



Parameter & Sample Type

Fluoride in Pure Water (0.1 – 10 mg/L) by direct measure

Introduction

Fluoride at low levels in pure water is determined by direct measurement with the Orion 9609BN combination fluoride electrode on an Orion Star meter or other Orion ISE meter using Orion prepared standards mixed with TISAB II buffer. The calibration is accurate from 0.1 – 10 mg/L fluoride ion.

References

1. Method 4500-F C, Ion Selective Electrode Method. Standard Methods for the Examination of Water and Wastewater, 20th Edition, 1998. APHA, AWWA, & WEF, Washington, D.C. www.standardmethods.org

Recommended Equipment

Orion Star Meter (Orion 1119000 or 1115000) or other Orion ISE meters without ISE temperature correction; Fluoride electrode (Orion 9609BN); Orion ATC 9270006MD, Orion 096019 paddle stirrer for Star meter or magnetic stirrer; 50 mL graduated cylinder;

Required Solutions

Fluoride standards at 1, 2, and 10 mg/L (040906, 040907, & 040908); TISAB II (940909); fill solution Optimum Results A (900061); deionized water (DI).

Solutions Preparation

0.5 mg/L check standard: measure 20.0 mL of 1 mg/L standard (Orion 040906) into a clean 50 or 100 mL beaker. Add 10.0 mL of TISAB II buffer and 10.0 mL of DI.

Meter Setup

Connect the electrode to meter and set up for stirring (magnetic or paddle stirrer). Follow meter manual setup for Fluoride by ISE measurement. Select resolution of 3 digits, concentration units of mg/L (ppm), auto-blank correction (nLin) set to auto, and read type continuous.

Electrode Setup

See the electrode manual for (assembling and) preparation of the electrode.

Electrode Performance Check

Check slope daily according to the electrode manual. Drift may be checked by comparing a 1 minute to a 2 minute reading. Results should agree with desired criteria. See troubleshooting section of manual if slope or drift problems.

Electrode Storage, Soaking, and Rinsing

Electrodes may be stored in Orion 040906 (1 mg/L fluoride standard with TISAB II) between measurements and overnight. For long periods of time, store dry. See electrode manual for details on dry

storage. Between measurements, rinse the electrode with DI water and gently blot dry.

Sample Preservation

Collect fluoride samples in plastic. No sample preservation is required. A collected sample is valid for 28 days. Refer to reference(s) and/or EPA 40 CFR Part 136.3 for details.

Sample Preparation

Fluoride testing by ISE method is a temperature sensitive procedure. For precise measurements, allow all the standards and the samples to reach room temperature before analysis. Standards are premixed with TISAB II – no preparation is required. For each standard, pour about 30 mL into a clean 50-mL beaker. For samples: measure 15.0 mL sample into a 50-mL clean beaker. Add 15.0 mL TISAB II to the beaker for a total volume of 30 mL. If using magnetic stirrer, add a clean dry stir bar.

Calibration

A three point calibration is performed at 1, 2, and 10 mg/L fluoride ion. After this three point calibration, results are accurate down to 0.1 mg/L. See fluoride graph on page 2.

Place the electrode(s) into the 1 mg/L fluoride standard with TISAB II (Orion 040906) and stir. Allow electrode to stabilize until readings change by no more than 2% per minute (0.5 mV/min), which is 0.020 mg/L per minute in the 1 mg/L standard. Then, begin the three point calibration, starting with the 1 mg/L standard, followed by the 2, and 10 mg/L standards. After calibration, the slope will be displayed and should be 54 mV/decade or higher. Note: the slope value for a three point calibration may be higher than 60 mV/decade due to the auto-blanking function of the meter. Check accuracy by reading the 0.5 mg/L check standard. Results should be close to 0.5 mg/L, within the user-determined tolerance. See page 2 for typical results. If reading is not acceptable, see troubleshooting section of the manual.

Analysis

Rinse electrode(s) and stirrer (if using Star meter) with DI water and blot dry. Place in the prepared sample and stir. A stable reading is usually achieved in 2-3 minutes. Verify that the sample has come to room temperature before recording the fluoride results. Record the fluoride value and the temperature.

Quality Control (QC)

Recommended QC procedures include: calibration and calibration verification, initial demonstration of laboratory capability and method detection limit determination, laboratory control samples (LCS), method blanks, matrix spikes (MS), sample duplicates, and independent reference materials. See references above for details.



Testing Results:

Fluoride conc (mg/L)	Result (mg/L)	mV	Resp time (min)	%R	RSD or %D
1	1.02	103.5	1	102%	1.3%
1	1.04	103	1.5	104%	
1	1.04	103	1.5	104%	
1	1.05	102.5	1.5	105%	
0.5	0.507	121.2	1.5	101%	3.2%
0.5	0.523	120.4	2	105%	
0.2	0.198	143.7	2	99%	4.5%
0.2	0.207	142.8	2	104%	
0.1	0.0895	160.6	2	90%	3.0%
0.1	0.0917	160.1	1.5	92%	
0.1	0.0961	159.2	1.5	96%	
0.1	0.0947	159.5	3	95%	

Note: fluoride response was tested between 0.1 and 10 mg/L. Calibration at 1, 2, and 10 mg/L gives accurate results down to 0.1 mg/L.

