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First Step Hazard Categorization

TECHNIQUES FOR IDENTIFYING UNKNOWN MATERIALS

Hazard Categorization

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Hazard Categorization

Begin by obtaining the critical information first, take action, then as the situation allows, collect additional information.

Emergency responders are overwhelmed by the need for information during hazardous materials emergency response situations. Are responders at risk? Is there immediate danger to the public? Is there a fire or explosion hazard? Should water be used? Should runoff be contained? Because questions like these surfaces during hazardous materials situations, responders are confronted with the need to rapidly identify the hazards associated with unidentified materials.

Many commercially available products are available to aid first responders and site managers in characterizing the hazards associated with unknown materials. These products range from inexpensive multi-test strips and color indicating tubes, to systems costing thousands of dollars using flow charts and wet chemistry tests. The inherent problem with these commercial systems is the trade-off between the amount of information obtained and the time and training required to use the information successfully and effectively. Test strips provide information quickly, but they do not give the incident commander or site manager the range of information necessary to make informed decisions. Test strips may also only be suited for liquid phase unknowns. Likewise, indicator tubes may specifically identify a chemical, or class of chemicals, but are only useful for gas or vapor unknowns. Additionally, flow chart/wet chemistry systems, although very thorough in the range of information that can be obtained, may require hazardous materials responders to spend over an hour working through the flow chart for each unknown.

It may take over an hour using a flow-chart test kit to identify water.

Another problem common to flow chart systems is the potential for critical error to occur. The results of many tests are subject to interpretation, and it is not uncommon for two users to interpret the same test result in two different ways. A misinterpretation of one result can lead the user down the wrong flow chart path leading to misidentification of the unknown material. A Hazmat responder with limited training and experience in interpreting test results can be blinded by the constraints of a flow chart, and subsequently miss critical or subsidiary hazards of an unknown material.

For these reasons, the ***First Step*** procedures were developed. The ***First Step*** procedures balance the use of strip tests with wet chemistry tests and supplies the incident commander with the critical information needed to make expedient, safe decisions. These tests can be performed by a user with limited experience and training in usually less than 5 minutes, regardless of the phase of the

unknown. Because the user is not restricted to a flow chart or to the tests available only on a commercial multi-test strip, the user is able to assemble pieces of the puzzle together and draw conclusions about the nature and hazards of an unknown material. The equipment required to perform these tests is commercially available from a number of sources, and a test kit with enough equipment to characterize 500 samples can be assembled for approximately $250.

FIRST STEP PROCEDURES

Critical Information

Rapid (5 min.)

Inexpensive

Limited Training Required

The ***First Step*** procedure is not intended to replace other commercially available hazard categorization systems or laboratory analyses. These systems have proved indispensable for providing confirmation tests and for further characterization of an unknown beyond the ***First Step*** procedures. For example, if a user performed the ***First Step*** procedures and determined the unknown to be an oxidizing acid, that information would be passed to the incident commander, actions could be taken based on those results, and a flow chart system, or other procedure could be subsequently used for determination of the specific acid species. Similarly, if after performing the ***First Step*** procedures the responder suspected the unknown to be ammonium hydroxide, an ammonia gas indicator tube could be used for confirmation.

First step can also be a valuable tool for container or removal sites where a large number of unknowns need to be categorized quickly for removal and disposal. Often in these cases, it's not the specific identification of the chemical or material that is important, rather the Department of Transportation (DOT) hazard class for placarding and transportation from site or the RCRA classification. Even unknown chemicals need to be properly packed and shipped within DOT regulation while maintaining proper chemical compatibility. The following are the DOT hazard classes, although the properties of every class are not determinable using ***First Step*** procedures.

Class 1: Explosives, may be indicated by the Hairpin Test.

Class 2: Gasses, difficult to discern using ***First Step***, Reference the R10 Air Monitoring and Sampling Decision Tree.

Class 3: Flammable and Combustible Liquids, indicated by the Flammability and Char Tests.

Class 4: Flammable Solids, indicated by the Flammability and Char Tests.

Class 5: Oxidizing Substances, Organic Peroxides, indicated by the Oxidizer and Peroxide Tests.

Class 6: Toxic and Infectious Substances, generally non-discernable for most toxic chemicals using ***First Step***, Cyanide Tests indicates toxicity.

Class 7: Radioactive Materials, non-discernable with ***First Step***.

Class 8: Corrosives, indicated with pH Test.

Class 9: Miscellaneous or Environmentally Hazardous Materials, chemicals with no other hazard identified by ***First Step*** are classified as Class 9 if not identified.

For materials with multiple Hazard Classifications, CFR 173.2a should be referenced for determining the Primary and Secondary hazard. As a general rule, the lower the class number, the larger the hazard associated, however, some hazard combinations deviate from this standard.

First Step Considerations

The ***First Step*** procedures consist of up to 11 separate tests. A description of each test procedure is outlined, and result interpretation is given for the materials most commonly encountered during emergency response incidents. For any test, whether it is a quick qualitative analysis test or rigorous laboratory analysis, there are limitations, such as interferences, and detection limits. The most common limitation of each test will be presented.

Although many of the tests require the unknown to react or illustrate its hazardous characteristic to interpret the outcome of the test, the amount of material used should be small enough to minimize the threat to the user. It is critical that the user be properly trained in hazardous materials safety and protects him or herself from any potential safety hazards that may be associated with the unknown material. It is strongly recommended that before starting to do ***First Step*** testing, that a hazard evaluation be conducted to determine what level of personal protective equipment is required. At a minimum, safety glasses, gloves, and a splash apron should be worn. Under some circumstances, full protective clothing, including supplied air for respiratory protection should be worn by anyone sampling, handling samples, or performing any of the following tests on unknown samples.

The tests should be performed in an area protected from weather, including wind, rain, and direct sunlight. Direct sunlight and wind can interfere with the user's ability to perform tests that require an interpretation of color and flammability. A black or shaded background is most useful for observing color during many of the tests.

If the materials to be characterized have multiple phases, care must be taken to separate each phase or matrix independently and then identify the major hazards associated with each phase. Direct-reading survey instrumentation (combustible gas indicator, photoionization detector, flame ionization detector, or radiation survey instruments), if available, are also valuable tools to aid in characterizing unknown materials as well as monitoring for health and safety purposes.

Many of the tests rely on paper test strips. While ideal for liquids, these can sometimes pose a problem for solids. If water soluble, the material left over from the Solubility Test may be used for paper testing. If non soluble, the user may need to smear the solids into the paper and allow more time to react to obtain the results.

First Step Procedures

1. Hairpin Test (Safety Step)

(Is the unknown explosive/reactive?)

Warning:

This must be performed on small amounts of the unknown and never directly inside any container.

When confronted with solids, particularly gray or dark solids, it is important to first confirm that they are not explosive or so reactive that testing them will present a safety hazard to the tester. This test is to identify potential explosives, organic peroxides, hydrides, or any other reactive material. The Hairpin Test must be performed on any unknown solid before continuing to the other tests. If the hairpin test is positive, testing on the unknown should **STOP** and no further chemical tests should be conducted. The unknown should be managed as a potential explosive/reactive, until confirmed or ruled out by a bomb technician or other qualified individual.

Dispense a small amount of the unknown on a watch glass. NEVER HOLD THE WATCH GLASS IN YOUR HAND FOR THIS OR ANY OTHER TEST. Make sure that there are not other ignitable materials near the watch glass. Hold the open ends of the hairpin with the metal tweezers and place the rounded end into the propane torch flame. The hairpin must be red-hot when it is touched to the unknown on the watch glass.

The plastic coating on a new hairpin will need to be burned off before completing the hairpin test on the unknown material

If there is a reaction or bright flame when the red-hot hairpin touches the unknown the test is positive and over. The material should be segregated and managed based upon the observed reaction.

If there is no reaction upon touching the hairpin to the unknown, move the hairpin around in the unknown in an attempt to cause a reaction. If there is no reaction, proceed to the next test.

2. Water Detection

(Is it water or water based?)

FALSE POSITIVE

A test indicates the presence of something that is actually not present.

An important part of characterizing any unknown material, particularly if it is liquid, is determining whether or not water is present. For this test, any commercially available water detection test strip can be used (such as Watesmo® Paper, Gallard-Schlessinger, Inc.). This test is not necessary on dry, solid samples. Keep in mind that something seemingly as obvious as the aqueous nature of an unknown may not be obvious to a user wearing appropriate respiratory and dermal protection.

Apply a drop of the liquid unknown directly to the test paper. A color change from white to blue indicates the presence of water.

FALSE NEGATIVE

A test indicates something is not present when it actually is.

Test Limitations: Methanol will give a positive result for this test. The test strips are detecting water that is hydrogen-bonded within the alcohol. Wet alcohols and solvents such as isopropanol or acetone can give a slight or delayed positive result as they are commonly diluted with water, and can also pull moisture out of the atmosphere, activating the paper. Some concentrated aqueous or deliquescent solutions, such as concentrated sodium hydroxide, do not liberate water easily to the test paper and may give a faint purple color or turn the paper red. Strong oxidizing solutions, such as concentrated nitric acid or sodium hypochlorite solutions, may oxidize the indicator dye in the test paper and give a false negative result or destroy the indicator pigment.

3. Water Solubility and Reactivity
(How does it react with water?)

Add a small amount of the unknown to a test tube containing ¼ inch of water. Note the generation of heat, bubbles, vapor, or precipitate that would indicate that the sample is water reactive. Observe whether the sample is soluble in water. If the sample does not immediately mix in water, try mixing it by shaking the test tube. Some materials, particularly solid dry inorganics (such as salts) may take some time to dissolve. Heating the sample may accelerate solubility. As you add the unknown, observe closely for density gradients, which will appear as clear, swirling lines in the water. Density gradients may indicate that a solid material is soluble, or that a liquid sample is something other than pure water. Look for one of the following results:

A material that reacts violently with water is considered a RCRA reactive waste and carries the hazardous waste number D003.

Soluble (ionic/polar): The unknown completely dissolves in water. A soluble liquid unknown could be an acid, base, alcohol (or other polar solvent), an aqueous solution (inorgainic salt dissolved in water), or even pure water. Watch carefully for density gradients as you add the unknown. Solid unknowns that are soluble are usually inorganic salts or polar organics. The terms **miscible** and **immiscible** may be encountered when considering the solubility of one liquid in another. **Miscible** means soluble without limits; for example, alcohol is miscible with water. Immiscible and insoluble essentially mean the same thing but refer to liquid in liquid and solid into liquid respectively.

SPECIAL INSTRUCTIONS: For unknown solids that are soluble, it is recommended to save the water solubility test. It can be used for some of the upcoming tests.

Immiscible and Floats (nonpolar): Insoluble nonpolar liquids with specific gravities less than 1.0 will float. These materials are usually hydrocarbons and oils. These materials may also be flammable.

Immiscible and Sinks (nonpolar): Insoluble nonpolar liquids with specific gravities greater than 1.0 will sink. These materials are usually chlorinated hydrocarbons (or mixtures of chlorinated hydrocarbons) such as dry-cleaning solvents (trichloroethylene, tetrachloroethylene) and PCBs.

Insoluble Solids: If a solid material does not dissolve in water, the specific gravity is usually not important. Heating the sample gently may aid solubility. Watch carefully for density gradients indicating that the material may be dissolving.

Reactive: If the unknown effervesces (generates bubbles), generates heat, or produces another product (a precipitate) or gas, the unknown is reactive. In the case of a reactive unknown, it may be useful to characterize the reaction product, such as the gas evolving from solution, for pH and flammability.

Test Limitations: Some materials, when mixed with water, may form a cloudy suspension, and it may be difficult to determine if the unknown is soluble. Try adding more water or less material when this issue is encountered.

PH IS NOT A LINEAR SCALE.

An acid with a pH of 1 is ten times more concentrated than a pH of 2, and 100 times more concentrated than a pH of 3.

4. pH Test

To test for pH, apply a small amount of a liquid unknown to a strip of commercially available pH paper and compare the color of the indicators to the standard chart. Note that pH measured by this procedure is only meaningful when measured as an aqueous solution. If the material is a solid or a liquid that did not test positive for the presence of water, dissolve the unknown material in water or mix with water by shaking in a test tube and test the resulting aqueous phase (use the water from the Water Solubility and Reactivity test). Vapors and gases can be tested by first wetting the pH paper, then holding it above the unknown (note the pH of the wet paper prior to exposing it to the unknown and look for a change).

A pH of less than 2 or greater than 12 is a DOT Class 8.

Test Limitation: Very strong oxidizing acids may destroy the indicator in the pH paper. If uncharacteristic color fading is noticed in the paper, try diluting the sample in half by adding an equal volume of DI water and re-testing. If the sample itself is colored or dark, interpretation of the final color of the test paper will have to be made with the original color of the sample in mind. You may also try dipping only the long edge of the pH strip in the solution using a capillary effect so the liquid bleeds across the paper. Similarly, if using non plastic pH strips, touch only the back side of the strip to the liquids and observe the opposite side.

A waste with a pH of less than 2 or greater than 12.5 is a RCRA corrosive waste and carries the hazardous waste number of D002.

5. Oxidizer Test

An oxidizer is considered a RCRA ignitable waste and carries the hazardous waste

Wet a piece of potassium iodide starch paper with 3 N hydrochloric acid (HCl) and apply a drop of the sample to the test paper. Solid unknown samples can be smeared directly onto the acid-wetted paper. A color change from white to black, brown, purple or blue indicates a positive result. Weak or dilute oxidizers may require one or two minutes for the color change to occur. Common oxidizers are halogens/halogenated compounds, nitrites/nitrates and peroxides. Any reaction during this test such as bubbling or color change of the unknown is probably a reaction with the HCl. This reaction should be confirmed by testing the unknown with HCl in a separate test tube.

Beware of any gas given off when reacting an unknown with an acid! If the unknown material contains cyanide, hydrogen cyanide gas will be produced. This is one of the reasons the user should be using a self-contained breathing apparatus for respiratory protection.

A positive for the Oxidizer test is a DOT Class 5.1 and Peroxide test is DOT Class 5.2.

Test Limitation: Colored samples make this test difficult to interpret. Very strong oxidizers, such as concentrated nitric acid, may destroy the test paper and give a false negative result, or the test paper may only change color where the outer fringes of the sample contacted the paper. If this is suspected, try diluting the sample in water, then testing the sample again.

Peroxide Test: For samples that test positive as an oxidizer, conduct the peroxide test. Apply a drop of the liquid or smear the solid onto the color indicator portion of the strip and compare to the standard chart.

6. Sulfide Test

Wet a piece of lead acetate paper with distilled water. Apply a drop of the unknown material to the wet test paper. REMEMBER: The water solubility test #3 above can be used for this test if it has not been contaminated. A color change of the test paper from white to black, or brown indicates a positive result.

Test Limitation: Weak solutions may require several minutes for a color change to occur. Colored samples make this test difficult to interpret.

7. Cyanide Test

There are several potential fast screening methods for cyanide. All the procedures have pros and cons; however, a reliable and perhaps the quickest procedures are presented here. Materials that tested below a pH of 12 will not contain a cyanide and need not be tested.

This test procedure may generate hydrogen cyanide gas! The user is warned to perform this test either in a laboratory hood, a well-ventilated area, or while on supplied air, and to ensure they are a safe distance from bystanders.

A cyanide or sulfide bearing waste is considered a RCRA reactive waste and carries the hazardous waste number D003.

Test Option #1: Cyanide Paper Method

Prepare a length of Cyantesmo® paper that will be inserted into a test tube. Bend the top end over to act as a stop so that the paper does not fall into the test tube and touch the unknown liquid. Make the test paper long enough so that it extends down into the test tube but will not touch the liquid. (about ¾ inch shorter than the test tube). You may also have to fold the paper lengthwise so that it will fit into the test tube.

In a test tube containing a small amount of the unknown in solution, (about ¼ inch), add two drops of concentrated sulfuric acid. The sulfuric acid must mix with the unknown. **Immediately** insert a length of cyanide test paper (such as Cyantesmo® paper, Gallard Schlessinger, Inc.) into the test tube so that there is at least an inch or more of dry paper above the liquid level in the test tube. The indication of a positive result will be in the dry portion of the test paper. If cyanide is present at a concentration above 50 parts per million (ppm), the paper will **turn blue** in the zone just above the liquid level, where it is reacting with the hydrogen cyanide gas. If no immediate change is observed, wait 15 minutes and then see if a change is visible.

A positive for the Cyanide Test is a DOT Class 6.

Test Limitation: The paper is sensitive to cyanide concentrations as low as 5 ppm; however, it may take up to 4 hours for the blue indicating color to appear at this concentration.

Note: This test may yield false negative results if the **unknown** is not wetted with distilled water. If the unknown is not in solution, the acid may not generate HCN

During training, it is recommended that the instructor make up this mixture and placed it at the cyanide test table for use by all the participants.

Test Option #2: Kotney Test Method

Step 1: Mix cyanide test #1 (ferrous ammonium citrate) with cyanide test #2 (ferrous ammonium sulfate) which will appear forest green or brown. **To make a solution of cyanide test #1, weigh out 10 grams of ferrous ammonium citrate and mix it with 50 ml of water. This is a saturated solution that can be stored for**

multiple days. When you are ready to do the Kotney Cyanide Test, place ½ inch of cyanide test #1 in a test tube and add a pea size amount of cyanide test #2, (ferrous ammonium sulfate) to test #1 and gently mix it. This amount of mixture should do about 2-3 Kotney Cyanide tests. If you will be testing a larger number of unknowns you may wish to mix enough for several tests, saving the mixture in a test tube for future tests.
Note 1: If you do not have some way to measure 10 grams, this is about 3 packets of sugar.

Step 2: Unknown Liquid: Add about 1/2 –inch of the unknown to a test tube.
Unknown Solid: Add ½ a pea-size amount of unknown to ½-inch of the water.
Step 3: Add 1/4 inch of the solution of cyanide test #1 & #2 to the test tube containing the unknown from step #1.
Step 4: Mix the solution well by taping the bottom of the test tube with your finger.
Step 5: Slowly add HCl Acid Test (3N) several drops at a time until a reaction occurs. You may wish to hold the test tube at an angle as the acid test comes in contact with the unknown solution so you may observe any reaction. You should start to see a reaction after 4-7 drops of HCl.

Observations:

a. **Dark blue precipitate**: Cyanide indicated. The blue color appears only when the solution is acidic and becomes deeper as more acid is added.

b. **Light blue precipitate**: Metal cyanides in which the cyanide anion is tightly bound are indicated.

c. **Blue-green or green liquid**: Very low level of cyanide indicated.

8. Flammability Test

This test is somewhat subjective and is a rough test for flammability and combustibility; however, when performed properly, it can be used to approximate the flash point of an unknown material.

A waste that has a flash point less than 140°F is a RCRA ignitable waste and carries the hazardous waste number of D001.

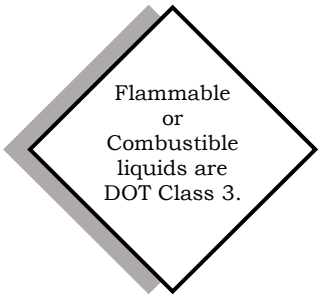
Place a nickel to quarter size pool of unknown in the center of the watch glass resting on the table. NEVER HOLD THE WATCH GLASS IN YOUR HAND FOR THIS OR ANY OTHER TESTS. At the level of the edge of the watch glass, slowly bring a lit match from eight inches away up to the pool of unknown in the watch glass. Continue to move the lit match nearer to the pool of unknown until the pool ignites or the match is extinguished in the pool of unknown.

Note the location of the match relative to the pool of unknown when the material ignites. The farther away the match is from the pool of unknown and when it ignites, the lower the flash point of the unknown. Some

combustible materials may require heating or even using the match as a wick. With such combustible materials it will require that you place the burned end of the match in the unknown and then turn the match up to use it as a wick and heat up the material until it ignites. This would be an indication that the unknown has a flash point in the combustible range.

Evaluation:

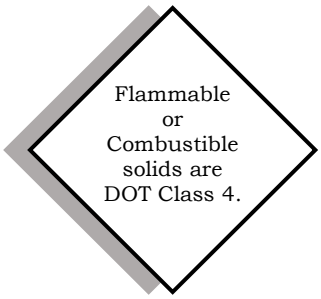
Extremely Flammable: The pool of liquid unknown ignites and continues to burn, before the match reaches the edge of the watch glass. For field characterization purposes, it should be considered extremely flammable. It will appear as if the flame jumps out to the lit match. The unknown is likely to have a flash point below 100°F.



Flammable: The pool of liquid unknown ignites and continues to burn, when the match reaches the edge of the watch glass or comes close to the pool, but does not touch the pool. For field characterization purposes, it should be considered flammable. The unknown is likely to have a flash point below 140°F.

Combustible: The pool of liquid unknown is difficult to ignite and needs to be heated with the match to continue burning. For field characterization purposes, it should be considered combustible. If the match must be used as a wick to burn the material on the match itself, it is definitely a combustible. The unknown is likely to have a flash point between 140 and 200°F. If the observations are borderline between flammable or combustible, classify as flammable.

Non-Flammable: If the pool of liquid unknown cannot be ignited and extinguishes the match upon contact, then the unknown, for field characterization purposes, should be considered nonflammable and is likely to have a flashpoint >200°F.



Additional information can be obtained by observing the burn characteristics of the flame. For example, alcohols will burn with an invisible or near invisible blue flame. A bright yellow flame will indicate the presence of an increasing number of carbon atoms in the unknown compound. Smoke in the flame is often an indication of heavy molecular weight compounds, such as oils, or compounds with oxygen atoms, such as ketones or aldehydes. Wispy, stringy. or spiderwebbing smoke may be an indication of aromatic compounds such as benzene, toluene, or xylenes.

Test Limitations: Flash points are estimations only and may be very inaccurate depending upon the individual performing the test and the environmental conditions. Alcohol flames are very difficult to see and may be missed unless the test is performed in a sheltered area with a black background to view against. The presence of sodium ions in a sample creates such a vivid yellow flame color that many users mistake it for combustion.

9. Beilstein Test

COMMON FLAME COLORS
Green - Cl, Cu, Br
Red - Li, Ca
Yellow/Orange - Na
Purple/Violet - K
Blue - Pb, Zn, As

PCBs in oil will cause a green flame color at about 500 ppm.

A green or blue flame indicates a DOT Class 6.

This test may be performed simultaneously with the flammability test. Use a thin looped copper wire and place a drop/small amount of material in the loop. Hold the loop up to the propane burner. Observe any immediate or residual color in the flame. Interpretation of this test may be difficult because the color may appear instantly or may only appear after prolonged heating of the copper wire in the hottest part of the burner flame (the tip of the inner cone of the flame). If no color is immediately apparent, continue to hold the copper wire loop in the flame until it is glowing red. Additionally, color may only be visible in a small portion of the flame, or may be overwhelmed by the presence of another cation or anion present, such as sodium. This test may have to be performed repeatedly to confirm the presence or absence of the color in the flame. To prevent cross contamination and interfering results, the copper wire must be burned completely clean of color prior to performing this test on another sample. If contamination remains on the wire, the end should be cut off and a new loop formed in the wire.

This test is a good procedure for identifying chlorinated (and halogenated) hydrocarbons. Chlorinated compounds such as dry-cleaning solvent and PCB oils give a brilliant green flame. The test is also useful for identifying anions or cations of inorganic salts.

Test Limitations: The presence of interfering ions, particularly sodium, will completely mask any competing color. Contaminated wires may interfere with test results; wire must be thoroughly cleaned, or the contaminated portion of the wire must be snipped off. If the sample is corrosive, copper ions may be liberated, resulting in a false positive green flame color. In some oils, PCBs may be present at a concentration that is high enough to be a health and environmental threat, but not high enough to give a visible green flame.

10. Iodine Saturation Test

This test estimates the degree of hydrogen saturation of an unknown and is only performed on liquid sample with solvent characteristics. Add a very small crystal (approximately the size of a pin head) of iodine to a test tube and then add about ¼ inch of the unknown liquid sample. Observe the color of solution after the iodine crystal dissolves. Alternatively, place a nickel sized portion of the liquid on a watch glass. Add a small crystal of iodine to the liquid and drag the crystal through the material. Observe the color of the streaks. Use the following list to help interpret the results. This is not a definitive list of compounds but will aid in establishing the compound class or family.

Color	Possible Compounds or Chemical Category
Red	Alkenes (double-bond compounds) Aromatics (benzene, toluene, styrene, xylene) Chlorinated compounds [trichloroethene (TCE), tetrachloroethene (PCE), chlorobenzene] Oils (turpentine, PCBs in oil)
Purple	Alkanes, hydrocarbons (saturated compounds) Thinners (kerosene, stoddard solvent, hexane) Chlorinated compounds (carbon tetrachloride, trichloroethane, methylene chloride)
Yellow/Orange	Oxygenated and polar compounds Alcohols (methanol, ethanol, isopropanol) Ketone [acetone, methyl ethyl ketone (MEK)] Acetates (ethyl acetate)
Brown/Muddy	Mixtures of two or more types of compounds Gasoline

Test Limitations: If too large of an iodine crystal is used, any compound or mixture test will turn deep red. Care and practice must be exercised to ensure a small enough crystal is used to obtain a valid result. With some samples, when water is added to the unknown mixtures (brown result), the mixture may clarify to a different result. This additional step can be useful in determining the components of a mixture.

11. Char Test

The char test is useful for determining whether an unknown material is an organic or inorganic compound and provides additional physical and chemical characterization of the unknown material. Begin by placing about ¼ inch of sample (solid or liquid) into a test tube. Slowly begin to heat the sample. Initially place the flame between the unknown and the test tube holder that is at the top of the test tube. If you heat the bottom of the test tube too quickly, it may cause the material to be propelled out of the tube posing a safety hazard. Make sure to never point the test tube towards another person and to heat the test tube evenly around the tube, otherwise it is likely to fracture.

If fumes or vapors are generated, note the pH of the fumes by placing a wetted pH paper in the fumes.

Again, slowly heat the sample. If fumes or vapors are generated, test the fumes for oxidizer by placing HCl wetted KI paper in the fumes and note the results.

After testing the fumes with pH and KI paper slowly heat the sample again, attempting to either boil a liquid sample off (evaporating the sample) or charring a solid sample. If vapors are given off, attempt to ignite them to determine whether they are flammable. When the vapors ignite, observe the flame. Flame characteristics include invisible flame or "pop" (hydrogen gas), hydrocarbon flames-clean, dirty, wispy "hanging spider webs", or other observations. Several possible results are outlined below.

Liquid Samples

Vapors that ignite - typical of organic liquids (solvents)
Vapors that do not ignite - typical of water

Charring residue - typical of organics
Non-charring or white residue - typical of dissolved inorganics

Solid Samples

Vapors that ignite - typical of organics
Vapors that do not ignite - typical of inorganics

Charring residue - typical of organics
Non-charring or white residue - typical of inorganics

Subliming Solids

Can be organic or inorganic (examples include naphthalene, phenol, sulfur, and some ammonium salts).

Vapors

Vapors with a pH of 9-11, and which are oxidizer positive are characteristic of nitrates, and are explosive when mixed with fuel products. These nitrates may yield negative oxidizer test, and neutral pH until heated and tested via the char pH and char oxidizer techniques.

Test Limitations: The test is difficult to perform on aqueous samples with dissolved salts. It takes considerable patience and practice to evaporate off water from a test tube without ejecting the entire sample. If the liquid in the test tube is heated too fast, it will boil out of the test tube. During initial training, the student should practice with water to achieve a heating technique that does not result in the sample boiling or exploding out of the test tube.

The char test can provide a great deal of information if conducted properly, and observations noted. Performance of the char test requires a little experience and practice to do it properly.

Test Equipment

Full ***First Step*** Kit (100 Samples):

The equipment necessary to perform the ***First Step*** Procedures can be assembled from commercially available supplies for approximately $250. Some material may be available at a drug store or grocery store. Reagent such as 3N hydrochloric acid can be ordered from a chemical supplier, or may be available for a nominal cost from a local school or university. The following list of materials should supply enough equipment for approximately 100 unknown samples.

Hardware

Propane/butane burner
Long-nose pliers
Copper wire (bare and clean, 10 gauge)
Iodine crystals (5 grams)
400 transfer pipettes (polyethylene)
2- Watch glasses (75 mm)
Test tube holder and rack
Spatulas
FisherBrand 500 Disposable Culture Tubes (12 x 75 mm) (About 5 tubes per sample)

Reagents

Distilled water
3 N Hydrochloric acid
Sulfuric acid, concentrated

Commercial Test Papers

1 box Water indicating paper
2 boxes pH paper
1 box Potassium iodide/starch paper
2 boxes Lead acetate paper
1 box Cyanide test paper
1 box Quantofix Peroxide Semi-quantitative test strips

Abbreviated ***First Step*** Kit (<10 Samples):

Watch Glasses x2
pH Paper
Watesmo Paper
Cyantesmo Paper
Potassium Iodide Starch Paper
Copper Wire
Pen
Propane Torch and Valve
3N HCl
Gloves
Iodine Crystals
FisherBrand Disposable Culture Tubes
Lead Acetate Paper
Metal Spatulas
Metal Scoop
Box of Matches
Tweezers
Hair pin
Test Tube Holder
D.I. Water
Concentrated Sulfuric Acid
Safety Goggles

Documentation

Test results and observations should be recorded on a form similar to the one attached.