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LABORATORY WORK No. 35
VOLUMETRIC ANALYSIS - TITRATION

■ **PRINCIPLE:**

Volumetric analysis (titration) is used for determining the content of a specific component in a sample. The essence of volumetric determination is the chemical reaction between a standard solution (of known concentration), which is usually added, and a given volume of a substance to be determined in a titration flask.
From the knowing volume of the standard solution, we can calculate the content of the substance in the titration flask.

When a chemical reaction proceeds quantitatively, just the **equimolar amount of substance** reacts and the **equivalence point** is reached.
We identify this point by:
- using the indicator (subjective method)
- using devices (objective method), eg. potentiometry, conductimetry etc.

Division of volumetric analysis methods:

1) **Neutralization analysis**
a) acidimetry - we titrate an alkali solution with an acid
b) alkalimetry - we titrate an acid solution by an alkali solution to determine the acid

2) **Precipitation methods**
- are based on formation of poorly soluble substances. We include argentometry to this group, a standard solution of $AgNO_3$ is used.

3) **Complexing methods and methods based on formation of less dissociable soluble compounds**
a) chelatometry - uses standard solutions of chelatones
b) mercurimetry - uses $Hg(NO_3)_2$ as titration agent

4) **Oxidation - reduction methods**
a) oxidimetry - standard solution is an oxidizer
b) manganometry - titration agent is $KMnO_4$
c) bichromatometry - titration agent is $K_2Cr_2O_7$
d) iodometry - titration agent is I_2
e) bromatometry - titration agent is $KBrO_3$
f) reductometry - is based on effects of substances having reducing properties, eg.
titanometry - reactions using $TiCl_3$

5) Diazotating methods - are used for determination of certain organic compounds. $NaNO_3$ is volumetric agent (eg. standard solution).

Indicators

Indicators are substances by which we can find out the equivalence point.
According to the usage, we divide indicators into these groups:

α) **Acid-base indicators** that react by changing colour depending on the concentration of H^+.

β) **Oxidation-reduction indicators** are substances having different colors depending on their different oxidation number.

χ) **The precipitation indicators** are used to identify the equivalence point by formation of an insoluble substance, mostly of different color than the product of the titration.

δ) **Chelatometric indicators** are used to indicate titration based on the formation of chelates with the reagent and indicator.

## TASK 1. *DETERMINATION OF THE PURITY OF ANHYDROUS NA_2CO_3 – ACIDIMETRY*

- **CHEMICALS:** anhydrous Na_2CO_3, HCl (c = 0,2 mol/l), methyl orange

- **AIDS:** scales, scoop, weighing boat, 100 ml volumetric flask, burette, titration flask, burner, stand, wire gauze, chemical pliers, pipette

- **PROCEDURE**: We weigh 0.5 g of the sample (anhydrous Na_2CO_3) on the analytical scales. We wash out the sample quantitatively into the volumetric flask of 100 ml. We pipette off 25 ml of the sample into the titration flask and we add 3 drops of methyl orange. We pour HCl (c = 0.2 mol/l) into the burette and titrate to the point when the colour changes from yellow to orange. Then we heat the mixture to the boiling point (we remove the free CO_2) and, after cooling, we titrate the yellow solution again until the orange colouring is reached. We repeat it 3 times. We calculate the arithmetic average of HCl consumption during the titration and substitute it for $V(HCl).10^{-3}$ in the calculation.

The reaction proceeds according to the equation:

$$Na_2CO_3 \; + \; 2HCl \; \rightarrow \; 2NaCl \; + \; CO_2 \; + \; H_2O \qquad Ft = 1/2$$

■ **APPARATUS:**
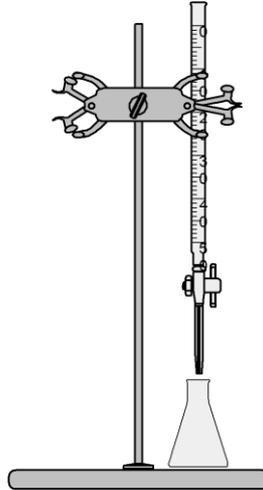
Picture: For titration prepare the stand, clamps, burette filled with HCl (c = 0.2 mol/l), titration flask with the sample.

■ **CALCULATIONS:**

$$w(\%) = \frac{M_{Na_2CO_3} \cdot V_{HCl} \cdot 10 \cdot c_{HCl} \cdot F_t \cdot F_z}{m}$$

$$F_z = 4$$
$$F_t = \frac{1}{2}$$
$$m = 0.5\,g$$

w = mass fraction (content of the substance in %)
Ft = titration faktor
Fz = dilution faktor
 m = weight in grams

■ **CONCLUSION**: Write down the calculated purity of Na_2CO_3 in %.

STUDENT´S SHEET no. 35
VOLUMETRIC ANALYSIS - TITRATION

1.Translate the Czech words into English:

1. laboratorní lžička
2. plynový kahan
3. laboratorní kleště
4. odměrná baňka
5. síťka
6. odměrná analýza
7. redox reakce

8. srážecí reakce
9. acido – bazická reakce
10. chemická rovnice
11. odměrné činidlo
12. standardní roztok
13. bod ekvivalence
14. zásada

2. Name five types of methods in volumetric analysis:

1.
2.
3.
4.
5.

3. Write in full or explain what these words or symbols mean:

HCl…………………………………………

 m…………………………………………...

 Ft…………………………………………...

Fz…………………………………………...

CO_2…………………………………………

V……………………………………………

c……………………………………………

w……………………………………………

3. **Fill in the right definition:**

When the standard solution is an acid, the method is called   ……………..……………..……

When the standard solution is an alkali, the method is called   ……..…………………………...

When the substance changes the oxidation number, the reaction is called   ………..………

 When a big complex is formed during the reaction, we speak about   ……..………..…….

4. Put the jumbled words into the right order:

1. We/ sample/ the /quantitatively /of /100 ml /into /a /wash /volumetric / out/flask.

2. with/ acid/ solution /We /the/ alkali/ titrate/.

3. average /Calculate/ HCl/ the /the/ arithmetic/ titration /of /consumption /of /during.

5. **Describe this picture – name the equipment and mark solutions for acidimetry:**
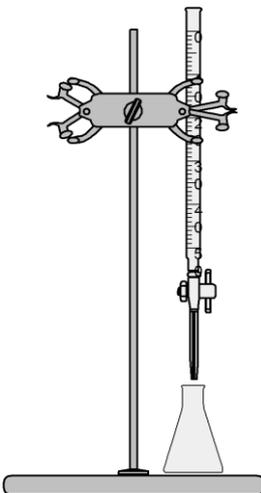
6. **Which indicators are used for acidimetry? Name at least three of them and the changes of their colouring.**