



BUILDING CHEMISTRY

LAB 1

FOUNDATIONS OF QUALITATIVE CHEMICAL ANALYSIS

DETECTION OF SELECTED IONS

THEORETICAL BACKGROUND

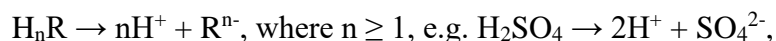
The knowledge of the chemical composition of substances provides information about the type of material, their components, or the kind of corrosion process has occurred. The knowledge of the chemical composition of the environment provides information about the possibility of an aggressive influence on building materials, and the anti-corrosion protection methods that should be applied.

In chemical analysis, the concept of ions recurs very often. An ion is an atom or molecule that has a non-zero net electrical charge. The two types of ions are:

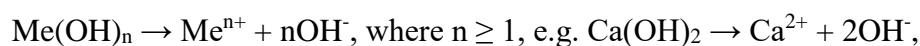
- positively charged ions - cations; Those are the hydrogen ions, metals ions, and some groups of elements which are positively charged, e.g. H^+ , Ca^{2+} , NH_4^+ ;
- negatively charged ions - anions; Those are the ions left after the removal of hydrogen atoms from an acid, and are called acid radicals.

Chemical compounds in aqueous solutions dissociate:

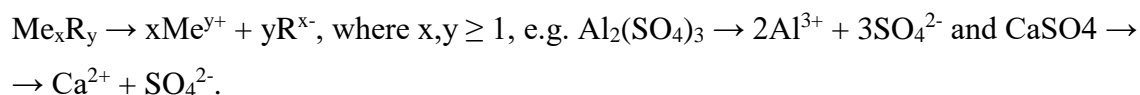
- acids – into hydrons and acid radicals



- bases – into metal ions and hydroxide ions



- salts – into metal ions or NH_4^+ and acid radicals



If x and $y = 1$, then valence of a cation = valence an of anion.



Methods for material composition analysis

There are two basic types of compound analysis:

- qualitative analysis
- quantitative analysis

In analytical chemistry, qualitative analysis is a method used for identification of compounds, i.e. gives answer to the question of what elements or functional groups are present in a substance, whereas quantitative analysis serves to determine the absolute or relative abundance (concentration) of ions, elements, or compounds in a sample.

Methods for analytical chemistry

Analytical chemistry consists of classical, wet chemical methods and modern, instrumental methods. In chemical analysis the following phenomena accompanying the identification process are observed:

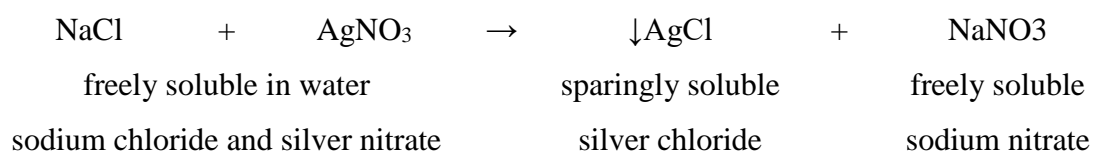
- residue precipitation,
- gas evolution,
- colored coordination complex formation,

Chemical analyses are usually carried out in aqueous solutions, however, identification of a material in its solid state is also possible. That is how the presence of the limestone (CaCO_3) in an aggregate is determined, or how the identification of metal alloys is carried out.

To determine the presence of the limestone in an aggregate, the solution of a mineral acid should be dropped on the material and the characteristic evolution of the CO_2 can be observed. The identification of metal alloys is carried out by pouring concentrated acid on the surface, followed by the use of proper reagents. As a result, a characteristic color on the sample can be observed. This method is also used in the identification of gold alloys.

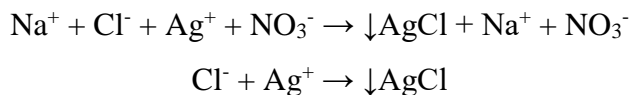
Precipitation reactions

An example of the precipitation reaction can be the identification of chlorine ion with the use of a silver complex (AgNO_3):





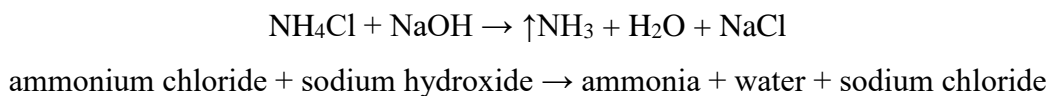
The downwards arrow next to the symbol of the sparingly soluble compound informs about the residue precipitation. Salts, as excellent electrolytes, are fully dissociated in aqueous solutions, which is why the chemical equation can be written down as follows:



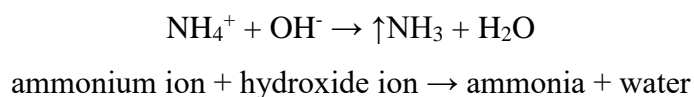
The deletion of the identical ions on both sides of the equation is possible due to the reaction between chloride ions and silver ions, i.e. freely soluble in water chloride complex reacts with soluble silver complex and produces sparingly soluble silver chloride. In practice, turbidity of the solution, or residue precipitation (depending on ion concentration), can be observed after mixing those two clear solutions.

Gas evolution reactions

In this type of chemical reactions the detection of ions is based on, a characteristic gas evolution or an odor change that occurs after the addition of proper reagents. An example of such reaction is ammonia evolution after the addition of a strong base to ammonium salts.

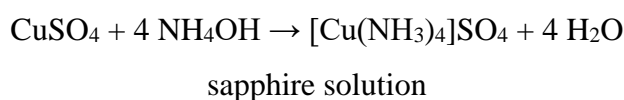


The upwards arrow next to the symbol of ammonia informs about the gas evolution. As with the previous example, the reaction can be written down in a simplified way, i.e.:



Colored coordination complex formation

Ion detection is based on the characteristic color of the precipitated substances after the addition of proper reagents. Both substances are initially colorless but due to the reactions between them a characteristic color is produced. A complex of tetraamminecopper (II) sulfate produced by a combining copper (II) sulfate (VI) and ammonium hydroxide can serve as an example.





Instrumental methods in analytical chemistry

The identification of substances is based on physical and physio-chemical processes that occur during the analysis. The substances can be in both solid and liquid state. Instrumental methods of analysis are based on the behavior of substances, such as:

- absorption or emission spectrum of substances (spectroscopic methods),
- heat of solution, enthalpy of reaction, enthalpy of fusion, melting point, freezing point, boiling point (thermal analysis, calorimetry),
- index of refraction (refractometry),
- structure of substances (X-ray diffraction, electron diffraction, microscopic techniques).

In instrumental methods the qualitative, as well as the quantitative information are produced.

One of the most common methods of detection of cations is a flame test. A flame test is an analytic procedure used in chemistry to detect the presence of certain elements, primarily metal ions, based on each element's characteristic emission spectrum. Table 1 presents some common elements and their corresponding colors.

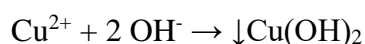
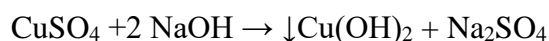
Table 1. Some common elements and their corresponding colors produced in a flame test

Element	Symbol	Flame color
Sodium	Na	Intense Yellow
Potassium	K	Lilac
Calcium	Ca	Brick red
Barium	Ba	Pale / Apple green
Copper	Cu	Green

Selected characteristic reactions of cations

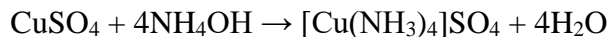
Cu^{2+} - copper (II) cation – blue salts solutions

- HCl – no residue,
- strong bases (NaOH, KOH) - blue gelatinous residue of copper (II) hydroxide $\text{Cu}(\text{OH})_2$,

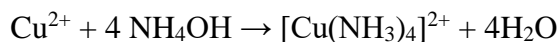




- ammonium hydroxide NH_4OH ; in excess amount, the substance reacts with copper (II) salts, e.g. copper (II) sulfate, and produces a sapphire coordination complex of tetraamminecopper (II) sulfate,



dark blue solution

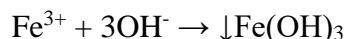
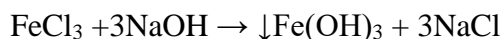


- copper salts in flame test produce green flame.

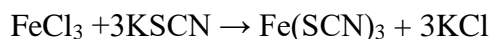
Fe^{3+} - iron (III) cation – yellow salt solution

- HCl – no residue,

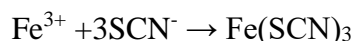
- bases (strong – sodium, potassium; weak – ammonium) – yellow gelatinous residue of iron (III) hydroxide $\text{Fe}(\text{OH})_3$



- potassium or ammonium thiocyanate (also known as rhodanide) produce a deep red coordination complexes of iron (III) thiocyanates – $\text{Fe}(\text{SCN})_3$. In a simplified way, the reaction can be presented as below:



deep red solution



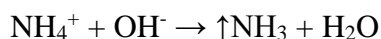
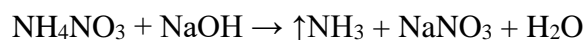
The reaction is very sensitive, and the color intensity of the complex depends on the concentration of the iron cations.

NH_4^+ - ammonium cation – colorless salts solutions

Ammonium salts are among the most water soluble chemical compounds and therefore do not produce any residue with common chemical reagents.

- HCl – no reaction,

- strong bases (sodium, potassium) – evolution of ammonia – NH_3 , a gas with characteristic pungent odor.





PRACTICAL SESSION

DETECTION OF CATIONS BASED ON CHARACTERISTIC REACTIONS

Equipment: glass test tube, test tube stand, pipette

Reagents: NaOH, HCl, H₂SO₄, K₂CrO₄, KI, (NH₄)₂CO₃

PROCEDURE

1. Performance of the characteristic reactions for selected cations according to table 2 (see theoretical background).
2. Performance of cation detection according to Figure 1 (see theoretical background):
 - pour app. 0.5-1.0 cm³ of on unknown cation solution into the test tube;
 - add the same amount of the reagent and observe the reaction;
 - note the ionic equations, visual effects and remarks;
 - perform the flame test for Cu²⁺, Ca²⁺, Ba²⁺, Na⁺, K⁺ ions: put the ion on the platinum wire or glass baguette and place in the flame; observe the color of the flame.

Figure 1 Pattern of cation detection

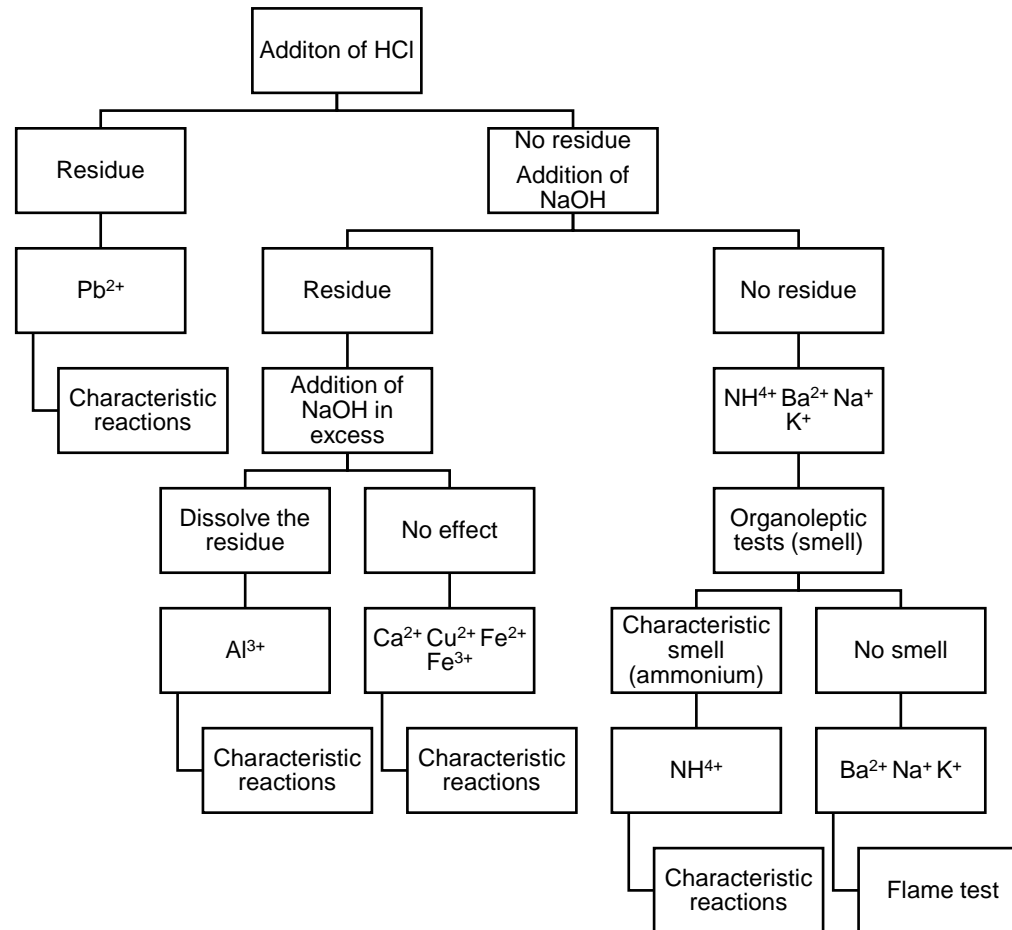




Table 2. Characteristic reactions of selected cations

Reagent Cation	Ion color	HCl (Hydrochloric acid)	NaOH (Sodium hydroxide)	(NH ₄) ₂ CO ₃ (Ammonium carbonate) in NH ₄ Cl i NH ₄ OH environment	H ₂ SO ₄ (Sulfuric acid)	NH ₄ OH (Ammonium hydroxide)	KI (Potassium iodide)	K ₂ CrO ₄ (Potassium (VI) chromate)	NH ₄ SCN (Ammonium thiocyanate)	Flame color
Pb ²⁺ (lead II)	colorless	PbCl ₂ white crystal residue	Pb(OH) ₂ white residue, soluble in excess	PbCO ₃ white residue	PbSO ₄ white residue	Pb(OH) ₂ white residue	PbI ₂ yellow residue	PbCrO ₄ yellow residue	-	-
Cu ²⁺ (copper II)	blue	-	Cu(OH) ₂ blue gelatinous residue	-	-	Cu[(NH ₃) ₄]SO ₄ dark blue-violet solution	-	-	-	green
Fe ³⁺ (iron III)	yellow	-	Fe(OH) ₃ yellow gelatinous residue	-	-	Fe(OH) ₃ yellow gelatinous residue	-	-	Fe(SCN) ₃ deep red solution	-
Al ³⁺ (aluminium)	colorless	-	Al(OH) ₃ white residue, soluble in excess	-	-	Al(OH) ₃ white residue	-	-	-	-
Ca ²⁺ (calcium)	colorless	-	Ca(OH) ₂ white residue, insoluble in excess	CaCO ₃ white crystal residue	-	-	-	-	-	brick red
Ba ²⁺ (barium)	colorless	-	-	BaCO ₃ white residue	BaSO ₄ white residue	-	-	BaCrO ₄ yellow residue	-	-
Mg ²⁺ (magnesium)	colorless	-	Mg(OH) ₂ white residue, insoluble in excess	-	-	Mg(OH) ₂ white residue	-	-	-	-
Na ⁺ (sodium)	colorless	-	-	-	-	-	-	-	-	yellow
K ⁺ (potassium)	colorless	-	-	-	-	-	-	-	-	lilac
NH ₄ ⁺ (ammonium)	colorless	-	↑NH ₃ characteristic smell	-	-	-	-	-	-	-



PRACTICAL SESSION

DETECTION OF CATIONS BASED ON CHARACTERISTIC REACTIONS

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