

## Preliminary instructions

- Always clean each and every instrument you use as carefully as possible (especially for ion detection this is a **requirement**). If you know/imagine how small an atom is you may imagine which amount of residue of another substance leads to false positives.
- Also if you got a salt, take a fraction of it into another (clean) test tube and solve it in H<sub>2</sub>O (clean distilled water!). This is your basic sample now.
- For each test take a small (!) probe of the basic sample into another test tube and make the respective test. Afterwards clean it. Take another bit and perform the next test. Never try to perform multiple tests on one and the same probe.

No joke: If you are allergic to certain substances or if you are color blind, please tell the supervisor or the professor. The latter one is hindering the first one may be dangerous.

## Proposal on the order of tests for ion detection

See the tables below for any detailed instructions. Follow the steps and always consider that, if you have one compound to detect, it will only contain **one anion** and **one cation**. Everything else would be a mix. So you can stop testing when you are sure you detected the correct ions. Also note, that the supervisor does not always work properly as well. My mixed probe was „to well“mixed and contained something else than listed. In such case ask for a **new and clean probe** instead of being pissed off because of their mistake!!!

1. First of all take a **look** on the salt or solution. Does it have a color? The color may already give a hint what ions it contains. See the following overview:

Color	Probably contains:
blue	<b>Copper</b> ions (Cu <sup>2+</sup> )
green	<b>Nickel</b> (Ni <sup>2+</sup> ) or <b>Chromium</b> (Cr <sup>3+</sup> ) ions
brown	Bi- (Fe <sup>2+</sup> ) or trivalent (Fe <sup>3+</sup> ) <b>Iron</b> ions

*But note, that this is not a final result.* It just gives a hint and makes life easier.

2. Perform a **flame test**. Possible results can be taken from the table for cations.  
Flame test instructions:
  - take a magnesia stick and anneal it using a burner, until the flame is colorless.
  - if you got a solid compound just put the tip of the stick in HCl (f) and afterwards into the solid compound (e.g. salt) such that some particles stick to the magnesia stick. If you got a liquid, just out the magnesia stick directly into the liquid. FYI: The burner „cleans“ the stick.
  - hold the tip of the stick into the flame of the burner (upper, usually colorless part of the flame). See the color and make your assumptions according to the table.
3. **Watchglass test**. For this test take two watchglasses and stick a moist UNITEST-strip (pH-test strip) onto one of them. On the other watchglass put a probe of you substance (may it be liquid or solid) and add some hydroxide solution to it. I recommend using NaOH solution. Afterwards put the first watchglass upside down on the watchglass with the substance and the test strip will turn blue if the substance contains NH<sub>4</sub><sup>+</sup> ions.
4. **Halogenides** (F<sup>-</sup>, Cl<sup>-</sup>, Br<sup>-</sup>, I<sup>-</sup>) and **phosphate** (PO<sub>4</sub><sup>3-</sup>). The „MOHR salt“ (AgNO<sub>3</sub>) added to a sample containing one of the above ions will give a precipitate. Note that a yellow precipitate can be the result of the presence of I<sup>-</sup> or PO<sub>4</sub><sup>3-</sup>. To verify/falsify either assumption you will have to perform another appropriate test (i.e. to detect PO<sub>4</sub><sup>3-</sup>).
5. Test for **sulfate** (SO<sub>4</sub><sup>2-</sup>) or **carbonate** (CO<sub>3</sub><sup>2-</sup>) using barium chloride or any other appropriate test method.
6. **Acetate** is the next. It is detected by adding a stronger acid (such as sulfuric acid - H<sub>2</sub>SO<sub>4</sub>) and testing the smell. Acetate produces acetic acid which smells just like vinegar.
7. „Asshole test“ for **nitrate** (NO<sub>3</sub><sup>-</sup>) - my favorite one - just as described in the table below.
8. **Barium** (Ba<sup>2+</sup>) and **Calcium** (Ca<sup>2+</sup>) using the „reverse test“ for sulfates and carbonates and adding HCl (f) to check whether the precipitate solves.
9. **Iron** (Fe<sup>2+/3+</sup>) ions using the tests described in the table below.
10. **Copper** (Cu<sup>2+</sup>) and **Zinc** (Zn<sup>2+</sup>) using 1. flame test, then 2. K<sub>4</sub>[Fe(CN)<sub>6</sub>] and at last NaOH.
11. **Nickel, Cobalt, Lead, Magnesium, Aluminium** in this order! All other missing ions afterwards.
12. As the very last test try detecting Manganese and Chromium. I personally do not trust (because of my experience) either of the tests and therefore would always preclude any other options before.

In general perform easy tests first and skip any tests if you are already sure you detected a specific ion. Try always to preclude ions you cannot detect. The last few tests are not really reliable/exact.

## Cation detection

<b>Ion</b>	<b>Flame test/borax</b>	<b>Other tests</b>
Li <sup>+</sup>	FT: red & yellow / orange	Cooking it with Na <sub>2</sub> HPO <sub>4</sub> (sodium hydro-phosphate) and NaOH (sodium hydroxide) gives a white precipitate (Ethanol aka C <sub>2</sub> H <sub>5</sub> OH promotes the reaction): $\text{HPO}_4^{2-} + 3 \text{Li}^+ \rightarrow \text{Li}_3\text{PO}_4 + \text{H}^+$
K <sup>+</sup>	FT: red & violet	$\text{ClO}_4^- + \text{K}^+ \rightarrow \text{KClO}_4$ (cold hydrochloric acid [sol.]; ethanol makes the reaction more sensitive)
Na <sup>+</sup>	FT: yellow (tends to orange)	none
NH <sub>4</sub> <sup>+</sup>	(sometimes orange) be careful, magnesia sticks produce a very similar color while annealing them.	$\text{NH}_4^+ + \text{NaOH} \rightarrow \text{Na}^+ + \text{NH}_3 + \text{H}_2\text{O}$ NH <sub>3</sub> can be detected by the so-called watchglass test. Using a watchglass with a moist pH-test stripe you will see a basic reaction (usually blue color on the stripe): $\text{NH}_3 + \text{H}_2\text{O} \rightarrow \text{NH}_4\text{OH}$ If you are skilled and experienced enough you may use a test-tube instead of a watchglass. You even may use smell testing ( <i>highly experienced people only!</i> ).
Mg <sup>2+</sup>	none	(NH <sub>4</sub> ) <sub>2</sub> HPO <sub>4</sub> (in NH <sub>3</sub> sol.) gives a white precipitate of Mg (NH <sub>4</sub> ) PO <sub>4</sub> · 6H <sub>2</sub> O
Ca <sup>2+</sup>	FT: red & green	A sated solution of K <sub>4</sub> [Fe(CN) <sub>6</sub> ] (German: „gelbes Blutlaugensalz“) and NH <sub>4</sub> Cl (with a surplus of ammonia) gives a white precipitate of Ca (NH <sub>4</sub> ) <sub>2</sub> [Fe(CN) <sub>6</sub> ] under cold conditions – so cool down the solution (e.g. under running water). Easier <b>as</b> below <b>with Barium</b> . It's not as reliable, but take a look on it!!!
Ba <sup>2+</sup>	FT: green	$\text{Me}^{2+} + \text{SO}_4^{2-} \rightarrow \text{MeSO}_4$ (white precipitate soluble in HCl) $\text{Me}^{2+} + \text{CO}_3^{2-} \rightarrow \text{MeCO}_3$ (white precipitate insoluble in HCl) Me stands for either Ba (Barium) or Ca (Calcium).
Zn <sup>2+</sup>	none	Zinc can be tested by the same method as copper (see Cu <sup>2+</sup> )
Mn <sup>2+</sup>	Borax: violet	$\text{Mn}^{2+} + 2\text{OH}^- \rightarrow \text{Mn}(\text{OH})_2$ (pink precipitate; maybe not reliable) Use preclusion if possible.
Cu <sup>2+</sup>	FT: green	Add ammonia sol. (with a surplus of NH <sub>3</sub> ). This gives a dark blue precipitate of a [Cu(NH <sub>3</sub> ) <sub>4</sub> ] <sup>2+</sup> salt. Easier: $\text{Cu}^{2+} + 2\text{OH}^- \rightarrow \text{Cu}(\text{OH})_2$ (light blue precipitate that gets black when heating because of CuO. Take sodium hydroxide - NaOH - for the OH <sup>-</sup> ions)
Co <sup>2+</sup>	Borax: blue	Adding NH <sub>4</sub> SCN (German „Ammoniumthiocyanat“) gives blue Co(SCN) <sub>2</sub> in neutral solution or H <sub>2</sub> [Co(SCN) <sub>4</sub> ] otherwise. While adding diethylether the blue changes into an organic phase.
Ni <sup>2+</sup>	Borax: brown-red	Adding C <sub>4</sub> H <sub>8</sub> O <sub>2</sub> N <sub>2</sub> (German „Diacetyldioxim“) gives a red and flaky precipitate of Ni(C <sub>4</sub> H <sub>7</sub> O <sub>2</sub> N <sub>2</sub> ) <sub>2</sub> Easier: $\text{Ni}^{2+} + 2\text{OH}^- \rightarrow \text{Ni}(\text{OH})_2$ (red to orange precipitate)
Pb <sup>2+</sup>	none	Adding K <sub>2</sub> CrO <sub>4</sub> (potassium dichromate) in an ammonia sol. gives a yellow crystalline precipitate of PbCrO <sub>4</sub> which is soluble in NaOH and HNO <sub>3</sub> .
Fe <sup>2/3+</sup>	none	Fe <sup>2+</sup> : K <sub>3</sub> [Fe(CN) <sub>6</sub> ] turns blue („Berliner Blau“) Fe <sup>3+</sup> : K <sub>3</sub> [Fe(CN) <sub>6</sub> ] turns blue („Turnbulls Blau“ & „Berliner Blau“)
Cr <sup>3+</sup>	Borax: green	$\text{Cr}^{3+} + 3\text{OH}^- \rightarrow \text{Cr}(\text{OH})_3$ (gray-green precipitate) Adding a Cr <sup>3+</sup> solution to a mixture of NaOH & H <sub>2</sub> O <sub>2</sub> gives a yellow solution
Al <sup>3+</sup>	none	$\text{Al}^{3+} + 3\text{OH}^- \rightarrow \text{Al}(\text{OH})_3$ (white precipitate). Then add fluoride ions (F <sup>-</sup> ): $\text{Al}^{3+} + 6\text{F}^- \rightarrow [\text{AlF}_6]^{3-}$ (called cryolith; use concentrated NaOH sol.) Better via preclusion.

The gray fields denote the unreliable test methods.

## Anion detection

<i>Ion</i>	<i>Tests</i>
F <sup>-</sup>	Fe (SCN) <sub>3</sub> is discolored by F <sup>-</sup> and PO <sub>4</sub> <sup>3-</sup> Fe (SCN) <sub>3</sub> can be produced by mixing NH <sub>4</sub> SCN and FeCl <sub>3</sub> (dark red, almost <b>bloody J</b> )
Cl <sup>-</sup>	Ag <sup>+</sup> + X <sup>-</sup> → AgX (colored precipitate)
Br <sup>-</sup>	Cl <sup>-</sup> : white (soluble in 1% NH <sub>3</sub> sol.)
I <sup>-</sup>	Br <sup>-</sup> : yellowish/very light yellow (soluble only in conc. NH <sub>3</sub> sol.) I <sup>-</sup> : yellow (not soluble in NH <sub>3</sub> sol.) <b>Note</b> , that phosphate (PO <sub>4</sub> <sup>3+</sup> ) shows a similar color!
NO <sub>3</sub> <sup>-</sup>	I call this „ <b>asshole test</b> “ as a tribute to my teacher from 7 <sup>th</sup> to 10 <sup>th</sup> grade. She called it exactly the same. This refers to the visual result, not to the odor as the name may suggest.  Add 1ml of sated iron (II) sulfate (FeSO <sub>4</sub> ) to 1ml of the suspect solution. Then cant the test tube and carefully and slowly drop conc. sulfuric acid (H <sub>2</sub> SO <sub>4</sub> ) down the tube. If the probe contains NO <sub>3</sub> - there would appear a brown ring at the surface of the solution. <b>Perform this experiment under the hood only, because of the poisonous gases produced by this mixture!</b> 2NO <sub>3</sub> <sup>-</sup> + 6Fe <sup>2+</sup> + 8H <sup>+</sup> → 2NO + 6Fe <sup>3+</sup> + 4H <sub>2</sub> O [Fe(H <sub>2</sub> O) <sub>6</sub> ] <sup>2+</sup> + NO → [Fe(H <sub>2</sub> O) <sub>5</sub> NO] <sup>2+</sup> + H <sub>2</sub> O
SO <sub>4</sub> <sup>2-</sup>	Ca(OH) <sub>2</sub> + X <sub>2</sub> <sup>+</sup> → CaX + 2OH <sup>-</sup> (white precipitate)
CO <sub>3</sub> <sup>2-</sup>	For X = SO <sub>4</sub> <sup>2-</sup> the precipitate is insoluble in HCl For X = CO <sub>3</sub> <sup>2-</sup> the precipitate is soluble in HCl and produces CO <sub>2</sub> gas
PO <sub>4</sub> <sup>3-</sup>	Fe (SCN) <sub>3</sub> is discolored by F <sup>-</sup> and PO <sub>4</sub> <sup>3-</sup> <b>Better:</b> Adding (NH <sub>4</sub> ) <sub>6</sub> Mo <sub>7</sub> O <sub>24</sub> · 4H <sub>2</sub> O (German: „Ammoniummolybdat“) which gives a yellow precipitate while heating
CH <sub>3</sub> COO <sup>-</sup>	While adding diluted H <sub>2</sub> SO <sub>4</sub> (or in principle any stronger acid) acetic acid (CH <sub>3</sub> COOH) is being produced – and as you know this smells like vinegar, which suffices as proof for acetate ions.

## Titration

Do not forget to note the vale of *f* from the flask – it's needed for the calculations.  
Also transform any masses to [mg] and any volumes to [100ml] (since you use a smaller amount to titrate).

<b>Iodometry:</b>	Relation : $n_{\text{Thio}} = n_{\text{Cu}}$
H <sub>2</sub> S <sub>4</sub> O <sub>6</sub> <sup>2-</sup> + 2I → S <sub>4</sub> O <sub>6</sub> <sup>2-</sup> + 2HI	Equation: $m_{\text{Cu}} = V_{\text{Thio}} \cdot c_{\text{Thio}} \cdot M_{\text{Cu}} \cdot f$
<b>MOHR's approach to detect halogenides</b>	Relation : $n_{\text{AgNO}_3} = n_{\text{Cl}^-}$
Ag <sup>+</sup> + Cl <sup>-</sup> → AgCl	Equation: $m_{\text{Cl}^-} = V_{\text{AgNO}_3} \cdot c_{\text{AgNO}_3} \cdot M_{\text{Cl}^-} \cdot f$
<b>Complexometry</b>	Relation : $n_{\text{EDTA}} = n_{\text{Zn}}$
M <sup>2+</sup> + [H <sub>2</sub> Y] <sup>2-</sup> → [MY] <sup>4-/2+</sup> + 2H <sup>+</sup>	Equation: $m_{\text{Zn}} = V_{\text{EDTA}} \cdot c_{\text{EDTA}} \cdot M_{\text{Zn}} \cdot f$
<b>Permanganometry</b>	Relation : $5 \cdot n_{\text{KMnO}_4} = 2 \cdot n_{(\text{COOH})_2}$
[no reaction equation]	Equation: $m_{(\text{COOH})_2} = V_{\text{KMnO}_4} \cdot c_{\text{KMnO}_4} \cdot M_{(\text{COOH})_2} \cdot f$
<b>Potentiometric titration (conductimetry)</b>	Relation : $n_{\text{NaOH}} = n_{\text{HCl}}$
NaOH + HCl → NaCl + H <sub>2</sub> O	Equation: $m_{\text{HCl}} = V_{\text{NaOH}} \cdot c_{\text{NaOH}} \cdot M_{\text{HCl}} \cdot f$

Test	Ion	Sample 1		Sample 2		Sample 3		Mixed sample	
		Flame test	Other test	Flame test	Other test	Flame test	Other test	Flame test	Other test
Flame test	Li <sup>+</sup>								
	K <sup>+</sup>								
	Na <sup>+</sup>								
	Ca <sup>2+</sup>								
	Ba <sup>2+</sup>								
	Cu <sup>2+</sup>								
	NH <sub>4</sub> <sup>+</sup>								
Mohr salt	Cl <sup>-</sup>								
	Br <sup>-</sup>								
	I <sup>-</sup>								
	F <sup>-</sup>								
	PO <sub>4</sub> <sup>3-</sup>								
Barium chloride	SO <sub>4</sub> <sup>2-</sup>								
	CO <sub>3</sub> <sup>2-</sup>								
Smell	CH <sub>3</sub> COO <sup>-</sup>								
Ass-hole	NO <sub>3</sub> <sup>-</sup>								
Remaining tests	Fe <sup>2+/3+</sup>								
	Zn <sup>2+</sup>								
	Ni <sup>2+</sup>								
	Co <sup>2+</sup>								
	Pb <sup>2+</sup>								
	Mg <sup>2+</sup>								
	Al <sup>3+</sup>								
	Cr <sup>3+</sup>								
Mn <sup>2+</sup>									
Conclusion:									

The order is about the same as in my proposal on the first sheet. The gray items in the „ion“ column denote that you should perform the flame test to check for their existence. The light blue rows are just for brevity.