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RECENT REVIEW OF THE QUECHERS SAMPLE PREPARATION METHOD FOR FOOD AND ENVIRONMENTAL SAMPLE ANALYSIS

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ABSTRACT

Creating reliable, environmentally responsible, and effective processes that ensure the traceability, safety, and caliber of their results is one of the main challenges facing researchers doing multi-residue analysis. The QuEChERS which stands for Quick, Easy, Cheap, Effective, Rugged, and Safe method has shown itself to be highly adaptable, yielding positive outcomes with a range of analytes. This method allows for versatility in the choice of solvents, salts, and buffers for salting-out partitioning, as well as the use of various sorbents throughout the cleanup process. QuEChERS is a more environmentally friendly sample preparation technique that fits perfectly with analytical chemistry's rising emphasis on sustainability. This review paper's goal is to illustrate the primary applications of the QuEChERS sample preparation method, with a focus on food and environmental investigations. It also covers important improvements in the history of sample preparation methods and offers insights into the classes of substances that have been effectively evaluated with this methodology.

Keywords: QuEChERS, Preparation, Analysis

INTRODUCTION

Anastassiades *et al.* released the QuEChERS method in 2003 after it was initially presented at the Fourth European Pesticide Residue Workshop in Rome in 2002. QuEChERS stands for Quick, Easy, Cheap, Effective, Rugged, and Safe (Veiga-del-Baño *et al.,* 2024). Agri-food and environmental analysis were transformed by this technique, which represented a breakthrough in analytical chemistry (Yao, 2023). The European Committee for Standardization's Standard Method EN 15662 and AOAC International's Official Method 2007.01 are two examples of buffered versions of the QuEChERS method, created by Anastassiades and Lehotay, they are now accepted as official techniques for identifying pesticide residues in fruits and vegetables (Yun *et al.,* 2024). Beyond analysis, the QuEChERS approach has influenced the creation of new commercial goods and marketing plans by makers of analytical instruments and providers of chemical reagents (Juhee *et al.,* 2024). Through a simplified procedure that consists of sample homogenization, solvent extraction/partitioning utilizing a salting-out method and dispersive solid-phase extraction (dSPE) with different sorbents and salts for cleanup, and analysis, the QuEChERS methodology reduces mistakes (Jain *et al.,* 2023). Both novices and seasoned analysts can use the procedure because of its ease of use, which speeds up sample processing (Santana-Mayor *et al.,* 2023).

The QuEChERS method's adaptability enables customisation to manage intricate matrices or particular extraction needs. Typical adjustments include the use of buffer salts, freezing-out methods for extremely fatty samples, alkaline hydrolysis for acidic chemicals, and new effective sorbents (Martínez *et al.,* 2024). In a variety of matrices, including environmental, food, feed, pharmaceutical, biological, and forensic samples, the QuEChERS method has proven to be highly effective and robust in the extraction and analysis of a broad range of analytes, including pesticides, mycotoxins, pharmaceutical residues, and illegal drugs (Varela-Martinez *et al.,* 2020). Multiclass approaches, based on the green chemistry idea of multi-residue analysis, have been used a lot. However, matrix interferences and co-extracted chemicals might affect the accuracy and reliability of the analysis and possibly damage the instruments, therefore the choice of extraction solvents and sorbents has a significant impact on the method's performance (Drabińska *et al.,* 2023). Consequently, in order to overcome these obstacles, method optimization is crucial (Santana-Mayor *et al.,* 2023)The extraction solvent, sample size and ratio, pH, types and quantities of salts for solvent extraction, sorbent types and quantities, and agitation time and mode are important considerations (Santana-Mayor *et al.,* 2023).

The sustainability features of the QuEChERS method are well known and in keeping with developments in green analytical chemistry (Hashim *et al.,* 2024). Compared to conventional methods, this approach drastically lowers the amount of solvent and glass material used, which lessens the environmental effect of production, shipping, and disposal (Santana-Mayor *et al.,* 2023). Anastassiades, for instance, found that this approach reduced solvent usage and expenses by around 95% and analysis time by about 90% when compared to a conventional methodology that is commonly used in Europe for pesticide analysis (Santana-Mayor *et al.,* 2023).

However, environmental exposure to toxic organic solvents is still a problem and contributes to pollution to some degree (Zhang *et al.,* 2024). Therefore, it is desirable to investigate the feasibility of alternative solvents, such as natural deep eutectic solvents (DESs), which are environmentally friendly and produce good results when combined with other techniques, in order to generate less waste and pollution (Santana-Mayor *et al.,* 2023). Additionally, the process's contaminant impact has been further reduced by the protocol's miniaturization, producing the so-called µ-QuEChERS (Kokosa, 2024). This miniaturization has proven advantageous not only in reducing the amount of reagents and solvents, but also in minimizing sample requirements, which is especially helpful for samples that are scarce or limited in availability, such as biological matrices. Nevertheless, it is important to take into account the QuEChERS method's limitations in spite of these benefits (Burato *et al.,* 2020). Compared to more conventional extraction methods like Solid

Phase Extraction (SPE), ultrasound-assisted extraction, and Pressurized Liquid Extraction (PLE), the QuEChERS approach has certain drawbacks despite its many benefits (Mandal et al., 2023). The comparatively lower enrichment factors, which may result in greater limits of quantification (LOQ), are one significant drawback (Mandal *et al.,* 2023). The robustness of the approach can occasionally be impacted by the presence of significant concentrations of co-extractives, especially in fatty matrices. QuEChERS is frequently used in conjunction with gas chromatography (GC) or liquid chromatography (LC) in conjunction with mass spectrometry (MS) or tandem mass spectrometry (MS/MS) to reduce interferences (Oymen *et al.,* 2022).

Furthermore, despite the QuEChERS method's flexibility, it necessitated adjustments in order to attain high recovery rates (usually above 85%) for a range of analytes across different matrices. This is both a benefit and a disadvantage. Numerous variations of the approach have been developed as a result of this flexibility, which might be troublesome because they are not harmonized (Elattar and El-Deen, 2024). Nevertheless, the QuEChERS method's versatility has prompted the creation of customized solutions, such as fully automated systems that can complete the process with little assistance from humans (Mandal *et al.,* 2023). When compared to more conventional extraction methods like matrix Solid Phase Dispersion, Liquid-Liquid Extraction (LLE), or SPE, the QuEChERS method's ease of use, speed, and affordability have all helped to increase its popularity (Lou *et al.,* 2023). Although initial investments in specialist equipment or sorbents may be required, it uses cheap materials, little solvent, and little equipment. Furthermore, QuEChERS uses sealed containers to minimize worker exposure to solvents and takes up little bench space, making it appropriate for small or transportable labs (Houliston, 2022).

By addressing the drawbacks of current multiclass multiresidue approaches, the QuEChERS methodology aimed to create an extraction technique that was both more efficient and environmentally beneficial (Santana-Mayor *et al.,* 2023). Sample size, sample pH, matrix constituents, extraction solvent type and ratio, agitation method, temperature, addition of salt and/or solvent, extraction time, and type and quantity of clean-up sorbents are just a few of the parameters that have been systematically studied and optimized to affect the method's efficiency (Santana-Mayor *et al.,* 2023). In order to strike a balance between simplicity, applicability, speed, selectivity, and analyte recovery, the final conditions were carefully chosen (Santana-Mayor *et al.,* 2023). Continuous advancements in analytical equipment, especially GC-MS(/MS) and LC-MS/MS, have broadened the field of analysis while preserving a high degree of selectivity since the introduction of QuEChERS (Tsiantas *et al.,* 2023). In order to achieve a balance in the purification operations, the cleaning procedure could be improved to reduce matrix effects and prevent instrument contamination. Protocols that extract a wider variety of compounds with varying natures (family and polarity) are preferred by this method. QuEChERSER (QuEChERS + Efficient and Robust Recovery) is an advancement of the technique that Lehotay and colleagues have presented (Santana-Mayor *et al.,* 2023). By making minor but significant changes, this updated method of rationalized sample preparation seeks to assess a wider variety of multiclass substances with different polarities (Santana-Mayor *et al.,* 2023). In QuEChERSER, for instance, 1–5 g of samples are used in liquid nitrogen instead of 10–15 g in QuEChERS; a 5 mL/g solvent-to-sample ratio of acetonitrile (ACN) (4:1, v/v) is used instead of 1 mL/g of ACN and water is added for dry samples; and 200 µL of the ACN phase is ultracentrifuged for LC analysis instead of salting-out and dSPE (Santana-Mayor *et al.,* 2023).

Unlike QuEChERS, which uses dSPE, QuEChERSER uses automated µ-SPE for GC analysis (Santana-Mayor *et al.,* 2023). Research has demonstrated that QuEChERSER has a 5% lower overall relative standard deviation (RSD) than QuEChERS, although employing a smaller test section. This suggests that QuEChERSER is more reproducible than QuEChERS (Rodríguez-Ramos *et al.,* 2024).. To minimize weak complexing agents (such magnesium and perhaps sodium) and to make it easier to transfer important analytes into the organic phase, the p-QuEChERS variation recommends utilizing a potassium hydrogen phosphate/dihydrogen phosphate mixture for the salting-out process (Santana-Mayor *et al.,* 2023). However, by employing a liquid-liquid extraction of the original CAN, the so-called FATChERS seeks to expand the method's usefulness in highly fatty matrices.

Applications of QuEChERS method

Numerous applications have made extensive use of the QuEChERS technique. These sections give a summary of the most current uses of the QuEChERS technique in food and environmental samples.

Food analysis

Food analysis, particularly that of fruits and vegetables, has been one of the main areas of application for the QuEChERS method (Narenderan et al., 2020); Ferracane et al., 2021; Wahab *et al.,* 2022; Dong *et al.,* 2023; Mabunda *et al.,* 2024). With little attention to evaluating the samples' bioactive potential, the majority of these applications concentrate on guaranteeing the safety of food for both humans and animals (Casado *et al.,* 2022; Benenguer et al., 2023; Mateus *et al.,* 2024). Examples of recent applications of the QuEChERS technique in food analysis are shown in Table 1 (Monteiro *et al.,* 2021; Cebi *et al.,* 2021; Hakami *et al.,* 2021; García-Vara *et al.,* 2022; Bakanov *et al.,* 2023; Mabunda *et al.,* 2024).

Over 50% of current applications, including the initial QuEChERS technology, have been utilized to find pesticide residues or the breakdown products of such residues in different foods (Feng *et al.,* 2020); (Rahman and others, 2021). In 2022, González-Curbelo et al.; in 2023b, Mandal *et al.,* in 2024, Antonio et al.; and in 2024, (Radowan, 2024). Other organic contaminants detected include furfural compounds (Mokh *et al.,* 2024), polycyclic aromatic hydrocarbons (PAHs) (Zhang *et al.,* 2020); (Peng and Lim, 2022); (Ostadgholami *et al.,* 2023) and (Trantopoulos *et al.,* 2024), alkaloids and other toxins (Casado *et al.,* 2022); (Fuente-Ballesteros *et al.,* 2023); (Jiao *et al.,* 2024), acrylamide (Sebastià *et al.,* 2023), plasticizers (Silva *et al.,* 2023); (Santini *et al.,* 2024), amines (Guo *et al.,* 2024), pharmaceuticals and veterinary drugs (Kalogeropoulou *et al.,* 2021); (Ninga *et al.,* 2022); (Kim *et al.,* 2023) ;(Brandi *et al.,* 2024), polychlorinated biphenyls (PCBs) (Reddy *et al.,* 2020); (Gamal *et al.,* 2024) (Kiani *et al.,* 2023), antioxidants (Santana-Mayor *et al.,* 2023), cannabinoids (Wylie *et al.,* 2020); (Reyes-Garcés and Myers, 2021);(López-Ruiz *et al.,* 2022) (Christodoulou *et al.,* 2023); , and estrogenic compounds (Sweeney *et al.,* 2021). Additionally, compounds with potential health benefits, such as flavonoids and phenolic acids, have also been analyzed using QuEChERS (Izcara, Perestrelo, *et al.,* 2022). The method has even been applied to analyze flame retardants, synthetic dyes, surfactants, illegal drugs, preservatives, steroids, amino acids, and other substances in food and feed (Tartaglia *et al.,* 2020);(Eyring *et al.,* 2021);(Makni *et al.,* 2022); (Marazuela, 2023); (Kadhum *et al.,* 2024). With the goal of creating more ecologically friendly and sustainable techniques, there is growing interest in integrating green chemistry concepts into analytical procedures. For instance, deep eutectic solvents (DESs) were used as extraction solvents and cleanup materials in the development of a modified, miniature QuEChERS process (Petrarca *et al.,* 2024). When employing gas chromatography-mass spectrometry (GC-MS) to analyze soyabean samples for pesticides, their approach produced good recovery rates and sensitivity, as well as enhanced extraction capacity and selectivity.

The QuEChERS approach has been used in numerous research to do multi-residue analysis of different chemical families. To extract 266 pesticides, 12 mycotoxins, 14 alkaloid toxins, and 3 Alternaria toxins from samples of maize and wheat, for example, Tölgyesi *et al.,* (2023) devised a multi-residue approach. This approach yielded good recovery values and limits of quantification (LOQs). Although the QuEChERS approach typically produces favorable outcomes, it occasionally fails to extract target chemicals and clean samples. Additionally, there is a tendency toward analytical procedures being smaller and simpler. For example, Bernardi *et al.,* 2020 used a modified QuEChERS approach to create a multi-residue method for extracting 197 pesticides, 56 veterinary medications, and 5 mycotoxins from animal feed samples (Santana-Mayor *et al.,* 2023). They diluted the final extract tenfold for ultra-high-performance liquid chromatography-mass spectrometry (UHPLC-MS/MS) analysis to account for considerable matrix effects, although this decreased the sensitivity of the approach.

PSA is one of several commercial clean-up sorbents that have been assessed to enhance compound detection with QuEChERS (Perestrelo *et al.,* 2019); (Bernardi et al., 2020); (Stringhini *et al.,* 2021); (Chen *et al.,* 2022); (Sadighara *et al.,* 2023); (Fontana *et al.,* 2024), C18 (Dong *et al.,* 2019); (Scordo *et al.,* 2020); (Tran-Lam *et al.,* 2021); (Casado, *et al.,* 2022); (Koloka *et al.,* 2023); (Han and Nam, 2024), graphitized carbon black (GCB) (Guo et al., 2024), EMR-lipid (Carro *et al.,* 2024), Z-Sep+ (Jung *et al.,* 2023); (Prata *et al.,* 2024), (Soriano et al., 2024), SAX (Kravos and Prosen, 2024), WAX (Tripathy *et al.,* 2024), CarbonX (Rodríguez-Ramos *et al.,* 2024), and multi-walled carbon nanotubes (MWCNTs) (Jiao and others, 2024). Additionally, functionalized nanomaterials have been investigated. To purify neonicotinoid pesticides from goji berries, for instance, (Elattar and El-Deen, 2024) created a modified QuEChERS approach that used porous boron nitride nanorods as a clean-up sorbent. This method performed better than traditional materials. The majority of applications now involve coupling high-resolution mass spectrometry (HRMS) systems with various analyzers, such as triple quadrupole (QqQ) (Perestrelo *et al.,* 2019); (Guo *et al.,* 2020); (Bessaire et al., 2021); (Stefanelli and Barbini, 2022); (Kaufmann *et al.,* 2023); (Nwachukwu et al., 2024), ion trap (IT) (Boti *et al.,* 2024), QTrap (Santana-Mayor *et al.,* 2023), quadrupole-time-of-flight (QToF) (Marín-Sáez et al., 2023), Orbitrap (Montemurro *et al.,* 2024), and single quadrupole (Q) (Yan et al., 2024), even though conventional detection systems are still used. More sensitivity and selectivity are offered by these sophisticated detecting technologies. The approach created by Kalogeropoulou *et al.,* (2021) to enhance standard laboratory analysis is an illustration of fusing these components with automation. By concurrently extracting 106 veterinary medications, 227 pesticides and metabolites, and 16 PCBs from catfish samples, this high-throughput technique, known as "QuEChERSER," achieved good recovery and repeatability for 349 chemicals in a complex matrix.

Environmental analysis

Pollution of air, water, and soil has been monitored using the QuEChERS method, mainly from pesticides (Acosta-Dacal *et al.,* 2021); Słowik-Borowiec *et al.,* 2022; Tsiantas *et al.,* 2023; Su, Liu, *et al.,* 2024); and other contaminants of emerging concern (CECs), including polycyclic aromatic hydrocarbons (PAHs) (Zhang *et al.,* 2020); (Baroudi *et al.,* 2022); (Prata *et al.,* 2024); and polychlorinated biphenyls (PCBs) (Kiani *et al.,* 2023); pharmaceuticals (Santana-Mayor *et al.,* 2023) quaternary ammonium compounds (QACs) (Bobic *et al.,* 2024), musks (Li *et al.,* 2023), bisphenols (Wang *et al.,* 2024), alkylphenols (Guo *et al.,* 2024), ultraviolet (UV) filters (Prata *et al.,* 2024), chlorinated paraffin (Lambert *et al.,* 2024), steroid hormones and synthetic oestrogens (Li *et al.,* 2023), brominated flame retardants (BFRs) (Fernandes *et al.,* 2023), among others. The QuEChERS procedure has been used to determine n-alkanes (lipid biomarkers that provide information about former environments) in addition to evaluating pollutants in various environmental compartments. In this instance, 29 n-alkanes and two isoprenoid acyclic hydrocarbons (phytane and pristane) in ancient sediment samples were evaluated using a QuEChERS-based procedure, which was followed by GC-MS analysis (Herrera-Herrera *et al.,* 2020). A sequential approach was used to optimize the process, which included assessing the type of extraction solvent, water addition, agitation mode, and the kind and quantity of clean-up sorbents.

Table 1: Some recent examples of the application of the QuEChERS method in food analysis

Sample (amount)	Sample (Analytes)	Solvents used	Extractions Salt	Clean-up steps	Analytical Techniques	%Recovery	Limits of Quantification	Remarks	References
Cereals (5 g)	266 pesticides, 12 mycotoxins, 14 alkaloid toxins, and 3 Alternaria toxins	9.9 mL ACN	4 g $MgSO_4$, 1 g NaCl, 1 g triNa, 0.5 g di-Na	-	HPLC- (QqQ)-MS/ MS (ESI+/)	67–103	0.05–200 µg/ kg	- Several ISs and surrogates were used. - Evaluation of 23 wheat or maize quality control and proficiency test.	Monteiro *et al.*, 2021
Bee products (1 g)	i) 20 PAHs ii) 56 pesticides	10 mL ACN	4 g $MgSO_4$, 1 g NaCl	200 mg $MgSO_4$, 300 mg C_{18}	i) GC-(IT)- MS (internal ionisation mode) ii) HPLC-DAD	i) 71-111 ii) 66-107	i) 0.24–0.78 µg/kg ii) ≤0.0262 µg/kg	- Deuterated and non-isotopically labelled surrogates were used.	Cebi *et al.*, 2021
Milk, salmon, lettuce, bread (1–5 g	16 PFASs	10 mL ACN (150 µL formic acid)	6 g MgSO4, 1.5 g NaCl	1) 900 mg MgSO4, 300 mg PSA, 150 mg GCB 2) 200 mg WAX SPE (3 mL tube)	UHPLC-(QTrap)- MS/MS (ESI-)	40–120	7–107 ng/kg	- Deuterated IS was used. - Analysis of 179 total diet study samples, including fruits and vegetables, breads, dairy products, animalderived foods, among others.	Hakami *et al.*, 2021
Jamb (1 g)	1 PAHs	10 mL ACN	1.0 g NaCl	1) 1.80 g $MgSO_4$, 400 mg PSA, 150 mg SAX, 50 mg C18 LLE extraction with 0.50mL n-hexane	GC-MS	55–113	0.6–1.5 µg/ kg	- Deuterated IS was used. - Method optimization was carried out Plackett-Burman experimental design. - Analysis of 6 jambu samples.	García-Vara *et al.*, 2022
Edible vegetable oil (0.4 g)	2 antioxidants, 3 photoinitiators, 3 plasticisers, 4 UV filters	4 mL ACN	-	800 mg $MgSO_4$, 40 mg PSA		60–106	0.15–0.51 mg/L	- Method optimization was carried out.	Mabunda *et al.*, 2024

ACN: acetonitrile; C_{18}: octadecylsilane; ChCl: choline chloride; DAD: diode array detector.

Table 2: Some recent examples of the application of the QuEChERS method in environmental analysis

Sample (amount)	Sample Analytes	Solvents Used	Extractions Salt	Clean-up steps	Analytical Techniques	% Recovery	Limits of Quantification	Remarks	References
Powder aerosol particles (10 mg)	14 PAHs	0.4 mL ACN/DCM (7:1, v/v)	20 mg Na_2SO_4/ NaCl (1:1, w/w)	8.0 mg PSA, 16 mg Na_2SO_4	HPLC	85–121	5.8–82.6 μg/L	-Method optimization was carried out using experimental designs.	Acosta-Dacal *et al.*, 2021
Soils (20 g)	3-chloroamide herbicides	15 mL water, 20 mL ACN (0.2%, v/v, formic acid)	4 g MgSO4, 1 g NaCl	UHPLC- (QqQ)-MS/ MS (ESI+)	90–104	1 μg/kg		- Analysis of 1010 soil samples, 616 surface water samples and 737 ground water was carried out.	Słowik-Borowiec *et al.*, 2022
Sewage sludge and hydrochar (1 g)	33 pharmaceuticals and metabolites	10 mL 0.1 M EDTA solution, 10 mL ACN (0.1%, v/v, acetic acid)	4 g $MgSO_4$, 1 g NaCl, 1 g tri-Na, 0.5 g di-Na	600 mg $MgSO_4$, 200 mg PSA, 75 mg of Z-Sep+	UHPLC-(Orbitrap)- MS (Ion Max ESI+)	51–104	0.8–24 μg/ kg	- Comparison of acetate and citrate buffer extraction solvents was carried out. - Method optimization was carried out.	Santana-Mayor *et al.*, 2023.
wastewater sludge and effluent,	10 pharmaceuticals and QACs	Manual: 3.5 mL water, 10 mL ACN	Manual: 4 g $MgSO_4$, 1 g NaOAc	Manual: 900 mg $MgSO_4$, 150 mg PSA	Manual: UHPLC-(Orbitrap)- MS and MS/ MS (ESI+)	Manual: >62		- Manual QuEChERS methods were developed.	Monteil-Rivera *et al.*, 2024
River sediments (10 g)	37 CECs	10 mL water, 10 mL ACN	2 g $MgSO_4$, 0.5 g NaOAc	200 mg EMRlipid	GC-(Q)-MS (EI)	71–117	0.025–200 μg/kg dw	- Evaluation of pesticides, polycyclic musks and biphenols was carried out. - Method optimization was carried out. .	Baroudi *et al.*, 2022
Wastewater sludge (0.5 g)	7 pharmaceuticals (β-lactams antibiotics) and one degradation product	g EDTAtreated sand, 5 mL 0.1 M EDTA solution, 10 mL ACN	4 g $MgSO_4$, 1 g NaCl, 1 g tri-Na, 0.5 g di-Na	Strata X™ SPE cartridge (styrene-divinylbenzene polymer)	UHPLC-(QTrap)-MS/MS (ESI+)	96–114	1.9–17.4 μg/kg	- Method optimization was carried out. - Clean-up step was performed using Solid Phase Extractions Analysis. - Deuterated ISs were used	Bobic *et al.*, 2024
Lake sediments (5 g)	LCCP standards (36 and 49% Cl)	5 mL water, 15 mL ACN	4 g $MgSO_4$, 1 g NaCl	1200 mg $MgSO_4$, 400 mg PSA, 400 mg C_{18}, 45 mg GCB	2D-UHPLC-(Orbitrap)- MS (ESI-)	81–95	1.38–7.00 μg/L	- Previous lyophilisation of samples was carried out. - Re-extraction with ACN. -	Prata *et al.*, 2024

Aquatic environment sediments (2 g)	11 CECs	1 mL water, 9 mL ACN, 3 mL acetone	2 g $MgSO_4$ and NaOAc	2 g MgSO4, 500 mg PSA, 500 mg C_{18}	HPLC-UV	64–109	5.0–86 ng/L	Evaluation of 3 pharmaceuticals, 4 steroid hormones, 4 xenoestrogens and 2 alkylphenols.	Guo *et al.,* 2024
Treated urban wastewater (10 mL)	107 CECs	10 mL ACN	4.0 g $MgSO_4$, 1.0 g NaCl, 1.0 g di-Na, 0.5 g tri-Na	750 mg $MgSO_4$, 125 mg Z-sep	HPLC- MS/MS	70-120 (95 analytes)	≤50 ng/L (95 analytes)	- Evaluation of 74 pharmaceuticals and 33 pesticides. - Comparison of five different QuEChERS extraction versions.	Santana-Mayor *et al.,* 2023
River water (10 mL)	Atrazine (herbicide) and two metabolites	5 mL ACN	3 g $MgSO_4$, 1 g of NaCl	100 mg MgSO4, 30 mg PSA, 50 mg C_{18}	HPLC-DAD	84–106	0.12–0.29 μg/L	- Previous filtration of samples. - QuEChERS and SPE extractions were compared.	Kim *et al.,* 2023
Soils (2.5 g)	6 QACs	4 mL water, 10 mL ACN	4 g MgSO4, 1 g NaCl, 1 g tri-Na, 0.5 g di-Na	900 mg MgSO4, 150 mg PSA, 150 mg C_{18}	LC-(IT)-MS/ MS (ESI+)	>53	-	- Comparison of commercial cleanup salts kits for waxed fruits and vegetables and pigmented fruits and vegetables.	Sadighara et al., 2023
Archaeological sediments (5 g)	29 n-alkanes and two isoprenoid acyclic hydrocarbons (pristane and phytane)	10 mL DCM	4 g $MgSO_4$, 1 g NaCl	900 mg $MgSO_4$, 150 mg PSA	GC-(Q)-MS (EI)	34–117	2.2–37.9 μg/kg	- Previous oven drying of samples. - Method optimization was carried out.	C. Li *et al.,* 2023
Sediments and plant tissues of Z. capensis (10 g)	13 OPPs	10 mL ACN	7.5 g $MgSO_4$, 1 g NaCl	150 mg $MgSO_4$, 50 mg PSA, 50 mg GCB	GC-(Q)-MS (−)	63–111	-	- Method optimization was carried out. - Deuterated and non-isotopically labelled surrogates were used.	Tsiantas *et al.,* 2023

ACN: acetonitrile; C_{18}: Octadecylsilane; DCM: dichloromethane; di-Na: sodium citrate dibasic sesquihydrate; DLLME: dispersive liquid-liquid microextraction; ECD: electron capture detector; EDTA: ethylenediaminetetraacetic acid.

Paddy fields (Wu *et al.,* 2023; Dong *et al.,* 2022) are included in the environmental matrix analysis (Table 2), (González-Curbelo et al., 2022) (Acosta-Dacal *et al.,* 2021). Among other things, Nguyen and Baduel (2023) show the validation yielded recovery values ranging from 34% to 117%, meeting international standards, following the optimization of the extraction process using 5 g of dry sample without water, 10 mL of dichloromethane (DCM), 4 g of MgSO4, 1.0 g of NaCl, and 900 mg of $MgSO_4$ with 150 mg of primary secondary amine (PSA) for clean-up.4 g of $MgSO_4$, 1.0 g of NaCl, and 900 mg of $MgSO_4$ with 150 mg of primary secondary amine (PSA) for clean-up, the validation produced recovery values ranging from 34% to 117%, meeting international standards. Five sediment samples from a Spanish Palaeolithic site were analyzed using the approach, which showed great sensitivity with LOQs ranging from 2.2 to 37.9 μg/kg and produced useful paleontological data. Additionally, the QuEChERS approach has been used for bioindicators including snails and pine needles (Baroudi *et al.,* 2022) and plant tissues (phytoremediators) (Bruzzoniti *et al.,* 2014). For instance, (Collimore and Bent, 2020) assessed the concentrations of 13 organophosphate pesticides (OPPs) in seagrass (*Zostera capensis*) and estuary sediments using a modified QuEChERS technique. The bioaccumulation of OPPs in seagrass was successfully assessed using this analytical technique, demonstrating the ability of *Z. capensis* to eliminate OPPs from urban settings. The circular economy movement has also brought attention to the reuse of agri-food waste in recent years, which calls for the safety of these materials to be assessed before they are utilized in new applications, particularly those meant for human consumption. In this regard, Shyamalagowri *et al.,* (2023) devised a method based on the QuEChERS technique, which was followed by GC separation and a variety of detection systems, to assess the presence of 30 CECs in vine canes (the inedible portions of grapevines), including 12 OCPs, 6 OPPs, 5 PCBs, and 7 BFRs.

The milling size of vine canes and the make-up of extraction and cleanup salts were among the experimental parameters that were optimized in this study. The smallest size of milled vine canes, the original QuEChERS extraction salt mix, and 3 mg of carbon for cleanup produced the best extraction efficiency and repeatability results. RSDs were less than 14%, and recovery values varied from 59% to 105%. Applying the validated methodology to 19 vine cane samples from various kinds confirmed the necessity of keeping an eye out for environmental pollutants in this antioxidant-rich and potentially beneficial substance.(Caratti and others, 2022). The most widely used extraction solvent is acetonitrile (ACN) [(Mahdavi *et al.,* 2021);(Khanehzar *et al.,* 2021);(Tran-Lam *et al.,* 2021);(Kecojević *et al.,* 2021);(Andjelković and Branković, 2023);(Cebi *et al.,* 2021);(Li *et al.,* 2024) (Iskandar *et al.,* 2024); (Liu *et al.,* 2024)], though other solvents like DCM with n-hexane (Sokołowski et al., 2023), acetone (Kadhum *et al.,* 2023), methanol (MeOH) (Tegegne *et al.,* 2023), and ACN acidified with formic or acetic acid have also been used. Analyte-matrix interactions may be impacted by acidic pH levels, which may encourage analyte dissolution during the extraction stage (Yang *et al.,* 2023). Sample sizes were generally between 0.5 and 20 g (1–10 mL for liquid matrices), while the extraction solvent quantities ranged from 0.4 to 20 mL [(García-Vara *et al.,* 2023); (Amin *et al.,* 2023); (Galindo *et al.,* 2024); (Fuente-Ballesteros *et al.,* 2024)]. Smaller sample sizes (e.g., 10 mg for atmospheric aerosol particles) have been tested (Yun *et al.,* 2023), and some research optimized sample sizes (Prata et *al.,* 2024). Hydrating the matrix can help the extraction solvent reach the sample when working with solid matrices. 0 to 15 mL were the ideal water addition volumes (Santana-Mayor et al., 2023). NaCl for the salting-out effect and anhydrous $MgSO_4$ as a phase-separating and drying agent (1.4–7.5 g) are examples of frequently employed extraction salts. To preserve matrix-interference compounds, substitutes such as improved matrix removal-lipid (EMR-lipid) (Santana-Mayor *et al.,* 2023) and diatomaceous earth (Bacha *et al.,* 2023) have also been employed. In certain instances, the cleanup phase was skipped, leading to a process that, depending on the matrix, might be better characterized as liquid-liquid extraction (LLE) or solid-liquid extraction (Sadighara *et al.,* 2023).

Beyond conventional manual techniques, recent advancements in the QuEChERS approach include the utilization of several mechanical aid modes for agitation (Horstkotte, 2023; Lee *et al.,* 2024). These include ultrasounds (Lou *et al.,* 2023), shaking platforms (Mou *et al.,* 2023), vortex agitation (Ferrari and Speltini, 2023; Wang *et al.,* 2024; Pratta *et al.,* 2024), and combinations of these techniques (e.g., vortex and ultrasounds). Ultrasounds and rotating shakers (Sayed *et al.,* 2023; Dong *et al.,* 2023; Vicari *et al.,* 2024). The goal of these techniques is to improve extraction efficiency and reduce the amount of co-extracted matrix compounds. Automation of sample preparation, aligned with Green Chemistry principles, is another significant advancement. Monteil-Rivera *et al.,* (2024) explored both manual and automated QuEChERS approaches for analyzing pharmaceuticals and QACs in soil, wastewater sludge and effluent, and biota samples (with automation applied only to biota matrices). In addition to lowering the overall analysis time to less than 40 minutes, the automated procedure called for smaller sample sizes, lower solvent volumes, and fewer extraction and cleanup sorbents. The data highlighted the necessity of continuous environmental monitoring by indicating inadequate pollutant removal during wastewater treatment. Despite being a proof of concept, this work demonstrates the QuEChERS method's scalability, versatility, and high-throughput potential. Other recent changes include using solid-phase extraction (SPE) rather than dispersive SPE (dSPE) for the cleanup step (Mabunda *et al.,* 2024) and combining QuEChERS with other extraction methods, such as DLLME, for additional analyte pre-concentration and derivatization prior to GC-MS analysis (Moreda-Piñeiro and Moreda-Piñeiro, 2023).

Gas chromatography (GC) and liquid chromatography (LC), frequently in conjunction with mass spectrometry (MS) or tandem mass spectrometry (MS/MS), are the primary methods used in the analysis of QuEChERS extracts from environmental samples for the separation and detection of target analytes (Song et al., 2020); (Tran-Lam *et al.,* 2021); (Słowik-Borowiec *et al.,* 2022). Single quadrupole (Q), triple quadrupole (QqQ), ion trap (IT), orbitrap, and triple quadrupole-linear ion trap (QTrap) are among the mass analyzers that have been used, especially in high-resolution mass spectrometry (HRMS) (Shi *et al.,* 2024). These devices usually use electron impact (EI) ionization sources and electrospray ionization (ESI) in both positive (ESI+) and negative (ESI-) modes, or a mix of the two.

Apart from MS, some investigations have used GC or LC in conjunction with traditional detectors such as diode array detectors (DAD), electron capture (ECD), fluorescence (FLD), flame photometric detectors (FPD), and UV-visible (UV-Vis) (Manggala *et al.,* 2023). Although they might not offer the same degree of sensitivity and specificity as MS-based techniques, these detectors give an alternate option for analyte identification.

CONCLUSION

The QuEChERS method, well-established for over two decades, continues to evolve in response to the changing social and environmental landscape. With the emergence of new food products, growing environmental concerns, and the significant impact of human activities, the scientific community is encountering new challenges. These challenges make sophisticated analytical methods necessary, particularly in sample analysis. The QuEChERS technique has remained a vital tool for processing food and environmental samples, with only slight improvements involving new extraction solvents and sorbent materials.

Recent developments have focused on broadening the method's applicability to a wider range of analytes and sample types. Key changes include the use of Deep Eutectic Solvents (DESs) and minor adjustments to the extraction solvents, as well as modifications in the types and amounts of salts, sample volumes, water addition, and agitation modes. Additionally, there have been advancements in the kinds and quantities of cleanup sorbents utilized, expanding beyond the official QuEChERS technique versions.

QuEChERS extracts have primarily been analyzed using chromatographic techniques. Looking forward, the diverse range of matrices and compounds, along with increasing sensitivity requirements, will drive advances in analytical techniques, likely leading to reduced consumption of solvents and sorbents. Automating the QuEChERS procedure remains essential for its continued development, with implementations like the QuEChERSER modification representing significant progress. Future studies might focus on enhancing the cleanup step to enable the QuEChERS process to recover hazardous heavy metals from various samples, expanding its use beyond pesticides and organic contaminants.

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