

LABORATORY WORK INSTRUCTIONS

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Work 4 Determination of Iron by Flame AAS Method

Material: volumetric flask 250.00 cm³ and 100.00 cm³ 9 pcs
Finnpipette 2 pcs (0.5 – 5.0 cm³, 0.1 – 1.0 cm³)
glass beaker 250 cm³
graduated cylinder 100 cm³
watch glass and glass rod, funnel and qualitative filter paper (Whatman 595½)
syringe, microfilter and glass beakers 100 cm³ and 50 cm³

Reagents: 0.1 mol/dm³ hydrochloric acid HCl
5 mol/dm³ hydrochloric acid HCl
Iron stock solution, 10,00 g/dm³ Fe (iron(III) nitrate solution)

In this work, the iron content of the iron tablet is determined with the flame AAS method. The iron(II) sulphate in the tablet is dissolved in hydrochloric acid and the amount of Fe ions is determined from the solution.

Preparation of samples:

1. An iron tablet is weighed with an analytical balance.
2. The tablet is dissolved in 100 cm³ 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 cm³ 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. The solution is aspirated into a 30 cm³ syringe and pressed through a microfilter into a 50 cm³ glass beaker.
6. From the microfiltered solution, three parallel samples are prepared as follows:
 - 1) 1.0 cm³ solution is pipetted into three 100.00 cm³ volumetric flasks.
 - 2) To the volumetric flasks, 1.0 cm³ 5 M hydrochloric acid 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 ($10\,000\text{ mg/dm}^3$), 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 1.0, 2.0, 3.0, 4.0 and 4.5 ppm iron.
3. To each volumetric flask 1.0 cm^3 5 M hydrochloric acid 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 at a wavelength of 248.3 nm with an oxidizing air-acetylene flame. The measurement period is $3 \times 4\text{ s} = 12\text{ s}$. The slit width of the monochromator is 0.2 nm. At the beginning of the measurement, the absorbance reading is zeroed with pure water.

The AAS software calculates the mass of iron (mg) in the tablet from the results of the parallel sample solutions. The mass of the tablet is used to calculate the tablet's iron content (mg/g). From the parallel results, a mean value is calculated. In this work, a margin of error for the results is calculated, which the teacher estimated to $\pm 3\%$ for the method.

Waste management:

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