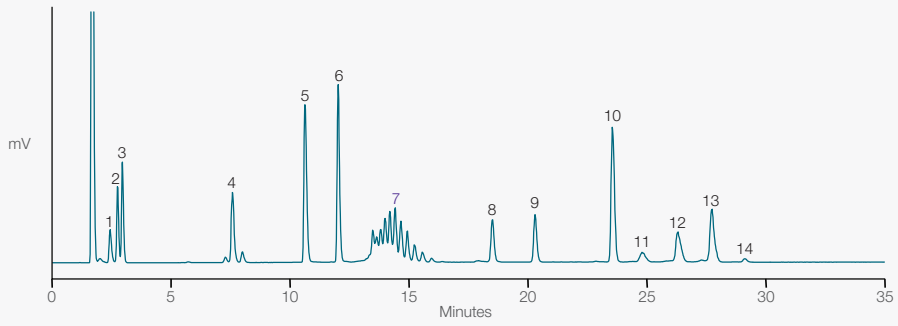
Acclaim Surfactant columns

A simple solution to difficult challenges

Thermo Scientific™ Acclaim™ Surfactant columns are high-efficiency specialty silica columns for separating anionic, nonionic, and cationic surfactants.

Separation of hydrotopic, cationic, nonionic, amphoteric and anion surfactants



Column	Acclaim Surfactant, 5 µm
Dimensions	4.6 × 150 mm
Mobile phase	A: CH_3CN, B: 0.1 M NH_4OAc, pH 5.2
Gradient	25% to 85% A in 25 min, then hold 85% A for 10 min
Temperature	30 °C
Flow rate	1 mL/min
Inj. volume	25 µL
Detection	ELSD
Peaks	1. Chloride
	2. Bromide
	3. Nitrate
	4. Xylene sulfonate
	5. Laurylpyridinium chloride
	6. Lauryldimethylbenzyl-ammonium chloride
	7. Triton X-100
	8. Cetyl betaine
	9. Decyl sulfate
	10. Dodecyl sulfate
	11. C_{10}-LAS
	12. C_{11}-LAS
	13. C_{12}-LAS
	14. C_{13}-LAS

Features

- 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 are designed for, and ideally suited to, the separation of a variety of different surfactants. The Acclaim Surfactant columns, both the Acclaim Surfactant and the Acclaim Surfactant Plus, incorporate 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 Acclaim Surfactant column is best suited for use with Ultraviolet (UV) and Evaporative Light Scattering (ELSD) detection and the Acclaim Surfactant Plus column for LC-MS detection (please refer to the Acclaim Surfactant Plus brochure for more information).

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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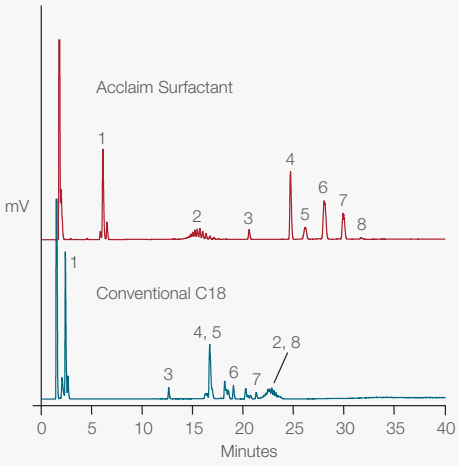
The separation of surfactants is typically accomplished using high performance liquid chromatography (HPLC). Reversed-phase and ion-exchange chromatography are the most popular approaches, but normal-phase and size-exclusion chromatography are also used, depending on the application. Although many HPLC 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.

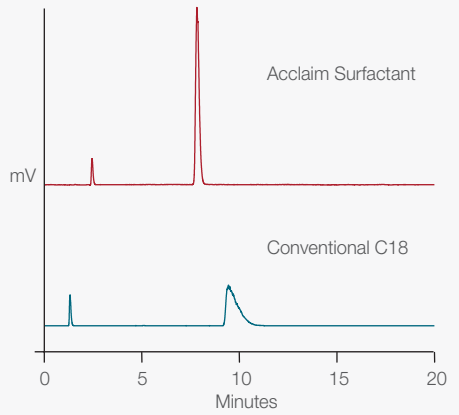
Excellent peak shapes for cationic surfactants

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.



Dimensions	4.6 × 150 mm
Mobile phase	A: Acetonitrile; B: 0.1 M NH_4OAc, pH 5.4
Gradient	25–85% A in 30 min, then hold at 85% A for 10 min
Temperature	30 °C
Flow rate	1 mL/min
Inj. volume	25 µL
Detection	ELSD
Peaks	1. Xylene sulfonate 2. Triton X-100 3. Decyl sulfate 4. Dodecyl sulfate 5–8. Dodecylbenzene sulfonate

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

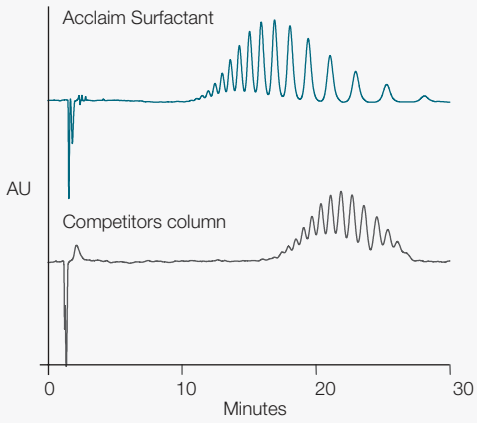


Dimensions	4.6 × 150 mm
Mobile phase	Acetonitrile/0.1M NH_4OAc, pH 5.4 v/v 40/60 for Acclaim Surfactant column 60/40 for Conventional C18 (Waters) column
Temperature	30 °C
Flow rate	1 mL/min
Inj. volume	5 µL
Detection	ELSD
Sample	Lauryldimethylbenzyl ammonium chloride (1000 ppm)

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.

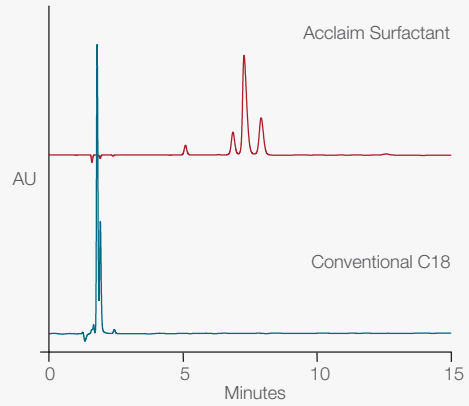


Dimensions	4.6 × 150 mm
Mobile phase	Acetonitrile/0.1 M $NH_4$0Ac, pH 5.4 v/v 40/60 for Acclaim Surfactant v/v50/50 for competitors column
Temperature	30 °C
Flow rate	1 mL/min
Inj. volume	10 µL
Detection	UV, 225 nm
Sample	Triton X-100 (1000 ppm)

Figure 3. Improved resolution between oligomers in ethoxylated surfactants.

Compatible with highly aqueous mobilephase 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.



Dimensions	4.6 × 150 mm
Mobile phase	Acetonitrile/0.1 M NH_4OAc, pH 5.4 v/v 30/70
Temperature	30 °C
Flow rate	1 mL/min
Inj. volume	5 µL
Detection	UV, 225 nm
Sample	Sodium xylene sulfonate (1000 ppm)

Figure 4. Analysis of a strongly hydrophilic hydrotrope.

Methods compatible with various detectors (ELSD, UV, MS conductivity detection)

Ultra-violet (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 Liquid Chromatography-Electrospray Ionization-MassSpectrometry (LC-ESI-MS) applications with little or no modifications, because both detectors share the same mobile phase requirements (Figures 1, 2, 8).

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.

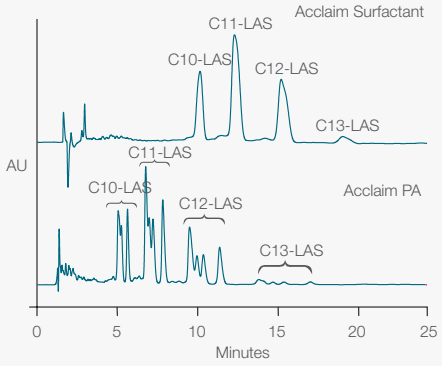
Broad range of applications

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.

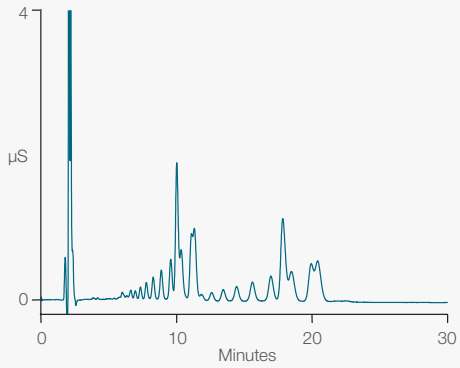
Linear alkylbenzenesulfonates (LAS) are the most widely used surfactants, due to their low cost and rapid degradation under aerobic conditions. The synthesis of LAS typically leads to a mixture of positional isomers that results in a very complex sample matrix that can be a challenge to separate effectively by chromatography. To simplify quantitative analysis, isocratic conditions are often used to produce only single peaks for the same size homologues 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 Thermo Scientific™ Acclaim™ Polar Advantage (PA) column gives more complex chromatograms.

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 6 shows the analysis of laureth sulfate using suppressed conductivity detection.



Dimensions	4.6 × 150 mm
Mobile phase	Acetonitrile/100 mM NH_4OAc, pH 5.4, v/v 70/30 for Acclaim Surfactant v/v 50/50 for Acclaim PA
Temperature	30 °C
Flow rate	1 mL/min
Inj. volume	5 µL
Detection	UV, 225 nm

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

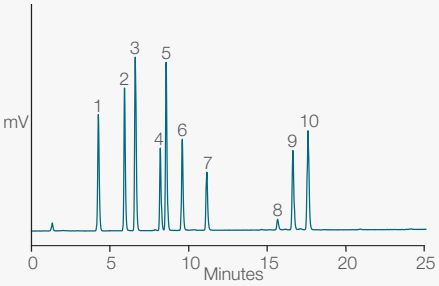


Column	Acclaim Surfactant, 5 µm
Dimensions	4.6 × 150 mm
Mobile phase	Acetonitrile/0.5 M H_3BO_4 and 20 mM NaOH v/v 55/45
Temperature	30 °C
Flow rate	1 mL/min
Inj. volume	10 µL
Detection	Suppressed CD (AMMS III)
Sample	Ammonium laureth sulfate

Figure 6. 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. As shown in Figure 7 and the linear response in Figure 8.



Time (min)	%A	%B
-12	20	80
0	20	80
25	80	20

Column	Acclaim Surfactant, 5 µm
Dimensions	4.6 × 150 mm
Mobile phase	A: Acetonitrile, B: 1% acetic acid
Temperature	30 °C
Flow rate	1 mL/min
Inj. volume	10 µL
Detection	ELS detector (gain 8, evaporating temperature 50 °C)
Peaks	(100–400 µg/mL each) 1. Lauryl pyridinium 2. Lauryldimethylbenzyl ammonium 3. Octylphenoxyethoxyethyl-dimethyl-benzyl ammonium 4. Cetyltrimethylammonium 5. Cetylpyridinium 6. Diethyl heptadecyl imizolinium – 1 7. Diethyl heptadecyl imizolinium – 2 8. 2M2HT quart – 1 9. 2M2HT quart – 2 10. 2M2HT quart – 3

Figure 7. Separation of cationic surfactants using ELSD.

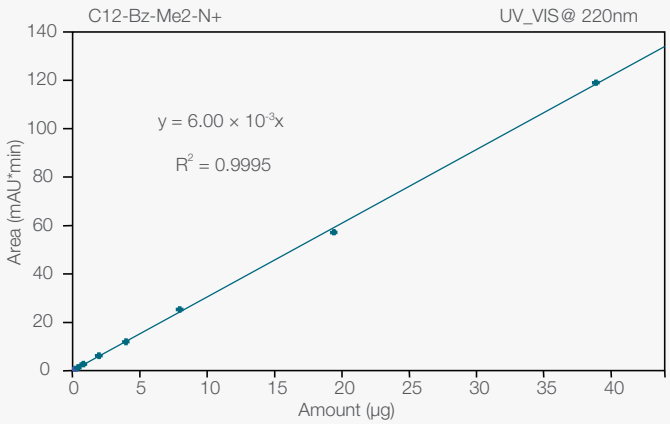
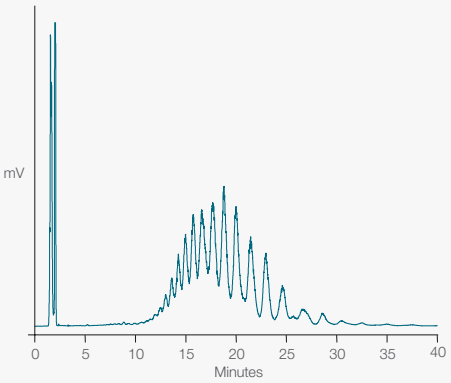


Figure 8. Calibration curve by UV detection.

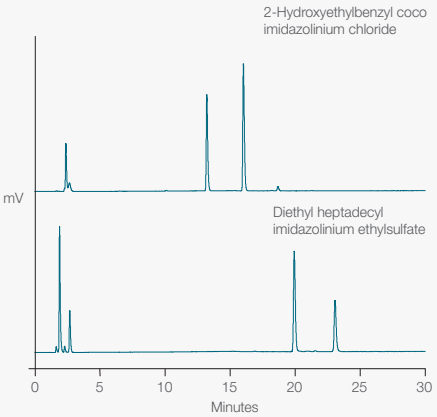
Nonionic surfactants

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 9–12 show chromatographic analyses of three individual nonionic surfactants using the Acclaim Surfactant column.



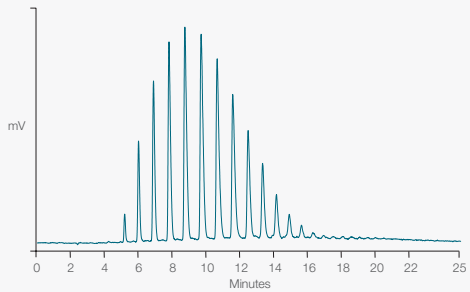
Column	Acclaim Surfactant, 5 µm
Dimensions	4.6 × 150 mm
Mobile phase	Acetonitrile/0.2 M NH_4OAc, pH 5.4 v/v 40/60
Temperature	30 °C
Flow rate	1 mL/min
Inj. volume	25 µL
Detection	ELSD

Figure 9. Separation of ethoxylated quats.



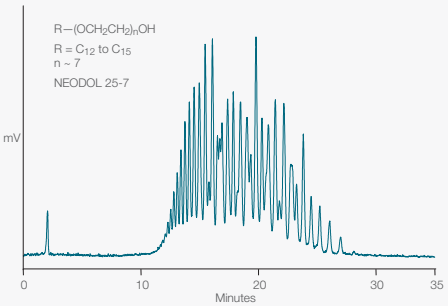
Column	Acclaim Surfactant, 5 µm
Dimensions	4.6 × 150 mm
Mobile phase	A: Acetonitrile; B: 0.1 M NH_4OAc, pH 5.4
Gradient	25–85% A in 30 min
Temperature	30 °C
Flow rate	1 mL/min
Inj. volume	10 µL
Detection	ELSD

Figure 10. Separation of quaternary imidazolinium compounds.



Column	Acclaim Surfactant, 5 µm
Dimensions	4.6 × 150 mm
Mobile phase	A: Acetonitrile; B: H_2O
Gradient	5–20% A in 25 min
Temperature	30 °C
Flow rate	1 mL/min
Inj. volume	10 µL
Detection	ELSD

Figure 11. Analysis of PEG monoethyl ether (MW-550).

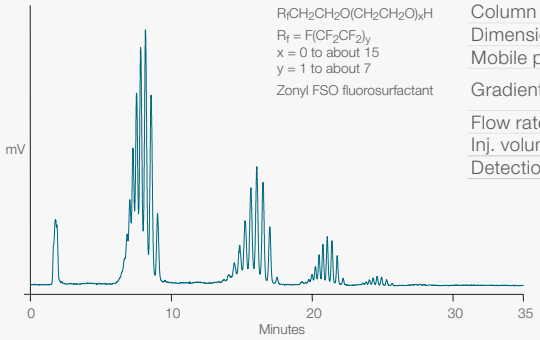


Column	Acclaim Surfactant, 5 µm
Dimensions	4.6 × 150 mm
Mobile phase	A: Acetonitrile; B: H_2O
Gradient	40–65% A in 30 min, then hold at 65% A for 5 min
Temperature	30 °C
Flow rate	1 mL/min
Inj. volume	10 µL
Detection	ELSD

Figure 12. Analysis of NEODOL 25-7.

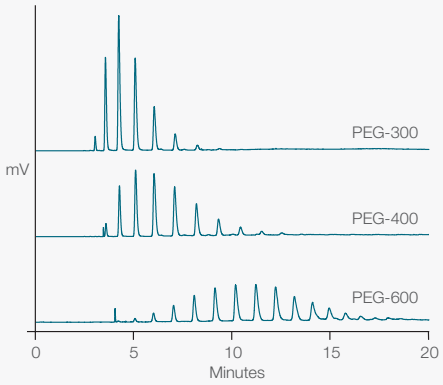
Polyethylene Glycols

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 14 illustrates the exceptional resolution of the Acclaim Surfactant column for individual oligomers in various PEGs.



Column	Acclaim Surfactant, 5 µm
Dimensions	4.6 × 150 mm
Mobile phase	A: Acetonitrile, B: H_2O
Gradient	40–70% A in 30 min, then hold at 70% A for 5 min
Flow rate	1 mL/min
Inj. volume	10 µL
Detection	ELSD

Figure 13. Analysis of ZONYL FSO fluorosurfactant.



Column	Acclaim Surfactant, 5 µm
Dimensions	4.6 × 150 mm
Mobile phase	A: Acetonitrile, B: H_2O
Gradient	3–15% A in 20 min
Flow rate	1 mL/min
Inj. volume	10 µL
Detection	ELSD

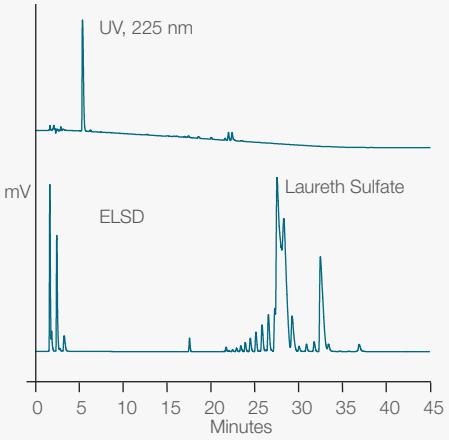
Figure 14. Separation of different polyethylene glycols.

Analysis of surfactants in consumer products

Figures 15–21 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.

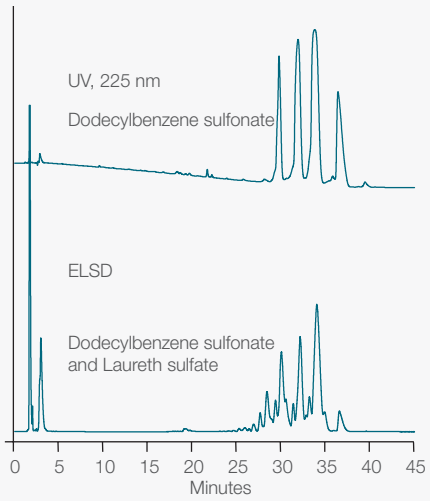
Reproducible manufacturing

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.



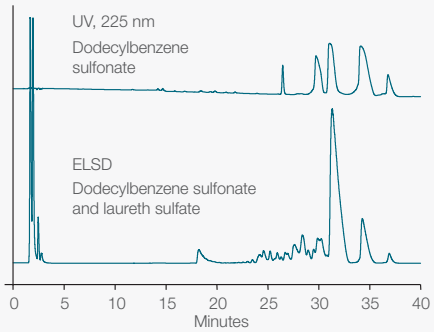
Column	Acclaim Surfactant, 5 µm
Dimensions	4.6 × 150 mm
Mobile phase	A: Acetonitrile, B: 0.1 M NH_4OAc, pH 5.4
Gradient	25–80% A in 30 min, then hold at 80% A for 15 min
Temperature	30 °C
Flow rate	1 mL/min
Inj. volume	10 µL
Detection	ELSD, UV

Figure 15. Analysis of a shampoo.



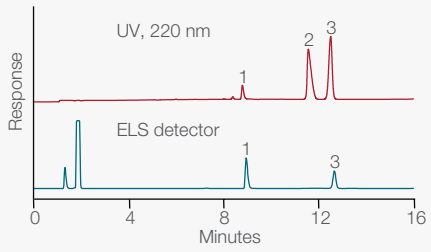
Column	Acclaim Surfactant, 5 µm
Dimensions	4.6 × 150 mm
Mobile phase	A: Acetonitrile, B: 0.1 M NH_4OAc, pH 5.4
Gradient	25–80% A in 30 min, then hold at 80% A for 15 min
Temperature	30 °C
Flow rate	1 mL/min
Inj. volume	5 µL
Detection	ELSD, UV
Sample prep	Dilute 10× with 70% acetonitrile, then filtered through 0.2 µm membrane

Figure 16. Analysis of a laundry washing detergent.



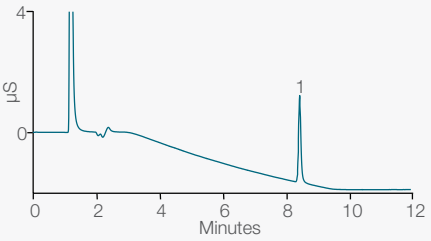
Column	Acclaim Surfactant, 5 µm
Dimensions	4.6 × 150 mm
Mobile phase	A: Acetonitrile, B: 0.1 M NH_4OAc, pH 5.4
Gradient	25–80% A in 30 min, then hold at 80% A for 10 min
Temperature	30 °C
Flow rate	1 mL/min
Inj. volume	10 µL
Detection	ELSD, UV
Sample prep	Dilute 10× with 70% acetonitrile, then filtered through 0.2 µm membrane

Figure 17. Analysis of a dishwashing liquid.



Column	Acclaim Surfactant, 5 µm
Dimensions	4.6 × 150 mm
Mobile phase	A: Acetonitrile, B: 100 mM ammonium acetate, pH 5.2, C: H_2O
Temperature	30 °C
Flow rate	1 mL/min
Inj. volume	10 µL
Detection	UV at 220 nm and ELS detector
Sample	Scope (direct injection)
Peaks	1. C16 – Pyridinium 2. Benzoate 3. Saccharinate

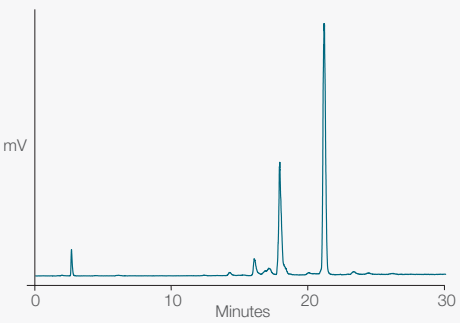
Time (min)	%A	%B	%C
-10	25	5	70
0	25	5	70
14	85	5	10
16	85	5	10



Column	Acclaim Surfactant, 5 µm
Dimensions	4.6 × 150 mm
Mobile phase	A: 70% Acetonitrile in D.I. water, B: 100 mM formic acid, C: D.I. water
Temperature	30 °C
Flow rate	1 mL/min
Inj. volume	15 µL
Detection	Suppressed conductivity detection (CSRS ULTRA II 4 mm suppressor, external water mode at 1.0 mL/min, current 44 mA)
Sample	Scope (fivefold dilution)
Peaks	1. Cetylpyridinium

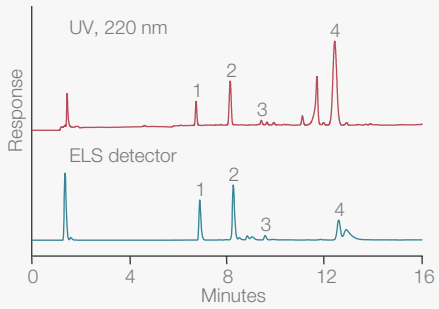
Time (min)	%A	%B	%C
-7	20	15	65
0	20	15	65
10	50	15	35
20	50	15	35

Figure 18. Analysis of a mouthwash.



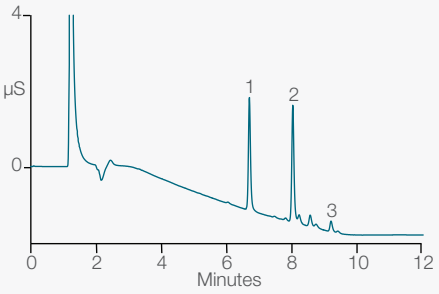
Column	Acclaim Surfactant, 5 µm
Dimensions	4.6 × 150 mm
Mobile phase	A: Acetonitrile, B: THF, C: 0.1 M NH_4OAc, pH 5.4
Gradient	40% A/10% B/50% C to 75% A/10% B/75% C in 20 min, then hold for 10 min
Temperature	30 °C
Flow rate	1 mL/min
Inj. volume	20 µL
Detection	ELSD
Peaks	Methyl bis[ethyl(tallowate)]-2-hydroxyethyl ammonium methyl sulfate (500×)

Figure 19. Analysis of a fabric softener.



Time (min)	%A	%B	%C
-10	25	5	70
0	25	5	70
14	85	5	10
16	85	5	10

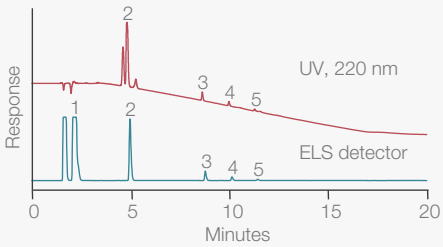
Column	Acclaim Surfactant, 5 µm
Dimensions	4.6 × 150 mm
Mobile phase	A: Acetonitrile; B: 100 mM ammonium acetate, pH 5.2, C: H_2O
Temperature	30 °C
Flow rate	1 mL/min
Inj. volume	10 µL
Detection	UV at 220 nm and ELS detector
Sample	Lysol disinfectant spray (direct injection)
Peaks	1. C_{12} – Dimethyl benzyl ammonium
	2. C_{14} – Dimethyl benzyl ammonium
	3. C_{16} – Dimethyl benzyl ammonium
	4. Saccharinate



Time (min)	%A	%B	%C
-7	20	15	65
0	20	15	65
10	50	15	35
20	50	15	35

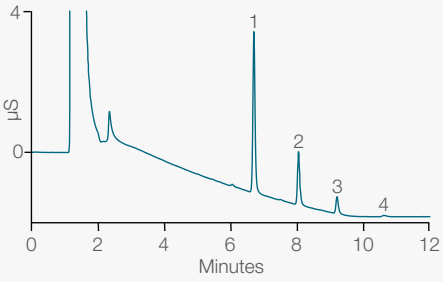
Column	Acclaim Surfactant, 5 µm
Dimensions	4.6 × 150 mm
Mobile phase	A: 70% Acetonitrile in D.I. water, B: 100 mM formic acid, C: D.I. water
Temperature	30 °C
Flow rate	1 mL/min
Inj. volume	15 µL
Detection	Suppressed conductivity detection (CSRS ULTRA II 4 mm suppressor, external water mode at 1.0 mL/min, current 44 mA)
Sample	Scope (fivefold dilution)
Peaks	1. Cetylpyridinium

Figure 20. Determination of benzalkonium salts in spray disinfectant, comparing UV, ELSD and Suppressed conductivity.



Time (min)	%A	%B	%C
-10	25	5	70
0	25	5	70
14	85	5	10
16	85	5	10

Column	Acclaim Surfactant, 5 µm
Dimensions	4.6 × 150 mm
Mobile phase	A: Acetonitrile, B: 100 mM ammonium acetate, pH 5.2, C: H_20
Temperature	30 °C
Flow rate	1 mL/min
Inj. volume	10 µL
Detection	UV at 220 nm and ELS detector
Sample	Scope (direct injection)
Peaks	1. C_{16} – Pyridinium
	2. Benzoate
	3. Saccharinate



Time (min)	%A	%B	%C
-7	20	15	65
0	20	15	65
10	50	15	35
20	50	15	35

Column	Acclaim Surfactant, 5 µm
Dimensions	4.6 × 150 mm
Mobile phase	A: 70% Acetonitrile in D.I. water, B: 100 mM formic acid, C: D.I. water
Temperature	30 °C
Flow rate	1 mL/min
Inj. volume	15 µL
Detection	Suppressed conductivity detection (CSRS ULTRA II 4 mm suppressor, external water mode at 1.0 mL/min, current 44 mA)
Sample	Scope (fivefold dilution)
Peaks	1. Cetylpyridinium

Figure 21. Comparison of UV, ELS and Suppressed conductivity detection of mouthwash.

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Column specifications

Specifications	
Starting material	Ultrapure silica
Particle size	5 µm
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 (Å)	120

Ordering information

Column	Format	Length (mm)	ID (mm)	Part number
Acclaim Surfactant	Analytical	150	2.1	068123
		150	4.6	063201
		250	4.6	063203
	Guard	10	2.1	069693
		10	4.6	069701

Acclaim Guard Holder ordering information

Guard holder	Part number
Thermo Scientific™ Acclaim™ Guard Cartridge Holder V-2	069580
Thermo Scientific™ Acclaim™ Guard Kit (Holder and coupler) V-2	069707
Guard to Analytical Column Coupler V-2	074188

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