

# Application of the Diacetylmonoxime Method to the Spectrophotometric Elemental Analysis System (SEAS) for in situ Urea Determination

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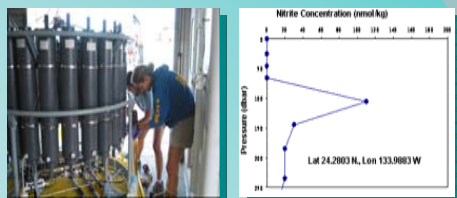
**Abstract:** The development of *in situ* chemical sensors for use in oceanographic research provides distinct advantages to shipboard and laboratory analysis such as a reduction in sample contamination, more rapid data acquisition, and increased sampling. The Spectrophotometric Elemental Analysis System (SEAS) has been developed to study subnanomolar analyte concentrations in seawater due to the incorporation of a liquid core waveguide as an optical cell.

Previous studies have proven SEAS ability to generate detailed nitrite and nitrate depth profiles throughout the water column with a depth capacity up to 500m. The diacetylmonoxime colorimetric method used to study urea concentration is being applied to the SEAS system. The optimization of this technique will provide more accurate and efficient means of studying dynamics of the oceanic nitrogen cycle.

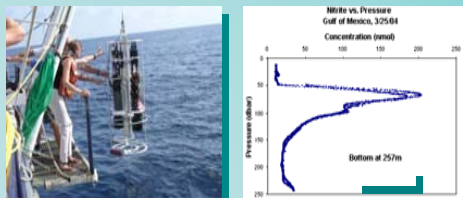
## Introduction:

The fully autonomous Spectrophotometric Elemental Analysis System (SEAS) offers an adaptable way to study chemical components of the marine system. The instrument design allows for the application of virtually any analytical method which involves colorimetric analysis. The system can also be used to detect fluorescent species. The incorporation of a liquid core waveguide (LCW) as the optical cell, further enhances its abilities by allowing the detection of analytes down to subnanomolar concentrations. This quality is in accordance with Beer's Law, in which longer path-lengths (up to 10m) enable detection of smaller concentrations.

The SEAS system is currently being modified for the determination of urea in the water column. Urea, an organic form of nitrogen, is excreted by fish, marine mammals, and zooplankton through metabolic processes. Urea also serves as a nutrient source for certain types of phytoplankton communities and is usually found in concentrations below 0.5  $\mu\text{M}$  in oligotrophic waters. In coastal areas, however, as a result of an increase in urea-based fertilizers, the concentrations are much higher and more variable than those in the open ocean. This increased usage of urea-based fertilizers warrants an efficient way to monitor these concentrations in order to assess the impact that anthropogenic inputs of urea has on coastal communities.

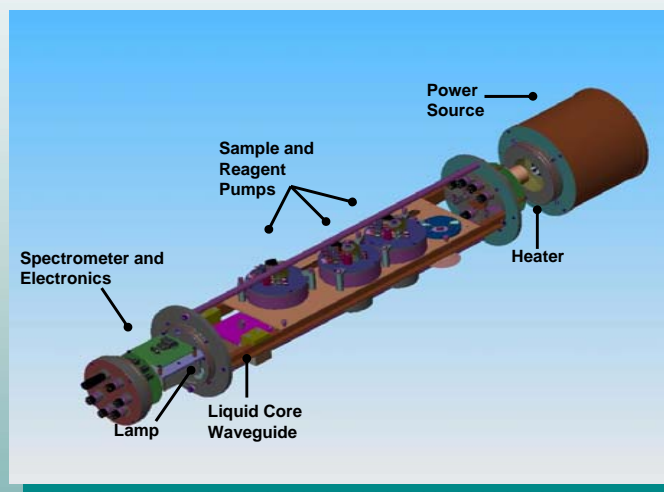


**Fig.1** Traditional methods using rosettes to collect water from different depths of the water column can be cumbersome. Each sample requires careful storage and time consuming laboratory analysis.



**Fig.2** In addition to drastically reducing the size of equipment needed to acquire a nutrient profile and the time required to process each sample, SEAS fully elucidates the picture of how analyte concentrations vary down the water column.

## Spectrophotometric Analysis System (SEAS)

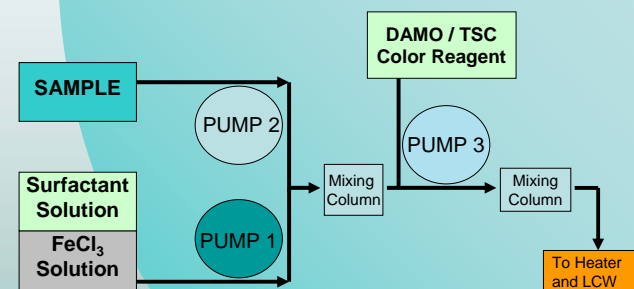


$$\text{Beer's Law: } A = \log(P_o / P) = e \cdot l \cdot c$$

## Current Applications

- Measurement of pH and  $\text{CO}_2$  fugacity
- Measurement of nitrogen and phosphorus species
- Trace metal analysis
- Total inorganic carbon

The system can also be modified to detect fluorescent species.



**Fig. 3** Schematic for urea experimental set-up on SEAS.

**Method:** In order to detect the presence of urea, standard spectrophotometric procedures are used. All reagents and standards are prepared with Milli Q water. Hexadecyl-trimethyl-ammonium bromide (HTAB) is being used as the wetting agent.

The surfactant solution and the iron chloride acidic solution are mixed prior to analysis and are then mixed with the sample in a 3:2 ratio (iron chloride solution: sample). After mixing the sample and surfactant, the color reagent is introduced into the solution. The solution is then delivered to the heater set around 90 °C. The spectrometer is set to a wavelength of 520nm.

**RESULTS:** Optimization of the method is progress. Adjustments are being made to parameters such as flow rates, heater setting, and tubing size.

## References:

- Callahan, *Environmental Science and Technology* (2004) 2: 587 – 593.  
Steimle, E. T., *Marine Chemistry* 77 (2002) 255-262  
Cozzi, S., *Marine Biology* (2004) 145: 843 – 848.