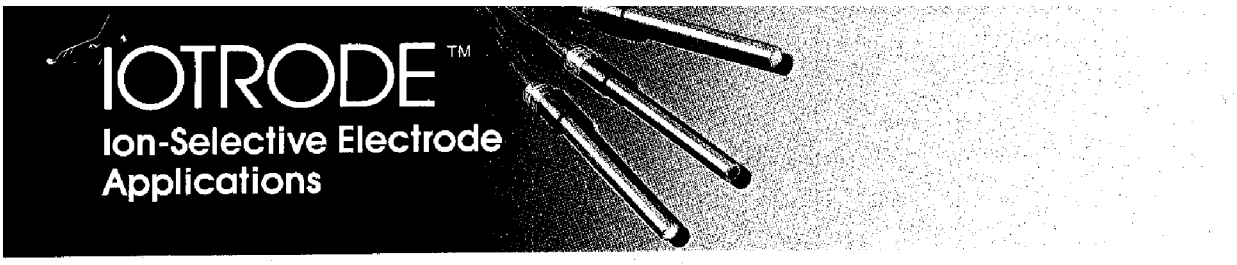


INTRODUCTION

The Iotron Nitrate Electrode is a liquid ion exchanger electrode with a thin, inert membrane. Nitrate in unpolluted and waste water in the range of 20-150 ppm can be determined by this method. Many ions interfere with nitrate determinations. To avoid the interferences a buffer solution is added to all samples and standards.

EQUIPMENT

Meter: pH/Millivolt meter. Readability of 1 mV required; 0.1 mV preferred. Specific ion meters will provide direct readout of final answer or concentration factor. Consult manual for millivolt measurement instructions.

Electrodes: 1. Iotrode(TM) AB 470 Nitrate Electrode. 2. Reference Electrode: RD-101 Calomel, Removable Sleeve; RD-201 Calomel, Non-Removable Reverse Sleeve; RD-301 Ag/AgCl, Removable Sleeve; or RD-401 Ag/AgCl, Non-Removable Reverse Sleeve.

Glassware: 1. 1 ml, 2 ml, and 10 ml pipets. 2. 150 ml beakers, plastic 3. 100 ml graduated cylinders 4. 100 ml, 1000 ml volumetric flasks 5. Magnetic stirrers and stirbars or glass stirring rods.

REAGENTS

1. 1000 ppm Standard Nitrate Solution: Weigh out 1.630 grams of Potassium Nitrate into a one liter volumetric flask. Dilute to mark with deionized water.

2. Ionic Strength Adjustor: Weigh out 1.24 grams Boric Acid into a one liter volumetric flask. Dilute with 900 ml of deionized water and dissolve solids. Dilute to mark with deionized water.

ELECTRODE SET-UP

Plug the sensing electrode into the G.E. or Glass jack of the meter. Fill the inner and outer chambers of the reference electrode with appropriate solutions. Plug the reference electrode into the REF or reference jack of the meter.

TECHNIQUE HINTS:

1. Stirring: Electrode response is improved if the samples and standardizing solutions are stirred at a fixed rate during measurements. If magnetic stirring is not available, stil solution one minute with a clean glass stirring rod before measuring.

2. Temperature: The slope of the sensing electrode and the absolute potential of the reference electrode are temperature dependent. Therefore,

samples and standardizing solutions should be at the same temperature.

3. Cleaning electrodes: Rinse both electrodes with a fresh portion of deionized water and blot dry with tissue between all measurements.

ELECTRODE CALIBRATION

The primary criteria for electrode performance verification is the span test. If 45 millivolts or greater change is observed for a decade change in concentration, the performance is considered satisfactory.

1. Put 50 ml of deionized water and 50 ml of ISA into a 150ml beaker. Place pH meter into the M.V. mode. Place electrodes in the solution to a minimum depth of one inch.

2. Pipet 1 ml of 1000 ppm nitrate standard into the solution. Stir thoroughly. Read stable electrode potential in millivolts and record value as E1.

3. Add 10 ml of 1000 ppm nitrate standard and stir thoroughly. Read stable electrode potential in millivolts and record as E2. Calculate S by E2 - E1. Assume S value as the slope of the electrode.

NOTE: If the slope value is below 45 mV, check electrode set up and recondition sensing electrode.

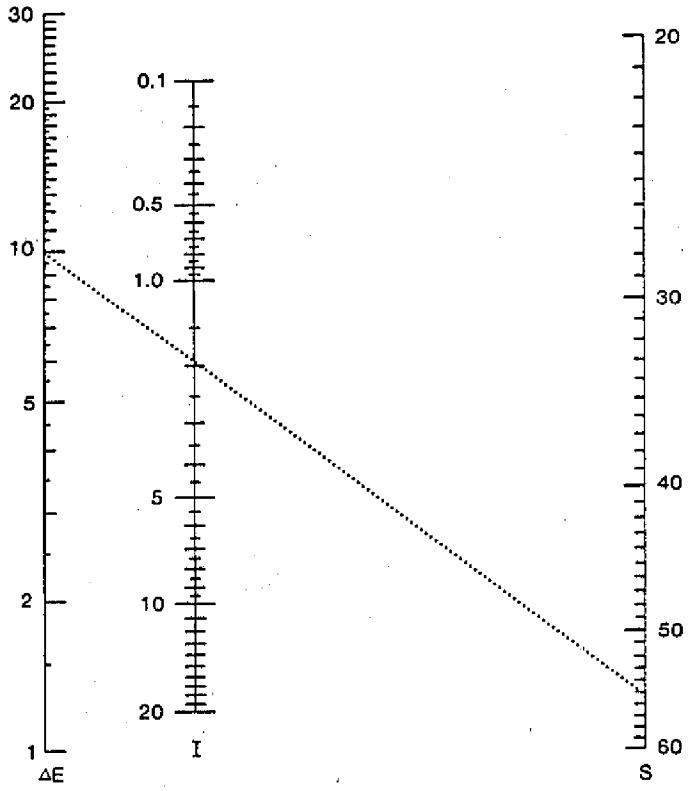
PROCEDURE

Sample Preparation: Dilute 50 ml of sample with 50 ml of ISA.

Sample Standardization: Add 100 ml of prepared sample solution to a 150 ml beaker. Place electrodes in the solution to a minimum depth of one inch. Read stable electrode potential in millivolts and record as E3.

Standard Addition: Pipet 1 ml of 1000 ppm nitrate standard into the solution. Stir thoroughly. Read stable electrode potential in millivolts and record value as E4. Calculate E by E4 - E3. NOTE: If E is more than 30 mV, dilute standard 1:10 with deionized water and divide final concentration by 10. If E is less than 8 mV, dilute fresh sample 1:10 with deionized water and multiply final concentration by 10.

Standard Addition Nomograph for use with Iotrode™ Electrodes



CALCULATIONS

To perform the calculation for standard addition, the following factors are needed:

V_S = Sample Volume
V_a = Volume of standard added
C^* = Concentration of standard
ΔE = Change in potential (see procedure section)
S = Slope of the electrode (see electrode calibration section)

$$\text{Concentration of Sample} = \frac{C^* \left[\frac{V_a}{V_a + V_S} \right]}{\left[\text{antilog} \frac{\Delta E}{S} \right] - \left[\frac{V_S + V_a}{V_S} \right]}$$

Note: If the sample is diluted, multiply sample concentration by appropriate dilution factor.

The standard addition nomograph method of calculation is valid when the increment of standard added to the sample is small compared to the volume. Therefore, if more than 1 ml of standard is added, please utilize the mathematical procedure above.

The following data must be known to utilize the nomograph: (See above for description of factors.)

ΔE, S, C^*, and V_S

Utilizing ΔE and S, draw a straight line to find I on the nomograph.

$$\text{Concentration of Sample} = \frac{(I)\,(C^*)}{V_S}$$

The concentration of Sample will be in the same units of concentration as C^*.

Example: ΔE = 10 mv
S = 55 mv
C^* = 1000 ppm
V_S = 100 ml
I = 2.0 (from Nomograph)

$$\text{Sample Concentration} = \frac{(2.0)\,(1000 \text{ ppm})}{100 \text{ ml}}$$

$$= 20 \text{ ppm}$$

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