Aalto-yliopisto
Kemian tekniikan
korkeakoulu

CHEM-A1280 Basic Laboratory Work in Chemistry (2 cr)

LABORATORY WORK INSTRUCTIONS

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Work 3 Determination of iron by spectrophotometric method

Material: volumetric flask 250.00 cm^3 and 100.00 cm^3 10 pcs
Finnpipette 2 pcs (0.5 – 5.0 cm^3, 0.1 – 1.0 cm^3)
glass beaker 250 cm^3
graduated cylinder 100 cm^3
watch glass and glass rod, funnel and qualitative filter paper (Whatman 595½)

Reagents: 0.1 mol/dm^3 hydrochloric acid HCl
1 mol/dm^3 sulfuric acid H_2SO_4
10 % hydroxylamine hydrochloride $NH_2OH{\cdot}HCl$
25 % sodium acetate CH_3COONa
0.5 % 1,10-phenanthroline hydrochloride $C_{12}H_8N_2{\cdot}HCl$
Iron stock solution, 10,00 g/dm^3 Fe (iron(III) nitrate solution)

In this work, the iron content of the iron tablet is determined absorptiometrically (colorimetrically). The iron(II) sulphate in the tablet is dissolved in hydrochloric acid and the amount of Fe^{2+} ions is determined from the solution.

Before the determination, the iron in the stock solution is reduced with hydroxylamine to divalent iron.

$$4\ Fe^{3+}(aq) + 2\ NH_2OH(aq) \rightleftharpoons 4\ Fe^{2+}(aq) + N_2O + 4\ H^+(aq) + H_2O(l)$$

A reducing agent is also added to the sample solutions to ensure that the iron in the solutions is divalent. The colour of the solutions comes from 1,10-phenanthroline, which together with Fe^{2+} ions form an orange-red complex $[Fe(C12H8N2)_3]^{2+}$.

$$Fe^{2+}(aq) + 3\ C_{12}H_8N_2(aq) \rightleftharpoons [Fe(C_{12}H_8N_2)_3]^{2+}$$

The molar absorption coefficient of the complex is about 11,000 $dm^3mol^{-1}cm^{1-}$ at the wavelength of 510 nm. The colour of the complex is stable when the pH of the solution is 2–9. The usual determination range is 0.4–8 ppm.

The accuracy of the determination is affected by the order in which the reagents are added, time, temperature and interfering ions. Interfering ions are silver, bismuth, copper, nickel, cobalt, perchlorate, cyanide, molybdate and tungsten.

Preparation of samples:

1. An iron tablet is weighed with an analytical balance.
2. The tablet is dissolved in 100 cm^3 of 0.1 M hydrochloric acid and the mixture is heated in a fume hood for 15 minutes with occasional stirring.
3. The insoluble residue is separated by filtering the solution into a 250.00 cm^3 volumetric flask. The instruments used for the dissolution and the filter paper are rinsed several times with a small amount of pure water to ensure a quantitative transfer of the sample solution to the volumetric flask.
4. The solution is allowed to cool to room temperature, after which the volumetric flask is filled to the mark with pure water and the solution is mixed thoroughly.
5. From the solution, three parallel samples are prepared as follows:
    1) 1.0 cm^3 solution is pipetted into three 100.00 cm^3 volumetric flasks.
    2) To the volumetric flasks, 4 cm^3 sulphuric acid, 10 cm^3 $NH_2OH{\cdot}HCl$ solution, 5 cm^3 sodium acetate solution and 10 cm^3 1,10-phenantroline solution is added.
    3) The volumetric flasks are filled to the mark with pure water and the solutions are mixed thoroughly.

Preparation of standard solutions:

1. From the iron stock solution (10 000 mg/dm^3), an iron solution with a concentration of 100 mg/dm^3 (ppm) is prepared in a 100.00 cm^3 volumetric flask.
2. From this solution, a series is prepared in 100.00 cm^3 volumetric flasks containing 0.5, 1.0, 2.0, 3.0, 4.0 and 5.0 ppm iron.
3. To each volumetric flask 10 cm^3 1 M sulphuric acid, 10 cm^3 $NH_2OH{\cdot}HCl$ solution, 12 cm^3 sodium acetate solution and 10 cm^3 1,10-phenanthroline solution is added.
4. The volumetric flasks are filled to the mark with pure water and the solutions are mixed thoroughly.
5. The reagent blank is prepared in the same way, but without the addition of iron.

Measurements and calculation of results:

The absorbance of the standard and sample solutions is measured after about 10 minutes of standstill at a wavelength of 510 nm. For the measurements, the reference cuvette contains pure water. Initially, the absorbance reading is zeroed when the reagent blank solution is placed in the sample cuvette. From the measured result of the standard solutions, a standard curve A = f(c) is drawn, and the standard curve equation is calculated.

The Fe^{2+} concentration of the sample solutions (mg/dm^3) is calculated using the standard curve equation. From the results of the parallel sample solutions, the mass of iron (mg) in the tablet and the tablet's iron content in the unit mg/g are calculated. From the parallel results, a mean value is calculated and the calibration error is determined with 95% reliability.

Waste management:

The cuvettes and pipette tips are rinsed with water, after which they are placed in mixed waste. The solutions are collected in the work's waste container for solutions.

Literature:

1. Toivonen, J. ja Yliruokanen, I., *Analyyttisen kemian harjoitustyöt: Kvantitatiivinen analyysi*, 12. korjattu painos, Otatieto Oy, Helsinki 2006, pp. 112 - 116.
2. Harris, D.C., *Quantitative Chemical Analysis*, 5. painos, W.H Freeman and Company, New York, 2000, pp. 863 -864.
3. Marczenko, Z., *Separation and Spectrophotometric Determination of Elements*, 2. painos, Ellis Horwood Limited, Chichester 1986, pp.330 – 333.
4. Jeffery, G.H., Bassett, J., Mendham, J. and Denney, R.C., *Vogel's Textbook of Quantitative Chemical Analysis*, 5. painos, Longman Scientific & Technical, 1989, pp. 690 – 692.