APPENDIX J

EXCERPT

University of California, Santa Barbara
Marine Science Institute - Analytical Laboratory
QUALITY ASSURANCE MANUAL

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University of California, Santa Barbara Marine Science Institute - Analytical Laboratory
QUALITY ASSURANCE MANUAL Revision Date: 5/31/2007

This is a summary of the Seawater micronutrient analysis (often referred to simply as nutrients or Flow Injection Analysis or FIA), taken from the UCSB and MSI Quality Assurance Manual, 2007 revision.

DETAILED ANALYTICAL PROCEDURES for Nutrient Analyses

Nutrient analyses are carried out with a Lachat Instruments (division of Zelweger Analytics) Model QuikChem 8000 Flow Injection Analyzer. This system is equipped for the simultaneous analysis of up to five nutrient species in aqueous samples. The autosampler holds racks which accommodate up to 120 sample tubes plus additional locations for blanks, standard solutions, and control samples.

- Nitrate + Nitrite
- Nitrite
- Ammonium/Ammonia
- Phosphate (ortho)

Instrument Description

Flow injection analysis is a continuous-flow technique for automated wet-chemical analysis, similar in overall operation to AutoAnalyzer instruments. In this system, solutions are continuously pumped, in appropriate proportions, into a reaction manifold, where they combine at the proper points and react to form detectable derivatives with the sample analyte. FIA differs from the more conventional bubble-segmented technique used by AutoAnalyzers in that (a) no bubble segmentation is used, (b) the fluid conduits are laminar flow channels, and (c) the sample is injected as a discrete plug into the carrier stream in order to effect the production of the detectable species. The output from the FIA detectors, which are simple colorimeters, is a peak-shaped signal corresponding to passage of the injected solution (now mixed with other solutions and having generated the detectable species) through the detector flow-cell. The height and area of the peak are proportional to the analyte concentration. Peaks are measured against baseline output for every injection.

A sample is aspirated from its sample tube in the autosampler racks and pumped through the sample loops in the injection valves - each analytical channel is equipped with a separate injection valve. Timing parameters are set to allow complete filling of the valves of all channels being utilized. Once the loop is filled, the sample is injected – injections for different channels may be staggered according to the time required to fill each loop. A complete analysis cycle takes 40 to 80 seconds, depending on which channels are being used.

Batch-Run Procedure

Several tasks must be performed in preparation for nutrients analysis. For highest efficiency, they should be performed in the sequence given. Once the preliminary tasks are completed, the sample run is initiated. For more details regarding the preliminary tasks consult the 2007 Quality Assurance Manual.

Sample Preparation

While the calibration standards are being run, prepare the samples for analysis. Remove samples from freezer and place in heating bath. Set timer to 40 minutes. Fill out the Sample Data Sheet with sample IDs in the order the samples will be run. When the calibration run is finished, open a new tray table and enter the sample ID information, including QC samples. Open the Data Quality Management (DQM) file and assure that control sample, check standard, and spike sample concentrations are correct. At the end of the 40 minutes, remove the samples from the heating bath, rinse the containers briefly with

DI water and place them in the dark to cool. Place the next group of samples in the heating bath, if appropriate. Set the timer for 40 minutes for both cooling samples and heating samples.

Second Calibration Run

If there were any problems with the first calibration run, or if it has been more than one hour since the run, perform another calibration run. Make sure there is sufficient standard solution in each of the sample tubes, select the appropriate tray table, and start the run.

Calibration Curve Check

When the calibration run is finished, examine the generated calibration curves. Make adjustments to peak-integration timing and other parameters as necessary to optimize the analysis conditions and the calibration curves. Print out the final calibration curves.

Analyze Samples

After the thawed samples have cooled, mix each sample thoroughly by inverting all containers at least five times, transfer them into sample tubes and place them in the autosampler racks. When all analysis conditions have been optimized, calibrations are acceptable, and sample data has been entered in the tray table, the batch run may be started. Check that the Auto DQM Schedule is correct, then choose Run Tray from the menu and assign the data file name. Sample-batch data file names also include the number of active channels, but use the first four letters of the investigator's name at the beginning and a 3-digit number corresponding to the sequential sample batch number run on the instrument at the end. Thus, a run analyzing only nitrate and phosphate for Clinton, for example, might have the file name Clin2150, where 150 represents the 150^{th} sample batch run on the FIA system (since installation). Start the run. Monitor the initial check standard and control sample results displayed on the computer screen as described in Section 5.2.3, Calibration Checks; be ready to abort the run if any problems appear. Continue to monitor the run, with special attention to the periodic QC samples, until the run is completed. Work up the data as described in Section 7.2.2, FIA Data.