

- Nitrate Analysis by Ion Selective Electrode

Purpose: to use an ion selective electrode to determine Nitrate in water and plant tissue.

Materials:

| | |
|---|---|
| pH/ ISE meter (Corning 150) | 100 ml volumetric flasks (6) |
| Nitrate combination electrode | 2 Molar ammonium sulfate (ISA) ³ |
| Ion stir Mixer | 100 ml plastic cup (8) |
| 1000 ppm NO ₃ -N ¹ | Dried, ground alfalfa |
| 100 ppm NO ₃ -N without ISA ¹ | Rotary shaker ² |
| 10 ml finnpipette and 6 tips | 1.0 ml dispensing pump |
| Kimwipes | DI water bottle |
| Semi-log paper | |
| 100 ml Graduated Cylinder | |

1. Located in refrigerator

2. Located on island in Analytic room. 3. ISA = Ionic Strength Adjustor

Procedure:

Preparation

Remove rubber cap from probe and place nitrate combination electrode in a 100 ppm NO₃-N solution (without ISA). If after 1 hour the mV reading is not stable, push top of electrode near cable three times to dispense gel. (It should click when you push it)

Make standards

- Make 100 ml of the following NO₃-N standards:
100ppm, 10ppm, 1ppm, .1ppm, .01 ppm and .001 ppm.
- Measure 50 mls of the standards into separate 100 ml plastic cups (This includes the 1000 ppm standard).
- Measure 50 mls of tap water into a 100 ml plastic cup

Extract alfalfa sample:

- Weigh .5 grams of alfalfa into a plastic cup.
- Add 50ml of DI water.
- Shake for 20 minutes on rotary shaker set to 150 RPM.

Take mV readings of solutions without ISA present

- Select absolute mV on the Corning Ion meter by pushing the **Mode** button until **ABS MV** appears in the display.
- Rinse electrode and mixer shaft and blot with a kimwipe.
- Place probe and mixer into solution and turn on mixer.
- Take readings by pushing the = button. (Pushing the **AUTO** button will cause an **A** to appear in the top right corner of the display. Auto causes the instrument to lock onto one value when the mV fluctuations are small.
- Starting with your lowest concentration, record the millivolt reading of all standards and samples (Including the tap water).
- Graph data on semi-log paper.

Take mV reading of solutions with ISA present

- Add 1ml of ISA to all of the solutions. (1 ml + 50 ml of solution)
- Take mV readings.
- Graph all data on semi-log paper.

Note:

- Samples and standards need to be at the same temperature i.e. room temperature.
- Check to make sure the nitrate membrane tip is on the electrode. If it isn't there it will be in the container it was stored in i.e. beaker. Ask lab tech for help if it isn't on the electrode.

Shutdown

- Rinse nitrate probe, blot dry and replace rubber cap.
- Turn off ion stir mixer.
- Put everything back where you found it (Don't leave materials by sink.)

ISA = Ionic strength adjustor

Problems

Drift

Problem: The mV readings for a solution won't stabilize. The values slowly go up or down. The values fluctuate wildly.

Solution: Push the top of the electrode near cable three times to dispense gel. (It should click when you push it) If this doesn't work, contact the lab technician.

Span

Problem: The millivolt difference between 10ppm NO₃-N and 100 ppm NO₃-N is less than 50mV.

Solution: Contact technician to install new sensing membrane.

Problem: The millivolt difference between 10ppm NO₃-N and 100 ppm NO₃-N is greater than 59mV.

Solution: Make new standards and add ISA.

Questions:

1. What is the precision of your measurements?
2. Discuss the resolution, working range and detection of limit of the electrode.
3. Discuss the effect of the ISA on the various solutions.
4. Compare the results obtained for the tap water and alfalfa before and after the ISA was added. If the ISA value is the true value then what is the accuracy of the value obtained without using ISA.