TRACE ELEMENT DETERMINATION BY PLASMA EMISSION SPECTROMETRY

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1. PURPOSE

The document provides a detailed procedure for the analysis of water samples by DC plasma emission spectrometry.

2. SCOPE

This procedure covers major, minor, and trace metal analysis by DC plasma emission spectroscopy of water samples used in chemical studies for the NNWSI project. The personnel covered by this procedure are those who perform the analyses of the samples.

3. PROCEDURE

Stepwise operating instructions for operating the Spectrametrics, Inc. DC plasma emission spectrometer may be found in the instrument manuals located in Room 314, Bldg. RC-1, TA-48. These manuals cover the areas of initial start-up, loading of standard data, and necessary maintenance.

The following stepwise procedure is to be implemented after the instrument has teen put into operation following the manufacturer's instructions.

1) Prepare an appropriate multi-element standard according to the procedure listed in TWS-CNC-11-11/78-154 for the preparation of working standards. This will be referred to as the "high standard". The concentration values for the elements in the standard shall be of the same order of magnitude as the expected values in the sample. If the sample values are completely unknown, then prepare a preliminary standard with 10 µg/mL of each of the metals to be analyzed for.
2) Set the mode switch to integrate, the time switch to five seconds, and the repeat switch to the desired number of replications.
3) Aspirate the high standard into the plasma and press the auto-range button.. If the status light comes on, press reset, determine what status messages are

being displayed, and take the appropriate action of either raising or lowering photomultiplier tube voltages.

4. If the photomultiplier tube voltages were changed, press auto-range again; if they were not, press high standard. If no status messages are displayed, allow the instrument to finish the high standard calibration routine.

5) Aspirate a blank solution, consisting of at least 17MΩ-cm water acidified with high purity nitric acid to 1% by volume, and press the low standard button.

6) Set the repeat switch to nine and the diagnostic toggle to the up position.

7) While aspirating the blank, press the sample button. The instrument will output measured values for each of the nine repeats, the mean value, and the standard deviation for each active channel. The limit of detection for each active channel is defined as twice the standard deviation of the blank. The output will also show raw data used by the instrument to calculate the results. This data should be checked at this time to ascertain that values for the high and low standards were properly input for all channels.

8) The limit of detection data should be compared to similar data produced in previous analytical runs. The limits of detection should be reproducible to within a factor of two from run to run under similar conditions. If the limits of detection are two times or more higher than those from previous runs, this indicates that the analytical system is not performing adequately and that proper remedial action must be taken. If any instrument readjustment or maintenance is carried out at this time, the calibration procedure must be repeated starting at step one.

9) After running the limit of detection and obtaining suitable results, update the low standard.
10) Change the repeat switch to indicate how many repeats are desired during analytical runs and disable the diagnostic mode.
11) Aspirate the high standard and press the sample button. When the measurement is finished, update the high standard.
12) Aspirate a sample and press the sample button.
13) After the data have been transferred to either the printer or the computer, the next sample may be run.
14) Run high and low standards at least every ten samples and update the calibration data.

4. QUALITY ASSURANCE

4.1 All procedures shall be carried out by qualified, certified staff members, or by technicians working under their supervision.

4.2 Equipment shall be calibrated by the operator as described in section 3 above.

4.3 Data shall be entered into the NNWSI central computer facility data file as described in memo TWS-INC7-2/83-1. Each data set includes the initials of the operator who performed the analysis.