**Parameter**
Nitrate

**Sample Type**
Wastewater

**Introduction**
The ion-selective electrode method is an EPA-approved test procedure to directly read nitrate in wastewater. Laboratory productivity is improved with quick, uncomplicated determinations. Use of the NISS buffer frees the ISE method from most interferences and holds the pH and ionic strength of all samples and standards constant. This procedure conforms to the EPA-approved test procedure from Standard Methods, as listed below.

**Reference**

**Result Statistics**
<table>
<thead>
<tr>
<th># Trials</th>
<th>Average</th>
<th>%CV</th>
</tr>
</thead>
<tbody>
<tr>
<td>3</td>
<td>11.8 mg/L</td>
<td>1.3 %</td>
</tr>
</tbody>
</table>

**Recommended Equipment**
5-Star Benchtop pH/ISE/Cond/DO meter (Orion 1119000) or other Orion ISE meter; ionplus® Nitrate Electrode (Orion 9707BNWP); benchtop stirrer (Orion 096019). Optional: printer (Orion 1010006); Star Navigator Software (Orion 1010007).

**Required Solutions**
1000ppm Nitrate as N standard (920707); 1.10 and 50mg/L nitrate as N calibration standards; Nitrate Interference Suppressor Solution (NISS) (Orion 930710); Optimum Results F fill solution (900046); Deionized water

**Solutions Preparation**
1mg/L Nitrate as N: 0.5mL 1000ppm std into 500mL volumetric flask, bring to volume with DI water.
10mg/L Nitrate as N: 5mL 1000ppm std into 500mL volumetric flask, bring to volume with DI water.
50mg/L Nitrate as N: 25mL of 1000ppm std into 500mL volumetric flask, bring to volume with DI water.

**Meter Setup**
Connect the electrode and stirrer to the Star Meter. Set measurement mode to ISE. In Setup mode, set resolution to 3, set the range to high, select mg/L as the unit, and set read type to auto. If all steps were followed correctly, the meter display will show three digits in the top line and "ISE: mg/L" to the right of the top line. For other Orion ISE meters, see meter instruction manual.

**Electrode Setup**
See the electrode manual for preparation of the electrode.

**Electrode Performance Check**
Check slope at least 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 for slope or drift problems.

**Electrode Storage, Soaking, and Rinsing**
See electrode manual for storage 1) between measurements, 2) overnight, and 3) for long periods of time. Between measurements, rinse the electrode with DI water and dry outer sleeve of electrode to remove drops before measuring the next sample. Do not wipe or rub the sensing element of the electrode.

**Sample Preservation**
Samples must be analyzed within 48 hours of collection and should be stored at <6 degrees Celsius. Refer to reference and/or EPA 40 CFR Part 136.3 for details.

**Sample Preparation**
Measure 10mL of sample and 10mL of NISS into a 50mL beaker. NISS must be added to all standards and samples. A larger sample size can be used if desired as long as NISS is added in a 1:1 ratio. For precise measurements, allow all standards and samples to reach the same temperature before analysis.

**Calibration**
Perform a three point calibration using 1.0 mg/L, 10.0 mg/L and 50.0 mg/L nitrate as N standards. After calibration, the electrode slope will be displayed and should be above 54mV/decade. Read a fresh portion of a standard to verify calibration. If reading is not acceptable, see troubleshooting section of electrode manual.

**Analysis**
Rinse electrode and stirrer with DI water and dry outer sleeve of electrode to remove drops. Place probe and stirrer in sample and measure. The concentration of the sample will be displayed. When a stable reading is achieved, the “ISE: mg/L” icon will stop flashing, the stable reading will be displayed and the stir probe will turn off.

**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.
### Electrode Log # 31

<table>
<thead>
<tr>
<th>Wastewater</th>
<th>ppm (mg/L) Nitrate</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample 1</td>
<td>11.80</td>
</tr>
<tr>
<td>Sample 2</td>
<td>11.60</td>
</tr>
<tr>
<td>Sample 3</td>
<td>11.90</td>
</tr>
<tr>
<td>Mean</td>
<td>11.77</td>
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<tr>
<td>Standard Deviation</td>
<td>0.153</td>
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<tr>
<td>%CV</td>
<td>1.30</td>
</tr>
</tbody>
</table>

**Typical Nitrate Calibration Curve**

- Electrode Potential (mV) vs. Log of concentration (mg/L)

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