1.0 PURPOSE

1.1 This establishes a method to be used in assaying the amount of sodium (Na) in aqueous samples. In particular, this method will be used to test radioactive samples in the radiochemistry hoods.

2.0 APPLICATION

2.1 This method covers the determination of dissolved sodium in decontaminated supernatant.

3.0 DISCUSSION

3.1 An ion selective sodium electrode allows sodium in aqueous samples to be measured quickly, simply, accurately, and economically.

3.2 The sodium electrode that will be used in this method houses both a sodium-sensing electrode and a reference electrode in one body.

3.3 Interferences

Lithium, potassium, rubidium, ammonia, silver, and thallium are all possible sources of interference if found in levels greater than ten times the concentration of sodium.

4.0 REFERENCES

4.1 Orion Ion Selective Sodium Electrode Manual.
Part II

5.0 EQUIPMENT

NOTE: All calibrated equipment is controlled per ACP 7.1.

5.1 Digital pH/mV meter
5.2 Ion Selective Sodium Electrode
5.3 Magnetic stirrer
5.4 Assorted glassware and plasticware

6.0 REAGENTS AND STANDARDS

NOTE: All reagents and solutions are prepared per ACP 8.1 and have a shelf life of one year unless otherwise stated.

6.1 ASTM type II water or equivalent.
6.2 Sodium Ionic Strength Adjustment Buffer (ISA) - 4M NH₄Cl and 4M NH₄OH. (This may be purchased as a solution or made: 21.4 grams NH₄Cl and 14.0 mL NH₄OH diluted to 100 mL.)
6.3 Sodium Standard Solution. (270.6 g NaNO₃ diluted to 1000 mL = 100,000 ppm)

7.0 SAFETY PRECAUTIONS

7.1 Follow standard laboratory safety procedures (ACP 7.2).

8.0 RECORDS

8.1 All measurement data and sample identification shall be recorded on the work sheet (Attachment A). The final result shall be recorded on the Analytical Request Sheet (ACP 5.1).

9.0 CALIBRATION AND CONTROL

9.1 Microprocessor ionanalyzer or pH meter - refer to the manufacturer's instruction manual for proper operation of the microprocessor ionanalyzer. Prepare calibration curves using a minimum of three standard solutions bracketing the expected concentration of the sample. The usual concentrations will be 10,000, 1,000, 100, 10 and 1 ppm Na.

9.1.1 Treat the standards as directed in 8.1 and measure the potential of each standard and record the millivolts or in parts per million read directly from the instrument. The standards and the sample must be at the same temperature.
9.1.2 Using Realm go through supervisor, worksheets, to calibration curve for SI. Enter MV readings for calibration standards to generate a curve. The computer will automatically calculate the equation of your calibration curve. As an alternative, (in case computer system is down), using semilogrithmic graph paper, plot the concentration of sodium in milligrams per litre on the log axis against the corresponding electrode potential, in millivolts on the linear axis when reading in millivolts.

9.1.3 Check the calibration curve every time when analyzing a set of samples.

9.1.4 For standards below 10 ppm, refer to Reference 4.1

9.2 A QC shall be ran to assure proper calibration.

9.3 The QC shall be in the range of the expected sample results. A QC shall be run with each set of samples. The QC may also be run every three hours as a check on the calibration curve.

9.4 A log book will contain the results of all QC samples per ACP 8.2; a divergence of greater than two standard deviations will require a retest of the spike. Two successive results greater than two standard deviations will require a review of the QC and/or the method by the Analytical Chemistry Manager.

10.0 PROCEDURE

10.1 Sample Treatment

10.1.1 Transfer 50 mL of the sample (or an adequate amount, depending on sample volume given to work with) to a plastic beaker. The sample temperature must be the same as that of the standards used in calibration.

10.1.2 Add the stirring bar and mix on the magnetic stirrer. Do not mix so rapidly that air bubbles are drawn into the solution.

10.1.3 Check level of inner filling solution and fill if necessary.

10.1.4 Immerse the electrode into the sample making sure sample volume is above collar of electrode.

10.1.5 Add (proper amount of) sodium ionic strength adjustment buffer, so that the ratio of buffer to sample is (1:10) (i.e., 50 mL sample, add 5 mL buffer).

10.1.6 Check the pH of the sample with pH paper; the pH must be greater than 6. If less than 5 ppm Na, pH must be greater than 9.
10.1.7 When the electrode comes to equilibrium, measure the electrode potential of the sodium concentration in millivolts.

10.2 Sample Measurement

10.2.1 Determine the sodium concentration by means of an ion analyzer.

10.2.2 Record the observed potential in millivolts, and convert to milligrams per litre of sodium by means of the calibration curve.

11.0 CALCULATIONS

N/A

12.0 ATTACHMENTS

Attachment A - Sodium Electrode Work sheet.
ATTACHMENT A

SODIUM ELECTRODE WORK SHEET

SAMPLE NAME ___________________________ LOG NUMBER ___________________________

SPECIAL INSTRUCTIONS __________________________________________________________

________________________________________

INSTRUMENT USED: ______________________________________________________________

CALIBRATION CURVE EQUATION = _______________________________________________________________________

<table>
<thead>
<tr>
<th>Standards</th>
<th>Conc</th>
<th>MV reading</th>
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<tbody>
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<td></td>
</tr>
<tr>
<td>Std #2</td>
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<tr>
<td>Std #3</td>
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<table>
<thead>
<tr>
<th>Sample ID</th>
<th>QC</th>
<th>Dilution Factor</th>
<th>MV Reading</th>
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</table>

> [Calculated Conc. (ppm)]

* To convert from ppm to wt. %: wt. % = Calculated Concentration (ppm) / Density of Sample x 10,000

ANALYST ___________________________ DATE ___________________________

APPROVED ___________________________ DATE ___________________________

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