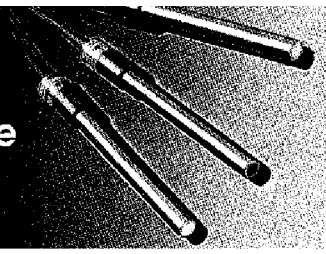
IOTRODE™
Ion-Selective Electrode Applications



Bulletin No. 000110

Iodide Analysis In Water

INTRODUCTION

The following procedure is valid for iodide concentrations as low as 0.5 ppm. Check the deionized water used in reagent preparation. The specific conductivity should be no greater than 0.2 Scm^{-1}. Sample solutions should be fresh and free of sulfide ions.

EQUIPMENT

Meter:

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

Electrodes:

1. Iotrode™ AB140 Iodide Electrode with polishing kit.
2. Reference electrode (Double Junction Type) Iotrode AB450 (Ag/AgC1 internal) or Iotrode AB455 (Calomel Internal)

Glassware:

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

REAGENTS

1. **Ascorbic Acid Antioxidant Solution (AAA):** Dissolve 80 grams NaOH in 500 ml of freshly boiled deionized water. Add 320 grams sodium salicylate very slowly, while stirring solution. Avoid formation of solid clumps. When all solid materials have dissolved, add 72 grams ascorbic acid. When solution has cooled, make up to one liter with deionized water and store in a plastic bottle. Shelf life is approximately two weeks.

2. **1000 ppm Iodide Standard:** Weigh out 1.308 grams of potassium iodide into a one liter volumetric flask. Add 500 ml deionized water and dissolve solid. Dilute to mark and store in dark plastic bottle.

ELECTRODE SET-UP

If the electrode is being used for the first time, please follow the instructions for polishing the membrane surface. 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 samples and standardizing solutions are stirred at a fixed rate during measurements. If magnetic stirring is not available, stir 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 55 millivolts or greater change is observed for a decade change in concentration, the performance is considered satisfactory.

1. Put 100 ml deionized water and 20 ml AAA into a 150 ml beaker. Place the 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 iodide standard into the solution. Stir thoroughly. Read stable electrode potential in millivolts and record value as E1.

3. Add 10 ml of 1000 ppm iodide 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 55 mV, check electrode set-up and repolish sensing electrode.

PROCEDURE

Sample Preparation: None.

Sample Standardization: Add 100 ml of sample solution and 20 ml AAA 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 iodide standard into the solution. Stir thoroughly. Read stable electrode potential in millivolts and record value as E4. Calculate ΔE by E4-E3.
Note: If ΔE is more than 30mV, dilute standard 1:10 with deionized water and divide concentration of standard by 10. If ΔE is less than 1 mV, increase milliliters of standard added to 10 ml.

CALCULATIONS

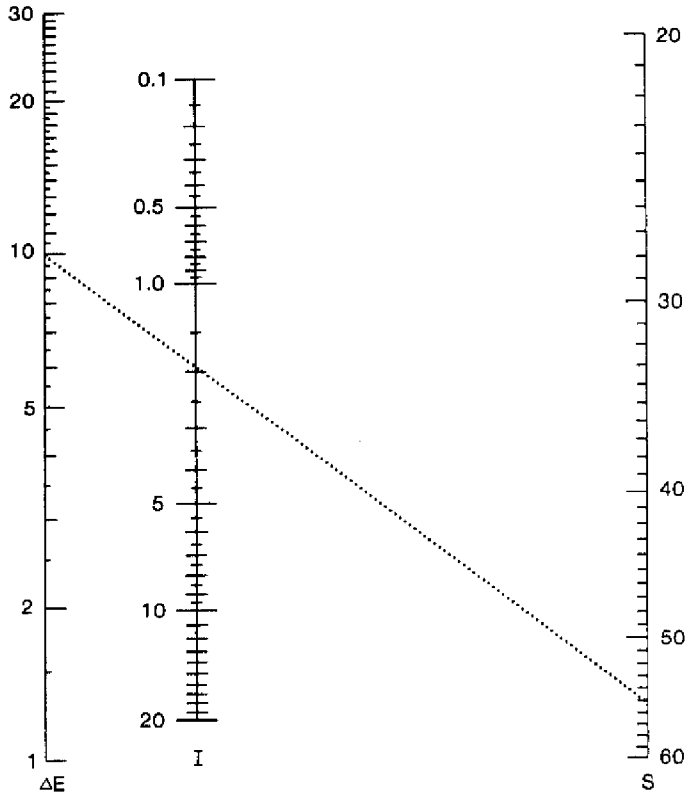
To perform the calculation for standard addition, the following factors are needed:

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.

Standard Addition Nomograph for use with Iotrode™ Electrodes



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)

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

$$= 20 \text{ ppm}$$