LABORATORY EXPERIMENT 6

TRANSITION METAL DETERMINATION USING COMPLEXOMETRIC TITRATION

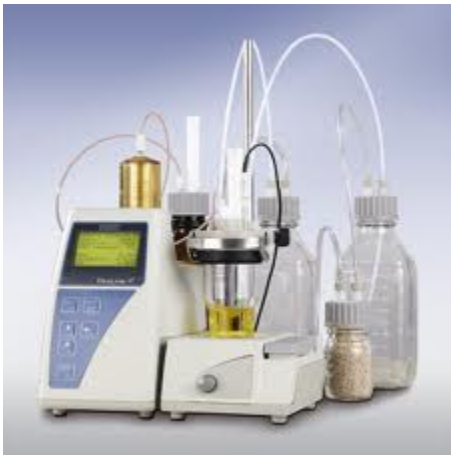
Complexometric titration with EDTA is an easy and convenient way to determine metals in many inorganic reagents and materials. Titration can be performed at various conditions (pH, t^o, masking agents) with a number of color metal-indicators.

$$M^{n+} + EDTA^{4-} \rightarrow MEDTA^{(n-4)}$$

Disodium salt of EDTA, $Na_2H_2EDTA \times 2H_2O$, can be obtained of high purity and can be used as a primary standard unless very high precision is desired.

General procedure is relatively simple: an appropriate quantity of solution is placed in the titration vessel (a beaker or an Erlenmeyer flask), pH is adjusted by addition of an appropriate buffer, necessary amount of suitable indicator is added and solution is titrated with EDTA .

Because of limited solubility of Na_2H_2EDTA in water, very common concentration of EDTA is 0.05 M (0.1 M is more difficult to prepare). In this experiment, we will employ two different types of burette: a basic single-speed titrator and a more versatile variable rate burette. The standard EDTA solution of known concentration will be provided.



The samples to be analyzed are all water soluble inorganic salts of various transition metals: Zinc, Cobalt, Nickel, and Copper. The metal can be easily identified by color of solution.

PROCEDURE:

In each case, start with weighing an exact amount of analyte (*ca* 100 mg) in water, placing it directly in titration vessel . Add 1-2 drops of diluted HCl to prevent hydrolysis.

Co: Add 1 g of hexamethylenetetramine and enough Xylenole Orange indicator to clearly see red-purple color. Heat it to 50-80°C, and titrate with EDTA to yellow color.

Alternatively, Co can be titrated with murexide as indicator (from yellow to violet).

Ni: Enough ammonia is added to convert all Ni(II) ions in a blue $Ni(NH_3)_6^{2+}$, add murexide indicator and titrate to violet color.

Cu: Add 1-2 g of ammonium acetate, several drops of PAR indicator solution, and titrate through purple to green color.

Zn: Add ammonia buffer with pH 9-10 to convert all Zn(II) ions in a colorless $Zn(NH_3)_4^{2+}$, add eriochrome black T indicator and titrate from purple to blue color.

As always, $$m_M = \frac{AW_M \times C_{EDTA} \times V_{EDTA}}{1000}$$

(AW_{Co} = 58.93, AW_{Ni} = 58.69, AW_{Cu} = 63.55, AW_{Zn} = 65.38)

$$\%\%M = (m_M/m_{sample}) \times 100$$

Report your result with appropriate uncertainty.