

Drug tests – Students Notes

By Doaa George, based on the workshop investigation by Claire Lenehan



Figure 1 – Forensic science exhibition II

Introduction

Forensic science is the science used to unveil the mysteries associated with crime. The name forensic has a Latin origin and means a public discussion or a market place. Criminality directly affects the public and solving crimes is essential to supporting a safe environment. When someone commits a crime, he/she always leaves some evidence behind at the crime scene and this evidence can be used to discover the culprit. If you have watched forensic files, you would have seen how different crimes have been solved using evidence found at the scene of the crime. Forensic scientists use a number of scientific tools to achieve their goal. In today's experiment you will take the role of a forensic scientist by using two popular techniques used in solving crime cases.

Risk analysis

Most of the chemicals you will be using are flammable and corrosive. Corrosive materials can damage body tissues including skin, eyes and the respiratory tract. You have to handle these chemicals with extra care and wear gloves and goggles at all times. It is best to conduct the experiments in a fume-hood.

You will be using organic solvents, therefore make sure you place the waste in the appropriate container.

Never look directly nor expose others to UV light as this can simply cause eye damage.

Questions

Do you think the experiments undertaken are reliable enough to prove or disprove a guilty verdict?

Aim

Use thin layer chromatography and chemical spot tests to identify the presence of a number of drugs.

Plan

Form groups of three and carefully read the method provided. Make sure you follow the safety precautions and assign a role for each member in the group. You will be doing two main experiments: Thin Layer Chromatography (TLC) and Chemical Spot Tests (CST). Make sure you familiarise yourself with both experiments before you start.

Materials

Part A: Chemical Spot Tests (CST)

The following reagents have been prepared for you.

- Marquis Reagent (10 ml of 37% formaldehyde added to 100ml of concentrated sulfuric acid)
- Nitric Acid Reagent (Concentrated Nitric Acid)
- Mecke's Reagent (0.5g of selenous acid dissolved in 100ml of concentrated sulfuric acid)
- Mandelin reagent (1g ammonium vanadates (metavanadate) dissolved in 100ml concentrated sulfuric acid)

The following **reference materials** have been prepared for you:

- 2-Chloroacetophenone (Mescaline mimic)
- Indole (LSD mimic)
- Aspirin
- Paracetamol
- Caffeine
- Panadeine powder (Paracetamol + codeine, opiate mimic)
- Nurofen Tablet (Ibuprofen)

The following **validation materials** have been prepared for you:

- No-Doz Tablet (caffeine)
- Aspirin Tablet

The following **unknown samples** are to be analysed:

- Unknown A (white powder)
- Unknown B (white powder)
- Unknown C (white powder)

Equipment required:

- 12-well porcelain test plate
- Pasteur pipettes
- Pencils
- Spatulas

- Colour reference chart
- Mortar and Pestle

Part B: Thin-Layer chromatography (TLC)

The following **reference materials** have been prepared for you:

- 1% w/v 2-Chloroactophenone (Mescaline mimic) in diethyl ether.
- 1% w/v Indole (LSD mimic) in diethyl ether.
- 1% w/v Aspirin in diethyl ether.
- 1% w/v Paracetamol in diethyl ether.
- 1% w/v Caffeine in diethyl ether.
- 1% w/v Panadeine (Paracetamol+Codeine (Opiate)) Tablet in diethyl ether.
- 1% w/v Nurofen (ibuprofen) Tablet in diethyl ether.
- 90:10 Diethyl ether : Methanol

The following **unknown samples** are to be analysed:

- Unknown A dissolved in diethyl ether
- Unknown B dissolved in diethyl ether
- Unknown C dissolved in diethyl ether

Equipment required:

- TLC plates and scissors
 - Beakers with watch glasses to cover (developing chamber)
 - Filter paper (11cm)
 - Capillary micropipettes (premade using Bunsen burner and Pasteur pipette)
 - UV-lamp
 - Iodine chamber
- Pencils and rulers (self provided)

Conduct

Make sure you understand the techniques you are using and why you are using them. Discuss with your team members to ensure you all know what you are doing. Read through the procedures before you start, if you have any questions or if you are unsure about any terms or steps, ask your teacher.

Procedure

Part A: Chemical Spot Tests (CST)

1. Label the wells of the porcelain test plate with the various substances to be tested.
2. Fill a disposable Pasteur pipette with the Marquis chemical spot test reagent.
3. Carefully apply 2 drops of the Marquis reagent to each well on the test plate. Dispose of pipette after use in appropriate container.
4. Make sure no colour change is observed at this stage as colour change indicates cross contamination.
5. Using separate spatulas to avoid cross-contaminating the drug samples, carefully transfer a small amount of each of the twelve substances (reference materials, validation materials

and unknown samples) into separate wells on a porcelain test plate (enough material that would fit on end of toothpick).

- After 5 minutes, note the final colour obtained for each drug sample. Fill in the following chart.

	Marquis	Nitric Acid	Meckes	Mandelin
2-chloroacetophenone				
Indole (LSD Mimic)				
Aspirin				
Paracetamol				
Caffeine				
Nurofen (Ibuprofen)				
Panadeine				
No-doz tablet				
Aspirin tablet				
Unknown A				
Unknown B				
Unknown C				

- Rinse the porcelain test plate with demineralised water into a large waste beaker and wipe dry using paper towel.
- Proceed to test the sample with the other three reagents by repeating the steps 2-7.

Part A: Thin Layer Chromatography (TLC)

- Prepare the developing chambers by adding approximately 5ml of mobile phase solvent to the wide-mouth jars.
- Cut circular filter paper into a rectangle and place into chambers to act as a wick.
- Cap each jar and allow chamber to equilibrate for 10 minutes while preparing your TLC plates.
- Obtain enough (3) TLC plates to test the drug standards and three unknown samples.
- Lightly draw the starting position by a pencil line ~ 1cm from the bottom of the plate. Mark off four positions on the plate with pencil and be sure to label what each spot represents.
- Obtain a capillary micropipette for each drug standard and your unknown samples.
- Immerse the end of the capillary micropipette into the vial until some of the sample is drawn in.
- Allow the drop at the end of the capillary to gently touch the plate on the starting line. Keep the spots small (~ 2mm) and concentrated by applying the sample 2-3 times and allowing the spot to dry between applications.
- Repeat this procedure for each of the sample being tested.
- To each plate, apply 3 drug standards and an unknown drug sample.
- Place the TLC plate in the chamber using forceps.
- Allow the solvent front to reach a level about 1 cm from the top of the plate.
- Remove the plate from the chamber and mark the position of the solvent front immediately. Set the plate on a paper towel in the fume hood to dry.
- Visualize the spots by illumination under a UV lamp.
- Trace around each spot lightly and mark the middle using a pencil.
- Place the plate in the iodine chamber for about 5 minutes.
- Trace each spot and note the characteristic including if a sample did not stain.

18. Measure the distance travelled by each component in the results section.
19. Calculate the R_f values. Record them in your notebook.

Analysis

Take photos of the results for both experiments to keep them as a record. Write down your observations, keep note of what worked well and what did not.

Thoroughly analyse the results obtained from the two experiments and make a rough estimate of what the unknowns could be in preparation for the discussion section.

Problem Solves

Discussion

Based on the TLC and CST results and other relevant sources if necessary, deduce the tentative identity of the compound(s) present in the three unknown samples. Include a detailed interpretation of your findings with adequate reasoning.

Conclusion

State whether the techniques used are efficient for revealing the unknown substances.

Do you have any recommendations to find more solid results or for future experiments?

Was your hypothesis supported?

Link to Figure 1 <https://www.flickr.com/photos/utslibrary/37317237446/in/photolist-p88F4L-XR8vMS-6LvoRA-XUzEhB-pAbfeJ-ebZXcc-cvezPm-cveA29-ebZXip-dV9B2q-cvexBG-cvezuh-cvey1b-ec6C41-ebZX6H-cvevc9-99aAYu-YRAB9o-cveuRf-hAA2n5-q2Gq2f-p83XoP-EZeuxu-dfJRGr-cveQhL-cvePxG-cveQFC-dfJV7S-gpXMeo-SHBjU2-YRAAzC-qsbhwG-pvtYZx-5zEjFG-qaNkMX-pvtxKK-StTzKd->

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