Nonionic ethoxylated surfactants, e.g., alkylphenol ethoxylates and fatty alcohol ethoxylates, are widely used in industrial and consumer products. Very often, HPLC is the method of choice for surfactant analysis (1). Depending on circumstances, determination of both EO number (degree of ethoxylation) and alkyl chain distributions is often needed. While EO distribution can be measured chromatographically with a normal phase (NP) column, the alkyl chain distribution is usually characterized on a reversed-phase (RP) column. Because both EO and alkyl chain separations may occur concurrently on a typical RP column, accurate quantification can be challenging. Acclaim Mixed-Mode HILIC-1 is a newly developed HPLC column that provides both RP and HILIC (hydrophilic interaction) properties (Figure 1).

Separations were performed with an Acclaim Mixed-Mode HILIC-1 column (4.6 x 150 mm, dp = 5 mm) on a modular Summit® HPLC System ( Dionex) equipped with a P680 gradient pump, ASI-100 Autosampler, TCC-100 column oven, and UVD 340 detector. Chromelon® 6.7 Chromatography Management Software (Dionex) was used for system control and data processing. A Sedex 85 evaporative light scattering detector (Sedere, Alfortville, France) was used to detect surfactants with weak or no chromophores.

Results and Discussion
Figure 2 illustrates the analysis of Brij 35 [lauryl alcohol condensed with 23 moles ethylene oxide, molecular formula: C12H25(OCH2CH2)23OH]. In RP conditions, all EO oligomers with the same alkyl chain collapse into a single peak, resulting in four well-separated narrow peaks (upper trace), unreacted PEGs (early eluting peak), and three ethoxylates corresponding to different alkyl chain lengths. In HILIC conditions, on the other hand, all EO oligomers are well separated (lower trace). Thus, the EO distribution can be determined on the same column. On a typical RP column, the determination of alkyl chain distribution is not possible.

Conclusion
Acclaim Mixed-Mode HILIC-1 provides both HILIC and RP properties, higher hydrophobic retention compared to a conventional HILIC column, and superior performance for determination of both EO number and alkyl chain distribution of nonionic ethoxylated surfactants (2,3).

References
(2) X. Liu, C. Pohl, J. Chromatogr. A (in press)

Figure 1: Dual operation modes: RP and HILIC. Mobile Phase: acetonitrile/0.1 M ammonium acetate, pH 5 [52/48 and 92/8(v/v) for RP and HILIC modes, respectively]; flow rate, 1 mL/min; injection volume, 10 mL; temperature, 30 °C; and detection, UV at 254 nm. Peaks: (1) Cytosine (0.1 mg/mL); (2) Naphthalene (0.1 mg/mL).

Figure 2: Analysis of ethoxylated alcohols in both RP and HILIC Modes. Mobile Phase: acetonitrile/0.1 M ammonium acetate, pH 5 [70/30 and 90/10 (v/v) for RP and HILIC separations, respectively]; flow rate, 1 mL/min; injection volume, 5 mL; temperature, 30 °C; and detection, ELS detector. Sample: Brij 35 (1 mg/mL).