradually add the second solution from the burette, the two solutions react. When this reaction is complete, you have reached the **end point**. So that you can tell when this happens, you add an **indicator**. It indicates the end point by suddenly changing colour.

The burette measures the volume of solution needed to reach the end point. You use this and the pipette volume to calculate the quantity of substance in the sample.
YOUR TASK
You must show that you can:
1. use a pipette to accurately measure out the required volume of a liquid
2. set up a burette, and control the tap to deliver a steady flow of liquid from the jet, a slow stream of drops and a single drop
3. correctly measure the volume of liquid delivered from the burette
4. carefully perform a titration, and accurately determine the end point

EQUIPMENT
- pipette and filler
- small funnel
- 100 cm³ beaker
- access to a balance
- sample B (an alkali of unknown concentration)
- wash bottle of distilled water
- burette
- clamp stand
- 250 cm³ conical flask
- solution A (an acid)
- screened methyl orange indicator

PROCEDURE
You will learn how to use the equipment, and then how to perform a titration. To practice using the pipette and burette, you will measure out volumes of water. Warning: Pipettes and burettes are fragile. The tips chip or break easily. Handle them gently and carefully.

There are three parts to the procedure.

PART A: USING A PIPEETTE
To fill the pipette you must suck the liquid up into it – but not with your mouth! There are various types of pipette filler. Your teacher will show you how to use yours.
1. Take about 50 cm³ of water in the beaker. Using the filler, suck water into the pipette, until the level is a few centimetres above the graduation mark.
2. Carefully allow the water to slowly run out, until the bottom of the meniscus is level with the graduation mark.
3. If it drops too far, suck it up and try again. Practise until you can reliably stop the meniscus on the mark.
4. Draw up a results table like Table A on the next page.
5. Place the conical flask on the balance. Tare the balance, to read zero.
6. Fill the pipette to the mark, and allow the water to run out into the flask. DO NOT blow.
7. Gently touch the tip of the pipette against the inside of the neck of the flask. A small amount of liquid will remain in the tip. This is deliberate. DO NOT blow it into the flask.
8. Read the balance, and record the mass of water in column 1 of Table A.
9. Empty the flask, and repeat steps 5 to 8 three more times. Record the masses in columns 2 to 4. (The flask does not need to be dry each time. Just zero it.)
10. Show the completed table to your teacher.
PART B: USING A BURETTE

1. Gently clamp the burette in the stand. Adjust the height so the conical flask just fits underneath the jet.
2. To fill the burette: make sure the tap is closed. Put the funnel in the top, and pour water in from the beaker, until the level is a few centimetres above the 0 mark. Place the beaker under the burette. Open the tap for a few seconds to fill the jet, so there is no air in it. Check whether the meniscus is on the scale; if not, run out more water until it is at or below 0.
3. Read the scale at the bottom of the meniscus. Record this reading, \( R_1 \), in column 1 of Table B. **Note:** You can make the meniscus show up more clearly, by holding something dark behind and slightly below it.
4. Place the conical flask under the burette.
5. Hold the tap by curling your **left** hand around the burette – thumb in front, fingers behind.
6. Hold the neck of the flask with your **right** hand. Lift it slightly, so the jet is about 1 cm inside the flask neck.
7. Open the tap fully (with your left hand). Gently swirl the flask (with your right hand) as the water flows in. During a titration, this will mix the two solutions. **Note:** If you are left-handed, it may be easier to swirl the flask with your left, and turn the tap with your right.
8. Close the tap when the level in the burette drops to somewhere between 19 and 21 cm³.
9. Run in a further 3 cm³, about 1 cm³ at a time. Swirl the flask well after each addition.
10. Carefully open the tap again, and adjust it to give a stream of separate drops – about one per second. In this way, add another 1–2 cm³ dropwise, while still swirling the flask.
11. By opening the tap very carefully, try to add one single drop, then close it. Practise this several times until you can control the tap to produce single drops one at a time.
12. Read the burette scale again. Record \( R_2 \) below \( R_1 \) in the table.
13. Subtract \( R_1 \) from \( R_2 \) to find the volume of water added to the flask.
14. Empty the flask. You do not need to dry it.
15. Repeat steps 4 to 13, recording your readings in table column 2. Run the water in quickly until the level drops to around 40 cm³. Continue dropwise, ending with single drops.
16. Now you have learned the techniques, ask your teacher to watch you.
17. Refill the burette, and repeat the procedure to show that you can perform the whole sequence correctly. Record your readings in column 3.

<table>
<thead>
<tr>
<th>Table A: Mass of 25.0 cm³ of water (g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Table B: Burette volumes</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
</tr>
<tr>
<td>Reading 1 (( R_1 ))</td>
</tr>
<tr>
<td>Reading 2 (( R_2 ))</td>
</tr>
<tr>
<td>Volume added (( R_2 - R_1 )) (cm³)</td>
</tr>
</tbody>
</table>
PART C: A SIMPLE TITRATION

Now you have learned to handle the equipment, you can try a simple acid-alkali titration. The purpose is to find out the concentration of an alkali – sodium hydroxide solution.

First you perform a ‘rough’ run. This tells you roughly how much acid is needed, and helps you perform the other runs more accurately.

1. Draw up a results table like Table C (next page).
2. Rinse out the flask with a little distilled water. Empty it, but you do not need to dry it.
3. Rinse out the burette with a little acid (Solution A). Run it out into the sink through the tap, to rinse that out too. Fill the burette with the acid. Don’t forget the jet.
4. Rinse out the pipette with the alkali [CARE: IRRITANT] by using the filler to suck it up (Sample B).
5. Pipette 25.0 cm³ of alkali into the flask. Remember: ignore any liquid that remains in the tip after you touch the tip of the jet against the flask neck.
6. Rinse the inside of the neck with a little distilled water, to wash all the alkali into the bottom of the flask.
7. Add three drops of screened methyl orange indicator.
8. Read the burette and record R₁ in the ‘Rough’ column.
9. Run in the acid, while swirling continuously, until the indicator begins to turn purple.
10. Run in more acid about 1 cm³ at a time, swirling after each. Stop when the mixture stays purple after swirling.
    Read the scale, record R₂ and calculate the volume used, V.
11. Empty and wash out the flask, then rinse it with distilled water as in step 2.
12. Repeat steps 5 to 8, recording R₁ in column 1. Calculate R₁ + V.
13. Run in acid, swirling continuously, until the reading is about 3 cm³ less than (R₁ + V).
14. Add more acid in a slow stream of drops until the indicator begins to change colour. It should show purple, but return to green after swirling.
15. Now add single drops, swirling well after each. Stop when the mixture stays greyish purple after swirling. This is your end-point.
16. Read the burette, then record R₂ and the volume used in column 1.
17. Fill the burette, and repeat the titration (steps 11 to 16) twice more. Record your results in columns 2 and 3.
18. Calculate the average volume of acid used – that is, the average of the volumes in columns 1, 2 and 3. (Ignore the ‘rough’ result.)

<table>
<thead>
<tr>
<th>Table C: Titration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Volume of alkali = 25.0 cm³</td>
</tr>
<tr>
<td>Reading 1 (R₁)</td>
</tr>
<tr>
<td>Reading 2 (R₂)</td>
</tr>
<tr>
<td>Volume of acid (R₂ – R₁) (cm³)</td>
</tr>
<tr>
<td>Average volume of acid used (cm³)</td>
</tr>
</tbody>
</table>
CATCHING THE DRUG CHEATS

{TEST PROCEDURE} PERFORM TITRATIONS

USING YOUR RESULTS

Calculate the concentration of the alkali (Sample B) from:

\[
\text{concentration of } B \text{ (in g dm}^{-3}\text{)} = 0.16 \times \text{average volume of acid A used}
\]

Give your answer to two decimal places.

The number in this formula depends on the chemicals used. It is 0.16 for your particular acid and alkali, but will be different for titrations with other chemicals.
**CATCHING THE DRUG CHEATS**

**TEST PROCEDURE** PERFORM THIN LAYER CHROMATOGRAPHY

**Chromatography** is a technique for separating and identifying the substances in a mixture. It gets its name from the Greek words for ‘colour’ (chromatos) and ‘writing’ (graphee). This is because, in a way, it writes the answer in the form of coloured spots, called a chromatogram.

To read a written word, you must look at both the letters and their positions. For example, ‘cat’ and ‘bat’ have different first letters, while ‘cat’ and ‘act’ have the same letters in different positions. Each set of letters tells you something different.

Similarly, to read a chromatogram and understand what it tells you, you must look at both the colour and positions of the spots.

In the example shown, chromatography was used to analyse the ink from a felt-tip pen. You may have done this yourself, using filter paper.

Look at the chromatogram spots. Which dyes (A, B, C or D) did the ink contain?

**Thin layer chromatography (tlc)** uses a sheet of plastic, coated in a thin layer of white solid. It is better than paper chromatography because:

- it works faster
- the spots don’t spread out so far

Despite its name, chromatography also works with colourless substances. The spots will be colourless, and therefore invisible. To make them show up, we use a ‘developer’ – a chemical that reacts with the colourless substances to form coloured ones.

Anti-doping laboratories use chromatography to analyse athletes’ urine samples. The chromatogram tells them which banned substances, if any, the urine contains. Laboratories use instrumental methods, such as **high pressure liquid chromatography (HPLC)**.

These methods are much more effective, but they work on the same principle. That is, they separate and identify substances in the urine. This detects any banned substances present.

Instead of spots, HPLC gives peaks – one for each substance in the mixture.

As with spots, the position of a peak tells us what substance produced it. Also, the size of a peak tells us how much of that substance was present in the sample.

**YOUR TASK**

You must show that you can use thin layer chromatography to identify an unknown substance and decide whether or not a sample contains a particular substance.

You must therefore be able to apply samples in suitable positions on a tlc plate, run the chromatogram, develop the chromatogram (if the samples are colourless), and interpret the chromatogram (decide what the spots tell you).
CATCHING THE DRUG CHEATS

{TEST PROCEDURE} PERFORM THIN LAYER CHROMATOGRAPHY

WHAT YOU NEED TO DO

You will set up and run two chromatograms to check for banned substances.
- Chromatogram 1 is to find out whether a food colouring contains an illegal dye.
- Chromatogram 2 is to find out whether a sample of water is contaminated with hazardous metals.

EQUIPMENT

- 2 x tlc plates
- thin glass tubes
- test samples for chromatograms 1 and 2
- developer solution (for 2 only)
- container with lid
- pencil and ruler
- distilled water
- dropper / Pasteur pipette

PROCEDURE

To save time, work with a partner. One person sets up chromatogram 1, while the other sets up 2. You can then run 1 and 2 at the same time. **Warning:** Handle tlc plates only by the edges, to avoid contaminating them with fingerprints. Samples B, K, L and M contain hazardous metals.

1. Draw a pencil line across the plate, 1 cm from the bottom, on the coated side. Be careful not to scrape away the coating.
3. Dip a glass tube into sample A. Briefly touch it on cross A, to put a spot of A onto the plate – no more than 2 mm diameter.
4. Using a clean tube for each sample, put tiny spots of the other seven samples onto their labelled crosses.
5. Let the spots dry, then add a second drop to each cross.
6. Remove the lid from the container and add a little distilled water, to a depth of about 5 mm. (The water level must be below the line on the tlc plate when you stand it in the container.)
7. Pick up the chromatograms by the edges. Carefully stand them upright in the container, leaning them against the wall. Make sure they don't touch each other.
8. Replace the lid, and leave the chromatograms to run. This will take several minutes.
9. When the water reaches about 1 cm from the top, remove the chromatograms from the container. Let them dry.
10. Empty the container, then put chromatogram 2 (B, K, L, M) back in, coated side up.
11. Using the dropper, dribble yellow developer solution all over the surface to reveal the spots.
12. Remove the developed chromatogram. Gently rinse off surplus developer under a slowly-running tap.

Show your chromatograms to your teacher, to prove that you can perform tlc successfully.
INTERPRETING YOUR CHROMATOGRAMS

Show your teacher that you understand how to ‘read’ your chromatograms correctly by answering the questions below. Look at the positions and colours of the spots. Compare A with X, Y and Z, and B with K, L and M.

**CHROMATOGRAM 1**

Samples A, X, Y and Z are food colourings made from dyes. Only certain dyes are safe to eat. It is illegal to use any other dyes in foods. The dyes in X and Z are safe, but Y is not.

1. How many dyes does each food colouring (X, Y and Z) contain?
2. How many dyes does colouring A contain?
3. Which dyes, (X, Y and Z) does colouring A contain?
4. Does colouring A contain an illegal dye?

**CHROMATOGRAM 2**

Sample K is a lead compound; L is a silver compound; M is a barium compound.

Sample B is water suspected of being contaminated with one or more of these metals.

1. Does your chromatogram show that the water contains any of these metals?
2. If so, which ones?
YOUR TASK
You are given four samples:
⋆ two known substances – B which is banned, and L which is legal
⋆ two unknown samples, P and Q, found in an athlete’s kit-bag.
You need to decide whether either (or both) of the unknown samples contains the banned substance.

EQUIPMENT
⋆ microscope
⋆ glass slides
⋆ mounted needle
⋆ lamp
⋆ samples

WHAT YOU NEED TO DO
1. Put a little of each sample on four separate glass slides. Label them so you know which is which.
2. Use the needle to spread out each sample, so you can see individual particles, not just a pile of crystals or powder.
3. Examine B and L under the microscope. Record your observations in your laboratory notebook – for example:
   ⋆ colour
   ⋆ shape
   ⋆ whether transparent or opaque
4. Take particular note of the differences between B and L. You need to be able to recognise them when you examine the unknown samples. Drawings may help.
5. Examine unknown samples, P and Q, recording your observations.
6. Use your observations to decide whether each of P and Q is:
   ⋆ the legal substance, L
   ⋆ the banned substance, B
   ⋆ mostly L with some B mixed in with it
   ⋆ mostly B with some L in it.
7. Report your findings, with your evidence.
YOUR TASK
You will be given a sample of an athlete’s urine to analyse. (Don’t worry, it’s not real!) The first test is to measure its specific gravity. You need to decide whether the sample has been tampered with (diluted or contaminated). If it has, its specific gravity will be lower or higher than when the sample was given by the athlete. The original value is marked on the sample.

EQUIPMENT

- balance
- 10 cm³ measuring cylinder
- dropper
- hydrometer, if available
- hydrometer tube (e.g. 250 cm³ measuring cylinder), if available
- urine sample

WHAT YOU NEED TO DO

1. Determine the specific gravity by measuring mass and volume, as you learned to do previously.
2. Also measure the specific gravity with a hydrometer, if available.
3. If your two values disagree, try to decide which is likely to be more accurate. Explain how you decided.
4. Note: ‘More accurate’ means closer to the true value. Getting an answer with more figures does not make it more accurate. For example, suppose the true specific gravity is 1.0256. The value 1.02641 is less accurate than 1.025.
5. Compare your result with the original specific gravity,
6. Decide whether or not your measurements suggest that the sample has been tampered with, and if so, whether it has been diluted, or been contaminated by adding something.
7. Report your findings, with your evidence.
CATCHING THE DRUG CHEATS

TEST PROCEDURE: DETECT SUBSTANCES WITH TLC

YOUR TASK
You will be given artificial urine samples, R and T, from two athletes. Test them to find out whether each sample contains banned substance F and/or G, or whether the athlete is ‘clean’.

EQUIPMENT
- tlc plate
- container with lid
- thin glass tubes
- dropper / Pasteur pipette
- pencil and ruler
- urine samples R and T
- solutions of banned substances F and G
- distilled water
- developer solution, D

WHAT YOU NEED TO DO
1. Prepare the tlc plate and apply spots of F, G, R and T. Record whether you are testing R₁ or R₂, and T₁ or T₂.
2. Run the chromatogram using distilled water.
3. Allow the plate to dry. Then develop it with solution D.
4. From your results, decide whether each urine sample contains F or G or both, or neither.
5. Report your findings, with your evidence. Remember to state which number samples you tested.
**{TEST PROCEDURE} SPOT TESTS**

A simple technique for qualitative analysis is to use **spot tests**. You take just one spot or drop of sample and add one drop of reagent. A spot test:

- requires only a tiny amount of sample
- can detect very low concentrations – sometimes less than one part in a million
- is very specific – each spot test detects one substance, so can distinguish between similar substances
- tells you whether a particular substance is, or is not, present in the sample

However, you need to know what you’re looking for, so you can choose the correct tests.

**YOUR TASK**

You must test two banned substances, H and J, with two spot test reagents, TR1 and TR2. Each reagent should give a deep colour with one banned substance, but not with the other.

Use these spot tests to decide whether samples W, X, Y and Z contain substance H or J, both, or neither.

**EQUIPMENT**

- spotting tile
- 6 x droppers
- solutions of banned substances H and J
- samples W, X, Y and Z
- dropping bottles of
  - TR1, ammonium thiocyanate
  - TR2, potassium hexacyanoferrate(III)

**WHAT YOU NEED TO DO**

You need to be careful to avoid cross-contamination. Use a separate dropper for each solution. Labelling the droppers H, J, W, X, Y and Z may help.

1. Draw up a results table like the one below.
2. Put one drop of H in each of two adjacent dips in the spotting tile. Add one drop of TR1 to one, and one drop of TR2 to the other. Record your observations in column H.
3. Repeat with J.
4. Your observations should show you how to recognise H and J.
5. Put one drop of W in two of the remaining dips. Do the same with X, Y and Z. Make sure you know which is which.
6. Decide what the spot tests tell you about samples W, X, Y and Z.
7. Report your findings, with your evidence.

<table>
<thead>
<tr>
<th></th>
<th>H</th>
<th>J</th>
<th>W</th>
<th>X</th>
<th>Y</th>
<th>Z</th>
</tr>
</thead>
<tbody>
<tr>
<td>with TR1</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>with TR2</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
YOUR TASK

Qualitative analysis of sample Z using spot tests showed that it contains banned substance H. You now need to perform quantitative analysis to find out how much – that is, the concentration of H in sample Z.

The concentration is very low – less than 50 mg/dm³. That is, less than 0.05 g in 1000 cm³, or 0.00005 g in 1 cm³.

At this low concentration, H is almost colourless. So you will add a chemical that reacts with H to form a coloured compound. You can then measure its concentration using colorimetry.

EQUIPMENT

- colorimeter and multimeter
- 10 cm³ measuring cylinder
- rack of six test tubes
- standard solution of H
- 2 x 100 cm³ beakers
- 25 cm³ measuring cylinder
- rubber bung
- unknown sample solution Z
- 2 x droppers
- glass stirring rod
- box to cover colorimeter
- ammonium thiocyanate solution

WHAT YOU NEED TO DO

PART A: PREPARE KNOWN SOLUTIONS

1. Carefully measure out 20.0 cm³ of H into a beaker.
2. Add 20.0 cm³ of ammonium thiocyanate solution to produce a red solution. (The mixture in the measuring cylinder will also turn red.)
3. Gently stir the solution to mix it well. Label the solution: Concentration = 50 mg/dm³.
4. Use this solution to prepare six test tubes of known concentrations. As before, use:
   - 10.0 cm³ (Label it 50 mg/dm³)
   - 8.0, 6.0, 4.0 and 2.0 cm³ topped up to 10.0 cm³ with water. (Work out their concentrations in mg/dm³ rather than as percentages.)
   - 10 cm³ of water only (Label it 0 mg/dm³)

PART B: CALIBRATE THE COLORIMETER

As before, use the six solutions to produce a calibration curve. The x-axis will be concentration in mg/dm³, not %.

PART C: ANALYSING UNKNOWN SAMPLE Z

1. Measure 5.0 cm³ of sample Z. Add 5.0 cm³ of ammonium thiocyanate, and mix well.
2. Take a colorimeter reading. Read off the concentration from the calibration curve.
3. Report your findings, with your evidence.
CATCHING THE DRUG CHEATS

{TEST PROCEDURE} MEASURE THE PURITY OF IBUPROFEN

YOUR TASK

Ibuprofen is a legal over-the-counter (OTC) medicine. Sample I is powdered ibuprofen tablet, which may, or may not, contain other ingredients. Your task is to find out how pure it is – that is, how much of the sample is actually ibuprofen.

Ibuprofen is an acid. So you can dissolve it, and titrate the solution with an alkali. Your result will tell you the mass of ibuprofen in sample I. From this you can calculate its purity.

Anti-doping laboratories can use similar methods to analyse banned substances in mixtures. Perhaps the substance has been disguised by mixing with a legal substance, such as sugar. They can determine the percentage of banned substance in the mixture.

EQUIPMENT

- pipette and filler
- small funnel
- 2 x 250 cm$^3$ conical flasks
- sample I (powdered ibuprofen)
- sodium hydroxide solution (an alkali)
- wash bottle of distilled water
- burette and clamp stand
- 100 cm$^3$ measuring cylinder
- access to a balance
- ethanol [HIGHLY FLAMMABLE AND HARMFUL]
- phenolphthalein indicator [HIGHLY FLAMMABLE]

WHAT YOU NEED TO DO

1. Draw up results tables like those below.
2. Weigh the container of sample I. Record the mass in the table.
3. Tip sample I into a conical flask. Reweigh the empty container and stopper. Subtract the masses to find the mass of sample I.
4. Carefully measure exactly 100 cm$^3$ of ethanol. Pour it into the flask. Swirl until sample I has completely dissolved and the solution is well mixed.
5. Using the other conical flask, titrate 25 cm$^3$ portions of this solution with the alkali (sodium hydroxide solution).
   - Use the pipette for I and the burette for the alkali.
   - The end-point is when the phenolphthalein indicator remains pink after swirling the flask.
   - Perform one rough titration, and two accurate ones, recording your readings in the table.
   - Use the 25 cm$^3$ of I solution remaining in the first flask for your third accurate titration.
6. Calculate the average volume of your three accurate titrations.
7. Calculate the mass of ibuprofen in sample I from:
   
   \[
   \text{mass of ibuprofen} = 0.0412 \times \text{average volume of alkali used}
   \]
8. Calculate the purity of sample I from:
   
   \[
   \text{percentage purity} = \frac{\text{mass of ibuprofen in I}}{\text{mass of sample I}} \times 100
   \]
9. Report your findings, with your evidence.
### Measure the Purity of Ibuprofen

**Test Procedure**

| Mass of container with sample | g |
| Mass of empty container       | g |
| Mass of sample                | g |

**Volume of I solution = 25.0 cm³**

<table>
<thead>
<tr>
<th>Rough</th>
<th>1</th>
<th>2</th>
<th>3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Burette reading 1</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Burette reading 2</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Volume of alkali used (cm³)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Average volume of alkali used (cm³)</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
The anti-doping programme is extremely important in helping to look after the integrity of sport and the health of athletes. A key part of the doping control process is the chemical/bio-chemical analyses, which are carried out in Accredited Laboratories.

Students are challenged to prove their proficiency at providing a laboratory service. It is a scaled down version of the International Accreditation process. They will have to show that they can assemble and use certain apparatus. They will have to show how well they can use tests to analyse samples, identify and/or measure specified substances.

Students should work in teams of about six people. Teams will need to manage their time effectively, sharing out different tasks. However, they should all practise the analytical techniques and, for stage 2, comparison of results should be included in their reports. At some point, it may be helpful if each team allocates different aspects of the checklists to individual team members. This could be:

- Quality control, ensuring that the correct test is carried out on the correct sample
- Internal Chain of Custody records
- Security
- Confidentiality
- Ensuring that each sample can be tracked
- Ensuring that the testers are traceable to each test carried out

**STARTER ACTIVITY**

There are separate notes for a starter activity, with an accompanying PowerPoint.

Students could also view the World Anti-Doping Agency videos: [www.youtube.com/wadamovies](http://www.youtube.com/wadamovies)

**THE ACTIVITIES**

It will be important for students to become conversant with the processes (and people) involved in an anti-doping programme. However, teachers may want their students to get started on practising the laboratory techniques first.

**STAGE 1**

Students will have to provide evidence that they can assemble and use apparatus to perform a titration, thin layer chromatography, light microscopy, specific gravity measurement, and colorimetry.

As these techniques are likely to be new to the students, it is more about their manual dexterity and following instructions than their results.

**STAGE 2**

When teams are ready, they can progress to stage 2. Given a sample, students will need to:

- assign it a reference number
- carry out a Qualitative analysis (microscopy, Thin Layer Chromatography or colour/spot test) or Quantitative analysis (specific gravity, colorimetry or titration)
- repeat the test as individuals, to show repeatability, or by other team members, to show reproducibility
- keep careful records to track and trace the sample and those who tested it
Teams should be asked to produce a dossier, showing their paperwork trail, how they kept everything confidential and secure, an evaluation of their test methods and evidence of their own research on doping control. The emphasis should be quality rather than quantity. So, if a team does not learn all the techniques or investigate all their samples, it can be balanced by the quality of their reports.

Teams are asked to produce a report with all their findings, showing that they are methodical with their paperwork and that they understand about confidentiality and security. They should also give an evaluation of their test methods.

A further part of the submission is evidence of their own research on doping control.

Becoming an accredited laboratory involves more than carrying out scientific tests. Students are therefore asked to include a small dossier about their laboratory. This can be photographs and statements but videos could be used. They are likely to need information from school staff, e.g. Health and Safety Statement, to help with this.

PRESENTATION

Students have not been given guidelines on how to present their evidence. Although test results are likely to be paper-based (or electronic, e.g. Word documents), photographs or video could usefully be used for other evidence.

As well as laboratory skills, teams should be judged on their skill in communicating scientific information.

Students could be reminded about some features of good communication:

- presentation – the process is important as well as the results
- using a mixture of verbal, written and visual communication
- present scientific information, rather than emotive arguments
- using scientific language and terminology correctly
- being able to talk knowledgeably about every aspect of the Challenge

Note: Where possible, encourage students to look at the resources online. Only print sheets that are strictly necessary.

HEALTH AND SAFETY

Much of this Challenge should be carried out in a laboratory, with all the usual rules applying. Although the tests and activities do not pose significant unusual hazards, teachers must carry out a risk assessment for their particular circumstances and student group. Specific health and safety guidance for the tests and activities is given in the relevant teacher/technician notes.
PROGRAMME OF STUDY FOR KEY STAGE 3

SCIENCE

KEY CONCEPTS
Scientific thinking
b. Critically analysing and evaluating evidence from observations and experiments.

Collaboration
a. Sharing developments and common understanding across disciplines and boundaries.

KEY PROCESSES
Practical and enquiry skills
a. Use a range of scientific methods and techniques to develop and test ideas and explanations.
b. Assess risk and work safely in the laboratory, field and workplace.
c. Plan and carry out practical and investigative activities, both individually and in groups.

Critical understanding of evidence
a. Obtain, record and analyse data from a wide range of primary and secondary sources, including ICT sources, and use their findings to provide evidence for scientific explanations.
b. Evaluate scientific evidence and working methods.

Communication
a. Use appropriate methods, including ICT, to communicate scientific information and contribute to presentations and discussions about scientific issues.

RANGE AND CONTENT
Chemical and material behaviour
b. Elements consist of atoms that combine together in chemical reactions to form compounds.
c. Elements and compounds show characteristic chemical properties and patterns in their behaviour.

Organisms, behaviour and health
b. Conception, growth, development, behaviour and health can be affected by diet, drugs and disease.
PROGRAMME OF STUDY AT KEY STAGE 3

SCIENCE

LEARN ABOUT
Organisms and Health
- Healthy body and mind
Chemical and material behaviour
- Atoms and chemical changes
- Elements, compounds and mixtures

LEARNING OUTCOMES
- Demonstrate a range of practical skills in undertaking experiments, including the safe use of scientific equipment and appropriate mathematical calculations.
- Use investigative skills to explore scientific issues, solve problems and make informed decisions.
- Research and manage information effectively, using Mathematics and ICT where appropriate.
- Show deeper scientific understanding by thinking critically and flexibly, solving problems and making informed decisions, using Mathematics and ICT where appropriate.
- Demonstrate creativity and initiative when developing ideas and following them through.
- Work effectively with others.
- Demonstrate self management by working systematically, persisting with tasks, evaluating and improving own performance.
- Communicate effectively in oral, visual, written, mathematical and ICT formats, showing clear awareness of audience and purpose.
CURRICULUM FOR EXCELLENCE

SCIENCES

BIOLOGICAL SYSTEMS: BODY SYSTEMS AND CELLS
By investigating some body systems and potential problems which they may develop, I can make informed decisions to help me to maintain my health and wellbeing. SCN 2-12a

MATERIALS: PROPERTIES AND USES OF SUBSTANCES
I have participated in practical activities to separate simple mixtures of substances and can relate my findings to my everyday experience. SCN 2-16a

MATERIALS: CHEMICAL CHANGES
I have collaborated in activities which safely demonstrate simple chemical reactions using everyday chemicals. I can show an appreciation of a chemical reaction as being a change in which different materials are made. SCN 2-19a
Through experimentation, I can identify indicators of chemical reactions having occurred. SCN 3-19a
KEY STAGE 3 PROGRAMME OF STUDY

SCIENCE

SKILLS
- Opportunities to carry out different types of enquiry (planning, developing, reflecting).

RANGE

Interdependence of organisms
3. The beneficial and detrimental effects of some drugs on the organs of the human body and other consequences of their use

The sustainable Earth
2. The physical and chemical properties of some elements, compounds and mixtures and how mixtures can be separated by simple techniques.
3. The differences between physical and chemical changes using some common examples.
These notes guide an introductory session to the challenge. The length of the session will depend, in part, on the amount of discussion generated, but is unlikely to exceed 20 minutes. The activity will engage students in the subject of anti-doping, by considering some of the underlying issues.

**Part one:** A PowerPoint will direct you through a series of questions and phrases to prompt discussion. These can be worked through fairly rapidly.

**Part two:** Introduce students to anti-doping tests, and carry out a demonstration to illustrate the tiny quantities of a substance that can be detected.

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**PART ONE: DISCUSSION (POWERPOINT PRESENTATION)**

The PowerPoint is attached to this document. Double-click this paperclip icon to open it. Or, go to View / Navigation Panels / Attachments.

**SLIDE ONE: TITLE SCREEN**

**SLIDE TWO: WHO’S TAKEN MEDICINE FOR ‘FLU, OR A BAD COLD, OR A NASTY COUGH?’**

You should get a good show of hands – most students have taken medicines for coughs and colds. **Click to reveal:** *Why did you take them?* Expect answers such as: ‘To get well’; ‘To feel better’

**SLIDE THREE: HAVE ANY OF YOU TRIED ALERTNESS DRINKS?**

You may need to explain that these are drinks such as Red Bull, Monster, Rockstar, Relentless, Red Devil, Solstis, Purdey’s. There may be some response. **Click to reveal:** *Why do people drink them?* Expect answers such as: ‘To keep awake’; ‘To keep going during exams’; ‘They make you feel good’; Explain that it is very similar to drinking a cup of strong coffee.

**SLIDE FOUR: HAVE YOU EVER HAD A SPORTS DRINK, SUCH AS LUCOZADE?**

You are likely to get some response. **Click to reveal:** *Why do athletes use them?* Guide answers towards: To replace fluid; To give energy from sugar; To replace salts lost through sweating; It is better for the body than plain water; Athletes could become unwell if they do not replace fluids and essential minerals and carbohydrate

*There are different types of sports drink for different purposes (isotonic, hypotonic and hypertonic), but that does not need to be explored here.*

**SLIDE FIVE: IS THIS CHEATING IN SPORT?**

**Click immediately** to reveal the two photographs. It is worth enabling a discussion here about boundaries:

What is the difference between using food and drink to keep healthy and taking supplements (whether banned or not)?; What is the difference between using drugs and physical aids?

**SLIDE SIX: DO YOU THINK THAT TOP ATHLETES USE...**

**Click immediately** to reveal these questions: …cough medicine? …alertness drinks? …energy drinks?

These are closed questions to obtain a simple response.

**SLIDE SEVEN: WHY ARE ENERGY DRINKS HEALTHY FOR SPORT?**

Aim to elicit the conclusions before revealing them on-screen. **Click to reveal:** …athlete health …fair competition
SLIDE EIGHT: WHY ARE ALERTNESS DRINKS LESS HEALTHY FOR SPORT?
Aim to elicit the conclusions before revealing them on-screen.
Click to reveal: …large quantities of caffeine …less fair competition …banned in some sports

SLIDE NINE: ARE COLD AND ‘FLU REMEDIES UNHEALTHY FOR SPORT?
Aim to elicit the conclusions before revealing them on-screen.
Click to reveal: …contain banned substances …unfair competition …sometimes!
Many OTC cold and ‘flu remedies, and cough syrups, contain pseudoephedrine hydrochloride, which is not allowed during competition. Pseudoephedrine is a stimulant used to make medicines non-drowsy. Athletes have to be extremely careful about anything they put into their bodies, particularly if they are feeling unwell. They must always refer to the most up-to-date sources of information on banned substances, and check the ingredients in all medicines. This includes OTC (Over the Counter) medicines that can be found on shop and supermarket shelves.
Click to reveal: So athletes need to be educated …

SLIDE TEN: WHAT IS DOPING?
The practice of using substances or methods to enhance performance is called doping. This includes not only the taking of banned drugs, but also methods that attempt to mask, or hide, the use of such substances. Furthermore, some substances are prohibited (not allowed) In-Competition (12 hours before a competition starts until it finishes), but their use is not prohibited (allowed) Out-of-Competition.
This sets up the next slide explaining what anti-doping organisations do, including educating athletes.

SLIDE ELEVEN: WHY HAVE ANTI DOPING ORGANISATIONS?
The likely answer will be … ‘To catch people who take drugs’. Although this is not incorrect, it is important to establish the underlying reasons for this, and highlight the role of educating athletes.
Anti-doping testing not only catches cheats, but acts as a deterrent to others who might be thinking about using banned substances or methods.
Anti-doping organisations also educate athletes (and other sports personnel) about the types of substances and methods that are prohibited, and the health and safety issues involved.
The ultimate goal is to achieve safe competition and provide a level playing field for all competitors.
Click to reveal: Doping control safeguards the integrity of sport and the health of athletes
[END OF POWERPOINT]
PART TWO: DEMONSTRATION

Athletes have to provide samples of urine or blood to be tested. Tests used in anti-doping laboratories are able to detect even the tiniest amount of a substance in a small sample of urine. Steroids, such as nandrolone, have to be detected in quantities as small as 2 nanograms in 1 ml of urine. 1 g = 1 billion nanograms.

So, that would be like knowing there’s just 5 g of a substance in an Olympic swimming pool. (Measure out 1 g and 5 g of salt, for example, to illustrate this quantity.)

DEMONSTRATION

You will need
- food colouring (any colour)
- 5 x test tubes
- test tube rack
- pipette
- stirring rod

What to do
1. Fill each test tube with water and place them in the rack.
2. Explain that it is easy to detect something when you can see.
3. Using the pipette, put two drops of food colouring into a test tube at one end.
4. Stir with the rod, and pipette two drops from this tube into the one next to it.
5. Explain that there may be other ways of detecting whether or not an additional substance is present. For example, by taste. (You are not going to demonstrate this though!)
6. Stir before putting two drops from this tube into the next.
7. Explain that as the amount of substance gets smaller and smaller, it is no longer possible to detect it by simply using our senses.
8. Continue this process.
9. Explain that each time you pipette drops from one tube to the next, the solution is becoming more diluted. In other words, there is less and less food colouring in the same amount of water.
10. Once the food colouring can no longer be seen, ask the students if there is any still present. Establish the fact that there is. The amount may be very little, but there must be some present – after all, they have watched you put drops of water containing food colouring into each test tube. The food colouring can decrease in quantity but it cannot disappear!
11. Remind the students that in anti-doping laboratories, scientists must be able to detect these miniscule amounts. Amazing isn’t it!
12. Explain that this Challenge will provide them with an opportunity to find out a little about the sophisticated techniques used in anti-doping labs, and to showcase their own ability to perform some techniques and procedures that can be used to identify and measure substances.
For accreditation, students must show themselves capable of setting up a simple microscope correctly, and bringing a sample into sharp focus.

The students’ procedure may require modification if the microscope to be used is significantly different from that shown. For example, if it has a single objective, single focusing knob, a condenser and/or diaphragm illumination control, a fixed body and moving stage, or built-in illumination.

Samples are normally viewed using transmitted light. Students need to appreciate that they must never use a microscope in direct sun, since the focussed sunlight would damage their eye. The mirror can be tilted in two planes, to reflect the light in the required direction (into the objective). If the daylight is dim, they can use a lamp instead – but they must shine it onto the mirror not onto the sample. This method does not work for opaque samples, which must be illuminated from above instead.

**EQUIPMENT**
- basic microscope
- prepared sample slides of a transparent material (eg salt or copper sulfate crystals, or photographic slide of blood cells), and an opaque material (eg pepper or iron filings)
- bench lamp

**HEALTH AND SAFETY**

Microscopes must NOT be set up in direct sunlight, since adjusting the mirror is likely to focus strong sunlight on the eye, causing serious damage. Preferably use a window away from the prevailing sun; otherwise use bench lamps. Microscope lamps (whether built-in or separate) need PAT testing.

**PROCEDURE**

Check how well the students’ microscopes correspond to the type of microscope illustrated. If necessary, amend the procedure accordingly.

Students are required to demonstrate they have succeeded in setting up and focusing the microscope correctly. They must describe in writing and/or draw what they see – detail will be dependent on how sharply they are able to focus the microscope. If sharing equipment, each student should show their competence individually.

If extension is necessary, the students could be asked to prepare their own slides.
For accreditation, students must show themselves capable of using simple equipment to determine the specific gravity of a liquid. This is obligatory in the testing of urine samples. World Anti-Doping Agency (WADA) rules require that, before sending a sample to the laboratory, its specific gravity (S.G.) is checked, using reagent strips and/or a refractometer. This is to ensure that it meets the requirement for analysis (S.G. ≥ 1.005). The laboratory measure the S.G. again prior to analysis. Comparison with the original value provides a check against contamination or tampering, such as dilution or substitution. (Second worksheet question.)

For students, using a hydrometer, if available, provides a simple and accurate method (subject to temperature variation). However, to help them understand the meaning of ‘specific gravity’, the worksheet instructs them to weigh an accurately measured volume of the liquid and also of water, then divide the results. This method corresponds to the definition of specific gravity.

\[
\text{specific gravity} = \frac{\text{mass of liquid}}{\text{mass of an equal volume of water}}
\]

If the students are already familiar with the concept of density, it would be worth discussing the difference between specific gravity and density – in particular, why the latter has units, but the former does not. Older and/or more able students could be challenged to argue whether or not the two properties always have equal numerical values.

**EQUIPMENT**

- balance measuring to 0.01 g, with tare facility
- 3 x 10 cm³ measuring cylinders
- 3 x droppers / Pasteur pipettes (drop size < 0.1 cm³)
- (optional) hydrometer, preferably covering at least 0.970 to 1.100 (a home-brew / wine-making hydrometer would be ideal)
- hydrometer tube (or measuring cylinder wider and taller than the hydrometer)
- sample liquids labelled X and Y (sufficient volume to almost fill the hydrometer tube)

To reinforce the point that some liquids are ‘lighter than the same volume of water’, one sample should have S.G. > 1.0, and the other < 1.0, eg cooking oil or ethanol. However, if a hydrometer is to be used, samples must be within the scale range. For example:

- 10% sodium chloride solution (S.G. ≈ 1.07)
- 10% ethanol solution (S.G. ≈ 0.98) [PURE ETHANOL IS HIGHLY FLAMMABLE, 10% SOLUTION IS LOW HAZARD]

**HEALTH AND SAFETY**

A risk assessment is required for all practical work, but this activity poses no hazards beyond those normally associated with using glassware, and the chosen sample liquids.
PROFESSIONS

Students are required to demonstrate their competence as a team and individually. It is suggested that students work in pairs, performing one method each on sample X, then swapping methods for sample Y.

MEASURING MASS AND VOLUME

Careful manipulation is required to measure out 10.0 cm³ accurately. The droppers must allow delivery of drops less than 0.1 cm³.

Students should appreciate that avoiding cross-contamination is paramount in anti-doping analysis. Therefore the procedure requires them to use a clean, dry measuring cylinder and dropper for each sample (third worksheet question).

Improved method

If time allows, students could use a more accurate, but longer, adaptation of the procedure.

They could record the mass of 1.0, 2.0, 3.0 … up to 10.0 cm³ and plot mass against volume.

Projecting the best-fit straight line (including the origin) to 10.0 cm³, should give a more reliable value (R) for the mass, than a single weighing (S).

Students may well need help in deciding the position of the best-fit line.

USING A HYDROMETER

Handling a hydrometer is simple enough. The problem for students will be in interpreting the scale. This is potentially confusing since it increases downwards, and the numbers may be truncated, showing only the last two digits.

Since hydrometers vary in range and scale markings, students will need to be shown how to read the scale on their particular version, and deduce the specific gravity.

For example:

- whether the reading shown here is 8, 12, 23 or 28,
- whether this means the specific gravity is 1.012 or 1.112.

As in method A, to reinforce the importance of avoiding cross-contamination, they are instructed to wash and dry the equipment between samples.
For accreditation, students must show themselves capable of setting up a colorimeter, calibrating it correctly, and using it to determine the concentration of a sample solution. Their procedure is based on using the ‘home-made’ colorimeter, details of which are given on the Factsheet: How to make a colorimeter.

Instead of using coloured filters, this simple version uses a coloured LED. As with filters, the colour should be complementary to the colour of the solution – that is, green for a red solution, or red for a blue or green solution.

Some preliminary testing is required to match solution S and the multimeter resistance range to the particular LED and LDR used. See below.

If a commercial instrument is to be used
- set it to ‘absorbance’ mode, not ‘transmission’
- pre-select the appropriate colour filter
- check whether the procedure requires modification, besides ignoring steps 2, 4 and 6.

**EQUIPMENT (PER STUDENT OR PAIR)**

- colorimeter and multimeter*
- 2 x 100 cm³ beakers
- 2 x droppers
- 10 cm³ measuring cylinder
- rack of six test tubes labelled 10, 8, 6, 4, 2 and 0
- rubber bung for test tube
- box (large enough to cover the colorimeter to exclude light)
- 35–40 cm² standard solution, labelled S
- 10–15 cm³ unknown sample solution, labelled U

* The resistance range required depends on the LDR used – up to 1 MΩ for NORPS12. Some pre-testing is required to find a combination of concentration and resistance range that gives resistance readings for the six tubes spread across the range.

Standard solution S can be any safe substance with colour complementary to the LED, for instance a food colouring. Add the concentrated original dropwise to 100 cm³ water, counting the drops, until the solution gives full-scale deflection on the colorimeter. Scale up the volume as required, allowing for consumption of S in the preparation of U.

Prepare solution U by dilution of S. It’s concentration, expressed as a percentage of S, must be known to the teacher, but not to the students. If desired, give different students different concentrations, labelled U₁, U₂ ... as required.

**HEALTH AND SAFETY**

A risk assessment is required for all practical work, but provided solution S is non-hazardous, there should be no safety problems with this procedure.
PROCEDURE
If a commercial colorimeter is to be used, you will need to instruct students in its correct use, including:

- not altering the filter, which you have pre-set
- handling sample tubes (cuvettes), avoiding fingerprints and liquid on the outside
- reading absorbance, not transmission.

If using the home-made colorimeter, ensure that students know which multimeter range to set, and which scale to read.

To produce a reliable calibration curve requires accurate concentrations. The volumes of $S$ and water in the known solutions must therefore be measured accurately, by bringing levels up to the calibration lines drop by drop. Students may need help in deciding where to draw the best-fit curve.

Meter readings must always be taken with the same volume (and therefore depth) of solution in the colorimeter tube, since absorption depends on path length through the liquid.

Competence in setting up the colorimeter may be judged by how well the readings form a smooth calibration curve. Any point significantly out-of-line should be re-measured.

Having determined the concentration of unknown $U$, students with sufficient scientific and mathematical understanding could be asked to estimate the uncertainty in their result, judged from the uncertainty in the positioning of their calibration curve.
For accreditation, students must show themselves capable of setting up and performing titrations. That is:

- using a pipette to accurately measure out the required volume of a liquid
- setting up a burette, and controlling the tap to deliver:
  - a steady flow of liquid from the jet  |  a slow stream of drops  |  a single drop
- correctly measuring the volume of liquid delivered from the burette
- carefully performing a titration, and accurately determining the end point
- calculating the concentration of a sample from their results, given the appropriate formula

Students will need to acquire and practise the techniques. It is suggested that you work through the worksheet with them, demonstrating the techniques, before allowing them to try for themselves. To avoid waste, they practise just with water, before attempting a simple acid-alkali titration.

The various steps in the techniques involve good examples of scientific reasoning and logical thought. They are therefore worth discussing with the students, asking them to suggest why certain actions are necessary for accurate results. They should appreciate that:

- a titration involves determining the volume of one solution that reacts with a known volume of the other
- the known volume is measured with a pipette; the unknown with a burette
- both volumes must be measured as accurately as possible
- the pipette and burette must be rinsed out with their respective solutions, to remove water droplets, which would otherwise dilute the solutions
- the titration flask must be clean, (rinsed with distilled water), but need not be dry, because the volumes of both solutions have already been measured before they are run into the flask
- the pipette is designed to not quite empty completely, and
  - if the tip is not touched against the flask, the volume will be too low
  - if the remaining liquid is blown out the volume will be too high
- it is not necessary to waste time adjusting the burette starting level to exactly 0.0, since the volume added is just the difference between the start and finish readings.
- for the same reason, it is not necessary to refill the burette after each run
- the purpose of a titration is to determine the concentration of one of the solutions, which may be the one in the pipette, or in the burette

The other concentration must be known in advance, as must the molar ratio in which the reagents react. For simplicity in this exercise, these factors are included in a single number, \( n \), in the calculation formula given to the students: concentration of alkali \( B = n \times \text{average volume of acid } A \text{ used} \)

The concentration is given in g dm\(^{-3}\), not mol dm\(^{-3}\), since Key Stage 3 students are unlikely to be familiar with moles.
EQUIPMENT

- 25 cm³ pipette and filler
- 50 cm³ burette
- small funnel (for filling burette)
- clamp stand or burette stand
- 100 cm³ beaker
- 250 cm³ conical flask
- access to a balance reading to two or three decimal places
- 0.10 mol dm⁻³ HCl, labelled ‘solution A’ (about 150 cm³ per student or pair)
- 0.08 mol dm⁻³ NaOH [irritant], labelled ‘sample B’ (about 150 cm³ per student or pair)
- screened methyl orange indicator
- wash bottle of distilled water

HEALTH AND SAFETY

Careful, gentle handling of pipettes and burettes will minimise the risks.

In particular, students should be warned against placing the pipette where it may roll off the bench, knocking the pipette or burette tip, forcing a jammed burette tap, and squeezing the burette too tightly in the clamp.

PROCEDURE

Students first practise using the pipette and burette. You will need to show them how to use your particular type of pipette filler.

You should judge their competence by observation, including whether the student interprets the inverted burette scale correctly.

In addition, completed Table A indicates the accuracy and consistency of their pipette work. The theoretical mass of 25.0 cm³ of pure water is given below.

<table>
<thead>
<tr>
<th>Temperature / °C</th>
<th>15</th>
<th>18</th>
<th>20</th>
<th>22</th>
<th>25</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mass / g</td>
<td>24.977</td>
<td>24.965</td>
<td>24.955</td>
<td>24.944</td>
<td>24.926</td>
</tr>
</tbody>
</table>

For the titration, screened methyl orange is specified, since the greyish end-point between green and purple is more obvious. Plain methyl orange may be used, but it is more difficult to distinguish the end-point in the gradual transition from yellow through orange to red.

If the indicator is changed, the colours in the procedure must be amended appropriately.

To calculate the concentration of alkali B, they are given the formula:

\[
\text{concentration of B (in g dm}^{-3}\text{)} = 0.16 \times \text{average volume of acid A used}
\]

Students are not expected to understand where the 0.16 comes from.

For 0.10 mol dm⁻³ HCl and 0.08 mol dm⁻³ NaOH, the expected end-point is about 20 cm³, and the concentration about 3.2 g dm⁻³, depending on the exact concentrations of the reagents.
For accreditation, students must show themselves capable of setting up, running and interpreting thin layer chromatograms.

The exercises echo anti-doping procedures, being based on detecting and identifying banned substances – here an illegal food dye and toxic metals in a water supply.

Students are likely to have used paper chromatography previously, but may need reminding. If time allows, it may be worth running paper and thin layer chromatograms of felt tip inks simultaneously, to demonstrate the benefits of the latter. This could also be used as an opportunity to show students how to spot small samples (1–2 mm diameter) onto the plate.

The introduction in the students’ worksheet points out that anti-doping laboratories use instrumental methods, such as HPLC. Students need no details, beyond knowing that these methods work on the same chromatographic principles of separating a mixture and identifying the components from their positions on the chromatogram.

In addition, instrumental methods are quantitative, since the peak sizes indicate the relative amounts of each component. The fact that ‘peak size’ is not the same as ‘peak height’ is not important at this stage.

The students run two chromatograms, one coloured (dyes), the other colourless (metal salts), which then requires developing to reveal the spots.

In practice, the dyes need not necessarily be food colourings – any water-soluble dyes will show the principle. Whether each dye is, or is not, actually food-safe is irrelevant. The metal ion concentrations in the supposed water sample are, of course, unrealistically high to ensure detection.

---

**EQUIPMENT**

- 2 x tlc plates (about 50 x 70 mm, cut from larger sheet)
- container with lid (eg tlc tank or screw top jar)
- capillary / melting point tubes
- (To help apply very small spots to the plate, the tubes can be drawn out to a smaller diameter in the centre, then carefully broken in half.)
- pencil and ruler
- distilled water
- test sample solutions (only minimal volumes are required, e.g. a few drops of each on a spotting tile, or watch glasses)

**X, Y and Z:** food colourings (use bought colourings diluted about ten times, or 0.1% solutions of powdered dyes), each containing different dye(s) – chosen so that a mixture of X and Z is a similar colour to Y, e.g. red, orange and yellow, or yellow, green and blue

**A:** mixture of X and Z

**K, L and M:** approx. 1 mol dm⁻³ Pb(NO₃)₂ [toxic], AgNO₃ [corrosive], and Ba(NO₃)₂ [harmful] or BaCl₂ [toxic]

**B:** mixture of K and L

**developer solution for B:** 0.25 mol dm⁻³ K₂CrO₄ [toxic]

**dropper / Pasteur pipette**
HEALTH AND SAFETY

Solutions of lead and some barium potassium chromate are toxic; silver nitrate is corrosive to skin. However, the quantities required are minimal.

Capillary spotting tubes are fragile, with sharp ends if drawn out, but safe unless mishandled.

PROCEDURE

It may be advisable to practise spotting onto filter paper beforehand, for students to learn how to produce the tiny spots required. You should also stress the importance of:

- avoiding finger prints on the plate, by handling it only by the edges
- not damaging the coating while marking out start positions
- avoiding cross-contamination by using a new spotting tube for each sample
- ensuring the solvent level will be below the start line when the plate is stood in the container
- keeping an eye on the running chromatogram, to remove it when the solvent front approaches the top.

To save time, a pair of students can simultaneously prepare and run one chromatogram each.

The running solvent must be distilled water, since chloride or sulfate ions in tap water would partially precipitate the metals in the lead, silver and barium solutions.

The invisible spots of colourless metal ions in chromatogram 2 are revealed by reaction with chromate ions, producing coloured chromates of the three metals. Rinsing off the unreacted developer restores the yellow plate to white, making pale yellow barium chromate more visible.

Note: If the tlc plate has a fluorescent material incorporated in the coating, you could demonstrate an alternative to using a developer. The invisible spots can be revealed by shining a UV lamp onto the undeveloped plate in a darkened room. The spots show up as dark patches against the fluorescent background, and their positions can be ringed with a pencil.

INTERPRETING THE CHROMATOGRAMS

Many students will have performed paper chromatography previously. Studying the example ink chromatogram in their worksheet, may jog their memory of what the spots tell them. If necessary, ensure that they realise that same-coloured spots at the same distance up the plate indicate that the samples contain the same substances.

It is not necessary to introduce the concept of Rf values at this level. Simple comparisons of position will suffice.

Applying the principal to their own chromatograms should enable them to deduce the contents of samples A and B, and answer the questions.

It would be worth pointing out that interpreting chromatograms in anti-doping cases is much less straightforward. Urine contains many more substances, so if tlc were used, the spots are likely to overlap. Instrumental methods, such as HPLC, are much more effective at separating and distinguishing the components of complex mixtures. Like food, drugs are broken down (metabolised) as they pass through the body, so the drug itself does not appear in the urine. The analyst must know what break-down products indicate the original presence of each particular drug.
To demonstrate proficiency, students must show themselves capable of using microscopy to decide whether unknown samples contain a ‘banned substance’.

They are provided with two crystalline materials, which are not readily distinguishable to the naked eye, but recognisably different under the microscope. One represents banned substance B, the other a legal substance L. The students study these, noting the distinguishing features of each.

They are then given two unknown samples, P and Q, supposedly found in an athlete's kit-bag. Their task is to examine these, and thus identify each sample as one of the following:

- pure B
- pure L
- mostly L with some B mixed in with it
- mostly B with some L in it.

If several teams of students are working, it is suggested that allocations of the four compositions to P and Q is varied across the teams.

P and Q may or may not be the same. Again, this could vary between teams.

Students are instructed to record their findings and evidence, and write a test report.

**EQUIPMENT**

- basic microscope
- mounted needle
- lamp

**SAMPLES**

B and L: two crystalline materials, with small crystals, which are not readily distinguishable by shape or size to the naked eye, but recognisably different under a microscope (such as sodium chloride and benzoic acid or calcium sulfate). Label these B and L, rather than with their actual identities.

P and Q: One of these should be pure B or L, the other a mixture of both, with one component predominant, in a ratio of about 1:3 or 3:1.

If several teams are allocated different samples, label them P₁, P₂ and so on.

**HEALTH AND SAFETY**

Microscopes must NOT be set up in direct sunlight. Preferably use a window away from the prevailing sun; otherwise use bench lamps.

Associated chemical hazards must be considered when choosing appropriate sample materials.

**PROCEDURE**

Students examine B and L, and note differences in their appearance. They then examine P and Q, comparing them with B and L, to deduce the identity of each.

Their worksheet instructs them to submit their findings and evidence. Given time, you might ask them to talk through their analyses as part of Quality Control, explaining how they came to their conclusions.
CATCHING THE DRUG CHEATS

{TEACHER NOTES} DETERMINE THE SPECIFIC GRAVITY OF URINE

To demonstrate proficiency, students must show themselves capable of measuring the specific gravity of simulated urine.

**EQUIPMENT**
- balance measuring to 0.01 g, with tare facility
- 10 cm³ measuring cylinder
- dropper / Pasteur pipette (drop size < 0.1 cm³)
- hydrometer (if used in the Accreditation exercise)
- hydrometer tube (or measuring cylinder wider and taller than the hydrometer)
- ‘urine sample’ (sufficient volume to almost fill the hydrometer tube)
  - To simulate urine, for each dm³ of water:
    - dissolve 5 g sodium chloride and 10 g urea
    - add 3 cm³ of 1 mol dm⁻³ ammonia solution
    - add yellow food colouring to give a realistic shade.
  - Measure the specific gravity and record this as ‘Original S.G.’ on the label. Some samples should be allocated to students ‘as is.’ Others should be diluted or extra solute added, to simulate tampering.

**HEALTH AND SAFETY**
A risk assessment is required for all practical work, but this activity poses no hazards beyond those normally associated with using glassware. The solutes are non-hazardous.

**PROCEDURE**
Using one or both methods (measuring mass and volume, and using a hydrometer), students determine the specific gravity of their sample and compare it with the ‘Original S.G.’ They use the comparison to decide whether or not the sample appears to have been tampered with.

If possible, students should use both methods. They can then compare the two results and try to decide which is likely to be more accurate, bearing in mind the procedures used.

This provides an opportunity to discuss experimental error and uncertainty in measurements. It is also worth discussing the meaning of ‘accurate’, to correct the common misconception that obtaining an eight figure answer on a calculator makes it accurate.
To demonstrate their proficiency, students must show themselves capable of using TLC to decide whether two ‘urine samples’ contain either or both of two supposedly ‘banned’ substances.

The samples are pale in colour, so require developing to reveal the chromatogram spots.

**EQUIPMENT (PER STUDENT OR PAIR)**

- tlc plate (about 50 x 70 mm, cut from larger sheet)
- container with lid (eg tlc tank or screw top jar)
- capillary / melting point tubes (to help apply very small spots to the plate, the tubes can be drawn out to a smaller diameter in the centre, then carefully broken in half)
- dropper / Pasteur pipette
- pencil and ruler
- distilled water
- test sample solutions (only minimal volumes required, e.g. a few drops of each on a spotting tile)
  - ‘banned substance’ F: approx. 1 mol dm\(^{-3}\) potassium (or ammonium) thiocyanate
    - **BOTH SOLIDS ARE HARMFUL, BUT SOLUTIONS ARE LOW HAZARD AT THIS CONCENTRATION**
  - ‘banned substance’ G: approx. 1 mol dm\(^{-3}\) potassium hexacyanoferrate(II)
  - developer solution, D: approx 0.5 mol dm\(^{-3}\) iron(III) chloride
    - **IRRITANT**
  - ‘urine samples’ R and T: water with yellow food colouring to give a suitable shade, then mixed to give:
    - R1 3:1 ‘urine’ + F
    - R2 2:1:1 ‘urine’ + F + G
    - T1 3:1 ‘urine’ + G
    - T2 no additions (‘clean’ athlete)

Allocate different combinations of R and T to different students or pairs.

Since the reagents involved are unrelated to actual anti-doping tests, their chemical identities should not be revealed. Label them only with their code letters.

**HEALTH AND SAFETY**

Capillary spotting tubes are fragile, with sharp ends if drawn out, but safe unless mishandled.

**PROCEDURE**

Students should have practised the techniques previously, but it may be advisable to remind them about taking care to avoid:

- finger prints on the plate
- damaging the coating while marking out the plate
- cross-contamination of the samples
- using too high a solvent level in the container
- allowing the solvent front to reach the top of the plate

Though the chromatogram spots are initially very pale, developing with Fe\(^{3+}\) ions converts F to deep red Fe(CNS), and G to Prussian blue. This should enable students to easily decide whether each ‘urine’ sample contains F or G, both or neither.

They are instructed to submit a brief report of their conclusions, which should include their chromatogram as evidence.
This activity should be given to students before they tackle the colorimetry exercise, since the spot tests establish that sample Z contains ‘banned substance’ H. They can then go on to determine its concentration using colorimetry.

Students perform simple spot tests to decide whether or not four samples contain either of two ‘banned substances’. These are just iron(II) and iron(III) ions, chosen because they give easily recognisable deep colours with common reagents – hexacyanoferrate(III) and thiocyanate ions respectively.

Students should appreciate that a spot test shows whether or not a specific substance is present. A separate test is needed for each substance, so spot testing to identify a complete unknown would be very laborious. However, they are quick and easy if you know what you need to detect.

Students may also realise that something similar to spot testing applies when a developer is used to reveal colourless spots on a chromatogram. There, however, the same reagent reacts with all the spots, and their identification involves position as well as colour.

**EQUIPMENT (PER STUDENT OR PAIR)**

- spotting tile with at least 12 cavities
- If unavailable, use chromatography paper with spotting positions marked in pencil on a 4x3 or 6x2 grid at 3 cm spacings. Fine droppers should then be used to avoid drops spreading too far and merging.
- 6 x droppers
- a few drops each of:
  - H: approx 0.1 mol dm\(^{-3}\) NH\(_4\)Fe(SO\(_4\))\(_{2}\)12H\(_2\)O [ammonium iron(III) sulfate.12-water]
  - J: approx 0.1 mol dm\(^{-3}\) freshly prepared (NH\(_4\))\(_2\)Fe(SO\(_4\))\(_{2}\)6H\(_2\)O [ammonium iron(II) sulfate.6-water]
  - W: J diluted about 10 times
  - X: distilled water made very pale yellow with food colouring
  - Y: mixture of W and Z, about 1:1
  - Z: H diluted about 10 times
- dropping bottles of:
  - TR\(_1\): NH\(_4\)CNS [ammonium thiocyanate] (or KCNS [potassium thiocyanate])
  - TR\(_2\): K\(_3\)Fe(CN)\(_6\) [potassium hexacyanoferrate(III)]

**HEALTH AND SAFETY**

There are no specific safety issues. All reagents are non-hazardous.

**PROCEDURE**

The procedure is straightforward, though inexperienced students may require a little practice at delivering single drops. They should easily identify H in samples Y and Z by the formation of deep red [Fe(H\(_2\)O)\(_5\)(SCN)]\(^{2+}\), and J in W and Y by the formation of Prussian Blue.

To reinforce the point that spot tests are very sensitive, the students could dilute one drop of H with nine drops of water (10x dilution) and test the diluted solution – then repeat this (100x dilution), and so on until the red coloration no longer appears. They should be able to reach 10 000x.
Prior to this activity, students should perform the Spot Tests exercise, from which they should find that sample Z contains ‘banned substance’ H.
To demonstrate proficiency with a colorimeter, students must generate the appropriate calibration curve and determine the concentration of H in sample Z.
To render H sufficiently coloured at low concentration, they react it with the reagent used in the spot test.

**EQUIPMENT (PER STUDENT OR PAIR)**

- colorimeter and multimeter
- Note: green LED required, or blue-green filter if commercial colorimeter used
- 2 x 100 cm³ beakers
- 2 x droppers
- 10 cm³ measuring cylinder
- 25 cm³ measuring cylinder
- glass stirring rod
- rack of six test tubes
- rubber bung
- box to cover colorimeter
- 25 cm² of standard solution of H: 50 mg dm⁻³ NH₄Fe(SO₄)₂·12H₂O [ammonium iron(III) sulfate.12-water]  
  Dissolve 0.50 g [IRRITANT WHEN SOLID] in 1 dm³, then accurately dilute by 10 (eg 25.0 to 250 cm³)
- 6–7 cm³ of sample solution Z: Known dilution of solution H, between 10 and 40 mg dm⁻³, set between the calibration values, such as 16 or 33 mg dm⁻³.  
  Various concentrations labelled Z₁, Z₂ etc may be allocated to different students. Concentrations must be known to teacher but not to students
- 30 cm² of 1 mol dm⁻³ ammonium thiocyanate solution [AMMONIUM THIOCYANATE IS HARMFUL WHEN SOLID]

**HEALTH AND SAFETY**

There should be no safety problems with this Procedure. The solutions are non-hazardous.

**PROCEDURE**

Students should already be familiar with the use of the particular colorimeter. If a commercial model is used, pre-set the filter to blue-green – approx. 490 nm if set by wavelength.
They make a red solution [iron(III) thiocyanate] corresponding to H concentration 50 mg dm⁻³, from which they prepare calibration samples. Concentrations are expressed as quantities rather than percentages. Check that they have calculated these correctly.
For compatibility with the calibration curve, the unknown sample Z is also be mixed with an equal volume of ammonium thiocyanate solution.
To demonstrate proficiency in titration techniques, students show that they can determine the percentage purity of an over-the-counter pharmaceutical, ibuprofen. Ibuprofen has low solubility in water, but is readily soluble in ethanol. Having a carboxylic acid (-COOH) group, it can simply be titrated against a known alkali.

Strictly, the solution should be prepared accurately in a volumetric flask. However, at this level, careful measurement of 100 cm³ in a measuring cylinder will suffice.

The sodium hydroxide solution should be freshly prepared, its concentration checked, and if necessary adjusted. If too low, a larger titre will be obtained, leading to a result above 100%.

It is also advisable to check whether the ethanol contains any acid impurity, which would again increase the titre and apparent purity. Titrate 25 cm³ of ethanol without ibuprofen against the sodium hydroxide added dropwise. If the titre is greater than 0.2 cm³, instruct students to subtract this from their average volume of alkali when calculating the mass of ibuprofen in step 7.

**EQUIPMENT (PER STUDENT OR PAIR)**

- 25 cm³ pipette and filler
- 50 cm³ burette
- small funnel (for filling burette)
- clamp stand
- 100 cm³ measuring cylinder
- 2 x 250 cm³ conical flasks
- access to a balance reading to two or three decimal places
- sample I: 0.8 ± 0.05 g powdered ibuprofen in weighing bottle or stoppered test tube
- 100 cm³ ethanol
- 100 cm³ 0.050 mol dm⁻³ sodium hydroxide (freshly prepared) [IRRITANT]
- phenolphthalein indicator
- wash bottle of distilled water

**HEALTH AND SAFETY**

Ethanol and phenolphthalein solution are harmful and highly flammable. All flames should be extinguished before the start, in case of spillage or breakage.

Sodium hydroxide at this concentration is an irritant, but the solid and any concentration higher than 0.5 mol dm⁻³ used to prepare the standard solution are corrosive.

**PROCEDURE**

If students have not previously met weighing by difference, the concept should be introduced prior to this exercise. The practicalities are simple enough, but the concept can be confusing when they are used to mass increasing during weighing out. The alternative is to tare the full container, and note the loss of mass when the sample is emptied out, but the resulting negative mass can also be confusing.
Stress that the sample must be completely dissolved in the ethanol, and the solution well mixed, otherwise each 25 cm³ portion will contain different amounts of dissolved sample.

After pipetting out three portions, they should perform their final run with the solution that remains in the original flask. Since only just 25 cm³ remains, pipetting would be tricky.

Students do not need to understand the origin of 0.0412 in step 7, which involves the molar mass of ibuprofen, 206.

For tablets that are close to 100% ibuprofen, the expected titre for 0.8 g is about 19 cm³.

To check student’s results, compare their percentage purity with the manufacturer’s figure.

- Weigh a known number of whole tablets (from the same batch as sample I).
- Determine the average mass per tablet.
- Compare this with the dose per tablet stated on the packet, converting mg to g.

\[
\text{percentage purity} = \frac{\text{stated dose per tablet}}{\text{average mass per tablet}} \times 100
\]

If desired, students could do this for themselves.
CATCHING THE DRUG CHEATS

{ROLE MODELS} JESSICA NEISEN

NAME: JESSICA NEISEN
ORGANISATION: QUEEN’S UNIVERSITY BELFAST, CCRCB
JOB TITLE: PHD STUDENT

1. WHAT DO YOU DO?
I’m a research student studying the interaction between cancers and the normal cells that surround them.

2. DESCRIBE YOUR TYPICAL WORK DAY
On a typical day, I grow cancer cells in flasks so that I can treat them in different conditions and run experiments testing gene expression and the activity of proteins involved in aggressive cancers.

3. WHAT HOURS DO YOU WORK?
Whatever is necessary. Typically I work 9-5, but if I have an experiment running, I’ll be in for late nights or weekends.

4. WHICH SUBJECTS DID YOU ENJOY MOST AT SCHOOL?
English! I was always interested in science, but I never liked my teachers, so normally dreaded going to class. Science was more what I wanted to do as a career as I felt studying English would have more limited career options. It has helped with my science writing though.

5. WHAT QUALIFICATIONS DO YOU HAVE?
I have a BSc (Hons) in Microbiology and an M.Phil in Molecular Oncology.

6. TO WHAT DEGREE WERE STEM SUBJECTS IMPORTANT IN GETTING YOUR JOB?
Absolutely crucial! The science part is obvious, but being familiar with maths and technology is a major part of my every day work and was essential for successfully completing my previous degrees.

7. WHAT WERE THE MAIN FACTORS THAT ATTRACTED YOU TO YOUR CURRENT JOB?
I started my PhD because it was a perfect combination of everything that I wanted to do with my life. I love the research side of it because it’s a different challenge every week, but before I did my degree, I seriously considered teaching as a career. Eventually science won out, but the beauty of academic research, for me, is that I get the best of both worlds.
8. HOW DID YOU GO ABOUT ENTERING INTO THIS CAREER/ GETTING EXPERIENCE AND DO YOU HAVE ANY ADVICE FOR SOMEBODY LOOKING INTO THE SAME CAREER?

I sent letters and emails everywhere! I applied for every research project I was interested in, talked to everyone I knew in the field until eventually something came through. There are always opportunities available, especially for short term projects, you just have to ask! Make sure you persevere. Classes, and eventually lectures are always going to be boring at some point because you have to learn all the basics before you specialise. Eventually you will get to do the things that interest you most. Then one day it’ll hit you that most of the stuff you didn’t enjoy at school is actually pretty useful.

9. WHAT ARE THE BEST/ WORST THINGS ABOUT YOUR JOB? WHAT DO YOU FIND MOST REWARDING ABOUT IT?

The worst bit is when there are technical issues with an experiment and having to work those out. There is nothing worse than having an experiment in your head, but not having the equipment or know-how to make it work. The best bit is when you finally get it to work!

10. WHAT ARE THE CHALLENGES OF YOUR JOB?

Not every idea is a good idea. It can be very hard going to work every day when you have had a spell of hypotheses that don’t turn out to be right.

11. WHAT HAS BEEN THE HIGHLIGHT OF YOUR CAREER SO FAR? WHAT HAS BEEN THE MOST EXCITING/ INTERESTING PROJECT YOU HAVE WORKED ON?

I get stupidly excited about really little things! Most recently I’m working with a group to develop a computer algorithm that automatically scores images of prostate tissue that have been stained for a protein that should tell doctors how aggressive a person’s cancer is.

12. HOW DO YOU HOPE TO PROGRESS IN YOUR FIELD OVER THE COMING YEARS?

One day I’d love to lead my own research group and be able to contribute to a whole range of projects and collaborate with other groups in the field.

13. WHAT PASSIONS AND INTERESTS DO YOU PURSUE IN YOUR PERSONAL TIME?

I love music so volunteer for a music charity that aims to engage people of all ages in music based careers. I also play softball and volleyball and help coach the university’s softball team.
NAME: RHIANNON LOWE

ORGANISATION: GLAXOSMITHKLINE (GSK)

JOB TITLE: SENIOR SCIENTIST

1. WHAT DO YOU DO?
I am a scientist in Safety Assessment. I look at the safety of drugs and I'm currently looking at the safety of gene therapy.

2. DESCRIBE YOUR TYPICAL WORK DAY
A typical day is varied but could include a mixture of lab work and desk based work including: DNA extraction, PCR (Polymerase Chain Reaction) and conference calls to our linked Italian gene therapy group.

3. WHAT HOURS DO YOU WORK?
37.5hrs a week but we are able to do flexitime

4. WHICH SUBJECTS DID YOU ENJOY MOST AT SCHOOL?
Science, Drama and History

5. WHAT QUALIFICATIONS DO YOU HAVE?
I have A-levels in Biology, Chemistry and Physics. I then completed a BSc in Applied Biology and a PhD in Immunology/Virology.

6. TO WHAT DEGREE WERE STEM SUBJECTS IMPORTANT IN GETTING YOUR JOB?
Essential, although I would say I have not yet needed my Physics A-level. I think two sciences at A-level would have been OK.

7. WHAT WERE THE MAIN FACTORS THAT ATTRACTED YOU TO YOUR CURRENT JOB?
A real sense of helping to improve people's lives and using my brain.

8. HOW DID YOU GO ABOUT ENTERING INTO THIS CAREER/ GETTING EXPERIENCE AND WHAT ADVICE WOULD YOU GIVE SOMEBODY LOOKING INTO THE SAME CAREER?
I would recommend doing work experience at any opportunity! I got my foot in the door by asking staff if I could do some volunteer work at the hospital, which led to me obtaining a 1st in my BSc final year project in diabetes research. We have letters at GSK everyday asking for work experience and we do try and offer as many placements as we can –so just go for it! The abpi website (www.abpi.org.uk) is also really helpful and has lots of case studies which may give an insight into jobs you may not have considered.
9. WHAT ARE THE BEST/WORST THINGS ABOUT YOUR JOB? WHAT DO YOU FIND MOST REWARDING ABOUT IT?
The best thing is that the work I am currently doing has a direct effect on patients. I also get lots of opportunities to keep learning; from invited guest lectures on site, conferences (one was in Monte Carlo!) to my company, GSK, funding my PhD.

10. WHAT ARE THE CHALLENGES OF YOUR JOB?
Juggling different projects and dealing with changing deadlines although it is also what makes it interesting. Time zones can be a challenge: next week I have a conference call with Utah that starts 3pm their time and ends 1.30am our time.

11. WHAT HAS BEEN THE HIGHLIGHT OF YOUR CAREER SO FAR? WHAT HAS BEEN THE MOST EXCITING/INTERESTING PROJECT YOU HAVE WORKED ON?
My Imperial college PhD ceremony last week at the Royal Albert Hall. Having the opportunity to complete a part time PhD (funded and supported by GlaxoSmithKline) whilst working full time with two young children was a challenge but absolutely worth it. The PhD was looking at a new treatment for viruses, in particular Hepatitis C, which has no vaccine and affects 1:12 of the world’s population.

12. HOW DO YOU HOPE TO PROGRESS IN YOUR FIELD OVER THE COMING YEARS?
In the gene therapy field we are really at the cutting edge, the study we are working on in collaboration is for ADASCID, where children are born with no immune system (bubble boy). I am looking forward to many more lives being saved due to gene therapy.

13. WHAT PASSIONS AND INTERESTS DO YOU PURSUE IN YOUR PERSONAL TIME?
I really enjoy volunteering in schools as a STEM Ambassador, hosting work experience, giving careers talks and taking workshops out to schools including anti-doping as GSK provided all anti-doping testing for the 2012 Olympic and Paralympic games. I also do mad science sessions for disabled children every holiday.

Inspire young people in science, technology, engineering and maths.
Become a STEM Ambassador.
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