

The use of Commercial test kit reagents for the determination of nitrate in sea water.

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Abstract

Nitrate is traditionally measured via sensitive yet cumbersome Copper-Cadmium reduction column and colorimetric measurement. Analysis by Nitrate Ion Selective Electrode (ISE) coupled with flow injection analysis can provide a rapid and automatable procedure for but unfortunately Chloride ions interfere significantly⁴. Precipitation of Chloride as insoluble Silver Chloride has been used to remove Chloride ions from solution enabling the use in brackish water samples. This approach is very expensive. This work reports the investigation of using chemical reactant solutions from commercial test kits for aquaria, where the amount of heavy metal waste eliminated. A method for sea water was developed using spectrometry in the visible region providing better precision than the test kit technique. The advantages of FIA^{1,2,3} with small sample sizes and rapid sample through put and automation is being investigated and preliminary results for the FIA method are reported.

Introduction

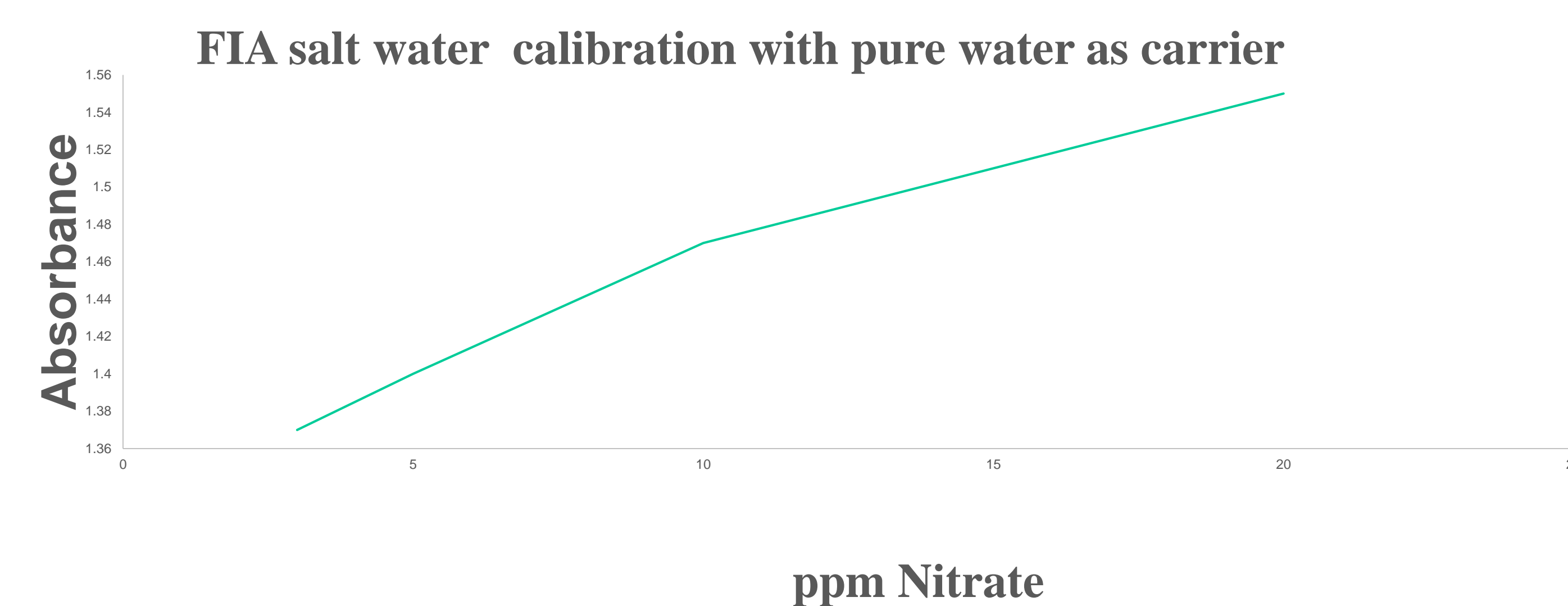
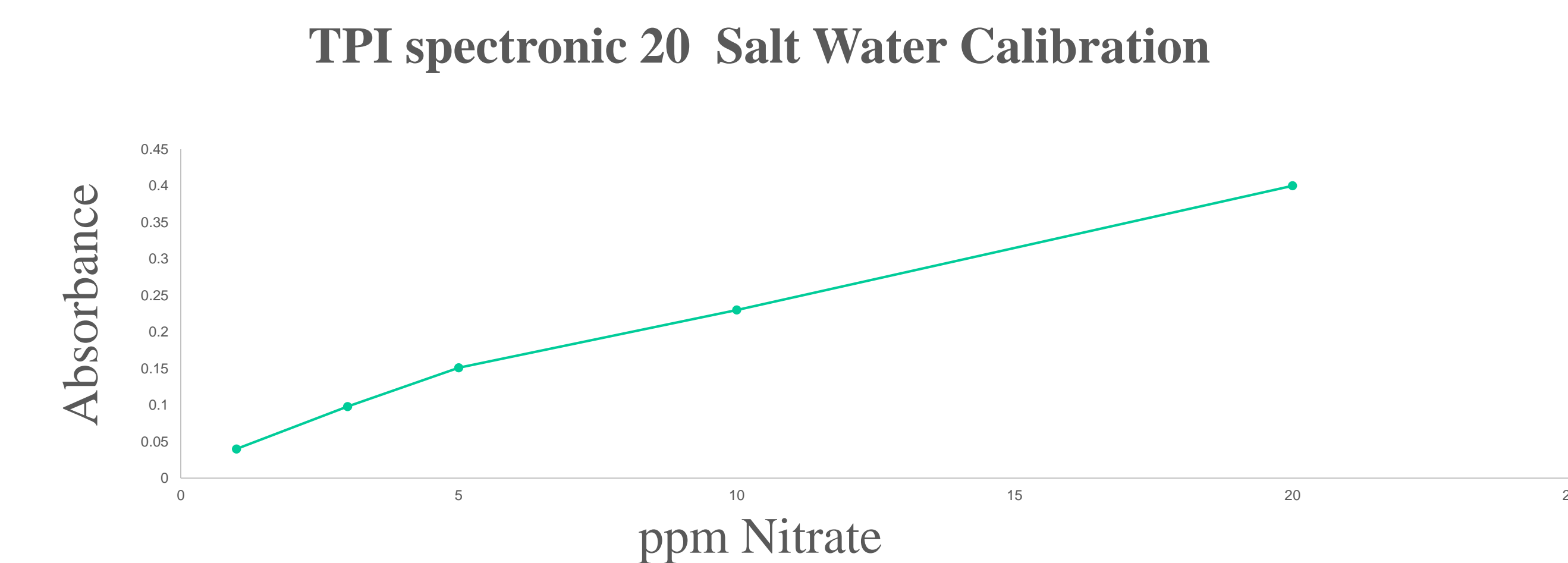
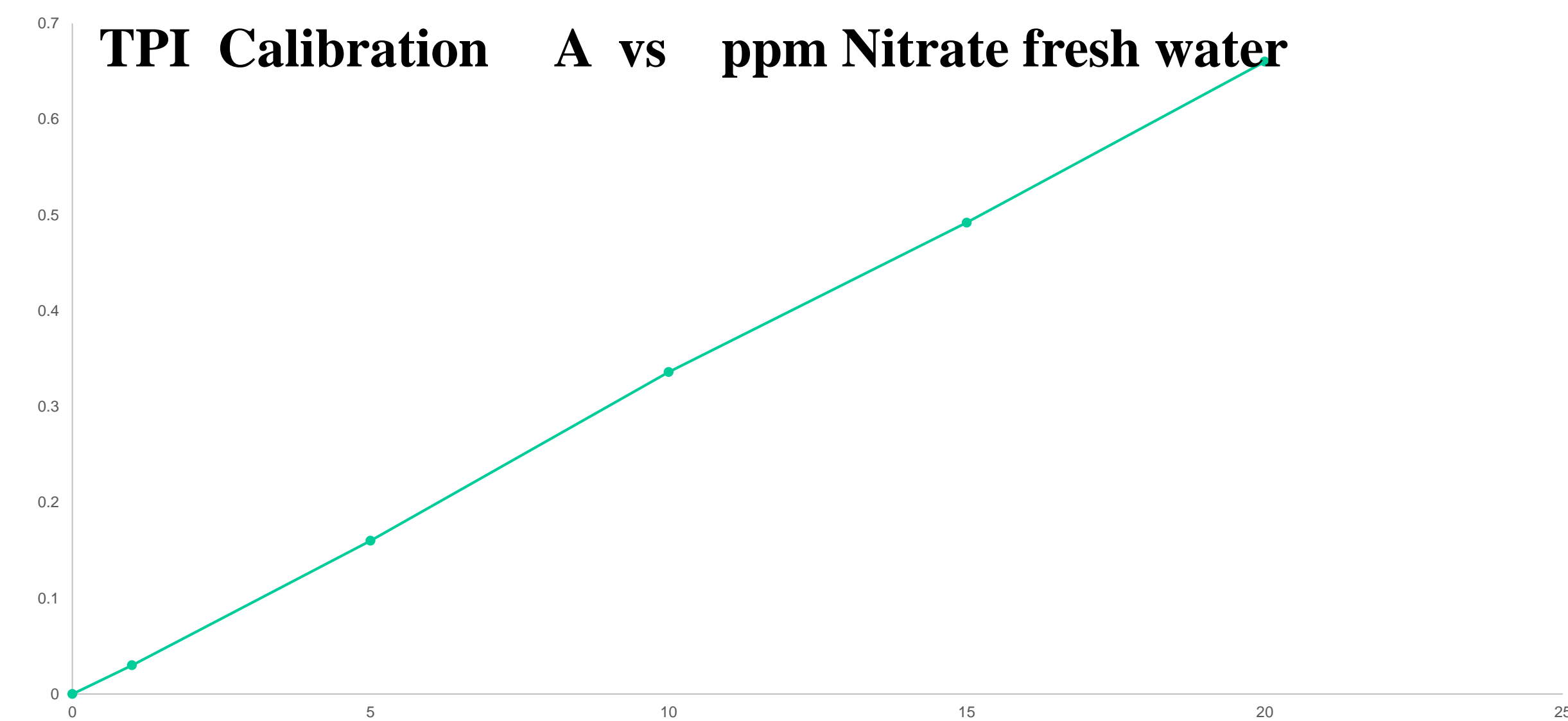
Nitrate is initially reduced to nitrite before developing the color Nitrite is commonly measured using the colorimetric measurement via the azo dye. In this study, replacement of the environmentally hazardous cadmium for the reduction with a commercial test kit reagents have been investigated for fresh water samples.. The test kit provides an estimate of nitrate levels via a color strip comparison after 5 minutes of mixing with the sample and two unidentified test kit reagents. An instrumental measurement was adapted to the test kit procedure which produce reasonable fast measurement which were accurate and precise. Flow Injection Analysis (FIA) can provide a multichannel flow approach in developing a continuous and automated method for monitoring nitrate.

Experimental

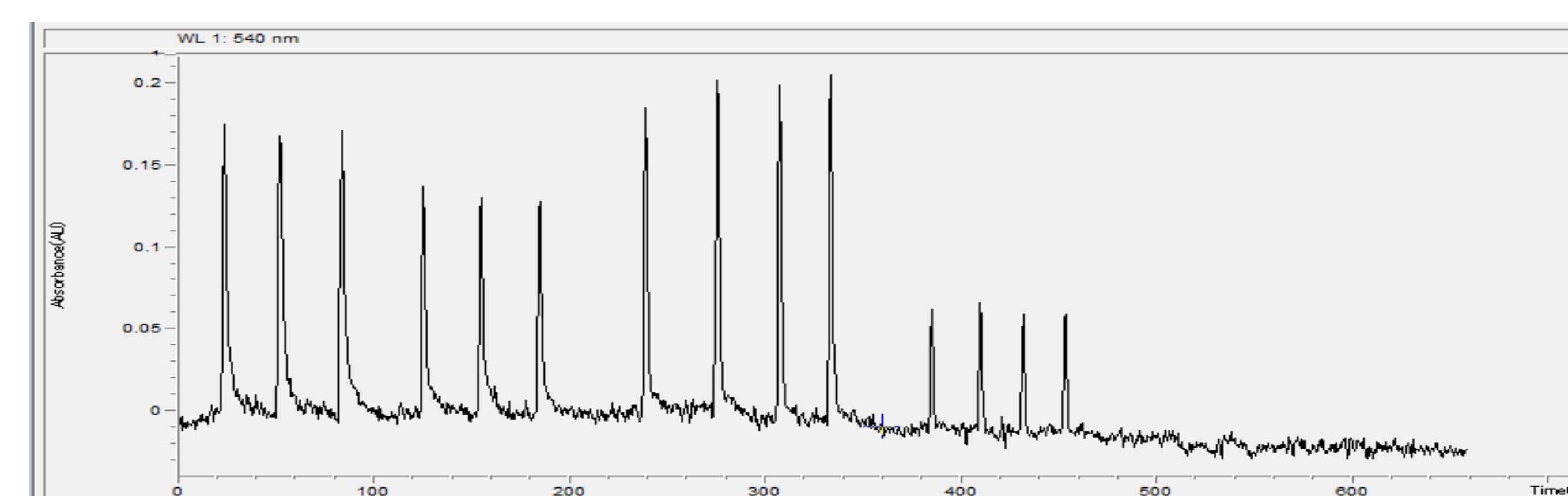
TPI test kits and included reagents were employed for colorimetric measurement at 540 nm detection wavelength in a Spectronic 20 visible spectrometer and in the FIA experiments, with Cary 8454 UV-VIS spectrometer with flow through cell as the detector. 60 μ L injection volumes and a flow rate of 1.25 mL per minute were employed via a Gilson MP3 peristaltic pump and a Rheodyne injection valve in a single line FIA.

The color development reaction proceed for 5 minutes to produce a stable color prior to measurement. In the FIA mode the color reactions were done in the loading syringe of the FIA system. Both water and saltwater blank solutions were investigated as the carrier solution.

Salt water based standards were all made with a background of 3.5 % Sodium Chloride Sigma-Aldrich ACS. Potassium Nitrate, Sigma-Aldrich ACS was used in ultra pure water from a Barnstead, E-pure; 17.9 megaohms for the standard solutions in the range 0 to 20 ppm nitrate in both fresh water and salt water. 3.5 m/m% sodium chloride was chosen as an approximation of sea water ³.



Typical FIA peaks



Results and Discussion

The aim of the research group was to develop a fast and where possible an automatable onsite method for nitrate in water without the waste problems associated with cadmium reduction. The commercial TPI test kit provides reagents that meet this goal and shows excellent results when used in visible spectroscopy.

The central panel of the poster shows the calibration plots for both fresh water and salt water solutions. The salt water plots shown are firstly for the manual test kit method employing a Spectronic 20 spectrometer for visible wavelength measurements and secondly for the flow injection measurement mode. Typical FIA peaks for this work are also shown.

The manual method shows good linearity with $R^2 = 0.99$ for fresh water and $R^2 = 0.99$ for salt water. The FIA plot also exhibits good linearity with $R^2 = 0.98$ and a relative standard deviation of 0.8% for triplicate injections.

Conclusion

The TPI test kit with a Spec 20 as a lab method gave excellent results with integer discrimination in the low ppm range for both fresh and salt water. The measurements made in the FIA mode gave comparable results but also with the ability to provide relative standard deviation data and the possibility a developing an automated method where the addition of the two TPI test Kit reagents could be added to the samples online via merging streams and the use of appropriate mixing coils before detection at 540 nm. Future work will involve the investigation of the online mixing techniques and the automation of test sample solutions directly from aquariums.

References

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