14A. Complexometric determination of nickel

Information about complexometry as an analytical method, EDTA as widely applied titrant, and the Complexometric indicators can be found in instruction no. 14 ("*The complexometric determination of calcium and magnesium in the same sample*").

Analysis of nickel is important especially in minerals and alloys. Nickel can be determined exactly by gravimetric method, where it is precipitated as dimethylglyoxime complex, see instruction no. 09. However, the gravimetric methods, although very precise, are also time consuming. If one does not need great accuracy but prefers to shorten the time of analysis, an exploit the inclination of Ni to form coordination compounds, also with EDTA. In this analysis, in the conditions applied, copper and cobalt can disturb and should be separated by extraction. Minor amounts of copper can be masked using sodium thiosulfate. Also iron(III), aluminum and manganese ions should be separated before titration or – if in minor amounts – masked using triethanoloamine. Ions of alkaline earth metals and lanthanides should be precipitated using fluoride anions. As seen from the above, for natural samples, containing many different disturbing additives, this analysis is not necessarily so competitive compared to gravimetry…

Determination of nickel using EDTA titrant is carried in alkaline environment. It is better if the environment is slightly alkaline at the beginning of reaction, and becomes more alkaline (from ammonia added). Indicator is murexide, changing color from yellow to violet.

Complexing of nickel(II) by EDTA goes rather slowly, i.e. titration should be performed not too fast.

Procedure

The sample will be a mineral or alloy – discuss with your teacher the method of its dissolution and possible identification (and separation or masking) of admixtures which disturb the analysis. Finally, nickel is transferred to solution and placed in a volume flask.

Attention: First titration is always for reference standard ("witness"). The sample should be overtitrated adding2-3 mL of titrant in excess. The following titrations are conducted until the color becomes identical with that of standard.

1. Add distilled water to the mark, mix carefully the content of the volume flask.
2. Transfer a portion of solution to the Erlenmayer flask, using a pipette. Dilute with distilled water to ca. 100 mL. Add murexide (little pinch), ca. 5 mL 10% NH_4CL and, drop by drop, concentrated ammonia to pH about 8.. pH can be checked using a litmus paper when preparing the standard, in the following analyses it is sufficient to add the same number of drops..
3. Titrate slowly using 0.01 M EDTA. When the color begins to change, add ca. 10 mL of concentrated ammonia and continue titration to complete change of color to violet.
4. Perform points 2-3 three times minimum.

Report

The report should contain the following:

- name and surname of student, date of analysis,
- reactions,
- all obtained results of titrations,
- calculated mass of nickel in grams in the original sample:
  $m_{Ni}=V_{EDTA}\cdot c_{EDTA}\cdot M_{Ni}\cdot W$, where W is commensure*/ of the volume flask with the pipette,
- a commentary, if necessary.

*/ commensure – the ratio of volumes of the volume flask to that of pipette.

Sources:
textbooks
Internet: Wikipedia and http://www.titrations.info/complexometric-titration