# CHAPTER 3

# CONCLUSION

## Uranium Determination

The fluorescence spectra of uranium as uranyl ion in aqueous solution show maximum intensity at pH of 8 with an excitation wavelength at 315 nm and an emission wavelength of 495 nm. At pH of 8, when uranium was in potassium dihydrogen phosphate - disodium hydrogen phosphate buffer the optimum emission intensity was obtained at an excitation wavelength of 270 nm and an emission wavelength of 515 nm. Sodium tetraborate can also be used as a pH 8 buffer for uranium giving maximum emission intensity at 495 nm after excitation at 340 nm.

Fluorescence spectra of uranium in sulfuric acid, phosphoric acid and mixtures of these acids agree with those previously reported (65). Uranium in acid phosphate solutions show maximum intensity at pH of 3.5, with an excitation wavelength of 250 nm and an emission wavelength of 515 nm. Dipotassium hydrogen phosphate - phosphoric acid was found to be a suitable buffer solution for uranium at pH 3.5.

A single line FIA manifold was developed from a batch procedure using sodium tetraborate (0.001 M) as carrier stream. The fluorescence intensity was measured at an excitation wavelength of 340 nm and an emission wavelength of 495 nm. GG (a piece of cellophane sheet), PP (a piece of polypropylene (atactic) sheet) and glass filters were also compared for use as emission filters. Glass filter gave the best result and PP performed better than GG. However, GG and PP were applied and gave linear calibration for a single line manifold in the range of 5 to 50 mg/l under optimised conditions.

A two line manifold was applied to the fluorimetric determination of uranium at pH 3.5 with dipotassium hydrogen phosphate - phosphoric acid as the carrier stream. Standards were injected into this stream which was then merged with (0.01 M) sodium hydroxide stream. The excitation wavelength was 250 nm and emission intensity at 495 nm was recorded.

The proposed FIA method was found to be more convenient and more rapid than the fusion technique. Other parameters in the two line manifold (flow rate, mixing coil length, sample volume) should be studied further in order to optimise the conditions. Furthermore, both proposed FIA methods should be developed further to lower the limit of uranium detection.

## Yttrium Determination

For the FIA determination of yttrium as the yttrium-Arsenazo III complex, an in-valve column packed with Amberlite IRC-50 resin was employed to preconcentrate yttrium ion which was sorbed onto the resin and desorbed using hydrochloric acid solution as eluent.

Conditions were optimised in two ways. The first was the conventional way in which the response was obtained by varying one factor while keeping the others at constant levels. The second was the simplex method which used a statistical approach to optimise factors in the system. Both approaches yielded similar optimised conditions.

Single standard calibration can be performed by varying the preconcentration time up to 7 minutes. The linear range was found up to 0.45 microgram of yttrium. Sample solution should have a pH of between 4 and 7. Yttrium ion can be adsorbed onto glass surface : Yttrium solutions of low concentration must therefore be stored in plasticware. Interferences effect were also studied (section 2.3.10). The proposed FIA procedure was applied to the determination of yttrium in tin tailing leachates : the results agreed with those obtained by an ICP-AES method. FIA method which has been developed is convenient and rapid, with economic time and reagent consumption and reduced contamination. The recommended method is suitable for trace yttrium determination.