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Review

# Chromatographic tools for analyzing and tracking non-ionic surfactants in the aquatic environment

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**Abstract**

This review describes the developments over the last few decades in the routine determination of two major types of non-ionic surfactants in environmental samples, i.e. alkylphenol ethoxylates (APE) and alcohol ethoxylates (AE). Commercial mixtures of these surfactants consist of several tens to hundreds of homologues, oligomers and isomers. Therefore, their identification and quantitation in the environment is complicated and cumbersome. The request for more specific analytical methods has prompted a replacement of all the separate steps in traditional, usually non-chromatographic methods of analysis of AP and APE by chromatographic tools. Thus, a 100-fold gain in detection limits has been achieved. Determination of AE and APE in aqueous samples is possible at the ppb level nowadays, when solid-phase extraction, including chromatographic cleanup, is applied, followed by liquid chromatography (LC). Reversed-phase LC resolves the various alkyl homologues, whereas normal-phase LC provides information on the ethoxylate oligomer distribution. Because of differences in (bio)degradation patterns observed in the aqueous environment between AE and APE, it is recommended to analyse AE by reversed-phase LC and APE by normal-phase LC. The more sophisticated hyphenated LC–MS techniques provide full information on isomer, oligomer and homologue distribution of both AE and APE.

*Keywords:* Water analysis; Reviews; Surfactants; Alkylphenol ethoxylates; Alcohol ethoxylates

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## 1. Introduction

Due to their specific physical chemical properties non-ionic surfactants find their application in the fields of industry, processing technology and science, with a major usage in detergents. These surfactants are used wherever their interfacial effects of detergency, (de)foaming, (de)emulsification, dispersion or solubilisation can enhance product or process performance. The properties of non-ionic surfactants which make them beneficial for detergents include their relative ionic insensitivity and their sorptive behaviour [1]. The production of non-ionic surfactants in Western Europe and the USA is a growing market and amounts to 750 000 t/a [2]. The most part of the group of non-ionic surfactants consist of alcohol ethoxylates (AE) and alkylphenol ethoxylates (APE) (Fig. 1). Because of their poor degradability and the formation of persistent metabolites the Parcom Member States decided to phase out the use of nonylphenol ethoxylates (NPE) by the year of 2000. In western Europe and the USA the APE in household detergents have been completely replaced by the AE. Mainly because of its lower price APE are still being used in substantial amounts in institutional and industrial applications.

Chronic toxicity data of non-ionic surfactants to algae, fish and (in)vertebrates range from 0.1 to 20 mg/l, dependent on substances and experimental conditions [3,4]. Environmental concern arose as a result of these effect levels and the release of large amounts of surfactants into the environment. Therefore surfactants have to pass ready biodegradability tests before they are put on the market. AE easily pass these biodegradability tests [2]. In contrast to the AE, the APE are not as rapidly or completely biodegraded. Biodegradation of APE is initiated by hydrophilic attack at the ethoxylate end in which the ethoxylate chain is sequentially shortened by one ethoxylate group at a time. Nonylphenol (NP) and its mono- and diethoxylated derivatives remain as the most persistent metabolites [5]. Lipophilicity of these compounds has been shown to decrease [6,7], and aqueous solubility to increase with increasing number of ethylene oxide groups per molecule [8]. Toxicity of non-ionic surfactants is probably based on non-specific narcotic interferences with membranes. As a result of the less hydrophobic alkyl moiety APE are generally less toxic than equivalent AE. The shorter the ethoxylate chain, the more toxic the compound [2]. NP and to a lesser extent the nonylphenol ethoxylates (NPE) have been recognized as having reproductive and endocrine disrupting effects [9].

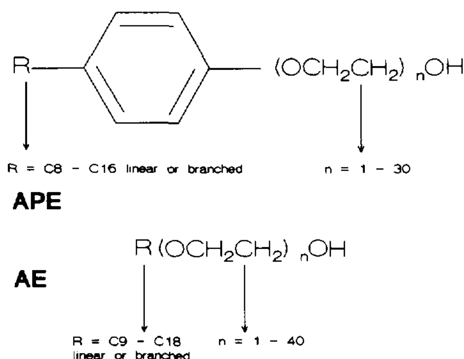


Fig. 1. Structure of alcohol ethoxylates and alkylphenol ethoxylates.

There are several analytical techniques available for studying the fate, effects and behaviour of surfactants in the environment. For the determination of linear alkylbenzenesulphonate and ditallowdimethylammonium chloride, the major anionic and cationic surfactants, in a broad range of environmental samples, specific HPLC methods exist today [10]. The more traditional methods for these compounds, based on colorimetry, lack specificity due to interferences by synthetic and natural substances [10].

An excellent overview of the environmental chemistry of non-ionic surfactants, including their detection, has been made by Holt et al. [2]. During the sixties and seventies analytical methods for the determination of non-ionics were optimised and standardised. Two principal methods emerged in Europe and the USA, respectively. The European method is based on solvent sublation followed by cation-exchange chromatography, precipitation of the non-ionics and potentiometric determination of the bismuth

consumed by the precipitate [11]. This method provides a reasonably good estimate of the total non-ionic surfactants (AE and APE). The somewhat simpler American method uses a similar sequence of sublation and ion-exchange chromatography, whereafter the non-ionics are determined colorimetrically by their cobalt thiocyanate complexes [12]. In both methods the response of the non-ionic surfactant depends on the molecular mass and the polyoxyethylene chain length. No response is observed with non-ionics containing four or less ethoxylate units. The traditional methods to analyse non-ionic surfactants usually overestimate their concentration in effluents of sewage treatment plants and river water [10,13,14].

The non-specificity of the above methods prompted attempts to develop methods with more resolving power. Not surprisingly, solutions were almost exclusively sought in chromatographic methods. Initial attempts applied thin-layer chromatographic methods [15]. The concurrent tremendous development of gas chromatography (GC) and high-performance liquid chromatography (HPLC), however, soon led to promising results in particular in the liquid chromatography area.

HPLC nor GC methods necessitate chemical reactions with metal cations, as in potentiometric or colorimetric detection methods for non-ionics. Apart from their enhanced specificity, the chromatographic techniques offer more sensitivity [10]. They can not only be applied to the final detection of the analytes, but also to sample extraction and preparation. Therefore, the chromatographic methods have almost completely taken over the analytical procedure for the routine determination of non-ionic surfactants from the more traditional methods. The purpose of the present paper is to present a concise overview of the latest achievements of chromatographic methods in the routine analysis of AE and APE in the aquatic environment. The chromatographic techniques are evaluated with regard to practicality, robustness, sensitivity and specificity. It is our aim to confine ourselves principally to those methods that have been shown to be applicable to environmental samples. In addition, some promising future techniques will be discussed.

### *1.1. Chemical structure*

Due to their wide range of masses and physical chemical properties the structure of non-ionic surfactants is of major importance in their analysis. Non-ionic surfactants, like other surfactants, possess a polar (hydrophilic) and a non-polar (hydrophobic) region. The polar region of the non-ionic surfactants considered here is provided by an ethoxylate chain (polyoxyethylene glycol) usually ranging from 2 to about 20 units (see Fig. 1). Sometimes a combination of ethoxylated and propoxylated parts comprise the hydrophilic structure [2]. The non-polar, hydrophobic region consists of linear or branched alkylphenols and alcohols. The most commonly used AE contain a linear and partially $\alpha$ methyl-branched hydrocarbon chain of between 12 and 15 carbon atoms. The APE usually contain a branched hydrocarbon chain of 8, 9 or 12 carbon atoms. The highly branched carbon chains originate from the polymerisation of propylene and butylene which gives rise to a wide number of isomers. The major part of the APE and some of the AE, viz. those with a chain of 11 or 13 carbon atoms, are derived from these branched materials. Thus, the non-ionic surfactants considered here consist of several hundreds of structurally different compounds with molecular masses ranging from about 200 to 1200. Their homologous behaviour is substantiated by their chromatographic retention characteristics, which have been shown to vary linearly with the number of ethoxylate groups [6]. Separation of all the different homologues, oligomers and isomers of the non-ionic surfactants in one analysis seems almost unachievable. This is why the analysis of non-ionic surfactants is one of the most difficult in the environmental analytical field.

## 2. Extraction and enrichment

The technique most commonly applied to extract non-ionics from aqueous samples has

been a procedure called sublation. In this technique, fine bubbles of an inert gas (typically nitrogen or helium) are dispersed through the aqueous sample, thereby transferring the surface active materials into an overlaying organic layer (usually ethyl acetate, because of its relatively high partition coefficient for APE compared to e.g. *n*-hexane or 1-octanol [16]). The coextracted ionic surfactants are subsequently removed by ion-exchange chromatography.

Centrifugal partition chromatography (CPC) has been used as an alternative to the sublation extraction of NPE [16]. In CPC, the waste or surface water acts as the mobile phase and the extractant as the stationary phase. Among 1-octanol, hexane and ethyl acetate, the latter solvent was selected as the most suitable organic extractant on the basis of prior shake-flask measurements of partition coefficients for each solvent–water system. The major advantage of CPC over sublation extraction is its ability to concentrate sample volumes of several litres into organic solvent amounts of several tens of ml. Disadvantages of CPC are in the formation of emulsions when a relatively large loading of the stationary phase occurs.

Among the various other extracting procedures suggested, such as, e.g., Soxhlet extraction [17] and steam distillation [18,19], preconcentration chromatography with macroreticular resins such as the Amberlite XAD resins in glass columns has been shown to be very successful in extracting organics, including non-ionic surfactants, from aqueous samples [20–22]. This procedure requires a carefully selected elution scheme to desorb the APE and AE and further cleanup by silica adsorption as well as liquid–liquid chromatography [23]. This method can be considered a predecessor of the nowadays widely applied technique of solid phase extraction (SPE) for extraction and enrichment of organics from aqueous samples.

SPE has been shown to be a very powerful and robust alternative to the traditional methods of extraction of organic compounds from various matrices [24,25]. Moreover, SPE offers the advantage of immediate analyte enrichment, thus reducing analyst time and the amount of necessary solvents significantly. Various types of commercially available adsorbents in prepacked cartridges enable the application of different separation mechanisms [25], thereby enhancing the versatility of the modern analyst.

As pointed out by Marcomini et al. [17], for surfactants every surface can act as a potential adsorbent. In principle, therefore, many materials may be suitable for adsorption chromatography-type separation and isolation. On the other hand, this behaviour also implies that chromatographic systems may easily become contaminated with surfactants prior to their actual use. For SPE of AE, APE and NP, commercial reversed-phase octadecylsilica cartridges have been applied successfully to extract and enrich them from water and wastewater samples [17,26]. Seawater samples could also be extracted and enriched in this way [27]. Solid-phase $C_{18}$ extraction disks have been applied as a pre-extraction step for non-ionics from aqueous solution with subsequent extraction of the disks by supercritical fluid extraction (SFE) [28]. Through SFE, (part of the) extraction and cleanup is combined into a single step. The technique is certainly promising, since a selective extraction of AE and APE was obtained [28]. Its applicability to environmental samples remains to be shown, however. An $\alpha$-hydroxyethylated styrene-divinylbenzene resin was used successfully for the preconcentration of a non-ionic from river water [29]. Even in the presence of artificial humic acids at a concentration of 100 mg $l^{-1}$ good recoveries were obtained. Serial combinations of ion-exchange resins and $C_{18}$ SPE have also been used for clean up and extraction of NPE, without distortion of the oligomer distribution [19].

Concludingly, as in many other extraction schemes for organic micropollutants, SPE is taking over the more time-consuming and elaborate methods traditionally used for extraction of non-ionics from aqueous environmental samples.

## 3. Cleanup

The separation of non-ionic compounds from other ionic surfactants is usually accomplished by ion-exchange chromatography. The non-ionics

pass the ion exchanger together with other, non-surfactant materials. The resulting mixture thus contains a large variety of non-ionics, which requires further cleanup and fractionation prior to final detection. Owing to the polymeric nature of the polyethoxylated compounds, physical properties of individual oligomers will vary over a wide range. Among the first attempts to tackle this problem were liquid–liquid extraction and several chromatographic techniques, including silica column adsorption chromatography [30]. The advantage of normal-phase chromatography over ion-exchange cleanup is that normal phase cleanup combines the properties of ion exchange and chromatography, i.e. binding of strongly ionised molecules and separation of the non-ionic substances [31].

Alternatively, flow injection with on-line cation-exchange solid-phase extraction can be used to separate non-ionics from charged interferences in the sample. This method has been used to separate a non-ionic emulsifier from a drug synthesis reaction medium, to which it was added as solubility enhancer, and detect it by UV [32].

RP-HPLC with diode-array detection has been used to fractionate ether insoluble organic extracts of raw and drinking water prior to the determination with FAB mass spectrometry [33]. The fractions contained a broad range of surfactants as AE, APE, fatty acids, polyethylene glycols, polyethylenepropylene glycol block polymers and alkyl ether sulphates.

Hence, the more traditional approach of using separate cleanup steps including ion exchange as well as liquid–liquid extraction tends to be overtaken by column adsorption chromatography, SPE and/or SFE [28] and related techniques.

## 4. Separation and detection

### *4.1. High-performance liquid chromatography*

The major advantage of HPLC is its ability to separate and quantitate the various homologues and oligomers by length of the alkyl and ethoxylate chains. Reversed-phase HPLC provides information about the alkyl chain length, whereas normal-phase HPLC resolves the ethoxylate oligomers. LC detection of non-ionic surfactants can be accomplished either directly, when a chromophore is present in the molecule (APE), or by derivatisation (of AE) with an appropriate reagent. In theory the same analytical techniques which are used for the analysis of AE can be applied to APE. Dependent on the derivatisation reagent, UV- or fluorescence detection is applied to the analysis of AE. APE posses a ring chromophore which enables direct UV (at 277–280 nm) or fluorescence detection using excitation and emission wavelengths of 230 and 302–310 nm, respectively. For the analysis of APE in environmental samples HPLC with fluorescence detection provides the most simple and suitable technique [2].

Reversed-phase HPLC is more suitable for quantitative analysis of AE in environmental samples than normal-phase HPLC because in reversed-phase LC the most commonly used $C_{12}$–$C_{15}$ AE are separated from NPE, which is the most abundantly used APE [31]. The total concentration of $C_{12}$–$C_{15}$ AE can be quantified with a commercial mixture of AE, e.g. Neodol 25/9, because the ethoxylate chain of AE is hardly affected by biodegradation in the environment [26]. The major biodegradation pathways of AE are intramolecular scission and $\beta$-oxidation of the hydrocarbon chain [2], which both leave the ethoxylate chain unchanged. An example of a reversed-phase chromatogram of commercial $C_{12}$–$C_{15}$ AE is shown in Fig. 2, demonstrating a typical homologue distribution. At trace levels in environmental samples reversed-phase HPLC analysis of AE can suffer from interferences by a high abundance of APE and highly branched AE ($C_{11}$ and $C_{13}$).

Thermospray LC–MS currently provides the most selective and sensitive analysis of AE [26,34]. This method allows the determination of total and individual AE species at the ppb level. In addition, the method distinguishes branched AE from linear ethylene-based AE, which may coelute.

Normal-phase HPLC is often applied to obtain information about the ethoxylate chain distribution of AE and APE. An example of a normal-phase HPLC chromatogram of NPE is shown in Fig. 3. APE are often quantitatively analysed by

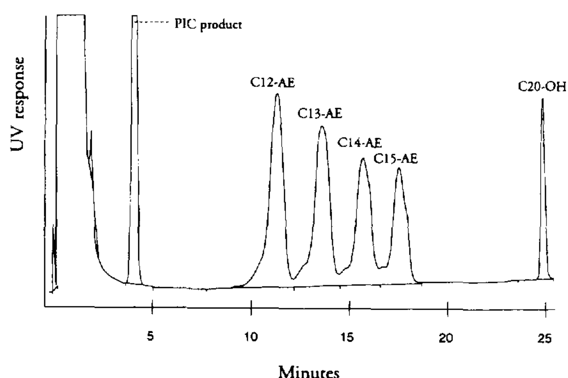


Fig. 2. Reversed-phase chromatographic separation of alkyl ethoxylate homologues. Conditions: column, 125 × 4 mm LiChrocart C-18, 5 μm; mobile phase, linear gradient elution, 80:20 (v:v) methanol–water → 100% methanol in 25 min; UV-detection, 235 nm.

normal-phase HPLC because biodegradation of APE involves stepwise shortening of the ethoxylate chain. Normal-phase HPLC enables the separation of the persistent alkylphenols and lower ethoxylated APE.

An overview of chemical analysis of AE and APE by normal and reversed-phase HPLC is given by Garti et al. [35]. It can be concluded from their review that a wide range of possible columns (ranging from, e.g., RP2 to RP18 in

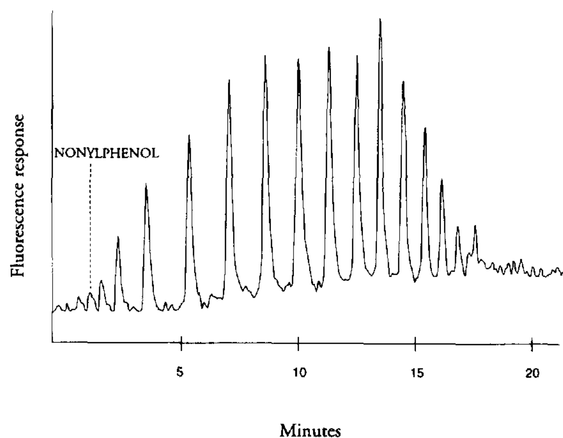


Fig. 3. Normal-phase chromatographic separation of APE oligomers. Conditions: column 100 × 4.6 mm $NH_2$-Hypersil, 3 μm; mobile phase, linear gradient, n-hexane–isopropanol–water 93.1:6.8:0.1 (v/v) → 44.1:49.9:6.0 in 20 min; fluorescence detection, excitation 230 nm, emission 295 nm.

reversed-phase), eluents and detection techniques can be used for the analysis of AE and APE.

Summarising the above, for the routine measurement of AE and APE, reversed-phase HPLC and normal-phase HPLC are recommended, respectively. LC–MS analysis is currently the only technique which enables the analyst to obtain complete information on the occurrence and distribution of homologues, oligomers and isomers at the ppb level.

### *4.2. Gas chromatography and supercritical fluid chromatography (SFC)*

Due to the low volatility of the higher ethoxylate oligomers, AE cannot be easily analysed directly by GC. Only a combination of trimethylsilyl derivatisation of AE and capillary high-temperature GC allows separation of $C_{12}$–$C_{18}$ AE with an ethoxylate chain length of as much as 21 EO units [36,37]. A drawback of high-temperature GC analysis is that sample degradation for the high molecular mass AE and APE might occur. High-temperature capillary columns are coated with a stabilized bonded polysiloxane film and allow for temperatures up to 400°C.

For analysis of the lower APE (NP and NPE with one and two ethoxylate groups) in effluents of sewage treatment plants a direct capillary gas chromatography–mass spectrometry method has been applied successfully [5]. Derivatisation techniques are also applicable to these lower APE. GC–ECD and GC–MS detection of NP and NPE with 1, 2 or 3 ethoxylate units can be used, e.g., after derivatisation to pentafluorobenzoyl derivatives [38]. As with the AE, the higher ethoxylates of the APE can only be analysed by GC after derivatisation and with the use of a high-temperature column. An additional advantage of derivatisation of AE is that it results in improved separation of oligomers [36].

Wee developed a GC method based on the cleavage of the AE by hydrogen bromide into alkyl bromides [30,40]. This method is comparable with reversed-phase HPLC in its provision of information on the alkyl distribution and its

sensitivity towards possible interferences from APE and branched isomers.

SFC in principle can be a very suitable means of analysis due to the high solubility of non-ionics in $CO_2$ [41]. SFC is even capable of analysing higher molecular mass AE [28] but has the disadvantage of not fully resolving the higher alkyl homologues [36]. As pointed out in the section on extraction, SFE–SFC has shown to be successful in selectively extracting and determining of AE and APE from aqueous samples [28]. Oligomer separation of nonylphenols by SFC was shown to be more complete than by HPLC, with shorter elution times [42]. However, to our knowledge these techniques have not yet been applied to environmental samples.

Compared to HPLC and traditional GC, high-temperature GC allows the separation of all the homologues and oligomers of the most commonly applied non-ionic surfactants. However, this technique has never been applied to environmental samples yet. Serious obstacles, such as coelution and interferences, still need to be surmounted.

## 5. Conclusions and future developments

The different steps of the traditional non-specific analytical procedures for the routine determination of non-ionic surfactants in aqueous environmental samples have been replaced gradually by more specific chromatographic procedures. Proper alternatives for the sublation extraction and ion-exchange cleanup are provided by solid-phase extraction and column adsorption chromatographic cleanup, respectively. Beside their specificity, these techniques are recommended because they allow automation of the entire analytical procedure. The application of chromatographic techniques in all separate steps of the entire procedure for the determination of non-ionics has led to significant improvements in the areas of homologue and oligomer separations as well as detection limits. In particular the various SPE techniques have improved the latter by more than 100-fold as compared to the traditional methods of analysis [29]. Analysis at the ppb level is no longer a problem.

The separation of all the different homologues, isomers and oligomers in one analytical run remains the most challenging research question in the analysis of non-ionic surfactants. For the determination of AE in aqueous samples this has been achieved by the combination of RP-HPLC and mass spectrometry. This hyphenated technique yields information on both alkyl and ethoxylate chain distribution, as well as on the occurrence of branched isomers. Further work along these lines regarding the analysis of APE is indicated.

Further improvement of oligomer and homologue resolution of AE and APE in environmental samples must be sought in high-resolution chromatography. Examples are high-temperature GC, capillary electrophoresis-based methods and capillary gel permeation chromatography. Separation of a complex $C_{12}$–$C_{18}$ mixture has been achieved by high-temperature GC [36]. However, its possible merits still need to be demonstrated on environmental samples.

A promising technique which has not been applied until now is the analysis of non-ionic surfactants in a coupled reversed- and normal-phase LC system. The reversed-phase LC separates and passes the alkylchain homologues to the normal-phase column where the ethoxylate oligomers are separated. Obviously, a proper choice of solvents or solvent exchange is required here. If desired, the sequence of normal- and reversed-phase separation can be inverted. Either on-line column switching or heart-cut type systems are feasible. In this way information can be obtained on both alkyl homologues and ethoxylate oligomers without the need of highly sophisticated detectors.

## References

[1] J. Falbe, Surfactants in Consumer Products, Springer-Verlag, Berlin, 1987.

[2] M.S. Holt, G.C. Mitchell and R.J. Watkinson, in O. Hutzinger (Editor), The Handbook of Environmental Chemistry, Vol. 3, Part F, Springer-Verlag, Berlin, 1992, p. 89.

[3] M.A. Lewis, Ecotoxicol. Environ. Safety, 20 (1990) 123.
[4] M.A. Lewis, Water Res., 25 (1991) 101.
[5] W. Giger, E. Stephanou and C. Schaffner, Chemosphere, 10 (1981) 1253.
[6] M. Szögyi and T. Cserháti, J. Planar Chromatogr., 5 (1992) 267.
[7] T. Cserháti, J. Biochem. Biophys. Methods, 27 (1993) 133.
[8] M. Ahel and W. Giger, Anal. Chem., 57 (1985) 2584.
[9] S. Jobling and J.P. Sumpter, Aquat. Toxicol., 27 (1993) 361.
[10] E. Matthijs and E.C. Hennes, Tenside Surf. Det., 28 (1991) 22.
[11] R. Wickbold, Tenside Surf. Det., 9 (1972) 173.
[12] S.L. Boyer, K.F. Guin, R.M. Kelley, M.L. Mausner, H.F. Robinson, T.M. Schmitt, R.C.Stahl and E.A. Setzkorn, Environ. Sci. Technol., 11 (1977) 1167.
[13] D. Brown, H. de Henau, J. Carrigan, P. Gerike, M. Holt, E. Keck, E. Kunkel, E. Mathijs, J. Waters and R.J. Watkinson, Tenside Surf. Det., 23 (1986) 190.
[14] D. Brown, H. de Henau, J. Carrigan, P. Gerike, M. Holt, E. Keck, E. Kunkel, E. Mathijs, J. Waters and R.J. Watkinson, Tenside Surf. Det., 24 (1987) 14.
[15] S.J. Patterson, C.C. Scott and K.B.E. Tucker, J. Am. Oil Chem. Soc., 44 (1967) 407.
[16] R.A. Menges, T.S. Menges, G.L. Bertrand, D.W. Armstrong and L.A. Spino, J. Liq. Chromatogr., 15 (1992) 2909.
[17] A. Marcomini, S. Capri and W. Giger, J. Chromatogr., 403 (1987) 243.
[18] M. Ahel and W. Giger, Environ. Sci. Technol., 21 (1987) 697.
[19] E. Kubeck and C.G. Naylor, J. Am. Oil Chem. Soc., 67 (1990) 400.
[20] P. Jones and G. Nickless, J. Chromatogr., 156 (1978) 87.
[21] T. Saito and K. Hagiwara, Fresn. Z. Anal. Chem., 312 (1982) 533.
[22] E.M. Thurman, T. Willoughby, L.B. Barber and K.A. Thorn, Anal. Chem., 59 (1987) 1798.
[23] P. Jones and G. Nickless, J. Chromatogr., 156 (1978) 99.
[24] M.W.F. Nielen, R.W. Frei and U.A.Th. Brinkman, J. Chromatogr. Library, Vol. 39A, Elsevier, Amsterdam, 1988, Ch.1, p. 5.
[25] P. de Voogt, Trends Anal. Chem., 13 (1994) 389.
[26] E. Matthijs, M.S. Holt, A.T. Kiewiet and G. Rijs, Environ. Sci. Technol., submitted.
[27] A. Marcomini, S. Stelluto and B. Pavoni, Int. J. Environ. Anal. Chem., 35 (1989) 207.
[28] M. Kane, J.R. Dean, S.M. Hitchen, C.J. Dowle and R.L. Tranter, Anal. Proc., 30 (1993) 399.
[29] I. Fujita, K. Nishiyama, Y. Nagano, K. Harada, M. Nakayama and A. Sugii, Int. J. Environ. Anal. Chem., 56 (1994) 57.
[30] M.J. Rosen, Anal. Chem., 35 (1963) 2074.
[31] A.T. Kiewiet, J.M.D. van der Steen and J.R. Parsons, Anal. Chem., 67 (1995) 4409.
[32] C. Moeder, N. Grinberg, H.J. Perpall, G. Bicker and P. Tway, Analyst, 117 (1992) 767.
[33] J. Rivera, F. Ventura, J. Caixach, M. de Torres and A. Figueras, Int. J. Environ. Anal. Chem., 29 (1987) 15.
[34] K.A. Evans, S.T. Dubey, L. Kravetz, I. Dzidic, J. Gumulka, R. Mueller and J.R. Stork, Anal. Chem., 66 (1994) 699.
[35] N. Garti, V.R. Kaufman and A. Aserin, in J. Cross (Editor), Nonionic Surfactants: Chemical Analysis, Marcel Dekker, New York, NY, Ch. 7, 1987.
[36] A.H. Silver and H.T. Kalinoski, J. Am. Oil Chem Soc., 69 (1992) 599.
[37] H.T. Rasmussen, A.M. Pinto, M.W. DeMouth, P. Touretzky and B.P. McPherson, J. High Resolut. Chromatogr., 17 (1994) 593.
[38] C. Wahlberg, L. Renberg and U. Wideqvist, Chemosphere, 20 (1990) 179.
[39] V.T. Wee, Adv. Identif. Anal. Org. Pollut. Water, 1 (1981) 467.
[40] N.J. Fendinger, W.M. Begley, D.C. McAvoy and W.S. Eckhoff, Environ. Sci. Technol., 29 (1995) 856.
[41] K.A. Consani and R.D. Smith, J. Supercrit. Fluids, 3 (1990) 51.
[42] Z. Wang and M. Fingas, J. Chromatogr. Sci., 31 (1993) 509.