

Nitrite FlowPlus Combination ISE Instructions.

The direct/ON nitrate combination ISE has a solid state PVC polymer membrane with an integral liquid reference system. The electrode is designed for the detection and analysis of Nitrite ions in aqueous solutions and is suitable for use in the laboratory and as a component in on-line analysers.

The Flow plus range incorporates two solid state ISE's in one 12 mm Epoxy body. One acts as the Sensor whilst the other is incorporated into the reference **system**. Your Flow plus combination ISE therefore does not require a separate reference electrode, making it convenient to use with small sample volumes. The reference half cell has a free-flowing liquid junction which will reduce drift and provide more stable readings than conventional ISE's. Flow plus electrodes are ideal for measuring awkward or dirty samples, which would clog conventional reference electrodes.

For after sales support and applications advice please contact your direct/ON distributor or e-mail sales@direct-ion.com.

Principle of operation.

The solid state sensor provides rapid analysis, as it does not require lengthy pre conditioning or an internal solution. Its shelf life is also longer than for standard ISE's.

The Flow Plus reference system has a liquid junction provided by a conical plug which can be flushed clean by rotating and then pressing the electrode cap thereby flushing electrolyte through the junction. If the sample is dirty the junction can be operated with a faster leak rate by opening the junction slightly. This operation is performed by rotating the cap fully clockwise followed by half a turn counter clockwise for normal aqueous samples, and one full turn for dirty samples

The Electrolyte passes over a rounded, polished, solid state ISE incorporated into the Electrodes inner shaft. The internal ISE varies in type according to the Ion being used as the sensor. The Electrolyte and the internal ISE therefore always have the same potential and contact is made between the two ISE's by the flowing electrolyte.

Flow plus combination ISE's have several technical advantages over conventional combination electrodes. These are clearly demonstrated upon use of the electrodes in real situations with awkward samples or in cases where stability, response times or drift cause a problem.

Equipment required

Flow plus 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 direct/ON 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 direct/ON 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.

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

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

Electrode Preparation

Your Nitrite Flow Plus combination comes complete with an optimized filling solution (Cat No. 6071). Please ensure that no other solution is used, as this will adversely effect the performance of the electrode.

The filling solution should be replenished each day before using the electrode and should be filled to a level around 20 mm below the fill hole to ensure a proper flow rate. The electrode filling solution level should always be at least 20 mm above the sample solution level.

The electrode should be allowed to stand in the lowest concentration standard solution being used for 1 hour prior to analysis. This solution should not contain ISAB/TISAB for this purpose.

Filling the electrode

1. Turn the white electrode cap fully clockwise thereby closing the liquid junction.
2. Using the filling solution and dispensing tip provided add a small amount of filling solution through the fill hole. Tip the electrode upside down so as to lubricate the O-ring at the top of the electrode. Return the electrode to the vertical position.
3. Open the liquid junction by turning the electrode cap one turn counter clockwise.
4. Drain the chamber by pressing down on the white electrode cap with your thumb. A few drops of filling solution will be released from the electrode junction and this will wet the internal cone.
5. Close the junction by rotating the cap fully clockwise and fill the electrode to just below the level of the fill hole.
6. Rotate the electrode cap half a turn counterclockwise prior to use.

Operation

1. Prepare the electrode as described in the last section.
2. Connect the electrode to the meter being used for analysis
3. 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.
4. Dispense 50 ml of each standard into analytically clean beakers (100 to 150 ml size is perfect)
5. Add ISAB/TISAB in the appropriate ratio. As a guide with sample concentrations in the 1 to 1000 ppm range 1 ml of a 4 Molar ISAB to 50 ml sample is satisfactory. For TISAB please read the label.
6. 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.
7. Repeat step 6 for all subsequent standards proceeding from lowest to highest.
8. Plot a calibration curve on semi log paper using mV values on the linear Axis and concentration on the log scale.
9. Rinse the electrode in deionised water and blot dry. Place the electrode in the sample and record the stable mV value.
10. 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 sales@direct-ion.com.

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.

Electrode Storage

The filling solution should not be allowed to evaporate as this causes crystallization. For overnight storage store the electrode in the lowest concentration standard.

For longer periods the filling solution should be drained and the inside of the chamber flushed with deionised water. The electrode should be stored with the protective cap in place to prevent any damage to the membrane surface.

Cleaning and Maintenance.

The Flow Plus ISE can be disassembled for complete cleaning however this is not usually necessary and should not be undertaken unless absolutely necessary.

If the junction becomes blocked between the cone and the body this can usually be rectified by flushing with filling solution. Rotate the cap a couple of turns counter clockwise, hold the electrode and with your thumb push the cap down and drain the chamber. Refill with filling solution.

If you need to clean the electrode more thoroughly please follow the steps below.

1. Rinse the outer body in warm running water.
2. Drain the chamber as above.
3. Unscrew the cap, slide the cap and spring up the cable.
4. Holding the electrode body, push down on the Grey threaded portion with thumb and forefinger until the cone is protruding from the bottom of the shaft.
5. Withdraw the inner body from the shaft by grasping the cone with a clean tissue.
6. Rinse the inner body and the electrode shaft with deionised water and allow to dry.

Reassembly

1. Moisten the O-ring on the electrode body with a drop of filling solution. Insert the screwthread end of the electrode body into the outer body of the electrode.
2. Push the inner body into the shaft using a gentle twisting motion. Take care not to handle the membrane surface. Stop when the bottom end is flush with the outer shaft.
3. Locate the spring on to the end of the body and screw the cap back on in a clockwise direction.
4. Refill with filling solution.

Care of the PVC Membrane

The PVC membrane surface should not be handled. For routine cleaning please rinse with deionised water and blot dry.

Should further cleaning be required immerse the tip in a warm dilute detergent solution for around 10 seconds and then rinse thoroughly with deionised water.

For long term or unattended storage the protective black cap should be placed over the electrode to avoid any potential damage.

The membrane should not be brought into contact with organic solvents, fats, oils and strong acids and alkalis.

Catalogue number 5071**Nitrite Flow plus 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	0.5 to 460 ppm
Slope	-30 to -59 mV per decade
Potential Drift	2 mV per day
Operating pH range	2 to 11
Temperature range	5 to 50 Deg C
Endpoint time	Typically 30- 60 seconds
Interferences. Ions with coefficients above 0.001.	Cyanide

Ordering Information.

Part Number	Description
5021	Nitrate Flow Plus Combination ISE. Range 0.4 to 62,000 ppm. Complete with filling solution, one metre cable and BNC connector.
5031	Potassium Flow Plus Combination ISE. Range 0.04 to 39,000 ppm. Complete with filling solution, one metre cable and BNC connector.
5041	Calcium Flow Plus Combination ISE. Range 0.02 to 4,010 ppm. Complete with filling solution, one metre cable and BNC connector.
5051	Ammonium Flow Plus Combination ISE. Range 0.9 to 9000 ppm. Complete with filling solution, one metre cable and BNC connector.
5221	Fluoride Flow Plus Combination ISE. Range 0.02 to 1,900 ppm. Complete with filling solution, one metre cable and BNC connector.
5261	Chloride Flow Plus Combination ISE. Range 1 to 35,500 ppm. Complete with filling solution, one metre cable and BNC connector.
6021	Filling Solution for Nitrate Flow plus ISE. 30 ml.
6031	Filling Solution for Potassium Flow Plus ISE. 30 ml.
6041	Filling Solution for Calcium Flow plus ISE. 30 ml.
6051	Filling Solution for Ammonium Flow Plus ISE. 30 ml.
6221	Filling Solution for Fluoride Flow Plus ISE. 30 ml.
6261	Filling Solution for Chloride Flow plus ISE.30 ml

For further information contact Sales@direct-ion.com