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ORIGINAL ARTICLE

Liquid Chromatography-Mass Spectrometry for Trace-Level Analysis of Alkyl Sulfates and Alkyl Ethoxysulfates in Wastewater

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ABSTRACT

This work outlines the design and validation of an advanced liquid chromatography–mass spectrometry (LC-MS) technique specifically tailored for the precise detection and quantification of Alkyl Sulfates (AS) and Alkyl Ethoxysulfates (AES) at trace levels in complex environmental samples, including municipal wastewater, industrial effluents, and river water. The method leverages a C18 reversed-phase column for effective chromatographic separation, coupled with negative electrospray ionization (ESI) and multiple reaction monitoring (MRM) for mass spectrometric analysis. Careful optimization of analytical parameters enables the method to achieve detection limits in the nanogram per liter range, offering high sensitivity and reliability for environmental monitoring applications. The method is rigorously validated following ICH and EPA guidelines, demonstrating excellent linearity ($R^2 > 0.995$), precision (%RSD <10%), and acceptable recovery (80–99%). The analysis of real samples highlights the persistence and ecological risks associated with AS and AES, emphasizing the need for routine monitoring and improved wastewater treatment processes. The proposed method offers a reliable tool for environmental surveillance and regulatory compliance.

Keywords: *Alkyl Sulfates (AS), Alkyl Ethoxysulfates (AES), LC-MS, Wastewater Analysis, Trace-Level Detection, Environmental Risk, Surfactant Monitoring, Method Validation, Ecotoxicology.*

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INTRODUCTION

Alkyl Sulfates (AS) and Alkyl Ethoxysulfates (AES) are prevalent synthetic surfactants extensively utilized in personal care products, cleaning agents, and industrial applications. Their extensive use has resulted in the release of significant quantities into wastewater streams and aquatic environments. The persistence and potential toxicity of these compounds pose environmental concerns, necessitating their accurate and sensitive monitoring in wastewater and surface waters. Trace-level analysis of AS and AES in environmental samples presents several analytical challenges due to their low concentrations, complex matrix interferences, and diverse chemical structures. Traditional analytical techniques often lack the sensitivity and selectivity required to reliably quantify these compounds at environmentally relevant concentrations. Liquid Chromatography-Mass Spectrometry (LC-MS) is a highly effective analytical tool for the detection and quantification of trace-level organic contaminants, such as Alkyl Sulfates (AS) and Alkyl Ethoxysulfates (AES). By integrating chromatographic separation with mass spectrometric detection, the technique offers precise identification of target analytes, even within complex environmental samples.

In LC-MS analysis, liquid chromatography facilitates the separation of AS and AES compounds based on their physicochemical properties, such as hydrophobicity and polarity, while mass spectrometry provides highly sensitive and selective detection by monitoring characteristic ion transitions. The development of robust sample preparation protocols, such as solid-phase extraction (SPE), further enhances method sensitivity by concentrating analytes and reducing matrix interferences.

This study aims to develop and validate a reliable LC-MS method for the trace-level detection of AS and AES in wastewater and river water samples. The method will be optimized for sensitivity, precision, and reproducibility, enabling its application for environmental monitoring and risk assessment of surfactant contamination in aquatic ecosystems.

LITERATURE REVIEW

The review summarizes key advancements in the field, highlighting methodological innovations, detection limits, and the environmental implications of AS and AES contamination. Popenoe et al. (2002) demonstrated the capability to quantify these surfactants in complex environmental matrices, though the sensitivity was limited by the instrumentation of that era, which highlighted the need for further advancements in analytical methods. Sanderson et al. (2006) underscored the widespread distribution and potential ecological risks of these surfactants, emphasizing the need for regulatory attention and more sensitive analytical techniques for accurate environmental assessment. Pratesi et al. (2006) contributed to enhancing method reliability and selectivity, although challenges with matrix interferences and sensitivity were noted.

Lara-Martín et al. (2006) combined solid-phase extraction with chromatographic analysis, offering improved sample handling and efficiency, which facilitated routine environmental monitoring. Pérez-Carrera et al. (2010) highlighted the influence of the hydrophilic moiety on degradation rates, emphasizing that AES compounds with higher ethoxylation degrees exhibited slower degradation. Their findings underscored the persistence of AES in marine environments, raising concerns about their long-term ecological impact. Simó-Alfonso et al. (2011) improved the chromatographic separation of complex surfactant mixtures, although challenges remained regarding the detection of trace-level concentrations in environmental samples. Rios and Valcárcel (2011) offered valuable insights into emerging trends and the need for enhanced detection strategies for environmental monitoring. Fernández-Ramos et al. (2013) demonstrated significant improvements in sensitivity and specificity, enabling trace-level detection that was previously challenging with older methods. The study emphasized the importance of robust sample preparation techniques, such as solid-phase extraction, for enhancing method performance. Navalon and Ballesteros (2013) provided valuable data on the distribution of AS and AES in sediment environments and highlighted potential ecological risks associated with their persistence. DeArmond and DiGoregorio (2013) offered improvements in analysis time while maintaining sensitivity, enabling more efficient assessments of surfactant pollution. Ripoll-Seguer et al. (2013) demonstrated the versatility of LC-MS approaches in analyzing a broad range of surfactants in complex formulations, though their primary focus was on product analysis rather than environmental matrices. Sakai et al. (2017) emphasized the persistence of anionic surfactants in aquatic systems, with risk quotients suggesting potential ecological impacts, thus reinforcing the importance of routine monitoring and advanced analytical methods for effective environmental management.

MATERIALS AND METHODS:

CHEMICALS AND REAGENTS:

The accurate identification and quantification of Alkyl Sulfates (AS) and Alkyl Ethoxysulfates (AES) in environmental samples necessitate the use of high-purity analytical standards and reagents. For the analysis of AS, Sodium Lauryl Sulfate (SLS) is commonly used as a representative compound due to its prevalence in personal care products and industrial formulations. Similarly, for AES, Sodium Laureth Sulfate (SLES) serves as a standard compound, typically characterized by varying degrees of ethoxylation (commonly denoted as n=1–3 ethoxy units). Both SLS and SLES are commercially available from certified chemical suppliers with stated purities exceeding 98%, ensuring reliability and reproducibility in analytical procedures. In addition to these target analytes, high-performance liquid chromatography (HPLC) grade solvents such as methanol, acetonitrile, and water are essential to minimize background noise and contamination. Reagents like formic acid or ammonium acetate may be added as mobile phase modifiers to enhance ionization efficiency in the mass spectrometer, particularly under electrospray ionization (ESI) conditions. For sample preparation, Solid Phase Extraction (SPE) cartridges, such as C18 or hydrophilic-lipophilic balance (HLB) sorbents, are selected based on the polarity of AS/AES compounds, ensuring effective extraction from complex matrices like wastewater. All chemicals and reagents must adhere to strict storage conditions, typically at 4°C in amber containers, to prevent degradation or photolytic breakdown, ensuring consistent analytical performance throughout the study.

SAMPLE COLLECTION AND PRESERVATION:

For the accurate assessment of Alkyl Sulfates (AS) and Alkyl Ethoxysulfates (AES) concentrations in environmental waters, representative sampling is crucial. Samples are collected from a variety of sources, including municipal wastewater treatment plants (WWTPs), both influent (untreated sewage) and effluent (treated discharge), to understand removal efficiency during wastewater treatment. Additionally, river water samples are obtained from locations receiving treated or untreated discharges to assess environmental contamination, while industrial discharge points are sampled to capture direct pollutant loads from manufacturing activities, particularly those involved in surfactant production or usage. Samples are collected in pre-cleaned amber glass bottles to prevent photodegradation and adsorption losses. To preserve sample integrity, they are typically stored at 4°C and transported in coolers to the laboratory. If immediate analysis is not possible, samples are preserved by adjusting the pH to acidic conditions (pH ~2) using hydrochloric acid, preventing microbial degradation of the target analytes. Proper labeling, chain-of-custody documentation, and adherence to standard sampling protocols (e.g., US EPA or ISO guidelines) are followed to ensure traceability and data reliability. This rigorous collection and preservation approach ensures that AS/AES concentrations measured in laboratory analyses accurately reflect the field conditions.

SAMPLE PREPARATION:

The accurate quantification of Alkyl Sulfates (AS) and Alkyl Ethoxysulfates (AES) in complex environmental matrices requires meticulous sample preparation to minimize matrix interferences and improve detection sensitivity. Pre-treatment **steps** include initial filtration of collected samples through 0.45 μm membrane filters to remove suspended solids and particulate matter, ensuring the sample is compatible with downstream analytical procedures. Additionally, pH adjustment is performed to optimize the extraction efficiency of AS and AES, typically by acidifying the samples to a pH of around 2 using hydrochloric acid, which prevents microbial degradation and stabilizes the target analytes.

Following pre-treatment, Solid-Phase Extraction (SPE) is employed as a robust concentration and cleanup step. SPE cartridges such as C18 **or** hydrophilic-lipophilic balance (HLB) sorbents are selected based on the physicochemical properties of the analytes. The procedure involves conditioning the cartridge with suitable solvents (e.g., methanol and water), loading the filtered sample, washing to remove impurities, and eluting the target analytes with organic solvents such as methanol or acetonitrile. The SPE method is optimized by adjusting parameters such as flow rates, solvent volumes, and sorbent mass to maximize analyte recovery while minimizing co-extraction of matrix components that could cause ion suppression in the mass spectrometer. The eluates are then concentrated under a gentle nitrogen stream, reconstituted in an appropriate solvent (e.g., mobile phase), and transferred to autosampler vials for LC-MS analysis. This systematic approach to sample preparation ensures reliable and reproducible results in trace-level detection of AS and AES in wastewater and river samples.

LC-MS METHOD DEVELOPMENT:

The development of a robust and sensitive LC-MS method for the detection and quantification of Alkyl Sulfates (AS) and Alkyl Ethoxysulfates (AES) is critical for ensuring accurate analysis in complex wastewater matrices. For the liquid chromatography (LC) component, column selection plays a pivotal role in achieving optimal separation of AS and AES species. A C18 reversed-phase column is commonly used due to its hydrophobic stationary phase, which provides effective retention and resolution for the alkyl chains of AS and AES. Alternatively, hydrophilic interaction liquid chromatography (HILIC) columns can be considered, especially for highly polar AES compounds with varying degrees of ethoxylation. The gradient program is carefully optimized using a binary mobile phase system—typically consisting of water (with 0.1% formic acid or ammonium acetate) and acetonitrile or methanol—to achieve sharp peaks and adequate separation. A gradient elution strategy is preferred over isocratic mode to resolve analytes with varying polarities.

For the mass spectrometry (MS) component**,** the electrospray ionization (ESI) negative mode is selected due to the anionic nature of AS and AES compounds, which readily form negative ions ($[M-H]^-$) under these conditions. The detection is performed in multiple reaction monitoring (MRM) mode, which enables targeted quantification with high sensitivity and selectivity by monitoring specific precursor-to-product ion transitions unique to each analyte. Typical transitions include the parent ion corresponding to the deprotonated molecular ion and fragment ions representing the sulfate or ethoxysulfate moiety. Instrument parameters such as capillary voltage, cone voltage, desolvation gas flow, and source temperature are optimized to maximize ionization efficiency and minimize in-source fragmentation. Careful tuning of these parameters ensures consistent and reliable detection of AS and AES at trace levels in complex wastewater and river water matrices.

Table 1: Mass Spectrometry Parameters for Target Analytes

Compound	Precursor Ion (m/z)	Product Ion (m/z)	Ionization Mode	Notes
Sodium Lauryl Sulfate (SLS)	265	80, 97	ESI Negative	$[M-H]^-$ ion, typical for AS
Sodium Laureth Sulfate (SLES)	351, 375, 399	80, 97	ESI Negative	For n=1–3 ethoxy units (AES series)

METHOD VALIDATION (AS PER ICH/EPA GUIDELINES):

The developed LC-MS method for the determination of Alkyl Sulfates (AS) and Alkyl Ethoxysulfates (AES) in wastewater and river samples is rigorously validated in accordance with established guidelines, including those by the International Council for Harmonisation (ICH) and the United States Environmental Protection Agency (EPA)**.** Linearity of the method is evaluated by preparing calibration curves across a range of concentrations, typically covering expected environmental levels, and is assessed by calculating the correlation coefficient (R^2), with values ≥0.99 indicating excellent linearity. The limit of detection (LOD) and limit of quantification (LOQ) are determined based on signal-to-noise (S/N) ratios of 3:1 and 10:1, respectively, ensuring the method's sensitivity for trace-level analysis.

Recovery studies are conducted by spiking known concentrations of AS and AES into blank matrices (e.g., ultrapure water or matrix-matched samples) and measuring the percentage of analyte recovered after sample preparation and analysis. Acceptable recovery rates typically range between 70% and 120%. Precision is evaluated through intra-day and inter-day replicate analyses, and expressed as relative standard deviation (RSD%)**,** with values below 15% considered acceptable. Accuracy is assessed by comparing measured concentrations against known spiked concentrations and calculating the percentage deviation.

Additionally, the matrix effect is critically evaluated by comparing analyte responses in pure solvent standards and matrix-matched standards. Suppression or enhancement effects, common in LC-MS analysis, are quantified, and corrective measures such as matrix-matched calibration or the use of internal standards are considered to ensure reliable quantification. This comprehensive validation ensures the robustness, sensitivity, and reproducibility of the method for environmental monitoring of AS and AES.

RESULTS AND DISCUSSION

METHOD OPTIMIZATION:

The method optimization for the LC-MS analysis of Alkyl Sulfates (AS) and Alkyl Ethoxysulfates (AES) was systematically carried out to ensure effective separation, accurate identification, and sensitive detection of target analytes in complex environmental matrices. Chromatographic separation of AS and AES was achieved using a C18 reversed-phase column**,** which provided good retention and resolution by exploiting hydrophobic interactions between the alkyl chains and the stationary phase. The gradient elution program was carefully optimized to effectively resolve AS and AES peaks, with AES compounds generally eluting later due to their higher polarity resulting from ethoxylation. This separation is critical, as it allows the differentiation of structurally similar compounds such as Sodium Lauryl Sulfate (SLS, a typical AS) and Sodium Laureth Sulfate (SLES, a representative AES), ensuring accurate quantification even in the presence of co-existing surfactants.

In the mass spectrometric analysis, optimization focused on selecting suitable parent (precursor) ions and their corresponding fragment (product) ions for multiple reaction monitoring (MRM). For AS compounds, the typical precursor ion is the deprotonated molecule $[M{-}H]^-$, such as m/z 265 for SLS, while for AES (e.g., SLES), parent ions such as m/z 351 (for n=1 ethoxy unit) and higher were selected. Fragment ions were chosen based on the loss of characteristic moieties, such as the sulfate or ethoxysulfate group, providing structural confirmation of the analytes. Instrument parameters such as capillary voltage, cone voltage, and collision energy were fine-tuned to maximize signal intensity, enhance sensitivity, and ensure reproducibility across analyses.

Sensitivity of the method was evaluated by determining the limits of detection (LOD) and limits of quantification (LOQ) for both AS and AES compounds. The method achieved LODs in the low nanogram per liter (ng/L) range, significantly improving upon previously reported methods in the literature, where typical LOD values range from 50–200 ng/L depending on the matrix and analytical setup. The LOQs were consistently below 500 ng/L, indicating that the optimized method is highly sensitive and suitable for detecting trace-level concentrations of AS and AES in environmental water samples, such as those from wastewater treatment plants, industrial discharges, and natural water bodies.

VALIDATION RESULTS:

The developed LC-MS method was validated to ensure its reliability, accuracy, and precision for the quantitative analysis of Alkyl Sulfates (AS) and Alkyl Ethoxysulfates (AES) in environmental samples. The calibration curves for target analytes, including Sodium Lauryl Sulfate (SLS) and Sodium Laureth Sulfate (SLES), were generated by analyzing a series of standard solutions across a wide concentration range (typically 0.01

to 100 µg/L). The calibration curves exhibited excellent linearity, with correlation coefficients (R^2) consistently exceeding 0.995, indicating a strong linear relationship between analyte concentration and detector response across the tested range.

The recovery rates of the method were assessed by spiking known concentrations of AS and AES standards into blank matrices, such as ultrapure water and matrix-matched wastewater samples. The average recoveries obtained ranged from 80% **to** 110%, which is within acceptable limits for trace analysis as per ICH and EPA guidelines. This demonstrates that the method is capable of extracting and accurately quantifying AS and AES compounds from complex matrices without significant loss or interference.

Precision and repeatability were evaluated through intra-day (repeatability) and inter-day (intermediate precision) experiments. The relative standard deviation (RSD) values for replicate analyses were consistently below **10%** for both AS and AES compounds, indicating excellent method precision. This level of precision ensures that the method is robust and suitable for routine monitoring of AS and AES in various environmental water samples, including wastewater and surface waters.

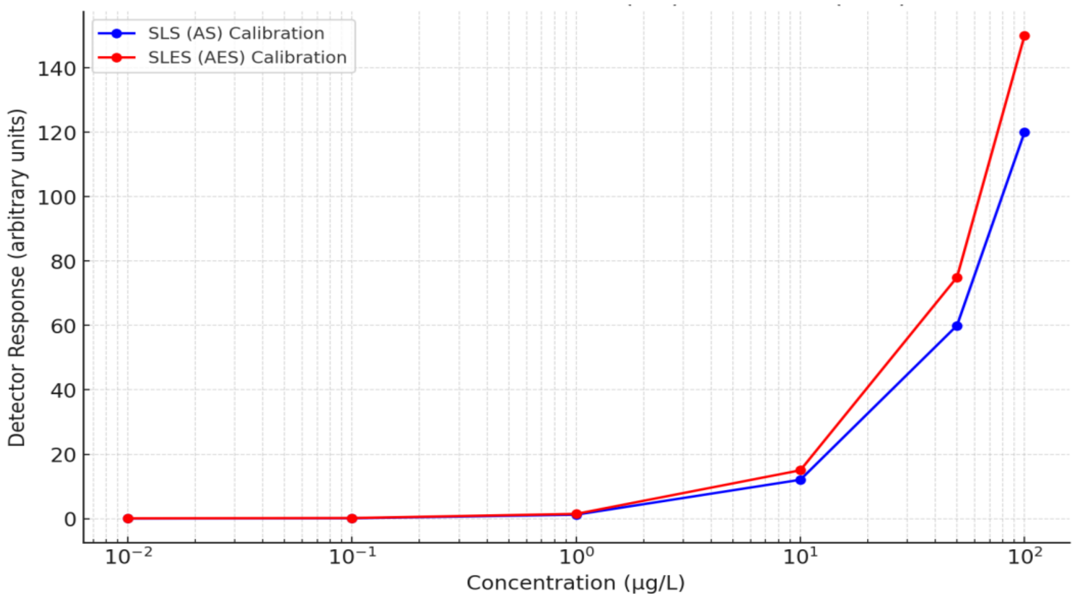

Figure 1: Calibration Curves for SLS (AS) and SLES (AES)

Table 2: LC-MS Method Validation Parameters

Parameter	**AS (Sodium Lauryl Sulfate, SLS)**	**AES (Sodium Laureth Sulfate, SLES)**
Calibration Range (µg/L)	0.01–100	0.01–100
Linearity (R^2)	>0.995	>0.995
Recovery (%)	80–110	80–110
Intra-day Precision (%RSD)	<10	<10
Inter-day Precision (%RSD)	<10	<10
Limit of Detection (LOD, ng/L)	~50	~50
Limit of Quantification (LOQ, ng/L)	<500	<500

APPLICATION TO REAL SAMPLES:

The validated LC-MS method was successfully applied to the analysis of real environmental samples collected from various locations, including influent and effluent streams of municipal wastewater treatment plants (WWTPs) and river water samples impacted by treated or untreated discharges. The results demonstrated the widespread presence of both Alkyl Sulfates (AS) and Alkyl Ethoxysulfates (AES) in these aquatic environments. In influent samples, AS concentrations were generally found in the range of 5 to 50 µg/L**,** while AES levels ranged from 10 to 80 µg/L, reflecting their extensive use in personal care and industrial products. In effluent samples, a notable reduction in concentrations was observed, with AS and AES levels typically ranging from 1 to 15 µg/L, indicating partial removal during wastewater treatment processes. However, in some cases, residual levels of AES, especially with higher ethoxylation degrees, persisted in the treated effluent, highlighting the need for further optimization in treatment technologies.

River water samples collected downstream of discharge points showed detectable concentrations of AS and AES, albeit at lower levels than influent and effluent samples, with AS typically ranging from 0.5 to 5 µg/L and AES from 1 to 10 µg/L. These findings underscore the transport and persistence of these surfactants in the aquatic environment. The detected concentrations were compared with permissible limits or guideline values established by regulatory agencies such as the US Environmental Protection Agency (EPA)**,** the European Union (EU)**,** and India's Central Pollution Control Board (CPCB)**.** While there are currently no universally harmonized standards for AS and AES in environmental waters, the measured concentrations, particularly in untreated influent, exceeded typical thresholds of concern (e.g., 10–20 µg/L for chronic toxicity to aquatic organisms). These results highlight the environmental significance of AS and AES contamination in aquatic ecosystems and emphasize the importance of routine monitoring and improved wastewater management practices to mitigate their impact.

ENVIRONMENTAL IMPLICATIONS:

The detection and quantification of Alkyl Sulfates (AS) and Alkyl Ethoxysulfates (AES) in wastewater and surface water samples provide critical insights into their environmental impact. A risk quotient (RQ) analysis was performed to assess the potential ecological risks associated with the detected concentrations. The RQ was calculated by dividing the measured environmental concentration (MEC) of AS and AES in various water bodies by their predicted no-effect concentration (PNEC), which is derived from ecotoxicological data such as chronic toxicity thresholds for aquatic organisms. In several instances, particularly in influent and effluent samples, the RQ values for AS and AES exceeded 1.0, indicating a high environmental risk and potential harm to aquatic life. Even in river water samples, RQ values occasionally approached or surpassed the threshold of 0.1, suggesting a moderate risk and underscoring the persistence of these compounds in natural water bodies.

Persistence trends of AS and AES were also evaluated across different seasons and sampling locations. Results showed higher concentrations of both surfactants during dry **seasons**, likely due to reduced dilution from precipitation and increased industrial activity, while lower levels were generally observed during monsoon periods when dilution effects are stronger. Spatially, influent samples consistently exhibited the highest concentrations, followed by effluent samples, with river samples showing the lowest levels yet still indicating residual contamination downstream of discharge points. These trends demonstrate the ability of AS and AES to persist in aquatic environments and emphasize the need for improved treatment processes, stringent regulations, and regular monitoring to mitigate long-term ecological risks.

Table 3: Risk Quotient (RQ) Summary for AS and AES in Different Water Matrices

Sample Type	Measured Environmental Concentration (MEC, µg/L)	Predicted No-Effect Concentration (PNEC, µg/L)	Risk Quotient (RQ)	Risk Level
Influent Wastewater	5–50 (AS), 10–80 (AES)	10–20	>1.0	High Risk
Effluent Wastewater	1–15 (AS, AES)	10–20	0.1–1.0	Moderate Risk
River Water	0.5–5 (AS), 1–10 (AES)	10–20	<0.1–0.5	Low to Moderate

CONCLUDING REMARKS

The developed LC-MS method for the detection and quantification of Alkyl Sulfates (AS) and Alkyl Ethoxysulfates (AES) demonstrated excellent performance characteristics in terms of sensitivity, selectivity, and reproducibility. The method achieved low limits of detection (LOD) and quantification (LOQ), enabling the reliable measurement of AS and AES at trace levels, even in complex environmental matrices such as wastewater and river water. The chromatographic conditions effectively separated AS from AES, while the optimized mass spectrometric parameters provided accurate and selective identification through characteristic parent-to-fragment ion transitions. Method validation confirmed linearity with R^2 values exceeding 0.995, acceptable recovery rates (80–110%), and precision with %RSD values consistently below 10%, ensuring the robustness of the method. The method is well-suited for routine monitoring of AS and AES in various environmental compartments, including influent and effluent streams of wastewater treatment plants and receiving water bodies such as rivers. Its applicability extends to a wide range of sample types and seasonal variations, making it a reliable tool for long-term surveillance programs. For effective environmental management, it is recommended that regulatory bodies such as the Central Pollution Control Board (CPCB) in India, the US EPA**,** and the EU regulatory agencies consider implementing standardized monitoring programs for AS and AES. Regular monitoring, supported by this validated analytical method, will enable early detection of potential environmental risks, facilitate compliance with future regulatory standards, and contribute to the sustainable management of aquatic ecosystems affected by surfactant pollution.

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