Polarized Microscopy Analysis of Unknown Materials

1. Scope

The purpose of this procedure is to determine an unknown material by microchemical analysis. Elements, cations and anions may be precipitated from a solution using specific reagents and each has a characteristic crystalline precipitate or colored solution.

2. Terms

anion – negatively charged ion

cation – positively charged ion

precipitate – the solid matter that separates from a solution

3. References

Bloss, F. Donald, An Introduction to the Methods of Optical Crystallography, Holt, Rinehart, and Winston, New York, 1961.

Chamot, E.M., Mason, C.W., Handbook of Chemical Microscopy Vol. I, John Wiley and Sons, Inc. New York, 1958.

Chamot,E.M., Mason, C.W., Handbook of Chemical Microscopy Vol. II, John Wiley and Sons, Inc. New York, 1940.

McCrone, W.C., Delly,J.G., The Particle Atlas, Vol. I-VI, Ann Arbor Science Publishers, Inc. 1973.

Behrens-Kley, Microscopical Identification of Organic Compounds, translated by Richard E. Stevens, Microscope Publications Ltd., 1969.

McCrone , W.C., Johnson, R.I., Techniques, Instruments and Accessories for Microanalysis: A User's Manual, Walter C. McCrone Associates, Inc., 1974.

Whitman, V. L. & Wills, W. F. Jr., "Extended Use of Squaric Acid as a Reagent in Chemical Microscopy, Vol 25, 1977

Wills, William F. Jr., "Squaric Acid Revisited" Microscope Publication Ltd., Vol 38 1990.

4. Examination Procedures

4.1. Evidence Types

Any item, material or substance that can be examined by microchemical testing.

4.2. Instruments and Equipment

Polarized Light Microscope
Glass slides
Cover slips
Forceps, tweezers, probes, tungsten needles
Droppers

4.3. Reagents and Chemicals

Specific reagents and conditions for each precipitation can be found in volume II of The Handbook of Chemical Microscopy. Some of the more common of these are listed below:

Microscopic Tests for Inorganic Ions

Ions	Reagents
Sodium	Uranyl acetate, squaric acid
Potassium	Chloroplatinic acid, perchloric acid, squaric acid
Ammonium	Chloroplatinic acid, iodic acid, squaric acid
Calcium	Ammonium carbonate, sulfuric acid
Strontium	Ammonium carbonate, iodic acid, squaric acid
Barium	Ammonium carbonate, potassium ferrocyanide, squaric acid
Magnesium	Ammonium hydroxide and phosphate
Zinc	Oxalic acid, potassium mercuric thiocyanate
Copper, lead	Zinc, potassium, mercuric thiocyanate
Mercury	Potassium bichromate, lead
Aluminum	Ammonium bifluoride, cesium sulfate
Tin	Zinc, cesium chloride
Arsenic	Ammonium molybdenate, cesium sulfate and

	potassium iodide
Chromium	Silver nitrate, lead acetate
Uranium	Thallous nitrate and ammonium carbonate
Fluoride	Sodium fluosilicate
Iron	Potassium ferrocyanide, potassium thiocyanate
Carbonate	Silver nitrate, calcium acetate
Nitrate	Silver nitrate, nitron sulfate
Cyanide	Silver nitrate, ammonium sulfide and ferric chloride
Chloride	Silver nitrate, thallous nitrate

4.4. Procedural and Chemical Precautions

Refer to the TBI Safety Manual for general safety requirements and hazard information regarding the use of reagents and solvents, and overall safety guidelines.

Protective attire, including laboratory coat, mask, gloves and eye protection should be used when working with clothing and/or bloodstained items.

Decontamination of a scientist's work area should be performed after each use, but shall be done after analyzing bloodstained items.

Hazardous chemicals shall be used in a chemical fume hood.

When handling concentrated acids, eye protection and laboratory coats shall be worn.

When diluting acids, always add acid to water.

When necessary, consult section and laboratory Material Safety Data Sheets (MSDS) regarding any chemical used in the Microanalysis section.

Label all generated solutions and reagents with appropriate warning stickers.

4.5. Limitations

The amount of sample available for analysis.

The amount of tests available to confirm an identification.

4.6. Procedure

Document submitted samples according to *Microanalysis Quality Assurance Policy*.

4.6.1. Identify one or more tests that may be applied to the identification of the suspected element or ion. Multiple tests are preferred although it is not always possible to find more than one microchemical test for a specific element or ion.

4.6.2. Prepare appropriate reagents as described in Vol. II of The Handbook of Chemical Microscopy or other reference text.

4.6.3. Any spot test or micro-chemical test method in a peer reviewed journal or academic text shall be performance checked prior to use. The check shall be documented in case notes and include the use of reference material(s) and blanks prior to use in casework.

4.6.4. Prepare a negative control from the solvent used to dissolve the unknown.

4.6.5. Prepare a positive control using a chemical compound that contains the specific element or ion being tested for.

4.6.6. Based on what the sample is perceived to be, the unknown sample is prepared for microscopy as described in Vol. II of The Handbook of Chemical Microscopy or other reference text.

4.6.7. Proceed with the analysis using the polarized light microscope. Normally, the reagent is dissolved in a small drop of water and made to run in contact with a test drop containing the unknown. Occasionally, it is best to add the reagent crystals directly to the test drop or to draw a reagent drop of solution across a dried residue from the test drop. The details of each test shall be studied before any actual testing. The negative control and the positive control shall be run before the unknown is tested.

4.6.8. The results from each reaction shall be thoroughly documented in case notes.

5. Measurement Traceability

There are no measurements of traceability associated with these tests.

6. Reference Materials

Known compounds with ions of interest to the particular case analysis.

7. Reports

Reporting results will vary depending upon the material analyzed and tests performed. Generally, the results should indicate the elements or ions identified. In most cases, no attempt to interpret the findings of the analysis will be provided. In some cases, it might be necessary to provide a limited list of items which might contain the elements or ions identified. Also, it might be necessary to identify compounds, elements, and ions, etc. which were not analyzed for.

Example: Microscopic examination and analysis of victim's shirt revealed a powder consistent with potato starch.