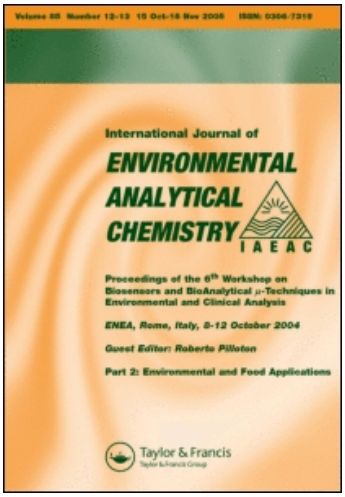


International Journal of Environmental Analytical Chemistry
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713640455

# Linear Alkylbenzene Sulfonates (LAS) in Sewage Sludges, Soils and Sediments: Analytical Determination and Environmental Safety Considerations

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**To cite this Article** De Henau, H. , Mathijs, E. and Hopping, W. D.(1986) 'Linear Alkylbenzene Sulfonates (LAS) in Sewage Sludges, Soils and Sediments: Analytical Determination and Environmental Safety Considerations', International Journal of Environmental Analytical Chemistry, 26: 3, 279 — 293
**To link to this Article: DOI:** 10.1080/03067318608077120
**URL:** http://dx.doi.org/10.1080/03067318608077120

*Intern. J. Environ. Anal. Chem.*, 1986, Vol. 26, pp. 279–293
0306-7319/86/2604-0279 $18.50/0

Printed in Great Britain



# Linear Alkylbenzene Sulfonates (LAS) in Sewage Sludges, Soils and Sediments: Analytical Determination and Environmental Safety Considerations†

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(*Received March 20, 1986*)

Linear Alkylbenzenesulphonates (LAS), a major anionic surfactant used in laundry products, can be measured specifically in the environment by instrumental analysis. In addition to a desulphonation-gas chromatography approach, a method based on high performance liquid chromatography has been developed. The main features of the methods are outlined, and LAS concentrations measured in sewage sludge, sediments and sludge amended soils are reported.

Knowledge of usage volumes, sewage treatment practices and environmental transport and transformation mechanisms has been used to predict concentrations of LAS. These calculated concentrations were found to agree well with those actually measured in the environment.

Both measured and calculated ambient concentrations of LAS are below those which could produce potentially adverse effects in representative surface water, benthic and terrestrial organisms.

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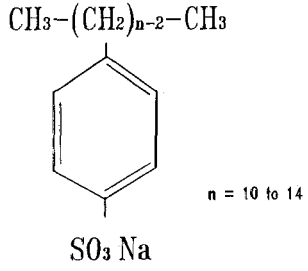
†Presented at the 16th Symposium on the Analytical Chemistry of Pollutants, Lausanne, Switzerland, March 17–19, 1986.

## INTRODUCTION

Linear alkylbenzene sulfonate (LAS) is an anionic surfactant used in household cleaning products. Its current volume of use is about 80,000 t/y in Germany[1] and 5,000 t/y in Switzerland.[2] Commercial LAS consists of alkyl chainlengths ranging from $C_{10}$ to $C_{14}$ as shown in Figure 1.




Linear alkylbenzene sulfonate (LAS)

FIGURE 1

A number of analytical techniques have been developed to measure LAS in environmental samples. In addition to the well known colorimetric procedure based on methylene blue (MBAS), more specific instrumental methods have been developed using gas chromatography (GC). These methods not only have a greater sensitivity but are also very selective. This has allowed the specific determination of trace levels of LAS in river water and sediment, sewage sludge and soil.

The purpose of this paper is to describe a newly developed procedure based on high performance liquid chromatography (HPLC) and to present results obtained from its application to environmental samples, particularly sludge, soil and sediment. In addition, results of environmental monitoring programs obtained with the gas chromatography-based methods are presented for comparative purposes, predicted concentrations in sludge and soil are reviewed and perspective is offered as to the significance of the LAS concentrations found.

## ANALYTICAL METHODS

Several analytical techniques exist to measure LAS in environmental samples. Undoubtedly, the most commonly employed one is the MBAS procedure based on the formation of a complex with the coloured methylene blue cation.[3-5] MBAS results are conservative since the method is not specific for LAS, but also responds to other anionic materials present in the sample. For example, Sullivan and Swisher[6] determined that only 10–20% of the MBAS concentration in river water was actually LAS, while Waters and Garrigan[7] found the fraction to be approximately 26%.

Several chromatographic procedures have been developed to measure LAS specifically in complex matrices. Swisher[8] and Sullivan and Swisher[6] used microdesulfonation followed by gas liquid chromatography of the resultant alkylbenzenes to determine the various homologs and isomers of commercial LAS. Waters and Garrigan[7] described a microdesulfonation/gas liquid chromatography procedure for the determination of LAS in river water that is sensitive to the μg/l level. Osburn[9] expanded the applicability of the desulfonation/gas chromatography procedure to include sludges and river sediments. McEvoy and Giger[10] adapted the methodology of Hon-Nami and Hanya[11] for the analysis of LAS in sewage sludge. Their approach involved the formation of alkylbenzenesulphonyl derivatives followed by gas chromatography/mass spectrometry (GC/MS).

Although the gas chromatographic methods are highly specific and sensitive, they are also very complex and time consuming. For this reason, we have determined the feasibility of measuring trace LAS concentrations by high performance liquid chromatography.

## THE HPLC METHOD

An outline of the preparation steps of the HPLC method is shown in Figure 2 and complete details are given by Matthijs and De Henau.[12]

Briefly, the sample is extracted under reflux with methanol. The methanol fraction is passed through an anion exchange resin to eliminate interfering nonionic materials, and the LAS is eluted from

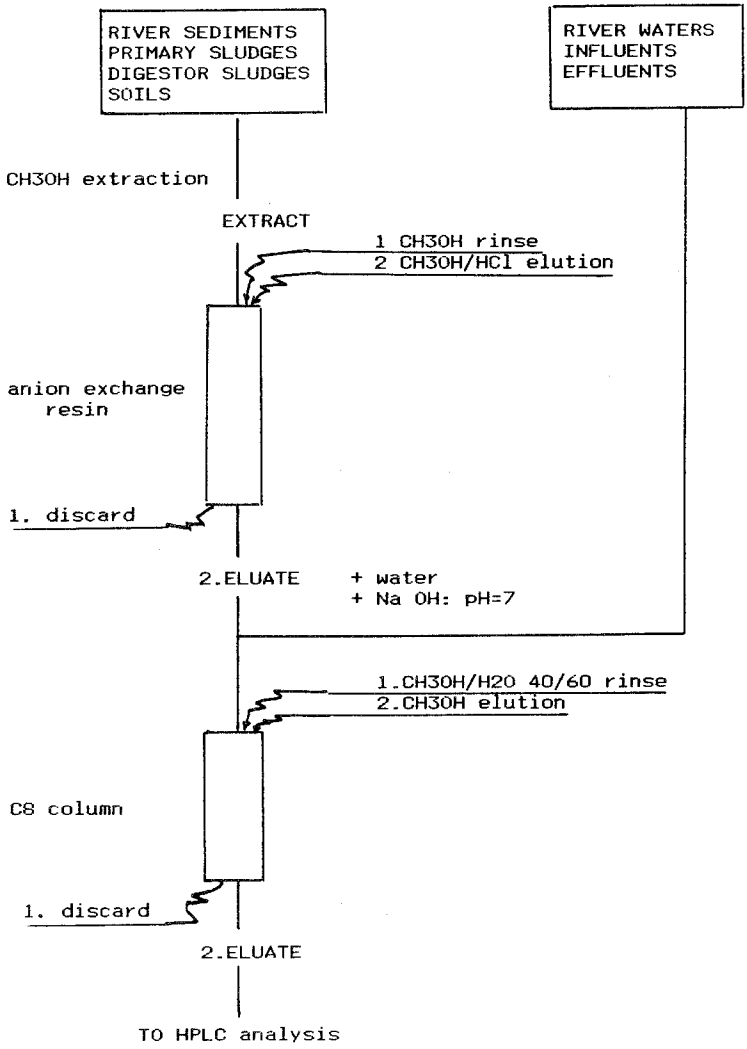



FIGURE 2 Preparative steps HPLC analysis of LAS.

the resin with acidic methanol. The eluate is diluted in water, adjusted to neutral pH and further purified through a small scale preparative octyl reversed phase silica column. The column is rinsed with a methanol/water solution followed by elution with pure methanol. The analysis of LAS in the eluate is by high performance liquid chromatography with UV detection of the benzene chromophore group. The various LAS homologs are separated by a water/acetonitrile/sodium perchlorate system, and the concentration of LAS quantified by the use of pure homolog standards.

Figure 3 presents a typical chromatogram obtained with this method. The detection limit of HPLC separation is 0.05 $\mu$g for each LAS homolog. The reproducibility of the entire method, expressed as



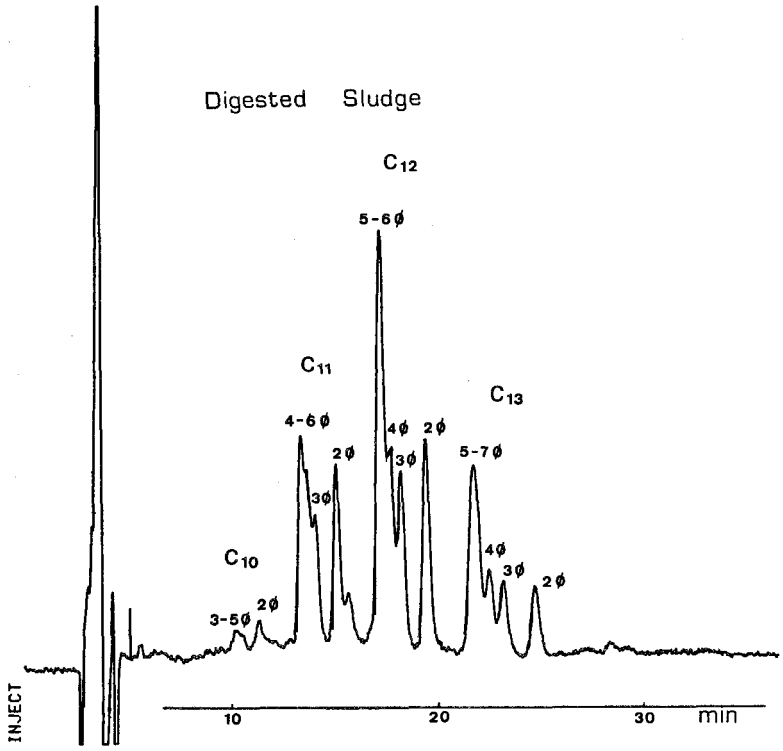



FIGURE 3

a relative standard deviation, is 6% for river water, sediment, raw and treated sewage and municipal sludges. Recovery of LAS spiked into aqueous, sediment and sludge samples is 94%, 87% and 84%, respectively. Required sample size for sludges is 1 g (dry solids) and 10 g for sediments (dry solids). The preparation steps require about 1 h per sample, which is considerably less than that required for the procedures based on gas chromatography.

## MONITORING RESULTS OBTAINED WITH SPECIFIC ANALYTICAL METHODS

Most studies to monitor LAS concentrations in sewage and surface water have used the nonspecific MBAS method. These have been reviewed by Sivak *et al.*[13] Table I presents the results from monitoring studies at sewage treatment plants in North America based on the desulfonation-gas chromatography methodology of Osburn.[9,14] The analyses were conducted on composite samples collected over extended time frames. Results from a program conducted by the U.S. Soap and Detergent Association at the Enid, Oklahoma activated sludge plant are included too.[15] Results of a pilot study in Germany, using the HPLC method described in this paper, are also shown in Table I. LAS concentrations in the raw (influent) sewage at the individual plants were in the range of 1–10 mg/l and removal was 90% or greater in all cases.

TABLE I

Summary of LAS monitoring for activated sludge treatment plants.

| Location | Average LAS concentration (mg/l) | | Average Removal (%) | Reference |
|---|---|---|---|---|
| | Influent | Effluent | | |
| Canada (3 plants) | 2.0 | 0.09 | 96 | 14 |
| United States (3 plants) | 3.8 | 0.06 | 98 | 14 |
| Enid, Oklahoma | 3.7 | 0.04 | 99 | 15 |
| Germany (8 plants) | 4.8 | 0.07 | 99 | — |

## Sewage treatment plant sludges

The program conducted by the Soap and Detergent Association employed Osburn's desulfonation-gas chromatography methodology in a detailed, mass balance study at the sewage treatment plant of Enid, Oklahoma.[15] This is a very efficient activated sludge plant serving about 70,000 persons. The mass balance information summarized in Table II shows that about 96% of the LAS entering the plant was biodegraded. The largest fraction of the LAS that was not biodegraded and thus left the plant, was associated with the sludge. The concentration of LAS (or any component from household products) in municipal sewage sludge can be estimated by the procedures described by Holman.[16] As shown in Table III, a



TABLE II

Fate of LAS at the Enid, Oklahoma activated sludge treatment plant.

| | kg LAS/day | % of Total |
|---|---|---|
| Amount entering plant | 105 | 100 |
| Amount leaving plant | | |
| Effluent | 1 | 1 |
| Sludge | 3 | 3 |

Reference 15

TABLE III

Prediction of LAS concentration in digested sludge.

$C_s = C_{is} * F_{is}/F_s * (R - D)$

$C_s$ = concentration in sludge
$C_{is}$ = concentration in the influent sewage
$F_{is}/F_s$ = ratio of sewage flow to sludge flow
$R$ = fraction removed by treatment process
$D$ = fraction biodegraded by the treatment process

$C_s = 3.3$ g LAS/kg dry solids for $C_{is} = 5$ mg/l
$(R-D) = 0.1$ (i.e 10% in sludge, 1% on effluent)
$F_{is}/F_s = 260$ (typical value)
sludge is 4% solids (typical value)

concentration of 3.3 g LAS/kg dry solids is predicted for digested sludge based on typical treatment parameters and assuming that: (a) the concentration of LAS in influent sewage is 5 mg/l, (b) 10% of the LAS is removed via sorption to sludge with no biodegradation during sludge digestion, and (c) 1% of the LAS is discharged in the effluent (biodegradation of LAS by the treatment process is thus about 90%).

Specific analytical methods have been used to measure concentrations of LAS in sewage sludge (Table IV) McEvoy and Giger[10] utilized GC/MS techniques in a survey of digested sludges in Switzerland, while desulfonation-gas chromatography was applied to digested sludges collected from various locations in the United States,[14] including Enid, Oklahoma.[15] Very recently, we have applied the HPLC method in a survey of digested sewage sludges collected from a number of treatment plants in Germany. The predicted LAS concentration in digested sludge (Table III) compares favorably with the values measured across a range of treatment plants. In addition, the concentrations determined by the different analytical methods are consistent and agree very well.



TABLE IV
Measured concentrations of LAS in digested sludge.

| Location | LAS concentration (g/kg dry solids) | | Reference |
|---|---|---|---|
| | Average | Range | |
| Switzerland (8 plants) | 5.6 | 2.9–11.9 | 10 |
| United States (3 plants) | 5.6 | 5.3–7.0 | 14 |
| Enid, Oklahoma | 5.5 | — | 15 |
| Germany (8 plants) | 6.2 | 1.6–11.8 | — |

Predicted value (Table III): 3.3 g/kg.

## Sludge-amended soil

LAS concentrations in soil resulting from the application of sewage sludge can be predicted from knowledge of LAS concentration in the

sludge and the application rate of sludge to soil, and assuming that no LAS biodegradation occurs.[16] Taking 6 g LAS/kg sludge solids as a representative value (Table IV), the concentration of LAS in sludge-amended soil is estimated to be 7 mg/kg on a mass basis (1.2 g/m$^2$ on an area basis) as shown in Table V.

Using the HPLC method, we measured LAS concentrations in a few soil samples collected in Germany and the United Kingdom (Table VI). These soils all have a history of several years of sludge application. The German soils are used for agricultural purposes, while the U.K. soil is used only for sludge disposal and therefore receives a higher application of sludge. The measured values are consistently lower than the value of 7 mg/kg estimated assuming no LAS biodegradation. Variations in parameters such as the actual application rate and the concentration of LAS in the specific sludge applied may also be a factor in the difference between measured and



TABLE V

Predicted concentration of LAS in sludge-amended soil.

| |
|---|
| *Predicted concentration* |
| Area basis: 1.2 g/m$^2$ |
| Mass basis: 0.007 g/kg or 7 mg/kg |
| *Assumptions* |
| LAS concentration in sludge: 6 g/kg |
| Application rate of sludge to soil: 0.2 kg/m$^2$ per year |
| Bulk density of soil: 1200 kg/m$^3$ |
| Depth of tillage: 15 cm |
| No biodegradation of LAS |

TABLE VI

Measured concentration of LAS in sludge-amended soil.

| Location | Concentration (mg/kg) |
|---|---|
| Germany | |
| (3 different locations) | 0.9, 1.1, 1.3 |
| United Kingdom | 2.2 |

Predicted value (Table V): 7 mg/kg.

predicted values. Nevertheless, these preliminary survey data indicate the applicability of the HPLC method to sludge-amended soils.

## LAS concentration in river sediment

LAS present in treated sewage discharged to receiving waters will partition between the water and sediment phases. The concentration of LAS in sediment is a function of a number of parameters, including biodegradation and sorption, distance from the sewage outfall, the dilution of the sewage upon discharge and the composition of the sediments. Complex mathematical models have, therefore, been used to predict the fate of LAS in river systems.[17,18] As reported by Games,[17] measured concentrations of LAS in both the overlying water and sediment rapidly decreased with increasing distance from the sewage outfall. Concentrations predicted by the model agreed well with measured values, indicating that elevated concentrations of LAS in sediments are expected to occur only within a relatively short distance below a sewage treatment plant outfall (Table VII).

The results of a pilot study to apply the HPLC method to sediment collected from German rivers are also presented in Table VII. The

TABLE VII

Measured concentration of LAS in river sediment.

| Location | LAS concentration | |
|---|---|---|
| | Overlying water (mg/l) | Sediment (mg/kg dry solids) |
| United States (in-depth study of one location—distance below sewage outfall)[a] | | |
| < 1 km | 0.27 | 275 |
| 12 km | 0.12 | 8.4 |
| 25 km | 0.08 | 2.7 |
| 48 km | 0.04 | 1.4 |
| Germany (survey of 14 locations below sewage discharges) | 0.01–0.09 | 1.5–174[b] |

[a]Reference 17.
[b]13 of the 14 values were below 25 mg/kg, 10 of the 14 values were below 10 mg/kg.

LAS concentrations in these sediments measured by HPLC are comparable to those determined in the more detailed, site-specific study which utilized desulfonation-gas chromatography.[17]

## BIODEGRADABILITY OF LAS IN VARIOUS ENVIRONMENTAL COMPARTMENTS

LAS biodegradation has been studied extensively by a number of researchers using laboratory-scale models of sewage treatment systems, model surface waters and natural waters.[13,19–25] When tested at realistic concentrations ($\mu$g/l level) in natural surface waters, LAS has been found to undergo rapid ultimate biodegradation. For example, a half-life of 1.4 days was observed in river water collected below the outfall of a sewage treatment plant.[20] Biodegradation was similarly rapid in the presence of sediments (half-life of 0.7 days).

The results of several recent studies of the kinetics of LAS ultimate biodegradation in surface waters, sediments, groundwater, subsurface soils and sludge-amended soil are summarized in Table VIII. Radiotracer techniques were used with various LAS chainlengths (i.e. $C_{10}$–$C_{14}$) uniformly labeled with carbon-14 in the aromatic ring or at specific positions in the alkyl chain. These data indicate that LAS undergoes rapid ultimate biodegradation in all environmental compartments studied. The half-lives were comparable to those observed for natural substrates such as glucose, fatty acids and cellulose.

TABLE VIII

Kinetics for the ultimate biodegradation of LAS in various environmental compartments.

| Test system | Half-life (days) | References |
|---|---|---|
| River water below sewage outfall | 1.0–1.4 | 14, 20 |
| River water below sewage outfall plus sediment | 0.7 | 20 |
| Sediments below sewage outfall | 0.6 | 14 |
| Groundwater plus subsurface soil | 1.1 | 23 |
| Soils | | |
| United States | 17–27 | 14 |
| United Kingdom | 8–10 | 24 |
| Japan | 12–22 | 25 |

LAS concentrations in soil would be reduced by more than 90% after a period of 3–4 half-lives, or about 75–100 days, following application of sludge. In the case of an annual application of sludge to the soil, this suggests that almost all of the LAS in the soil will have biodegraded by the time of the next application. The same applies to aerobic river sediments where ultimate biodegradation of LAS shows a half-life of about 1 day. Accordingly, measured levels of LAS in sludge, sediment and soil likely represent a steady-state condition where continual input of LAS from use and disposal of products by consumers is balanced by sorption and biodegradation mechanisms.

## LAS TOXICITY AND ENVIRONMENTAL SAFETY CONSIDERATIONS



The toxicity of LAS to aquatic life has been the subject of extensive study. An in-depth review of these investigations is beyond the scope of this paper and the reader is referred to literature sources for details. [13,21,22,26–32] Key results from these references are that the toxicity of LAS to aquatic life increases with increasing alkyl chainlength; the acute toxicity of commercial LAS is typically in the range of 1–10 mg/l; transient biodegradation intermediates are from 100 to 10,000 times less toxic than the parent molecule; and that ambient concentrations in river water are well below thresholds for chronic effects.

There are fewer reports concerning the toxicity of LAS to benthic organisms. Consequently, a program is underway at our U.S. laboratory to develop a standardized test procedure to evaluate chronic effects on sediment dwelling biota. Preliminary results from these studies[14] indicate that life-cycle exposure of an aquatic insect (*Chironomus riparius*) to sediment concentrations up to 500 mg/kg of LAS had no adverse effects on survival or emergence of adults (Table IX). Sorbed LAS does not appear to be bioavailable to the midge. Rather, it was LAS in the interstitial water that correlated with toxicity. The chronic $EC_{50}$ (about 2 mg/l) was comparable between tests conducted without sediment and tests where desorption from LAS-spiked sediment resulted in about 2 mg/l in interstitial water. Therefore, the presence of LAS in river waters and sediments

TABLE IX

Toxicity of LAS to sediment Biota and commercial plant seedlings.

| Organism | Results | References |
|---|---|---|
| Benthic midge *Chironomus riparius* | No effect at sediment concentrations up to 500 mg/kg | 14 |
| | (Measured concentration in sediments: 1–275 mg/kg) | Table VII |
| Commercial plant seedlings—barley, radish, pea, cucumber, tomato, lettuce | No effect at soil concentrations up to 0.4 g/m$^2$ (2.2 mg/kg) | 24 |
| | (Measured concentrations in soil: 0.9–2.2 mg/kg) | Table VI |



at concentrations reported here do not appear to represent a hazard to aquatic life.

Most toxicity studies with terrestrial plants have examined the effects of LAS solutions sprayed directly onto the plant or used for watering the soil.[13,21,22] Sivak *et al.*,[13] concluded that even when plants are grown in LAS solutions (i.e. conditions of maximum LAS bioavailability), adverse effects occur only at concentrations greater than 50 mg/l. Information developed recently by the Unilever Company[24] indicates that the majority of commercial plant seedlings tested showed visible signs of toxicity only at an LAS concentration of 1000 mg/l in water applied to the soil and to the foliage as a spray (1000 mg/l in the applied water was estimated to be equivalent to approximately 40 g/m$^2$ or 220 mg/kg, using data in Table V). Cucumber was the most sensitive species tested which showed effects at LAS concentrations of 100 mg/l (4 g/m$^2$ or 22 mg/kg). No reduction in growth or visible toxicity occurred in any species at LAS concentrations of 10 mg/l or less (0.4 g/m$^2$ or 2.2 mg/kg). These data were obtained under stress conditions and when compared to the measured concentrations of 0.9–2.2 mg/kg, suggest LAS in sludge applied to soil does not represent a hazard to terrestrial plants.

## CONCLUSIONS

This paper has provided a brief overview of analytical methods having specificity for LAS in environmental samples and described a new procedure based on HPLC. Additionally, results obtained from application of HPLC and other methods were summarized, as was biodegradation and toxicity data with emphasis on the sludge, soil and sediment compartments. It can be concluded from the information presented that:

- Analytical methods having specificity, sensitivity and reproducibility are now available for measurement of LAS in solid as well as aqueous matrices.
- A newly developed HPLC procedure having advantages of simplicity and rapidity has been developed that provides information comparable to that obtained with existing gas chromatographic-based methods.
- LAS is highly removed in sewage treatment with most of the removal due to biodegradation, and undergoes ultimate biodegradation in surface water, sediment, groundwater and soil at a rapid rate.
- Measured concentrations of LAS in sludge, soil and sediment compartments are predictable and represent a balance between input and removal by sorption/biodegradation mechanisms.
- Ambient concentrations of LAS in surface water, sediment and sludge-amended soil do not pose a hazard to aquatic or benthic organisms, or terrestrial plants.
- Further research with a variety of terrestrial organisms using sludge-amended soil containing known concentrations of LAS, and measurement of LAS concentrations in soil would further support the conclusion that LAS poses no hazard to organisms living in sludge-amended soil.



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