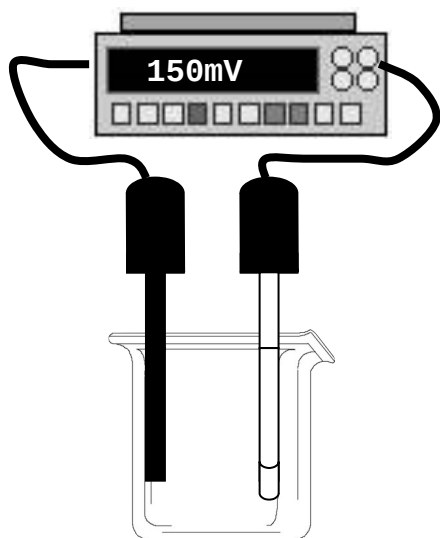
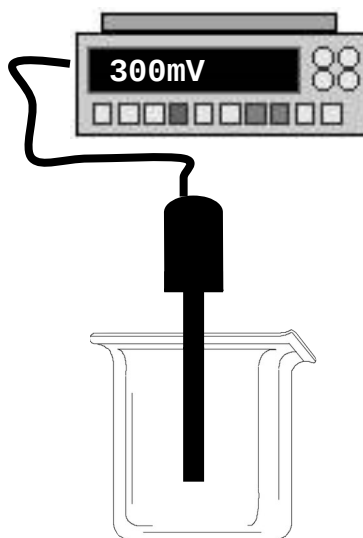


SENTEK

Ion Selective Electrode OPERATING INSTRUCTIONS



Mono/Reference



Combination

P
E
R
C
H
L
O
R
A
T
E

SENTEK

Unit 6 & 7 Crittall Court, Crittall Drive, Springwood Industrial
Estate, Braintree, Essex, CM7 2SE

Tel: +44 (0) 1376 340 456 Fax: +44 (0) 1376 340 453 Email: sentekuk@btconnect.com

Operating Instructions

Perchlorate Ion Selective Electrode

This Document includes operating instructions for two types of Ion Selective Electrode. A single sensing Half Cell Perchlorate Electrode which requires a separate Reference Electrode, and a Combination Perchlorate Electrode which has the Reference built into the outer shaft.

For extremely low level work the Half Cell plus Reference Electrode should be used. For general applications the Combination is more convenient.

Depending on the type of ISE chosen you will need to disregard some information for instance if you have the Combination Electrode then disregard any comments about reference electrodes.

A quick guide is included with a more involved section following.

SENTEK

Unit 6 & 7 Crittall Court, Crittall Drive, Springwood Industrial

Estate, Braintree, Essex, CM7 2SE

Tel: + 44 (0) 1376 340 456 Fax: +44 (0) 1376 340 453 Email: sentekuk@htconnect.com

Quick Guide to Perchlorate

ISE

The combination ion selective electrode has a solid state PVC polymer membrane with an integral *dri*TEK reference electrode. The electrode is designed for the detection and analysis of Perchlorate ions in aqueous solutions and is suitable for use in the field in the laboratory and in on line analyzers.

Installation

Connect the ISE to the mV or ion meter.

Remove the black protective cap and keep it in a safe place.

The ISE can be used immediately but pre soaking for 5 minutes in a 100ppm Perchlorate solution is recommended.

The ionic strength of the standards and solutions should be kept constant between all standards and samples. This is achieved by the simple addition of an Ionic strength adjustment buffer (ISAB).

Ammonium Sulphate is ideal. A typical addition would be 1 ml of 1 molar ISAB to 100 ml of standard and sample.

For low level measurements below around 50 ppm in relatively pure samples no ISAB is needed.

No temperature correction is necessary however standards and samples should be measured at the same temperature.

Begin calibration from the lowest concentration standard to avoid cross contamination. Calibration should cover the anticipated range of the samples.

Rinse tip with deionised water between measurements.

Avoid strongly acidic or alkaline samples, strong detergents and organic solvents.

Storage and Maintenance

Avoid touching the membrane surface.

After use rinse with deionised water, replace protective cap and store dry in its box.

If performance becomes sluggish, rinse with dilute detergent, rinse with deionised water and immerse the tip in a 1000ppm Perchlorate solution for 1 hour.

Specification

Perchlorate is a monovalent anion. A 1 molar solution of Nitrate contains 198.898 grams per litre of Perchlorate.

A 1000 ppm solution is equivalent to 0.005 moles per litre

Refer to the table below for the full specification:

Parameter	Specification
Overall length	155 mm
Body Diameter	12 mm
Cap Diameter	16mm
Connector	BNC
Cable length	180 mm
Resistance at 25 Deg C	< 2.5 Meg Ohm
Concentration Range	0.2 to 99,500 ppm
Slope	-54 to -59 mV per decade
Potential Drift	2 mV per day
Operating pH range	0 to 11
Temperature range	5 to 50 Deg C
Endpoint time	Typically 10 to 30 seconds
Interferences. Ions with coefficients above 0.001.	Iodide Nitrate. Thiocyanate

Operating Instructions for the Perchlorate Ion Selective Electrode

1. Characteristics

The Ion Selective Electrodes are "All Solid State" meaning they have a solid state membrane, an internal solid contact and contain absolutely no liquid: they have important advantages:

- Easy to handle, require only a few minutes preconditioning prior to usage.
- Stability of electrochemical potentials resulting in a very low signal drift and eliminating the necessity for frequent re-calibration.
- Short response time which is of importance when used in automatic analysers, titrators, process monitoring and process control.
- Function in any position: upside down for small sample voluminous.
- No leaking of liquids.
- Practically no limitation of storage time
- Can be stored and transported below 0°C (32°F)

2. Applications

ISE's are used in a wide range of applications:

- General use in Analytical Laboratories.
- Usage out in the field.
- Continuous and long time monitoring of industrial and environmental parameters.
- On-line process control.
- Automatic Analysers, Titrators, Clinical Analysers.

The Perchlorate ISE is especially used for:

Analysis of explosives, solid propellants, solid fuels.

3. Specifications

Mechanical:

Body	:	Diameter 12mm, Length
120mm		
Total Including Cap	:	Diameter 16mm,
Length 150mm		
Cable Length	:	1.0m, shielded
Connector	:	BNC (if no other specified)
Electrode Weight	:	20g

Electrical:

Resistance	:	Less than 2.5 Megohm
Response Time	:	Less than 10 s (30 s)
Potential Drift	:	Less than +/- 1mV
Re-calibration	:	Once a day

Chemical:

Preconditioning Solution :	10^{-1} M NaClO ₄
Preconditioning Time :	15 Minutes
Admissible pH Range :	pH 0 to pH 11
Admissible Temperature :	5°C to 50°C
Optimal Temperature :	25°C
Recommended TISAB :	0.1 M CH ₃ COONa
Reference Electrode :	Double Junction
Outer Filling Solution :	0.1 M CH ₃ COONa
Electrode Slope (25°C) :	54 to 59 mV/Decade
Linear Measuring Range :	1 to 2×10^{-5} M 10^5 to 2ppm
Total Measuring Range :	1 to 2×10^{-6} M 10^5 to 2×10^{-1} ppm
Detection Limit :	Less than 2×10^{-6} M 2×10^{-1} ppm

Selectivity Coefficients:

I-Ion k (ClO ₄ ,I)	=	2×10^{-2}
SCN ⁻ -Ion k (ClO ₄ ,SCN)	=	3×10^{-2}
NO ₃ ⁻ -Ion k (ClO ₄ ,NO ₃)	=	2×10^{-3}
Cl ⁻ -Ion k (ClO ₄ ,Cl)	=	3×10^{-4}
H ₂ PO ₄ ⁻ -Ion k (ClO ₄ ,H ₂ PO ₄)	=	2×10^{-4}
CH ₃ COO ⁻ -Ion k (ClO ₄ ,CH ₃ COO)	=	10^{-4}

4. Electrode Preparation and Maintenance

Remove protective cap and immerse the electrode for the specified time in the recommended preconditioning solution. When soaking is complete, rinse the Electrode with distilled water and dry : the Electrode is now ready for calibration.

When an Electrode shows a slow response or low slope, the membrane may be poised or coated with a film. Try to rejuvenate the membrane by soaking the Electrode in the preconditioning solution for 12 hours. If this procedure is not successful, polish the membrane with very fine emery paper and soak for 15 minutes in the preconditioning solution.

Avoid exposing the membrane to high concentrated solutions for long periods of time.

Avoid touching the membrane.

After measurement, rinse the electrode with distilled water and dry. Replace the protective cap and store.

5. Measuring with ISE's

An ISE is always used with a Reference Electrode : Select type as recommended and fill with the specified solutions. Make sure the liquid junction is not clogged.

The electrochemical potential E between the ISE and the Reference Electrode indicates the activity a of the ion to be determined:

$$E = E^{\circ} + S \cdot \log a$$

$$E^{\circ} = \text{Constant Potential } S + \text{Slope of the Electrode}$$

The ISE and the Reference Electrode are connected to a suitable pH Meter, Ion Meter or Computer Interface. Refer to the manual for each device.

During calibration and measurement, the solutions are softly stirred with a magnetic stirrer.

Ensure the calibration and sample solutions have the same temperature (+/- 2°C).

Ensure the Electrode is rinsed and dried between measurements to avoid carry-over or contamination.

When reading the meter, wait until the value is stable.

6. Calibration

Calibration is carried out by using concentration standards. For a correct calibration, the calibration solutions must have the same ionic strength as the sample solution. This is achieved by adding TISAB (Total Ionic Strength Adjustment Buffer) to the calibration and sample solutions. In solutions with low ionic strength (less than 10^{-4}M) like water, this is not necessary.

When the equation in Paragraph 5 is drawn on a semi-logarithmic paper (potential E linear on the vertical axis and concentration logarithmic on the horizontal axis) the graph is a straight line. Therefore, it is necessary to make a 2 point (better 3 or 4 point) calibration measurement in order to plot the calibration curve.

The concentration standard is usually diluted in powers of 10 (dilute 1 part with 9 part of distilled water).

7. Direct Potentiometry

It may be necessary to pretreat the sample before taking a measurement: complexing interfering ions and decomplexing the ion to be determined. For accurate results, the calibration and sample solutions should have the same matrix (background) and all the reagents added to the sample should be added to the calibration solutions.

When the Electrodes are immersed in the sample and the reading is stable, the measured electrochemical potential allows the concentration of the ion to be determined from the calibration curve.

8. Standard Addition Method

The concentration of the sample can be determined by adding a certain amount (usually 10% of the sample volume) of a concentrated standard (approximately 20 times the expected sample concentration) to the sample and measuring the change in the electrochemical potential. The sample concentration can be calculated by the formula:

$$C_p = C_s \frac{V_s}{(V_p + V_s) \cdot A - V_p} : A = 10$$

C_p	Concentration of Sample	C_s	Concentration of Added Standard
V_p	Volume of Sample	V_s	Volume of Added Standard
$E_2 - E_1$	Potential Difference	S	Electrode Slope

This method is simple to perform and the results are reliable. The precision is in the range of 2% to 5%. The slope of the Electrode is known from previous calibrations.