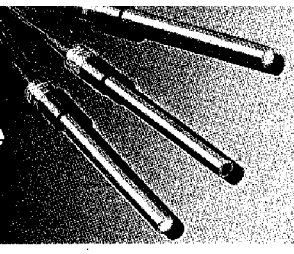
IOTRODE™
Ion-Selective Electrode Applications



Bulletin No. 000103

Determination Of Sulfide In Water

INTRODUCTION

A major contributor to odor in drinking water is sulfide in the form of H_2S. Utilizing the Iotrode™ Sulfide Electrode the user may determine sulfide in most water samples very simply. The only restriction is no mercury ions be present.

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:
Measuring Electrode: Iotrode AB-120 Sulfide ISE.
Reference Electrodes—Double Junction—Refillable
Calomel — RD-101 Removable Sleeve
RD-201 Non-Removable Sleeve
Ag/AgCl — RD-301 Removable Sleeve
RD-401 Non-Removable Sleeve

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

REAGENTS

1. **Sulfide Anti-oxidant Buffer (SAOB):** Dissolve 80 grams NaOH in approximately 500 ml of freshly boiled deionized water and slowly add 320 grams sodium salicylate while stirring. When all the solid has dissolved, add 72 grams ascorbic acid and dissolve. Cool the solution to room temperature and dilute to 1000 ml with deionized water. Store in tightly closed plastic bottle, preferably in a refrigerator. Allow solution to warm to room temperature before use. Shelf life of solution is approximately one week. If solution turns brown, discard.
2. **25% SAOB:** Dilute SAOB 1:4 with deionized water. Prepare fresh daily.
3. **1000 ppm Sulfide Standard:** Dissolve 0.75 grams $Na_2S\ 9H_2O$ in 100 ml of 25% SAOB solution. Determine the sulfide concentration by filtrating 25 ml portion with 0.1M lead nitrate. Store in tightly closed plastic bottle. Shelf life is one week.
4. **0.1M Lead Nitrate:** Weigh out 33.120 grams $PbNO_3$ into a 1000 ml volumetric flask. Dilute to one liter with deionized water. 1 ml = 3.2066 mg sulfide.
5. **Water Sample:** As much as possible, the sample must be kept out of contact with the atmosphere. When collecting samples, dilute immediately 1:1 with SAOB and keep tightly stoppered in a plastic bottle. Do not allow samples to age over one week.
6. **Outer Junction Reference Electrode Filling Solution:** Dilute 10 grams KNO_3 to 100 ml with deionized water.

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 25 millivolts or greater change is observed for a decade change in concentration, the performance is considered satisfactory.

1. Put 50 ml deionized water and 50 ml 25% SAOB 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 sulfide standard into the solution. Stir thoroughly. Read stable electrode potential in millivolts and record value as E_1.

3. Add 10 ml of 1000 ppm sulfide standard and stir thoroughly. Read stable electrode potential in millivolts and record as E_2. Calculate S by E_2-E_1. Assume S value as the slope of the electrode.

PROCEDURE

Sample Preparation: (1:1) dilution with SAOB upon collection.

Sample Standardization: Add 100 ml of 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 E_3.

Standard Addition: Pipet 1 ml of 1000 ppm sulfide standard into the solution. Stir thoroughly. Read stable electrode potential in millivolts and record value as E_4. Calculate ΔE by E_4-E_3.
Note: If ΔE is more than 15mV, **dilute standard** 1:10 with deionized water and divide final concentration by 10. If ΔE is less than 5mV, **dilute fresh sample** 1:10 with deionized water and multiply final concentration by 10.

CALCULATIONS

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

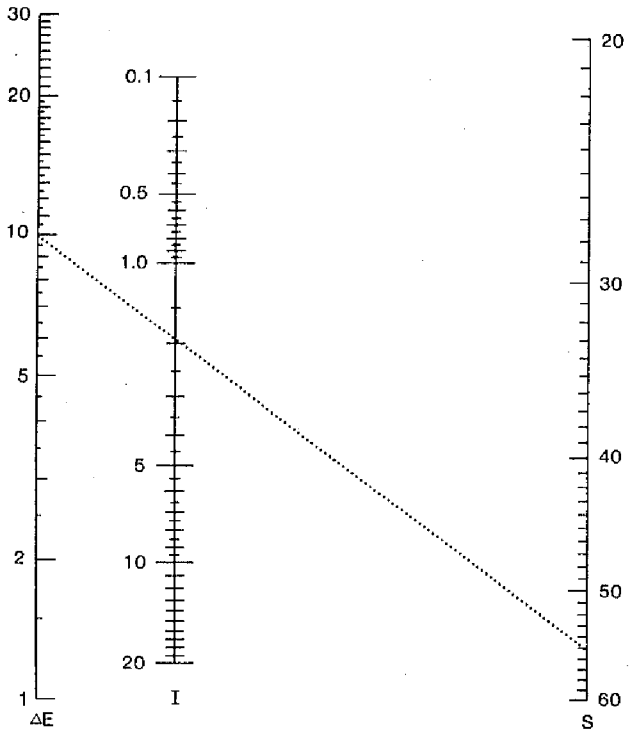
V_S = Sample Volume
V_a = Volume of standard added
C^* = Concentration of standard
ΔE = Change in potential (see procedure section)
S = Slope of the electrode (see electrode calibration section)

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

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

Dilution factor = 2

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)

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

$$= 20\text{ ppm}$$

REFERENCES:

Baumann, E.W., 1974, Determination of parts per billion sulphide in water with the sulfide-selective electrode, Anal. Chem., 46, 1345.