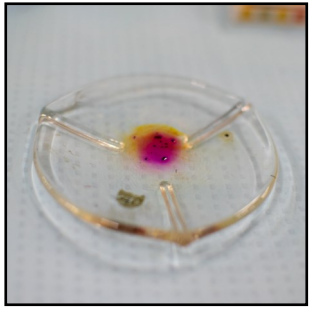
First Step Hazard Categorization

Safety Precautions

Conducting any hazard categorization of an unknown inherently poses a number of risks. It is your responsibility to minimize those hazards as much as possible while conducting a hazard categorization.



- Always assume the unknown you are working with is hazardous and treat it as such.
- Conduct tests in an area protected from weather including wind, rain, and direct sunlight.
- Use air monitoring equipment during testing including a PID and gamma radiation detector. Use additional monitoring equipment if appropriate and available.
- Wear safety glasses and gloves at all times. Consider additional PPE including Tyvek and APRs if conditions warrant. An eyewash station should be close by and easily accessible.
- Inspect all glassware for damage before use. Damaged glassware should not be used as it may break or explode during a test.
- Never hold a watch glass in your hand while conducting a test, and always use test tube holders when heating a test tube.
- Always point test tubes away from yourself and other people while conducting each test. Heated test tubes may create a projectile and launch the unknown if not properly heated.
- Clean up between each test to avoid unexpected reactions between unknowns and to maintain an organized work space.

Department of Transportation Hazard Classifications

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 or water reactive emitting flammable gas, indicated by the flammability, solubility 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 8 can be further described as either Acidic or Basic for classification.

Class 9: Miscellaneous or Environmentally Hazardous Materials, unknowns with no other hazard identified by ***First Step*** are classified as Class 9.

Test 1 — Hairpin Test (Safety Step)

Materials: Large hairpin, tweezers, watch glass, propane torch.

Tests for solid explosives, organic peroxides, hydrides and other reactive compounds.

Place a small amount of solid material on the watch glass.

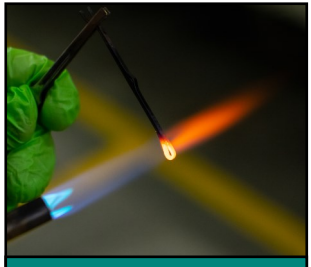
Hold the open ends of the hairpin with the metal tweezers and place the rounded end into the propane torch flame and heat to be red-hot.

Touch the hairpin to the material and look for a 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**, the test is **negative** and you can proceed to the next test.

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 A FLAME OR REACTION IS OBSERVED,
DO NOT PROCEED WITH ANY OTHER TESTS!



Heating the hair pin to red hot.

The reaction of a positive hairpin test.

Test 1 — Hairpin Test Results Definitions

Observation	Interpretation of Observation:	Result to Record
Sparking, flames, or explosion observed	Positive—DO NOT CONTINUE TESTING	+
No reaction	Negative	–

Solid Description Definitions

Solid Type:	Definition
Powder	Dry, bulk solid composed of many very fine particles that may flow freely when shaken or tilted.
Granules	A medium-sized particle with an irregular shape.
Chunks	Irregularly shaped pieces of a solid, pencil eraser size or larger
Crystals	A solid with repeating geometric pattern. Crystals can vary in color and size.
Flakes	A thin flat solid that may be visibly fragmentable.
Pellets	A compressed solid product, usually with small rounded or spherical shape.
Pills	A compressed powder in a uniform shape, generally manufactured for consumption.

Test 2 — Water Detection Test

Materials: Transfer pipet, tweezers, watch glass, Watesmo® test paper.

This test is not necessary for dry solid samples.

Apply a drop of the liquid unknown directly to the test paper on the watch glass.

Look for a color change from white to blue. A **blue** color change indicates the presence of water or a water based solution.

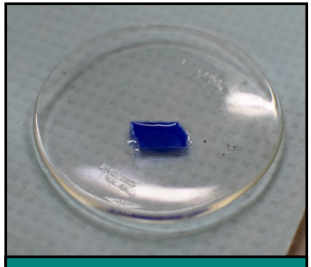
Test Limitations:

Methanol can give a **positive result**. The test strips are detecting water that is hydrogen-bonded within the alcohol.

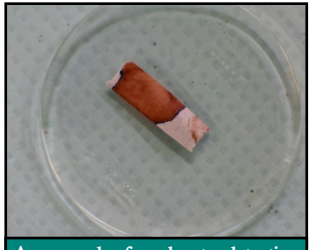
Wet alcohols and solvents such as isopropanol or acetone can give a slight or delayed **positive result** as they are commonly diluted with water, and can also pull moisture out of the atmosphere, activating the paper.

Concentrated aqueous or deliquescent solutions, such as sodium hydroxide, do not liberate water easily to the test paper resulting in a faint **purple** or **red** color depending on the concentration.

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.



A positive water detection test.



An example of a red water detection test due to a deliquescent solution.

Test 2 — Water Detection Test Results Definitions

Observation	Interpretation of Observation:	Result to Record
Paper turns blue	Positive for water.	+
No blue color observed	Negative for water.	–
Sample is a Solid	Water Test not applicable.	N/A
Paper turns red or purple	May be concentrated aqueous or deliquescent solutions, such as concentrated sodium hydroxide.	Record Observation
Sample is either opaque or an oxidizer that makes a color determination difficult	Opaque samples may obscure reaction and oxidizers will destroy the indicator pigment.	Record Observation

Test 3 — Water Solubility and Reactivity

Materials: Test tube, transfer pipet/scoopula, DI Water.

Add a small amount of the unknown to a test tube containing 1/4 inch of water.

Observe the test for **heat**, **bubbles**, **vapors** or **precipitate** indicating water **reactivity**.

If the sample does not immediately mix in water, try agitating the test tube. Again if nothing happens you may try heating the test tube.

Observe for solubility and density gradients.

If the unknown **completely dissolves** in the water, it is **soluble/miscible** and **ionic** or **polar** indicating it may be an acid, base, alcohol, aqueous solution, inorganic salt, or pure water.

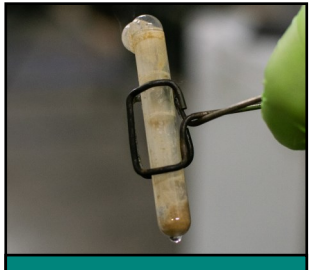
If the unknown is **insoluble/immiscible** and **floats**, its **non-polar** with a specific gravity <1 and indicative of hydrocarbons and oils.

If the unknown is **insoluble/immiscible** and **sinks**, its **non-polar** with a specific gravity >1 and indicative of chlorinated hydrocarbons or PCBs.

If a solid **does not dissolve** and no density gradients are observed, its **insoluble**.

If the unknown **reacts**, test the gas evolving from the solution for **pH** by holding a pH test strip above the tube. Test for **flammability** by using a match to attempt to ignite the evolved gas.

An immiscible floating liquid.



An example of a water reactive solid.

Test 3 — Water Solubility and Liquid Terminology Definitions

Solubility Test Observation	Interpretation of Observation:	Result to Record
Unknown solid that completely dissolves in water.	May be polar or inorganic	Soluble
Unknown liquid will completely mix homogeneously with water.	May be polar or inorganic	Miscible
Unknown liquid does not dissolve in water and floats, Specific gravity is < 1.	May be nonpolar or organic, volatile possibility	Immiscible and floats
Unknown liquid does not dissolve in water and sinks, Specific gravity is > 1.	May be nonpolar, organic chlorinated possibility	Immiscible and sinks
Unknown solid does not dissolve and floats in water, density is < 1.	May be nonpolar or organic	Insoluble and floats
Unknown solid does not dissolve and sinks in water, density is > 1.	May be nonpolar or organic	Insoluble and sinks
Unknown solid does not dissolve and stays suspended in water, density is close to or equal to 1.	May be nonpolar or organic	Insoluble solids
Unknown solid or liquid that may bubble, produce a gas, or feel unusually hot or cold when introduced to water.	Potential flammable gas could be generated	Reactive
Viscosity Test Observation	**Interpretation of Observation:**	**Result to Record**
Less surface tension than water, little-to-no meniscus. (Example: Acetone)	May be a solvent and/or volatile	Less Than Water
Behaves like water with a similar viscosity.	May be an aqueous solution	Water-like
Light, free-flowing oil. (Example: Mineral Oil)	May be indicative of organics and hydrocarbons	Light Oil
Oil that moves slower than water, stick to the sides of glass briefly. (Example: Motor Oil)	May be indicative of organics and hydrocarbons	Medium Oil
Thick, slow-moving oil. May stick to the sides of glass for considerable time. (Example: Molasses)	May be indicative of organics and hydrocarbons	Heavy Oil
Solid gel or jelly-like substance. May appear liquid but can hold a physical shape without a container.	Could be organic or inorganic, perhaps a consumer product.	Gel
Thicker than gel substance, usually a waste and a mixture of solids and liquids.	Indicative of wastes. Could be organic or inorganic	Sludge
Turbidity Test Observation	**Interpretation of Observation:**	**Result to Record**
Completely transparent, details can be seen through the material if looked through.	May be a pure chemical or mixture	Clear
Partially Transparent, allows some light to pass through, but details cannot be seen if looked through.	May be pure, color may indicate type	Translucent
Not transparent, little to no light can pass through.	Longer chain solutes or contents that block light.	Opaque

Test 4 — pH Test

Materials: pH paper, transfer pipet, tweezers, watch glass, test tube and DI water for soluble solids.

This test is not necessary for insoluble dry solids or immiscible liquids.

For liquids, apply a drop of the liquid unknown directly to the pH paper on the watch glass.

For soluble solids, apply a drop of the solid dissolved in water (use mixture from Test 3—Water Solubility) directly to the pH paper on the watch glass.

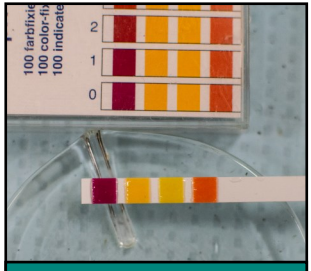
Observe the color change and compare to the color indicator on the standard chart. A pH of greater than 12 or less than 2 indicates the unknown is corrosive.

Vapors and gasses can be tested by pre-wetting the pH paper and exposing it to the unknown gas.

Test Limitations:

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, it may be difficult to discern a color change. Retest by diluting the sample in half by adding an equal volume of DI or by dipping the long edge of the pH strip in the solution so the liquid bleeds across the paper.



A acidic pH result (<2 pH).

A basic pH result (>12 pH).

Test 4 — pH Test Results Definitions

Observation	Interpretation of Observation:	Result to Record
0-2	Strongly Acidic (Corrosive)	Record exact pH value as determined by the pH test strips
2-4	Acidic	
4-6	Weakly Acidic	
7	Neutral	
8-10	Weakly Basic	
10-12	Basic	
12-14	Strongly Basic (Caustic)	

Test 5 — Oxidizer Test

Materials: Transfer pipet/scoopula, watch glass, potassium iodide starch paper, 3N HCl.

Wet the potassium iodide starch paper with the 3 N HCl.

If the unknown is **liquid**, apply a drop of the unknown to the starch paper.

If the unknown is **solid**, smear the unknown directly into the starch paper.

Observe the paper for a color change from white to **black**, **purple** or **blue** which indicates a **positive** result.

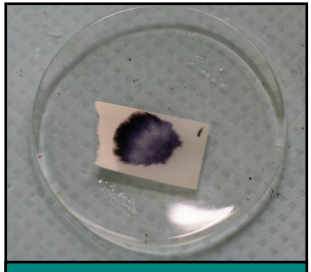
If the test is **positive**, the unknown is likely an oxidizer and may be **halogenated**, a **nitrite** or **nitrate,** or a **peroxide**.

Weak oxidizers may require a few minutes for a color change to occur.

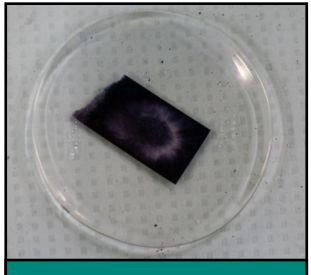
Strong oxidizers, like nitric acid, may destroy the test paper giving a **false negative** result, or the test paper may only change color where the outer fringes of the sample contacted the paper. Try diluting the sample and retesting.

Any **bubbling** or **color change** of the unknown is likely a reaction with the HCl and should be tested in a separate test tube.

Peroxide Test: For samples that test positive as an oxidizer, a peroxide test can be performed if test strips are available. Apply a drop of the liquid or smear the solid onto the color indicator portion of a Quantofix® Peroxide test strip and compare to the standard chart.



A positive oxidizer test.



An example of a strong oxidizer.

Test 5 — Oxidizer Test Results Definitions

Observation	Interpretation of Observation:	Result to Record
Will not react with Oxidizer test paper.	Substance is not an oxidizer	No
Will react with Oxidizer test paper causing color change to black, purple, or blue gradually (can develop after a few minutes).	Not a strong oxidizer	Weak
Will react with Oxidizer test paper causing color change to black, purple, or blue immediately. If strong enough, a white circle may appear on test strip.	Unknown is an oxidizer, may need to test for peroxides	Strong
Compound is darkly colored/opaque or has other properties making a clear determination difficult to discern.	Not a clear enough result to make a determination	Inconclusive

Test 6 — Sulfide Test

Materials: Transfer pipet/spatula, watch glass, lead acetate paper, DI water.

Wet a piece of lead acetate paper with DI water.

If the unknown is liquid, apply a drop of the liquid unknown directly to the test paper on the watch glass.

If the unknown is solid, apply a drop from the solubility test directly to the test paper on the watch glass.

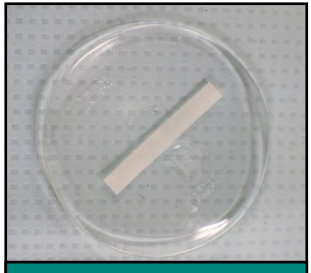
If a color change from white to **black** or **brown** is observed, the test is **positive**. The color change may require several minutes to develop.

If no change is observed, the test is **negative.**

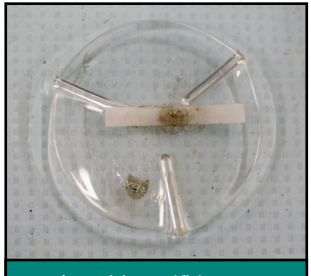
Test Limitations:

Weak solutions will require additional time for color change to occur.

Colored samples may make this test difficult to interpret. Sometimes the color change is easier to observe from the back side of the paper.



A negative sulfide test.



A positive sulfide test.

Test 6 — Sulfide Test Results Definitions

Observation	Interpretation of Observation:	Result to Record
A color change from white to **black** or **brown** is observed.	Positive for sulfide	+
No color change is observed.	Negative for sulfide	–

Test 7 — Cyanide Test, Option # 1

Materials: Test tube, Cyantesmo® test paper, concentrated sulfuric acid, DI water.

If the unknown is solid, mix a small amount into about 1/4 inch of DI water in a test tube.

If the unknown is liquid, add about a 1/4 inch of unknown to the test tube.

Cut off a length of Cyantesmo® test paper and fold the end of the paper so it can hang on the edge of the test tube. The bottom of the paper should be just above the top of the liquid.

Add 2-3 drops of the sulfuric acid to the test tube

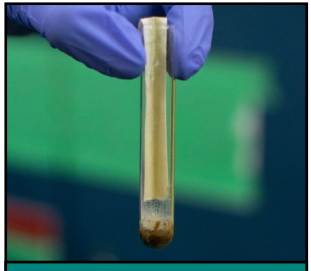
If the end of the paper turns blue, the test is **positive** and the cyanide gas released is at a concentration above 50 ppm in the test tube.

If no immediate change is observed, wait 15 minutes to see if a change develops.

Test Limitations:

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.

A **false negative** may occur if the unknown is not wetted before adding the acid.



A negative cyanide test.

A positive cyanide test.

Test 7 — CN Test #1 Results Definitions

Observation	Interpretation of Observation:	Result to Record
End of paper turns blue above surface of unknown	Positive for cyanide	+
No color change is observed	Negative for cyanide	–
Cyanide test does not need to be performed if pH Test Result is less than 10	No concern about HCN generation	N/A

Test 7 — Cyanide Test, Option # 2

Materials: Transfer pipet, test tube, DI water, 3N HCl, ferrous ammonium citrate (RE2332), ferrous ammonium sulfate (RE2333) (These can both be found in the HazCat 2.0 Kits)

Add 1/2 inch of cyanide test solution #1 to a clean test tube (test tube #1).

Add a pea size amount of cyanide test solution #2 to test tube #1 and gently mix.

For **liquid** unknowns, add 1/2 inch of the unknown to a clean test tube (test tube #2).

For **solid** unknowns, add a 1/2 peas sized amount to 1/2 inch of DI water in a clean test tube (test tube #2).

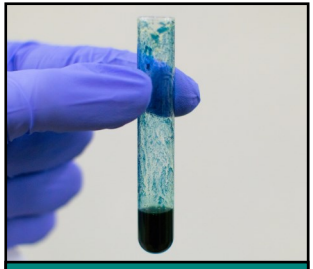
Add 1/4 inch of solution from test tube #1 to test tube #2. Mix the solution by tapping the test tube with your finger.

Add 4-7 drops of 3N HCl slowly to test tube #2 until a reaction occurs.

If a **dark blue precipitate** is observed, cyanide is present. The color will deepen as more acid is added.

If a **light blue precipitate** is observed, tightly bound metal cyanide anions are present.

If **blue-green** or **green** liquid is observed, very low concentrations of cyanide are present.



A positive cyanide test.

A low concentration cyanide test.

Test 7 — CN Test #2 Results Definitions

Observation	Interpretation of Observation:	Result to Record
A blue precipitate or blue-green or green liquid is observed	Positive for cyanide	+
No color change is observed	Negative for cyanide	–
Cyanide test does not need to be performed if pH Test Result is less than 10	No concern about HCN generation	N/A

Test 8 — Flammability Test

Materials: Transfer pipet, watch glass, matches.

Add a nickel to quarter sized pool of unknown to the center of a watch glass.

At the level edge of the watch glass, slowing bring a lit match towards the pool of the unknown starting from approximately 8 inches away.

Continue to move the match until the pool ignites or the match is extinguished.

Observe the location of the match when the pool ignites and the burn characteristics.

If the pool ignites before the match reaches the edge of the watch glass, the unknown is **extremely flammable** with an estimated flash point below **100˚F**.

If the pool ignites when the match reaches the edge of the watch glass or as it approaches the pool, the unknown is **flammable** with an estimated flash point between **100 - 140˚F**.

If the pool is difficult to ignite and needs to be heated with the match to burn, or if the match needs to be used as a wick to burn, the unknown is **combustible** with an estimated flash point **> 141˚F**.

If the pool cannot be ignited or if the match is extinguished upon contact, the unknown is **non-flammable**.

Test Limitations: Flash points are estimations only and may not be accurate depending upon the individual performing the test and the environmental conditions.

An example of a extremely flammable liquid.

A combustible liquid, using the match as a wick.

Test 8 — Flammability Test Result Definitions

Observation	Interpretation of Observation:	Result to Record
Pool of sample on watch glass ignites before match reaches the edge. (<100°F)	Shorter chain hydrocarbons that volatilize	Extremely Flammable
Pool of sample ignites on watch glass as it reaches the edge or as it approaches the pool. (100-140°F)	Contains hydrocarbons	Flammable
Difficult to ignite pool and need continual heat by using the match as a wick to burn. (140-200°F)	Longer chain hydrocarbons	Combustible
Material cannot be ignited or match is extinguished upon contact with the sample. (>200°F)	No flammability concerns, may be aqueous solution / inorganic	Non-Flammable

Test 9 — Beilstein Test

Materials: Transfer pipet, tweezers, copper wire with a small loop, propane torch.

Apply a drop or small amount of solid unknown to the copper loop.

Place the loop in hottest point of the torch flame (the tip of the inner cone of the flame).

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.

If **green** is observed, the unknown may contain **chlorine**, **copper**, or **bromine**.
If **red** is observed, the unknown may contain **lithium** or **calcium**.
If **yellow** or **orange** is observed, the unknown may contain **sodium**.
If **purple** or **violet** are observed, the unknown may contain **potassium.**
If **blue** is observed, the unknown may contain **lead**, **zinc**, or **arsenic.**

Test Limitations:

The presence of interfering ions, particularly sodium, will completely mask any competing color.

If the sample is corrosive, copper ions may be liberated, resulting in a false positive green flame color.

Note: Color change may be quick and not as vibrant at lower concentrations. When possible, try performing this test in a darker environment in order for color changes to be more apparent.


Green Flame


Red Flame


Orange Flame


Purple Flame


Blue Flame


Normal Flame

Test 10 — Iodine Saturation Test

Materials: Transfer pipet, test tube, watch glass, iodine crystal, spatula.

This test is only necessary for unknowns with properties of organic liquids or solvents.

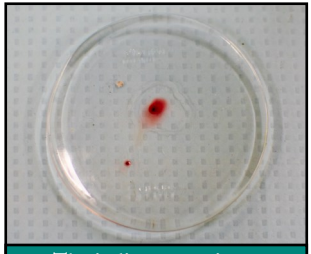
There are two options for this test: Add a very small crystal (approximately the size of a pin head) of iodine to a test tube and then add about ¼ inch of the unknown liquid sample.

OR

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.

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 mixture will turn deep red or brown.



The iodine test using a watch glass.

Four color examples of the iodine test (from left to right, xylene, hexane, methanol and gasoline)

Test 11 — Char Test

Materials: Test tube, test tube holder, propane torch, transfer pipet/spatula.

The char test is useful for determining if an unknown is an organic or inorganic compound and provides additional physical and chemical characterization of the unknown material.

Place a 1/4 inch of material in the bottom of a test tube. Hold the test tube at the top with the test tube holder.

Slowly heat the test tube with the propane torch heating the area above the material first and slowly moving down across the material to heat it evenly.

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.

Test vapors for flammability by attempting to ignite them with the torch while heating the sample. If they do not ignite, continue heating and check again to confirm. You can also check pH and oxidation properties using the indicator papers from earlier tests.

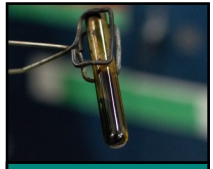
Continue heating the sample attempting to either boil a liquid sample off (evaporating the sample) or charring a solid sample.

Liquid Samples: Vapors that **ignite** indicate **organic liquids** (solvents), Vapors that **do not ignite** indicates water solutions, **Charring residue** indicates **dissolved organics**, **Non-charring** or **white residue** indicates i**norganics.**

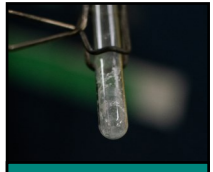
Solid Samples: Vapors that **ignite** indicate **organics**, Vapors that **do not ignite** indicate **inorganics, Charring residue** indicates **organics**, **Non-charring or white residue** indicates **inorganics.**

Subliming Solids: Can be organic or inorganic (examples: 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 a negative oxidizer test, and a neutral pH until heated and tested via the char pH and char oxidizer techniques.



An organic char test.



An inorganic char test.

A sublimating solid.

Test 11 — Char Test Results Definitions

Char Test Observation	Interpretation of Observation:	Result to Record
Blackened residue remains on glass tube.	Likely Organic	Charring Residue
White or other colored residue remains on glass tube.	Dissolved solute in solution, likely aqueous/ inorganic	Non-charring residue
Sticky, generally dark looking residue that does not "dry" completely.	Likely Organic	Tar
An unknown that goes from a solid to gas, skipping the liquid phase.	Examples: Phenol, Ammonium salts, Sulfur	Subliming Solid
No visible residue remains on glass tube.	Pure compound, or small chain organic	No residue
Vapor Test Observation	**Interpretation of Observation:**	**Result to Record**
Dark or black smoke visible.	Likely Organic	Dark Vapors
Smoke appears stringy or forms short webs.	Likely Organic	Spiderweb Vapors
Vapors will catch fire when exposed to an ignition source.	Indicative of Organics	Vapors that Ignite
Vapors will not catch fire when exposed to an ignition source.	Indicative of Inorganics	Vapors that don't Ignite