

Inorganic Analysis

The Inorganic Analysis includes the Metal Analysis Tests and the Anions Chart. The Main Solids and Liquids Charts may direct you to complete the Metal Analysis Tests if an anion has already been identified on the Main Chart. If the cation, metal portion, of an inorganic compound has already been identified on the Main Chart then you may have been directed to only the Anions Chart. When you have successfully identified both the cation and the anion then the two names (endpoints) are put together (cation first, anion last) to form the name of the compound.

Metal Analysis Tests

The Metal Analysis Tests are a series of screening tests used to identify the cation (metal portion) of a metal salt and to identify mixtures of metal salts dissolved in water. Neutral or acidic aqueous solutions should be screened for metals. Basic solutions are unlikely to contain metals, as most metals (arsenic and mercury are exceptions) will precipitate out of basic solutions.

These tests can be conclusive, but are generally meant to quickly direct the HazCat user toward a metal confirmation test. Most of these tests favor one or several metals, allowing for the identification of certain metals in the presence of other metals. A very high concentration of one metal may mask other metals present.

Use the Inorganic Analysis Summary Form to record the observations.

REAGENTS NEEDED

Plastic Beaker		Sodium Hydroxide	RE2308
Distilled Water	RE2300	Thioacetamide	RE2309
3N Hydrochloric Acid	RE2301	Hydrogen Peroxide	RE2310
Nitric Acid	RE2302	Diphenylcarbazide	RE2311
Hydrochloric Acid	RE2303	Dithizone	RE2312
Sodium Borate	RE2305	Filter Paper	RE2341
Potassium Ferrocyanide	RE2306	pH Test strips	HW4048
Ammonium Hydroxide	RE2307	Methanol	RE2325

INSTRUCTIONS

- Set up a **Filter Paper RE2341** by soaking the paper with just enough Potassium Ferrocyanide **RE2306** to wet the entire filter paper. Place the paper on a paper towel and allow it to dry.
- Line up nine test tubes in the test tube rack in front of numbers 1-9 on the Tube Manager.
- Liquid:** Do a pH Test on the unknown liquid. If the pH is less than 3, fill test tube **#1** with the unknown liquid and go to Step 6. If the pH is greater than 3, fill the Plastic Beaker with the unknown liquid to the ½ fl oz (15ml) mark.
 - Adjust the pH to less than 3 by adding 1 drop of Nitric Acid **RE2302**. Repeat as necessary until the pH is less than 3. Go to Step #5.

Solid: Add 2 spoonfuls of the unknown to a plastic beaker.

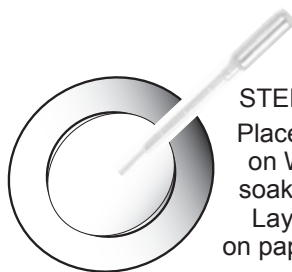
- Fill the Plastic Beaker with Distilled Water **RE2300** to the 10ml mark. Stir well. If the solid dissolves add 12 drops of Nitric Acid **RE2302** and go to Step 4. If it does not dissolve continue.
 - Add Nitric Acid **RE2302** to the Plastic Beaker up to the ½ fl oz (15ml) mark. Stir well. If the solid dissolves go to Step 4. If it does not dissolve continue.
 - Add Hydrochloric Acid **RE2303** to the Plastic Beaker up to the 20ml mark. Stir well. Go to Step #4.
- Add Distilled Water **RE2300** to the Plastic Beaker up to the 25ml mark.
 - Fill test tube **#1** with the unknown solution from the plastic beaker. Refill test tube **#1** as needed.

Section VI - Inorganic Analysis

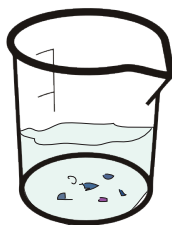
6. Add 2 spoonfuls of Sodium Borate **RE2305** to test tube **#2**. Add 6 drops of the solution from test tube **#1** to test tube **#2**.
7. Add ½ inch of Potassium Ferrocyanide **RE2306** to test tube **#3**.
8. Add ½ inch of Ammonium Hydroxide **RE2307** to test tube **#4**.
9. Add ½ inch of Sodium Hydroxide **RE2308** to test tube **#5**.
10. Add ½ inch of Hydrochloric Acid **RE2303** to test tube **#6**.
11. Add 12 drops, ¼ inch of the unknown solution from test tube **#1** to each of test tubes **#3, 4, 5, and 6**. Allow the drops to run down the side of the test tube. **DO NOT SHAKE THE TEST TUBES**.
12. Refer to the charts for Metal Analysis Tests III, IV, V, and VI on the pages that follow and highlight any colors observed on the Summary Form Step 12. If a white solid has appeared in test tube **#6**
– do the Cadmium Test now to further screen for lead, mercury and silver, then continue test.
13. Heat the contents of test tube **#2** with the inner blue cone of the torch flame. The Sodium Borate **RE2305** will puff up. Continue to heat and melt the puffed up solid until it flows back to the bottom of the test tube. This may require rotating the test tube slowly in the torch flame. Heat until it becomes a red hot molten mass at the bottom of the test tube.
14. Put the test tube into the Erlenmeyer Flask to cool. The test tube may shatter as it cools.
15. Dip the looped end of a **Flame Test Wire** into the unknown solution in test tube **#1**.
16. Place the looped end of the **Flame Test Wire** at the tip of the inner blue cone of the torch flame. Observe for color changes in the flame. Repeat as needed. Refer to the chart for Metal Analysis Test I and highlight any color observed on the Summary Form Step 16.
17. Pour the glass pieces from the Erlenmeyer Flask onto a piece of white paper. Examine the broken glass for colors. Refer to the charts for Metal Analysis Test II and highlight the glass color(s) observed on the Summary Form Step 17.
18. Drip one drop of the unknown solution from test tube **#1** onto the center of the **Filter Paper** made in Step 1. Set aside and allow to dry.
19. Examine test tubes **#3, 4, 5 and 6** again to observe if any new layers of a different color or if any gels or solids have formed and highlight any new colors/gels/solids observed on the Summary Form Step 19.
20. Add 12 drops of the unknown solution from test tube **#1** to test tube **#7**. Add 3 drops of Thioacetamide **RE2309**. Shake well. Refer to the chart for Metal Analysis Test VII and highlight any colors observed on the Summary Form Step 20.
21. Add 3 drops of Hydrochloric Acid **RE2303** to test tube **#7** and shake well. Wait for 1 minute and then refer to the chart for Metal Analysis Test VII and highlight any colors observed on the Summary Form Step 21.
22. *Gently warm* the liquid in test tube **#7** by passing it in and out of the torch flame for one minute. Do not boil the liquid. Set the test tube back in the test tube rack and wait for 2 minutes. Refer to the chart for Metal Analysis Test VII and highlight any colors observed on the Summary Form Step 22.
23. Add 12 drops of ammonium hydroxide **RE2307** to test tube **#7**. Shake well. Wait for 1 minute and then refer to the chart for Metal Analysis Test VII and highlight any colors observed on the Summary Form Step 23.
24. Add ½ inch of **RE2325** to test tube **#8**. Remove the lid from **RE2311** and insert a clean pipette. Tap the bottom of the bottle with the pipette tip and stir the solid inside. Place the pipette into test tube and mix well by squeezing the pipette several times. Dispose of the pipette. Add 12 drops of the unknown solution from test tube **#1**, shake and observe as it reacts for 2 minutes. Refer to chart for Metal Analysis Test VIII and highlight any colors observed on the Summary Form Step 24.

25. Add 1 drop of ammonium hydroxide **RE2307** to test tube **#8**, wait for 1 minute and 30 seconds without moving the test tube before shaking lightly. Wait 1 more minute and observe the test tube to see if the color changes, fades or darkens. If no color change occurred repeat for up to 4 more drops of **RE2307**. Refer to the chart for Metal Analysis Test VIII and record any new colors and possible metals on the Inorganic Analysis Summary Form Step 25.
26. Add 12 drops of Dithizone **RE2312** to test tube **#5**. Mix well with a pipette. Allow to settle. Refer to the chart for Metal Analysis Test IX and highlight any colors observed on the Summary Form Step 26.
27. Observe any colored rings or spots that have formed on the **Filter Paper**, compare to the colors on the Metals Analysis Test III Chart and highlight any colors observed on the Summary Form Step 27.
28. Summarize any metals indicated by highlighting them on the Inorganic Analysis Summary Form. Do the confirmation tests for those metals indicated (the page number for each confirmation test is listed on the form). In many cases there are more than one test that can be used to confirm a metal, however for some metals a confirmation test does not exist.
To identify the anion go to the Anions Chart at the end of this Section.

Metal Analysis Test Set-Up



STEP #1
Place Filter Paper
on Watch Glass,
soak with RE2306
Lay Filter Paper
on paper towel to dry







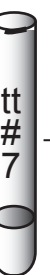




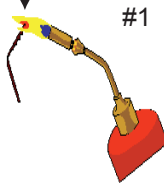



STEP #3

Liquid: Add ½ inch of unknown liquid. If pH is > 3 add drops of RE2302.

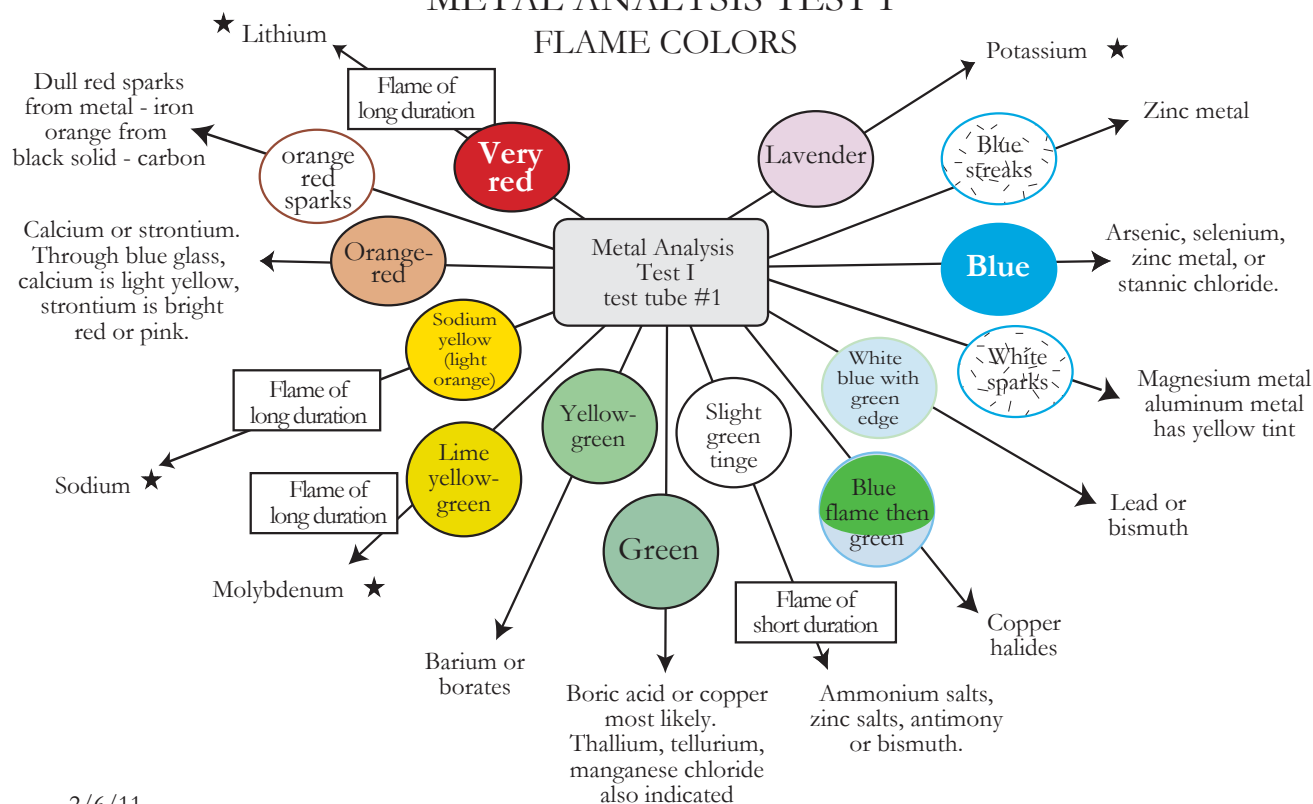
Solid: Add 2 spoonfuls of unknown solid. Add ¼ inch of RE2300. Stir. If solid does not dissolve add ¼ inch of RE2302. Stir. If solid does not dissolve add more RE2302. Stir. If solid does not dissolve add ¼ inch of RE2303.

(set up 8 test tubes as shown below, #5 will be used for multiple tests:)

	MA Test II	MA Test III	MA Test IV	MA Test V	MA Test VI	MA Test VII	MA Test VII	MA Test VII	MA Test VIII	MA Test IX	
											
Unknown solution made in STEP #s 3-5 	Add ¼ inch of RE2305, add 6 drops from test tube #1	Add 1ml of RE2306 add 12 drops from test tube #1	Add 1ml of RE2307 add 12 drops from test tube #1	Add 1ml of RE2308 add 12 drops from test tube #1	Add 1ml of RE2303 add 12 drops from test tube #1	Add 12 drops from test tube #1 add 3 drops of RE2309 to test tube #7	Add 3 drops of RE2303 to test tube #7 wait 1 min	 Gently warm test tube. #7 do not boil wait 1 min	Add 12 drops of RE2307 to test tube #7 wait 1 min	Add ½" of RE2325 to the #9 test tube Add solid from RE2311 bottle with pipette tip and stir. Add 12 drops from test tube #1	Add 1ml of RE2312 to the #5 test tube at STEP #25

METAL ANALYSIS TEST I

FLAME COLORS



2/6/11

Observe the flame color through the:

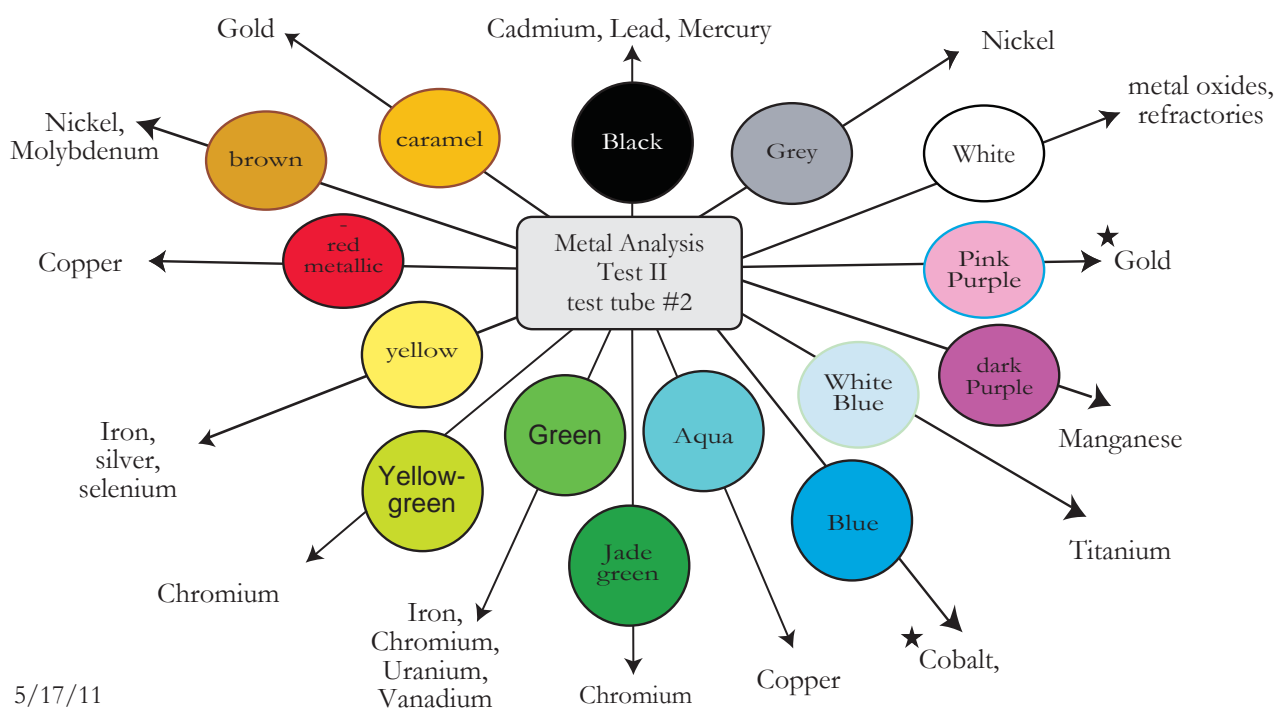
Blue Glass - Calcium is light yellow, Strontium is brighter, Potassium is red/blue, Sodium is invisible

Green glass - Calcium is orange, Strontium is yellow, Potassium is blue/green

METAL ANALYSIS TEST II

Sodium Borate Glass Colors

RE2305



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METAL ANALYSIS TEST III

Potassium Ferrocyanide Colors

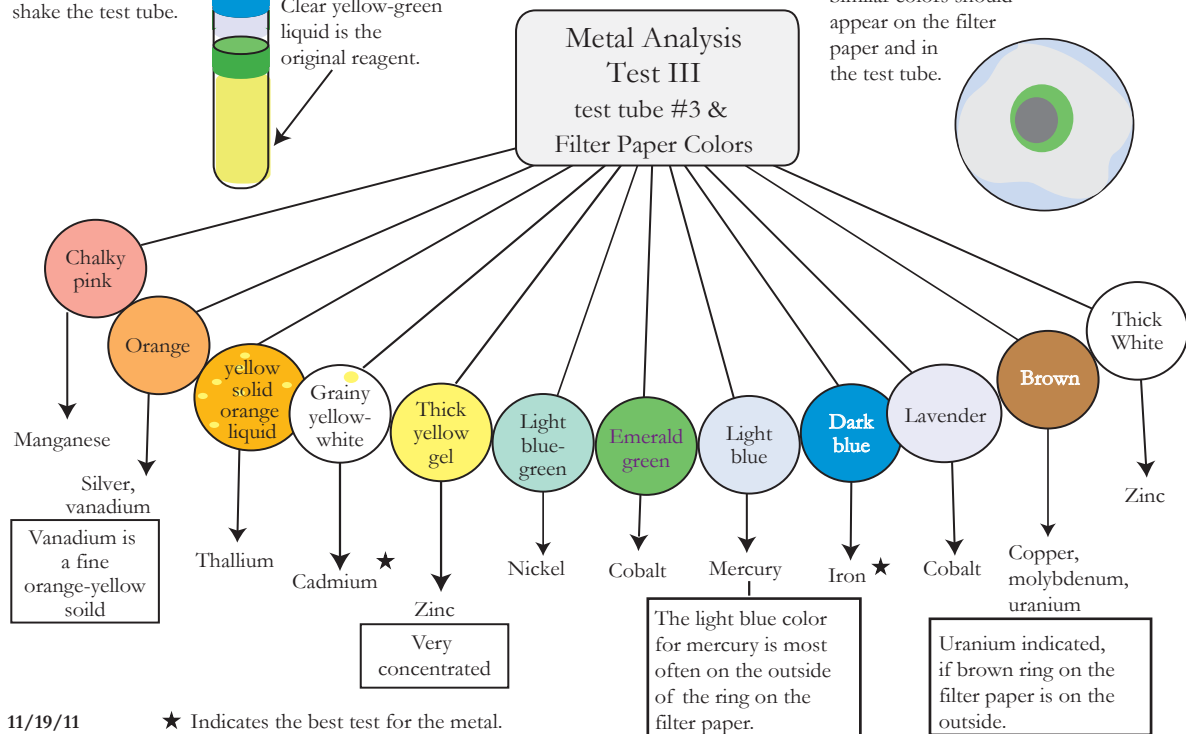
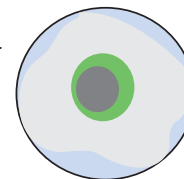
RE2306

Layers may form over a few minutes. Be careful not to shake the test tube.



Clear yellow-green liquid is the original reagent.

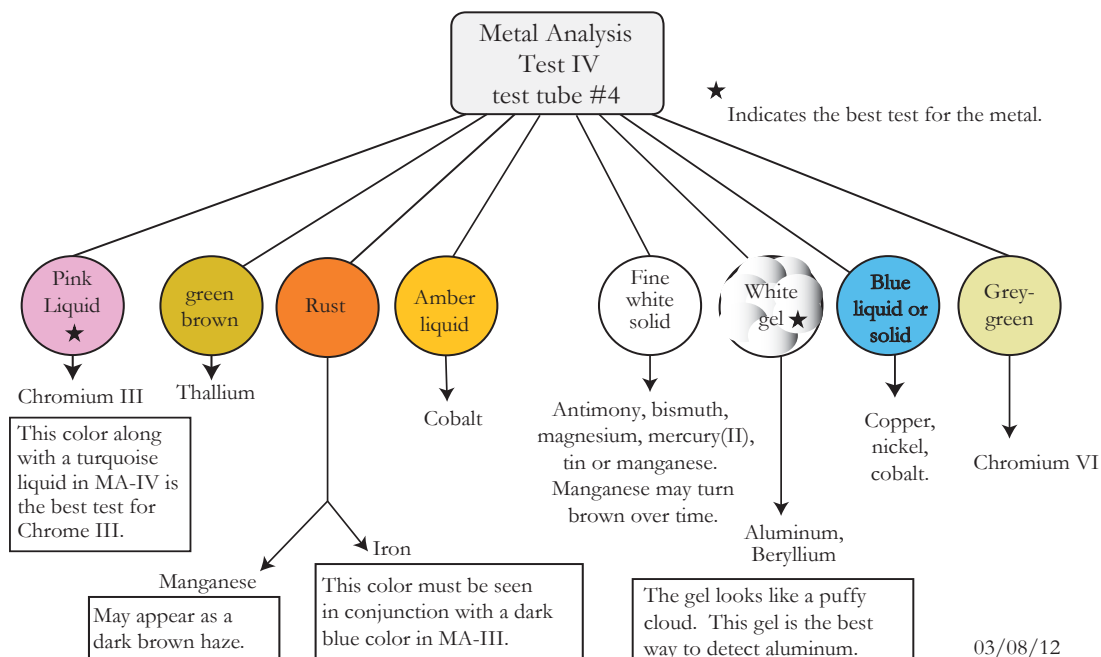
Similar colors should appear on the filter paper and in the test tube.



METAL ANALYSIS TEST IV

Ammonium Hydroxide Colors

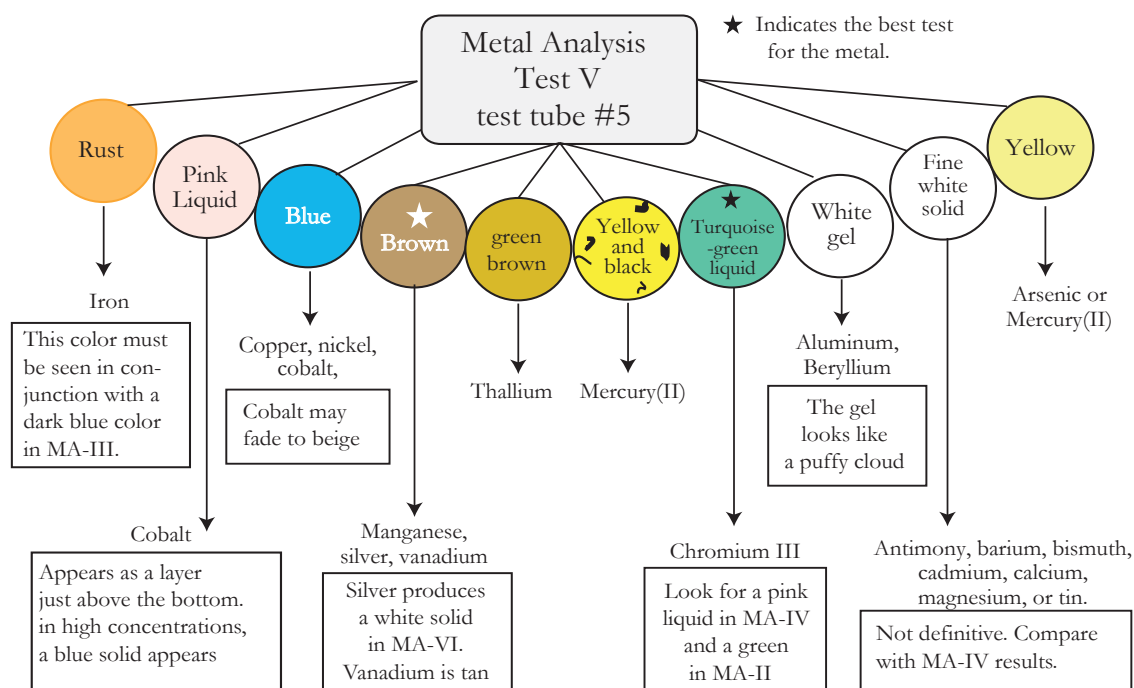
RE2307



METAL ANALYSIS TEST V

Sodium Hydroxide Colors

RE2308

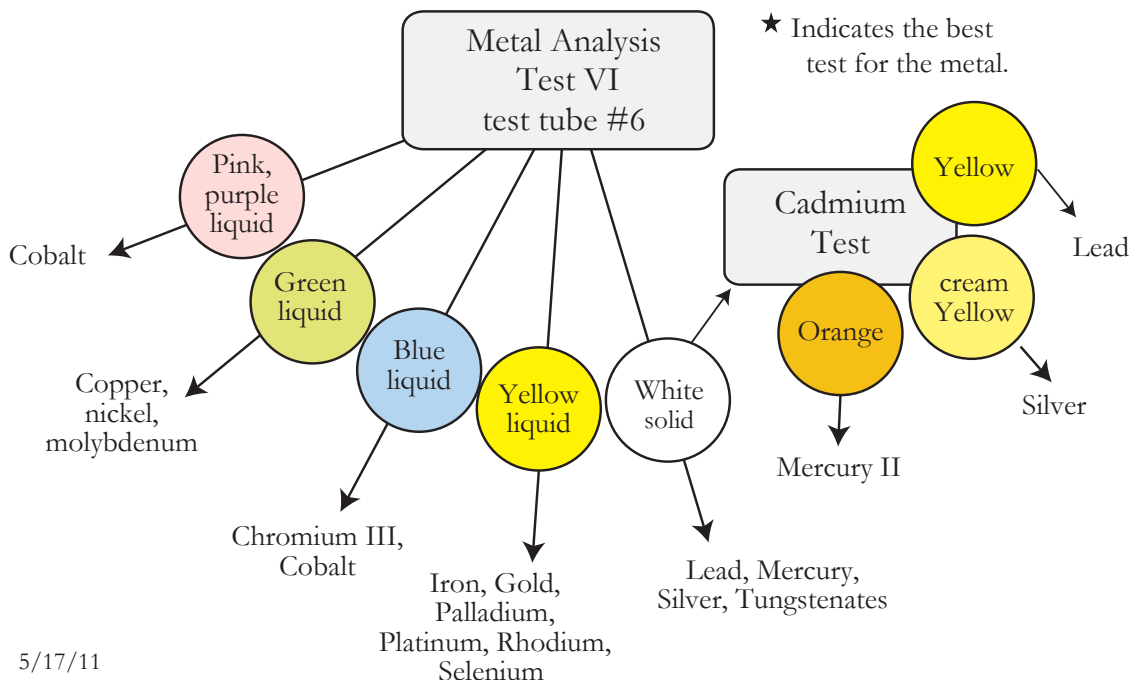


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METAL ANALYSIS TEST VI

Hydrochloric Acid Colors

RE2303



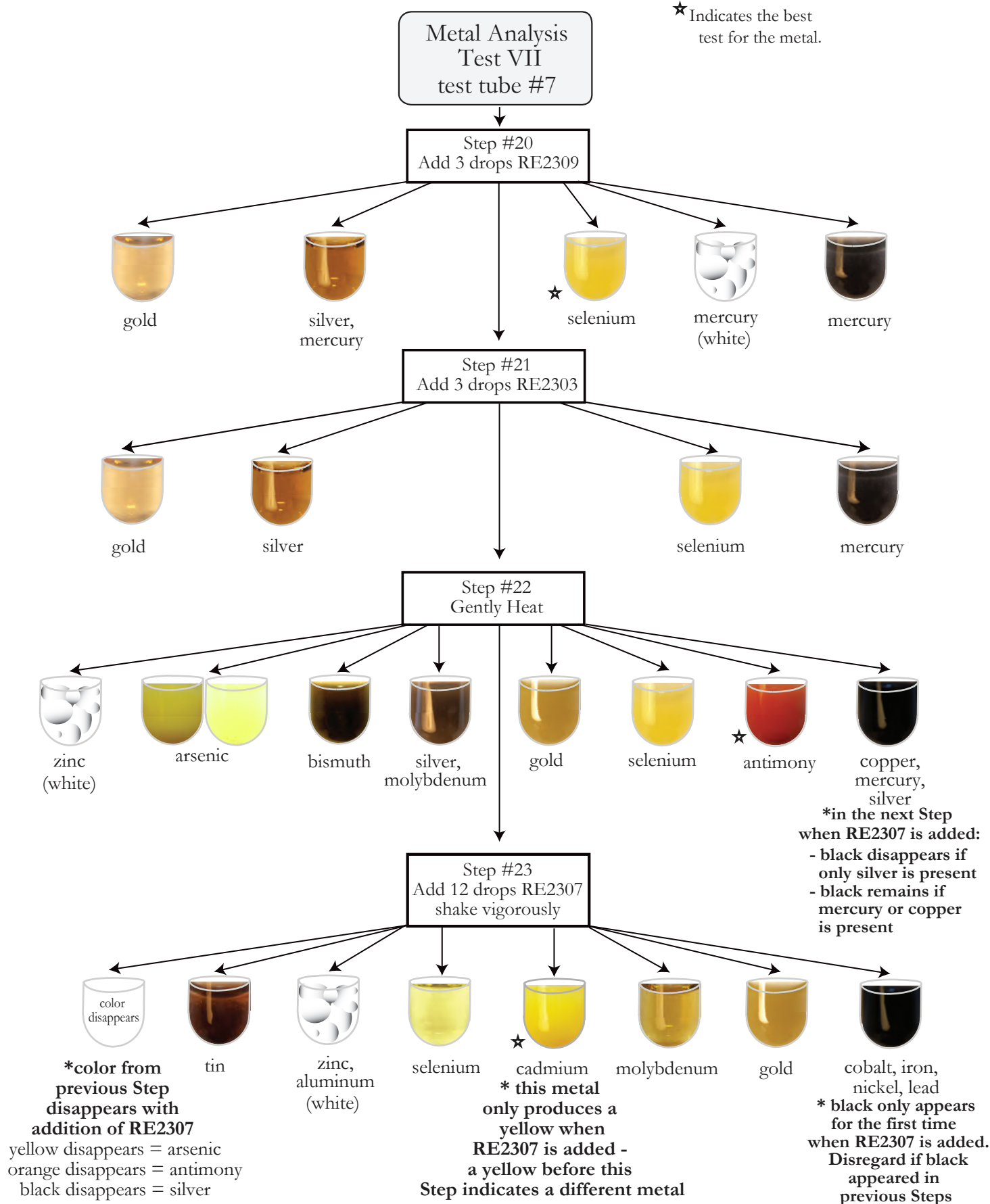
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METAL ANALYSIS TEST VII

Metal Sulfide Colors

RE2309

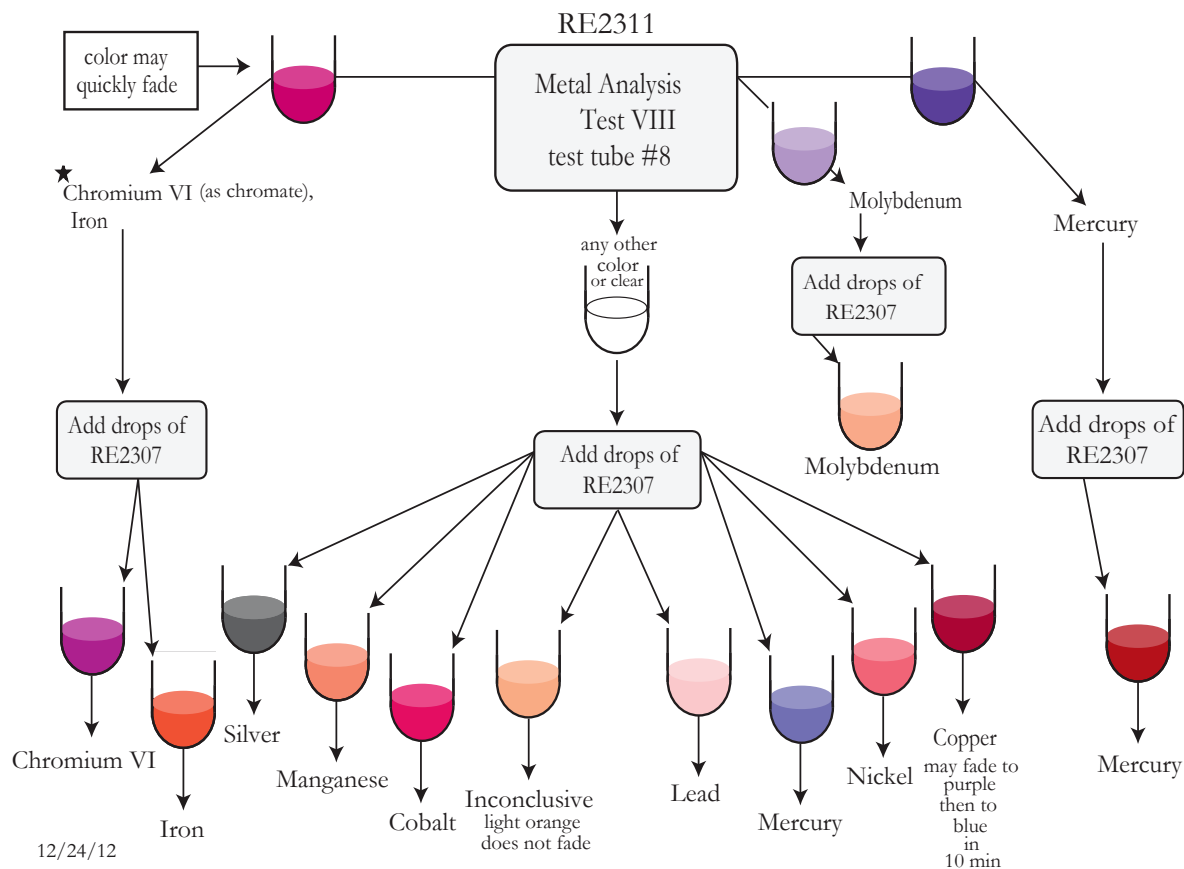
★ Indicates the best test for the metal.



METAL ANALYSIS TEST VIII

Diphenylcarbazide Colors

★ Indicates the best test for the metal.



METAL ANALYSIS TEST IX

Dithizone Colors

RE2312

★ Indicates the best test for the metal.

