

Operating Instructions & Technical Specifications

directION Sulphide Combination ISE.

The *directION* combination ion selective electrode has a solid state crystalline membrane with an integral driTEK reference electrode. The electrode is designed for the detection and analysis of Sulphide 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 20 minutes in a 100 ppm Sulphide solution (adjusted to pH13-14) is recommended.

The ionic strength of the standards and solutions should be kept constant between all standards and samples. The pH also needs to be controlled and the Sulphide Ions prevented from oxidising. This is achieved by the simple addition of a Sulphide anti oxidant buffer(SAOB). Typical ratio is 1: 1 sample or standard plus SAOB.

SAOB Preparation.

To approximately 600ml of distilled Water in a 1000ml beaker add 200 ml of 10 N NaOH, 35g of ascorbic acid and 67g of Disodium EDTA. Stir completely until everything dissolves. Dilute to the 1000 ml mark with distilled water. Prepare fresh every couple of weeks. Handle with care.

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

After use rinse with deionised water , wipe clean with a tissue or lint free cloth, 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 1000 ppm Sulphide solution for 1 hour.

Should further conditioning be requires rub down the crystal surface with a very fine emery paper until the surface has a grey shiny appearance. Stand electrode in 100 ppm Sulphide solution for 20 minutes.

Specification:

Sulphide is a divalent anion.

Refer to the table below for the full specification:

Catalogue number 3225 Sulphide *directION* Combination ISE

Parameter	Specification
Overall length	155 mm
Body Diameter	12 mm
Cap Diameter	16mm
Connector	BNC
Cable length	1000 mm
Resistance at 25 Deg C	< 2.5 Meg Ohm
Concentration Range	0.003 to 32,100 ppm
Slope	-24 to -29 mV per decade
Potential Drift	2 mV per 8 hr
Operating pH range	13 to 14
Temperature range	5 to 50 Deg C
Endpoint time	Typically 10 to 30 seconds
Interferences: Ions with coefficients above 0.001.	Silver Mercury

Recommendations for successful analysis.

directION combination ISE's can be used with any pH/mV meter or Ion meter. If the meter does not have a BNC socket and you have a BNC electrode please contact your *directION* distributor who will arrange to have the correct plug fitted. Adapters are also available if the same electrode has to be used on more than one meter.

Meters with a 0.1 mV resolution are recommended whilst dedicated Ion meters will provide direct concentration readouts saving time and effort in constructing calibration curves and performing calculations. Your *directION* distributor can advise on the most suitable meter.

Magnetic stirrer/stirrer bars are recommended for laboratory analysis. Please operate at the lowest constant speed available.

Semi-logarithmic (4-cycle) graph paper is required for preparing calibration curves when you are using a mV meter.

Required Solutions

Distilled or deionised water will be required to prepare Standards, ISABs and to rinse the electrode between measurements.

1000 ppm Stock Standard solution. Used for preparation of Standards. (Prepared by customer)

ISAB. Used to adjust the Ionic strength of all standards and samples. Typical addition is 1 ml of 4 Molar ISAB to 50ml of all standards and samples.

Operation

1. Connect the electrode to the meter being used for analysis
2. Prepare a series (at least 2) of standards that bracket the expected sample concentration. This is best done by serial dilution of the stock solution. Ideally standards should be a decade in concentration apart e.g. 1, 10, and 100 ppm.
3. Dispense 50 ml of each standard into analytically clean beakers (100 to 150 ml size is perfect)
4. Add ISAB/TISAB in the appropriate ratio. As a guide with sample concentrations in the 1 to 1000 ppm range 1ml of a 4 Molar ISAB to 50 ml sample is satisfactory. For TISAB(Fluoride analysis only) please read the label.
5. Rinse the electrode with deionised water and blot dry with a lint free cloth and place in the lowest standard. When the reading is stable record the mV value.
6. Repeat step 6 for all subsequent standards proceeding from lowest to highest.
7. Plot a calibration curve on semi log paper using mV values on the linear Axis and concentration on the log scale.
8. Rinse the electrode in deionised water and blot dry. Place the electrode in the sample and record the stable mV value.
9. Using the calibration curve determine the unknown sample concentration.

Hints and tips.

1. Ensure that the temperature of all standards and samples are the same to reduce errors.
2. Using a magnetic stirrer for laboratory analysis is recommended but not essential. It is however important to have the stirrer set on a low constant speed which must be reproducible for all measurements.
3. Prior to sample measurement ensure that the electrode is thoroughly rinsed with deionised water. It is worth performing this rinse twice given the possibility of carryover being greatest in high concentration solutions.
4. Prepare standards by serial dilution.
5. Make sure your electrode is conditioned by leaving the tip in the lowest concentration standard for 1 hour prior to analysis.

Methods of Analysis.

We have described direct potentiometry above. This method is simplified by using a direct reading ion meter. There are several other methods, which are useful. For full advice or copies of applications method sheets please contact info@edt.co.uk

Known Addition: An incremental technique where the potential of the sample solution is measured followed by addition of a small volume of a higher concentration standard solution. The new potential is measured and from difference in the two values, and using the known electrode slope, the unknown concentration is determined,

This method is ideal for samples whose matrix is not entirely clean or aqueous. In these instances comparisons with clean standards is not appropriate thereby making direct potentiometry unsuitable. Known addition works because both standard and sample are measured in the same matrix.

Typical sample volume is 50 ml, typical standard volume is 5 ml. The standard should be approximately 100 times the sample concentration for accurate analysis.

Sample Addition: An incremental technique where the potential of a dilute standard solution is measured followed by the addition of a small volume of more concentrated sample. The new potential is recorded and the difference noted. Using this value (and the predetermined electrode slope) the unknown concentration is determined.

This method is ideal for dirty or viscous samples with an awkward matrix. The sample however needs to be relatively concentrated i.e. at least 100 times the Electrodes linear detection limit. The analysis does have the benefit of only requiring a small volume.

The sample matrix is basically broken down by dilution with the standard and therefore analysis is carried out in the same media.

End Point Titration: Flow Plus combination ISE's are ideal end point indicators and will produce a significant potential change at the equivalence point. The Ion in question must be contained in the titrand or the titrant and must therefore be in excess or absence at the end point.

Applications Advice:

There are three types of directION ISE all of which have advantages in certain applications or circumstances. It is important to select the correct ISE for your application. The typical situations are listed below with the most appropriate ISE type. When in doubt contact your local directION distributor or e mail info@edt.co.uk for further advice or support.

Half Cell ISE with separate reference:

Traditionally the most common set up with a double junction reference electrode filled with the appropriate filling solution which is usually the ISAB or a dilution of the ISAB.

This set up is ideal in low concentration work where the outer filling solution of the reference can be diluted to minimise Interference or Ionic Strength problems. It is also advisable when several ISEs are being used simultaneously as this will be both economical and practical. Don't forget, if the reference fails it is fairly low cost to replace. Combination ISE's are more expensive and either a reference or sensor failure will mean a full electrode replacement.

Half cell ISE's are also easier to maintain or polish (crystalline membranes only) and therefore can have a longer lifetime.

Double junction reference electrodes are also slightly faster responding due to the liquid electrolyte, but do have the disadvantage of needing to be re filled and to be stored wet.

Combination directION ISE with built in driTEK reference:

This is the most practical way to perform Ion analysis in the field and is perfect for non skilled operators. The ISE and reference are combined in one body, which is stored dry and requires no or little maintenance.

The connector is a BNC which will fit all meters without having to worry about the configuration of the reference half cell connector.

Because the reference has a gel electrolyte it is both fairly concentrated and cannot be replenished. This means that working at low levels requires more care (particularly conditioning) and in these cases the Flow Plus or Half Cell options are preferable.

The reference electrolyte cannot be replenished and so the electrode lifetime is less than that of a Half Cell and a reference however the practicality of this set up outweighs any lifetime issues if you need to work in the field or require rapid analysis with unskilled operators.

Flow Plus Combination ISE:

This type of ISE combines the benefits of both of the above options. There is no need for a separate reference as it is a combined unit. It also has a liquid refillable reference which enables low concentration work to be performed but with the added advantage of being stored dry due to the ability to flush clean the electrode after use.

The sensor is also easy to maintain as the electrode can be taken apart and re assembled.

There are only two considerations which are; that you need a reference electrolyte for operation and that it is slightly more expensive than the conventional Combination ISE.

For further information visit www.edt.co.uk