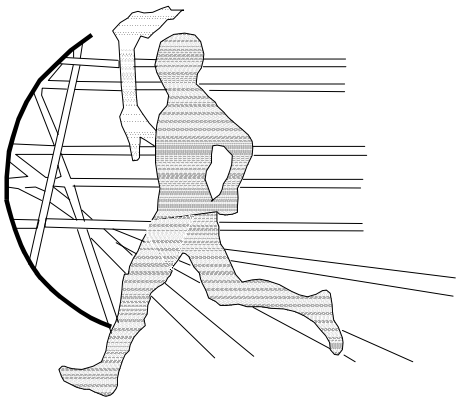

London and District Science Olympics

Annual Competition

MYSTERY SOLUTIONS

Sponsored by the Department of Chemistry,
University of Western Ontario

Grades 11 & 12 Chemistry

Thames Valley District School Board

London District Science and Technology Fair Inc

The London District Catholic School Board

Faculty of Science - *The* University *of* Western Ontario

http://www.ldstf.ca/olympics/

MYSTERY SOLUTIONS

RULES

Chemistry Grades 11 & 12

1. THE EVENT

Teams will be provided with a specified set of testing reagents, droppers and other necessary glassware and a flame source. Eight unknown solutions will be provided in separate numbered vials.

The team will be given twenty minutes to identify the ion type in each vial.

The team will have 5 minutes at the end of the event to clean the glassware.

Students may bring in (a) one 8.5"x 11" sheet of paper with any notes they wish written using both sides and (b) the Rules for the event.

2. UNKNOWNS

Unknown solutions in three categories identified as

a) anion only (3 unknowns)

b) cation only (3 unknowns)

c) both cation and anion (2 unknowns)

Teams will be given about 15 mL of each solution and the individual tests should be run on samples of about 1 mL.

Fresh solution should be used for each new test.

3. SCORING

Teams will be judged on the number of anions and cations determined correctly.

Ties will be broken by questions on the chemistry behind the analytical procedures (e.g., write an equation for the test for the carbonate ion).

1. ANION UNKNOWNS

You will be provided with about 15 mL of 3 solutions and you will be asked to determine the anion present in each. Each of these anions will be one of acetate, bromide, carbonate, chloride, iodide, or phosphate as their sodium salt as shown below.

sodium acetate	CH_3CO_2Na
sodium bromide	NaBr
sodium carbonate	Na_2CO_3
sodium chlorine	NaCl
sodium iodide	NaI
sodium phosphate	Na_3PO_4

It is up to you to devise the quickest scheme to do this using the reagents and procedures which follow :

Cl^- Br^- I^-

Solution remains mildly acidic.
pH approximately 4

Figure 2 *Categories of Anions. pH ≤ 7*

$CH_3CO_2^-$ CO_3^{2-} PO_4^{3-}

Solution becomes somewhat basic
pH = 7-10

Figure 3 *Categories of Anions pH > 7*

The anions can be conveniently broken into 2 categories based upon the pH of the solution when they dissolve. Use pH paper, not litmus paper, if available.

Individual anions

CHLORIDE Cl^-

Add a few drops of dilute nitric acid and then add about 1 mL of silver nitrate ($AgNO_3$) solution. A white precipitate of AgCl separates.

BROMIDE Br^-

Add a few drops of dilute nitric acid and then add about 1 mL of silver nitrate solution. A cream coloured precipitate of AgBr forms.

IODIDE I^-

Add a few drops of dilute nitric acid and then add about 1 mL of silver nitrate solution. A yellow coloured precipitate of AgI forms.

Note: The three halides (chloride, bromide and iodide) all give precipitates with silver ions but these can be distinguished by their colours. With a little practice you should have no trouble telling these apart. However, to confirm your choice, you can add an equal volume of concentrated ammonium hydroxide to each test tube containing the precipitate. After shaking, the silver chloride will redissolve; the silver bromide will mostly redissolve; and the silver iodide will remain undissolved.

To further distinguish between bromide and iodide:
Add about 1 mL of paraffin oil and a few drops of chlorine water. A red or orange colour in the paraffin oil layer indicates bromide; a violet colour indicates iodide.

ACETATE $CH_3CO_2^-$

This solution will have a pH of about 7. Add about 1 mL of silver nitrate solution. A white precipitate will form slowly. This precipitate will redissolve upon the addition of about 1 mL of dilute nitric acid. The halide's precipitate will not redissolve.

CARBONATE CO_3^{2-}

Add about 1 mL of dilute nitric acid and look for the formation of bubbles of carbon dioxide.

PHOSPHATE PO_4^{3-}

To 1 mL of the phosphate solution add 1 mL of dilute nitric acid and warm almost to boiling. Then add 1 mL of ammonium molybdate $(NH_4)_2MoO_4$ solution and a yellow coloured precipitate will form.

2. CATION UNKNOWNS

You will be provided with about 15 mL of 3 solutions and you will be asked to determine the cation present in each. Each of these cations will be present as either the chloride or nitrate as listed below.

Aluminum chloride	$AlCl_3$
Ammonium chloride	NH_4Cl
Barium nitrate	$Ba(NO_3)_2$
Copper (II) nitrate	$Cu(NO_3)_2$
Iron (III) nitrate	$Fe(NO_3)_3$
Sodium chloride	$NaCl$
Strontium nitrate	$Sr(NO_3)_2$
Zinc chloride	$ZnCl_2$

Your task is to identify the specific cation present in each solution as quickly as possible using the reagents and procedures which follow:

ALUMINUM Al^{3+}

Add about 0.5 mL of ammonia solution (ammonium hydroxide). A gelatinous white precipitate will form.

AMMONIUM NH_4^+

Add about 0.5 mL of a dilute sodium hydroxide solution. Warm the test tube in the hot water bath provided. Immediately test the vapours emitted from the test tube with moist pH paper. A basic pH should be observed.

BARIUM Ba^{2+}

Add about 0.5 mL of a sodium sulfate solution. A white precipitate should form. Both Ba^{2+} and Sr^{2+} give this result. Barium ions can be confirmed using a FLAME TEST (See below).

COPPER (II) Cu^{2+}

Add about 0.5 mL of an ammonia solution (ammonium hydroxide) and shake. A deep blue colour indicates Cu^{2+} ions were present. Copper (II) can also be confirmed by a flame test.

IRON (III) Fe^{3+}

Add a few drops of a dilute sodium hydroxide solution. A copious brown precipitate should be observed.

SILVER Ag^+

Add a few drops of dilute nitric acid and then add some drops of a sodium chloride solution. A white precipitate should be observed.

SODIUM Na^+

There is no simple test for sodium ions. Use a flame test.

STRONTIUM Sr^{2+}

Add about 0.5 mL of a sodium sulfate solution. A white precipitate should form. Both Ba^{2+} and Sr^{2+} give this result. However, Sr^{2+} gives a flame test quite different than Ba^{2+}.

ZINC Zn^{2+}

Add a few drops of an ammonia solution (ammonium hydroxide) and a white precipitate should form. However, if you add about 0.5 mL of a concentrated ammonia solution it should redissolve. Compare this result with aluminum.

3. COMPOUND UNKNOWNS (BOTH CATION AND ANION)

You will be given about 15 mL of 2 solutions and you will be asked to identify both the cation and the anion present in each and thus deduce the identity of the compound that has been dissolved.
The two compounds will be composed of the cations and anions from the previous tests. You should use the tests presented earlier for the anions and cations separately.

4. FLAME TESTS FOR CATION IDENTIFICATION

Put the end of a splint into the solution so that some of the solution adheres to it. Adjust the bunsen burner / butane torch so as to produce a blue flame. Apply the unknown solution to the tip of the inner blue flame. Do not burn the splint as this will affect the colour of the flame.

RESULTS

Barium	Green
Copper	Green
Sodium	Intense yellow/orange (colours whole flame)
Strontium	Bright red

You must try these tests with known compounds in order to become familiar with the colours, as words cannot describe the colours adequately.

5. A LIST OF REAGENT SOLUTIONS AND NECESSARY APPARATUS

The following is a list of reagent solutions and apparatus that will be available on the day of the contest. You should design your schemes with this in mind. If you have a problem locating specific reagents, do not hesitate to contact the co-ordinator.

REAGENT SOLUTIONS

Ammonium hydroxide	1.0 M, Conc
Ammonium molybdate	0.5 M
Chlorine water	Cl_2 / water
Nitric acid	1.0 M
Silver nitrate	0.1 M
Sodium chloride	0.5 M
Sodium sulfate	1.0 M
Sodium hydroxide	1.0 M
Paraffin Oil	

APPARATUS

Test tubes and holder
Disposable pipettes
pH paper
Bunsen burner / Butane torch
Wooden splints

6. PREPARATION INSTRUCTIONS FOR REAGENTS AND UNKNOWNS

After you develop a scheme and procedure for analyzing the possible unknowns you will receive, it wouldn't hurt at all to practice. This means that you must prepare your own unknown test solutions and reagent solutions. The following list should prove helpful. About 10 mL of each of these should prove more than sufficient for any practice round.

REAGENTS

Ammonium hydroxide 1 M

Add about 1 mL of concentrated ammonium hydroxide to 10 mL of distilled water.

Ammonium molybdate 0.5 M

Add about 1 gram of the solid to about 10 mL of distilled water.

Nitric acid 1 M

Add about 1 mL of concentrated nitric acid to 10 mL of distilled water. Be careful with concentrated nitric acid for it can burn you and put holes in your clothes.

Silver nitrate 0.1 M

You want about 0.2 grams in 10 mL of distilled water. If this solution is cloudy add a few drops of dilute nitric acid. Two or three medium sized crystals will suffice to give you 0.2 g. Don't get this solution on your hands as they will turn black.

Sodium chloride 0.5 M

You want about 0.5 g in 10 mL of distilled water. Ten pinches of salt is about right.

Sodium hydroxide 1 M

Three pellets of sodium hydroxide in 10 mL of distilled water is about right. The pellets may be a little slow to dissolve but just give them time.

Sodium sulfate 1 M

You want about 1.4 g in 10 mL of distilled water. About ten pinches of sodium sulfate will be about right.

Unknowns

To make a solution of your unknowns use the equivalent of two or three generous pinches of the solid in about 2.5 cm (height) of distilled water in a test tube. The solids may be a little slow to dissolve. A little shaking, stirring, and gentle warming will help but so will patience.

The following compounds are recommended for making up unknowns but other soluble salts can be used.

Aluminum chloride
Ammonium chloride
Barium nitrate
Copper (II) nitrate
Iron (III) nitrate
Silver nitrate
Sodium acetate
Sodium bromide
Sodium carbonate
Sodium chloride
Sodium iodide
Sodium phosphate
Strontium nitrate
Zinc chloride

7. SAFETY IN THE lABORATORY

Safety is the first priority in all lab work.

The University safety policy, devised by Occupational Health and Safety, mandates "maximal skin coverage" for all laboratory personnel, including students. Ankle-length pants and socks that cover the ankle are required. No shorts or capris are permitted. Exposed midriffs are not allowed. Long hair must be tied back. Shoes must completely cover your feet: toes, sides, heel and the entire upper foot. Running shoes are generally acceptable. Sandals, open-toed shoes, ballerina shoes, open-top shoes, and shoes with cut-outs or openings are not acceptable.

Safety glasses will be provided. Make sure you wear safety glasses at all times in the laboratory.

Read and understand all instructions prior to starting the procedure. If you do not understand anything ask the supervisor.

If you spill any materials or break glassware contact the supervisor before attempting a cleanup.

Do not carry out unauthorized experiments.