

## CH 424 Instrumental Analysis Lab

### Fluorometric Determination of Aluminum in Aqueous Sample

(Reproduced with a minor modification from CH 424 Instrumental Analysis Course Manual, spring 1996, written by Jim Peterson)

#### Introduction

An extremely useful and reasonably sensitive fluorometric method for the determination of aluminum is based upon the formation of its complex with 8-hydroxyquinoline (8-quinolinol) and subsequent extraction into solvents such as methylene chloride. The fluorescence does not vary critically with time, pH of extraction, or amount of fluorophore used. Significantly, the presence of iron in amounts equivalent to the aluminum concentration does not seriously interfere with the analysis.

#### Reagents

Prepare accurately a stock solution approximately 100 ppm in aluminum by dissolving potassium aluminum sulfate in water to a final volume of 250 mL. Dilute this solution volumetrically in water to obtain 1 L of a working standard, *ca.* 2 ppm in aluminum. Add 6 mL glacial acetic acid to excess water in a 100-mL volumetric flask, then dissolve 2 g 8-quinolinol in this solution and make up to the mark with water (nominally 2% 8-quinolinol in 1 M acetic acid). Add 45 g of ammonium acetate and 15 mL 28% (w/w) ammonia solution to a 1-L volumetric flask and make up to the mark with water. Methylene chloride (dichloromethane) is required for the solvent extraction step.

#### Procedure

To approximately 100 mL water in a 250-mL separatory funnel add 2 mL of the 8-quinolinol solution and 2 mL of the ammonium acetate buffer. The pH should be  $8.0 \pm 1.5$ . Extract with two 15.0-mL portions of methylene chloride, transferring these to a 50-mL volumetric flask and topping up to the mark with methylene chloride. This constitutes the blank for the fluorometric measurements.

Now prepare a series of standards containing *ca.* 3, 6, 9 and 12  $\mu\text{g}$  of aluminum by adding, respectively 1.5, 3.0, 4.5 and 6.0 mL of the working aluminum standard to the 100 mL water initially in the separatory funnel and then following the same procedure as above. Measure the fluorescence of these solutions at 520 nm, using an excitation wavelength of 365 nm and construct a calibration curve. Subsequently, the best fit to these data should be determined by a least squares regression analysis. **Be careful handling the fluorescence cells--they are expensive.**

Obtain an aqueous aluminum solution of unknown concentration from your instructor. Pipet a 10.0 mL aliquot of this into a 250-mL separatory funnel containing

approximately 100 mL water, then proceed as for the calibration standards. Measure the fluorescence of the sample and determine the concentration of aluminum in the unknown as it was supplied to you. Report your result as ppb and in terms of 95% confidence limits.

### Questions

- (i) Draw the structure of 8-quinolinol and describe the form of its complex with aluminum.
- (ii) What are the principle interferences in determinations of this kind?
- (iii) Man has no known dietary requirement for aluminum, so why might clinical chemists be interested in aluminum analysis?

### References

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