Thermo Scientific

Acclaim Surfactant Plus

Product Manual

P/N: 065530-01 March 2013

Thermo SCIENTIFIC

Product Manual

for

Acclaim Surfactant Plus Columns

Acclaim Surfactant Plus, 3µm, Analytical Column (4.6 x 150mm), P/N 078950
Acclaim Surfactant Plus, 3µm, Analytical Column (3.0 x 150mm), P/N 078951
Acclaim Surfactant Plus, 3µm, Analytical Column (3.0 x 100mm), P/N 078952
Acclaim Surfactant Plus, 3µm, Analytical Column (2.1 x 250mm), P/N 078953
Acclaim Surfactant Plus, 3µm, Analytical Column (2.1 x 150mm), P/N 078954
Acclaim Surfactant Plus, 3µm, Analytical Column (2.1 x 100mm), P/N 078955
Acclaim Surfactant Plus, 5µm, Analytical Column (4.6 x 250mm), P/N 082767
Acclaim Surfactant Plus, 5µm, Analytical Column (4.6 x 150mm), P/N 082768

Acclaim Surfactant Plus, 5µm, Analytical PEEK column (4x150mm), P/N 078956

Acclaim Surfactant Plus, 5µm, Guard Column (4.6 x 10mm), P/N 082773
Acclaim Surfactant Plus, 5µm, Guard Column (3.0 x 10mm), P/N 078959
Acclaim Surfactant Plus, 5µm, Guard Column (2.1 x 10mm), P/N 078960

Revision History:

Revision 01, February 27, 2012, Original Publication.

Revision 02, July 10, 2012, Added 5µm analytical columns (P/N's 078948 & 078949).

Revision 03, March, 2013, Added new column format and updated part numbers. Part number 065530 replaces 065460.

For Research Use Only. Not for use in diagnostic procedures.

Safety and Special Notices

Make sure you follow the precautionary statements presented in this guide. The safety and other special notices appear in boxes.

Safety and special notices include the following:

 **SAFETY**	***Indicates a potentially hazardous situation which, if not avoided, could result in death or serious injury.***
 **WARNING**	***Indicates a potentially hazardous situation which, if not avoided, could result in damage to equipment.***
 **CAUTION**	***Indicates a potentially hazardous situation which, if not avoided, may result in minor or moderate injury. Also used to identify a situation or practice that may seriously damage the instrument, but will not cause injury.***
 **NOTE**	***Indicates information of general interest.***
IMPORTANT	***Highlights information necessary to prevent damage to software, loss of data, or invalid test results; or might contain information that is critical for optimal performance of the system.***
Tip	***Highlights helpful information that can make a task easier.***

Contents

1. Introduction

The Acclaim™ Surfactant Plus column is a state-of-the-art, high-efficiency, silica based, specialty column for the determination of different types of surfactants, including anionics, nonionics, cationics, and amphoterics, for a wide range of samples, such as consumer products, pharmaceuticals, food & beverages, environmental samples, etc.

It offers the following features:

- Ideal selectivity for simultaneous separation of anionic, nonionic, cationic, and amphoteric surfactants
- Well suited to the determination of cationic surfactants
- Excellent resolution for ethoxylated surfactants
- Compatible with various detection methods, including charged aerosol detector (CAD), mass spectrometer (MS), evaporative light scattering detector (ELSD), suppressed conductivity detector, UV-Vis detector, etc
- Capable of retaining highly hydrophilic compounds, such as hydrotropes

The Acclaim Surfactant Plus column, based on novel mixed mode chromatography technology and advanced bonding chemistry, consists of both reversed-phase and anion-exchange retention mechanisms. Its column chemistry is designed in such way that cationic, nonionic, amphoteric and anionic surfactants elute in different time frames, which is helpful for peak identification. In addition, the surface silanol activity is effectively deactivated so that cationic surfactants elute in symmetrical and efficient peaks compared to those obtained on any other columns. The advanced surface modification process ensures hydrolytically stable and reproducible surface, resulting in excellent compatibility with MS, CAD and ELSD, as well as column-to-column consistency.

Specifications and Operating Conditions

Operating pH range:	2.5 –7.5 (3.0 – 6.0 recommended)
Operating temperature:	5 – 50 °C
Flow rate:	0.60 – 1.80 mL/min for 4.6-mm i.d. column 0.30 – 0.90 mL/min for 3.0-mm i.d. column 0.15 – 0.45 mL/min for 2.1-mm i.d. column
Storage solution:	MeCN/20 mM NH_4OAc, pH5 v/v 90/10 or pure acetonitrile
Aqueous compatibility:	0 – 100% aqueous mobile phase
Organic compatibility:	0 -100% common HPLC organic solvents (max 20% THF for PEEK columns)

CAUTION

Always use buffered solution for analysis and storage.
Avoid sudden pressure surge.

Table 1 Operating pressure and flow rate specification

Particle Size	Column Dimensions	P/N	Maximum Pressure (Recommended)	Typical Flow Rate (Recommended)
3µm	2.1 x 100mm	078955	6,000 psi	0.15 – 0.45 mL/min
	2.1 x 150mm	078954	6,000 psi	0.15 – 0.45 mL/min
	2.1 x 250mm	078953	10,000 psi	0.15 – 0.45 mL/min
	3.0 x 100mm	078952	6,000 psi	0.30 – 0.90 mL/min
	3.0 x 150mm	078951	6,000 psi	0.30 – 0.90 mL/min
	4.6 x 150mm	078950	6,000 psi	0.60 – 1.80 mL/min
5µm	4.6 x 150mm	082768	6,000 psi	0.80 – 2.00 mL/min
	4.6 x 250mm	082767	6,000 psi	0.80 – 2.00 mL/min
	4.0 x 150mm (PEEK)	078956	3,500psi	0.60 – 1.20 mL/min

Table 2 Guard Cartridge and Holder information

	Particle Size	**Column Dimensions**	**P/N**	**Required Holder**
Guard	5µm	2.1 x 10mm	078960	P/N 069580
		3.0 x 10mm	078959	P/N 069580
		4.6 x 10mm	082773	P/N 069580

2. Getting Started – A Step-by-Step Procedure

It is recommended that you run the column performance test upon receiving your new Acclaim Surfactant Plus column. The purpose of such test is to ensure no damage has occurred during shipping. Steps 1 - 5 below outline the necessary steps to perform this validation test. Test the column using the conditions described on the Quality Assurance (QA) Report enclosed in the column box. Repeat the test periodically to track the column performance over time.

NOTE

Slight variations may be observed on two different HPLC systems due to differences in system electronic, plumbing, operating environment, reagent quality, column conditioning, and operator technique.

2.1 Step 1 – Visually inspect the column

Report any damage to Thermo Fisher Scientific. Depending upon the nature of the damage, we may request that you ship the damaged column back to us for a replacement.

2.2 Step 2 – Prepare mobile phase

To obtain reliable, consistent and accurate results, it requires that mobile phases are free of non-volatile, ionic or spectroscopic impurities. Therefore, Maintaining low trace impurities and low particulate matters in mobile phases helps to obtain better sensitivity, and protect the column and system components.

2.2.1 De-ionized Water

The de-ionized water used to prepare the mobile phase should be Type 1 Reagent Grade water, or HPLC Grade Water. The deionized water must be free of ionized impurities, organics, microorganisms and particulate matters. Many commercial water purifiers are designed for HPLC applications and are suitable for these applications.

NOTE

Whenever applicable, degas the aqueous component and solvent component separately before mixing them together. Excessive purging or degassing of mobile phases should be avoided because it may change mobile phase composition.

2.2.2 Solvents

The solvents used must be free from particulate, ionic or UV-absorbing impurities. Use of ultrahigh purity solvents, HPLC grade, will usually ensure that your chromatography is not affected by impurities in the solvent.

2.2.3 Mobile phase preparation

Because many surfactants have low or no chromophore, universal detectors, such as CAD and ELSD, are often used. Operation of both types of detectors requires the use of volatile mobile phases, containing ammonium acetate, ammonium formate, acetic acid, or formic acid. The quality of these buffer salts or acids are critical for good detection and only high-purity (99.9% or better) reagents should be used. Depending on specific application, the mobile phase may consist of an organic solvent (e.g. acetonitrile or methanol) and a buffer solution (e.g. ammonium acetate buffer). Both pre-mixed and proportioning valve generated mobile phases give satisfactory results. For an isocratic method, the use of proportioning valve provides better flexibility in method optimization, while the pre-mixed mobile phase provides less baseline noise and better system-to-system reproducibility. For a gradient method, mobile phase must be generated with the proportioning valve.

2.2.3.1 Preparation of 100 mM, pH5.0 ammonium acetate buffer

1. Weigh 7.78 g ammonium acetate (e.g. Fisher, LC/MS OPTIMA 99% min, A114-50 or equivalent) and 2.0 g of acetic acid (Fisher, Glacial, 99.9%, A38-500) in a 1-L reservoir bottle.
2. Add 998. 0 g of D.I. water to same bottle.
3. Sonicate the resulting solution for 10 min to remove dissolved gases.

2.2.3.2 Preparation of performance test mobile phase (acetonitrile: 100 mM, pH5.0 ammonium acetate = 50:50 v/v)

1. Weigh 391.1 g acetonitrile (HPLC grade) in a 1-L reservoir bottle.
2. Add 500.0 g of buffer prepared by the method described in "Section 2.2.3.1" to the same bottle.
3. Sonicate the resulting solution for 2 min to remove dissolved gases.

2.3 Step 3 – Set up the LC system

The column can be used on any LC system that is equipped with a LC pump, a column oven, an injector (or an auto-sampler), and a detector. For surfactants that have chromophore, a UV or a DAD detector can be used. For a nonvolatile analyte with no or very weak chromophore, an CAD or ELS detector should be used, which requires the use of a volatile mobile phase, such as ammonium acetate buffer. The system should be thoroughly primed till free of non-volatiles before use.

2.4 Step 4 – Condition the column

When a new column is used for the first time, it should be washed thoroughly with acetonitrile/100 mM ammonium acetate, pH5 (80:20, v/v) for 20 column volumes then with the mobile phase for 20 column volumes before any injection is made.

When switching to a new mobile phase, make sure that the new mobile phase is compatible with the previous mobile phase in the column to avoid column clogging due to precipitation. The column should be fully conditioned before any injection is made (e.g. 20 column volumes).

When switching from a nonvolatile (e.g. phosphate buffer) mobile phase to a volatile (e.g. ammonium acetate buffer) mobile phase, the column must be washed thoroughly off-line with acetonitrile/100 mM ammonium acetate (50:50, v/v) for at least 20 column volumes and then with acetonitrile/100 mM ammonium acetate (75:25, v/v) for 10 column volumes before equilibrated with the desired mobile phase for 20 column volumes.

2.5 Step 5 – Reproduce the chromatogram in the Quality Assurance Report

Perform the column performance test using the conditions described in the Quality Assurance Report, and compare the result with the one in the report. After the column is fully equilibrated, multiple injections should be made until the reproducible retention is obtained.

NOTE

Due to various reasons, such as difference of LC systems, mobile phases, oven temperature control, etc, you may observe slightly different retention time from that in the report.

2.6 Step 6 – Real sample analysis

Once the column performance is satisfactorily confirmed in Step 5, the column is ready for real sample analysis.

NOTE

It is required that guard columns be used with the analytical column for real-life samples. It is recommended that the column performance test be performed periodically to monitor the condition of the column.

3. Considerations in Method Development

3.1 Ionic Strength (or Buffer Concentration)

Ionic strength (buffer concentration) affects retentions of ionic or ionizable analytes. Ionic strength increase results in significant retention decrease for anionic surfactants, slight retention increase for cationic surfactants, and virtually no effect on retentions of nonionic or amphoteric surfactants. To analyze anionic surfactant containing samples, an ammonium acetate or ammonium formate buffer should be used rather than acetic or formic acid solution.

3.2 Organic Solvent

Mobile phase organic solvent content affects hydrophobic retention of all types of surfactants – higher organic solvent content results in lower retention. While the Surfactant Plus column is compatible with all common HPLC solvents, such as acetonitrile, methanol, ethanol, hexane, etc, acetonitrile is the preferred solvent because it provides higher efficiency, lower back pressure, and low UV background. On the other hand, if the detection method is CAD or ELSD, acetone can be a lower cost and lower toxicity replacement to acetonitrile.

3.3 Mobile Phase pH

Mobile phase pH has insignificant effect on the charge state of surfactants. However, the charge state of surface silanols is pH dependent, thus affecting retentions of ionic surfactant to some extent. In general, pH increase results in lower retention for anionic surfactants, higher retention for cationic surfactants, and little effect on retentions of nonionic and amphoteric surfactants. Considering all factors, the recommended operating pH range is 3 to 5.8, conveniently and effectively provided by ammonium acetate, ammonium formate, acetic acid or formic acid buffer systems.

3.4 Buffer Types

CAUTION

The Acclaim Surfactant Plus must always be used or stored in buffered mobile phases.

This column is designed for applications using volatile buffers, such as ammonium acetate, which is compatible with CAD, ELSD, MS and UV at (>225 nm). The column can be also used with phosphate buffers.

When analyzing UV-active surfactants, UV detection combined with phosphate buffer may be used. Whenever possible, set the UV detection at multiple wavelengths including one at 210 nm. When dealing with analytes with no chromophore, CAD or ELS detection combined with a volatile buffer (e.g. ammonium acetate) should be considered. Make sure mobile phase is free of non-volatiles and all channels of LC system are thoroughly washed with non-volatile mobile phase components. When dealing with a mixture of analytes with and without chromophore, it is beneficial to put UV and CAD (or ELS) detectors in serial right following the analytical columns. In this case, it requires the use of a volatile mobile phase (e.g. ammonium acetate). Due to the use of acetate in mobile phase, UV detection is u set at a wavelength greater than 225 nm.

3.5 Isocratic vs. Gradient

The Acclaim Surfactant Plus column is fully compatible with isocratic and gradient conditions. Because surfactants are much different in hydrophobicity and/or charge, a gradient method is usually advantageous to resolve different types of surfactants within the same analysis. In this case, the gradient condition provided in the lot qualification test (shipped with the column) is a good starting point. On the other hand, it is sometimes easier to develop an isocratic method for certain surfactants so that the analysis time can be shortened for higher throughput and simpler system requirement.

4. Column Care

4.1 Mobile phases

Mobile phase must be buffered. All mobile phases should be freshly prepared and used for no longer than five days. No phosphate buffers older than three days should be used. All chemicals and solvents should be at the highest available quality. In-liner filters are recommended.

4.2 Guard cartridges

When analyzing real life samples, a guard cartridge must be used with the analytical column, and replaced periodically depending on the nature of the sample. Failing to do so will result in rapid column deterioration and premature column failure.

4.3 Column storage

The column may be stored in mobile phase for a short period of time, such as overnight. For long-term storage, use acetonitrile/20 mM ammonium acetate, pH5 (90:10 v/v) or pure acetonitrile as the storage solution.

4.4 Operating pH: pH 2.5 to 7.5

While the pH limit for the Acclaim Surfactant Plus is 2.5 to 7.5, it is highly recommended that the column be used between pH 3 and pH 6, which is covered by buffer range of ammonium acetate and ammonium formate buffers.

4.5 Operating temperature: 5 to 50 ºC

For surfactant analysis, separation can usually be optimized by adjusting mobile phase ionic strength and/or organic solvent content. The typical temperature for routine analysis is between 20 to 30 °C. To extend the column lifetime, elevated temperature is not recommended and should be avoided.

4.6 Flow rate and pressure

The operating flow rates are column inner diameter dependent (0.60 – 1.80 mL/min for 4.6-mm i.d. column; 0.30 – 0.90 mL/min for 3.0-mm i.d. column; 0.15 – 0.45 mL/min for 2.1-mm i.d. column). The mid-point value of the flow rate range is usually a good flow rate to use. The operating pressure limit depends on column length and the pressure that the column was packed at. For example, the pressure limit of a 150-mm column is 6,000 psi provided that the flow rate limit is not exceeded. It is important not to expose the column to pressure surge.

4.7 Column washing procedure

When the column washing practice is needed, such as deteriorated column performance and/or excessively high backpressure, the following procedure can be tried to restore the column performance.

For a 3.0-mm i.d. column used in ammonium acetate buffer:

1. Wash the column with 20 mM ammonium acetate solution/acetonitrile v/v 50/50 for 5 column volumes at a flow rate of 0.3 mL/min
2. Wash the column with 200 mM ammonium acetate solution/acetonitrile v/v 80/20 for 20 to 50 column volumes at a flow rate of 0.3 mL/min (to remove strongly retained ionic species).
3. Wash the column with 20 mM ammonium acetate solution/acetonitrile v/v 20/80 for 20 column volumes at a flow rate of 0.3 mL/min (to remove strongly retained hydrophobic compounds).
4. Equilibrate the column with the mobile phase for a minimum of 20 column volumes.

NOTE

Above washing can be conveniently performed by in-situ proportional valve mixing the following three components using acetonitrile, DI water and 200 mM ammonium acetate solution.

NOTE

If above treatments fail to improve the column performance, replace it with a new one.

5. Frequently Asked Questions

1. What is the Acclaim Surfactant Plus?
   The Acclaim Surfactant Plus column is a state-of-the-art, high-efficiency, silica column designed for the determination of different types of surfactants, including anionics, nonionics, cationics, and amphoterics, for a wide range of samples, such as consumer products, pharmaceuticals, food & beverages, environmental samples, and etc.

2. Why do I need the Acclaim Surfactant Plus?
   The Acclaim Surfactant plus is the most versatile, high-performance specialty column for determinations of surfactants. Surfactants are widely used in the consumer product, industrial, agricultural, and pharmaceutical markets, in products as diverse as pesticides, detergents, petroleum products, cosmetics, and pharmaceuticals. Their separation and identification can be difficult due to both the diversity of surfactants and the complexity of sample matrices. Its column chemistry is designed in such way that cationic, nonionic, amphoteric and anionic surfactants elute in different time frames, which is helpful for peak identification. In addition, the surface silanol activity is effectively deactivated so that cationic surfactants elute in much improved symmetrical and efficient peaks compared to those obtained on any other columns. The advanced surface modification process ensures hydrolytically stable and reproducible surface, resulting in excellent compatibility with MS, CAD and ELSD, as well as column-to-column consistency.

3. How does the Acclaim Surfactant Plus work?
   The Acclaim Surfactant Plus column, based on novel mixed mode chromatography technology and advanced bonding chemistry, consists of both reversed-phase and anion-exchange retention mechanisms. It provides hydrophobic retention and electrostatic repulsion to cationic surfactants, hydrophobic retention and electrostatic attraction to anionic surfactants, and hydrophobic retention only to nonionic and amphoteric surfactants. As the result, different types of surfactants elute in the order of cationic, nonionic, amphoteric and anionic surfactants.

4. When do I need the Acclaim Surfactant Plus?
   You should consider using Acclaim Surfactant Plus when you are working with the applications that involve determinations of surfactants, including anionic, cationic, nonionic and amphoteric surfactants.

5. What factors should I consider for method development using Acclaim Surfactant Plus?
   Organic solvent content and ionic strength (or buffer concentration) in the mobile phase are two most effective and convenient factors for optimizing chromatographic conditions. Sometimes, mobile phase pH may affect retention and selectivity (refer to Section 3 - Considerations in Method Development).

6. What mobile phases should I use with Acclaim Surfactant Plus?
   The Acclaim Surfactant Plus column is designed for applications using volatile buffers, such as ammonium acetate, which is compatible with CAD, ELSD, MS and UV at (>225 nm). The column can be also used with phosphate buffers when required by applications.

   When analyzing UV-active surfactants, UV detection combined with phosphate buffer may be considered. Whenever possible, set the UV detection at multiple wavelengths including one at 210 nm. When dealing with analytes with no chromophore, CAD or ELS detection combined with volatile buffer (e.g. ammonium acetate) should be considered. Make sure mobile phase is free of non-volatiles and all channels of LC system are thoroughly washed with non-volatile mobile phase components. When dealing with a mixture of analytes with and without chromophore, it is beneficial to put UV and CAD (or ELS) detectors in serial right following the analytical columns. In this case, it requires the use of a volatile mobile phase (e.g. ammonium acetate). Due to the use of acetate in mobile phase, UV detection is often set at a wavelength greater than 225 nm.
   When using suppressed conductivity for detecting cationic surfactants, formic acid or acetic acid should be used with acetonitrile in the mobile phase.

7. What should I do before starting using Acclaim Surfactant Plus?
   Before using the column, please read this User Guide carefully, and contact Thermo Fisher Technical Support if you have any questions regarding the use of this column.

8. How to store an Acclaim Surfactant Plus column?
   The column can be stored in mobile phase for short period of time. It is required to use acetonitrile/10 mM ammonium acetate, pH5 (90:10 v/v) or pure acetonitrile as the long-term storage solution.

9. Why is Acclaim Surfactant Plus “the column of choice” for analyzing cationic surfactants?
   Determination of cationic surfactants is challenging on most silica based reversed-phase column due to the undesired ion-exchange interactions between surfactant silanols and cationic surfactants. As the result, cationic surfactants often elute as tailing peaks with excessive retention. Mobile phases containing high concentration of salts (e.g. NaCl, $NaClO_4$, etc) are used to improve peak shape. However the resulting methods are incompatible with MS, CAD, or ELSD. On Acclaim Surfactant Plus, the surface silanol activity is effectively deactivated so that cationic surfactants elute in much improved symmetrical and efficient peaks compared to those obtained on any other columns. Therefore, Acclaim Surfactant Plus is the most suitable column for the determination of cationic surfactants among all columns in the market.

10. Compared to reversed-phase columns (e.g. C18), why is the Acclaim Surfactant Plus advantageous for determination of anionic and nonionic surfactants?
Although reversed-phase columns can be used to determine anionic or nonionic surfactants, peak co-elution can often be observed due to unmatched selectivity. The Acclaim Surfactant Plus column provides both reversed-phase and anion-exchange retention mechanisms. It provides hydrophobic retention and electrostatic repulsion to cationic surfactants, hydrophobic retention and electrostatic attraction to anionic surfactants, and hydrophobic retention only to nonionic and amphoteric surfactants. As the result, different types of surfactants elute in the order of cationic, nonionic, amphoteric and anionic surfactants. Moreover, the selectivity can be optimized by adjusting mobile phase organic solvent content as well as buffer concentration to prevent co-elution between different surfactants or between other interferences and the surfactant of interest.

11. Can I use Acclaim Surfactant Plus to determine nonionic ethoxylated surfactants?
Yes. Acclaim Surfactant Plus provides excellent resolution between oligomers with different number of ethylene oxide units in various nonionic ethoxylated surfactants such as Triton X-100, polyethylene glycols (MW < 1500 Dalton), polyethylene glycol alkyl ethers, etc (See Section 6 – Applications for reference).

12. What detectors can be used with the Acclaim Surfactant Plus?
This column is compatible with commonly used detectors for HPLC applications, including CAD, ELSD, UV, MS, RI, etc. Volatile mobile phase must be used when CAD, ELSD, or MS is used as the detection method. For routine surfactant analysis, CAD is the preferred detector due to its higher sensitivity and better RSD.

13. Can suppressed conductivity detection be used with the Acclaim Surfactant Plus?
Cationic surfactants may be detected selectively and sensitively by suppressed conductivity detection (SCD) as shown in Figure 6.16. For compatibility with ion chromatography systems and detectors, Thermo Scientific recommends P/N 078956, packed in all-PEEK column hardware, and designed for this application. Formic acid or acetic acid is the recommended electrolytes for this application. Use a CSRS 300 suppressor in external water mode with mobile phases containing up to 40% acetonitrile. Use a CMMS 300 for chemical mode suppression with mobile phases containing any amount of acetonitrile. For complete instructions, refer to the manuals for the suppressors.

14. Must I use guard cartridge with an Acclaim Surfactant Plus analytical column?
Yes. Guard cartridges protect the more expensive analytical column by trapping highly retained components and particulates from the mobile phase or the sample.

15. What should I do if the column shows deteriorated performance?
Make sure the proper connections. Refer to “Section 4.7 Column washing procedure” for details.

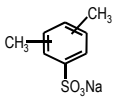
16. What should I do if the column exhibits excessively high backpressure?
First, make sure that the mobile phase is freshly prepared and filtered before use and that the sample is free of particulates. Then, back-flush the column for certain amount of time (e.g. 10 to 30 min) while monitoring the change in column pressure. If problem persists, replace with a new column.

6. Applications

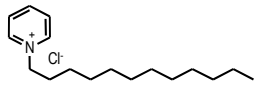
6.1 Mixture of Cationic, Nonionic, Amphoteric and Anionic Surfactants

Hydrotrope, Cationic, Nonionic, Amphoteric and Anionic Surfactants

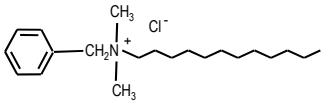
Sodium Xylene Sulfonate



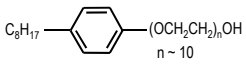
Laurylpyridinium Chloride



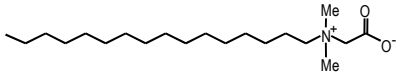
Lauryldimethylbenzyl Ammonium Chloride



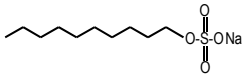
Triton X-100



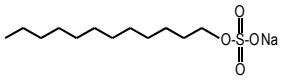
Cetyl Betaine



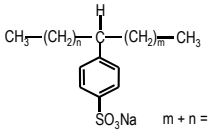
Sodium Decyl Sulfate



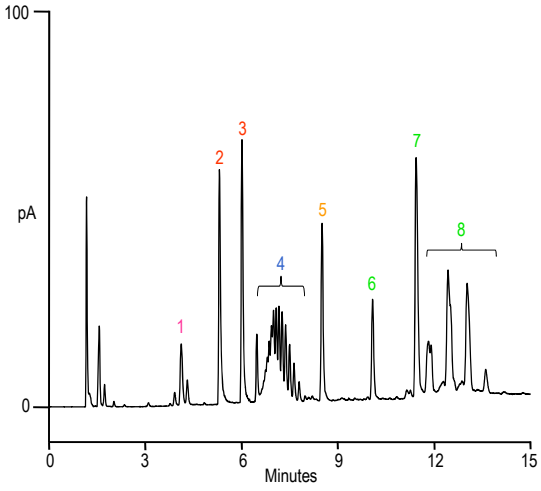
Sodium Dodecyl Sulfate (SDS)



Sodium Dodecylbenzene Sulfonate (LAS)



Separation of Cationic, Nonionic, Amphoteric & Anionic Surfactants



Column: Acclaim Surfactant Plus, 3 µm
Dimensions: 3.0 x 150 mm
Gradient:

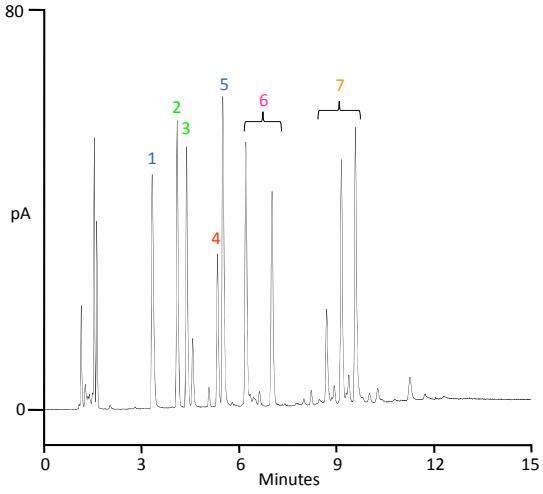
Time (min)	Acetonitrile	0.1 M Ammonium Acetate, pH5
-8	25	75
0	25	75
10	80	20
15	80	20

Temperature: 30 °C
Flow Rate: 0.6 mL/min
Inj. Volume: 5 µL
Detection: Corona *ultra* (Gain = 100 pA; Filter = med; Neb Temp = 20°C)
Peaks: (100 - 400 µg/mL each)

1. Xylene sulfonate
2. Laurylpyridinium
3. Lauryldimethylbenzyl-ammonium
4. Triton X-100
5. Cetyl betaine
6. Decyl sulfate
7. Dodecyl sulfate
8. Linear alkylbenzene sulfonate (LAS)

6.2 Cationic Surfactants

Separation of Cationic Surfactants



Column: **Acclaim Surfactant Plus**, 3 µm
Dimensions: 3.0 x 150 mm
Gradient:

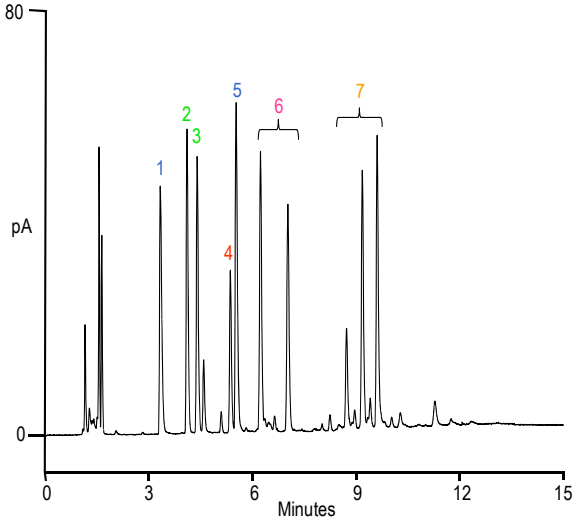
Time (min)	Acetonitrile	0.1 M Ammonium Acetate, pH5
-8	35	65
0	35	65
8	85	15
15	85	15

Temperature: 30 °C
Flow Rate: 0.6 mL/min
Inj. Volume: 5 µL
Detection: Corona *ultra* (Gain = 100 pA; Filter = med; Neb Temp = 20°C)

Peaks: (200 - 400 µg/mL each)
1. Lauryl pyridinium
2. Lauryldimethylbenzyl ammonium
3. Octylphenoxyethoxyethyl-dimethylbenzyl ammonium
4. Cetyltrimethylammonium
5. Cetylpyridinium
6. Diethyl heptadecyl imizolinium
7. Dimethyldihydrogenated tallow ammonium (2M2HT)

6.3 Hydrophobic Alkyl Quaternary Amines

Separation of Cationic Surfactants



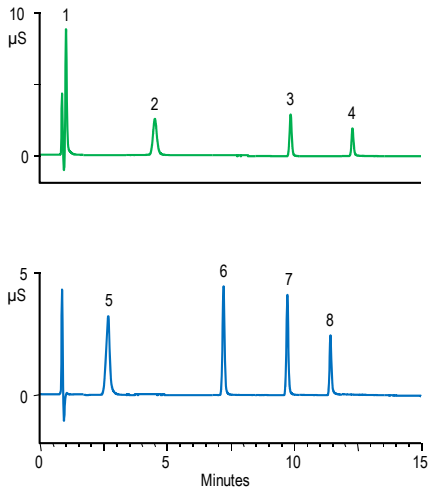
Column: Acclaim Surfactant Plus, 3 µm
Dimensions: 3.0 x 150 mm
Gradient:

Time (min)	Acetonitrile	0.1 M Ammonium Acetate, pH5
-8	35	65
0	35	65
8	85	15
15	85	15

Temperature: 30 °C
Flow Rate: 0.6 mL/min
Inj. Volume: 5 µL
Detection: Corona *ultra* (Gain = 100 pA; Filter = med; Neb Temp = 20°C)

Peaks: (200 - 400 µg/mL each)
1. Lauryl pyridinium
2. Lauryldimethylbenzyl ammonium
3. Octylphenoxyethoxyethyl-dimethylbenzyl ammonium
4. Cetyltrimethylammonium
5. Cetylpyridinium
6. Diethyl heptadecyl imizolinium
7. Dimethyldihydrogenated tallow ammonium (2M2HT)

Separation of Alkyl Quaternary Amines Using Conductivity Detection



Column: Acclaim Surfactant Plus, 3.0 µm
Dimension: 3.0 x 150 mm
System: ICS 3000
Mobile phases: A: Acetonitrile
B: 100 mM Formic acid
C: Water

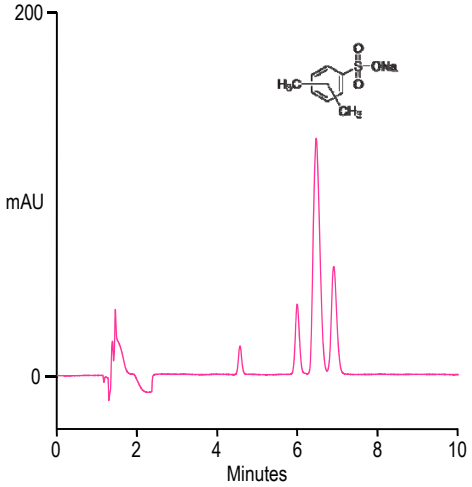
Gradient times:

Time (min)	%A	%B	%C
-12	5	5	90
0	5	5	90
12	40	5	55
20	40	5	55

Flow rate: 0.500 mL/min
Injection: 5 µL
Temperature: 25 °C
Detection: Conductivity with blank subtraction
Suppressor: CSRS300-2mm (external water 1.0 mL/min, current = 8 mA)
Peaks:
1. Tetrabutylammonium
2. Tetrapentylammonium
3. Tetrahexylammonium
4 Tetraheptylammonium
5. Decyl-trimethylammonium
6. Dodecyl-trimethylammonium
7. Tetradecyl-trimethylammonium
8. Hexadecyl-trimethylammonium

6.4 Xylene Sulfonate (Hydrotrope)

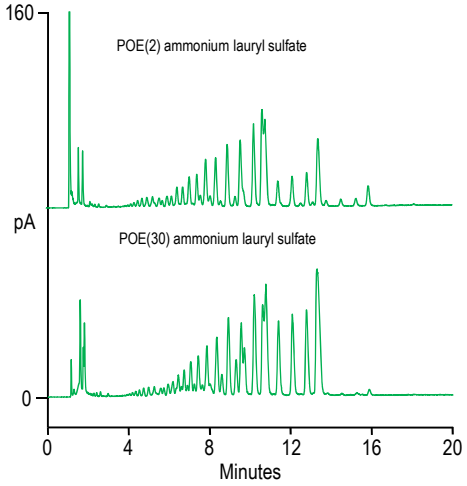
Determination of Xylene Sulfonates



Column:	Acclaim Surfactant Plus, 3 µm
Dimensions:	3.0 x 150 mm
Mobile Phase:	30/70 v/v CH_3CN/20 mM (total) NH_4OAc, pH5
Temperature:	30 °C
Flow Rate:	0.60 mL/min
Inj. Volume:	2 µL
Detection:	UV, 225 nm
Sample:	Sodium xylene sulfonate (1 mg/mL)

6.5 Laureth Sulfates

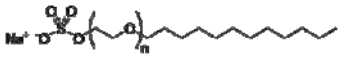
Profile of Ethoxylated Lauryl Sulfates



Column:	Acclaim Surfactant Plus, 3 µm
Dimensions:	3.0 x 150 mm
Gradient:	

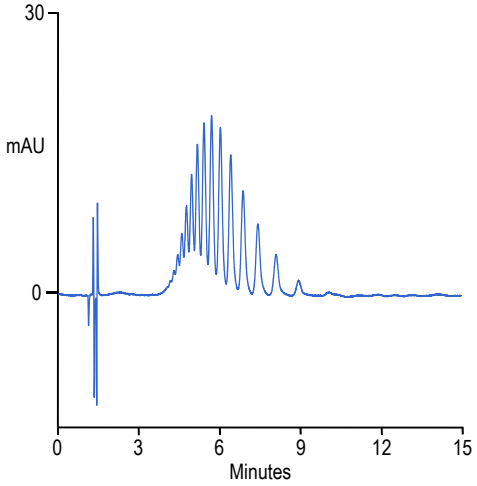
Time (min)	Acetonitrile	0.1 M Ammonium Acetate, pH5
-10	45	55
0	45	55
15	75	25
20	75	25

Temperature:	30 °C
Flow Rate:	0.60 mL/min
Inj. Volume:	2 µL
Detection:	Corona *ultra* (Gain = 100 pA; Filter = med; Neb Temp = 20°C)
Sample:	10 mg/mL each

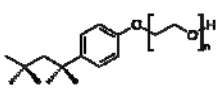


6.6 Triton X-100

Triton X-100

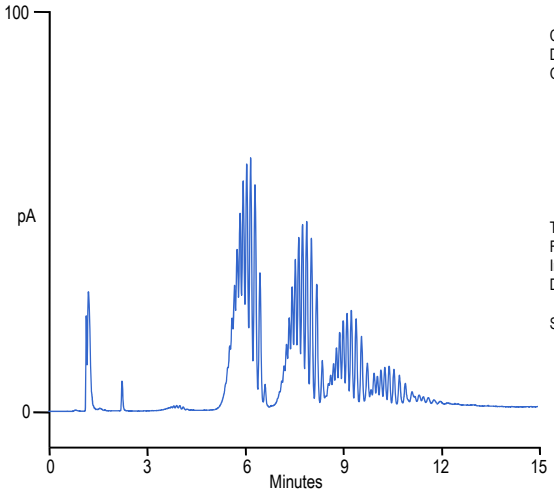


Column:	Acclaim Surfactant Plus, 3 µm
Dimensions:	3.0 x 150 mm
Mobile Phase:	40/60 v/v CH_3CN/10 mM (total) NH_4OAc, pH5
Temperature:	30 °C
Flow Rate:	0.60 mL/min
Inj. Volume:	2 µL
Detection:	UV, 225 nm
Sample:	Triton X-100 (1 mg/mL)



6.7 Zonyl FSO Fluorosurfactant

Zonyl FSO Fluorosurfactant



Column: Acclaim Surfactant Plus, 3 µm
Dimensions: 3.0 x 150 mm
Gradient:

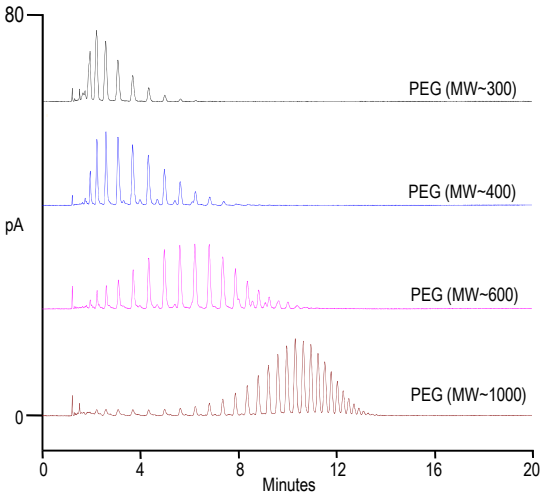
Time (min)	Acetonitrile	0.1 M Ammonium Acetate, pH5
-8	30	70
0	30	70
10	60	40
15	60	40

Temperature: 30 °C
Flow Rate: 0.6 mL/min
Inj. Volume: 5 µL
Detection: Corona *ultra* (Gain = 100 pA; Filter = med; Neb Temp = 20°C)
Sample: Zonyl FSO Fluorosurfactant (10 mg/mL)

$(C_2H_4O)_x(CF_2)_yC_2H_5OH$

6.8 Polyethylene Glycols (PEGs)

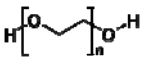
Polyethylene Glycols (PEGs)



Column: Acclaim Surfactant Plus, 3 µm
Dimensions: 3.0 x 150 mm
Gradient:

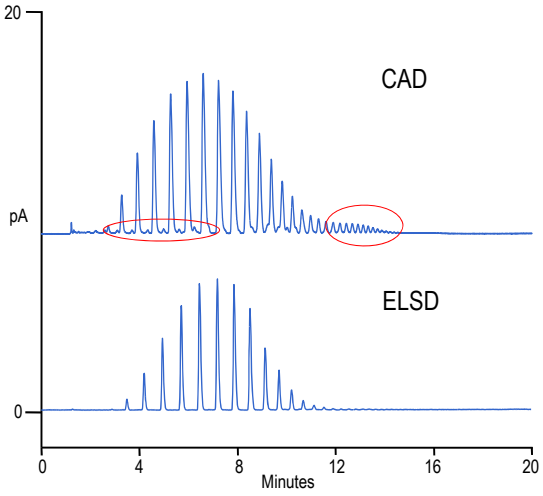
Time (min)	Acetonitrile	0.1 M Ammonium Acetate, pH5
-8	2	98
0	2	98
20	20	80

Temperature: 30 °C
Flow Rate: 0.6 mL/min
Inj. Volume: 2 µL
Detection: Corona *ultra* (Gain = 100 pA; Filter = med; Neb Temp = 20°C)
Sample: (2.5 mg/mL each)
PEG (MW~300)
PEG (MW~400)
PEG (MW~600)
PEG (MW~1000),



6.9 Polyethylene Glycol Monoethyl Ether (MW ~ 550)

Sensitivity Comparison CAD vs. ELSD



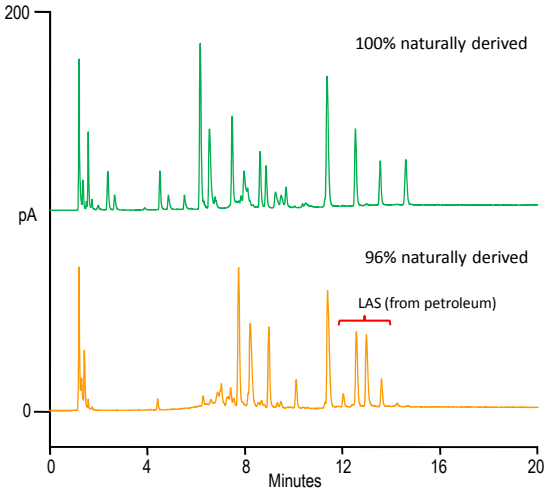
Column: Acclaim Surfactant Plus, 3 µm
Dimensions: 3.0 x 150 mm
Gradient:

Time (min)	Acetonitrile	0.1 M Ammonium Acetate, pH5
-8	2	98
0	2	98
20	20	80

Temperature: 30 °C
Flow Rate: 0.6 mL/min
Inj. Volume: 2 µL
Detection: Corona *ultra* (Gain = 100 pA; Filter = med; Neb Temp = 20°C)
Sedex-85 ELSD (Gain = 10; Evap. Temp = 50°C
Sample: PEG Monoethyl ether (MW ~ 550), 5 mg/mL

6.10 Laundry Detergents

Laundry Detergents



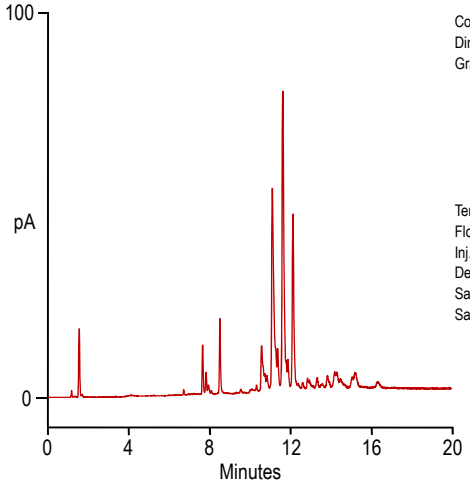
Column: Acclaim Surfactant Plus, 3 µm
Dimensions: 3.0 x 150 mm
Gradient:

Time (min)	Acetonitrile	0.1 M Ammonium Acetate, pH5.2
-8	25	75
0	25	75
10	80	20
20	80	20

Temperature: 30 °C
Flow Rate: 0.6 mL/min
Inj. Volume: 2 µL
Detection: Corona *ultra* (Gain = 100 pA; Filter = med; Neb Temp = 20°C)
Sample: Llaundry detergents (20x dilution)

6.11 Fabric Softener

Fabric Softener



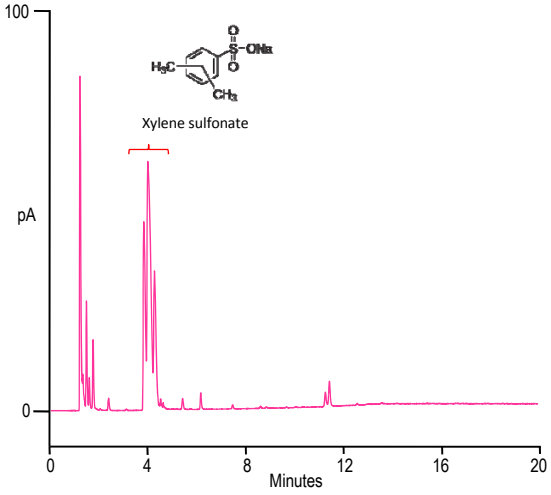
Column: Acclaim Surfactant Plus, 3 µm
Dimensions: 3.0 x 150 mm
Gradient:

Time (min)	Acetonitrile	0.1 M Ammonium Acetate, pH5
-10	30	70
0	30	70
10	85	15
20	85	15

Temperature: 30 °C
Flow Rate: 0.60 mL/min
Inj. Volume: 1 µL
Detection: Corona *ultra* (Gain = 100 pA; Filter = med; Neb Temp = 20°C)
Sample: Fabric Softener (40x dilution)
Sample Preparation:
1. Dissolve 0.50 g of sample in 20 mL of 75% ethanol
2. Sonicate the mixture for 5 min
3. Filter the suspension through the 0.2-µm membrane filter

6.12 Hand Soap

Liquid Hand Soap



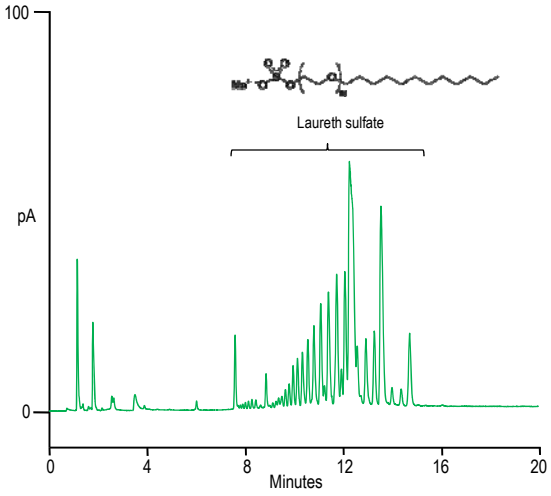
Column: Acclaim Surfactant Plus, 3 µm
Dimensions: 3.0 x 150 mm
Gradient:

Time (min)	Acetonitrile	0.1 M Ammonium Acetate, pH5.2
-8	25	75
0	25	75
10	80	20
20	80	20

Temperature: 30 °C
Flow Rate: 0.6 mL/min
Inj. Volume: 1 µL
Detection: Corona *ultra* (Gain = 100 pA; Filter = med; Neb Temp = 20°C)
Sample: Foaming hand soap (40x dilution and filtered)

6.13 Shampoo

Shampoo



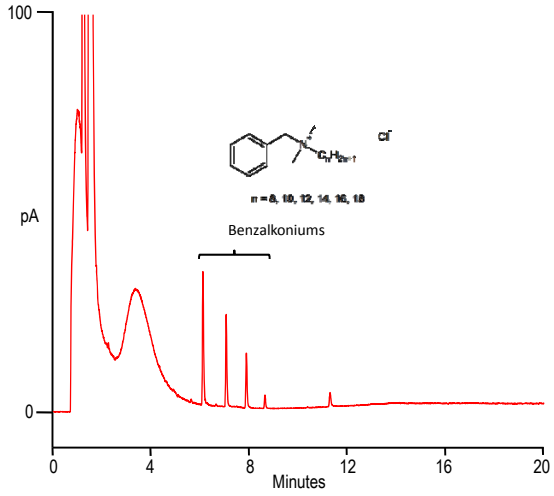
Column: Acclaim Surfactant Plus, 3 µm
Dimensions: 3.0 x 150 mm
Gradient:

Time (min)	Acetonitrile	0.05 M Ammonium Acetate, pH5.2
-8	25	75
0	25	75
10	80	20
20	80	20

Temperature: 30 °C
Flow Rate: 0.6 mL/min
Inj. Volume: 2 µL
Detection: Corona *ultra* (Gain = 100 pA; Filter = med; Neb Temp = 20°C)
Sample: Shampoo (40x dilution and filtered)

6.14 Eye Drops

Eye Drops



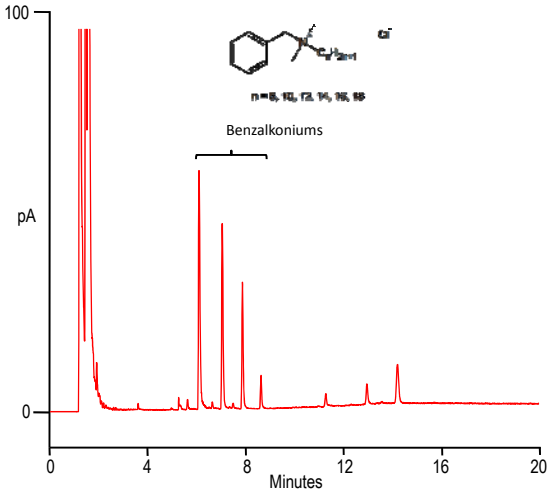
Column: Acclaim Surfactant Plus, 3 µm
Dimensions: 3.0 x 150 mm
Gradient:

Time (min)	Acetonitrile	0.1 M Ammonium Acetate, pH5.2
-8	25	75
0	25	75
10	80	20
20	80	20

Temperature: 30 °C
Flow Rate: 0.6 mL/min
Inj. Volume: 5 µL
Detection: Corona *ultra* (Gain = 100 pA; Filter = med; Neb Temp = 20°C)
Sample: Nasal spray (filter and inject)

6.15 Nasal Spray

Nasal Spray



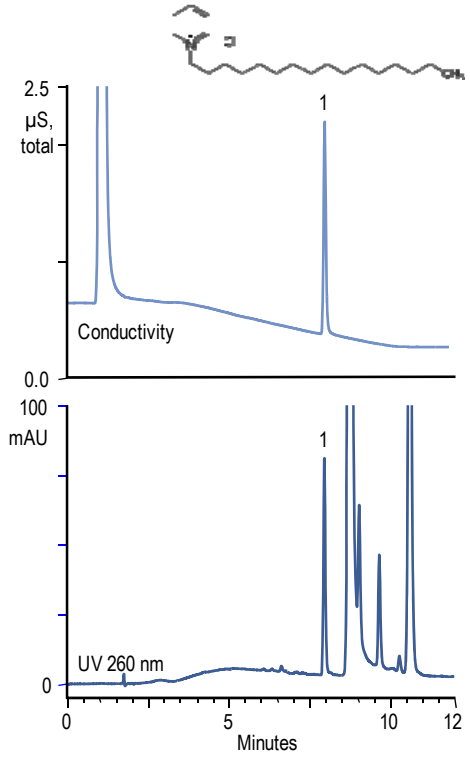
Column: Acclaim Surfactant Plus, 3 µm
Dimensions: 3.0 x 150 mm
Gradient:

Time (min)	Acetonitrile	0.1 M Ammonium Acetate, pH5.2
-8	25	75
0	25	75
10	80	20
20	80	20

Temperature: 30 °C
Flow Rate: 0.6 mL/min
Inj. Volume: 5 µL
Detection: Corona *ultra* (Gain = 100 pA; Filter = med; Neb Temp = 20°C)
Sample: Nasal spray (filter and inject)

6.16 Mouthwash

Selective Detection of Cationic Surfactant in Mouthwash with Suppressed Conductivity Detection



HPLC Conditions

Column:	Acclaim Surfactant Plus
Dimensions:	5 µm, 4.0 x 150 mm (PEEK)
System:	ICS 3000
Mobile Phases:	A: Acetonitrile B: 100 mM Formic acid C: Water

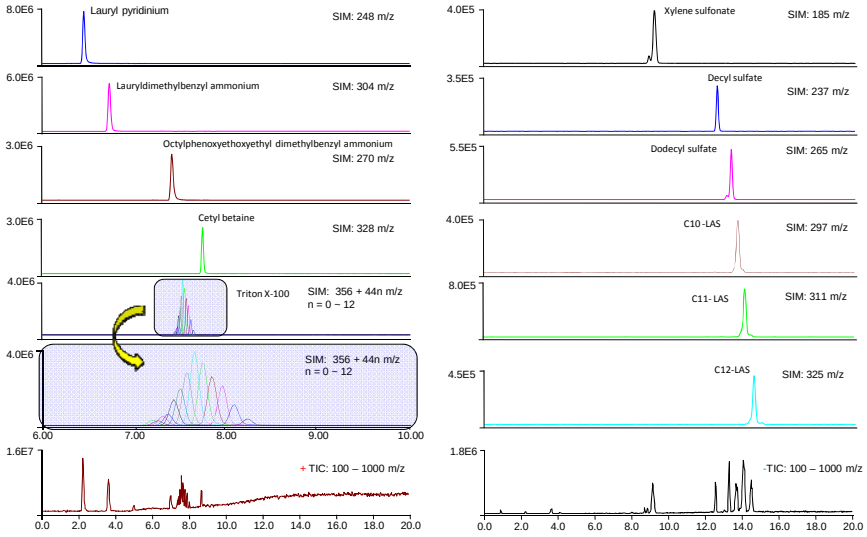
Gradient Program:

Time (min)	%A	%B	%C
-8	5	5	90
0	5	5	90
7	40	5	55
12	40	5	55

Flow rate:	0.800 mL/min
Injection:	5 µL
Temperature:	25 °C
Detection:	UV 260 nm Conductivity
Suppressor:	CSRS300-4mm External water 1.0 mL/min 12 mA
Peaks:	1. Cetylpyridinium

6.17 Separation and Identification of Different Surfactants by LC-ESI-MS

Simultaneous Analysis of Cationic, Nonionic, Amphoteric and Anionic Surfactants by LC-ESI-MS



Chromatographic Conditions

System:	UltiMate 3000 RSLC
Column:	Acclaim Surfactant Plus, 3-µm
Dimension:	2.1x150-mm
Temp:	30 °C
Mobile phase:	A: D.I. water
	B: 100mM ammonium acetate, pH 5
	C: Acetonitrile

Gradient:

Time (min)	%A	%B	%C
-10	65	5	30
0	65	5	30
1	65	5	30
8	10	5	85
20	10	5	85

Flow Rate: 0.3 mL/min

MS Conditions

System:	MSQ Plus single quadrupole MS
Interface:	Electrospray ionization (ESI)
Probe Temp.:	450 °C
Needle Vol.:	3 kV
Nebulizer Gas:	Nitrogen at 85 psi
Scan Mode:	Polarity switching full scan
	100 – 1000 m/z