Standardisation of Iron(III) Solutions with Potassium Chlorate

Standardisierung von Eisen(III)-lösungen mit Kaliumchlorat

Standardisierung von Eisen(III) mit Kaliumchlorat

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The following procedure for the standardisation of iron(III) solutions is recommended:

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Mix an aliquot of ferric alum solution with 5 ml of 0.5 N oxalic acid, add sufficient dilute H_2SO_4 to keep the acidity at 1 N in a total of 20 ml and expose to bright sunlight or Philips 120 W high pressure mercury vapour lamp for 15—20 min. Treat the iron(II) formed with 12 M H_2SO_4 to give an acidity of 8—9 M and cool the mixture. Add 5 ml of syrupy phosphoric acid and titrate with 0.1 N $KClO_3$ solution using 0.1 ml of 0.01 M ferroin as indicator till the disappearance of the red colour. The indicator correction is 0.07 ml. The error is $\pm$ 0.3%.

When using a potentiometric method the potential break amounts to 330—380 mV per 0.04 ml of 0.1 N $KClO_3$.

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Rapid Extraction and Direct Spectrophotometric Determination of Iron(III) with Thiothenoyltrifluoroacetone

Schnelle Extraktion und direkte spektralphotometrische Bestimmung von Eisen(III) mit Thiothenoyltrifluoraceton

Best. von Eisen(III); Spektralphotometrie; Extraktion mit Thiothenoyltrifluoraceton

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Thiothenoyltrifluoroacetone (STTA) has been employed for the extraction and direct spectrophotometric determination of iron(III) at trace concentrations at pH 4.8—5.0 in the presence of 0.8 M sodium acetate. A bright red coloured complex was measured spectrophotometrically at 490 nm. At this wavelength the molar absorptivity is 2.45×10^{-4} while sensitivity (Sandell's) is 0.0023 µg/cm². The reproducibility is $\pm$ 1.2%. The extraction is quantitative between pH 4.7—5.2; it started decreasing at pH > 5.4 and is negligible beyond pH 7.0. The extraction is 50% with 0.2 M but is quantitative between 0.8—2.0 M of sodium acetate. No extraction is possible in absence of acetate ion. Similar effect was also observed by others [1].

The verification of Beer's law at 480, 490, 500 nm showed that the system conforms to this law in the concentration range of 0.05—5 µg/ml of iron(III) at 490 nm in the presence of 0.8 M acetate ion. The

complex is stable for at least 48 h, beyond which it is dissociated.

The variation in concentration of the reagent from 0.0025—0.001 M and volume of the reagent (0.001 M STTA-CCl_4) from 2.5—15 ml showed that the extraction is quantitative with 10 ml of 0.001 M of the reagent. The extraction is incomplete with dilute or less volume of the reagent. At high reagent concentration no appreciable change in extraction was observed.

The study of the effect of diveres ions on extraction of iron showed that the alkali and alkaline earth ions do not interfere upto a ratio of 1:150. Cd^{2+}, Mo^{6+}, Ag^+, Be^{2+}, Al^{3+}, SCN^-, CN^-, F^-, SeO_3^{2-} and tartrate are tolerated upto ratio of 1:100. Ions showing strong interferences are Cu^{2+}, Co^{2+}, Ni^{2+}, Bi^{3+}, Pd^{2+}, citrate, ascorbate and EDTA. Tolerance of some of the ions could be improved by tartrate (Pb), phosphate (W), fluoride (U) and cyanide (Hg, Au, Mn).

Procedure. To an aliquot of the sample solution containing about 12 µg of Fe(III) add sodium acetate, such that the concentration is 0.8 M in a total volume of 25 ml. Adjust the pH to 4.8 with 1 M H_2SO_4 or NH_4OH, add 10 ml of 0.001 M STTA solution in carbon tetrachloride and shake for 10 min. Transfer the bright red coloured organic phase into a 10 ml volumetric flask and measure the absorbance at 490 nm against reagent blank. Prepare a calibration curve according to the same procedure.

Reference

1. Goldstein, G., Menis, O., Manning, D. L.: Anal. Chem. **32**, 400 (1960).

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