CHAPTER 3

CONCLUSIONS

FIA procedures for the determination of phosphate nitrite nitrate chromium and calcium have been developed.

The fluorescence-quenching method for the determination of orthophosphate was investigated by modifying the one reported by Motomizu[19]. The proposed FIA method was based on the fluorescence quenching of Rhodamine B by phosphomolybdate. The fluorescence intensity of Rhodamine B was found to decrease with the increase in phosphate concentration. It was found to be sensitive and reliable ; the determination range of 0.05-0.1 mg/l, % RSD of 1.3 for 0.08 mg/l PO_4^{3-} (n=12) , and the sample throughput of 36-40 injections/h. Interference effect was negligible, except for Al(III) , Sn(II) and As(V). Suspended solids in the sample were found to interfere in the method because of self absorption effect. The method was applied to the determination of phosphate in natural water samples. The results obtained by the proposed procedure agree with the results obtained by normal colorimetric-FIA method.

For the proposed FIA method of the sequential determination of NO_2^- and NO_3^- , which was based on the colour formation by the diazotization with sulfanilamide after coupling with N-(1-naphthyl)-ethylenediamine , was investigated by using a single colorimetric detector. A copperized Cd-column was used to reduce NO_3^- to NO_2^- before injecting the sample to the system. This method , the determination ranges were 0.01-0.3 mg/l of NO_2^- and 0.05-0.3 mg/l of NO_3^- , % RSD of 1.2 for 0.05 mg/l NO_2^- (n=15) and 7.7 for 0.07 mg/l NO_3^- (n=43) , and the sample throughput was 180 injections/h. The method was applied to the determination of NO_2^- and NO_3^- in a spiked water sample. Recoveries were found to be 93 % for NO_2^- and 100 % for NO_3^-.

FIA sequential determination of Cr(VI) and Cr(III) , based on the complex formation of 1,5-diphenylcarbazide (DPC) / Cr(VI) in an acid solution , was investigated by using a single colorimetric detector. An in-valve C18 column was used for the separation of some interferences by retaining the Cr-DPC complex and eluting with methanol. The cerium(IV) was used to oxidized Cr(III) to Cr(VI). Using the manifold described in the section 2.5.2. 3 curves can be constructed as shown in Figure 2.45. A simultaneous determination of Cr(VI) and Cr(III) in a mixture can be made. The proposed procedure was demonstrated for the spiked drinking water. Recoveries were 100 % and 117 % for Cr(VI) and Cr(III) respectively. Interferences were also studied.

FIA for calcium using Murexide was made by using ethylenediamine buffer of pH = 9.0-9.5. A calibration curve was found to be linear for 10-120 mg/l Ca. Interference by Mg was found when a green LED was used. Calmagite-EDTA solution was also applied for hardness determination. A calibration consisted of two linear range, 10-40 and 40-180 mg/l Ca (25-100 mg/l and 100-450 mg/l as $CaCO_3$). This was applied for the determination of hardness of a water sample.