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Comparison of Colorimetric and Ion Chromatographic methods for Aqueous Fluoride Analysis

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Abstract

Fluoride concentrations in drinking water samples were determined by three different analytical techniques, SPADNS Colorimetric method, ion chromatography (IC) and Eriochrome Cyanine R based Palintest® fluoride test using a YSI 9500 Photometer. The present study was done to investigate, whether above methods determine the same or different fluoride species, such as free fluoride, weakly complexed fluoride and total fluoride. Performance characteristics in terms of precision and accuracy have been obtained for the three methods using standard fluoride solutions. Standard deviations of the triplicate analysis for the three methods were less than ±0.312 for all the standard solutions. For 1.0 ppm solution, IC and SPADNS methods produced <5% error and Palintest produced 7% error. In addition to standard solutions, thirty freshwater samples were also analyzed from each method and each reading was triplicated. Statistical analysis of the data by t-test (paired) indicated that there is a significant difference between IC and Palintest readings ($p<0.05$) and IC and SPADNS readings ($p<0.05$) whereas there is no significant difference in Palintest and SPADNS readings ($p>0.05$). Statistical analysis of the data revealed that the observed differences are most likely due to the presence of Ca, Mg and Al ions. In order to determine the effects of Mg and Al ions on the analysis of fluoride, a 0.8ppm fluoride solution was spiked with different amounts (0.5-100.0 ppm for Mg, 0.1-20 ppm for Al) of metal ions and let the solutions to equilibrate to about 24 hours. Equilibrated samples were analyzed by the SPADNS and Palintest. According to the results, increasing amounts of Mg, underestimated the fluoride concentration. There is a 27% and 13% underestimation of fluoride when the Mg concentration is 80.0 ppm for Palintest and SPADNS methods respectively. In a similar way, increasing amounts of Al underestimated the fluoride concentration with a 37% and 30% underestimation for Palintest and SPADNS methods respectively at 20.0 ppm Al. The underestimation of the fluoride concentration in the presence of Mg and Al can be attributed to the formation of strong metal-fluoro complexes, which do not break during the analysis. Acid distillation of the samples to remove the metal ion interferences significantly reduced the error of the analysis. This research suggests that different Fluoride analysis methods measure different pools of Fluoride in same aqueous solution.

Key words: Fluoride, Ion chromatography, SPADNS method, Palintest method