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# Lauryl Alkylbenzene Sulfonates in the Urban Water Cycle (Toulouse, France)

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## **Abstract**

Application of the European Water Framework Directive requires Member States to have better understanding of the quality of surface waters in order to improve knowledge of priority pollutants. Xenobiotics in urban receiving waters are an emerging concern. This study proposes a screening campaign of laurylalkylbenzene sulfonates in a separated sewer system. An analytical method by solid-phase extraction and liquid chromatography coupled with mass spectrometry detection was developed providing satisfactory detection and quantification limits. Ten sites and seven types of waters (wastewaters, treated wastewaters, ground water, rainwaters, roof collected waters, run-off waters and carwash wastewaters) were investigated over one year in Toulouse (France) using quantitative monitoring. Ground, rain and roof collected water concentrations are similar to treated wastewater levels. Run-off water was the most polluted and impacted the aquatic environment. Washing station were responsible for 75% of the LAS load in stormwaters. The wastewater treatment plant reduced laurylalkylbenzene sulfonates concentrations by 90% before discharge into the environment. The results showed that laurylalkylbenzene sulfonates concentrations were variable over time and space in all urban water compartments. No correlation between laurylalkylbenzene sulfonates concentrations and global water pollution parameters was observed.

**Keywords:** Surfactant; Pollution; Quantification; liquid chromatography; mass spectrometry

## **Introduction**

Linear alkylbenzene sulfonates (LAS) are synthetic anionic surfactants, produced since the 1960s. They were introduced to substitute highly branched alkyl benzene sulfonates which were slowly biodegradables [1]. In 2003, worldwide consumption of LASs has been estimated at about 18 million tonnes [2]. Thus, it is the most surfactants used in detergents and cleaning products (shampoos, personal care products ...). They consist of a mixture of homologues differing in the number of carbons in the aliphatic chain. For each homologous, positional isomer exist depending on the position of the benzene sulfonate group on the aliphatic chain. Commercially, produced LASs are mixtures containing homologues with alkyl chains ranging from 10 to 13 carbon atoms [3]. Their detergency depends mainly on the position of the benzene sulfonate group from the alkyl chain and the length of this channel [4]. These surfactants enter into wastewater treatment plants (WWTP) and are dispersed into the environment through effluent discharges into surface waters. They could also be discharged directly in surface waters [1]. LASs were found in range of concentrations of 1.6 to 3.7  $\mu\text{g}\cdot\text{L}^{-1}$  in drinking waters and between 14 and 155  $\mu\text{g}\cdot\text{L}^{-1}$  in river water [5]. In WWTP, reported values of LASs are between 104 and 1920  $\mu\text{g}\cdot\text{L}^{-1}$  in wastewaters and between 11 and 872  $\mu\text{g}\cdot\text{L}^{-1}$  in treated wastewaters [6]. Evaluation of LAS levels in environmental samples were principally performed by high-performance liquid chromatography (HPLC) coupled with UV-detection ([7]; [8]) or fluorescence detection ([3]; ([9]; [10]; [13])). Methods using mass spectrometric detection were more rare [6]. Thus, the aim of this study was to develop and optimize an analytical method to quantify the LASs in different water samples by liquid chromatography coupled with mass spectrometry detection in order to determine LAS concentrations in the urban water cycle of Toulouse (France).

## **Experimental**

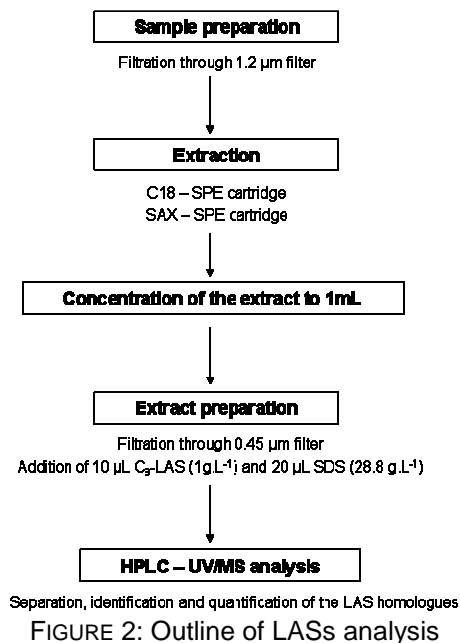
### **Reagents and standards**

All chemicals used were analytical quality. Methanol, acetonitrile and hydrochloric acid 37% Multisolv HPLC grade were purchased from Scharlau (France). N-hexane Suprasolv and ammonium acetate were obtained by VWR Merck (France). Condea Chimie SARL supplied the Marlon ARL which is a commercial surfactant powder containing 80% of C<sub>10</sub>–C<sub>13</sub> LAS. This commercial homologue LAS mixture has the following homologue mass distribution: C<sub>10</sub> (14.3%), C<sub>11</sub> (35.7%), C<sub>12</sub> (30.8%), and C<sub>13</sub> (19.2%). C<sub>8</sub>-LAS from Alfa Aesar (France) was used as internal standard at 10 mg.L<sup>-1</sup> in each extract. Sodium DodecylSulfate (SDS; 99% purity) was purchased from Acros Organics (France). It was added in extract at the concentration of 2 mM. GF/C filters (glass microfibre; 1.2 µm) from Whatman (France) were used for filtration of water samples. Lichrolut RP-18e SPE cartridges (500 mg; 3 mL) were obtained by VWR Merck (France) and SAX (Strong Anionic eXchange) SPE cartridges (500 mg; 3 mL) were purchased from Varian (France). Syringe filters in PTFE with a 0.45 µm pore size from VWR Merck (France) were used to filtrate extract before analysis.

### **Sample collection**

Nine sites were investigated in Toulouse in order to evaluate the LASs contamination. Ground water was collected under a normally frequented urban road. Rainwaters were collected in a zone free from any overhanging interference. Roof collected waters were taken from buildings in the town centre. The town of Toulouse is equipped with a separated sewer system where pollutants in wastewater system could not mix with the stormwater. The two main outlets of Toulouse (France) were selected in order to evaluate the stormwater quality. Outlet 1 fed from an urbanised catchment area of 439 hectares. Outlet 2 drains water of a low urbanised area which corresponds to a catching area of 1428 hectares. Three carwashes were investigated: truck carwash which trucks were washed manually, self-service carwash for car and motorcycles equipped with self-service high-pressure water jet and petrol station carwash equipped with self-service high-pressure water jet and washing roller brushing. These three sites discharged wastewaters into the stormwater network after a pre-treatment process composed of a scrubber and an oil separator. The Toulouse wastewater treatment plant (WWTP) was also investigated. This treats about 125 000 m<sup>3</sup> per day of wastewaters and discharges its effluent into the River Garonne. It is composed of a pre-treatment grid, sand trap and degreaser plus three treatment units: G1 (400 000EH), G2 (150 000EH), G3 (250 000EH), followed by a nitrification unit G4 (800 000EH) which treats all water from G1, G2 and G3, before discharging it into the River Garonne. In this study, wastewater entering unit G1 (biological treatment using activated sludge) and treated wastewaters (after unit G4) were investigated. Figure 1 shows the locations of the sampling sites.





Water samples were filtered through GF/C filters to remove suspended solids in order to avoid clogging of the cartridge. Water sample extractions were performed with Lichrolut RP-18e SPE cartridges (500mg; 3mL) pre-conditioned with 10 mL of methanol followed by 10 mL of milliQ-water. 1000 mL of water sample was transferred to the SPE cartridge using a vacuum manifold system (Supelco). SPE cartridge was rinsed with 5 mL of milliQ-water followed by 5 mL of methanol/milliQ-water mixture (20/80; v/v). LAS were eluted with 10 mL of methanol. This extract was then transferred to a SAX SPE cartridge pre-conditioned with 3mL of n-hexane followed by 10 mL of methanol. LAS were eluted with 2 mL of hydrochloric acid/methanol mixture (20/80; v/v). The final extract was concentrated under a gentle stream of nitrogen and redissolved in 1mL of methanol. The extract was filtered through a 0.45 µm syringe filter. Then, 10 µL of internal standard C<sub>8</sub>-LAS at 1 g.L<sup>-1</sup> and 20 µL of SDS at 28.8 g.L<sup>-1</sup> were added to the extract.

### Sample analysis

Chromatographic analysis was performed on a Dionex high performance liquid chromatography (Dionex, France) equipped with a SOR100 degasser, a P680 HPLC pump, a ASI 100 autosampler, a TCC 100 thermostated column compartment and a UVD340U ultraviolet diode array. A mass spectrometer MSQ Surveyor (Thermo Finnigan, France) was connected on line. The system was computer-controlled with data acquisition and processing using Chromeleon (Dionex) software.

LASs separation was carried out using a Lichrospher® 100 RP-18e (250x4 mm id, 5µm) column protected by Lichrospher® 100 RP-18e (4x4 mm id, 5µm) guard column (VWR Merck, France). The injection volume was set at 30 µL, the column was thermostated at 25°C and the flow rate was 0.8 mL.min<sup>-1</sup>. The elution was performed with a gradient composed of milliQ-water (A) and a solution of acetonitrile/milliQ-water (80/20; v/v) containing 2 mM of ammonium acetate (B). The gradient programme for the first 3min was 30% B and was increased linearly to 100% B over 10 min, then kept isocratic for 2 min and decreased linearly to 30% B over 2 min (Figure 3).

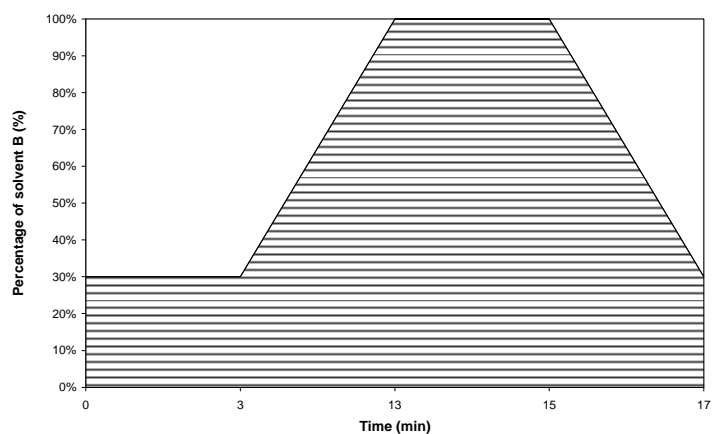


FIGURE 3: Diagram of gradient used for the separation of LAS homologues. Flow-rate=0.8 mL.min<sup>-1</sup>

LASs detection was carried out using UV detector at 224 nm and mass spectrometer equipped with an ESI probe in negative mode (Figure 4 and 5). The ESI conditions were as followed: probe temperature 350°C, capillary tension 3 kV, ion fragmentation energy 80V. The fragments (m/z) used for the identification and quantification of LASs were: 297 (C<sub>10</sub>-LAS), 311 (C<sub>11</sub>-LAS), 325 (C<sub>12</sub>-LAS), 339 (C<sub>13</sub>-LAS), 270 (C<sub>8</sub>-LAS) and 183 as common fragment ion.

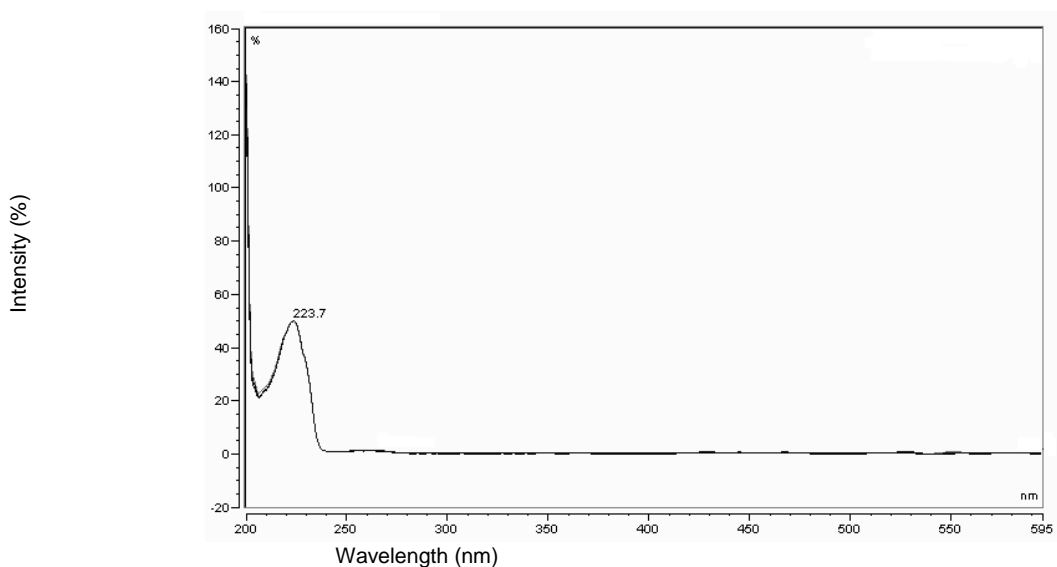


FIGURE 4: UV spectrum of LAS

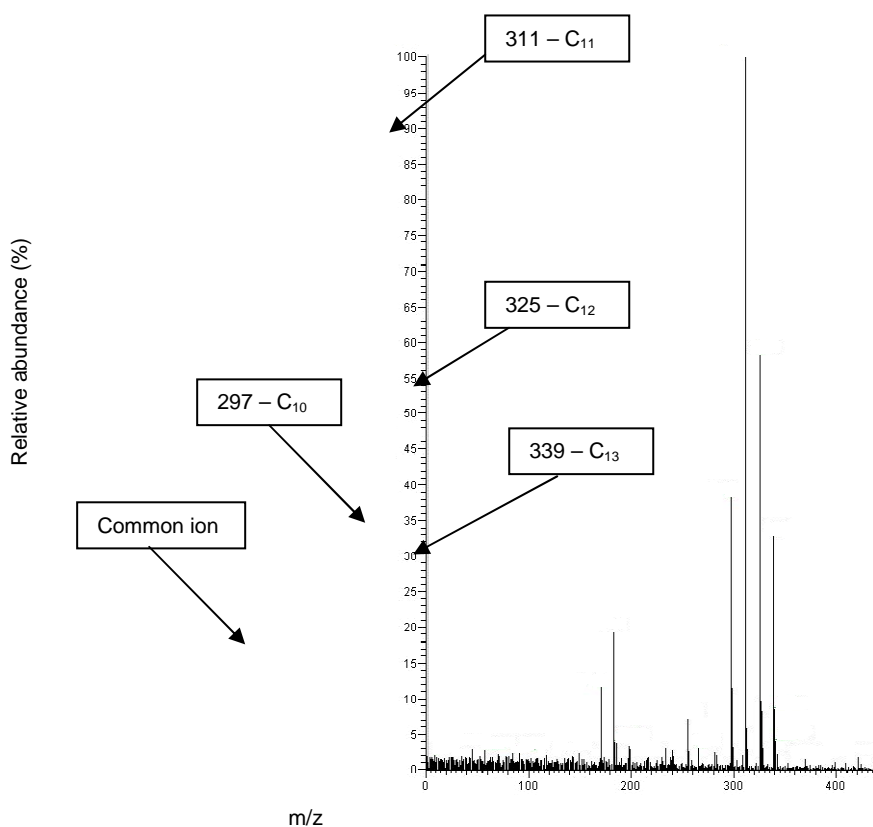


FIGURE 5: Mass spectrum of LAS

### Multivariate data analysis

The multivariate data analysis simulation was performed using the commercial software XL stat. The objectives of Principal Component Analysis (PCA) are to find and interpret hidden complex and casually determined relationships between dataset. The key idea is to study the data structure in a reduced dimension while retaining the maximum amount of variability present in the data. A matrix of pairwise correlations among compounds concentrations is decomposed into eigenvectors, which, are sorted in descending order of their corresponding eigenvalues. In this work, the variables were standardized in order to ensure that they have equal weights in the analysis (mean is equal to zero and the standard deviation is equal to the unit). Then, the calculation of the covariance matrix by identifying the eigenvalues and their corresponding eigenvectors was carried out.

## Results and discussion

### Chromatographic separation, calibration and limits of detection

Figure 6 shows the chromatogram resulting from the methodology described above applied to a standard mixture of LASs. LASs were separated in 17 min, with retention time of 11.1 min, 11.9 min, 12.6 min and 13.3 min for C<sub>10</sub>, C<sub>11</sub>, C<sub>12</sub>, C<sub>13</sub>-LAS, respectively. The quantitative calculations are made from the peak area corrected with C<sub>8</sub> peak area. LASs were determined as the sum of homologous C<sub>10</sub> to C<sub>13</sub> LASs. The calibration curve was obtained with standard solution of LASs. The linear range was set between 0.01 µg.L<sup>-1</sup> and 3 mg.L<sup>-1</sup> for the sum of LASs (C<sub>10</sub>-C<sub>13</sub>), with regression coefficient (r<sup>2</sup>) above 0.99.

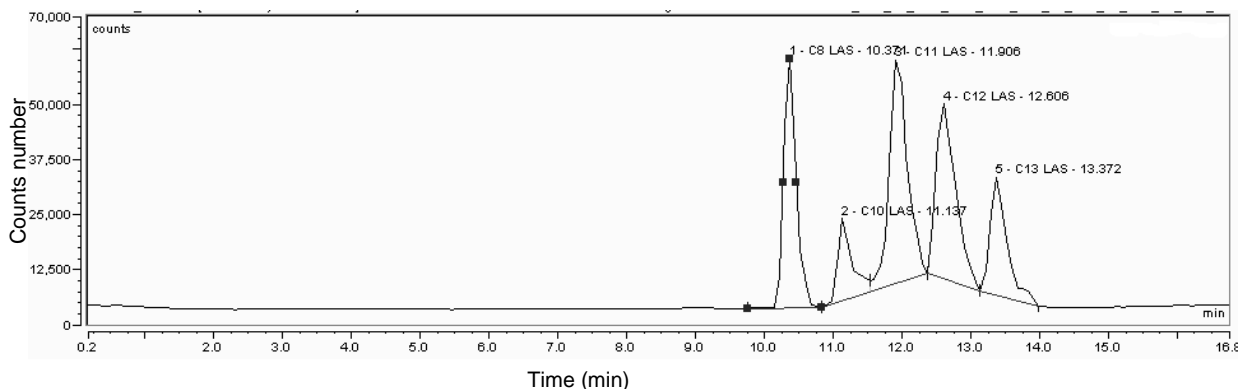


FIGURE 6: HPLC–MS chromatogram of LASs standard mixture at 5 mg.L<sup>-1</sup>

The precision of the chromatographic determination was evaluated by standard solution of 5 mg.L<sup>-1</sup>. The standard solution was analysed ten times and the relative standard deviation (%) was calculated. The repeatability of the chromatographic determination was found to be 7% for the sum of LASs. Reproducibility of the method was tested on EVIAN water spiked with three concentrations of the sum of LASs (0.7, 10 and 300 µg/L). Extraction was repeated three times for each concentration at different days. The  $\Sigma$  LAS (C<sub>10</sub>-C<sub>13</sub>) percentages of recovery for each concentration were respectively in the 120 ± 5 %, 84 ± 6 % and 60 ± 3 %. The percentages of recovery were good for the concentration of 0.7 and 10 µg.L<sup>-1</sup>. For the concentration of 300 µg.L<sup>-1</sup>, the recovery was lower than other due to the possible saturation of the cartridge during the extraction time. In an other hand, the matrix which is moderately mineralized could also interact with the sorbent and induce a decrease of the percentages of recovery. Limit of detection, determined as 3 standard deviations above the mean blank signal, was 0.01 µg.L<sup>-1</sup> for the sum of LASs. Limit of quantification was determined as 10 standard deviations above the blank signal and was equal to 0.03 µg.L<sup>-1</sup>. The method developed in this paper allows to obtain limits lower than those obtained by other works ([3]; [10]; [12]; [13]).

#### Concentrations in environmental samples

LAS concentration was determined for the ten types of water investigated in Toulouse. The results of analysis were summarized in Table 1. The minimum, maximum, mean and standard deviation (SD) values were calculated. Values less than the quantification limit were taken as zero for statistical calculations.

TABLE 1: Statistical data on LAS concentrations found in studied waters

Type of water	n	Minimum (µg.L <sup>-1</sup> )	Maximum (µg.L <sup>-1</sup> )	Mean (µg.L <sup>-1</sup> )	SD (µg.L <sup>-1</sup> )
Ground water	4	0.01	0.03	0.02	0.01
Rainwaters	4	0.03	0.21	0.09	0.08
Roof collected waters	4	0.02	0.09	0.04	0.03
Run-off waters	20	6.20	920	252	244
Truck carwash	5	0.005	53.0	14	23.0
Self-service carwash	5	8 100	64 000	20 120	24 540
Petrol station carwash	5	46.0	3 000	719	1 280
Wastewaters	8	2.70	6.80	3.84	1.36
Treated wastewaters	8	0.08	0.14	0.11	0.02

A schematic representation and box plot representation of LASs concentration in the different waters was shown in Figure 7 and 8 respectively.



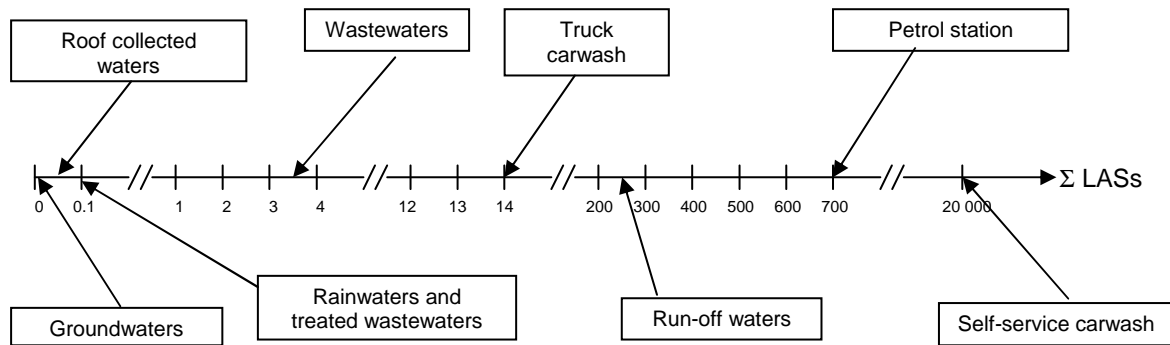


FIGURE 7: Schematic representation of mean concentration of LASs in studied waters

According to Figure 7 and Table 1, wastewaters from self-service carwash were the most polluted water and presented LAS values higher than those observed in literature for wastewater ([13]; [6]). The petrol station carwash studied discharges  $700 \mu\text{g.L}^{-1}$  of LASs in mean into the stormwater network. LAS concentrations observed from petrol station carwash were in order of magnitude of values observed for surface waters in England [9] and in Spain [12]. LAS concentrations found in wastewater from truck carwash are low compared with data from surface waters in England (mean= $147 \mu\text{g.L}^{-1}$ ) [9] and in Spain (mean= $44 \mu\text{g.L}^{-1}$ ) [12]. The difference of LAS concentrations between self-service carwash and petrol station carwash could be explained by the difference between carwash processes, perhaps high-pressure water jet consumes more detergent with LASs than washing roller brushing. Moreover, the self-service carwash could be more used than the petrol station carwash.

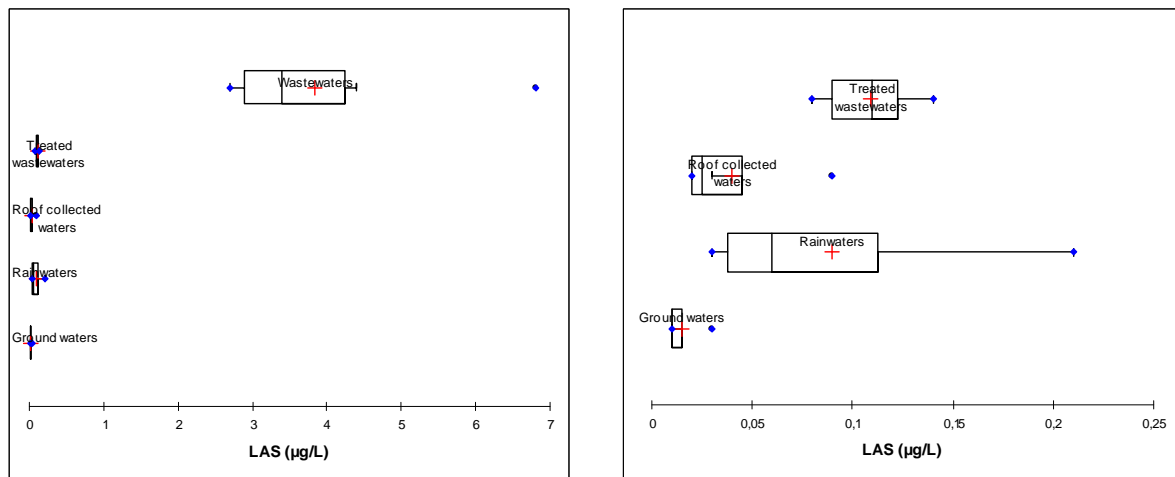


FIGURE 8: Box plots of LAS concentrations by sampling site, noting the minimum and maximum values, the median (—) and the mean (+)

The wastewater treatment plant (WWTP) reduced LAS concentrations by 90% before discharge into the environment. LAS concentrations in wastewaters from WWTP were lower than those found in the literature for wastewaters from WWTP in Spain (mean= $837 \mu\text{g.L}^{-1}$ ) [6], in Jordan (mean= $10.21 \mu\text{g.L}^{-1}$ ) [8] and in Italy (mean= $6329 \mu\text{g.L}^{-1}$ ) [13]. The levels observed in WWTP entry are lower than those of wastewaters from carwash. A Spanish study reported LAS mean concentrations for treated wastewaters ( $590 \mu\text{g.L}^{-1}$ ) higher than those observed in this study [6]. Two other studies reported LAS values of  $2.59 \mu\text{g.L}^{-1}$  [8] and  $68 \mu\text{g.L}^{-1}$  [13] which were higher than values found in treated wastewaters in Toulouse.

Mean values observed for the two outlets were comparable to concentrations found for surface water in Turkey (mean= $224 \mu\text{g.L}^{-1}$ ) [11] and in England [9]. Ground water, rainwaters and roof collected waters have low concentrations of LASs. Rejected waters by the outlets to the environment were more polluted than those rejected by WWTP. Wastewaters entering in unit treatment G1 of WWTP were less concentrated in LASs than stormwaters.

As carwashes are allowed to reject wastewaters into the stormwater network after a pretreatment, a comparison of loads from carwashes to pollution flows at outlets was performed. Thanks to an inventory of the organizations owning discharges licences and a modelisation scenario, we manage to

establish the composition of the two outlets: two self-service carwashes, two petrol station carwashes and one truck carwash. Thus, for run-off waters (outlet 1 and 2), 75% of the LAS pollution could be attributed to the carwash discharges [13].

### Correlation study

Principal Component Analysis was carried out in order to find relationships between LAS and global parameters of water pollution. More than LAS levels, samples were measured for pH, conductivity, turbidity, chemical oxygen demand (COD), nitrogen (Nt), phosphorus (Pt), suspended solid (MES) and volatile suspended solid (MVS). A data matrix, with columns representing the different samplings (observations) and rows corresponding to the measured parameters (variables), was constructed. A total of 48 complete observations were selected for analysis. Table 2 presents the statistical data on the different parameters measured.

TABLE 2: Statistical data on different parameters concentrations found in studied waters

Variable	Unit	Observations	Minimum	Maximum	Mean	SD
LAS	$\mu\text{g.L}^{-1}$	48	0.0	920.0	104.2	224.1
DCO	$\text{mg.L}^{-1}$	48	29.0	890.0	157.7	230.6
PT	$\text{mg.L}^{-1}$	48	0.0	9.7	1.8	2.8
NT	$\text{mg.L}^{-1}$	48	4.0	81.0	27.3	24.7
pH	-	48	4.1	8.7	7.4	0.9
Conductivity	$\mu\text{S.cm}^{-1}$	48	7.5	971.0	458.9	293.1
Turbidity	NTU	48	0.8	304.7	49.8	93.5
MES	$\text{mg.L}^{-1}$	48	2.9	448.0	69.1	112.3
MVS	% MES	48	15.3	100.0	65.6	26.2

The Principal Component Analysis showed that of the 9 components, the first component (F1) accounted for about 59.6 % of the total variance, the second component (F2) accounted for about 16.7 % of the total variance and the third component (F3) accounted for about 9.9 % of the total variance of the dataset. The loadings for the three first components and square cosines are presented in Figure 8.

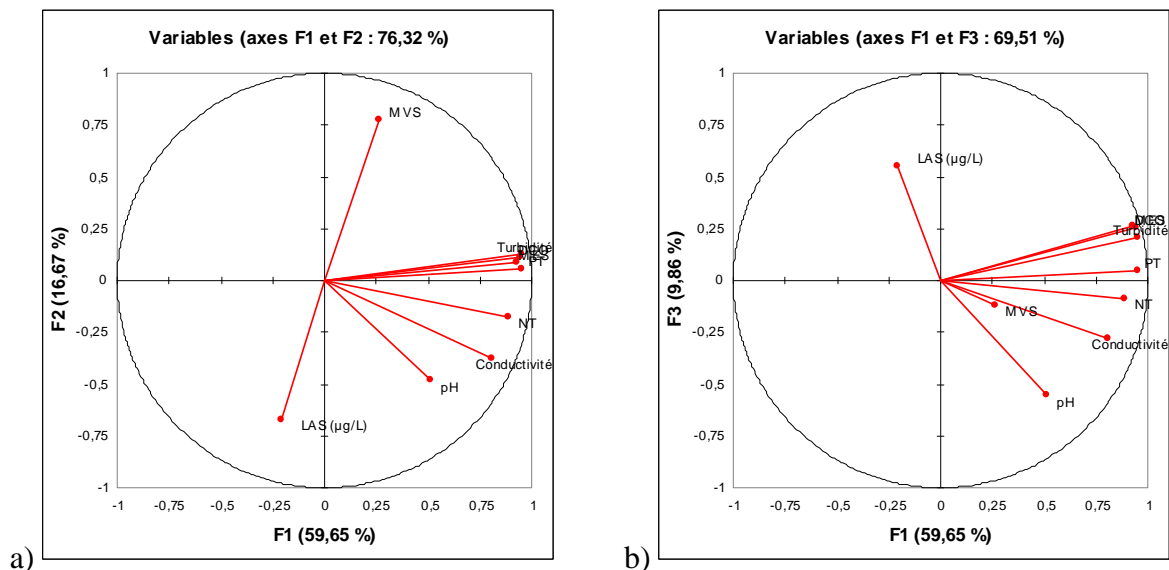


FIGURE 8: The square cosines for all variables in a) components F1 and F2 and b) components F1 and F3

A variable is increasingly well represented by a component as the corresponding value of the square cosine approaches the unit. Almost all variables are well represented by the first three components, F1, F2 or F3, that collectively explain 86.2 % of the total variance of the dataset. The variables that primarily contributed to the first eigenvector were principally turbidity, chemical oxygen demand, total phosphorous, total nitrogen, suspended solid and conductivity. The second eigenvector was mainly

related to volatile suspended solid. The third eigenvector was mainly related to LAS (Figure 8). It is clear that turbidity, chemical oxygen demand, total phosphorous, and suspended solid are correlated ( $R^2 > 0.90$ ). Orthogonal variables are significantly not correlated; it seems to be the case between all the global pollution parameters and the level of LAS in waters. Moreover, it is important to note that LAS and MVS are not negatively correlated ( $R^2 = -0.40$ ) even if they seem to be symmetrically opposed regarding the center of the circle.

## **Conclusion**

An analytical method for determination of LAS contents in water samples by SPE extraction and HPLC-UV/MS quantification was developed. The method provided satisfactory detection and quantification limits ( $LOD = 0.01 \mu\text{g.L}^{-1}$  and  $LOQ = 0.03 \mu\text{g.L}^{-1}$ ). This method was applied to water sampled in Toulouse (France). Wastewaters, treated wastewaters, ground water, rainwaters, roof collected waters, run-off waters and carwash wastewaters were sampled and analysed. Determination of LAS levels in these waters showed that run-off waters were more polluted than treated wastewaters or wastewaters. LAS concentrations observed in carwash discharges were not negligible. These wastewaters were discharged into the stormwater network and impacted stormwater quality. The results showed that laurylalkylbenzene sulfonates concentrations were variable over time and space in all urban water compartments. A multivariable analysis was performed and no correlation between LAS levels and global water pollution parameters was observed.

## **Acknowledgment**

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