

Unknown Chemical Guidance

An unknown is defined as a chemical in an unlabeled container for which the identity is undetermined. Federal, state, and local regulations all specifically prohibit the transportation, storage, or disposal of unknown wastes. In addition, hazardous waste disposal companies will not accept unknowns without proper analysis. Therefore, unknown or unlabeled chemicals all require analysis prior to disposal, which can easily cost \$1,000 or more for a single sample. Unknown chemicals present serious legal and safety problems for the University. **EH&S will not accept any "unknown" wastes for disposal. The burden of costs incurred in the identification of unknown wastes lies with the department that generated the waste.**

Preventing Unknown Chemicals

Many unknown chemicals are generated due to a lack of good housekeeping and good laboratory safety practices. Unknown and unlabeled chemicals can be prevented by:

- Labeling all containers (including beakers and test tubes) properly. This should be done even when creating reagent solutions for temporary use. Labeling will also prevent using the wrong material accidentally.
- Inspecting containers and labels in the lab inventory periodically and replacing fading or deteriorating labels that are not legible.
- Labeling containers using full chemical names, not abbreviations, chemical structures, or formulas.
- Archived research samples are often stored in boxes containing hundreds of small vials. Label the outside of the box with the chemical constituents, paying special attention to regulated materials such as radioactives, solvents, heavy metals, and other toxic materials. If the samples are nonhazardous, label them as such.
- Maintaining an accurate chemical inventory list.
- Require all reaction mixtures stored in lab glassware to be labeled with chemical composition, the date they were formed, and the name of the lab worker responsible.
- Require all workers to properly identify any unknown materials before they leave the area or lab.
- In the event of lab staff turnover or graduation, require all materials be identified and dispose of all materials and samples that are no longer in use or wanted.
- Submit frequent waste pick up request forms to reduce the amount of hazards in your laboratory to reduce risk and create more lab space.

Identifying Unknown Materials

Every effort should be made to properly identify an unknown chemical prior to contacting EHS. The following steps should be taken to help identify unknown and unlabeled chemicals:

- Consult with the Principal Investigator (PI) or Lab Manager about the type of work that was being conducted. Eliminating certain chemicals as a possibility helps narrow down the determination and also helps with final waste disposal. This is especially important for materials that may contain mercury, polychlorinated biphenyls (PCBs), polyfluoroalkyl substances (PFAS), dioxins, and furans, as they require special handling for disposal.
- Ask area personnel about the container.
- Contact groups that previously used that area and see if they can provide any information on the identity of the materials.
- Review projects current in process.

Disposal of Unknown Materials

If the identity of the material cannot be determined through all of the steps identified above, EHS will remove the material and dispose of it. However, EHS requires three tests be performed and the results recorded on the hazardous waste tag prior to pick up. These tests can reduce the high cost of testing required to have unknown chemical waste transported off campus and are required to legally transport waste offsite. The three tests consist of a *Reactive Test, pH Level,* and a *Flammability Test.* A Reactive Sulfide Test and a Reactive Cyanide Test are also required before chemicals can be transported but may not be readily available to the labs on campus. These can be arranged by EH&S if needed. EH&S will not remove any wastes for disposal without a properly filled out hazardous waste tag on the container, and a properly submitted Hazardous Waste Request. In addition, based on the number of unknowns in a lab, EHS will charge the generator or department a fee per bottle for the analysis and identification of the waste.

Visually evaluate the material, if the container is swollen, compromised, damaged or compromised, or shows visible evidence of crystal growth within the container, contact EHS immediately.

Unknown Testing Procedures

Test #1 – Water Miscibility/Reactivity (Mandatory in order to have Unknown Chemicals picked up by EH&S)

This test is to determine if the unknown material reacts with water, and also whether the material is soluble in water, or whether it sinks or floats when added to water.

Liquid Unknown

Add a small amount of unknown liquid to a new test tube. Slowly allow one drop of deionized water to flow down the inside of the test tube into the unknown liquid. Any endo- or exothermic (i.e. heat or cold) reactions are considered positive for water reactivity and the results must be noted on the hazardous waste tag attached to the unknown. Note, if the unknown material exhibits any signs of reacting with water do not complete any further testing and contact EHS for proper disposal.

If unknown liquid does not react to water as identified above, add a small amount of the unknown liquid to a test tube containing deionized water to determine if the material sinks, floats, or is soluble in water.



Examples: Unknown Material Floats on Water

Unknown Material Sinks in Water (Water Dyed Green)



Solid Unknown

Use a spatula to obtain a small amount of the material as possible and place on a watch glass. Place one drop of water onto the side of the watch glass and allow to run down into the unknown solid. Any endo- or exothermic (i.e. heat or cold) reactions are considered positive for water reactivity and the results must be noted on the hazardous waste tag attached to the unknown. Note, if the unknown material exhibits any signs of reacting with water do not complete any further testing and contact EHS for proper disposal.



Example of Water Drop Being Added to Solid Unknown

If unknown solid does not react to water as identified above, add an additional amount of the solid to a test tube with deionized water to determine if material is soluble in water. Record results of testing on hazardous waste tag attached to the unknown.

Test #2 – pH (Mandatory in order to have Unknown Chemicals picked up by EH&S)

Liquid

Dip the pH paper into the unknown liquid and compare the test strip to the pH chart on the container. Note the results of the test on the hazardous waste tag attached to the unknown.



Example: Unknown liquid has a pH of 0 indicating an acidic solution

Solid

If material from has been determined not to be water reactive, place a small amount of the solid material on a watch glass with enough deionized water to dissolve or wet the solid and dip the pH strip into the solution. Note the results of the test on the hazardous waste tag attached to the unknown.



Example: pH strip being dipped into unknown solid/water solution

Test #3 – Flammability (Mandatory in order to have Unknown Chemicals picked up by EH&S)

Liquid

Dip cotton swab into unknown liquid and pass-through flame of Bunsen burner. Note results of flammability test as either "positive" or "negative" on hazardous waste tag.



Example: Positive Flammable Reaction

Example: Negative Flammable Reaction



Solid

If unknown did not react with water, wet the cotton swab with deionized water and then roll into the solid unknown to coat the swab with the unknown solid material. Pass the cotton swab through the flame of a Bunsen burner. Note the results of the flammability test as either "positive" or "negative" on the hazardous waste tag.



Example: Positive Flammable Reaction



Example: Negative Flammable Reaction



Test #4 - Reactive Sulfide Test (This test is optional and may not be readily available to UT

Laboratories but can greatly reduce the cost of having Unknown Chemicals removed from campus.)

To test for reactive sulfides in an unknown substance, the most common and reliable method is the lead acetate test. This involves placing a portion of the sample in an acid solution and observing the gas that evolves. If the sample contains sulfides, the evolved gas will react with *lead acetate paper*, turning it dark brown or black.

Detailed Steps:

- 1. Prepare the sample: Place a small amount of the unknown sample (1 ml or 1 gram) in a culture tube.
- 2. Acidify the sample: Add a few drops of dilute acid (e.g., hydrochloric acid) to the sample.
- 3. Prepare the lead acetate paper: Wet a piece of lead acetate paper with a drop of water.
- 4. **Test for sulfide:** Immediately place the wetted lead acetate paper over the culture tube, keeping the paper close to the surface of the solution.
- 5. **Observe the paper:** If the lead acetate paper turns dark brown or black, it indicates the presence of reactive sulfides.

Additional Considerations:

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Safety:

Sulfides can produce toxic gases like hydrogen sulfide (H2S), so it's crucial to work in a well-ventilated area and wear appropriate personal protective equipment (PPE).

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Confirmatory tests:

The lead acetate test is a qualitative test, meaning it indicates the presence or absence of sulfides but not the concentration. For more precise quantification, other methods like *silver nitrate test* or spectrophotometry can be used.

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Hazardous waste classification:

If sulfides are present, the waste may be classified as reactive hazardous waste according to **EPA regulations**.

Test #5 - Reactive Cyanides Test (This test is optional and may not be readily available to

UT Laboratories but can greatly reduce the cost of having Unknown Chemicals removed from campus.)

Method 9012B, also known as the **Distillation and Colorimetric Analysis of Cyanide**, is used to determine both total cyanide and cyanide amenable to chlorination, but it does not measure reactive cyanides. It primarily detects inorganic cyanides, including soluble salts and complexes.

Here's a breakdown of what Method 9012B does and doesn't do:

What Method 9012B does:

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Determines Total Cyanide:

It measures the total amount of cyanide present in a sample, including both simple salts and complexed cyanides.

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Measures Cyanide Amenable to Chlorination:

It specifically identifies the amount of cyanide that can be readily converted to chlorine and other derivatives in the presence of chlorine.

What Method 9012B does NOT do:

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Determine Reactive Cyanide:

It does not specifically measure "reactive" cyanide, which refers to cyanide that is more likely to be involved in chemical reactions or to react with other substances.

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Distinguish Between Different Cyanide Species:

While it measures total cyanide and cyanide amenable to chlorination, it doesn't differentiate between various forms of cyanide (e.g., simple cyanide salts, complexes with metals, etc.).

How Method 9012B works:

- 1. **Distillation:** The sample is distilled under acidic conditions to liberate free cyanide gas (HCN) from both simple cyanide salts and metal cyanide complexes.
- 2. **Collection:** The released HCN gas is collected in a solution.
- 3. Colorimetric Analysis: The HCN is then analyzed colorimetrically, often using a reaction with chloramine-T and pyridine-barbituric acid, or other reagents.