Nitrate Measurement Protocols

Basic theory: The nitrate ion selective electrode is a combination electrode with a positive and a negative half cell. The negative half cell is bathed in a fixed level of nitrate (negatively charged) solution behind a membrane which is selectively sensitive to nitrate ions. The positive half cell is bathed in ammonium ions from the ammonium sulfate gel solution which is dispensed by depressing the button on the top of the electrode body. The nitrate level is read by determining a millivolt potential difference between the inside and outside of the nitrate-sensitive membrane. We set that millivolt potential equal to a given ppm concentration by placing the electrode in a known nitrate standard, and then setting the meter's millivolt reading equal to the concentration in ppm. (example: 128.4 mV may be the reading the meter gives when placed in a solution of 1ppm nitrate) Between any two millivolt/concentration readings, the meter interpolates a straight line. At very low concentrations of nitrate (<3ppm), the millivolt differences are actually a curve. Low level calibration is a separate procedure from those outlined below.

Dosatron Measurements – 1, 6, 50, 55ppm nitrate

A. Calibrate at 1, 5, 10, 50, 70 ppm

Materials List:
nitrate concentrate (Hach standards10, 100 ppm)
liquid ISA (ionic strength adjuster)
10ml volumetric flasks (one clean for 1, 5, 50, 70ppm standards)
.1-5ml repipettor + tips
9 25-40ml beakers, 9 small stir bars, all clean
stir plates
kimwipes
double deionized (de-I) water (500ml +)
fine-tipped washbottle containing double de-I water
re-pipettor + clean tips
disposable gloves
paper towels
prop for nitrate electrode
meter to read electrode
data sheets/ scribble paper + writing tool
watch or timer

1. In the hour before calibration, the electrode should be conditioned in 100ppm nitrate solution, without ISA in it.

2. Set up solutions for calibrating the electrode (1ppm, 5ppm, 10ppm, 50ppm, 70ppm). Using volumetric containers as needed measure 10ml.of each solution into a marked tiny beaker; add 10ml of ISA (ionic strength adjuster). Set a rinsed and dried stir bar into each beaker and turn on gentle stirring.

Note: Yes, you now have not 1ppm, but .5ppm; not 5ppm but 2.5ppm, not 10ppm but 5ppm, 25ppm, not 50ppm, 35, not 70. The samples to be measured will be diluted with ionic strength adjuster (ISA) in the same fashion, so the scale of measurement is the same for both calibration standards and samples. If a dry form of ISA is to be used, the solutions will be set up differently.

3. Rinse the electrode with double de-I water & gently pat dry with fresh kimwipe. Set electrode into first (lowest level) calibration solution; press the gel dispenser on electrode top

4. Clear previous calibrations on meter:
a. press "standardize" on meter
b. press "clear"
c. press "standardize" again
d. choose "ppm"
e. key in the "1.0"ppm dilution; press "enter" key.
f. read instructions to see that you are ready to set the meter to accept this calibration . If ready and at least **10 minutes** have elapsed since you put the probe into the calibration solution, press "enter". Note the millivolt reading quickly before the meter accepts this calibration.

5. After the meter accepts this calibration, remove the electrode from the calibration solution; rinse with double de-I water; gently blot dry.

6. Place the electrode into the next highest calibration solution (5ppm), dispense gel, allow to equilibrate for 5 minutes. Press "standardize", key in "5.0" ppm, press enter, note mV reading. After the meter accepts this calibration point, proceed.

7. Repeat step 6 until the 4 calibration points are established. **This calibration should be reliable for about an hour.**

B. Measure sample nitrate

Collecting materials List:
Clean carrying tub
Clean labeled sample bottles
Watch or timing device
Data Sheets
Combination to Chew lab
liquid ISA

1. Collect representative samples of solutions (1, 6, 50, 55ppm + tap water): Run the solutions out of sampling tube into waste bucket for at least 1 minute; rinse sample container with solution at least twice; collect 100ml or more of sample; cap tightly.

2. Take samples to Chew lab in clean tub: **do not bring dirty containers into the Chew lab**.

3. Measure solutions starting with the lowest concentration first.
a. measure 10ml of solution 1 into tiny beaker with fresh repipettor tip; add 10ml of ISA with new repipettor tip + clean stir bar; turn on stirring; continue to set up your solutions this way as you have time while electrode is equilibrating.
b. rinse electrode with double de-I water; gently blot dry with clean kimwipe.
c. place electrode into prepared sample solution 1; dispense gel.
d. allow to equilibrate
e. when the reading "settles out", seems to hover around a point, take the reading, record it. This usually happens about 1 minute after placing the electrode in the solution. If reading continues to change rapidly, dispense more electrolyte gel into solution. If this doesn't work, make a note, take reading anyway & move to the next solution. Contact Ellen for help if problem continues.
f. be ready with the next highest sample: 10ml of sample solution + 10ml of ISA + clean stir bar in small clean beaker.
g. rinse electrode with double de-I water; blot with clean kimwipe, place in next prepared sample, dispense gel, let reading settle out, record.
h. follow with the remaining samples.
i. after final sample, rinse electrode and place it in 30 to 100ppm nitrate (no ISA in the resting solution!) until next calibration. If conditioning for low level nitrate reading, see separate procedure.

C. Clean up
All glassware and other re-useable equipment is washed after the day's measurements are done. Three rinses of di water at the tap and one of double de-I will suffice. Place each piece in a position where it will drain and dry by the next measurement date.