User Guide Surfactant Ion Selective Electrode



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Introduction

This user guide contains information on the preparation, operation and maintenance for the surfactant ion selective electrode (ISE). General analytical procedures, electrode characteristics and electrode theory are also included in this user guide. The surfactant electrode is a potentiometric sensor that indicates the endpoint in a titration of anionic or cationic surfactants in solution.

Technical Support Chemists can be consulted for assistance and troubleshooting advice. Within the United States call 1.800.225.1480 and outside the United States call 978.232.6000 or fax 978.232.6031. In Europe, the Middle East and Africa, contact your local authorized dealer. For the most current contact information, visit www.thermo.com/contactwater.

For the latest application and technical resources for Thermo Scientific Orion products, visit www.thermo.com/waterapps.

Surfactant Plastic Membrane Half-Cell ISE, Cat. No. 9342BN

The surfactant half-cell electrode must be used with the double junction reference electrode, Cat. No. 900200. The surfactant half-cell electrode is available with a BNC connector, Cat. No. 9342BN. Electrodes with a BNC connector can be used on any ISE or mV meter with a BNC connection.

The 9342BN surfactant half-cell electrode is packaged with one 475 mL bottle of 0.05 M Hyamine® 1622 titrant, one 60 mL bottle of 0.01 M sodium lauryl sulfate (SLS) standard and one 475 mL bottle of sample additive with diluted Triton® X-100.

Required Equipment

 Thermo Scientific Orion 960 titration system, equivalent titration system or a pH/mV meter with a 0.1 mV resolution and a manually controlled reagent delivery system with pipets or burets.

Surfactant electrodes can be used on any titration system or pH/mV meter with a BNC connection. The electrodes can also be used on meters with a variety of inputs when an adapter is used. Visit www.thermo.com/water for details.

- Thermo Scientific Orion surfactant half-cell electrode, Cat. No. 9342BN.
- 3. Double junction reference electrode, Cat. No. 900200.
- Magnetic stirrer or Thermo Scientific Orion stirrer probe, Cat. No. 096019. The stirrer probe can be used with 3-Star, 4-Star and 5-Star benchtop meters.
- 5. Volumetric flasks, graduated cylinders and beakers.
- 6. Distilled or deionized water.
- Double junction reference electrode filling solutions. Use inner chamber filling solution, Cat. No. 900002, and 3 M KCI electrode filling solution, Cat. No. 810007, with the double junction reference electrode.

Note: Do not use the outer chamber filling solution that ships with the double junction reference electrode because it contains interferences for surfactant measurements.

- Titrant (working solution) 0.05 M Hyamine 1622, Cat. No. 654201. The working solution is recommended for the titration of anionic surfactants. Sodium lauryl sulfate (SLS) is recommended for the titration of cationic surfactants. Alternative titrants may be used depending on the nature of the sample.
- Surfactant standard 0.01 M sodium lauryl sulfate (SLS), Cat. No. 654202. The surfactant standard is used to standardize the titrant.

- Sample additive diluted Triton X-100, Cat. No. 654203.
 The sample additive is added to all samples to keep the electrode clean.
- pH adjustor the selection of a pH adjustor depends on sample requirements. For acid samples use 0.01 M HCl and for alkaline samples use 0.1 M NaOH. The rule of thumb is acid for both anionic and cationic surfactant analysis, alkaline for polacrylates.
- Electrode rinse solution acidic distilled water. Add 50 mL of 0.1 M HCl to 1000 mL of distilled or deionized water. Store the solution in a wash bottle. For polyacrylate analysis, substitute 0.1 M NaOH for 0.1 M HCl.

Electrode Setup

Surfactant Half-Cell Electrode Preparation

Remove the surfactant electrode from the packaging. To ensure electrical continuity, shake the electrode down like a clinical thermometer. Rinse the surfactant electrode with distilled water and then soak it in a 0.0001 M sodium lauryl sulfate (SLS) solution at least 10 minutes prior to use.

To prepare the 0.0001 M SLS – add 1 mL of the 0.01 M SLS, Cat. No. 654202, to a 100 mL volumetric flask, dilute to the mark with distilled water and mix well.

Double Junction Reference Electrode Preparation

Prepare the reference electrode according to the reference electrode user guide. Fill the reference electrode with inner chamber filling solution, Cat. No. 900002, and 3 M KCl, Cat. No. 810007, as the outer chamber filling solution.

Note: Do not use the outer chamber filling solution that ships with the 900200 double junction reference electrode because it contains interferences for surfactant measurements.

Measurement Units

Any convenient unit of measurement can be used – moles per liter (M), parts per million (ppm), etc. If using an automatic titrator other than the 960 titrator, consult the manufacturer's instructions for information on units of measurement. If doing the titration manually, convert the molarity of the sample determined by the titration to another appropriate unit of measurement.

Sample Requirements

Expected sample concentration should be within the range of 1×10^{-2} to 1×10^{-5} M. Higher concentrations may be diluted. The sample volume or weight used should give an endpoint volume between 1 to 5 mL when titrated using the 960 titrator.

Anionic surfactants, sulfated and sulfonated surfactants may be titrated with Hyamine 1622. Acidify the sample pH to between 2.5 and 4.5 with HCl or 1.0 M citric acid.

Polyacrylates may be analyzed if the sample pH is adjusted to between 10 and 11 with 0.1 M NaOH.

Cationic surfactants should be titrated with an appropriate anionic species such as sodium lauryl sulfate. Acidify the sample to pH 3 with HCl or citric acid. Avoid excessive foaming that can result in erroneous results. Stir moderately. Dilute the sample as much as possible.

Sample additive, diluted Triton X-100, should be added to all samples to help keep electrode clean and working properly.

Measuring Hints

- Stir all solutions at a uniform, moderate rate and avoid excess foaming. Place a piece of insulating material, such as Styrofoam or cardboard, between the magnetic stir plate and beaker to prevent measurement errors from the transfer of heat to the sample.
- Concentrated samples (over 0.01 M) should be diluted to between 1 x 10⁻⁴ to 1 x 10⁻⁵ M before measurement.
- Always rinse the electrode with distilled water between measurements and shake the electrode to remove the water and prevent sample carryover. Do not wipe or rub the electrode sensing module.
- Add 1 mL of sample additive to every 50 mL of sample.
- Adjust the pH according to the method that is being used.
- Recondition the electrode daily before storage.
- Use slightly acidic distilled water to rinse the electrode.
- After immersing the electrode in a solution, check the electrode sensing surface for air bubbles and remove air bubbles by reimmersing the electrode in the solution and gently tapping it.
- The double junction reference electrode fill hole must be open during measurements to ensure a uniform flow of filling solution.

Electrode Storage

Surfactant Half-Cell Electrode Storage

Rinse the electrode between measurements with acidic distilled water.

Recondition the electrode when response becomes noisy, sluggish or non-reproducible. Soak the electrode in slightly acidic distilled water for one hour, and then soak the electrode in a 10-4 M SLS solution for 10 minutes.

Store the electrode dry.

Double Junction Reference Electrode Storage

The double junction reference electrode may be stored in a 0.1 M KCl solution between sample measurements and up to one week. The filling solution inside the electrode should not be allowed to evaporate, as crystallization will result.

For storage longer than one week, drain the reference electrode, flush the inside with distilled water and store the electrode dry.

Electrode Maintenance

Double Junction Reference Electrode Flushing

If the area between the electrode outer body and inner cone becomes clogged with sample or precipitate, flush the area with filling solution or distilled water.

- Hold the electrode body with one hand and use your thumb to push down on the electrode cap to drain all of the filling solution out of the electrode.
- Fill the electrode with distilled water and then push down on the cap until all the water is drained from the chamber. Repeat this procedure until all of the sample or precipitate is removed from the electrode.
- Fill the electrode with fresh filling solution up to the fill hole.
 Push down on the cap to allow a few drops of filling solution to drain out of the electrode and then replenish the lost filling solution.
- Rinse the electrode with distilled water.

Cleaning the Surfactant Sensing Module

If the electrode is exposed to high levels of interfering ions, it may drift and become sluggish in response. When this happens, restore normal performance by soaking the electrode for an hour in slightly acidic distilled water and then soaking it for ten minutes in a 10-4 M SLS solution. If soaking the electrode does not restore normal electrode performance, replace the electrode.

Titrant Preparation

Select an appropriate working solution (titrant). The 0.05 M Hyamine 1622 solution, Cat. No. 654201, is recommended for the titration of anionic surfactants. Sodium lauryl sulfate (SLS) is recommended for the titration of cationic surfactants. Alternative titrants may be used depending on the nature of the sample.

Determine the concentration of working solution needed for analysis from the appropriate Thermo Scientific Orion 960 titrator system application or from **Table 1**.

Table 1
Recommended Working Solution Concentrations

Expected Sample Concentration (M)	Recommended Working Solution Concentration (M)
0.001 to 0.05	0.05
0.0001 to 0.001	0.005
0.00001 to 0.0001	0.001

Adjustments in concentration of working solution may need to be made depending on the concentration of your sample and your method. To prepare standards with a different concentration use the following formula:

$$C_T = (C_S * V_S) / V_T$$

 C_{T} = concentration of titrant

 V_{T} = volume of titrant

 C_S = concentration of sample

 V_s = volume of sample

Dilute the 0.05 M Hyamine 1622 solution to the appropriate concentration or prepare a selected titrant as required. Standardize the titrant by titrating it against a known concentration of SLS and then back-calculating to determine the exact concentration. Use the following formula:

$$C_T = (C_{STD} * V_{STD}) / V_T$$

 C_T = concentration of titrant

 V_T = volume of titrant

 C_{STD} = concentration of standard

 V_{STD} = volume of standard

Working Solution Preparation

Select an appropriate working solution (titrant) according to the method you are using. For anionic surfactants use the Hyamine 1622 provided. For cationic surfactants, sodium lauryl sulfate or another appropriate anionic surfactant is recommended.

Determine the concentration of working solution (titrant) needed for your analysis from the appropriate 960 titration system application. If not using a 960 titration system application, calculate the necessary working solution concentration to provide an equivalence volume of between 2 to 5 mL. See **Table 1**.

The following information is based on analyzing for anionic surfactants utilizing Hyamine 1622 as the titrant and 0.01 M sodium lauryl sulfate (SLS) as the standard.

- Dilute the 0.05 M Hyamine 1622 solution to the appropriate concentration. See **Table 1** for recommended solution concentration.
- Standardize the working solution by titrating against a known concentration of SLS and then back-calculating to determine the exact concentration of the working solution. The 960 titrator does this automatically for you using technique 11, STANDARDIZE SYSTEM. See **Table 2** for recommended volumes
- Carefully pipet the volume of standard determined above into a clean beaker.
- 4 Add 50 mL of distilled water
- Add 3 mL of 0.01 M HCl. The pH should be between 2.5 and 4.5. If not, add more HCl in small increments until the correct pH is reached.
- 6. Add 1 mL of sample additive.
- Thoroughly rinse the electrodes, stirrer and dispenser probe, then lower into beaker.
- 8. Follow the display messages and action steps to standardize the working solution.

- The concentration of the working solution, in molarity, will be displayed. If greater accuracy is desired, repeat the method three or four times and average the results.
- Record the working solution concentration on the bottle. If more than one AUTO DISPENSER is in use with the 960 titrator, also record which AUTO DISPENSER was used.

Table 2 Standard Volume Matrix

Approximate Matrix	Volume of 0.01 SLS Standard (mL)	Endpoint Volume (mL)	Predose Volume
0.05	10	2	1
0.005	2	4	2
0.01	0.5	5	3

960 Titration System Displays and Actions for Working Solution Procedure

1-Identify the electrode as X-in the EA940 and return to AUTOCHEM STANDBY2-Press speed and enter 7 to begin3AUTOCHEM STANDBYPress yes4Use SAVED method?Press no5XXXXX XXXXXXXX? (technique x)Enter 116STANDARDIZE SYSTEM (technique 11)Press yes7Standard by: vol 1 weight 2Press 18PRINTOUT: short 1 long 2Press 29M result unit selected No action necessary, momentary display10Reaction ratio = x.xxxx?Press 1 and then press yes11mV READINGS by No action necessary, momentary display12TIME Press 1 STABILITY Press 2Press 5 and then press yes14BEFORE analysis stir x.x sec?Press 5 and then press yes15Continuous stirring required?Press yes16SAMPLE ID NUMBER required?Press no17DISPENSER is No action necessary, momentary display		Display	Action
7 to begin 3 AUTOCHEM STANDBY Press yes 4 Use SAVED method? Press no 5 XXXXX XXXXXXX? Enter 11 6 STANDARDIZE SYSTEM (technique x) 7 Standard by: vol 1 weight 2 8 PRINTOUT: short 1 long 2 9 M result unit selected No action necessary, momentary display 10 Reaction ratio = x.xxxx? Press 1 and then press yes 11 mV READINGS by No action necessary, momentary display 12 TIME Press 1 Press 1 STABILITY Press 2 13 x.x sec Press 5 and then press yes 14 BEFORE analysis stir x.x sec? 15 Continuous stirring required? Press no 16 SAMPLE ID NUMBER required? Press no	1	-	in the EA940 and return to
4 Use SAVED method? Press no 5 XXXXX XXXXXXXX? Enter 11 6 STANDARDIZE SYSTEM (technique x) 7 Standard by: vol 1 weight 2 8 PRINTOUT: short 1 long 2 9 M result unit selected No action necessary, momentary display 10 Reaction ratio = x.xxxx? Press 1 and then press yes 11 mV READINGS by No action necessary, momentary display 12 TIME Press 1 Press 1 STABILITY Press 2 13 x.x sec Press 5 and then press yes 14 BEFORE analysis stir x.x sec? 15 Continuous Press yes 16 SAMPLE ID NUMBER required? 17 DISPENSER is No action necessary,	2	-	
5 XXXXX XXXXXXXX? (technique x) 6 STANDARDIZE SYSTEM (technique 11) 7 Standard by: vol 1 weight 2 8 PRINTOUT: short 1 long 2 9 M result unit selected No action necessary, momentary display 10 Reaction ratio = x.xxxx? Press 1 and then press yes 11 mV READINGS by No action necessary, momentary display 12 TIME Press 1 STABILITY Press 2 13 x.x sec Press 5 and then press yes 14 BEFORE analysis stir x.x sec? Press 5 and then press yes 15 Continuous stirring required? Press no 16 SAMPLE ID NUMBER required? 17 DISPENSER is No action necessary,	3	AUTOCHEM STANDBY	Press yes
(technique x) 6 STANDARDIZE SYSTEM (technique 11) 7 Standard by: vol 1 weight 2 8 PRINTOUT: short 1 long 2 9 M result unit selected No action necessary, momentary display 10 Reaction ratio = x.xxxx? Press 1 and then press yes 11 mV READINGS by No action necessary, momentary display 12 TIME Press 1 Press 1 STABILITY Press 2 13 x.x sec Press 5 and then press yes 14 BEFORE analysis stir x.x sec? 15 Continuous stirring required? 16 SAMPLE ID NUMBER required? 17 DISPENSER is No action necessary,	4	Use SAVED method?	Press no
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weight 2 8 PRINTOUT: short 1 long 2 9 M result unit selected No action necessary, momentary display 10 Reaction ratio = x.xxxx? Press 1 and then press yes 11 mV READINGS by No action necessary, momentary display 12 TIME Press 1	6		Press yes
 2 M result unit selected No action necessary, momentary display Reaction ratio = x.xxxx? Press 1 and then press yes mV READINGS by No action necessary, momentary display TIME Press 1 Press 1 STABILITY Press 2 x.x sec Press 5 and then press yes BEFORE analysis stir x.x sec? Continuous stirring required? SAMPLE ID NUMBER required? DISPENSER is No action necessary, 	7		Press 1
momentary display 10 Reaction ratio = x.xxxx? Press 1 and then press yes 11 mV READINGS by No action necessary, momentary display 12 TIME Press 1 Press 1 STABILITY Press 2 13 x.x sec Press 5 and then press yes 14 BEFORE analysis stir x.x sec? 15 Continuous Press yes 16 SAMPLE ID NUMBER required? 17 DISPENSER is No action necessary,	8	9	Press 2
11 mV READINGS by No action necessary, momentary display 12 TIME Press 1 Press 1 STABILITY Press 2 13 x.x sec Press 5 and then press yes 14 BEFORE analysis stir x.x sec? 15 Continuous Press yes 16 SAMPLE ID NUMBER required? 17 DISPENSER is No action necessary,	9	M result unit selected	
momentary display 12 TIME Press 1 STABILITY Press 2 13 x.x sec Press 5 and then press yes 14 BEFORE analysis stir x.x sec? 15 Continuous stirring required? 16 SAMPLE ID NUMBER required? 17 DISPENSER is No action necessary,	10	Reaction ratio = x.xxxx?	Press 1 and then press yes
STABILITY Press 2 13 x.x sec Press 5 and then press yes 14 BEFORE analysis Press 5 and then press yes 15 Continuous Press yes 16 SAMPLE ID NUMBER Press no 17 DISPENSER is No action necessary,	11	mV READINGS by	•
14 BEFORE analysis stir x.x sec? 15 Continuous Press yes 16 SAMPLE ID NUMBER required? 17 DISPENSER is No action necessary,	12		Press 1
stir x.x sec? 15 Continuous Press yes 16 SAMPLE ID NUMBER required? 17 DISPENSER is No action necessary,	13	x.x sec	Press 5 and then press yes
stirring required? 16 SAMPLE ID NUMBER required? 17 DISPENSER is No action necessary,	14	,	Press 5 and then press yes
required? 17 DISPENSER is No action necessary,	15		Press yes
,	16		Press no
	17	DISPENSER is	•

	Display	Action
18	AUTOMATIC Press 1 PIPET Press 2	Press 1
19	ELECTRODE No. to be used?	Enter the electrode channel number and then press yes
20	STANDARD VOLUME = xx mL	Enter the volume of standard pipeted into the beaker and press yes
21	STANDARD x.xxxxxxM?	Enter the actual lot analysis concentration of the standard that is printed on the back of the bottle and then press yes
22	TITRANT increments by	No action necessary, momentary display
23	constant mL 1 constant mV 2	Press 1
24	Constant increment mL? = xx	Enter 0.25 and then press yes
25	PRE-DOSE VOLUME x.xx mL?	Enter the amount from Table 2 and then press yes
26	MAX TOTAL TITRANT VOL = x.xx mL?	Press 6 and then press yes
27	SAVE METHOD?	Press yes
28	save as METHOD 1?	Press yes or enter desired number and then press yes
29	1:X- ELECTRODE IN SOLUTION?	Press yes to begin analysis

Analytical Techniques

A variety of analytical techniques are available to the analyst. The following is a description of these techniques.

Sample Analysis By Manual Titration

The surfactant electrode is a potentiometric endpoint indicator for the analysis of surfactants in solution. General titration procedures for the analysis of an anionic surfactant are as follows:

- Before the titration, rinse the surfactant electrode with an acid rinse solution and blot dry with a soft, lint-free tissue. Fill the double junction reference electrode with fresh filing solution, Cat. No. 900002 in the inner chamber and Cat. No. 810007 in the outer chamber.
- Connect the electrodes to the pH/mV meter and set the
 meter to the mV measurement mode. Pipet 50 mL of the
 unknown into an appropriate sized beaker. Add 3 mL of
 0.01 M HCl and 1 mL of diluted Triton X 100 to the beaker.
 Insert the electrodes into the solution and stir using a
 magnetic mixer. Wait until the mV reading is stable (drift is
 ± 1 to 2 mV/minute).
- 3. Fill a 10 mV buret with 0.05 M Hyamine 1622. Once the mV reading is stable, add Hyamine in increments of 0.5 mL to 1.0 mL at the beginning of the titration, then in increments of 0.1 mL to 0.25 mL in the region of the endpoint. Record the electrode potential after each addition of titrant. The endpoint is at that volume of titrant where the potential changes dramatically in the positive direction. Continue the titration 1 to 2 mL past the endpoint. On standard coordinate graph paper, plot milliliters of Hyamine added versus millivolt readings. The point of greatest inflection is taken as the endpoint. Calculate the unknown surfactant concentration as follows:

$$C_{unknown} = (C_{titrant} * V_{titrant}) / V_{unknown}$$

 $C_{unknown}$ = concentration of the unknown

 $C_{titrant}$ = concentration of the titrant (Hyamine 1622)

V_{titrant} = volume of titrant in milliliters

 $V_{unknown}$ = volume of unknown in milliliters

This basic procedure may be modified depending on the sample concentration and method used.

Sample Analysis Using the 960 Titration System

The surfactant electrode is an endpoint indicator. The titration technique of choice is the second derivative analysis. Alternatively the first derivative technique may be used.

Two examples of methods using the 960 titrator for the analysis of anionic surfactants are included. Different types of samples may require method modification or alternative sample preparation.

Sodium Lauryl Sulfate In Toothpaste

The second derivative technique is used for the calibration of SLS in toothpaste. The toothpaste is weighed, dispersed in water, and analyzed directly. No extraction procedures are required.

Typical Results

Five analyses yielded: Mean Value: = 1.2 (%w/w)

Rel. Std. Dev.: = 0.83% of mean value

Solution Preparation

Use the 0.05 M Hyamine 1622 titrant. No dilution is necessary. For best results standardize the titrant with the 0.01 M SLS standard provided.

Equipment Preparation

Plug prepared electrodes into input 1 or 2. Identify electrode as X- in the EA940 meter. Flush the AUTO DISPENSER with 20.0 mL of deionized water. Flush the AUTO DISPENSER with 5 mL working solution, 0.05 M Hyamine 1622.

Sample Preparation

Accurately weigh about 1.0 grams of toothpaste into an analysis beaker and enter the exact weight into the 960 titrator. Add 50 mL of distilled or deionized water.

Add 3 mL of 0.01 M HCl, (sample pH should be between 2.5 and 4.5). Add 1 mL of sample additive.

Note: Set stirrer speed to the 9 o'clock position. Rinse the electrodes, stirrer and dispenser probe thoroughly with acidic distilled water after each analysis.

Writing Method

Parameter	Entry
Technique	7, second derivative
Printout	Long
Result unit	% w/w
Reaction ration	1.000
Blank value	0.0000
mV readings by	Time
Reading criterion	3.0 seconds
Before analysis stir time	40.0 seconds
Continuous stirring	Yes
Sample ID number	Yes
Dispenser type	Automatic
Electrode No. 1 to be used	YES
Titrant concentration	0.0500 M
Titrant increment by	mV
Constant increment	10.0
Pre-dose volume	0.5 mL
Maximum total titrant	2.000 mL
End titration at	Endpoint
1 endpoint expected	Yes
Sample weight	Enter actual weight

% Anionic Surfactant Concentration In Detergents

% w/w anionic surfactant concentration in detergents is measured after diluting the sample.

Typical Results

1.0 M citric acid is recommended for acidification.

For five samples:
Mean = 3.662% w/w anionic
Std. deviation = 0.45%
Analysis time = 3 min.
Solution Preparation = Use the 0.05 M Hyamine 1622 titrant.
No dilution is required.

Sample Preparation

Weigh 5 grams of detergent and dissolve in approximately 300 mL of deionized water. Add solution to a 500 mL volumetric flask and then add deionized water to the mark. Take 20 mL of sample solution, add to beaker with 1 mL of 1 M citric acid and then add 25 mL of deionized water. Add 1 mL sample additive (diluted Triton X-100). The sample weight is entered as 0.2 grams.

Writing The Method

Parameter	Entry
Technique	6, First Derivative
Printout	Long
Result unit	% w/w
Reaction ratio	1.00
Molecular weight of sample (formula weight)	80.0 (formula weight of SO ₃)
Blank value	0.0000
mV readings by	Time
Reading criterion	6 seconds
Before analysis stir time	5 seconds
Continuous stirring	Yes
Sample ID number	Yes
Dispenser type	Automatic
Titrant concentration	0.05 M
Titrant increment by	mL
Constant increment	0.1 mL
Pre-dose volume	0.0000
Maximum total titrant volume	2.5 mL
End titration at	Maximum volume

Electrode Characteristics

Electrode Response

A typical titration for anionic surfactants, utilizing the 960 as the titrator, takes between 2 and 5 minutes. Time for analysis may vary depending on the sample, titrant, method and equipment.

Reproducibility

The standard deviation for the titration of pure SLS with Hyamine 1622 was found to be 0.1% using the 960 titrator.

Limits of Detection

The lower limit of detection is approximately 10-5 M for anionic surfactants. Careful technique and selection of titrant may allow lower levels to be detected for some sample types.

Samples which have a concentration greater than 10⁻² M must be diluted prior to measurement.

Electrode Life

Each electrode will last approximately six months with normal laboratory use, but the actual lifespan of the electrode will depend on the type of samples that the electrode is used in. Before replacement, refer to the **Troubleshooting** section to make sure that the difficulties are caused by the electrode.

The electrode is warranted for six months under normal laboratory use. Proper use of the sample additive and strict adherence to cleaning and reconditioning procedures will help ensure the maximum life for the electrode.

Temperature Effects

The range of operation for the surfactant electrode is 0 to $40\,^{\circ}\text{C}$. Use at other temperatures may permanently destroy the membrane.

Interferences

Any organic anion or cation which resembles the species of interest may interfere with the measurement.

pH Effects

The operating pH range for the surfactant electrode is pH 2 to 12. Use at other pH values may adversely affect the membrane.

Ideally, for the analysis of anionic, sulfated and sulfonated surfactants, the pH should be between 2.5 and 4.5.

Other samples may require pH adjustment to different ranges. For example, analysis of polyacrylates requires adjustment to pH 10.

Theory of Operation

The surfactant electrode is an endpoint indicator for the determination of anionic surfactants in solution. It may also be used for the determination of cationic surfactants.

The reaction which takes place when titrating a sulfated or sulfonated anionic surfactant with Hyamine 1622 is as follows:

Where:

R = surfactant carbon chain

R⁴N⁺ = Hyamine ion

Troubleshooting

Follow a systematic procedure to isolate the problem. The measuring system can be divided into four components for ease in troubleshooting: meter, electrode, sample/application and technique.

Meter

The meter is the easiest component to eliminate as a possible cause of error. Thermo Scientific Orion meters include an instrument checkout procedure and shorting cap for convenience in troubleshooting. Consult the meter user guide for directions. When using the 960 titrator, make sure that the instrumentation and reagent delivery system are operating properly. See the 960 titrator user guide.

Electrode

- 1. Rinse the electrode thoroughly with distilled water.
- 2. Verify the electrode performance by performing a titration using a known standard.
- If the electrode fails this procedure, review the Measuring Hints section. Clean the electrode thoroughly as directed in the Electrode Maintenance section. Drain and refill the reference electrode with fresh filling solution.
- 4. Repeat the titration using a known standard.
- It the electrode fails this procedure again, determine whether the surfactant or reference electrode is at fault.
 To do this, substitute a known working electrode for the electrode in question and repeat the titration using a known standard.
- If the electrode passes the procedure, but measurement problems persist, the sample may contain interferences or complexing agents, or the technique may be in error.
- 7. Before replacing a faulty electrode, review this user guide and be sure to thoroughly clean the electrode; correctly prepare the electrode; use the proper filling solution and standards; correctly measure the samples and review the **Troubleshooting Checklist** section.

Sample/Application

The quality of results depends greatly upon the quality of the reagents. Always prepare fresh reagents when problems arise, it could save hours of frustrating troubleshooting! Errors may result from contamination of prepared standards or titrant, accuracy of dilution, quality of distilled water, or a mathematical error in calculating the concentrations.

The electrode and meter may operate with standards, but not with the sample. In this case, check the sample composition for interferences, incompatibilities or temperature effects.

Refer to the **Sample Requirements**, **Interferences** and pH Effects sections.

Technique

If trouble persists, review operating procedures. Review analysis sections to be sure proper technique has been followed. Verify that the expected concentration of the ion of interest is within the limit of detection of the electrode. Check the method of analysis for compatibility with your sample.

Assistance

After troubleshooting all components of your measurement system, contact Technical Support. Within the United States call 1.800.225.1480 and outside the United States call 978.232.6000 or fax 978.232.6031. In Europe, the Middle East and Africa, contact your local authorized dealer. For the most current contact information, visit www.thermo.com/contactwater.

For the latest application and technical resources for Thermo Scientific Orion products, visit www.thermo.com/waterapps.

Warranty

For the most current warranty information, visit www.thermo.com/water.

Troubleshooting Checklist

- No electrode filling solution added –
 Fill the reference electrode with filling solution up to the fill hole. Refer to the Electrode Preparation section for details.
- Incorrect electrode filling solution used –
 Refer to the Electrode Preparation section to verify that the correct electrode filling solution was used.
- Electrode junction is dry –
 Push down on the electrode cap to allow a few drops of filling solution to drain out of the electrode.
- No reference electrode present –
 The surfactant half-cell electrode require a separate reference electrode, Cat. No. 900200.
- Reagents are contaminated or made incorrectly –
 Prepare fresh reagents. Refer to the Measurement Hints
 and Analytical Techniques sections.
- Sample not completely added, diluted or poor pipeting –
 Take care when adding sample or diluent to beaker that no
 sample is splashed onto sides of beaker. Use an automatic
 pipet for best results when measuring sample by volume.
- Sample too concentrated or working solution too dilute Dilute sample or select a different concentration of working solution.
- Sample too dilute or working solution too concentrated Make sure expected sample concentration is greater than 10-5 M. Dilute the working solution
- Solutions at different temperatures –
 Allow solutions to reach the same temperature.
- Air bubble on sensing module –
 Remove air bubble by reimmersing the electrode in solution.
- Electrode not properly connected to meter Unplug and reconnect the electrode to the meter.
- Meter or stir plate not properly grounded –
 Check the meter and stir plate for proper grounding.
- Static electricity present –
 Wipe plastic parts on the meter with a detergent solution.
- Defective meter –
 Check the meter performance. See the meter user guide.

Ordering Information

Cat. No.	Description
9342BN	Surfactant half-cell electrode, BNC connector (requires separate reference electrode)
900200	Double junction reference electrode, pin tip connector
900002	Inner chamber filling solution for the double junction reference electrode, 5 x 60 mL bottles
810007	Outer chamber filling solution for the double junction reference electrode, 5 x 60 mL bottles
654201	0.05 Hyamine 1622 titrant, 475 mL bottle
654202	0.01 M sodium lauryl sulfate (SLS) standard, 60 mL bottle
654203	Sample additive, diluted Triton X-100, 475 mL bottle
654204	Non-ionic surfactant reagent, 475 mL bottle
654205	Non-ionic surfactant titrant, 475 mL bottle

Specifications

Minimum Level

1 x 10-5 M - Pure SLS that can be titrated

Maximum Level

 $5 \times 10^{-2} M$ – Pure SLS that can be titrated with 0.05 M Hyamine

pH Range

2 to 12

Temperature Range

0 to 40 °C

Electrode Resistance

5 megohms

Size - 9342BN Surfactant Half-Cell Electrode

Body Diameter: 12 mm

Body Length: 110 mm

Cap Diameter: 16 mm

Cable Length: 1 meter

^{*} Specifications are subject to change without notice

Thermo Fisher Scientific **Water Analysis Instruments**

North America 166 Cummings Center Beverly, MA 01915 USA Toll Free: 1-800-225-1480 Tel: 1-978-232-6000 Dom. Fax: 1-978-232-6015 Int'l Fax: 978-232-6031

Europe P.O. Box 254, 3860 AG Nijkerk Wallerstraat 125K, 3862 CN Nijkerk, Netherlands Tel: (31) 033-2463887 Fax: (31) 033-2460832

Asia Pacific

Blk 55, Ayer Rajah Crescent #04-16/24, Singapore 139949 Tel: 65-6778-6876 Fax: 65-6773-0836

www.thermo.com/water

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