

14B. Complexometric determination of bismuth

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 bismuth is important especially in minerals and alloys. Bismuth is a component of many alloys, like the Wood alloy (it melts at temperature as low as +68°C. The Wood alloy contains 50% Bi).

Bismuth can be determined by instrumental method, in particular spectroscopy. However, it reacts easily with EDTA forming stable coordination compounds even in acids, so this method is worth consideration, too. Ions of Fe(III), mercury, thorium and zirconium can disturb this reaction. If only Fe⁺³ are present, one can reduce them to Fe⁺², using ascorbic acid. Lead does not affect this analysis, even if its excess is few thousand times. On the other hand, complexing anions like phosphates, citrates, tartrates, oxalates of fluorides are undesirable.

Complexation reaction of Bi with EDTA should be carried in acid environment, pH 2-3. The indicator is pirocatechol violet (100 mg in 100 mL water), changing color from blue to yellow, or xylenol orange (color changing from red to yellow). Reaction is rather slow, so titration shouldn't be carried 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, bismuth is transferred to solution and placed in a volume flask.

Attention: First titration is always for reference standard ("witness"). The sample should be overtitrated adding 2-3 mL of titrant in excess. The following titrations are conducted until the color becomes identical with that of standard.

1. In the volume flask adjust the pH to about 1 by adding cautiously of dilute ammonia or dilute nitric acid. Add distilled water to the mark, mix carefully the content of the volume flask.
2. Transfer a portion of solution to the Erlenmeyer flask, using a pipette. Dilute with distilled water to ca. 100 mL. Add the indicator (ca. 1 mL).
3. Titrate slowly using 0.01 M EDTA 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 bismuth in grams in the original sample:
$$m_{\text{Ni}} = V_{\text{EDTA}} \cdot C_{\text{EDTA}} \cdot M_{\text{Ni}} \cdot W$$
, 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, In particular „Vogel's textbook of quantitative chemical analysis", G.H. Jeffery, J. Bassett, J. Mendham, R.C. Denney, Longman, Great Britain, 5th edition, 1989.