Sampling in combination with process knowledge as critical factors for the reliability and accuracy of laboratory testing

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In today's highly complex and demanding market of analytical services, it is of outmost importance that laboratories safeguard the reliability and accuracy of their results. Amongst many other important factors, our experience during the last three decades has demonstrated that a carefully planned sampling protocol is not by itself adequate in satisfying above requirement. It shall always be born in mind that making correct sampling decisions necessitates good knowledge of the actual process that is being monitored.

Data collected through our involvement in the commissioning and subsequent operation of large-scale projects (e.g. Seawater Reverse Osmosis Plants - SWRO's, sewage treatment plants and renal wards in healthcare establishments), will be presented. These will provide well justified real-life examples of how critical can process knowledge be in the design and monitoring of sampling activities. Particular reference will be made to the impact on the reliability and also interpretability of test results.

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Ensuring quality of the analytical process in a research laboratory

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Validation and verification of analytical methods for testing of new materials or new specific methods often pose a line of problems and require alternative approaches. The necessary analytical instrumentation, the available CRM or standard procedures are among the problems encountered. Firstly, in the research laboratory usually the equipment is tailored to the quantity and size of the samples tested. Often we need to scale down the procedure to lower weight or volume, which could alter the analytical behavior of the standard procedure used for CRM testing. Secondly, although we chose the CRM with characteristics as close as possible to the studied material, often an appropriate CRM is not commercially available. For example, analysing CRM of soils is usually a procedure for estimating the analytical behaviour of the applied method for testing mine tailings, however the discrepancy of the matrix composition or analytes content, as well as their state and speciation in the sample, doesn't allow the method to be fully studied. In some cases, standard addition approach could help for at least approximatively estimation of the uncertainty of measurement, but the attending the chemical equilibrium of added analyte with matrix to the level which reflects the conditions in the CRM sample or testing samples could be difficult to attend, especially in solid samples. Thirdly, when studying the behaviour of newly developed procedure for estimation of specific characteristics of the sample (for example, study of alkaline reactivity of mine tailing or fly ashes as precursors for geopolymer obtaining) the problems are even more pronounced. Applying the procedure to available CRM of soils or mine tailing doesn't provide the informative data as the reference or consensus values are not available for this specific procedure. An alternative approach was to study the behaviour of pure components and phases of the sample in the tested media. However, we found that we are scaling up the method and the obtained data could only roughly present the behaviour of the method in the tested materials. Fourthly, to ensure the quality control, CRM and in-lab control samples were used in our laboratory. However, we found that the differences in particle size in standard and testing samples highly influenced the obtained results and sometimes they appeared non-informative to be applied for quality control. In the research laboratory typically one analyses limited number different samples in different conditions applying modified procedures which limits the obtaining of reasonable quantity of data for constriction of useful control charts. Nevertheless the mentioned problems we are still applying the standard procedures to available CRM for initial estimation of the uncertainty of measurement and the method behaviour in the research laboratory. More through estimation needs development of specific protocols.

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Analytics for phosphorus recycling from sewage sludge and mineral fertilizers

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Phosphorus is an essential element for all organisms. Along with other essential elements such as nitrogen and potassium, phosphorus enables the growth of plants and is therefore widely used in fertilizers. However, phosphate rock is a finite resource which has been listed by the EU as a critical raw material [1]. As nowadays large quantities of phosphorus residues are lost in sewage sludge, several European countries have decided to recycle phosphorus from sewage sludge in the future. Various techniques have been proposed to extract phosphorus from sewage sludge and reuse it for the production of new fertilizers. These novel practices come with new analytical demands for the industry as well as for public laboratories.

Another challenge in the field of environmental analytics is the analysis of mineral fertilizers in Switzerland. The concentration of several heavy metals (As, Cd, Hg, Pb and U) are regulated for mineral fertilizers in Switzerland. However, the products are rarely controlled due to the lack of accredited laboratories in this field. The last campaign of mineral fertilizers analysis in 2021 has however shown that up to 46% of the tested products did not meet the requirements [2], which highlights the importance of more frequent measurement campaigns for mineral fertilizer.

The aim of this project is to establish the analytical methods and infrastructure for enabling analysis in the fields of phosphorus recycling and mineral fertilizers meeting the requirements of metrological traceability. As a first step we participated at a proficiency testing for the analysis of metals in sewage sludge. The production of a reference material such as sewage sludge or sewage sludge ash which could be used in the process of phosphorus recycling is part of this project. Additionally, we aim to establish an accredited method for mineral fertilizer analysis in Switzerland.

[1] Regulation of the European Parliament and of the Council: establishing a framework for ensuring a secure and sustainable supply of critical raw materials and amending Regulations (EU) 168/2013, (EU) 2018/858, 2018/1724 and (EU) 2019/1020 (2023).
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Equipment Qualification

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In the early 1990s it was not longer sufficient for the laboratories to do things right, they had to be documented. Many laboratories achieve this through formal quality systems, generally implemented in accordance with one or more of the major internationally recognised quality standards: ISO 9000 series, GMP, GLP and ISO Guide 25. However, these standards were intentionally very general, in order to be as widely applicable as possible. They set out general requirements, such as that equipment must be fit for purpose, properly maintained and calibrated with national or international reference materials, but did not specifically define what was required other than calibration or how this should be achieved.
It was not clear where and when formal Equipment Qualification is appropriate and how it should be documented. Specifically in the highly regulated pharmaceutical industry regulatory bodies increasingly turned their attention to this area. There was the usual danger of overreaction, fragmentation and new bureaucracy.
In order to establish a generally accepted position, guidelines have been developed by Eurachem-UK and LGC (under the VAM initiative) together with the Pharmaceutical Analytical Science Group (PASG). The working group has brought together a broad cross-section of equipment manufacturers, representatives of accreditation bodies and regulatory authorities, and users of analytical equipment [1, 2]. The guidance sets out an approach to EQ based on four stages of qualification; design qualification (DQ), installation qualification (IQ), operational qualification (OQ) and performance qualification (PQ).

This concept did not really gain a foothold in the ISO standards 9000ff and ISO/IEC 17025 and related standards until today.
In contrast, the concept was quickly adopted in the pharmaceutical industry. It was generally extended to all equipment, including production, infrastructure and logistics. EQ with the four xQ steps became an important supporting element in GMP [3,4].

After the turn of the millennium, processes became more important in practice compared to individual subsequent actions.
EQ was newly understood as a process starting with the idea of acquiring a device until its disposal. User requirements newly became a key document of EQ. The life cycle is closely linked to data integrity issues. This new concept is gaining great acceptance in GMP [5].

Eurachem is considering the development of a guideline on EQ.

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Pyramid of Quality

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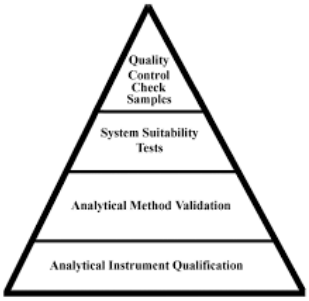
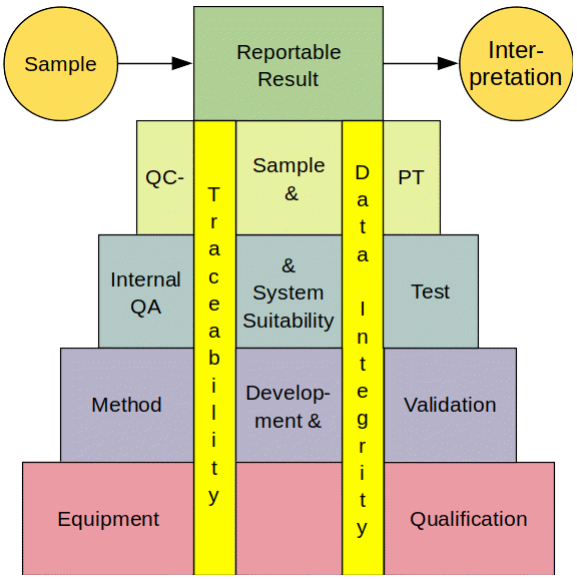
In an analytical target profile, an analytical problem with the boundary conditions is defined. The selection of the methodology, the approach and the parameters serve to achieve this target. Various principles of good analytical practice have been defined to assist the laboratory in achieving the target.
With the listing of principles it is difficult to show the dependencies between the individual maxims. Graphical representations are good for illustrating prerequisites and influences.



In USP <1058> Analytical Instrument Qualification, the triangle of data quality is defined. It shows as a basis the qualified equipment used to develop, validate, and perform an analytical method. Quality assurance is performed using System Suitability Tests and Quality Control Check Samples.
In the quality pyramid, additional aspects and their links are shown graphically. The pyramid covers the part of measurement within the analytical process.



Measurement uncertainty arising from sampling and analytical steps of dissolution test

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Dissolution is used to determine the rate and extent of drug release from the dosage form into a dissolution medium, which allow to assess the batch-to-batch variability. Considering that the dissolution test is used predict the vivo performance of the drug as well, it is important to guarantee the quality and reliability of dissolution test results. The aim of this work was to evaluate the measurement uncertainty arising from sampling and analytical steps of dissolution test of prednisone tablets. Dissolution test was performed using 900 mL of purified water as dissolution medium and a dissolution apparatus equipped with paddles rotating at 50 rpm for 30 minutes. Quantification was performed by UV spectrophotometer. Uncertainty arising from sampling was estimated using the duplicate method (empirical approach), using 17-sampling target, two samples for each sampling target, and three replicas for each sample, totalizing 102 analyses. Uncertainty arising from analytical steps considered the uncertainty from dissolution step (estimated using Monte Carlo method and regression equation obtained using DoE) and uncertainty from quantification step [1]. Overall uncertainty value was found to be 2.2%, which is below the target uncertainty value (u^t=2.5%). The contribution of uncertainty sources were uncertainty from sampling (24%), uncertainty from dissolution step (29%), and uncertainty from quantification step (47%). The results of dissolution test should be compared to the specification limits (Q). According to the pharmacopeia requirements, the batch of the medicine should be declared compliant if the dissolved amount of prednisone for six tablets are above the specification limits +5% (Q+5%=85%). Since the measured values for all six tablets (96.5%, 94.0%, 96,4%, 95.3%, 96.0%, and 96.9%) were above the multivariate acceptance limit (89.4%, calculate as the standard uncertainty multiplied by multivariate coverage factor) [2], the batch of the prednisone tablets was declared complaint, with a reduced total risk of false decision (total risk value below 5%).

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Total combined global risk assessment applied to pharmaceutical equivalence of medicines

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Pharmaceutical equivalence is an important step in determining the interchangeability between medicines, particularly when bioequivalence is not required or applicable. Pharmaceutical equivalence study ensures that test and reference medicines gave the same active ingredient, strength, dosage form, and quality, which indicate they are identical in terms of their chemical and physical properties. Usually, pharmaceutical equivalence is assessed using a single batch of generic medicine and a single batch of reference medicine. However, the regulatory agency may require ongoing monitoring of the generic medicine's quality to ensure that it continues to meet the standards set by the regulatory body. To the best of our knowledge, there is no work in the literature that allow one to assess the total combined risk of a pharmaceutical equivalence study not yet performed (e.g. what are the risk of a false pharmaceutical equivalence decision for two medicines – test and reference – that will be manufactured in the future?) [1]. This risk of false decision for a future batch that will be tested is defined as global risk, which is estimated using Bayesian statistics [2]. The aim of this work was to assess the total combined global risk of false decisions regarding the pharmaceutical equivalence (non-equivalence) of test and reference medicines. Test and reference medicines of ofloxacin ophthalmic solution were subjected to assay (HPLC), pH determination, potency (agar diffusion), identification (HPLC and TLC), volume, and sterility test. In addition, test and reference medicines of ofloxacin tablets were subjected to assay for ofloxacin content (HPLC), friability test, potency (agar diffusion microbiological assay), identification (HPLC and UV), hardness, appearance, disintegration test, uniformity of dosage unit test, and weight. Particular and total global risks were estimated using Monte Carlo method (MCM) implemented in a MS-Excel spreadsheet. The consumers' risk values were calculated as function of the historical mean for quantitative analysis (e.g. assay, pH, potency, volume, friability, etc.) or the probability of a compliant batch for qualitative analysis (e.g. identification, sterility test, appearance, etc.). For quantitative analysis, the risk values differ for tests with interval specification limits (e.g. assay, potency, etc.) when compared to those tests with minimum or maximum specification limits only (e.g. volume, friability test, hardness, etc.). For qualitative analysis, the higher the probability of a compliant batch the lower the consumers' risk values. The total combined global risk of a false pharmaceutical equivalence decision can be significantly high (above of 5%) for some of the simulated conditions. Reduced risk values were obtained when historical means were far from the specification limits and the probability of a compliant batch is high, or when the standard deviation values are low.

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Measurement uncertainty evaluation of microbial enumeration test for medicines

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Microbial quality is an important aspect that should be assessed to guarantee the efficacy and safety of medicines. Despite of all efforts to ensure the reliability of microbial enumeration test, there will always be some uncertainty associated with the measured value, particularly when is expected a reduced microbial load [1]. The aim this work was to evaluated measurement uncertainty of microbial enumeration test for medicines using a bottom-up approach. Ten different medicines were intentionally contaminated with three levels (10^3, 10^4, and 10^5 CFU/mL) of bacteria (*Staphylococcus aureus*, *Pseudomonas aeruginosa*, and *Escherichia coli*) and fungal (*Aspergillus brasiliensis* and *Candida albicans*). Aliquots of 10 mL of contaminated medicines was subjected to decimal serial dilutions (1:10, 1:100, and 1:1000) and aliquots of 1 mL of each dilution were transferred to Petri plates (three replica per dilution for each microorganism). Portions of 15-20 mL of tryptic soy agar (TSA) and Sabouraud dextrose agar (SDA) were placed into the plates for bacteria and fungal, respectively. Plates containing TSA were incubated at 30-35 ºC for 2-3 days, while plates containing SDA were incubated at 20-25 ºC for 5-7 days. Monte Carlo method and Poisson-lognormal regression [2] were used to assess how measurement uncertainty is related to the microbial load. Uncertainties from dilution factors, repeatability between plate microbial counts, and recovery of microbial counts in comparison to microbial counts of reference material were considered. Measurement uncertainty is important to guarantee the reliability and quality of enumeration test results and it should be taken into account in order to decide whether a medicine batch is compliant or non-compliant to the specification limits.

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Multivariate guard-bands applied on multiparameter evaluations of medicines

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Medicines are essential for maintaining good health and treating diseases and illnesses. The quality, efficacy, and safety of medicines are usually verified by analytical results of drug's identity, purity, strength, and composition. In this context, measurement uncertainty is a critical aspect for the quality and reliability of analytical results, as it can use to assess the risk of false conformity decisions that can have serious consequences. The pharmacopeial compendia usually adopt a simple acceptance rule, that does not that into account the information from measurement uncertainty. In this work, we compared the decision making using simple acceptance rule and decision rule with the use of guard-band (that takes into account the use of measurement uncertainty information) for multiparameter evaluation of ofloxacin ophthalmic solution and acyclovir topical cream. Ciprofloxacin ophthalmic solution medicines were subjected to volume measurements, pH determination, density determination, assay (HPLC), potency (agar diffusion) and drop test. Acyclovir topical cream samples were subject to weight measurements, bacterial and fungal enumeration tests, and assay (UV). Multivariate guard-band widths were calculated by multiplying the standard uncertainty (u) by an appropriate multivariate coverage factor (k') [1]. The multivariate coverage factor (k') was obtained by Monte Carlo method and Goal Seek tool implemented in a MS-Excel spreadsheet [2]. According to the simple acceptance rule (that do not take into account the measurement uncertainty information), all the results obtained for ciprofloxacin ophthalmic solution and acyclovir topical cream are within the specification limits. However, there is an increased risk of false conformity decisions for assay of ciprofloxacin and drop test. In other words, when considering a decision rule with the use of guard-bands (that takes into account the measurement uncertainty information), assay and drop test results are not compliant (are out of the acceptance limits obtained using guard-bands). Decisions made using simple acceptance rule and decision rules with the use of guard-band may differ. Therefore, the use of information of measurement uncertainty in conformity (non-conformity) assessment is highly recommended to ensure the proper efficacy, safety, and quality of medicines.

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[2] C.M. da Silva, F.R. Lourenço, *Journal of Pharmaceutical and Biomedical Analysis*, **2023**, *222*, 115080.

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[2] Nationale Marktkampagne Dünger 2019/2020: Schlussbericht. Wirschafts-, Energie- und Umweltdirektion Kanton Bern (2021).
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Quality Characteristics of Performance of the Determination of SARS-CoV-2 in wastewater. The case study of Athens.

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Wastewater-based Epidemiology (WBE) is a non-invasive and cost-effective chemical tool. During the pandemic caused by the SARS-CoV-2, the role of WBE is continuously upgraded and gains popularity as an early warning tool that provides additional information on the COVID-19 prevalence. Many scientists worldwide are monitoring the virus load in samples from the influents of Wastewater Treatment Plants (WWTPs) since the beginning of the pandemic. However, the determination of SARS-CoV-2 in wastewater remains a challenge even nowadays when virus has gone endemic. Assessment of bias between protocols, matrix effect and different sampling techniques (flow proportional, grab sample etc.) are the main problems of SARS-CoV-2 determination and hampers methods harmonization. On contrary, according to EU and WHO directives, monitoring of SARS-CoV-2 in wastewater is a trend analysis tool, while absolute quantification is not the purpose of WBE surveillance [1,2]. The aim of this study is to present the case of SARS-CoV-2 surveillance in wastewater from Attica region (Greece) and the applied approaches to ensure the quality in the whole measurement. Since August 2020, when surveillance has started, two different methods were used. A PEG-precipitation and a semi-automatic protocol based on membrane preconcentration were applied while Real Time-PCR was used as detection technique for both methods. Duplication between samples and PCR measurements was applied as an internal quality control tool for the measurement and dilution as a tool for assessment of inhibitors. Using the RANOVA3 software, ANOVA and Robust ANOVA a top-down evaluation of the uncertainty of both approaches was achieved. To obtain representative results, periods with both high and low wastewater loads were used for the uncertainty estimations. Moreover, uncertainty factor was calculated due to the logarithmic distribution of microbiological measurement. Finally, all the characteristics were taken into consideration while comparing protocols and evaluating fitness for purpose. It is the first time, that a SARS-CoV-2 surveillance method was used in a top-down approach.

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European Metrology Network for Pollution Monitoring POLMO

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EURAMET, the association of National Metrology Institutes (NMI) in Europe, has promoted in the last few years the creation of European Metrology Networks (EMNs) to analyse the European and global metrology needs and address these needs in a coordinated manner. EMN members will then formulate common metrology strategies including aspects such as research, infrastructure, knowledge transfer and services.

The EMN for Pollution Monitoring (POLMO) has been approved in May 2022 during the last General Assembly of EURAMET.

Robust metrology for monitoring pollution of air, water and soil, will be vital to achieving zero pollution and carbon neutrality, which are the ambitions set out in both the European Union and the United Nations strategies.

POLMO aims at creating a sustainable metrology infrastructure to support European directives and international regulations, targeting pollution monitoring, using smart specialisation to maximise outcomes from currently available research resources.

This EMN will initially focus on chemical, biological and radionuclide pollution in air, water, and soil, with a scope to expand its expertise to other pollutants such as light and noise in the future.

https://www.euramet.org/european-metrology-networks/pollution-monitoring

European Metrology Network for Safe and Sustainable Food

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The provision of safe, high-quality food is vital for human health, and innovation in the food sector will be needed to protect the environment, ensure sustainability, and respond to future needs.

EURAMET, the association of National Metrology Institutes (NMI) in Europe, approved in May 2022 the European Metrology Network (EMN) for Safe and Sustainable Food (EMN Food).

This network will promote a harmonised approach to ensure traceability in food measurements, matrix reference materials and calibration standards. This will allow National Metrology Institutes (NMIs) and Designated Institutes (DIs) across Europe to respond to stakeholder needs with confidence and quality. Furthermore, the EMN Food aims to assess current capabilities within the network, transfer knowledge between network members, support provisional developments and create a research hub for food safety.

The support to new business opportunities and innovation in the food safety field will increase the competitiveness of the market, ensuring sustainability and reducing environmental impact.

https://www.euramet.org/european-metrology-networks/safe-and-sustainable-food

VALIDATION OF A METHOD FOR ACID TREATMENT AND SUBSEQUENT DETERMINATION BY ICP-OES OF HEAVY METALS IN SOILS AND INDUSTRAIL TALINGS

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The most used precursors for producing of geopolymers are metakaolin, fly ash and granulated blast furnace slag. A large number of studies show that the mine tailings, whose utilization is an extremely important worldwide, are candidates for precursors for these new materials. The geochemical composition of wastes is crucible for the structure and physical properties of the newly synthesized material. The composition of mine wastes is not only with structural importance for geopolymerization process, but also from ecological importance, as a component of future wastes. The aqua regia digestion is not total digestion technique but is powerful method for digestion of all environmentally available elements with exception of that bounded in silicate structures, which are considered non-mobile in the environmental conditions. This method could be applied not only for fast assessment of the toxicity of mine tailings and the geopolymers on its base, but also for evaluation of the degree of encapsulation of hazardous materials.

In the present study, an open aqua regia digestion method of mine tailings and fly ashes followed by ICP-OES measurement was validated for determination of As, Cd, Cr, Cu, Pb, Ni and Zn. The selected elements are noted in the European Commission standards for non-agricultural soils. The limits of detection and quantification were determined. Emission lines free from spectral interference were selected. Accuracy, repeatability, reproducibility were evaluated. Results are reported with their expanded uncertainty. Applicability of the method was assessed by z-score.

Acknowledgment: This study is supported by the Bulgarian National Science Fund under the contract KP-06-DO02/5 "RecMine – Environmental footprint reduction through eco-friendly technologies of mine tailings recycling" in the frame of ERA-MIN3 program, Horizon Europe.

Objective assessment of the evolution of microplastic contamination in sediments from a vast coastal area

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The environmental pollution by microplastics is well recognized. Microplastics were already detected in various matrices from distinct environmental compartments worldwide, some from remote areas. Various methodologies and techniques have been used to determine microplastic in such matrices, for instance, sediment samples from the ocean bottom. In order to determine microplastics in a sediment matrix, the sample is typically sieved through a 5 mm mesh, digested to remove the organic matter and density separated to isolate microplastics from the denser part of the sediment [1]. The physical analysis of microplastic consists of visual analysis under a stereomicroscope to determine particle size, colour, and shape. The chemical analysis is performed by an infrared spectrometer coupled to a microscope (micro-FTIR), allowing the identification of the chemical composition of microplastic, i.e., the type of polymer.

Creating policies and legislation to control and manage (micro)plastic pollution is essential to protect the environment, namely the coastal areas. The developed regulation must be supported by the known relevance and trends of the pollution type.

This work discusses the assessment of contamination trends of a 700 km^2 oceanic area affected by contamination heterogeneity, sampling representativeness and the uncertainty of the analysis of collected samples [2]. The methodology developed consists of objectively identifying meaningful variations of microplastic contamination by the Monte Carlo simulation of all uncertainty sources. This work allowed to unequivocally conclude that the contamination level of the studied area did not vary significantly between two consecutive years (2018 and 2019) and that PET microplastics are the major type of polymer. The comparison of contamination levels was performed for a 99% confidence level. The collected information on the environmental area is crucial for the objective and binding determination of microplastic contamination relevance.

[1] V. Morgado, L. Gomes, R.J.N.B. Silva, C. Palma, *Sci. Total Environ.*, **2022**, *832*, 155053.
[2] V. Morgado, C. Palma, R.J.N.B. Silva, *Environ. Sci. Technol.*, **2022**, *56*, 11080-11090.