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Investigation of matrix interference in extractive-photometric analysis of surfactants in water samples

Orziqulova Shohsanam Rustam qizi
teacher
Department of Chemistry, Jizzakh State Pedagogical University
Uzbekistan, Jizzakh

Sultonov Marat Mirzayevich
doctor of Chemical Sciences, Professor
Department of Chemistry, Jizzakh State Pedagogical University
Uzbekistan, Jizzakh

Исследование матричной интерференции в экстракционно-фотометрическом анализе поверхностно-активных веществ в водных образцах

Орзиқулова Шохсанам Рустам қизи
преподаватель
кафедра химии, Джизакский государственный педагогический университет
Республика Узбекистан, Джизак

Султонов Марат Мирзаевич
д-р хим. наук, проф.
кафедра химии, Джизакский государственный педагогический университет
Республика Узбекистан, Джизак

Abstract

Extractive-photometric determination remains a widely used approach for surfactant analysis in natural and wastewater samples, but matrix constituents can interfere with the ion-association and colour-forming reactions on which it relies. This study systematically compared published interference data to develop a unified framework for understanding matrix effects in extractive-photometric surfactant determination. Twenty-one primary interference studies, together with one supporting review, covering anionic, cationic, and nonionic surfactant methods were analyzed by

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interferent type, reagent chemistry, sample matrix, and reported mitigation strategy. Three principal sources of interference were identified: competing inorganic ions and residual oxidants, coexisting surfactants of different ionic classes, and organic matrix constituents such as humic substances and proteins, addressed by five mitigation strategies — chemical masking, selective pre-extraction, physical separation, procedural control, and selection of interference-resistant reagents. Reagent chemistry proved a fundamental determinant of matrix tolerance: reagents based on inner-sphere coordination with the surfactant resist inorganic salt interference substantially better than conventional outer-sphere ion-pairing dyes. Interferents tolerated individually could also produce substantial bias when present simultaneously in saline matrices, showing the limits of single-interferent validation. The resulting framework offers a mechanistically informed basis for selecting reagents and sample-treatment procedures for surfactant determination in real environmental waters, including waters with variable salinity and organic load.

Аннотация

Экстракционно-фотометрическое определение остаётся основным методом контроля поверхностно-активных веществ (ПАВ) в природных и сточных водах, однако реальные матрицы содержат компоненты, мешающие цветной ионно-ассоциативной реакции. В работе систематически сопоставлены опубликованные данные по мешающему влиянию и предложена единая классификация матричных помех при экстракционно-фотометрическом определении ПАВ, построенная на анализе двадцати одной первичной работы и одной обобщающей публикации, охватывающих анионные, катионные и неионогенные ПАВ. Выделены три источника помех — конкурирующие неорганические ионы и остаточные окислители, ПАВ другого ионного класса и органические компоненты матрицы (гуминовые вещества, белки), — устраняемые пятью стратегиями: химическим маскированием, селективной предварительной экстракцией, физическим разделением, процедурным контролем и выбором устойчивого к помехам реагента. Химия реагента оказалась ключевым фактором солевой устойчивости: реагенты, образующие внутрисферные комплексы с анионом ПАВ, устойчивее к неорганическим солям, чем внешнесферные ионно-ассоциативные красители. Интерференты, безопасные по отдельности, могут давать значительную неаддитивную погрешность при совместном присутствии в реальной солёной матрице, что ограничивает достаточность проверки на одном интерференте. Предложенная система даёт практическую, основанную на химии основу для выбора реагентов и этапов предварительной обработки при анализе ПАВ в реальных экологических водах с переменным солевым составом и органической нагрузкой.

Keywords: surfactants, matrix interference classification, extractive-photometric determination, ion-pair extraction, water analysis.

Ключевые слова: поверхностно-активные вещества, матричные помехи, экстракционно-фотометрическое определение, экстракция ионных пар, анализ вод.

Introduction

Surfactants reach surface and ground waters mainly through wastewater discharge. Their amphiphilic structure makes them useful as detergents and hard to isolate cleanly for analysis [15]. Extractive-photometric determination, based on a coloured ion pair with an oppositely charged dye, remains a widely used routine approach because it needs no expensive equipment [6, 15]. Anionic surfactants are usually determined with methylene blue or related azo dyes [6, 10]; nonionic surfactants through coordination of the polyoxyethylene chain with an alkali-metal cation, extracted as picrate, tetrabromophenolphthalein, or thiocyanatocobalt(II) complexes [5, 9, 20, 21]; cationic surfactants with anionic dyes such as methyl orange [1, 4].

Problems appear once these methods leave the standard-solution stage. Common ions (Cl^-, NO_3^-, Ca^{2+}, Mg^{2+}, Fe^{3+}) bias the signal in either direction, depending on the dye [6]. Less obvious species matter too: residual chlorine interferes with cationic-dye methods unless reduced beforehand [13], and tolerance limits for sulfide, thiocyanate, iodide, and humic acids differ tenfold between reagents [10, 16, 18]. Surfactants of different ionic classes interfere with each other as well: anionic surfactants suppress the nonionic signal by competing for the coordinating cation [5, 20], while cationic surfactants inflate it by forming their own ion pair [5]. Storage and filtration add bias through adsorption losses [20], and since the response depends on alkyl chain length, the calibration standard chosen affects accuracy in ways tied to the unknown sample composition [16].

Reported countermeasures include masking interfering ions, pre-extracting the interfering surfactant, reducing residual oxidants, solid-phase extraction, and redesigning the procedure itself [1, 3, 4, 10, 11, 13, 16, 17, 18, 21]. However, these approaches are usually reported within individual analytical systems, making cross-method comparison difficult. Therefore, the objective of this study was to critically compare and classify matrix interference in extractive-photometric surfactant determination across anionic, cationic, and nonionic analytical systems. Particular attention was given to the chemical origin of interference, the dependence of matrix tolerance on reagent chemistry and sample composition, and the effectiveness of reported mitigation strategies. By comparing primary studies under a common analytical framework, this work aims to distinguish general interference mechanisms from reagent-specific effects and to establish practical criteria for selecting chromogenic reagents and sample-treatment procedures for analysis of surfactants in real water matrices.

Materials and Methods

A literature survey was conducted and approximately 60–70 publications related to extractive-photometric determination of surfactants were screened. From these, twenty-one primary studies meeting the inclusion criteria were selected for detailed comparative analysis; one additional review article [15] was consulted for general background and is not part of the criteria-based comparison. The selected papers span nearly five decades of the field: foundational reagent-development studies published between 1978 and 2013 are complemented by recent studies (2022–2026) that re-examine cross-class and matrix interference using solid-phase extraction and green-solvent alternatives. All were published in peer-reviewed analytical chemistry journals, including Analyst, Analytica Chimica Acta, Talanta, Analytical Sciences, Analytical Methods, Chemosphere, Microchimica Acta, ACS ES&T Water, and Sensors & Diagnostics. Three inclusion criteria were applied: the method

had to be an ion-pair extraction applicable to anionic, cationic, or nonionic surfactants; the article had to report quantitative tolerance limits, recoveries, or an explicit description of at least one matrix effect; and enough experimental detail (reagent, extraction solvent, wavelength, and sample matrix) had to be given to compare studies against each other. Papers describing extraction procedures without interference data, or reporting only calibration and sensitivity, were excluded even where they concerned the same reagent systems, since they added no comparative information on matrix effects.

For each source, the reported interferents were sorted into three categories: common inorganic ions and residual oxidants, coexisting surfactants of a different ionic class, and organic matrix constituents such as humic substances and proteins. Where an interferent could plausibly belong to more than one category (for example, a species acting both as a competing ion and, at higher concentration, as an oxidant), it was assigned to the category under which the original authors reported the effect. Mitigation strategies described in each source were grouped by underlying mechanism rather than by the specific reagent in which they first appeared, giving five categories: chemical masking, selective pre-extraction, physical separation (washing or ion-exchange), procedural control (reduced extraction volume or contact time), and selection of an interference-resistant reagent. Original concentration units were kept as reported instead of being converted to a common scale, since commercial surfactant mixtures often have an uncertain average molar mass, and forcing a conversion would have introduced error not present in the source data. Where two studies reported apparently conflicting tolerance for the same ion under similar conditions, both values are reported and the likely source of the discrepancy (reagent, solvent, or extraction ratio) is noted in the discussion.

Results and Discussion

Anionic surfactants: the methylene blue problem and its alternatives

The methylene blue (MB) method is a widely used anionic-surfactant procedure but is intrinsically susceptible to interference from other extractable anions, since ion pairing does not by itself distinguish the target surfactant from them. Fytianos et al. [6] found Cl^-, Ca^{2+}, and Fe^{3+} tolerable for the MB dye at natural-water concentrations, while Mg^{2+} biased results above 500 ppm and NO_3^- above 20 ppm; Ródenas-Torralba et al. [18] confirmed similar limits and additionally reported exceptional tolerance to Triton X-100 (>10 g/L).

Reagent chemistry, not just concentration, determines chloride tolerance. Ho and Lin [8] and Kasahara et al. [11] each found that a weaker, more selective ion-pairing reagent reduces chloride bias, with Kasahara's reagent remaining interference-free above seawater salinity where the classical ethyl violet dye already precipitates. Crisp et al. [3] explained why: outer-sphere ion pairs, such as methylene blue's, bind indiscriminately and extract small hydrated anions like chloride along with the surfactant, whereas their inner-sphere copper reagent coordinates the surfactant oxygen directly — a bond chloride cannot form — leaving the method essentially free of seawater-matrix interference. The lesson: chloride sensitivity is not intrinsic to ion-pair extraction but tracks how indiscriminately the reagent binds, and can be designed out rather than only corrected for afterward.

Organic co-solutes are a separate problem. Pedraza et al. [16] tabulated over twenty interferents for MB versus the surfactant-dye binding degree method and found humic acid raises the MB blank while proteins such as albumin depress the signal, and proposed C18 solid-phase pretreatment. Moskvin et al. [14] explained why: humic acids act two ways at once, forming their own extractable complex with MB (raising the blank) while competing with the surfactant for the dye (lowering the signal). Their fix — an intermediate acidic column wash at pH 10 — eliminated humic-acid interference up to 20 mg/L, well above typical river concentrations.

Procedural and solvent changes offer a third route, moving from simplification toward full solvent replacement. Jurado et al. [10] cut MB extraction to a single step; Pereira and Rocha [17] reduced solvent volume via microextraction with no significant interference; and two recent studies remove chloroform altogether — dos Santos et al. [19] using a hydrophobic natural deep eutectic solvent with smartphone readout (LOD 2.0 μg/L), and Yoon et al. [22] a 3:1 methyl isobutyl ketone–1,2-dichloroethane mixture that cuts phase separation to under 3 minutes and tolerates a broad range of pH, ionic strength, and anions.

Cationic and nonionic surfactants

Cationic-surfactant methods vary just as much by reagent, and cross-class effects can run either direction. Biswas and Pal [1] found thirteen coexisting ions and other surfactant classes biasing chrome azurol S determination, restoring real-water recovery from below 23 % to 75–90 % only after EDTA masking — a reagent-specific pattern seen with Evtushenko's methyl orange method [4] as well. Korzhan et al. [12] showed the opposite can also occur: a coexisting nonionic surfactant enhanced rather than degraded the Coomassie Brilliant Blue G signal for cetylpyridinium chloride. The scale of cross-class bias was recently quantified directly in seawater: Bramblett and Frossard [2] found anionic surfactants underestimate cationic-surfactant colorimetric results by up to 83 %, an effect largely removed by analysing sequential solid-phase extracts separately rather than combined.

Nonionic determination has its own logic, since the reagents used respond to ion association with other surface-active species rather than simple inorganic ions. Tôei et al. [20] and Favretto et al. [5] both found anionic and cationic surfactants cross-interfering with nonionic determination, resolved by pre-extraction. Inaba [9] showed why single-interferent testing can mislead: LAS, PEG-300, and humic acid were individually mild, but combined with 0.6 M NaCl pushed the result to 180 % of the true value. Yokoyama et al. [21] solved this structurally with a triple-stage cartridge that removes cationic and anionic surfactants before the nonionic fraction reaches the colour-forming reaction, eliminating rather than merely tolerating the interference.

Synthesis

Table 1 summarises the picture: three principal interference sources — competing inorganic ions and residual oxidants, cross-class surfactants, and organic matrix constituents — met by five countermeasures. These sources rarely act in isolation, as the combined-interferent data above show. Among the five countermeasures, procedural control (smaller volumes, shorter contact times [17]) suppresses interference fairly generally, almost regardless of chemistry; chemical masking is narrowly targeted but reliable for a known interferent, such as EDTA for Fe^{3+} [1, 3, 4, 8]; and

physical separation — a simple wash [14] or full ion-exchange fractionation [21] — tends to outperform correcting for interference mathematically. Reagent selection is the least-discussed strategy but, as shown above, the most fundamental, since it converts salt tolerance from an optimisation outcome into an inherent reagent property. Recent work extends this same logic across all four amphiphilic classes at once: Grisillon et al. [7] combined a graphitized-carbon-black SPE protocol with optimised ion-pair spectrophotometric detection to achieve separate quantification of cationic, nonionic, weak-anionic, and strong-anionic surfactants at nanomolar sensitivity, confirming physical fractionation before colour development as the most effective route to matrix-independent determination.

Table 1.

Reported interferents and mitigation strategies in extractive-photometric surfactant determination

Surfactant class	Analytical system	Main interferent(s)	Observed effect	Mitigation	Ref.
Anionic	Methylene blue	Mg^{2+}; NO_3^-	Positive bias above 500 ppm (Mg^{2+}); small positive bias above 20 ppm (NO_3^-)	Kept within tolerance limits	[6]
Anionic	Ethyl violet / p-xylene	Halides; Fe^{3+}	Positive bias (halides, own ion pair); negative bias (Fe^{3+}, competes for dye); other cations negligible	Extract washing (halides); EDTA masking (Fe^{3+})	[8]
Anionic	Co(5-CF_3-PADAP)$_2^+$ vs. Ethyl violet / Co(5-Cl-PADAP)$_2^+$	Cl^-; cationic surfactant; perchlorate	Cl^- severely biases weaker reagents (ethyl violet, Co(5-Cl-PADAP)$_2^+$), not Co(5-CF_3-PADAP)$_2^+$ (stable to 0.7 mol/dm³); cationic surfactant (−87.9%) and perchlorate (−4.8%) still bias it	Reagent selection resolves Cl^-; other two remain minor, unresolved	[11]
Anionic	Diaquabis(ethylenediamine)copper(II)	Sulfide; Fe^{3+}	Only these two interfere; major seawater ions do not	H_2O_2 oxidation (sulfide); EDTA masking (Fe^{3+})	[3]
Anionic	Methylene blue (flow)	Ca^{2+}, Mg^{2+}, Cd^{2+}, Cl^-, NO_3^-, oxalate, Triton X-100	Tolerable within quantified limits for each ion	Operated within quantified tolerance limits	[18]
Anionic	Methylene blue (extraction-chromatography)	Humic acid	Dual mechanism: raises background and competes with analyte	Intermediate acidic column wash	[14]
Anionic	MB / DDABr–CBBG	S^{2-}, SCN^-, I^-, phenol, humic acid, albumin	Interferes at reported concentrations	C18 SPE pretreatment	[16]
Anionic	Methylene blue (simplified)	Filtration	Causes surfactant loss in the classical protocol	Single-step extraction, no filtration	[10]
Anionic	Methylene blue (microextraction)	Broad ion/organic panel	No significant effect	Small volume, short contact time	[17]
Anionic	4-(4-N,N-dimethylaminophenylazo)-2-methylquinoline	Residual chlorine (ClO^-)	Positive interference	Ascorbic acid reduction	[13]
Cationic	Methyl orange	Cu^{2+}; Fe^{3+}	Cu^{2+} harmless; Fe^{3+} interferes above 0.5 µg/mL	EDTA masking; $Fe(OH)_3$ precipitation above 5 µg/mL	[4]

Surfactant class	Analytical system	Main interferent(s)	Observed effect	Mitigation	Ref.
Cationic	Chrome azurol S / 1-butanol	Cl^-, NO_3^-, SO_4^{2-}, PO_4^{3-}, CO_3^{2-}, Ca^{2+}, Mg^{2+}, Pb^{2+}, Fe^{3+}, Ni^{2+}, Cu^{2+}, SDS, Triton X-100	13 ions bias signal at low μM; real-water recovery <23% unmasked	EDTA masking (4 g/L); recovery restored to 75–90%	[1]
Cationic	Coomassie Brilliant Blue G	Triton X-100 (nonionic surfactant, organised medium)	Signal enhanced and colloidal system stabilised, not degraded, by coexisting nonionic surfactant	Reagent-chemistry effect exploited rather than removed (opposite-direction cross-class effect)	[12]
Nonionic	TBPE-K	Anionic surfactants; filtration/storage	Anionic surfactants bias the signal; filtration and storage reduce recovery	Controlled sample handling	[20]
Nonionic	Picrate	Anionic and cationic surfactants	Cross-class interference	Preliminary extraction/removal step	[5]
Nonionic	Co(II)–PAR (toluene)	LAS, PEG-300, humic acid, NaCl	Individually mild; LAS+humic acid+NaCl combined — strong non-additive bias	Ion-exchange pretreatment for saline/high-load samples	[9]
Nonionic	Ferric thiocyanate (toluene)	Cationic and anionic surfactants	Eliminated, not just tolerated	Sequential SCX–SAX–C18 ion-exchange fractionation	[21]
Anionic	Methylene blue (NADES microextraction)	None reported (green-solvent alternative)	Chloroform-free NADES extraction, smartphone colorimetry	Solvent substitution (NADES replaces chloroform)	[19]
Cationic	Colorimetric quantification (seawater SPE extract)	Anionic surfactants (complex mixtures)	Underestimation of cationic surfactant concentration by up to 83%	Sequential two-step SPE, separate quantification per extract	[2]
Anionic / Cationic / Nonionic	SPE (graphitized carbon black) + ion-pair spectrophotometry	Cross-class surfactant mixtures	Separate quantification of 4 surfactant classes at nanomolar sensitivity	Sequential-elution SPE fractionation before colour development	[7]
Anionic	MIBK–1,2-dichloroethane (3:1)	F^-, Cl^-, Br^-, I^-, NO_2^-, NO_3^-, CN^-, phosphate, carboxylates (15 anions tested)	Minimal interference except Br^-/I^- above natural-water concentrations	Solvent selection (replaces chloroform); stable pH 3–10, ionic strength to 2 mol/L	[22]

Conclusion

Matrix interference in extractive-photometric surfactant determination can be grouped into three principal sources: competing inorganic ions and residual oxidants, cross-class surfactants, and organic constituents such as humic acids and proteins. These sources may interact rather than act independently under realistic sample conditions, and no single reagent handles all three at once. Five countermeasures appear across the literature: chemical masking, selective pre-extraction, physical separation by washing or ion exchange, procedural control such as smaller extraction volumes or shorter contact times, and choosing a reagent whose coordination chemistry is inherently resistant to the expected interferent, an option that is the least discussed of the five, though arguably the most fundamental. That last option has a real mechanistic basis: reagents forming inner-sphere complexes with the surfactant can provide substantially greater salt tolerance than conventional outer-sphere ion-pairing dyes, however carefully the latter are optimised.

The main contribution of this study is the integration of these five countermeasures into a single classification, applicable across anionic, cationic, and nonionic surfactants rather than to one dye system at a time. This classification rests on two distinct underlying principles: physical removal of the interferent before colour development, and structural resistance built into the reagent itself, rather than a list of unrelated tricks. A further finding, drawn directly from the combined-interferent data of Inaba [9], is that interferents harmless on their own can combine into a substantial, non-additive error in a real saline matrix: LAS, humic acid, and NaCl each stayed within tolerance individually, yet together produced approximately 80 % positive bias. This shows that single-interferent spiking, the default validation approach in most of the reviewed literature, is not sufficient by itself to establish a method's reliability in a real matrix. Recent evidence (2022–2026) reinforces this framework: solid-phase fractionation before colour development continues to resolve cross-class interference in modern seawater and freshwater applications [2, 7, 19], confirming that the two governing principles identified here — physical removal of the interferent and reagent-level structural resistance — remain valid as analytical methods evolve.

These findings are particularly relevant to environmental waters with variable salinity, hardness, and organic load. Method validation should therefore consider the dominant combination of matrix interferents rather than relying on single-interferent tests alone.

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