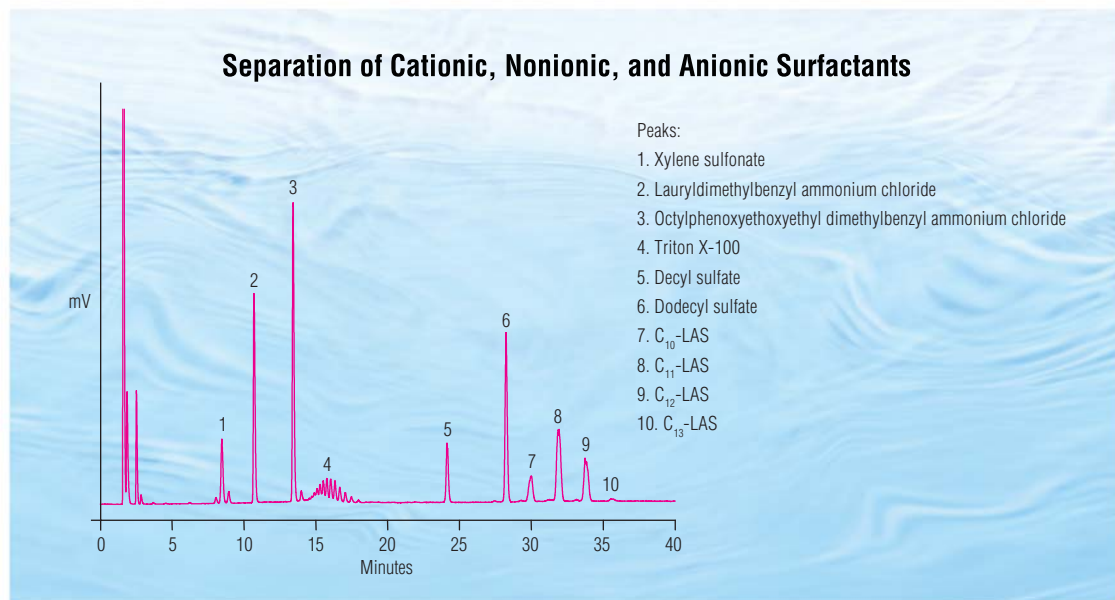


## Acclaim® Surfactant Column A Simple Solution to Difficult Challenges



*Acclaim Surfactant columns are high-efficiency specialty silica columns for separating anionic, nonionic, and cationic surfactants:*

- *Ideal selectivity for separation of anionic, nonionic, and cationic surfactants*
- *Excellent peak shapes for cationic surfactants*
- *Improved resolution for ethoxylated surfactants*
- *Compatible with highly aqueous mobile phases*
- *Methods compatible with various detectors*
- *Broad range of applications*

### **Ideal Selectivity for the Separation of Anionic, Nonionic, and Cationic Surfactants**

The Acclaim Surfactant column is a new column designed for, and ideally suited to, the separation of a variety of different surfactants. This column incorporates a proprietary silica-based bonded phase that offers ideal selectivity and unprecedented capacity for separating cationic, nonionic and anionic surfactants in a single run. The simple, volatile mobile phases are compatible, with mass spectrometry detection which

facilitates the application of this column to trace-level analyses of surfactants in various matrices, including pharmaceutical formulations and environmental samples.

Surfactants are widely used in industrial, agricultural, and pharmaceutical markets, in products as diverse as pesticides, detergent powders, petroleum products, cosmetics, and pharmaceuticals. Their separation and identification can be a challenge due both to the diversity of surfactants and complexity of the sample matrix.

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The separation of surfactants is typically accomplished using HPLC. Reversed-phase and ion-exchange chromatography are the most popular approaches, but normal-phase and size-exclusion chromatography are also used, depending on the application. Although many HPLC stationary phases are available and have been used for the analysis of surfactant formulations, none of these columns have been designed specifically for this application, nor are they capable of separating anionic, nonionic, and cationic surfactants in a single chromatographic run. Figure 1 shows the difference between a conventional C18 column and the Acclaim Surfactant column for the separation of a mixture of anionic and nonionic surfactants. The Acclaim Surfactant column provides excellent separation, whereas the C18 column fails to resolve all the surfactants under the same conditions.

### Excellent Peak Shapes for Cationic Surfactants

Reversed-phase chromatography, using a C18 column, is often used for the separation of anionic surfactants. When analyzing cationic surfactants, however, it is often difficult to obtain sharp, symmetrical peaks due primarily to the presence of free silanols. The novel bonding chemistry of the Acclaim Surfactant phase allows for effective deactivation of free silanols toward positively charged cationic surfactants, resulting in excellent peak shapes, as shown in Figure 2. By comparison, a C18 column tested under similar conditions demonstrates an extended retention time and peak tailing.

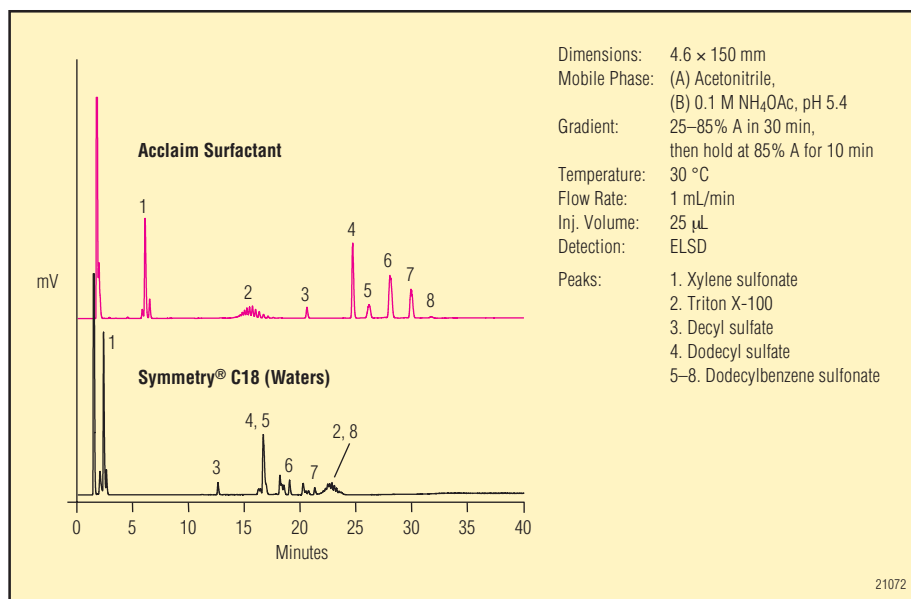


Figure 1. Separation of a mixture of surfactants showing remarkable selectivity.

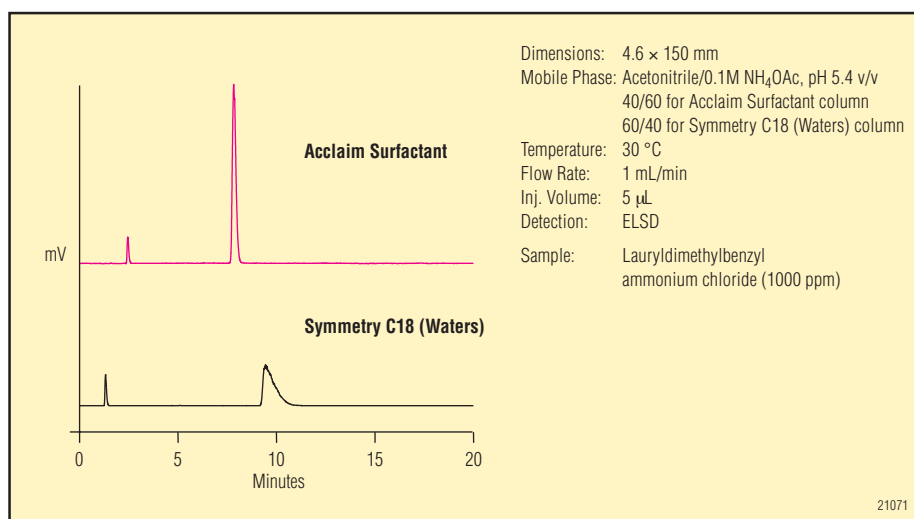


Figure 2. Analysis of a cationic surfactant showing excellent peak shape.

## Improved Resolution for Ethoxylated Surfactants

As a consequence of its novel column chemistry, the Acclaim Surfactant column exhibits a unique polarity that provides significantly improved resolution for individual oligomers of ethoxylated surfactants compared with conventional C18. Figure 3 provides a comparison between the Acclaim Surfactant column and a conventional C18 for the characterization of Triton X-100. The Acclaim surfactant column exhibits significantly improved resolution between the oligomers.

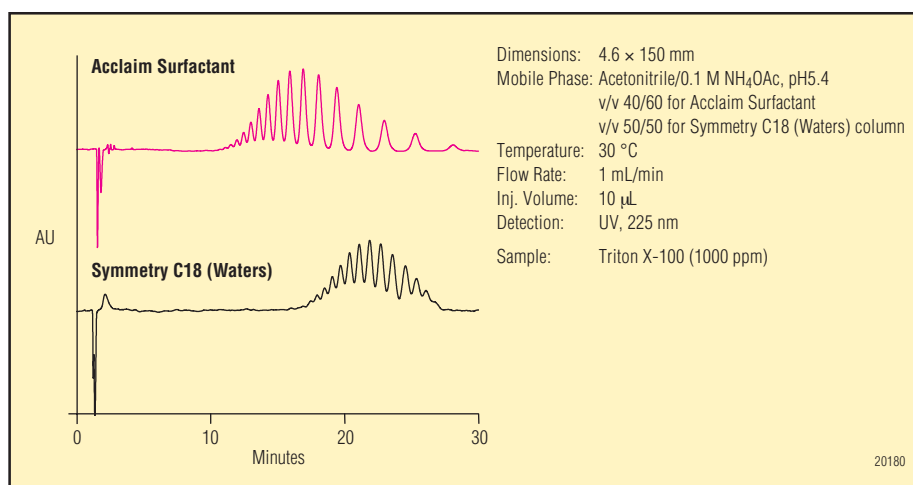


Figure 3. Improved resolution between oligomers in ethoxylated surfactants.

## Compatible with Highly Aqueous Mobile Phase Conditions

High-density C18 columns are often unsuitable for analyzing strongly hydrophilic hydrotropes, such as sodium naphthalene sulfonate and xylene sulfonate. The problem arises because these analyses require a highly aqueous mobile phase that often leads to undesirable “dewetting”. As illustrated in Figure 4, the novel chemistry of Acclaim Surfactant column provides excellent resolution between isomers of xylene sulfonate, while under the same condition little or no retention is observed on the conventional C18 column.

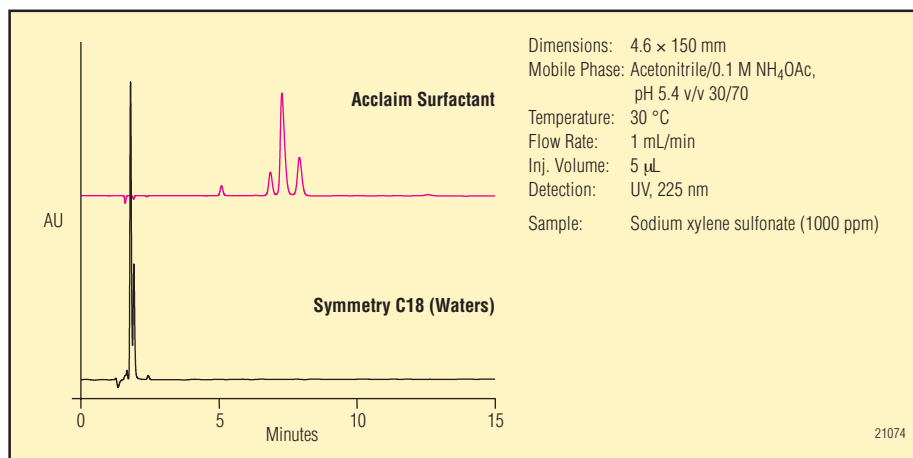


Figure 4. Analysis of a strongly hydrophilic hydrotrope.

## Methods Compatible with Various Detectors (ELSD, UV, MS Conductivity Detection)

UV absorbance is the most popular detection method in HPLC, due to its ease of use and sensitivity (Figures 3–5 and 18–20). The drawback with this approach is that the analyte must have a chromophore to be detected and many surfactants do not.

Although refractive index (RI) detection is a universal detection method, capable of detecting all analytes, it is incompatible with gradient methods, exhibits low sensitivity, and thus is only used when other detection methods are not applicable.

Evaporative light-scattering detection (ELSD) is not only a universal detection method, but also is compatible with gradient methods and is far more sensitive than RI. In addition, methods developed with ELSD can be easily transferred to LC-ESI-MS applications with little or no modifications, because both detectors share the same mobile phase requirements (Figures 1, 2, 8, 11–14 and 16–22).

Mass spectrometry (MS) is an inherently sensitive and universal method and has become the widely accepted tool for characterization of organic compounds. The soft ionization techniques, such as electrospray ionization (ESI), have greatly increased

the applicability of MS detection to surfactant analysis. As shown in Figures 6, 10, and 15, the Acclaim Surfactant column can be used for the analysis of anionic, cationic, and nonionic surfactants using LC-ESI-MS and ammonium acetate eluents.

Suppressed conductivity detection can also be used for surfactant analysis and provides certain advantages for analyzing trace levels ionic surfactants in complex matrices. Figures 7 and 9 show the separation of various anionic and cationic surfactants on the Acclaim Surfactant column, using a borate buffer and acetic acid mobile phases, respectively.

## Broad Range of Applications

### Anionic Surfactants

Anionic surfactants account for 60% of surfactant use in the United States, where they are popular ingredients in detergent powders. This popularity arises because of their effectiveness compared with other surfactants in particulate soil removal, especially from natural fabrics, and because they are easily spray-dried.

**Linear alkylbenzenesulfonates** (LASs) are the most widely used surfactants, due to their low cost and rapid degradation under aerobic conditions. The synthesis of LAS typically leads to a mixture of positional isomers that results in a very complex sample matrix that can be a challenge to separate effectively by chromatography. To simplify quantitative analysis, isocratic conditions are often used to produce only single peaks for the same size homologous species. As shown in Figure 5, LAS can be separated on the Acclaim Surfactant column into simple, single peaks corresponding to a homologous series, whereas the Acclaim PA column gives more complex chromatograms.

**Alkyl sulfates** are the sulfuric acid esters of linear alcohols. They are frequently employed as additives in cosmetics and detergents. Figure 6 shows the analysis of lauryl sulfate, a major ingredient in shampoo, on an Acclaim Surfactant column using LC-ESI-MS and an ammonium acetate eluent.

**Alkylether sulfates** are prepared by adding oxyethylene groups to an alcohol that is then sulfated. Oxyethylation enhances water solubility and foaming, making these surfactants ideal components in shampoos and detergents. Figure 7 shows the analysis of laureth sulfate using suppressed conductivity detection.

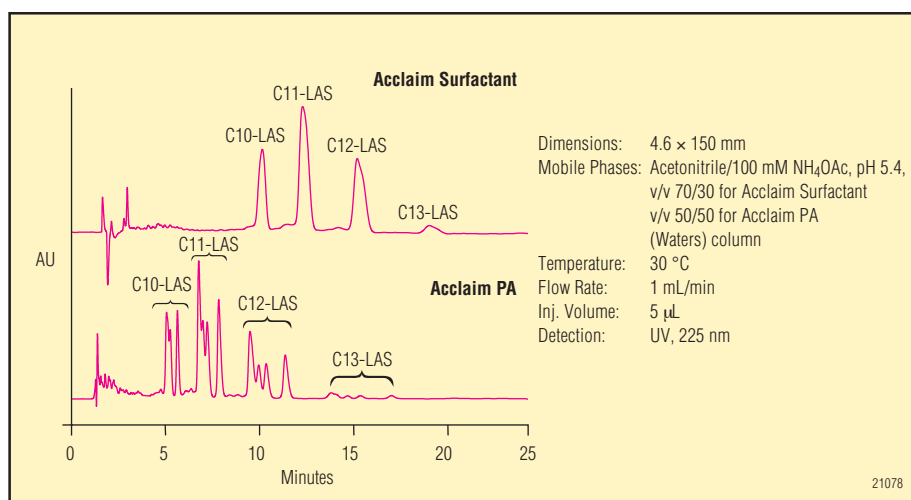


Figure 5. Analysis of sodium dodecylbenzene sulfonate (LAS).

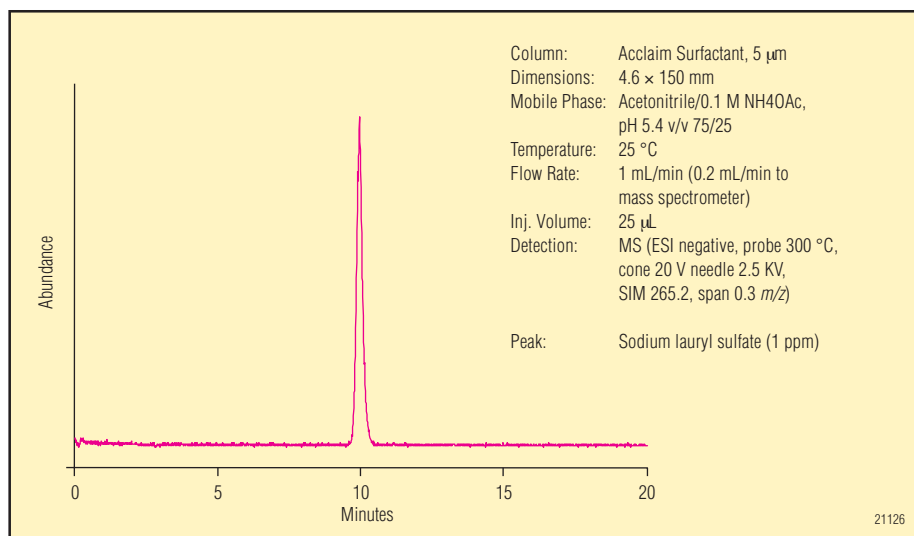


Figure 6. Analysis of sodium lauryl sulfate using LC-ESI-MS.

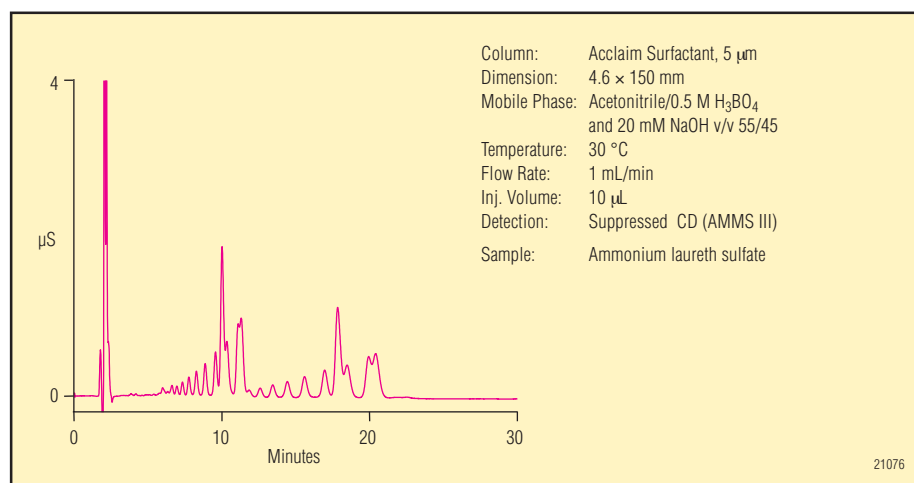
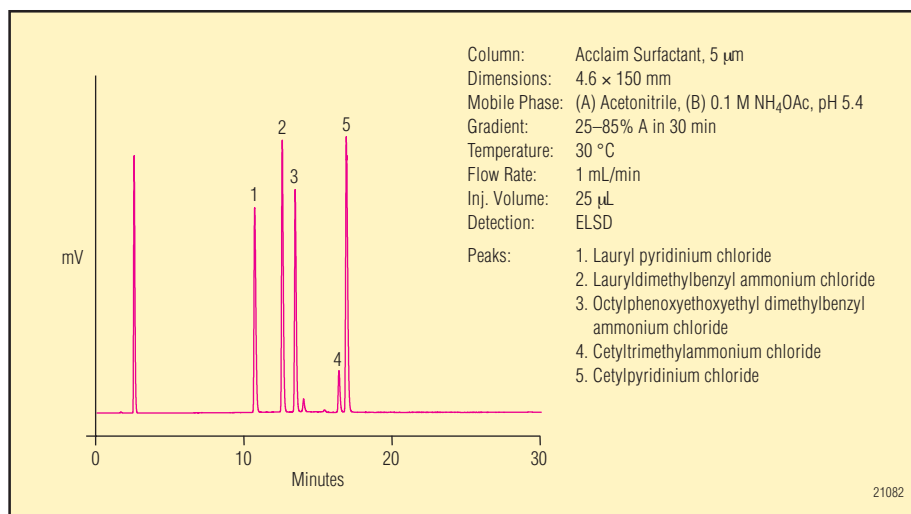


Figure 7. Analysis of ammonium laureth sulfate using conductivity detection.

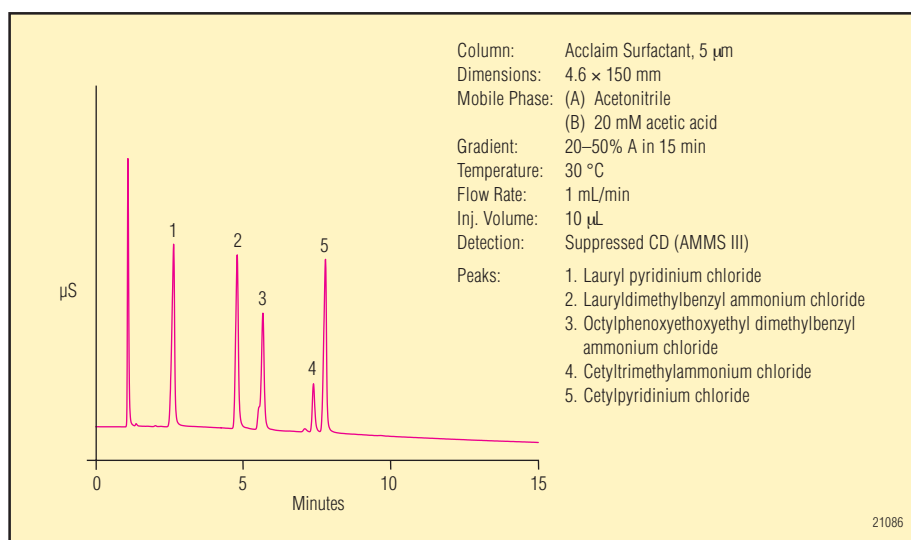
## Cationic Surfactants

Cationic surfactants are used as fabric softeners, corrosion inhibitors, and antimicrobial agents. The most popular cationic surfactants include alkyl quaternary ammonium salts, benzylalkylammonium salts, pyridinium salts, ester quats, ethoxylated quats, and quaternary imidazolium compounds.

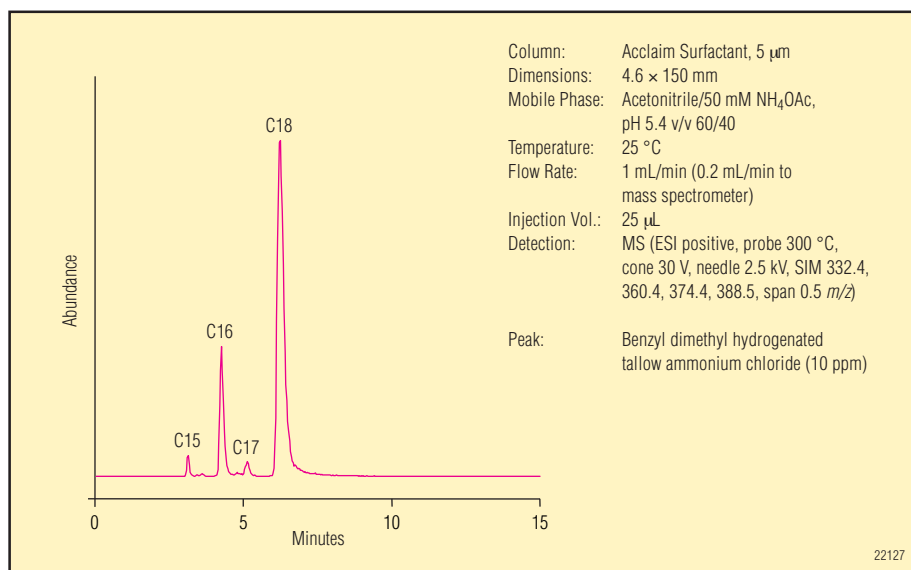
Figures 8–12 present examples of chromatographic analysis using the Acclaim Surfactant column.



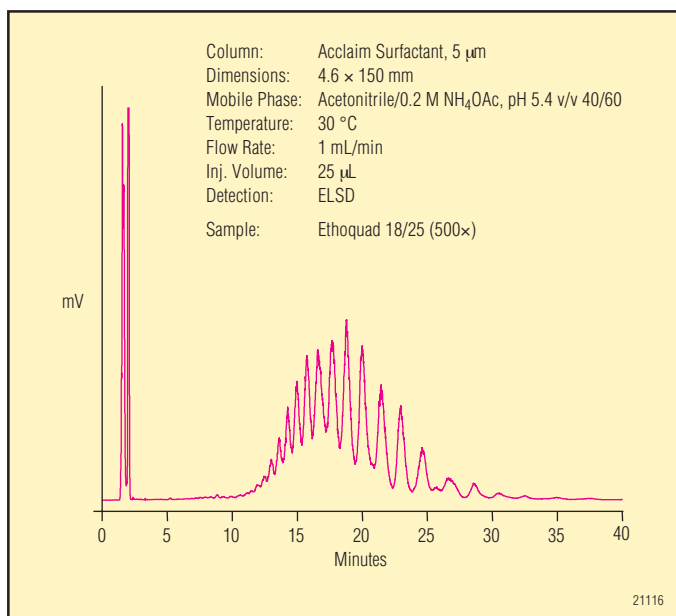
**Figure 8.** Separation of cationic surfactants using ELSD.



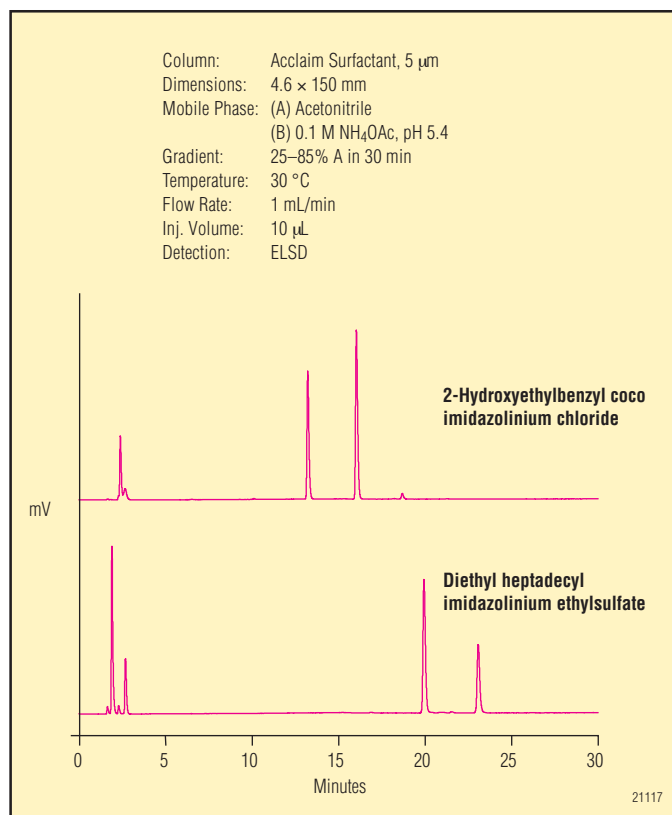
**Figure 9.** Separation of cationic surfactants with suppressed conductivity detection.



**Figure 10.** Analysis of quats using LC-ESI-MS.



**Figure 11.** Separation of ethoxylated quats.



**Figure 12.** Separation of quaternary imidazolinium compounds.

### Nonionic Surfactants

Nonionic surfactants account for about 40% of the worldwide consumption of surfactants. Most nonionic surfactants are considered low-foaming products, have good cold water solubility, and low critical micelle concentration. Their compatibility with cationic fabric softeners makes them preferable in certain formulations. Figures 13–16 show chromatographic analyses of three individual nonionic surfactants using the Acclaim Surfactant column.

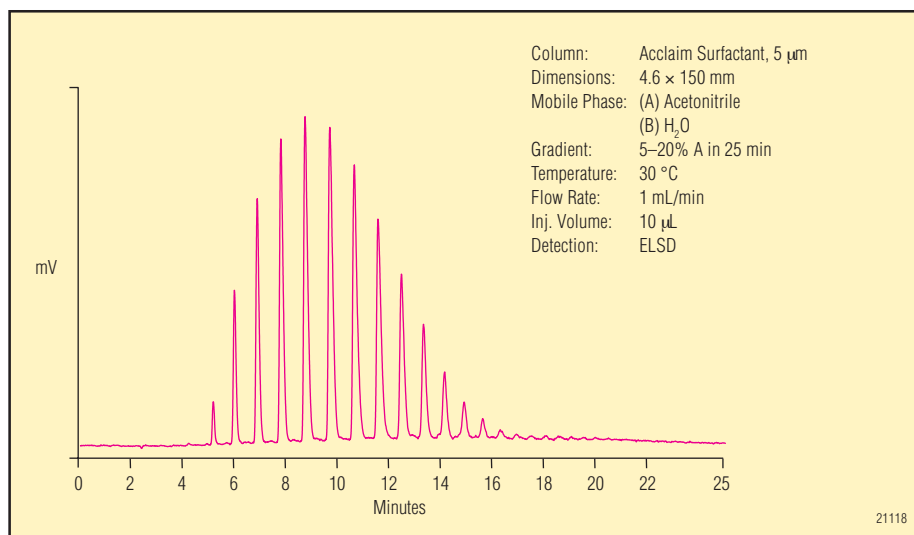


Figure 13. Analysis of PEG monoethyl ether (MW-550).

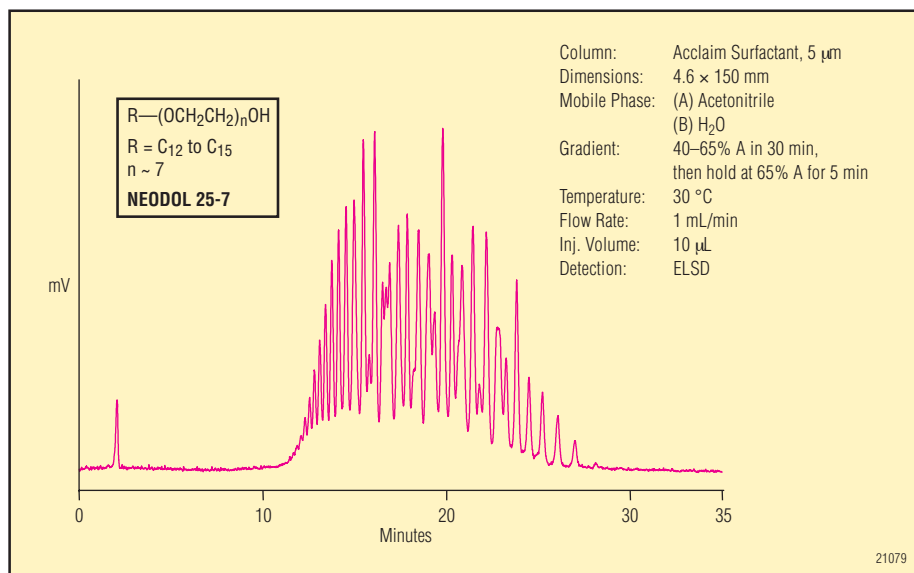


Figure 14. Analysis of NEODOL 25-7.

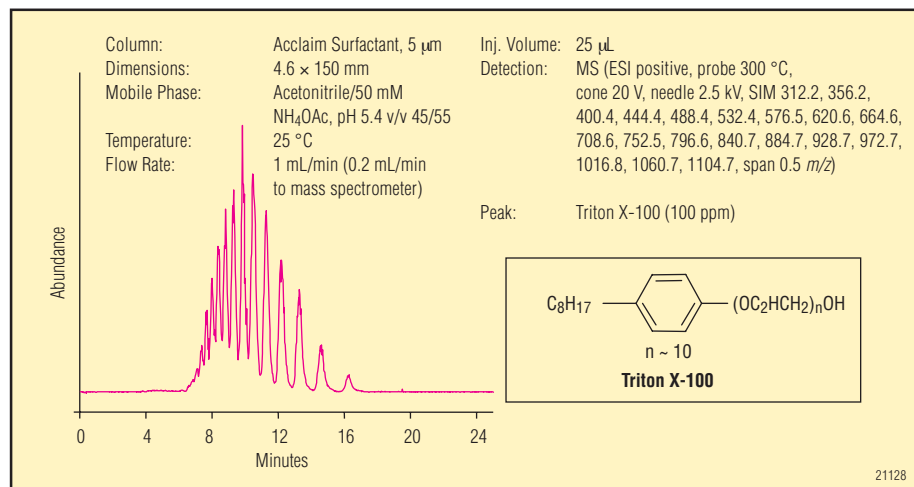


Figure 15. Analysis of Triton X-100 using LC-ESI-MS.

### Polyethylene Glycols (PEGs)

Polyethylene glycols (PEGs) are often nonsurfactant impurities found in ethoxylated surfactants, typically in the range of 1–10%. The oligomer distribution is similar to, but broader than that of the surfactant. Figure 17 illustrates the exceptional resolution of the Acclaim Surfactant column for individual oligomers in various PEGs.

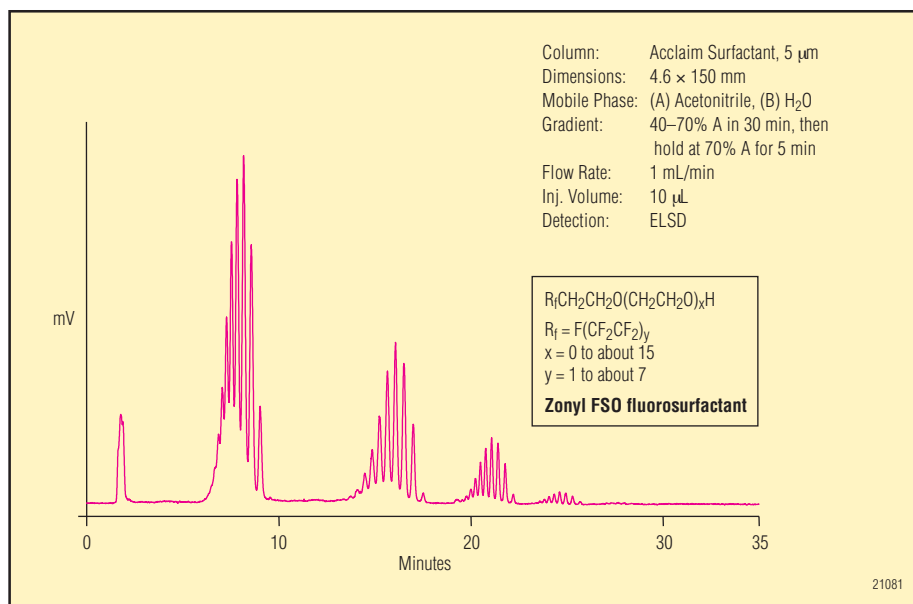


Figure 16. Analysis of ZONYL FSO fluorosurfactant.

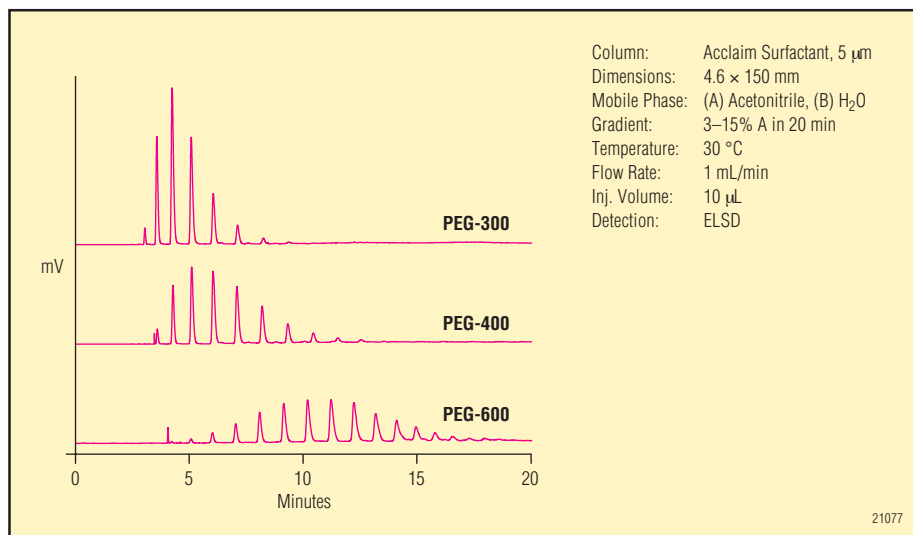


Figure 17. Separation of different polyethylene glycols.



## Analysis of Surfactants in Consumer Products

Figures 18–22 demonstrate the applicability of the Acclaim Surfactant column for analyzing a variety of consumer products, such as shampoo, laundry detergent, dish washing liquid, mouthwash, and fabric softener.

### Reproducible Manufacturing

To meet the exacting needs of our customers, each Acclaim Surfactant column is manufactured to stringent specifications to ensure column-to-column reproducibility. Each column is shipped with a lot validation sheet showing the test results and specifications for the lot of bonded silica packed into the column. In addition, each column is individually tested and shipped with an individual test chromatogram validating the column performance, with respect to selectivity, capacity, and efficiency.

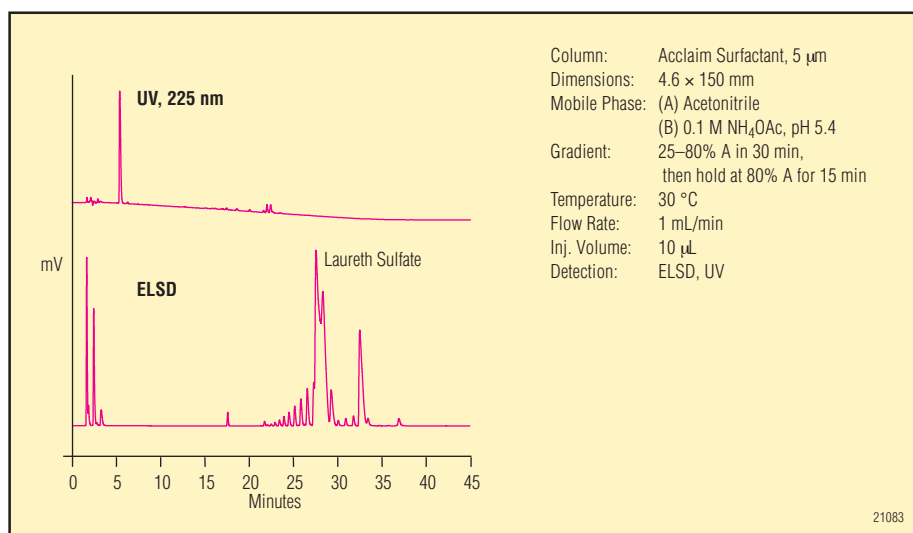


Figure 18. Analysis of a shampoo.

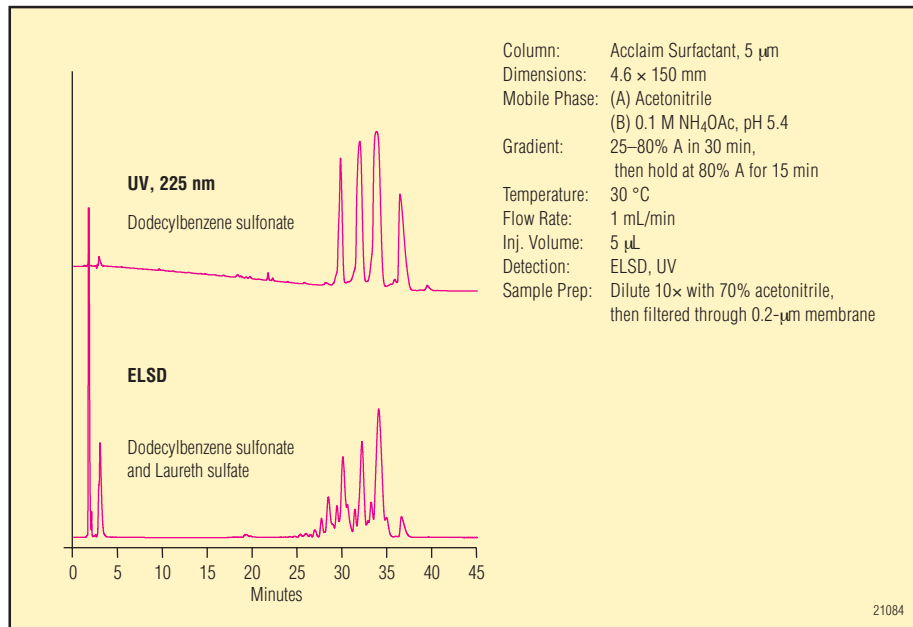


Figure 19. Analysis of a laundry washing detergent.

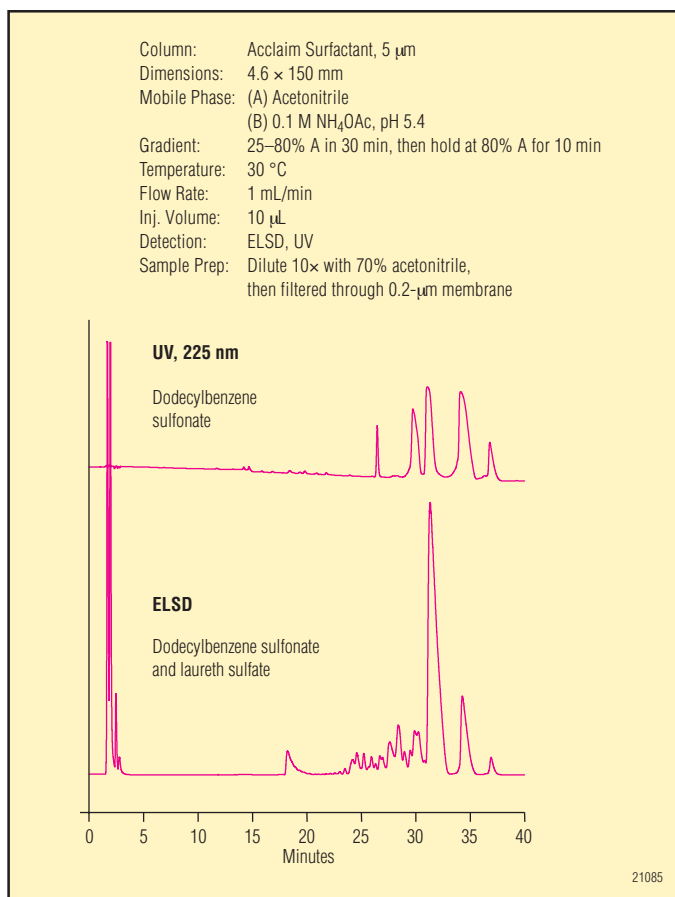


Figure 20. Analysis of a dishwashing liquid.

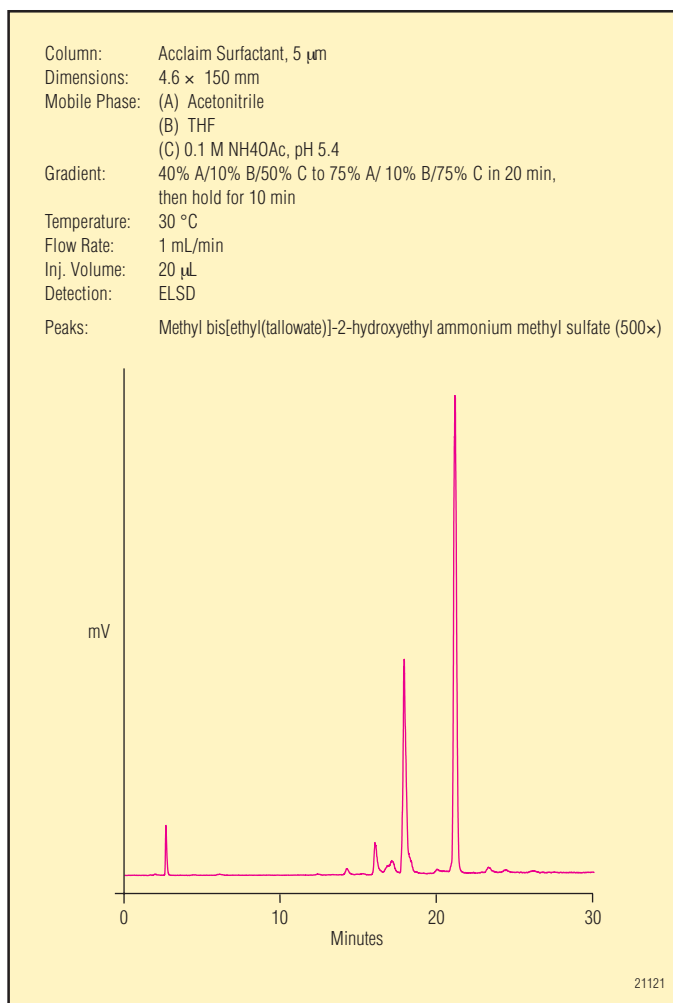


Figure 22. Analysis of a fabric softener.

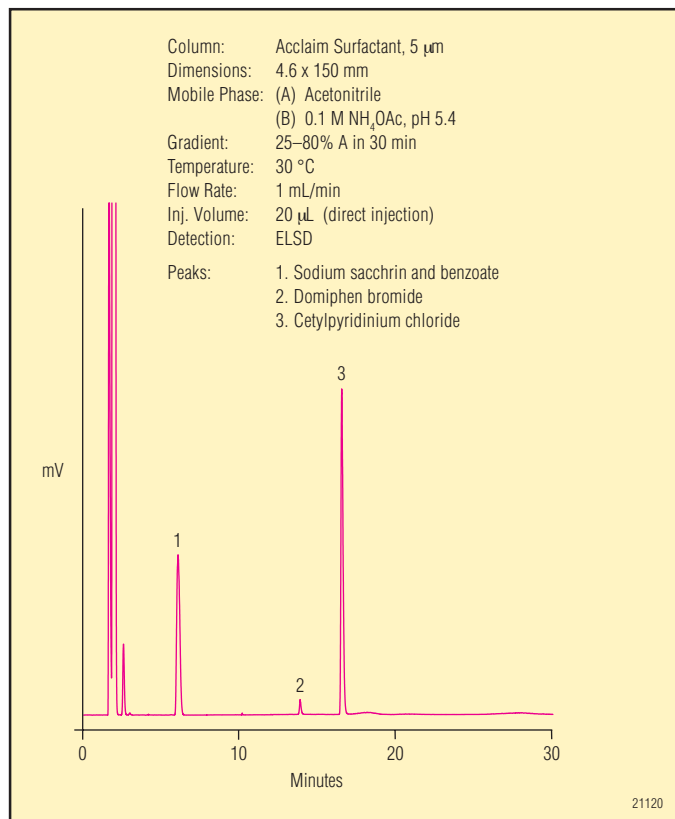


Figure 21. Analysis of a mouthwash.

## SPECIFICATIONS

*Starting Material:*  
Ultrapure silica

*Particle Size:*  
5  $\mu\text{m}$

*Particle Shape:*  
Spherical

*Particle Size Distribution (40/90):*  
1.2

*Total Carbon Content (%):*  
12%

*Endcapped:*  
Yes

*Metal Impurity (ppm) Na, Fe, Al:*  
<10.0

*Pore Volume (mL/g):*  
0.9

*Average Pore Diameter ( $\text{\AA}$ ):*  
120

*Surface Area ( $\text{m}^2/\text{g}$ ):*  
300

*pH range:*  
2.5–7.5

*Temperature:*  
<60  $^{\circ}\text{C}$

## ORDERING INFORMATION

To order in the U.S., call (800) 346-6390 or contact the Dionex Regional Office nearest you. Outside the U.S., order through your local Dionex office or distributor. Refer to the following part numbers.

<b>Product Description</b>	<b>Part Number</b>
Acclaim Surfactant Analytical Column (4.6 $\times$ 150 mm) .....	063201
Acclaim Surfactant Analytical Column (4.6 $\times$ 250 mm) .....	063203
Acclaim Surfactant Guard Cartridges (4.3 $\times$ 10 mm), 2 ea .....	063215
Acclaim Guard Kit (holder and coupler) .....	059526
Acclaim SST Guard Cartridge Holder .....	059456
Guard to Analytical Column Coupler .....	059457



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\* Designed, developed, and manufactured under an NSAI registered ISO 9001 Quality System.



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