CHAPTER 4

CONCLUSIONS

The procedures for determination of Fe(II), Fe(III), and phosphate based on flow injection analysis and related techniques were developed.

Determination of iron by FIA using salicylic acid as a color reagent

The method for determination of Fe(III) using salicylic acid, which is cheap and easily available in a drug store, was for the first time proposed by using FI spectrophotometry. The method is simple, fast, precise, accurate and requires only small amounts of sample. It is an economical method suitable for determination of iron in the range 1 - 20 mg l^{-1} with the detection limit of 0.46 mg l^{-1} and the relative standard deviations (n = 3) over this range varied from 1.4-5.4%. The method has been demonstrated for an assay of iron in pharmaceutical preparation samples. The results obtained were in good agreement with the standard USP method.

Determination of Fe(II) and Fe(III) by Capillary Electrophoresis

Attempts were made to develop capillary electrophoresis for simultaneous determination of Fe(II) and Fe(III). Conditions to separate Fe(II) from the Fe(III) peak would be possible, but Fe(III) can be reduced to Fe(II) by PAR during separation period. So the speciation of Fe(II) and Fe(III) cannot be achieved.

Determination of Fe(II) and Fe(III) by voltammetry

Voltammetry was proposed with the same objective as capillary electrophoresis. Pyrophosphate and phosphate were used as complexing ligands and supporting electrolyte for differentiating the reduction peaks of Fe(II) and Fe(III). It was found that both the reagents can separate peak potentials due to Fe(II) and Fe(III). But due to reduction of Fe(III) to Fe(II) giving contribution to the peak due to Fe(II), speciation of Fe(II) and Fe(III) was not achieved either.

Phosphate determination using flow injection analysis with electrochemical detection

Attempts were made for using amperometric detection as a simple FI system to investigate phosphate by reduction of 12-molybdophosphate. It was found that the detection limit was 3.4 μg P l^{-1}. The linearity lined between 50-500 μg P l^{-1}. The relative standard deviations were 1.3% and 9.5%. The method can be applied to salinity seawater samples. This is a major advantages over the FIA spectrophotometry using molybdenum blue method. A low level phosphate can be determined by using an on-line preconcentration system. A detection limit of 0.18 μg P l^{-1} was achieved and a linear calibration of 0.1-10 μg P l^{-1} was obtained.

Novel on-line sulfide removal FI system for Phosphate determination

Interference due to sulfide in phosphate determination could be destroyed by on-line oxidation with potassium permanganate. Potassium permanganate was selected as it is easily available, cheap, less toxic and the excess amounts in the line can be on-line destroyed by ascorbic acid. The method has been demonstrated for the efficiency of sulfide removal from water samples without the oxidation of organic phosphate. The linearity range and the detection limit obtained from this method were 50-1000 μg P l^{-1} and 17.3 μg l^{-1}, respectively. The relative standard deviation ($n = 3$) over this range (50-1000 μg P l^{-1}) varied from 0.3-12.4%. The results obtained by the proposed method were in good agreement with the conventional FI method with off-line sulfide removal by acidification.

Simultaneous determination of phosphate and silicate using stopped FIA

Simultaneous determination of phosphate and silicate in water samples was developed by using a simple laboratory-designed semiautomatic stopped-FI analyzer. The results obtained agreed with the standard method. The method can be applied to water samples containing phosphate and silicate in the range of 0.6-10 mg l^{-1} and 4-15 mg l^{-1}, respectively.