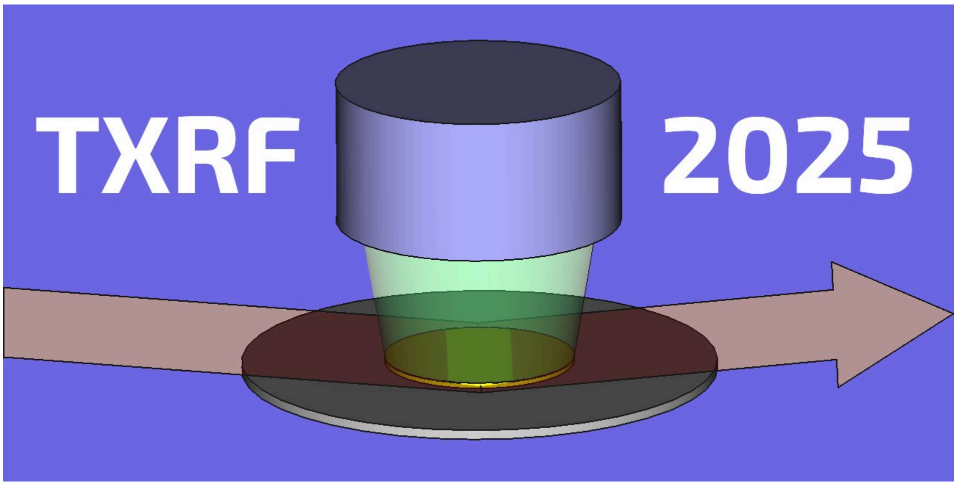

20th International Conference on Total Reflection X-ray Fluorescence Analysis and Related Methods

September 9-12, 2025
Jan Kochanowski University, Kielce, Poland

Program and book of abstracts

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Monday, 8th September 2025

18:00	Registration and Welcome Reception (co-sponsored by Bruker)	

Tuesday, 9th September 2025

08:00	Registration	

TXRF History/Biomedical applications
(Chairperson: Maria Luisa Carvalho)

09:00	TXRF-2025 organizers	TXRF 2025 OPENING SESSION
09:15	Joanna Chwiej (Invited)	“FROM BULK TO SPATIAL INSIGHT: TXRF AND MICRO-XRF IN BIOMEDICAL INVESTIGATIONS”
10:00	Ramon Fernández-Ruiz	“A BRIEF HISTORY OF TXRF TECHNOLOGICAL EVOLUTION”
10:25	Ilona Stabrawa	“ANALYSIS OF ELEMENT CONCENTRATIONS IN THE BLOOD SERUM OF PATIENTS WITH RHEUMATOID ARTHRITIS (RA)”
10:45	Coffee Break	
11:15	Christina Streli	“TWENTY EDITIONS OF THE TXRF 2025 CONFERENCE”
11:45	Aleksandra Wilk	“TXRF AND ICP-OES ANALYSIS OF MAGNETITE AND MAGHEMITE NANOPARTICLE BEHAVIOUR *IN VIVO”*
12:10	Jasna Jablan	“EVALUATION OF BLOOD COLLECTION TUBE MATERIAL IMPACT ON TRACE ELEMENT QUANTIFICATION IN SERUM USING TXRF”
12:35	Milena Piotrowska	“STUDY OF ELEMENT CONCENTRATONS IN BLOOD SERUM OF PATIENTS RECEIVING PARENTERAL NUTRITION USING TXRF”
13:00	Conference Photo/Lunch	

Biological and environmental applications (Chairperson: Diane Eichert)

14:15	Hagen Stosnach	“POTATO PETIOLE ANALYSIS BY MEANS OF TXRF IN CONTRAST TO ICP-OES ANALYSIS”
14:40	Nand Lal Mishra	“ELEMENTAL DETERMINATIONS IN WATER SAMPLES OF MAHESHARA LAKE, GORAKHPUR, INDIA BY TOTAL REFLECTION X-RAY FLUORESCENCE”
15:05	Sebastian Hauser	“GREEN ANALYTICAL STRATEGIES FOR TRACE ELEMENT QUANTIFICATION IN BIOLOGICAL SAMPLES USING TXRF AND NANOLITER DEPOSITION TECHNIQUES”
15:30	Coffee Break	
16:00	Tsugufumi Matsuyama	“APPLICATION OF FREEZE CONCENTRATION TECHNIQUE FOR IMPROVING SENSITIVITY OF TXRF ANALYSIS”
16:30	János Osán	“TXRF ANALYSIS FOR ELEMENTAL COMPOSITION AND METAL UPTAKE OF CLAYS FOR RADIOACTIVE WASTE REPOSITORY”
17:15	**Poster session/Lab visit**	

FROM BULK TO SPATIAL INSIGHT: TXRF AND MICRO-XRF IN BIOMEDICAL INVESTIGATIONS

Joanna Chwiej[1], Karolina Olbrich[1], Marzena Rugiel[1], Kamil Kawon[1], Aleksandra Wilk[1], Aldona Kubala-Kukus[2,3], Agnieszka Drozdz[1], Katarzyna Matusiak[1], Zuzanna Setkowicz[4]

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The total reflection X-ray fluorescence (TXRF) is a powerful tool of quantitative elemental analysis, offering high sensitivity, minimal sample preparation, and the ability to measure its very small volume. The capacity of TXRF for simultaneous multi-element detection with good precision makes it especially suited for studying trace elements in biological samples such as tissues, body fluids, and cell cultures. Therefore, the method is more and more frequently used in primary research focused on the identification of various physio- and pathological processes, in disease diagnostics, metabolic profiling, and pharmaceutical development.

Many of such investigations are carried out based on animal models being an essential component of biomedical science. Offering invaluable insights into complex physiological processes, the progression of various diseases, enabling the development and testing of treatments in a controlled, ethically regulated environment, the experimental animals serve as a critical bridge between basic research and clinical applications.

Elemental analysis of animal tissues with TXRF method has, however, some limitations including the difficulties with the quantification of lower-Z elements or the lack of spatial information on the distributions of the elements within examined tissues. The answer to these problems can be the use of additional, supplementary X-ray fluorescence (XRF) based technique, namely, synchrotron XRF microscopy.

During the talk, the utility of both methods to resolve of various biomedical problems will be compared. The presented examples will include the elemental anomalies of brain and other tissues, and body fluids associated with: the glioma development [1,2], the influence of ketogenic diet on brain postnatal development [3,4] and glial scar formation as well as the metabolism and fate of iron oxide nanoparticles [5,6,7].

Acknowledgements

This work was partially financed by the funds granted to the AGH University of Krakow in the frame of the “Excellence Initiative – Research University” project (Action 4: A system of university grants for research carried out with the participation of doctoral students and young scientists, PL-Joanna Chwiej).

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A BRIEF HISTORY OF TXRF TECHNOLOGICAL EVOLUTION

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Total Reflection X-Ray Fluorescence (TXRF) spectroscopy has undergone significant evolution since its theoretical foundations were established in the 1920s and its practical development began in the 1970s. Early discovered by Compton in 1923 [1], it was rediscovered approximately fifty years later by Yoneda and Horiuchi in 1971 [2], who proposed the ingenious idea of using total reflection of X-ray phenomena over a flat reflector surface for analytical X-ray fluorescence analysis. Later, the conceptual and practical development of Aiginger and Wobrauschek [3] during the 1970s, and Knoth and Schwenke [4] during the 1980s, established the technical and methodological basis of TXRF as a powerful analytical technique in trace element detection. A first commercially available TXRF device was presented by Seifert & Co., Ahrensburg, Germany, in 1980. Nearly at the same time, a simple module built by Wobrauschek and distributed by the IAEA (International Atomic Energy Agency), Vienna, Austria, became available.

The first International TXRF Conference was held in 1986, in Geesthacht, Germany. From this moment, we can consider that TXRF spectrometry was born as we know it today. From this event to the present, technological advancements have dramatically improved its sensitivity, resolution, and applicability. *Detectors:* The transition from proportional counters to Si(Li) detectors, accompanied by the use of liquid nitrogen, or more recently to actual SDD detectors, is an illustration of their improvement. *Optics:* The ability to define the energy band and geometry of the incident beam over the reflector with high brightness is another fundamental improvement resulting from new optics developments during this period, including high-reflectivity single or double multi-layer monochromators with curved optics. *Sources:* The transition from the classical Seifert-type glass tube, equipped with a weighted transformer and toxic oil baths, weighing several hundred kilograms, to the actual electronic cards associated with present high-brightness micro X-ray sources, which achieve the same typical 50 kV of excitation, is also a representative example of its technological evolution.

This work highlights key milestones and cutting-edge innovations that continue to position TXRF as a vital tool in ultra-trace elemental analysis.

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ANALYSIS OF ELEMENT CONCENTRATIONS IN THE BLOOD SERUM OF PATIENTS WITH RHEUMATOID ARTHRITIS (RA)

Ilona Stabrawa[1,2,*], Paulina Żarnowiec[3], Aleksandra Krzemień[1], Piotr Terlecki[1], Aldona Kubala-Kukuś[1,2], Dariusz Banaś[1,2], Jolanta Wudarczyk-Moćko[2], Karol Szary[1,2], Grzegorz Wesołowski[1], Katarzyna Durlik-Popińska[3], Natalia Jagiełło[1], Oliwia Soból[1]

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Rheumatoid arthritis (RA) is a chronic disease of the musculoskeletal system that leads to the destruction of cartilage and periarticular structures. Although RA is an autoimmune disease, it can also be related to mineral imbalances in the body. The aim of this study was to analyze the concentrations of selected elements in the blood serum of patients with RA and to compare them with those of a healthy control group [1].

Blood serum samples were collected from a total of 130 individuals: 30 healthy subjects and 100 patients diagnosed with rheumatoid arthritis. Elemental concentrations were determined using total reflection X-ray fluorescence (TXRF) spectroscopy. This technique enables the simultaneous detection of macroelements (e.g., Ca, K, S, P) and trace elements (e.g., Fe, Zn, Cu, Mn, Cr, Se, Pb) in small sample volumes. Measurements were performed using a Picofox S2 TXRF spectrometer at the Institute of Physics, Jan Kochanowski University in Kielce.

The obtained data were statistically analyzed to identify correlations between element concentrations and disease status. Additionally, clinical parameters (such as age and disease duration) and serological markers (including RF IgA, RF IgM, RF IgG, CCP, ANA, and anti-CarP antibodies) were analyzed for intercorrelations, allowing for a broader perspective on the biochemical profile of RA patients. The obtained results may contribute to the identification of diagnostic and prognostic biomarkers, thus supporting the development of personalized medicine in the treatment of joint diseases.

Acknowledgements

This work was co-financed by the Minister of Science (Poland) under the "Regional Excellence Initiative" program (project no.: RID/SP/0015/2024/01).

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TWENTY EDITIONS OF THE TXRF 2025 CONFERENCE

Ch. Streli[1,2]*, P. Wobrauschek[1,2], A. Kubala-Kukuś[3,4]

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In the year 1923 Arthur H. Compton described for the first time total reflection of X-rays under grazing incidence of the primary beam [1, 2]. Nearly fifty years later, in 1971, Yoneda and Horiuchi discovered the possibility to use the phenomenon of external total reflection of X-rays at a flat reflector surface for spectra analyses [3]. The promising idea was abandoned and was re-evaluated in 1974 by Aiginger and Wobrauschek in Austria and early 1980th by Knoth and Schwenke in Germany. Few papers were published until 1986. They deal with theoretical estimation [4] and the first spectrometer prototype and its features [5]. A first commercially available device was presented by Seifert & Co., Ahrensburg, Germany in 1980 and nearly at the same time a simple module built by Wobrauschek and distributed by the IAEA (International Atomic Energy Agency) Vienna, Austria were available. Nearly 50 modules, as non-profit systems for academic use only, has been installed in developing countries around the whole world under the auspices of the IAEA. The Institute of Physics of Jan Kochanowski University, the organizers of TXRF 2025 conference, started work with the TXRF technique based on Wobrauschek's module.

After long years of experimental work on instrumental development, the first International Conference on Total Reflection X-ray fluorescence Analysis and Related Methods (also known as TXRF conference) was held in 1986, in Geesthacht (near Hamburg), Germany. Subsequently, eighteen more conferences have been organized in different countries on a biannual basis. The biannual TXRF conference is a forum for experts and users of TXRF and related techniques. The scope is to jointly present and discuss recent advances, latest research, and new perspectives in different fields of TXRF from the fundamentals to the applications.

Notable international conferences on TXRF have been held in various locations, including Geesthacht, Germany (1986, 1992); Dortmund, Germany (1988, 1996, 2011); Vienna, Austria (1990, 2000); Tsukuba, Japan (1994); Eindhoven, The Netherlands (1996); Austin, TX, USA (1998); Madeira, Portugal (2002); Awaji Island, Japan (2003); Budapest, Hungary (2005); Trento, Italy (2007); Gothenburg, Sweden (2009); Osaka, Japan (2013); Denver, CO, USA (2015); Brescia, Italy (2017); Girona, Spain (2019); Clausthal, Germany (2023); and Kielce, Poland (2025) [6].

Presented work summarizes the twenty editions of the TXRF conference.

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TXRF AND ICP-OES ANALYSIS OF MAGNETITE AND MAGHEMITE NANOPARTICLE BEHAVIOUR *IN VIVO*

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Superparamagnetic iron oxide nanoparticles (SPIONs), due to their unique physicochemical properties, have great potential for applications in several branches of medicine. Among others, they may be used as contrast agents for magnetic resonance imaging and in cancer therapy based on the local hyperthermia [1]. The features of SPIONs, such as the nanometre size, the high surface area to volume ratio provide also great opportunities to modify the particles for specific technological solutions and new implementations. On the other hand, SPIONs could induce possible cytotoxic effects to by mechanical damages or by disturbing the homeostasis of chemical processes occurring in cells.

The properties of SPIONs can be modified with various coatings and ligands to suit its purpose, while their most commonly used core material is magnetite - containing iron atoms on two oxidation states: +2 and +3. An alternative to magnetite is maghemite being its oxidized form and containing iron atoms only on the +3 oxidation level [2]. The differences in oxidation levels, thermic stability, and the source of magnetic moments between magnetite and maghemite can influence the biokinetics an distribution of SPIONs in the human body.

To achieve the purposes of this study 14 groups of Wistar rats (6 animals each) were examined. These were male and female rats treated with the PEG coated SPIONs with 10 nm magnetite or maghemite core, from which the organs were taken 2 and 24 hours, and 7 days from the injection of nanoparticles. Additionally, naive norms were investigated. The solutions of SPIONs were administered to rats at a dose of 0,5 mg Fe per kg of body mass, which is an equivalent of doses of SPIONs used in humans as contrast agents. After appropriate time from nanoparticles administration, the animals were sacrificed, and their tissues were taken and frozen in liquid nitrogen. After microwave-assisted digestion in HNO_3 the samples were enriched with Ga internal standard and measured via TXRF spectroscopy using Rigaku Nanohunter II spectrometer. For the purpose of more precise lower-Z elements analysis, the ICP-OES method and Agilent 5100 ICP-OES spectrometer were employed.

Analysis of concentrations of P, S, K, Ca obtained with ICP-OES, and Fe, Cu, Zn and Se determined with TXRF revealed differences in the accumulation of examined elements between both sexes as well as in the biokinetics, fate and effects of SPIONs with various cores. Even though this study has preliminary character, its results point to oxidative stress and/or increased immune system activity occurring in the response to the examined nanoparticles action.

Acknowledgements

This work was partially financed by the funds granted to the AGH University of Krakow in the frame of the “Excellence Initiative – Research University” project (Action 4: A system of university grants for research carried out with the participation of doctoral students and young scientists, PL-Joanna Chwiej).

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EVALUATION OF BLOOD COLLECTION TUBE MATERIAL IMPACT ON TRACE ELEMENT QUANTIFICATION IN SERUM USING TXRF

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The increasing recognition of the essential role that trace and ultratrace elements play in human metabolism — in addition to their potential toxic effects — has increased the clinical demand for accurate and sensitive analytical methods. As instrumental techniques reach ever lower detection limits, concern about contamination during sample collection and handling has become increasingly important. According to the Clinical and Laboratory Standards Institute (CLSI) guidelines [1], contamination can occur throughout the preanalytical process, with sampling materials and storage containers playing an important role.

The aim of this study was to evaluate the effects of different blood collection tubes on trace element concentrations measured in serum by comparing standard tubes with a red cap with certified trace element-free BD Vacutainer® tubes (dark blue cap).Blood sampling was performed on 20 adult volunteers who gave informed consent and was approved by the Ethics Committee of the Dubrava University Hospital (reference: 2025/0128-5). Blood samples were centrifuged at 2,000×g for 10 minutes to obtain serum within 2 hours of blood collection. All serum samples were aliquoted and stored at −20 °C in Eppendorf tubes until analysis. The serum samples were analysed undiluted and diluted 1:1 with ultrapure water to check the influence of the serum matrix. Element concentrations were determined using total reflection X-ray fluorescence spectrometry (TXRF), a powerful multi-element method that is particularly suitable for clinical applications due to the small sample volume required, minimal sample preparation and the ability to detect trace elements at low concentrations. TXRF is considered an environmentally friendly analytical technique as it significantly reduces the use of reagents, waste generation and energy consumption compared to conventional wet chemical methods and other spectroscopic techniques. This is in line with the principles of green analytical chemistry and makes TXRF an environmentally friendly choice for routine monitoring of trace elements [2]. The results show that the type of sampling tube has a significant impact on the trace element content of clinical samples, highlighting the need for standardised pre-analytical protocols. Furthermore, this study underlines the suitability of TXRF as an environmentally friendly, cost-effective and sensitive method for routine trace element analysis in biological samples.

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STUDY OF ELEMENT CONCENTRATIONS IN BLOOD SERUM OF PATIENTS RECEIVING PARENTERAL NUTRITION USING TXRF

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Parenteral nutrition is a medical procedure aimed at maintaining or improving the nutritional status of patients who, due to their health condition, are unable to meet their nutritional needs through natural oral intake. Clinical nutrition is a multi-component process that includes: assessment of the patient's nutritional status, evaluation of the patient's nutritional requirements, provision of the an appropriate daily amount of macronutrients, micronutrients, fluids, and ongoing monitoring of the therapy [1]. In the context of the complexity of the parenteral nutrition process, it is important to monitor the patient's condition. The assessment of the content of elements in the patient's body can be performed on the basis of the analysis of the elemental composition of the patient's serum. For this purpose, the total reflection X-ray fluorescence method (TXRF) [2] can be applied, which is a powerful analytical technique widely utilized for elemental analysis in biological samples, including human serum.

Presented studies concentrate on the elemental analysis of human serum samples of 48 patients receiving parenteral nutrition using TXRF technique. The serum elemental analysis was performed two times: on the first day of parenteral nutritional treatment (measurement I) and seventh day after starting (measurement II). In the studies, also serum samples for the control group (50 persons) were analyzed, which allowed for comparison with the values obtained for the studied group. The TXRF analysis of the serum elemental composition was, for both groups, also supported by anthropometric, biochemical and immunological studies. During the TXRF study, the following elements were determined in the human serum samples of studied and control groups: P, S, Cl, K, Ca, Cr, Mn, Fe, Ni, Cu, Zn, Se, Br, Rb, Sr and Pb. On the first stage of the studies, the changes in concentration of Cr and Se (supplemented in studied group) and Br were discussed [3]. Next, the analysis of censored data, observed in the case of Mn, Ni, Rb, Sr and Pb, were performed [4].

The current analysis focuses on the examination of changes in other elements determined in serum. In the studies, the values of descriptive statistics parameters were calculated and the types of concentration distributions in serum were verified. Next, the contents of elements were compared between measurement I and II for studied group) and between the studied and control groups. Correlations between the content of elements were discussed. An additional aspect of the analysis is the verification of the dependence of the content of elements on anthropometric, biochemical and immunological parameters.

This work was co-financed by the Minister of Science (Poland) under the "Regional Excellence Initiative" program (project no.: RID/SP/0015/2024/01).

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POTATO PETIOLE ANALYSIS BY MEANS OF TXRF IN CONTRAST TO ICP-OES ANALYSIS

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Several factors put an increased pressure on the development of new seeding potato varieties. The change of global climate demands new potato varieties, which can withstand draught and higher temperatures.

A second pressure factor is the global transformation of the fertilizer sector. With the goal of alleviating fertilizer supply shortages, the United States has launched a “Global Fertilizer Challenge”, which the EU has joined. Brazil has launched a national fertilizer strategy, while Russia is seeking to strengthen its influence in Africa through the donation of fertilizers [1]

In the research facilities of seeding potato producers new varieties are tested under various climate and fertilizing conditions. Of course, the results can be assessed by the final harvest yield and the state of the complete plant. But for a more accurate measurement of the general well-being and the available nutrient levels within the potato plant, the analysis of the petiole is applied.

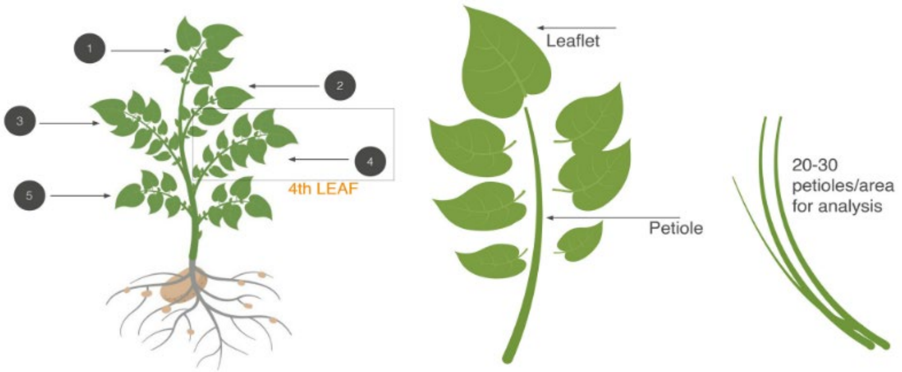

Figure 1: The usual procedure is to collect the youngest mature potato leaf from each of 20 to 30 plants, using an ordered pattern across the crop area, then remove the leaflets, retaining only the petioles for analysis [2]

The petiole collection procedure is described in figure 1. After collection, the petioles re frozen and later defrosted. After weighing the petiole juice was pressed out applying a texture analyzer. The juice was subsequently filtered using a syringe filter for the chemical analysis.

In this presentation the analysis of 45 samples, measured by ICP-OES and TXRF, are compared and evaluated. The results show that ICP-OES has its strength in the analysis of the macronutrients including Na and Mg while for the analysis of micronutrients like Zn and Cu the strong demanded dilution of the samples caused issues with the detection limits.

For TXRF analysis the common restrictions for the light elements B and Na were observed. The light macronutrients could be analyzed after adjusting the calibration values to compensate sample thickness effects. The micronutrients Mn, Fe, Cu, Zn and Ni could easily be analyzed.

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ELEMENTAL DETERMINATIONS IN WATER SAMPLES OF MAHESHARA LAKE, GORAKHPUR, INDIA BY TOTAL REFLECTION X-RAY FLUORESCENCE

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A knowledge of elements present in water samples in large water bodies is necessary for assessment of pollution in these water bodies and possible application of the water for sustainable development [1]. Maheshara lake is a large lake in the north side of Gorakhpur City, India which is a fast-developing city as an attractive tourist spot, growing industrial city and a transport hub having large scale movement of rail, road and air transport. The surrounding areas of the Gorakhpur city have very fertile land producing wheat, paddy, vegetables various fruits e.g. Mangoes, Water melon, muskmelon, potato, green vegetable etc. with the help of surrounding water bodies e.g. Maheshara lake, Ramgarh lake and Rapti river. Large number of tourists come to the city to visit the famous Gorakhnath temple, Gita Press, Ramgarh lake, nearby Kushinagar, a famous Buddhist tourist place, and while transiting to Nepal. The growth of Gorakhpur City and adjoining areas shall require large amount of water in future and the assessment of water quality in the surrounding water bodies may be helpful for its use for the city development. Total Reflection X-ray Fluorescence (TXRF) analysis of such samples can be beneficially utilized for such study [2-3]. The present work was done with this aim.

Water samples from Maheshara lake were collected in the rainy season in the month of August 2024 and analyzed using a low Z - High Z TXRF spectrometer. The TXRF spectra for low Z elements were measured using Cr Kα (5.41 keV) excitation source and vacuum sample chamber whereas medium and high Z elements were measured using Rh Kα (20.22 keV) source and ambient atmosphere sample chamber. The spectra were processed using EDXRF32 and PyMCA programs. The amounts of different elements present were determined using TXRF spectra and concentration of Ti and Ga internal standards. The TXRF spectra of the samples did not show presence of any harmful elements except F. The elements detected in the spectra of water samples are F, Na, Mg, Al, Si, S, Cl, K, Ca, Fe, Ni, Cu, Zn Rb, Sr etc. Some water samples collected in different months were also analyzed for trace elements to study the seasonal variation in the trace element concentrations.

The study indicates that trace elements present in the Maheshara lake water are well within the specified limits in drinking water prescribed by Bureau of Indian Standards (BIS). Also, the quality of Maheshara lake water is similar to underground water used for drinking. It was found that concentration of trace elements generally increases with time elapsed after rainy season probably due to concentration of trace elements as a result of evaporation of water, slow dissolution and leaching of elements from soil as well as due to mixing of some waste water in it. This study shall be useful in assessing possible usage of Maheshara lake water for drinking, irrigation, cattle feeding, washing etc. Further studies on other water bodies around Gorakhpur city shall be another step towards future sustainable development of the area.

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GREEN ANALYTICAL STRATEGIES FOR TRACE ELEMENT QUANTIFICATION IN BIOLOGICAL SAMPLES USING TXRF AND NANOLITER DEPOSITION TECHNIQUES

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Green Analytical Chemistry, i.e. the assessment and consideration of environmental, energy and safety aspects, is increasingly becoming part of the overall evaluation of analytical methods [1]. This has led to a new focus on the development and research of improved analytical methods that are more sustainable or 'greener'. Simplified sample preparation such as suspensions and partial digestions for trace element determination by total reflection X-ray fluorescence (TXRF) in various biological samples have been published previously [2,3]. However, matrix- or salt-rich samples such as culture media can affect performance and therefore complicate sample preparation. In this case, sample deposition by nanoliter dispensers can reduce the formation of salt crusts compared to conventionally prepared sample carriers and can therefore help to make sample preparation easier and greener [4].

The aim of this study was the evaluation of different sample preparation procedures for the reliable and green determination of trace elements in biological samples. First, trace elements were analyzed in a series of real-world samples, i.e., mouse spleen cells, mice bone and *Haloferax volcanii* H119 using newly developed TXRF methods by means of different suspension agents. These were validated by recoveries of selected elements in biological reference materials and solid-sampling high-resolution continuum source graphite furnace atomic absorption spectrometry (SS-HR-CS-GFAAS). Second, conventionally prepared sample carriers were compared with sample carriers prepared using a nanoliter dispenser (M2 Automation, Berlin, Germany). For this purpose, low nanoliters of prepared suspensions of real-world samples as well as biological reference materials were applied in a grid pattern and the resulting analytical figures were compared to sample carriers with a manually pipetted spot of the same total volume. In addition, the greenness of the analytical procedure and sample preparation method was evaluated using the Analytical Greenness Calculator (AGREE) and the Analytical Greenness Metric for sample preparation (AGREEprep). The greenness evaluation showed a better result for TXRF measurement compared to GFAAS and a better AGREEprep score for suspension in diluted acid compared to concentrated nitric acid.

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APPLICATION OF FREEZE CONCENTRATION TECHNIQUE FOR IMPROVING SENSITIVITY OF TXRF ANALYSIS

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It is important to measure trace elements in drinking water. Standard values of metal elements in drinking water are determined by World Health Organization (WHO). Total reflection X-ray fluorescence (TXRF) analysis is useful technique for determining trace elements in various aqueous samples1). In this method, a sample solution was dropped onto a substrate, and then dried. Incident X-rays at very low glancing angle were irradiated to the dried residue. Emitted fluorescent X-rays were observed at a detector. In this study, to improve the sensitivity of TXRF analysis, freeze concentration technique was employed. When water molecules crystalize, a high concentration area of impurity elements is present. Therefore, we considered that the sensitivity of TXRF analysis will be improved by applying freeze concentration technique to the sample preparation method.

For evaluating the proposed method, portable TXRF device 200FA (Ourstex Co. Japan) was used2). The target of the X-ray tube is W, and the silicon drift detector is equipped for observing fluorescent X-rays. Cr was selected as a target element, because the standard value of Cr in drinking water is determined by WHO. Test samples contained with Cr etc. were placed on a home freezer. After the test sample was frozen, it was moved from the home freezer. And we collected the exuded solution. Exuded test sample of 5 μL was mixed with an internal reference solution (15 μL), and then an aliquot of mixing solution (10 μL) was dropped onto a quartz glass. When TXRF analyses of exuded solutions were performed, the tube voltage and current of the X-ray tube were operated at 40 kV and 0.2 mA, respectively. The measurement time was set to 600 s. By applying freeze concentration technique, a net intensity of Cr Kα peak was improved. In this presentation, we will discuss the effect of impurity elements and recovery etc.

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TXRF ANALYSIS FOR ELEMENTAL COMPOSITION AND METAL UPTAKE OF CLAYS FOR RADIOACTIVE WASTE REPOSITORY

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In Europe, the final disposal of high-level radioactive waste (HLW) is planned in deep geological repositories. Their long-term safety is guaranteed by natural and engineered barriers, preventing radionuclides from entering the biosphere. An important element of the engineered barriers is the backfill material surrounding the waste packages. Due to their beneficial physico-chemical properties and high cation exchange capacity, bentonites are considered as a potential backfill material in several waste storage concepts. As the natural barrier, Boda Claystone Formation (BCF) has been selected for a potential host rock of the HLW repository in Hungary. Several of the safety approaches for the geological disposal of HLW rely on the strong uptake of radio-contaminants by clay minerals. Divalent transition metals Ni^{2+} and Co^{2+} were selected to model cationic activation and corrosion products.

Metal uptake of bentonites and clay-rich rocks was studied by recording sorption and desorption isotherms on powdered samples suspended in synthetic porewater with Ni^{2+} and Co^{2+} ions added at different concentrations. Radionuclide retention of B75 bentonite (Czechia) and BCF albitic claystone was evaluated. TXRF was applied for elemental analysis of both the solid and liquid phases. The powdered clays were analyzed in the suspension form using powdered geological standard reference materials for validation. For analysis of the liquid phases, the calibration samples were prepared in synthetic porewater for matrix matching.

The elemental composition of bentonite and albitic claystone samples obtained by suspension TXRF was in line with conventional XRF results of pelletized samples. For the liquid phase, radiotracers are usually applied in order to cover a large concentration range. The high-concentration part (0.01-10 mM) of the isotherms for Co^{2+} and Ni^{2+} could be recorded by means of TXRF using inactive chemicals. Although the porewater contains significant amount of salt (0.02 M NaCl), a reliable calibration could be obtained for TXRF for the elements of interest. The obtained results are consistent with ICP-OES results and sorption isotherms based on batch experiments involving radiotracers.

Comparison of the obtained sorption and desorption isotherms indicate irreversibility of sorption for both Ni^{2+} and Co^{2+} ions at the highest concentrations. This behaviour was supported by modelling considering an 80% Ca-montmorillonite content. The experimental isotherm was underestimated for high concentrations indicating and additional uptake mechanism through formation of a new phase. Based on the results, the B75 bentonite binds Ni^{2+} and Co^{2+} ions to a similar extent as the BCF albitic claystone, contributing to the retention of cationic radionuclides [1,2].

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Wednesday, 10th September 2025

08:30	Registration	
Environmental and industrial applications (Chairperson: Peter Wobrauschek)		
09:00	Eva Marguí (Invited)	“TXRF-BASED METHODS FOR THE ANALYSIS OF WASTE PRODUCTS FROM FOSSIL-FUELED POWER PLANTS”
09:45	Giacomo Siviero	“ELEMENTAL ANALYSIS OF CRITICAL RAW MATERIALS IN LITHIUM-ION BATTERY BLACK MASS: A TXRF-BASED APPROACH”
10:15	Tobias Schwank	“TRACE ANALYSIS OF COPPER, ZINC, LEAD, MANGANESE, CHROMIUM, COBALT AND NICKEL IN WORKPLACE AIR USING SUSPENSION-ASSISTED TXRF”
10:45	Coffee Break	
Particulate Matter (Chairperson: Peter Wobrauschek)		
11:15	Laura Borgese (Invited)	“ELEMENTAL ANALYSIS OF PARTICULATE MATTER: PRELIMINARY RESULTS FROM THE PROJECT "SMART-AIR"– SUSTAINABLE METHODS IN AIR RESEARCH QUALITY”
12:00	Armin Gross	“AN INNOVATIVE APPROACH MEASURING METAL CONCENTRATIONS IN AIRBORNE PARTICULATE MATTER”
12:30	Diane Eichert	“X-RAY FLUORESCENCE-BASED STUDIES OF PARTICULATE MATTER ON FILTERS”
13:00	Lunch Break	
14:15	Excursion	

TXRF-BASED METHODS FOR THE ANALYSIS OF WASTE PRODUCTS FROM FOSSIL-FUELED POWER PLANTS

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There is considerable interest in monitoring selenium (Se) and other metals in wastewater treatment processes and effluents from the power generation sector, such as flue gas desulfurization (FGD), for effective management and compliance with environmental regulations. Traditionally, this analysis has been done only in a laboratory environment using standard test methods such as inductively coupled plasma mass spectrometry (ICP-MS). However, there is an industry-wide need for a fast, in-situ analytical method suitable for process monitoring that could replace or significantly reduce the 2-week turnaround time associated with sending samples to third-party labs for ICP-MS analysis. Total reflection X-ray fluorescence spectrometry (TXRF) is a well-established analytical technique for trace multielement determination in various types of liquid and solid samples, offering a faster turnaround time that is of great interest to the industry. The use of this technique for Se and other metals determination in natural waters, drinking water, soils and industrial waste effluents has increased in popularity in recent years due to improvements in spectral sensitivity leading to the achievement of limits of detection at the ppb level and increased versatility that allows performing analysis onsite or near site [1-2].

In the present study, a comprehensive suite of analytical methodologies based on TXRF were developed for the determination of Se and other elements in a range of liquid and solid samples from fossil-fueled power plants, including FGD wastewater (FGDWW) and coal fly ash. The results indicated that, for most types of FGDWW samples – including leachates and impacted groundwater – trace levels of Se can be determined directly by depositing a few microliters of an internally standardized sample onto a reflective carrier. However, more complex samples from the initial stages of the physico-chemical treatment system, which can contain high levels of dissolved species (e.g., bromine) and suspended solids, require prior isolation of Se to overcome the spectral interference between Se-K_α and Br- K_α lines. In all cases, TXRF results were compared with those obtained using the standard ICP-MS method. In the case of multielemental analysis of coal fly ash, a detailed study was conducted to evaluate the use of suspensions as a rapid and practical method for sample preparation. Satisfactory analytical results in terms of accuracy and precision were obtained via empirical calibration using a set of certified fly ash reference materials for quantification. The total analysis time per sample was approximately 20 minutes (5 min for sample preparation + 15 minutes for measurement), significantly shorter compared to traditional preparation methods involving fused beads and pressed pellets for conventional XRF, or acid digestion for ICP-based techniques. Additionally, the use of suspensions eliminated the need for costly and hazardous reagents required for ash decomposition, thus minimizing potential health risks to laboratory personnel and aligning this approach closely with the principles of green analytical chemistry.

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ELEMENTAL ANALYSIS OF CRITICAL RAW MATERIALS IN LITHIUM-ION BATTERY BLACK MASS: A TXRF-BASED APPROACH

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End-of-life management and recycling of batteries have become more urgent than ever, due to steeply increasing demand, environmental concerns, limited resources and geopolitical tensions. The European Union aims to establish itself as a global leader in battery recovery, in the framework of the European Green Deal and the Circular Economy Action Plan [1]. After establishing a unified regulatory framework governing the entire life cycle of batteries placed on the EU market, the focus has been set on developing a methodology to calculate and verify recycling efficiency and recovery of critical raw materials, as lithium and cobalt, and strategic raw materials, as nickel and manganese. In order to succeed, a reliable elemental analysis of the spent battery material to recycle, the black mass, is of paramount importance: its variability in lithium-based compounds and the presence of graphite make the task challenging [2], especially for analytical techniques requiring sample solubilization, due to the possible presence of undissolved carbonaceous materials, which may cause troubles in quantification, e.g. ICP-MS. Moreover, the scarcity of suitable reference materials affects the accuracy of methods based on instrument calibration.

In view of the mentioned challenges, TXRF spectrometry may prove itself useful, being a versatile technique as regards the form of sample presentation and the quantification method by internal standardization. Moreover, it has been successfully used in the analysis of batteries previously [3]. In this work we evaluate and optimize sample preparation strategies, focusing on sample presentation to the spectrometer, to quantify critical metals such as cobalt, nickel, and manganese in NMC and LCO black masses [4]. Being the expected mass fraction of the analytes of interest between 1% and 30%, two important aspects are particularly addressed in order to minimize matrix effects and detector artifacts [5], while providing adequate detection capabilities: the mass of the dry residue on the reflector and the internal standard amount. The proposed methodology involves a first measurement of the as-is sample to both estimate its approximate composition by using absolute sensitivities and assess the presence of detector artifacts. Then, the dilution factor and the amount of internal standard are tuned accordingly to proceed with a more accurate quantitative analysis. The results are assessed by preliminary comparisons with complementary analytical techniques.

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TRACE ANALYSIS OF COPPER, ZINC, LEAD, MANGANESE, CHROMIUM, COBALT AND NICKEL IN WORKPLACE AIR USING SUSPENSION-ASSISTED TXRF

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The evaluation of metal exposures in workplace air remains an ongoing and contentious issue in the context of occupational health and safety guidelines and regulations. Recent reductions in occupational exposure limits (OELs) for several metals, including carcinogenic metals such as Co and Ni, have increased analytical challenges in monitoring trace metals in workplace air [1]. The metals of interest typically occur as particulate matter in fumes and dust, that can be collected as inhalable or respirable fractions on membrane-filters using portable air sampling systems, stationary or person related in the breathing zone of workers [2]. The OELs and the actual air concentrations of analytes at typical industrial workplaces (e.g. foundries, welding-shops, electroplating facilities, etc.) can vary from ng/m^3 up to mg/m^3 [3], depending on occupation, material and technical health and safety measures. The personal related sampling for inhalable or respirable fractions, required for the specific assessment of the workplace, leads to low total sampling volumes of 1,2 m^3. For lowest limits of quantification, analysis is therefore usually performed using sensitive but time-consuming, material and labor- intensive spectrometric techniques such as AAS, ICP-OES, and ICP-MS following open vessel or microwaveassisted acid digestion. Due to its well-known advantages, suspension-assisted TXRF seems to be a powerful alternative technique to the established methods mentioned above for the analysis of airborne particulate matter. However, this requires consideration of the analytical challenges that come with particle fractions of complex composition (e.g. total dust quantity, particle size distributions, bulk matrices, homogeneity in dispersion and sample application, spectral interferences and calibration strategy). Combined with the lack of normative specifications for validation of non-digestive methods in the current standards EN 482 [4] or ISO 21832 [5], the validation and establishment of TXRF routineapplications for the assessment of metals in workplace air is quite difficult.

Here we will present a cross-validation study for a routine-compatible, suspension-assisted TXRF analysis method of seven analytes in workplace air samples using microwave assisted digestion with subsequent ICP-MS analysis validated according to normative specifications [6, 7] as a reference. This will include the development of an internal validation scheme and the analytical performance characteristics determined with Bruker S2 PICOFOX (Mo- and W-source) and Bruker T-Star instruments. Furthermore, we will show the applicability of the method in interlaboratory aerosol-onfilter proficiency tests and its performance, limits and pitfalls with real world workplace air samples.

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ELEMENTAL ANALYSIS OF PARTICULATE MATTER: PRELIMINARY RESULTS FROM THE PROJECT "SMART-AIR"– SUSTAINABLE METHODS IN AIR RESEARCH QUALITY

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The SMART-AIR project (Sustainable Methods in Air Research qualiTy: elaboration and validation of reliable elemental quantification via correlative measurements based on certified and "green" techniques for particulate matter) was funded by the Italian Ministry of University and Research under the PRIN 2022 call and officially started in February 2025. The project builds on the outcomes of the COST Innovators Grant IG18130 AIR CAL (https://www.cost.eu/actions/IG18130/), with the goal of advancing the elemental analysis of airborne particulate matter (PM) collected on filters.

SMART-AIR focuses on the development and validation of optimized protocols for sample preparation and analysis, targeting the quantification of Pb, As, Cd, and Ni using complementary atomic spectrometric techniques (see Figure 1). Preliminary results will be presented from Total Reflection X-Ray Fluorescence (TXRF) measurements performed on filters loaded with different PM aerosol analogues [1]. These analogues were prepared by nebulizing either multi-element metal salt solutions combined with polydisperse micro- and nano-plastic suspensions, or by using certified PM10 reference material powders. The loading strategies are designed to replicate the elemental composition and particle size distribution typical of PM found in industrial emissions, workplace environments, and urban air.

A key objective of SMART-AIR is to ensure the reliability and comparability of elemental analysis results across techniques and laboratories, thereby supporting their adoption in regulatory air quality monitoring and standardization efforts.

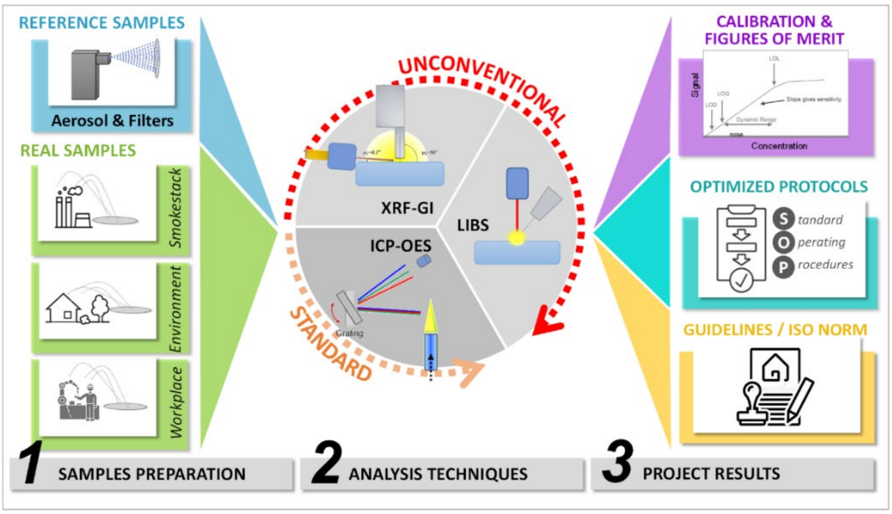

Figure 1: Graphical abstract of the SMART-AIR project.

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AN INNOVATIVE APPROACH MEASURING METAL CONCENTRATIONS IN AIRBORNE PARTICULATE MATTER

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The chemical composition of airborne particulate matter (PM) is important in terms of hazard potential and has therefore led to target values in the EU (Directive 2008/50/EC). The recently launched European project MI-TRAP (MItigating TRansport-related Air Pollution in Europe, https://mitrap-project.eu/) aims to establish a network of monitoring stations strategically located near transport emission hotspots by employing innovative monitoring devices, advanced data analysis, and management plans.

An innovative approach to sampling and measuring PM is based on a new cascade impactor with an arrangement of classifying nozzles optimized for total reflection X-ray fluorescence (TXRF) analysis of the collected particles. TXRF offers detection limits in the picogram range absolute mass and therefore represents a great potential for elemental analysis of heavy metals in aerosol particles [1]. The impactor consists of 4 stages (PM10, PM2.5, PM1, PM0.2) and collects particles in circular areas with a diameter of about 5 mm or less in the center of the sample carrier for full TXRF analysis and is designed to provide low blank values and minimum cross-contamination between subsequent sampling periods.

A first campaign in the framework of the MI-TRAP project was scheduled at the INFN facilities in Florence, Italy in December 2024. The objective of the campaign, which involved 14 runs with sampling times ranging from 1 h to 16 h, was as follows:
1) Immediate TXRF measurement after sampling (1 h sampling and 15 min measurement time). 2) Site by site comparison of impactor/TXRF approach with a commercial monitoring system (Xact 625i, SailBri Cooper, Inc.).
3) Prove of quantitative results with reference-free SR-XRF (PTB, Berlin) and PIXE (INFN, Florence).
4) Development of particle size-specific calibrations.

On-site TXRF measurements with a portable spectrometer enabled the detection of elements such as Cr, Cu, Ni, As and Pb in all runs, even at low concentrations in the pg/m³ range. Applying a laboratory spectrometer at different excitation conditions the heavy elements Mo, Sn and Sb could be quantified. Cd was only measurable with longer sampling times. Comparisons with SR-XRF and PIXE are presented in detail in this paper.

Based on the promising studies, the impactor/TXRF approach is currently being used as part of MITRAP measurement campaigns in various traffic situations in selected European cities (road, railroads, ports and airports). Depending on the pollution situation, shorter sampling times down to 15 minutes are also being tested.

This project has received funding from the European Union's Horizon Europe program under grant agreement No 101138449 — MI-TRAP.

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X-RAY FLUORESCENCE-BASED STUDIES OF PARTICULATE MATTER ON FILTERS

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Under the European Green Deal, the zero-pollution action plan (Directive (EU) 2024/2881) outlines a target of improving air quality by 2030, with a focus on $PM_{2.5}$. It also introduces an obligation to monitor additional pollutants such as ultrafine particles and sets new thresholds for potentially toxic elements such as Pb, Cd, As, Ni and Hg that national environmental agencies must regulate to. Hence, the directive necessitates additional PM monitoring, characterization and controls which in turn requires reliable and accurate elemental analysis techniques underpinned by calibration procedures employing fit-for-purpose reference materials (RM). Unfortunately, suitable RMs for PM on filters are crucially lacking, with no adequate concentration ranges for PMs covering the targeted applications. In addition, both for environmental, safety and economic reasons, analytical scientists are increasingly leaning towards green analytical chemistry. This concept aims at providing environmentally friendly alternatives to established analytical practices, developing cost-saving opportunities and ensuring laboratory personnel safety whilst preserving the classic analytical figures of merit (e.g., accuracy, sensitivity, selectivity).

Within these two frameworks, X-ray Fluorescence (XRF) may find an opportunity to establish itself. Indeed, despite its appealing capabilities (e.g., simultaneous elemental and chemical analysis that may be employed for qualitative, quantitative and screening purposes), XRF is still considered behind on the list of analytical techniques to be employed for the elemental characterization of matter. XRF-based techniques present, however, interesting advantages. Total Reflection X-ray Fluorescence (TXRF), as an example, enables experimentalists to simultaneously determine the elemental compositions with detection limits in the low picogram range. As for XRF microscopy (μ-XRF) or tomography, they provide spatially resolved information of the sample surface or deep volume composition, respectively, typically at the micrometer scale. When a synchrotron is used as an X-ray source, elemental sensitivity down to the femtogram range (TXRF) and spatial resolution at the nanometer scale (μ-XRF) may be achieved. Moreover, XRF is usually regarded as a greener, sustainable analytical technique in comparison to other wet analytical methods.

Here, we illustrate the potential of XRF-based techniques to the study of particulate matter on filters. In particular, we present data from a set of calibration samples characterized with XRF under grazing incidence conditions. We demonstrate that the retrieved net intensities may be used to build an external calibration curve, and allow for quantification procedures for PMs captured on air filters at environmentally relevant levels. The quantification yields uncertainties comparable with those obtained by other elemental analysis techniques. We also present results from a PM source apportionment study, using a combination of XRF-based techniques, including X-ray absorption spectroscopy and imaging.

Acknowledgments

This work was supported by the COST Innovator Grant IG18130 “AIR-CAL” supported by COST (European Cooperation in Science and Technology), and by the Italian Ministry of University and Research within the framework of the “Research Projects of National Relevance, PRIN 2022”, project "SMART-AIR", CUP: D53C24003250006.

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Thursday, 11th September 2025

08:30	Registration

Techniques related to TXRF (Chairperson: Giancarlo Pepponi)

09:00	Kouichi Tsuji (Invited)	“X-RAY FLUORESCENCE ANALYTICAL TECHNIQUES RELATED TO X-RAY TOTAL REFLECTION”
09:45	Martin Radtke	“FROM TOTAL REFLECTION TO GRAZING EXIT: BAMLINE'S 20+-YEAR JOURNEY IN X-RAY FLUORESCENCE INNOVATION”
10:15	Kirsten Siebers	“NANOPLASTIC CHARACTERIZATION WITH T- AND GI-XRF AS PART OF A CORRELATED MICRO-SPECTROSCOPIC APPROACH”
10:45	Coffee Break	

Techniques related to TXRF (Chairperson: Giancarlo Pepponi)

11:15	Manouel Pichois	“MoS_2 QUANTIFICATION OF OVERLAPPED ED-XRF SPECTRA USING SIMULATION AND COMBINED MEASUREMENTS”
11:45	Ebrahim Gholami Hatam	“MODELING COMPLEX X-RAY TRAJECTORIES: A 3-D SIMULATION FOR PATH LENGTH AND YIELD CORRECTION IN MICRO-PIXE ANALYSIS OF ROUGH SURFACES”
12:10	Grzegorz Wesołowski	“APPLICATION OF TOTAL REFLECTION X-RAY PHOTOELECTRON SPECTROSCOPY IN STUDIES OF METALLIC NANOLAYERS”
12:35	Intissar Djouada	“STRUCTURAL AND MORPHOLOGICAL ANALYSIS OF EVAPORATED Ni FILMS ON GaAs (001) AND Si (111)”
13:00	Lunch Break	

Experimental systems and procedures (Chairperson: Dariusz Banaś)

14:15	Dieter Ingerle (Invited)	“GIMOXS: A VERSATILE GI/TXRF SPECTROMETER FOR CHARACTERIZATION OF NANOMATERIALS IN THE LAB FROM C TO U”
15:00	Evgeny Demenev	“A MODULAR, LOW-COST MICROCONTROLLER-BASED READ-OUT SYSTEM FOR ENERGY DISPERSIVE X-RAY SPECTROSCOPY”
15:30	Coffee Break	

Experimental systems and procedures (Chairperson: Dariusz Banaś)

16:00	Diane Eichert	“18115-4: A NEW ISO PROPOSAL FOR A STANDARD FOCUSED ON TXRF TERMINOLOGY”
16:30	Markus Krämer	“X-RAY OPTICS IN TXRF AND GIXRF PRIMARY RADIATION: HOW TO GET YOUR MOST EXCITING BEAM”
17:00	Franziska Sand	“FILM-LIKE RESIDUE PREPARATION BY PICOLITER PRINTING”
20:00	Conference Dinner	

X-RAY FLUORESCENCE ANALYTICAL TECHNIQUES RELATED TO X-RAY TOTAL REFLECTION

Kouichi Tsuji, Ayuri Domyo, Shino Ideriha, Tomohiro Nishiyama, Yuka Hirayama

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The phenomenon of total reflection of x-rays has been applied to various x-ray analytical techniques. I (KT) will introduce studies on TXRF and related techniques in my laboratory. In total reflection x-ray fluorescence (TXRF), primary x-rays are totally reflected on a flat surface of the reflector, and its penetration depth is a few nm levels. Therefore, surface analysis of flat substrates such as Si wafer is possible. In TXRF, an x-ray detector such as a Si drift detector (SDD) is placed just above the reflector. This special geometry is suitable for trace analysis. In a TXRF trace analysis, a small amount of the sample is placed on a flat substrate, and a monochromatic primary x-ray is irradiated to the substrate. We can scan the position of the SDD under total reflection condition, leading to scanning type surface elemental imaging [1]. It is also possible to place an x-ray camera instead of the SDD under grazing incidence condition. In this case, full-field type surface-sensitive elemental imaging is possible [2]. After TXRF instrument was commercialized, the research on TXRF has moved to sample preparations [3,4]. Although TXRF has been applied to various samples, the importance of evaluating analysis volume of the TXRF instrument used has been discussed [5]. In addition, the shape of the sample on the reflector affects TXRF analysis, thus it would be useful to measure surface morphology by confocal microscope. Also, XRF elemental mapping, which can be done by micro-XRF spectrometer, would be helpful for TXRF analysis. TXRF analysis of biological sample sliced by a microtome will be shown.

A grazing exit (or emission) XRF (GE-XRF) is a related technique to TXRF [6]. The same sample as TXRF has been measured by GE-XRF. Since XRF can be produced by monochromatic primary x-rays and white x-rays, x-rays emitted from the x-ray tube are directly irradiated on the sample without monochromator. Thus, an enhancement of XRF intensity is expected. In addition, a micro x-ray beam can be applied, leading to micro-GEXRF [7]. The detection limit of GEXRF is comparable to that of TXRF [8].

X-ray total reflection phenomenon has been applied to x-ray focusing optics like polycapillary lens, leading to micro-XRF analysis [9]. In this experimental setup, the sample is usually placed to perpendicularly to the micro beam. A new XRF experimental arrangement using a capillary optic will be discussed.

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FROM TOTAL REFLECTION TO GRAZING EXIT: BAMLINE'S 20+-YEAR JOURNEY IN X-RAY FLUORESCENCE INNOVATION

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For more than two decades the hard-X-ray BAMline at the BESSY II synchrotron has served as a versatile platform for non-destructive micro-analysis. Its high photon flux and energy tunability support a suite of complementary methods that now span three dimensions of information: elemental sensitivity down to the femtogram level, chemical speciation, and depth resolution in the sub-micrometre regime [1].

Early work concentrated on synchrotron-radiation-induced total reflection X-ray fluorescence (SR-TXRF). A dedicated SR-TXRF station routinely achieves detection limits below 100 fg Ni and, by exploiting the monochromatic beam, extends to TXRF-XANES for speciation at trace concentrations[2].

Spatially resolved analytics were optimized by integrating the Color X-ray Camera (pnCCD + polycapillary optics). Operating as a single-photon counter, the camera delivers full-field XRF maps with ~50 µm effective pixel size and <160 eV energy resolution, recording tens of thousands of spectra in seconds. This provided an experimental testbed to quantify TXRF-specific matrix effects such as shading in picolitre droplet residues [3].

To probe buried interfaces the beamline recently adopted surface-sensitive Grazing Exit (GE) schemes. GE-XAS/-XANES, realised with angle-resolved pnCCD detection, separates fluorescence lines (e.g. Cr-Kα) and extracts oxidation-state profiles through layered alloys and coatings with sub-µm depth resolution. Case studies on corrosion of compositionally complex alloys illustrate the power of correlating lateral and depth information [4].

A current focus is reducing the typically long dwell times of sequential GE-XANES scans. By coupling Bayesian optimisation to the monochromator control we demonstrate intelligent energy grids that maintain spectral fidelity while cutting acquisition time by up to two orders of magnitude[5].

The next development stage will merge coded-aperture X-ray imaging with SR-TXRF. A reconfigurable mask placed in the incident beam will cast multiplexed illumination patterns on the sample; compressed-sensing reconstruction, assisted by on-the-fly machine learning, will convert the encoded fluorescence into high-fidelity elemental maps up to ten times faster than current full-field approaches while preserving sub-100 fg detection limits.

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NANOPLASTIC CHARACTERIZATION WITH T- AND GI-XRF AS PART OF A CORRELATED MICRO-SPECTROSCOPIC APPROACH

Kirsten Siebers[1], Dieter Ingerle[2], Lieke van den Burg[1], L.D.B. Mandemaker[1],
B.M. Weckhuysen[1], Peter Wobrauschek[2,3], Christina Streli[2,3], Florian Meirer[1]
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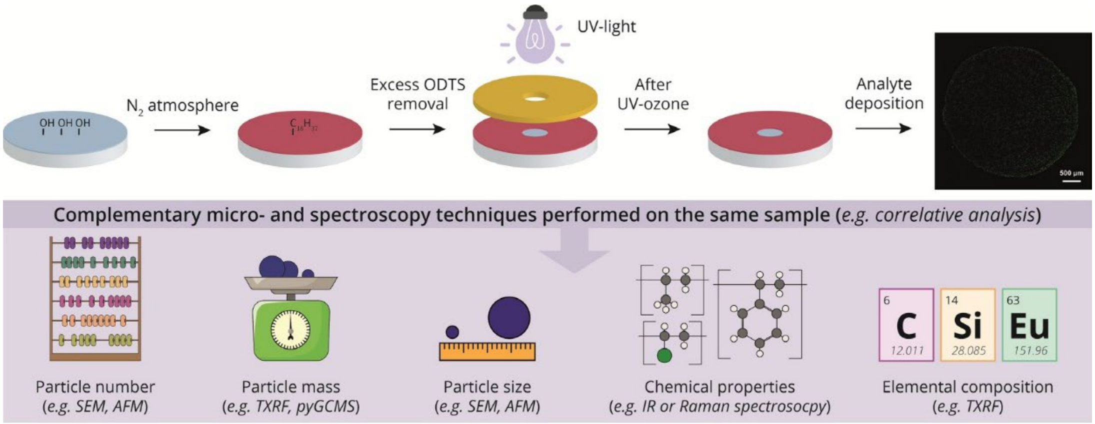
Total reflection X-ray fluorescence spectroscopy (TXRF) is a powerful tool for trace-elemental analysis of a wide range of elements, both qualitatively as well as quantitatively. Depending on the design of the instrument, also grazing incidence X-ray fluorescence spectroscopy (GI-XRF) can be performed with the same setup. In that case the fluorescence intensity is monitored as a function of the glancing angle of the incoming X-ray beam providing additional information about the nature of the sample such as being more film- or particle-like. [1] Therefore, the information obtained from combined T- and GIXRF measurements could aid the analysis of nanoplastics in the environment. In addition, the nondestructive nature of the techniques makes them fit well into a correlative micro-spectroscopy approach, which allows for a more holistic characterization of nanoplastic-related samples. [2] However, there are two reasons why measuring nanoplastics with TXRF is not straightforward. Firstly, common lab-based instruments are usually limited to higher Z elements, while nanoplastics consist mainly of carbon (Z=6). Secondly, nanoplastics samples are suspensions instead of solutions which can cause deviations from the necessary thin-film approximation for TXRF.

To overcome the first challenge, a new instrument was developed with optimal characteristics to measure both light elements, as well as metals by means of a modular design (GIMOXS: Grazing Incidence Modular Optics X-ray Spectrometer). [3] During initial testing, a lower detection limit of 7 ng was obtained for carbon, which makes it possible to detect nanoplastics at concentrations in the nanogramregime. To overcome the second challenge, an advanced sample preparation method was developed. Here, the analyte was forced to dry in a well-defined hydrophilic area on a hydrophobized reflector to provide control over the position of the residue. [4] The hydrophilic region was matched to the field-ofview of the TXRF detector to maximize the spread of the analyte. The formation of a coffee-stain around the periphery of the hydrophilic area was prevented by Marangoni flow assisted drying. [5] In this talk, the advanced sample preparation method for correlative micro-spectroscopy as well as its application for the quantification of nanoplastics by TXRF on a newly developed spectrometer will be presented.



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MoS_2 QUANTIFICATION OF OVERLAPPED ED-XRF SPECTRA USING SIMULATION AND COMBINED MEASUREMENTS

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As 2D TMDs (Transition Metal Dichalcogenides) deposition methods are now being developed to meet industrial demands, on 200 mm and 300 mm wafer scales, there is a crucial need to implement characterization techniques compatible with clean rooms environments and able to measure the composition and the thickness at such dimensions.

X-Ray Fluorescence (XRF) is a promising approach to that problematic, and challenging samples like MoS_2, proved to be successfully measured using wavelength dispersive (WD) detectors [1]. However, energy-dispersive (ED) systems present a lower spectral resolution, which causes sulfur (S) peaks to be fully overlapped by molybdenum (Mo)-*L* lines. Sulfur intensities are thus, not directly accessible, making the quantification challenging.

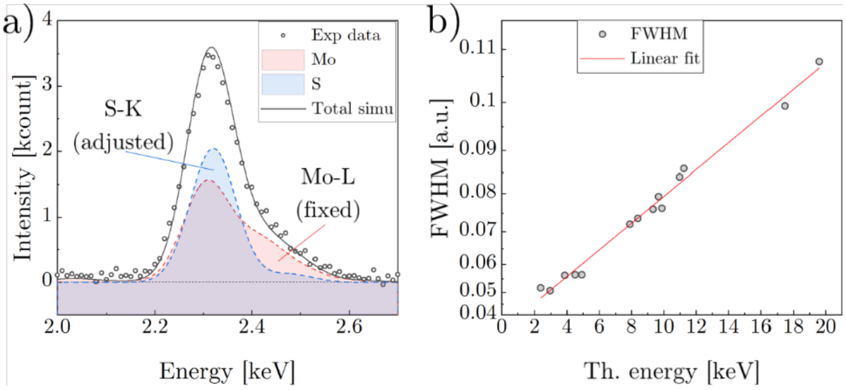

Figure 1: a) MoS_2 spectrum obtained under Cu-K_α monochromatic excitation (8.05 keV), showing overlapped Mo-*L* lines and S-*K* lines. Mo-L spectrum is simulated based on a combined Mo-*K* measurement. S-*K* is simulated and adjusted in order to fit the experimental data. b) Instrumental dependency of isolated peaks' full width at half maximum over their fluorescence energy.

We show that sulfur spectra can be determined by combining spectra of isolated Mo-*K* lines in order to simulate Mo-*L* contribution in the overlapped energy range. These simulations rely on databases in order to obtain spectra with theoretical intensity ratios and fluorescence energies. Instrumental parameters such as the full width at half maximum and the detector energy offset have been characterized and taken into account for the simulated spectra to be faithful to an instrumental result. Obtained concentrations are shown to be in agreement with reference WDXRF analysis.

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MODELING COMPLEX X-RAY TRAJECTORIES: A 3-D SIMULATION FOR PATH LENGTH AND YIELD CORRECTION IN MICRO-PIXE ANALYSIS OF ROUGH SURFACES

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Accurate modeling of X-ray path length is crucial for quantitative X-ray spectroscopy, significantly influencing intensity and detectability, particularly in Micro-PIXE/XRF analysis of samples with complex topographies [1]. This study presents a comprehensive 3-D simulation model developed to investigate X-ray path length and resultant yield variations due to surface roughness. The model simulates surface topography in 1-D, 2-D, and 3-D, integrating key physical parameters such as stopping power, X-ray production cross-sections, photon attenuation, and local photon creation to estimate X-ray intensity maps. A critical feature is its ability to account for severe surface roughness where X-rays may exit and re-enter the sample multiple times en route to the spectrometer. The model was validated by calculating X-ray path lengths and their effects on accumulated X-ray yield using both mathematically generated topographies and experimental Micro-PIXE data from pure Ti and structured brass alloys, acquired with a four-segment silicon drift detector (SDD) [2]. Results demonstrate a remarkable correlation between detailed path length calculations and observed X-ray yields, highlighting the substantial impact of microscale roughness. This approach promises to enhance the accuracy of X-ray intensity predictions, leading to more precise elemental composition analysis in Micro-PIXE and related X-ray spectroscopy techniques.

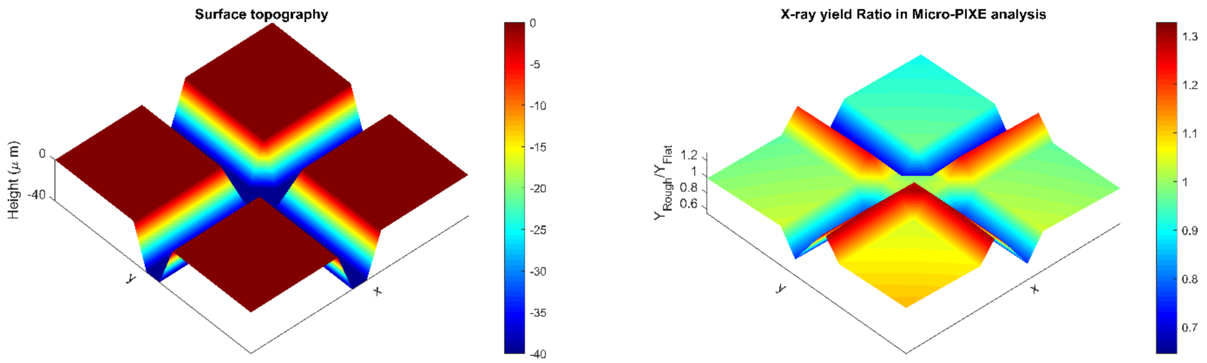

Figure 1: (Left) Simulated 3-D cross-shaped surface topography with a 40 µm indentation. (Right) Corresponding X-ray yield ratio (Y_{Rough}/Y_{Flat}) in Micro-PIXE, showing topographic influence on detected X-ray intensity.

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APPLICATION OF TOTAL REFLECTION X-RAY PHOTOELECTRON SPECTROSCOPY IN STUDIES OF METALLIC NANOLAYERS

Grzegorz Wesołowski[1,*], Aldona Kubala-Kukuś[1, 2], Dariusz Banaś[1, 2], Karol Szary[1, 2], Ilona Stabrawa[1, 2], Paweł Jagodziński[1, 3], Marek Pajek[1], Michał Borysiewicz[4], Oskar Sadowski[4] Juliusz Chojenka[5], Ilaria Carlomagno[6], Juan Reyes Herrera[6]

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X-ray photoelectron spectroscopy (XPS) is a widely used technique for analyzing the surface composition and chemical state of materials. By directing a low-energy X-ray beam (e.g., Al-Kα) onto the sample at a fixed angle, it enables the emission and detection of photoelectrons from the top few nanometers of the surface [1]. A notable enhancement of this technique can be achieved by reducing the incidence angle of the X-ray beam below the critical angle for total external reflection, which results in a stronger surface signal and lower background. This modified geometry is referred as total reflection X-ray photoelectron spectroscopy (TRXPS) [2,3].

In the present study, both conventional XPS and TRXPS geometries were employed to investigate nanolayers of Ti (10 nm, 50 nm, and 100 nm), Au (10 nm, 50 nm, and 100 nm), and Pd (100 nm), deposited on silicon substrates. The main goal was to determine the surface elemental composition and homogeneity of the nanolayers, as well as to evaluate the detection limit of XPS depending on the incidence angle. Measurements were performed using the SPECS mono-XPS system at the Institute of Physics, Jan Kochanowski University in Kielce.

XPS spectra were recorded at standard (35° and 10°) and total reflection angles (1.5° for Ti, 2.2° for Pd, and 2.4° for Au). The C 1s (C–C) peak at 284.8 eV was used for energy calibration. The registered photoelectron spectra gave information about the nanolayer elemental composition while detailed analysis of photoelectron peaks (e.g. C 1s, O 1s, Ti 2p, Pd 3d, and Au 4f) allowed for comparison of spectral parameters such as peak intensity, FWHM, and background level across different measurement geometries. The obtained parameters were used in calculation of detection limit for various incidence angle. The results confirmed that using angles below the critical angle significantly enhances the intensity of photoelectron peaks and reduces background, which leads to improved detection limits.

The nanolayers were also characterized by application of X-ray reflectometry (XRR) and grazing incidence X-ray fluorescence (GIXRF). The measurements were performed at the Elettra-Sincrotrone Trieste. XRR and GIXR measurements allowed for a comprehensive characterization of the morphology and roughness of the nanolayers.

This work was co-financed by the Minister of Science (Poland) under the "Regional Excellence Initiative" program (project no.: RID/SP/0015/2024/01).

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STRUCTURAL AND MORPHOLOGICAL ANALYSIS OF EVAPORATED Ni FILMS ON GaAs (001) AND Si (111)

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Ni thin films are deposited on GaAs(001) and Si(111) substrates at room temperature using thermal evaporation under vacuum. The effects on the film growth mechanism, microstructure, grain size, and surface morphology are studied. The resulting film structure is investigated by X-ray diffraction (XRD), it is found that the samples deposited on GaAs (001) are crystallized in fcc-Ni single-crystal; however, the samples Ni/Si(111) are all polycrystalline with the <111> texture. Atomic force microscopy (AFM) reveals topographic images of very smooth surfaces for most samples, while some samples present topographies of rough surfaces with roughness varying from 3.36 nm to 6.65 nm.

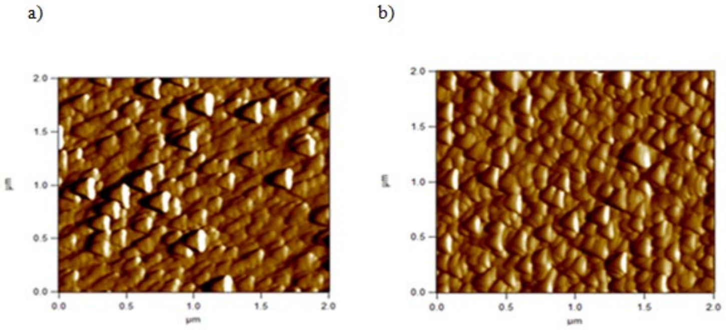

Figure 1: AFM image, (a) for the 600nm Ni/Si (1 1 1), (b) 800nm Ni/GaAs (001) samples. Scale in the axes: 2 µm x 2 µm.

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GIMOXS: A VERSATILE GI/TXRF SPECTROMETER FOR CHARACTERIZATION OF NANOMATERIALS IN THE LAB FROM C TO U

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[3]Atominstitut, TU Wien, Vienna, Austria

GIXRF in comparison to TXRF increases the incident angle of a monochromatic low-divergence exciting beam from zero up to several times the critical angle, thereby varying the penetration depth and the beam interferences.

The obtained measurement data contain information on composition, density, and thickness of layers in the nanometer range, depth distributions of implanted atoms (especially if combined with X-ray reflectometry (XRR)) or the size and composition of nanoparticles.

The sensitivity for the elements depends strongly on the excitation energy, thus the Grazing incidence modular X-ray spectrometer (GIMOXS) was designed to use a Cr anode X-ray tube for light elements down to Carbon and a Mo anode for heavier elements. In both cases the beam is monochromatized and parallelized by a suitable 1-dimensional parallel beam multilayer X-ray optics. In order to keep the setup modular and flexible, the optics assembly is housed in a separate vacuum chamber, which is connected by standard KF-flanges to the vacuum chamber containing the sample.

Apart from the small X-ray fluorescence cross section of light elements, i.e., elements below Magnesium, which can be mitigated by using a Cr X-ray tube, the analysis of light elements, poses significant other challenges. The high absorption of the produced low-energy X-ray photons in air has to be eliminated by using vacuum conditions also in the sample chamber. The natural limitation of the low X-Ray fluorescence and high Auger-Meitner electron yield has to be accepted. In order to reduce the absorption for low energy X-rays in the detector window, the detector is equipped with an ultra-thin SiN window as well as an optimized electron trap.

The sample stage is flexible for Si wafers of any shape as well as quartz reflectors with 30 mm diameter. Due to past experience with non-centered samples, x-y translation stages were added as a novel feature to allow scanning of the surface of the sample. The translation and rotation of the sample is computer controlled.

The reflected beam from the sample leaves the vacuum chamber and impinges on a fluorescence screen monitored by a CCD camera, which can be used for alignment or X-ray reflectivity measurements with limited dynamic range.

During the commissioning at Utrecht University the following detection limits have been achieved: Carbon 7 ng, Natrium 82 pg, Titan 0.3 pg, Rubidium 0.8 pg.

Further recent results will be presented.

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A MODULAR, LOW-COST MICROCONTROLLER-BASED READ-OUT SYSTEM FOR ENERGY DISPERSIVE X-RAY SPECTROSCOPY

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We present a cost-effective and power-efficient signal readout and data processing system tailored for X-ray spectroscopy with energy dispersive silicon detectors, based on a 32-bit ARM microcontroller. Designed with scalability in mind, this compact solution addresses the growing demand for high-channelcount, low-maintenance electronics in industrial and portable X-ray fluorescence systems, including TXRF.

Historically, microcontroller-based spectroscopy systems have been constrained by performance limitations and high dead time, rendering them impractical for demanding applications. Our work overcomes these challenges with a fully integrated, highly customizable architecture that leverages modern microcontrollers to deliver reliable performance while minimizing system complexity, component count, and cost.

Targeting silicon drift detector (SDD) arrays, the system enables signal acquisition and processing from two independent channels using a single microcontroller, with only four external components per channel. It achieves zero dead time and supports a throughput of up to 700,000 events per second per microcontroller (500,000 per channel), all with a current draw below 20 mA per channel. As an example Figure 1 shows a 16 channel system with 9 microcontrollers (8 for read-out, 1 for communication) has been realized for an energy dispersive silicon strip detector and a single MCU system designed for a 2 SDD detectors.

This combination of low power consumption, minimal hardware requirements, and high performance makes the system exceptionally well-suited for scalable detector arrays and industrial environments where space, power, and cost constraints are critical. Its modularity and ease of integration also make it an attractive solution for OEM developers and embedded TXRF systems requiring reliable, high-throughput spectral data acquisition.

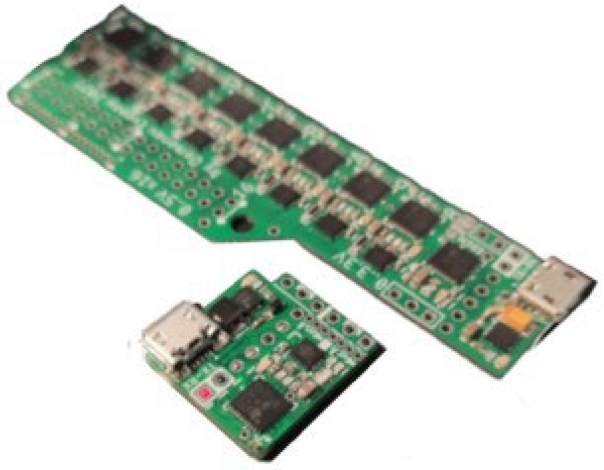

Figure 1: Image of a single MCU read-out board for a 2-channel detector and a 9 MCU board designed for a 16 channel detector.

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18115-4: A NEW ISO PROPOSAL FOR A STANDARD FOCUSED ON TXRF TERMINOLOGY

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Total Reflection X-ray Fluorescence (TXRF) is a technique that presents appealing capabilities, as it enables a simultaneous multi-elemental analysis of a sample which can be of qualitative, quantitative or screening character. A TXRF chemical measurement process, when considered holistically, combines knowledge in instrumentation, physics, analytical chemistry (sampling, sample preparation and handling), metrology, quality management, software and data analysis. The ease-of-use along with access to dedicated commercial instruments means that TXRF is a technique that can be employed by a broad range of people from very different backgrounds and from diverse fields for very different surface chemical analyses. Those conducting TXRF can be materials scientists, chemists, physicists, or biologists with those making use of the data and results extending beyond this group into other disciplines.

The wide range of disciplines, expertise, skill level and the individualities of national users have led to divergence of meanings being attributed to specific terms and, again, different terms being used to describe the same concept, approach, or analysis. To avoid the consequent misunderstandings between communities and to facilitate the clear exchange of information, as well as to provide basis for a TXRF normative framework, it is essential to clarify the concepts needed when undertaking a TXRF analysis. This is motivated further by the need for correct reporting of metadata with the move to open access and FAIR data repositories, and by the advent of artificial intelligence for the treatment and exploitation of the data.

Thus, it is critical that the terms relating to all aspects of a TXRF experiment are established in one place, their concepts clarified and their validity ensured across all scientific disciplines. This is the purpose and justification for the development of this new ISO standard [1]. The ISO 18115 standard defines, and gives references for terms for surface chemical analysis, and is composed by several parts. ISO 18115-1 covers general terms and those used in spectroscopy, ISO 18115-2 terms used in scanningprobe microscopy and 18115-3 focuses on optical interface analysis including ellipsometry, Raman and nonlinear optical techniques. The part four “Terms used in Total Reflection X-ray Fluorescence” will extend the standard. ISO 18115-4 will help in ensuring consistency, precision, and efficiency in TXRF analysis, and in fostering exchanges within scientific communities.

This presentation will highlight the rationales behind the ISO 18115-4 standard, and demonstrates that the terminology for TXRF is not fully autonomous and only based on terms sourced in physics, but is a consequence of the existence of intra-, inter-, multi- and trans-disciplinary relations. It will also discuss how this terminology standard is reflecting and supporting the contents of the VAMAS pre-normative activity [2] aiming at describing the TXRF chemical measurement process, via the elaboration of procedures and validation steps.

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X-RAY OPTICS IN TXRF AND GIXRF PRIMARY RADIATION: HOW TO GET YOUR MOST EXCITING BEAM

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The concept of TXRF is based on the assumption of a monochromatic, parallel, homogeneous X-ray beam hitting the sample at a fixed very small (or - in the case of GIXRF - varying) grazing angle. In reality, however, this is usually not the case, for physical and also for technical reasons.

A simple way to confine the radiation emitted from the source that reaches the sample is a slit system. A plane crystal monochromator can have a similar effect, just selecting the photon energy of the characteristic emission line of the tube. Curved multilayer mirrors on the other hand can significantly increase the photon flux illuminating the sample.

Here, the options with higher photon density and better beam parallelism (lower divergence) can have somewhat contrary effects. Further, the lateral distribution of photons per area unit varies when applying different optical devices. This can be particularly important with large sample areas or changing illumination areas in GIXRF set-ups.

Thus, different options of TXRF/GIXRF set-ups are evaluated in terms of illumination area, intensity distribution and spectral composition of the exciting beam. Results are presented together with ideas and perspectives for possible improvements in existing systems or new designs.

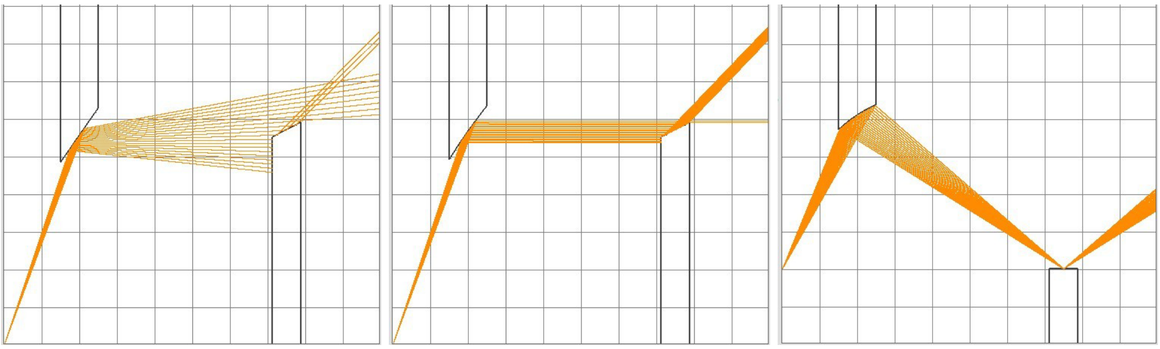

Figure 1: Effect of different primary beam optics on divergence and intensity distribution.

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FILM-LIKE RESIDUE PREPARATION BY PICOLITER PRINTING

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An accurate sample preparation is fundamental for the success of ultra-trace analysis techniques with total reflection X-ray fluorescence (TXRF) [1]. In TXRF, particulate and film-like residues need to be differentiated since the morphology has an influence on the detected sensitivity and thus on the quantification of the respective element [2]. Film-like residues are thinner than particulate residues and therefore only excited by the X-ray standing wave field caused by interference phenomena of the totally reflected X-ray beam [2,3]. The morphology of residues can be characterized using angle scanning TXRF. Film residues show a strong increase in the angle-dependent behavior before reaching the critical angle until a maximum is reached. In comparison to particulate residues, no plateau-like progression is observed. The intensity decreases again after the critical angle is exceeded. [3] Common sample preparation methods such as pipetting often lead to so-called coffee rings, in which a large part of the sample solution dries up as a particulate ring-shaped residue [2,4]. As an alternative to conventional pipetting, small sample volumes in the picoliter range (~160 pL) can be applied onto a sample carrier with a defined morphology, which also enables the preparation of film-like deposits [5,6].

Our in-house build picoliter printer is operated with commercially available ink-jet cartridges, multiple linear stages and 3D printed parts and is able to deliver user defined patterns of liquid ethylenediaminetetraacetic acid (EDTA) stabilized elemental standards. The specimen prepared on quartz glass carriers were measured using a table-top TXRF spectrometer (S4 T-STAR®, Bruker Nano GmbH, Berlin, Germany) with a Mo X-ray tube (Kα = 17.5 keV). Angle-resolved measurements were performed with the same instrument using Goebel mirrors. The shape of the printed residues was determined using an atomic force microscope (AFM).

By varying the printing parameters such as the quiet time and the droplets per position the influence on the film formation behavior can be investigated. In addition, the surface quality of the sample carriers and different additives were also taken into account. The preparations were first carried out with the element Nickel, but could be applied to other elements of the periodic table in the future to investigate their element-dependent film formation tendency.

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Friday, 12th September 2025

08:30	Registration	
TXRF applications (Chairperson: János Osán)		
09:00	Frithjof Mähler	“X-RAY EMISSION SPECTROSCOPY - AN SUPPORTING TOOL FOR TXRF MEASUREMENTS?”
09:35	Ukyou Yanagisawa	“EVAPORATIVE CONCENTRATION OF SOLUTION ONTO A HYDROPHILIC SPOT PRESERVED ON A FLUORINE-COATED GLASS SLIDE FOR HIGH-SENSITIVITY TXRF ANALYSIS”
10:10	Horoshi Yoshii	“DETECTION OF U AND PU IN THE PRESENCE OF SR BY HYBRID MEASUREMENT OF TXRF AND SPONTANEOUSLY EMITTED CHARACTERISTIC X-RAYS”
10:45	Coffee Break	
TXRF applications (Chairperson: János Osán)		
11:15	Abdallah Shaltout	“VALORIZATION THE IMPORTANCE OF THE RECYCLING OF DIFFERENT TYPES OF STEEL SLAGS BASED ON X-RAY SPECTROMETRY’S”
11:45	Arslan Usman	“CORRELATIVE SPECTROSCOPY AND QUANTIFICATION OF NMC ALLOYS USING XRF, SEM AND NANO GPS TECHNIQUE”
12:15	Regina Stachura	“ANALYSIS OF ELEMENTAL COMPOSITION OF WOMEN’S AND MEN’S NAILS USING TOTAL REFLECTION X-RAY FLUORESCENCE (TXRF) METHOD”
12:45	**TXRF 2025 Closing Remarks**	
13:00	Lunch	

X-RAY EMISSION SPECTROSCOPY - AN SUPPORTING TOOL FOR TXRF MEASUREMENTS?

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Total X-ray reflection spectrometry (TXRF) is used for the direct quantitative analysis of environmental samples from *e.g.* aerosols in impactors or for suspension assisted analysis of solid samples. These sample collection methods have the advantage that the sample is not changed between the collection and the quantitative analysis. This gives the opportunity that the elemental species are quasi-not chemically changed and can be determined in addition to the quantitative analysis. The oxidation state of transition metals can be important for an toxicological interpretation of the measured amount of the metal. A prominent example is Chromium where the oxidation state +6 is toxic and the oxidation state +3 is an essential trace element.

For the speciation of aerosol samples collected for TXRF measurements commonly X-ray absorption near edge structure (XANES) is used. Due to the low sample amount and the need of measurements done in fluorescence mode these measurements are done exclusively at synchrotron facilities. XANES measurements require an homogeneous sample in the area of the X-ray beam which is difficult to achieve for bigger spot sizes which are used in laboratory devices.

Another technique for elemental speciation is X-ray emission spectroscopy (XES). Most prominent in literature are measurements of the fine structure of the K$_\beta$ line of transition metals. With these spectra information about the spin state, the oxidation state and the coordination of the analyte can be gathered. For the analysis of small sample amounts the K$_\beta$ line is not advantageous due to its lower intensity. The K$_\beta$ is sensitive to the oxidation state of the element as well. In measurements of the fine structure of the Chromium K$_\alpha$ line significant differences between the oxidation states +3 and +6 can be observed.

In this work we explore the capabilities of an laboratory X-ray emission spectrometer for the speciation of small sample amounts on TXRF sample carriers

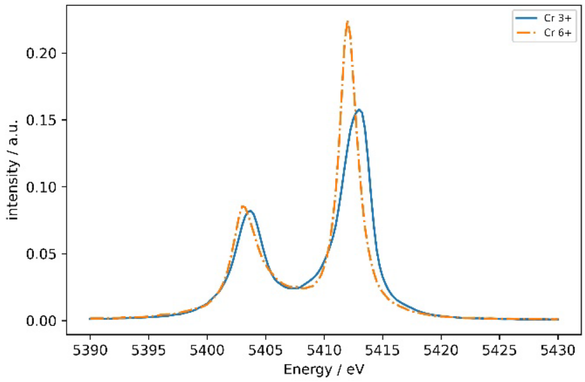

Figure 1: Area normalized K$_\alpha$ X-Ray Emission spectrum of Chromium in the oxidation states +6 and +3.

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EVAPORATIVE CONCENTRATION OF SOLUTION ONTO A HYDROPHILIC SPOT PRESERVED ON A FLUORINE-COATED GLASS SLIDE FOR HIGH-SENSITIVITY TXRF ANALYSIS

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Total reflection X-ray fluorescence (TXRF) analysis is typically utilized to analyze trace elements. Our research group has employed this technique to analyze uranium in solution [1]. During the TXRF analysis of a liquid sample, several microliters of the solution were dropped onto a fluorine-coated glass slide and dried. However, the sensitivity of the measurement can be improved by increasing the volume of the sample dropped onto the measurable area. In principle, a solution of the same concentration can be measured with tenfold sensitivity by dropping 10 times its volume onto the same spot size. In this study, we propose a more sensitive TXRF analysis method by applying a fluorine coating to the outside part of a hydrophilic slide glass, except for the central spot, which allows 1 mL of the solution to be dropped.

The hydrophilic surface-treated glass slide was covered on one side with polyimide tape, and the outer tape was cut off, leaving a 6 mm diameter of making area in the center. Then, a fluororesin solution (Fine Chemical Japan Co., Ltd.) was sprayed onto the surface, and after drying, the masking tape was removed. The sample was prepared by diluting a multielement standardized solution of XSTC-4470 (Spex CertiPrep Inc.) containing 10 ng/μL of uranium, and 1 mL of this solution was dropped onto the hydrophilic spot of the glass slide. The dropped solution was evaporated under redused pressure to shrink onto a 6 mm-hydrophilic spot, followed by adding 10 μL of 1 ng/μL Ga solution as an internal standard, and then dried completely. All measurements were performed using a TXRF spectrometer NANOHUNTER-II (Rigaku Co., Ltd.), at a tube voltage and current of 50 kV and 12 mA, respectively.

As illustrated in Figure 1, the solution on the fluorine-coated area shrinks to the hydrophilic spot during the drying process. This facilitates TXRF analysis with higher sensitivity than conventional methods.

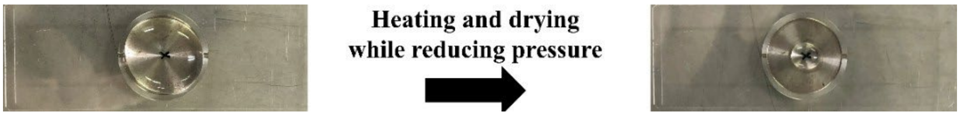

Figure 1: Droplets before and after drying. The droplets of 1 mL shrink onto the hydrophilic spots and are then thoroughly dried before being analyzed.

This study was performed under the auspices of the Secretariat of the Nuclear Regulation Authority, Japan.

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DETECTION OF U AND Pu IN THE PRESENCE OF Sr BY HYBRID MEASUREMENT OF TXRF AND SPONTANEOUSLY EMITTED CHARACTERISTIC X-RAYS

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TXRF analysis of samples containing U and Pu was previously performed by our group [1]. However, when a sample contains Sr, the analysis of Pu will be impeded because of overlapping of the Pu Lα peak (14.28 keV) with the Sr Kα peak (14.17 keV). The major isotopes of Pu spontaneously emit characteristic X-rays of their daughter nuclide, U, upon internal conversion after alpha decay [2]. Using a TXRF analyzer, the spontaneously emitted characteristic X-rays can be detected by setting the X-ray tube output to zero so that no X-rays are irradiated onto the sample (Figure 1). Portable TXRF analyzer 200-FAS (Ourstex) is a modified version of the commercially available 200-TX [1,3] and can measure the spontaneously emitted characteristic X-rays by setting the incident X-ray power to zero. This method enables analyzing Pu even in the presence of nonradioactive Sr.

In this study, we developed a method to detect U by TXRF analysis and Pu by measuring the spontaneously emitted characteristic X-rays by dropping a solution containing U, Pu, and Sr onto a glass plate. Although spontaneously emitted characteristic X-rays are detected during TXRF analysis, the intensity of their signal is considerably lower than that of the background signal, and they do not interfere with the analysis.

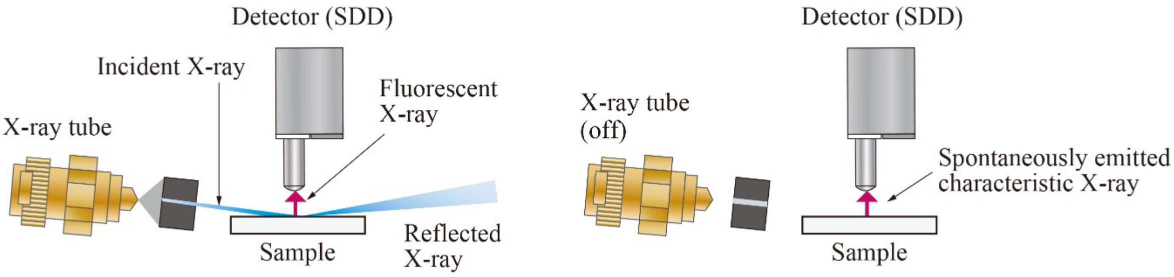

Figure 1: Overview of TXRF and spontaneously emitted characteristic X-ray measurements.

This study was performed under the auspices of the Secretariat of the Nuclear Regulation Authority, Japan.

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VALORIZATION THE IMPORTANCE OF THE RECYCLING OF DIFFERENT TYPES OF STEEL SLAGS BASED ON X-RAY SPECTROMETRY'S

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To prevent the deterioration of natural resources and contribute to sustainable development, the valorization and recycling of various types of steel slags have been emphasized. In this study, six different types of steel slags were collected, characterized, and investigated, namely: electric arc furnace (EAF) slag, ladle arc furnace slag treated with Si and Al (LAF-Si and LAF-Al), steel fume dust, mill scale, and refractory materials. High-resolution wavelength dispersive X-ray fluorescence (WDXRF) spectrometry with multi-dispersive crystals was utilized for accurate quantitative elemental analysis of the different types of steel slags. Additionally, an X-ray diffractometer was employed to study and estimate the quantitative phase analysis of the components in the steel slag samples.

The mill scale and EAF slags were found to have estimated iron oxide concentrations of up to ~95% and ~40%, respectively. The high concentration of iron oxides makes these materials attractive for further reduction and recycling processes. Moreover, the calcium oxides and other Ca compounds in the LAF slag reach up to ~50%. Additionally, zinc oxides are enriched in the steel fume dust, with concentrations of up to ~35%.

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CORRELATIVE SPECTROSCOPY AND QUANTIFICATION OF NMC ALLOYS USING XRF, SEM AND NANO GPS TECHNIQUE

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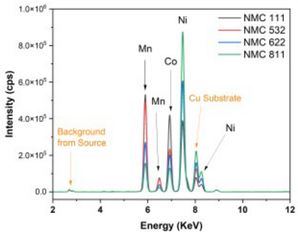
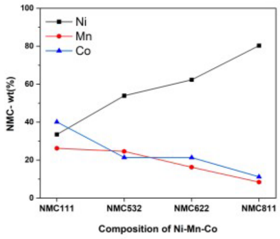
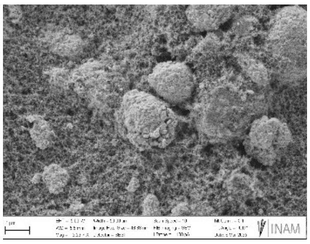
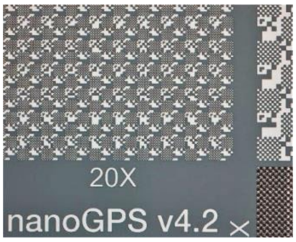
This study presents a scale-bridging approach for the quantification and characterization of NickelManganese-Cobalt (NMC) alloys using X-ray fluorescence (XRF) spectroscopy and nano GPS tagging. Accurate elemental composition measurement is crucial for optimizing NMC cathodes in lithium-ion batteries (LIBs), influencing their energy density, cycling stability, and safety. We employed micro

X-ray fluorescence (μ-XRF) for high-resolution elemental mapping across different NMC compositions (NMC111, NMC532, NMC622, and NMC811). Our findings demonstrate the impact of X-ray source current on the fluorescence yield, revealing self-absorption effects at higher currents, particularly in Ni-rich compositions. Cation mixing analysis showed increased Ni^{2+} migration in Ni-rich cathodes, while Co-rich samples exhibited enhanced structural stability. SEM analysis revealed particle morphology, indicating that Ni-rich NMCs tend to fragment into smaller agglomerates due to higher surface reactivity. To enhance measurement precision across different analytical instruments, we implemented nano GPS tags, achieving region of interest (ROI) relocation with ±3 μm accuracy. This study highlights the importance of advanced X-ray spectroscopy techniques and nano GPS-assisted correlative microscopy for precise elemental quantification, microstructural analysis, and defect detection in NMC materials. These findings contribute to the development of more efficient and stable next-generation lithium-ion batteries.









ANALYSIS OF ELEMENTAL COMPOSITION OF WOMEN'S AND MEN'S NAILS USING TOTAL REFLECTION X-RAY FLUORESCENCE (TXRF) METHOD

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The analysis of elemental composition in the human body is gaining increasing importance due to its diagnostic and epidemiological potential. Trace and macro elements are essential for numerous physiological processes, and any imbalance - whether deficiency or excess - can contribute to the development of chronic diseases, including cancer. Recently, studies have adopted nails as a potential biomarker of exposure to many metals due to their ability to accumulate elements over time and their resistance to external contamination. Among the various biological materials used in elemental composition analysis, such as blood or urine, nails allow monitoring of exposure to elements going back even to several months. In addition, nail samples can be collected without the need for invasive procedures, easily and long-term stored [1-3].

Total Reflection X-ray Fluorescence (TXRF) is an advanced analytical technique used for qualitative and quantitative multi-elemental analysis. It is characterized by high sensitivity, minimal sample consumption, and the ability to analyze liquid or solid samples, making it well-suited for trace element detection in biological matrices [4].

In this study, Total Reflection X-ray Fluorescence (TXRF) spectroscopy was used to determine the concentrations of selected elements in fingernail samples collected from people residing in the Świętokrzyskie Voivodeship (Poland). The study included 25 participants: 15 men and 10 women, of whom 10 were smokers and 15 were non-smokers. Elemental analysis was performed using a Picofox S2 spectrometer, at the Institute of Physics, Jan Kochanowski University in Kielce. Due to the need to convert the material into a liquid, an effective nail mineralization procedure was developed using concentrated nitric acid (V). After 24 hours of mineralization, homogeneous solutions were obtained, to which an internal standard (gallium) was added. The following elements were quantified: Si, P, S, Cl, K, Ca, Ti, V, Cr, Mn, Fe, Ni, Cu, Zn, As, Se, Br, Rb, Sr, and Pb.

The results revealed notable differences in elemental composition between men and women, as well as between smokers and non-smokers. These changes may be linked to physiological factors, environmental exposures, diet, and lifestyle habits. The study demonstrated the utility of TXRF for the determination of trace elements in biological samples and showed the diagnostic potential of nail-based element analysis in clinical and environmental health research.

Acknowledgements: This work was co-financed by the Minister of Science under the "Regional Excellence Initiative" program (project no.: RID/SP/ 0015/2024/01). The facility functioning is supported by Polish Ministry of Education and Science (project 28/489259/SPUB/SP/2021).

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POSTER CONTRIBUTIONS

DETERMINATION OF MANGANESE OXIDATION STATE IN AN ARTIFICIAL SLAG USING XANES SPECTROSCOPY

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Slag can be valuable as a secondary raw material for the circular economy or e-mobility, as it contains useful elements such as lithium and manganese. Their reprocessing and reutilization are becoming increasingly important. Understanding the formation of slags and the influence of certain elements, such as manganese, is particularly important, as it forms different compounds and phases in slags. In those, manganese is present in different oxidation states which can be determined with X-ray absorption nearedge structure analysis (XANES). This laboratory-based XANES method can be used to analyse both, crystalline and amorphous samples, as slags often contain a mixture of them.

The specimen was prepared by sol-gel combustion methods, and the resulting precursor was subjected to different temperature profiles to change the crystallinity grade. The probed sample system consists of a slag analogue with the composition Li-Mn-Ca-Si-O (see [1]). Programme 1: Melting at 1573 K, slow cooling in the furnace at 0.3 $K \cdot min^{-1}$ for a crystalline slag, see [2]. Programme 2: Melting at 1673 K and removal of the crucible from the furnace for a fast-cooling and amorphous slag, see [1]. For the measurement, the slags were prepared with a binder in the form of pellets. A laboratory-based spectrometer (easyXES100-extended, easyXAFS LLC, Renton, USA) is used to record the XANES data in transmission mode. The oxidation state is determined by linear combination fitting (LCF) of the measured sample spectra, in comparison with measured reference spectra.

Initial results show that the analyzed slowly cooled and crystalline slag mainly matches the reference spectra of Li_2MnO_3 and $LiMnO_2$, thus revealing the presence of Mn^{3+} and Mn^{4+}. Using LCF, a composition of Mn^{3+} of 21.4±2.9 % and Mn^{4+} of 78.6±1.3 % was determined. The fast-cooling and amorphous slag also shows similarities with the references Li_2MnO_3 and $LiMnO_2$. Thus, the oxidation state Mn^{3+} and Mn^{4+} are also present here. However, the LCF shows a different composition than the slowly cooled slag, with Mn^{3+} of 91.7±2.0 % and Mn^{4+} of 8.3±2.0 %. Both slags exhibit the presence of Mn^{3+} and Mn^{4+}. However, different proportions of the oxidation state are evident. Therefore, it cannot be ruled out that different cooling rates and possibly differences in melting temperature impact the present proportions of the manganese oxidation state. It is possible that the rapidly cooled slag has a lower Mn^{4+} content because more oxygen escapes from the melt due to the higher temperature which leads to a reduction of manganese. This condition is maintained by rapid cooling of the slag. While oxygen remains in the system in the slowly cooled slag. Consequently, it is important to be able to influence the oxidation state during melting and solidification.

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TXRF AS AN ANALYTICAL TOOL TO EXPLORE THE POTENTIAL OF SEWAGE SLUGE ASHES FOR METAL REMOVAL IN WATERS

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Transforming waste materials into valuable products plays a crucial role in promoting sustainability and protecting the environment. A good example for that is the potential use of sewage sludge ashes as adsorbent materials for metal removal in contaminated water samples. Some recent studies have demonstrated that the incorporation of ash, either as-produced or after pre-activation by acidic treatment, into immobilized polymeric- beads can be a good way to increase the adsorption capacity of these materials and make them easier to be manipulated in real conditions [1]. During bead formation, sodium alginate and cellulose-based materials, along with ash (both direct and activated), are brought into contact with calcium chloride, resulting in a stable and uniform bead structure. The size of these beads is confined based on the specific application.

In the present study, both ash powders and polymeric-ash beads have been evaluated for the specific removal of zinc from aqueous solutions. Specifically, three different adsorbent systems have been considered: (1) activated ash powder, (2) activated ash immobilized with sodium alginate to form beads, and (3) activated ash combined with sodium alginate and cellulose-based materials to form composite beads. To study the adsorption efficiency of these materials, firstly the adsorption isotherms for each material were plotted and fitted. The isotherm model fitted for all 3 materials was Langmuir isotherm which is a monolayer adsorption on a homogeneous surface.

After, to study in detail the time dependency of the adsorption process, batch kinetic studies were carried out. In this context, we explored the analytical potential of TXRF to monitor the Zn content, in the supernatants obtained from the adsorption experiments, in a fast and simple way. Due to the microanalytical capability of TXRF, the possibility of using a reduced number of tubes (from 8 to 1 tube) in batch adsorption tests was also evaluated. In all cases, results were compared with those obtained by ICP-OES and good agreement was found, above all for sewage sludge beads. Additionally, in the case of activated ash powder, the direct determination of Zn adsorbed onto the material after the kinetic study was also investigated. For that, 50 mg of material were suspended in 1 mL of a 1% Triton X-100 solution and directly analyzed by TXRF. With the possibility to analyze soil suspensions, sample manipulation is reduced, less amounts of reagents are needed, and sample preparation is faster in comparison with other spectrometric techniques. Furthermore, an approximation of the multi-elemental composition of the material can be also obtained. In summary, the TXRF technique provides a simple, inexpensive and greener analytical tool with a high potential in these type of adsorption studies.

The equilibrium concentration used for kinetic studies was 30 ppm for all materials and tested for 24 hours. All 3 materials obeyed pseudo-second order kinetics. TXRF and ICP analyses showed similar adsorption trends. Ash exhibited rapid but irregular adsorption, while beads showed slower, more uniform adsorption with higher capacity and easier recovery.

Despite health and environmental risks, ash's adsorption ability enables efficient, eco-friendly contaminant removal, supporting a waste-to-waste wastewater treatment approach.

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STUDY OF ZINC ADSORPTION IN REAL-TO-LIFE MICROPLASTICS BY MEANS OF TXRF ANALYSIS

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At present, water pollution by microplastics (MPs) is a significant environmental issue, affecting aquatic ecosystems and human health. One of the main concerns about MPs is the possible enrichment of pollutants due to their large specific surface and chemical properties. In particular, the role of microplastics as vector and carrier for trace metals in the environment is still poorly understood. In this contribution, Total reflection X-ray fluorescence spectrometry (TXRF) technique was used to monitor the metal content adsorbed to the MPs through batch adsorption studies. In this case, Zn was used as a metal model due to its widespread presence in different types of natural and wastewater samples.

Usually commercial, pristine and spherical MPs prepared at specific sizes are considered in laboratory tests. Some recent publications have shown the limitations of using these commercial materials as environmentally representative MPs. For that, more “real-to-life” MPs, prepared from micronized fragments of single-use plastics, were used instead. Specifically, MPs made of polyethylene (PE), polypropylene (PP) and polystyrene (PS) with different sizes (<63 µm and 63-80 µm) were prepared and used [1]. It is interesting to mention that MPs in the natural environment can undergo aging processes that can lead to changes in the surface and physicochemical properties of MPs and their bioavailability and adsorption capacities. For that, the same MPs were also submitted to an aging process with UV light (8 weeks at 42 °C) to simulate the effect of sunlight on MPs once they have entered the aquatic environment.

To study in detail the time dependency of the adsorption process, batch kinetic studies were carried out. For all the experiments 20 mL of aqueous solutions enriched with 1 mg/L Zn were put in contact with 20 mg of MPs at different times. After the adsorption process, solutions were filtered using a 0.45 µm Nylon filter. An aliquot of 1mL of the solution was spiked at 0.5 mg/L with Rh (internal standard) and 10 µL of the standardized sample were deposited on the quartz reflector for TXRF analysis. For comparison purposes, obtained TXRF results were compared with those obtained by ICP-OES analysis. Additionally, the loaded MPs collected onto the Nylor filter were also analyzed by Energy dispersive X-ray fluorescence spectrometry (EDXRF).

Results obtained demonstrated that Zn adsorption depends on the polymer type but mostly on the aging processes, being the aged MPs ones that exhibited a higher Zn adsorption capacity. From data obtained it was also verified that the adsorption kinetics was significantly faster for aged MPs.

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OPTIMIZING ELEMENTAL ANALYSIS OF *PIPER NIGRUM* BY TXRF

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The accurate determination of elemental concentrations in food matrices is essential for assessing nutritional value, ensuring food safety, and evaluating potential health benefits. In this study, the effect of different milling time and consequent particle size distibution on the accuracy of the elemental analysis of black peper (*Piper nigrum*) was evaluated. Pepper samples were ground for 5, 10, and 15 minutes and then analyzed by total reflection X-ray fluorescence spectrometry (TXRF) to quantify nutritionally and toxicologically relevant elements such as Mn, Fe, Ni, Cu, Zn, Rb, and Sr [1]. At the same time, the particle size distribution was measured to evaluate its effect on the accuracy of the elemental analysis by TXRF. The results showed significant variations in element concentrations associated with grinding time, suggesting both mechanical alteration of the sample matrix and potential contamination by the grinding equipment during prolonged processing. Particle size analysis showed considerable variability, highlighting the inherently random nature of the grinding process. In particular, the 5-minute grinding samples showed the widest distribution and the largest particle fragments. After 10 minutes, differences were observed mainly in the D90 values, while after 15 minutes the variability became smaller but was still observed in the D50 and D10 ranges.

These results underline the need for standardized grinding procedures in analytical workflows, as both element recovery and particle uniformity are influenced by grinding time. In addition, the random nature of the grinding process must be taken into account when interpreting elemental data from ground food samples, especially when trace-level accuracy is required [2].

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TEMPERATURE AND IKRUM DEPENDENCE IN TIMEPIX 3 USING KATHERINE READOUT & EVALUATION ON EFFICIENCY OF THE LINEAR COMPENSATION METHOD.

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The Timepix3 detector and Katherine readout system form a powerful duo for space exploration. Timepix3 captures high-resolution data on cosmic rays and X-rays, while Katherine enables remote control and data analysis—crucial for spacecraft safety. This setup is vital for studying radiation and protecting astronauts and electronics, especially in extreme space temperatures. This study investigates the effect of temperature fluctuations on the energy analysis of the XRF material spectrum as well the alpha particles using the Timepix3 detector and the Katherine readout system. The experiments were carried out in a controlled climate chamber, with temperatures ranging from 10°C to 40°C for material spectrum analysis and from -40°C to +60°C for comparison of equalization results. Equalization parameters, such as adjustment bits, masked pixels, and threshold settings, were analysed and stored within these temperature ranges. The performance of the Timepix3 in both Time-Over-Threshold (TOT) and Photon Counting (PC) modes was evaluated, focusing on environmental temperature variations and IKrum adjustments. IKrum is a leakage current compensation which impacts the output of the Charge Sensitive Amplifier (CSA). Two Equalization approaches were tested: (1) applying room-temperature equalization across varying temperatures and IKrum values, and (2) re-equalizing at each temperature. Both methods showed energy shifts and changes in spectral resolution due to instability in the detector and fluctuations in noise. It also evaluates the global linear compensation method's efficiency in enhancing detector performance under varying thermal conditions and IKrum inputs. Moreover, the study aims to investigate whether the cluster shape and energy of alpha particles changes depend on the distance between source and detector as well as the analysis of the changes in energy, under various environment conditions and detector setting as XRF material spectrum analysis. Through the experimental investigations using Timepix3 within controlled temperature environments, this study focuses on the interaction between IKrum variations, temperature fluctuations, and the precision of spectrum analysis using the global linear compensation method, aiming to improve data precision essential in fields such as space radiation measurement where the Timepix 3 is crucial.

MECHANISM UNDERLYING TRANSPORT OF AN ANTICANCER COPPER(II) COMPLEX INTO A549 LUNG CANCER CELLS AND ACROSS CELL MONOLAYERS

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Copper complexes with promising anticancer properties must have a well-understood mechanism of penetration into cancer cells. In these studies, the ability of copper(II)/1-allylimidazole complex [Cu(1-allim)$_4$(NO$_3$)$_2$] to enter cancer cells and diffuse through the monolayer was investigated using laser interferometry, TXRF, flow cytometry, and TEM. Since copper complexes must cross the plasma membrane to enter the cell, studies of the interactions between membrane lipids and copper complexes are necessary to fully understand the mechanism underlying their efficacy. The [Cu(1-allim)$_4$(NO$_3$)$_2$] complex studied in this document penetrates A549 cancer cells. Studies using laser interferometry, flow cytometry, TXRF and TEM have shown that the complex diffuses well through the cell monolayer and enters the cancer cells. Studies with a liposome model (fluorescence spectroscopy and DLS) have shown that the [Cu(1-allim)$_4$(NO$_3$)$_2$] complex crosses the membrane by endocytosis and passive diffusion, without damaging the membrane. The obtained results facilitate the understanding of the penetration of copper complexes with anticancer properties into cells.

DETECTION OF SMALL AMOUNTS OF METAL IONS USING FLUORESCENT SENSORS AND THE TXRF METHOD

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A novel fluorescent sensor based on a pyrazoloquinoline derivative, referred to as PQL3 (Figure 1) was synthesized. The structure of the compound suggests its potential for cation detection. The spectroscopic properties of PQL3 were investigated using UV-Vis spectroscopy (absorption and steady-state fluorescence). PQL3 absorbed violet light and emitted green light. A low fluorescence quantum yield was observed in polar solvents. In the next step, the fluorescence response of the sensor dissolved in acetonitrile was evaluated in the presence of various metal ions such as Zn^{2+}, Ca^{2+}, Pb^{2+}, Mg^{2+} and Ba^{2+}.

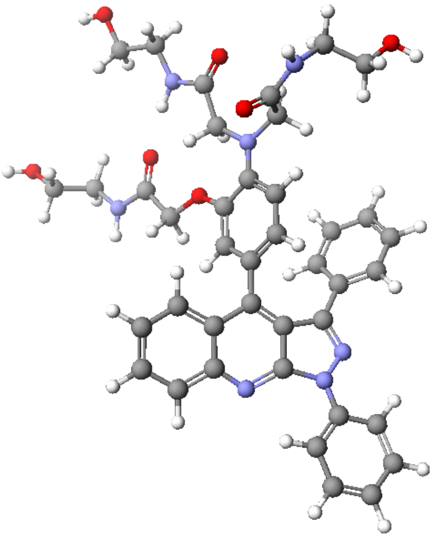

Figure 1: Structure of a fluorescent sensor based on a pyrazoloquinoline derivative substituted with phenols at position 1 and 3 and phenol with 2-hydroxyethylamide and N-(2-hydroxyethyl)-2-hydroxyacetamide at 4 position.

PQL3 in acetonitrile gave a fluorescent response to all tested ions. Due to the lack of selectivity, the possibility of using the sensor to determine very low concentrations of the tested ions was investigated. The dependence of fluorescence intensity on the concentration of each ion was determined and the detection limits of these ions were determined using PQL3.

Finally, the concentrations of Zn, Ca, Pb, Mg, and Ba were compared with results obtained using the Total Reflection X-ray Fluorescence (TXRF) method, conducted with a commercially available TXRF Picofox S2 spectrometer (Bruker) at the Institute of Physics, Jan Kochanowski University in Kielce. This system uses a 50 W excitation lamp with a molybdenum anode, operating at 50 kV and 1000 µA. Fluorescent X-rays from the sample were detected using a Peltier-cooled silicon drift detector (XFlash) with an energy resolution of approximately 149 eV for the Mn–Kα line. Qualitative and quantitative analyses of characteristic spectra were performed using the Spectra 7 software supplied with the spectrometer.

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A HIGHLY EFFICIENT DOWNCONVERTER FROM Er^{3+}, Yb^{3+}, AND Ce^{3+} DOPED PHOSPHATE GLASS FOR NIR LASERS

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Being one of the most important tools in industrial and medical fields, the red-NIR laser has caught a lot of attention in research studies to improve its emission efficiency and the stability of its lasing materials, both thermally and mechanically. In this regard, to enhance the emitting intensity of the redNIR laser generation from Er3+ ions inserted into a glass network, the effect of Yb3+, Nd3+, and Ce3+ ions on the emitted laser beam were studied. First, a host glass network of 44P2O5-15ZnO10Pb3O415NaF-15MgF2-1Er2O3 (PZPbNMEr^{3+}) was proposed as a red-NIR lasing material and was reinforced by 0.5 and 1 mol% of Yb3+ (PZPbNMEr^{3+}:0.5Yb^{3+} and PZPbNMEr^{3+}:1Yb^{3+}), Nd3+ (PZPbNMEr^{3+}:0.5Nd^{3+} and PZPbNMEr^{3+}:1Nd^{3+}), or Ce3+ (PZPbNMEr^{3+}:0.5Ce^{3+} and PZPbNMEr^{3+}:1Ce^{3+}) ions (labeled as PZPbNMEr^{3+}:RE^{3+}).

The structural variations resulting from compositional changes were examined by XRD, density, FTIR, and Raman spectra, which showed an increase in the glass network tightness. Here, the Raman measurements were not suitable for examining the structural properties of such glasses, as the used wavelength excited the studied glasses, producing photoluminescence instead of obtaining the characteristic bands of the glass network.

The glass tightness was positively reflected in the thermal stability and elasticity of the considered glasses, reflecting their suitability as lasing media. Optically, all the distinctive absorption bands of the Er3+, Yb3+, Nd3+, and Ce3+ ions appeared in the optical absorption spectra in the region of 200–2500 nm. Additionally, a significant impact of the induced strain or crystal field of the added RE3+ ions on the optical properties were observed.

A successful excitation was conducted by the green light of 525 nm to the host glass PZPbNMEr^{3+}, generating five wavelengths, one in the red region at 631 nm and four in the NIR at 748, 801, 1034, and 1527 nm. A significant enhancement to the intensity and red shift of the formed bands were observed with the inclusion of Yb3+, Nd3+, and Ce3+ ions, and new NIR bands were generated. Additionally, an energy transfer between Er3+/Yb3+ and

Er3+/Nd3+ was observed. Accordingly, the considered PZPbNMEr^{3+}:RE^{3+} glasses had highly efficient photoluminescence in the red-NIR region alongside their high thermal stability and good elasticity, making them a suitable choice for laser media.

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DEVELOPMENT OF AN XRF MEASUREMENT CELL FOR ELEMENTAL QUANTIFICATION AND MAPPING OF NEXT-GENERATION BATTERY MATERIALS

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Quantitative elemental analysis and spatial distribution in in-situ and operando conditions are crucial for understanding the composition and chemical processes in next-generation battery systems. Total reflection X-ray fluorescence (TXRF) and micro-X-ray fluorescence (µXRF) can be used as powerful techniques for analyzing trace impurities and monitoring charge carriers and the dissolution of active elements in electrodes, separators, and electrolytes, providing a foundation for optimizing battery efficiency and ensuring long-term reliability[1-3]. This work presents the design, fabrication, and testing of an easily assembled XRF measurement cell compatible with in-situ battery investigation, incorporating a Kapton® window as an X-ray-permeable interface. Elemental quantification and mapping along longitudinal axis of sodium-ion Swagelok battery cells at various operational states were performed, revealing that the use of thin Kapton® foils (~25-50 µm) enhances analytical accuracy of active material detection under in-situ condition. The developed measurement cell offers a practical and accessible laboratory-scale alternative to synchrotron-based XRF facilities, overcoming challenges associated with complex instrumentation, high operating costs, and limited beamtime availability.

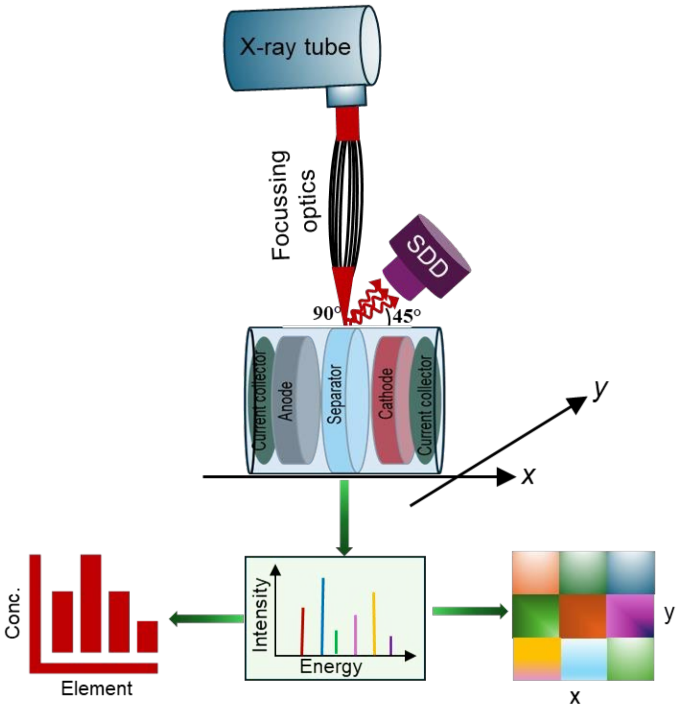

Figure 1: Schematic diagram showing elemental quantification and mapping investigation of a battery cell aligned in the in-house developed XRF measurement cell.

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DETERMINATION OF POTENTIALLY TOXIC ELEMENT CONTENT IN WILD MUSHROOMS AND ITS POTENTIAL BIOACCESIBILITY TROUGH TOTAL REFLECTION X-RAY FLUORESCENCE

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Forest wild mushrooms grow widely under suitable conditions of humidity and temperature. Many mushroom species are edible, both for animals and humans, and are essential components of numerous culinary traditions. Some may have toxic properties, either due to their composition or their bioaccumulation potential of potentially toxic elements (PTE's) [1].

From an environmental perspective, fungi could be useful in the cleanup process of contaminated soils, given their ability to accumulate metals and survive in adverse conditions, where the plants used for bioremediation are not sufficiently resistant. Various X-ray fluorescence techniques are suitable for this type of study, as they do not require chemical sample preparation. The simultaneous detection of all elements is another advantage. However, most XRF instrumental designs have limited sensitivity (1 $\mu g\ g^{-1}$), insufficient for many elements, especially those with high toxicity, even at low concentrations [2]. In this context, the use of TXRF analysis, with its greater sensitivity and ease of sample preparation, seems appropriate not only for a more precise elemental determination, but also to provide data of interest for bioavailability studies.

In this work, using a TXRF instrumentation (W target tube), we studied metal uptake in sixteen edible and non-edible mushrooms species, collected in different areas of Catalonia (NE, Spain). Samples were collected from both mining-contaminated and uncontaminated sites. Prior to the analysis, samples were prepared by suspension method [3], but also the bioaccesible concentrations of metals and metalloids were determined using the HCl digestion single extraction method proposed by Baek et al [4].

Mushroom samples collected from mining waste areas show higher levels of metals such as copper, zinc, selenium, and lead. Also notable is the presence of silver and cadmium, which, using the aforementioned instrument setup, can be determined by their characteristic K-α lines.

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EVALUATION OF THIOL SELF-ASSEMBLED MONOLAYER ON GOLD NANOPARTICLES USING TOTAL REFLECTION X-RAY FLUORESCENCE ANALYSIS

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This study presents a simple method for the evaluation of thiol self-assembled monolayers (SAMs) prepared on gold nanoparticles on a substrate using total reflection X-ray fluorescence (TXRF) analysis. SAMs are applied to various sensors using gold nanoparticles, and their properties such as the quantity of thiol molecules are needed to evaluate for accurate sensing. In this study, gold nanoparticle substrates were produced based on Ref. [1] as follows: First, a quartz glass substrate with a dry residue of 10 □L of an $HAuCl_4$ solution and an indium-tin oxide (ITO) substrate were placed facing each other in a container. Then, both substrates were simultaneously heated for 20min in air. The heating temperature increased from room temperature to about 170°C in 3 min, and it reached about 180°C in 20 min after the heating started. To prepare SAMs on gold nanoparticles produced on ITO substrates, gold nanoparticle substrates were incubated in 1 mM 1-dodecanethiol (1-DT) or 1 mM 4-mercaptobenzoic acid (4-MBA) ethanol solution overnight and washed by ethanol. TXRF analysis of the substrates was carried out by a portable TXRF spectrometer [2] in air for 600 s. As shown in Figure 1, the peak intensity corresponding to S Kα lines increased due to thiol molecules, which was observed as the peak broadening of Au Mα, Mβ lines from the gold nanoparticles. These results suggested that TXRF analysis can be applied to confirm the formation of SAM on gold nanoparticles.

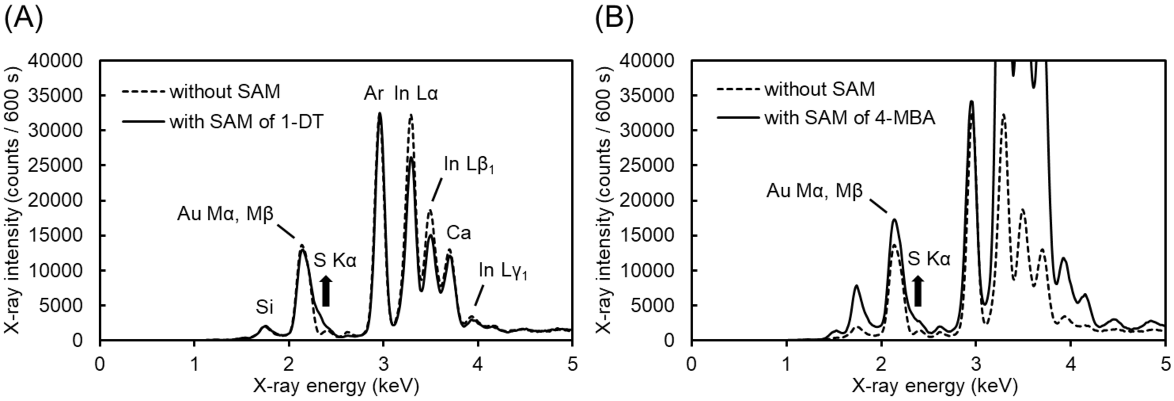

Figure 1: Representative TXRF spectra of SAM of (A) 1-DT and (B) 4-MBA on the gold nanoparticles in the Xray energy range up to 5 keV.

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CHARACTERIZATION OF THE ATI TXRF SPECTROMETER FOR THE DETECTION OF LOW Z ELEMENTS

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TXRF offers distinct advantages for detecting low-Z elements excited by X-rays. These elements emit fluorescence at relatively low energies—typically in the range of a few hundred eV—and their signal intensities are significantly weaker compared to those of medium-Z elements. To achieve reliable measurements, minimizing absorption is essential.

Air absorption can be effectively eliminated by conducting measurements under vacuum conditions. Additionally, self-absorption within the sample is reduced by applying it as a thin film. To further enhance sensitivity to low-energy radiation, a detector equipped with an ultrathin window (UTW) is required. In this setup, a UTW made of 40 nm silicon nitride (Si_3N_4) coated with 30 nm of aluminum minimizes energy loss at the detector entrance.

A specially designed TXRF vacuum chamber was developed for this purpose. It features a 1900 W Cr X-ray tube and an internal Ni/C multilayer monochromator [1,2]. The chamber is adapted to house a 70 mm^2 silicon drift detector (SDD, Amptek OEM module) with the aforementioned UTW. The detector is mounted with a custom-designed electron trap and collimation system tailored to the collimated active area of 50 mm^2, ensuring precise beam alignment and minimized background noise.

This vacuum chamber accommodates flexible sample holders of various shapes, as no fixed reference plane is required. Motorized linear and rotational stages—controlled via PC— enable automated adjustments of the X-ray footprint and incident angle, allowing for automated angular scans. Beam positioning is monitored through a fluorescence screen inside the chamber, which is observed using a CCD camera.

With these enhancements, the system is capable of detecting elements down to carbon. Detection limits, sensitivities, and quantitative performance data will be presented.

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THE ANALYSIS OF SPIONS METABOLISM AND TOXICITY BASED ON THE ELEMENTAL COMPOSITION OF BLOOD SERUM DETERMINED WITH THE TXRF METHOD

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Due to its properties, the total reflection X-ray fluorescence (TXRF) spectroscopy enables analysis of small volume liquid samples with low concentration of elements, including elements occurring at trace levels. For this reason, it is an ideal method for measuring the elemental composition of biological samples such as blood or plasma.

Superparamagnetic iron oxide nanoparticles (SPIONs) due to their small size, high surface-to-volume ratio and unique physicochemical properties open up a number of application possibilities in biomedicine. They can be used as MRI contrast agents, targeted drug carriers, as well as cancer therapy agents using the phenomenon of local hyperthermia. On the other hand, the same SPIONs properties are associated with an increased risk of toxicity for cells, tissues or organs. Studies conducted so far, based on animal models, indicate that SPIONs can disturb the elemental homeostasis of various organs.

In this project the TXRF and vibrational spectroscopy methods were used to verify whether the administration of such nanoparticles to animals can also affect the elemental and molecular composition of serum. Data obtained in this project can be a source of information about metabolism and potential toxicity of drugs based on iron oxide nanoparticles.

Comparison of plasma composition of female and male specimens indicated naturally occurring gender differences. After administration of nanoparticles to animals changes in levels of elements involved in iron metabolism, such as copper, were observed. A decrease in the levels of selenium and zinc, elements that play important roles in the immune system and have antioxidant effects, was also observed throughout the experiment. Changes in molecular composition such as the decrease in the level of lipids relative to proteins after SPIONs administration were observed, among others, using additional methods of vibrational spectroscopy.

PRODUCTION OF A MULTIFUNCTIONAL PLANT CRM WITH CERTIFIED CONTENT OF SELECTED ELEMENTS FOR USE IN SPECTRAL ANALYSIS

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In laboratory practice, it is necessary to use reference materials whose composition, due to the level of analyte concentrations and the nature of the matrix, corresponds as closely as possible to those of the tested analytical samples. Reference materials are used at all stages of the measurement process, including calibration of measuring equipment, method validation, and quality control of results. They are also used in interlaboratory comparisons and in assessing the proficiency of participating laboratories. A reference material fulfills its function only when each user receives an object with the same properties for testing. This can be achieved in two ways: by distributing the same sample of the material or by distributing material samples with the same parameters (homogeneous, stable during storage, stable from the moment of production to use).

The homogeneity assessment process involves comparing the results obtained from randomly taken samples of the bulk item of reference material. This process takes place after the material has been placed in the appropriate containers and includes testing of two types of homogeneity: within-bottle homogeneity and between-bottle homogeneity. In order to minimize the impact of potential inhomogeneity within a single package on the certification result of a given characteristic, it is necessary to determine the minimum amount (mass) of the reference material sample, assuring the same properties. This value is evaluated by the producer of CRM's and included in the uncertainty budget of the certified value.

In the described case, means, strawberry fruits and leaves, enriched with emerging elements, As, Cd, Hg and Pb, both expected to be the measurements towards the examination of their homogeneity was performed. The vessels with the material were divided into nine sets. One vessel was randomly selected from each set. Four samples were taken from each vessel. Each portion of material was subjected to microwave-assisted digestion in a closed system. Quantitative analysis of selected elements in the samples was performed using inductively coupled plasma mass spectrometry (ICP-MS). Then, an assessment of homogeneity was performed, including statistical analysis of the results using Grubbs and Shapiro-Wilk tests using two-factor ANOVA analysis and estimation of uncertainty related to the material's inhomogeneity.

Acknowledgments

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DEVELOPMENT OF A SOFTWARE FOR SIMULATIONS OF X-RAY REFLECTIVITY CURVES

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The X-ray reflectivity (XRR) technique, theoretically developed by Parratt [1], is a well-established, nondestructive and non-contact [2] structural characterization method used to study the properties of materials relevant to many research fields, such as physics, chemistry, biology, and materials science. This method allows the determination with high precision of the density, thickness and roughness of the layers both for films and multilayer samples. Determining such nanolayer properties is crucial in most technological applications of thin films [3] due to the fact that the properties of thin layers depend on their thickness.

The idea of XRR is to register the intensity of primary X-ray after reflection from the sample surface as function of the incidence angle, the values of which range from 0° to approximately 3°-4°. Changes in measured reflectivity indicate differences in density, thickness, roughness and period of the nanolayer samples. Therefore, it is crucial to correctly model the experimental XRR curves.

Presented studies concentrate on the development of a software for simulations of X-ray reflectivity curves. The software implements the recursive method calculation of XRR which considers the effects of dynamic scattering and absorption. The reflectivity coefficient is calculated for a multilayer structure having N layers as a function of the incidence angle θ. The recursive calculation starts from the last layer, then the values of reflectivity are calculated on each interface. Structural imperfections, such as interface roughness affecting the X-ray reflectivity curves, were implemented in the calculations. Finally, the reflectivity of the whole multilayer system is calculated in dependence of the incidence angle.

Studies present the technical aspects of the software and XRR curves calculated for different bulk samples, mono- and multilayers systems with various properties (density, thickness and roughness of interface). Systematic discussion of XRR calculations as well as the factors influencing the XRR curves will be presented and discussed.

The calculated XRR curves were applied for modeling the experimental data collected at the XRF beamline at Elettra Sincrotrone Trieste. In the XRR measurements, the silicon (Si), titanium (Ti), gold (Au) and molybdenum (Mo) bulk samples were studied. The XRR curves were also measured for multilayer structures, including Ti, Au, titanium dioxide (TiO_2) and Pd films of varying thicknesses (from 1 nm to 100 nm) deposited on a quartz or silicon. The characteristics of nanolayers: density, thickness and roughness, were estimated from XRR curves using the developed software. The obtained thicknesses of the layers were compared with nominal values. In the study, also uncertainties of the determination of the nanolayer characteristics were discussed.

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AN INTERLABORATORY COMPARISON FOR XRF UNDER GRAZING INCIDENCE CONDITIONS (TXRF, GIXRF AND ANGLE-RESOLVED XRF)

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X-ray fluorescence (XRF) analysis under grazing incidence (GI) enables reliable and reproducible determination and quantification of ultra-trace elements in a sample. It also allows for detailed characterization of the three-dimensional elemental distribution, including both lateral spatial mapping and depth profiling. The fundamental quantification principle involves correlating the X-ray fluorescence intensities – which vary laterally depending on the varying angles of grazing incidence of the exciting X-rays - with the total elemental content as well as the spatial distribution within the sample. XRF under grazing incidence is a powerful technique that can be employed across a broad range of scientific and industrial applications.

Currently there is an ongoing project within the German WIPANO program to design, prepare and characterize samples for an international interlaboratory comparison. Targets of this project are studies about artificial multi-element thin film samples with different mass depositions on different substrates, covering different angular regimes of XRF (e.g. GIXRF and angle-resolved XRF), as well as challenges in terms of dimensional sizes, matrix variations and complexity in quantification. These samples will be thoroughly pre-characterized by means of X-ray reflectivity (XRR), reference-free XRF [1] and GIXRF and then distributed to all interested participants of a round-robin activity.

In this work, we discuss the design ideas and principles behind these rather complex samples. We also present plans of the interlaboratory comparison and show initial results of theoretic and experimental validations.

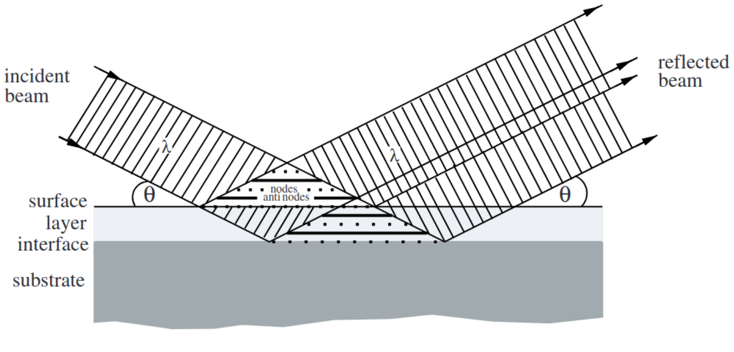

Figure 1: X-ray standing wave (XSW) field for a flat surface in the case of grazing incidence [2].

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TOTAL REFLECTION X-RAY FLUORESCENCE ANALYSIS OF BIOLOGICAL SAMPLES SLICED USING MICROTOME

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We have studied direct Total Reflection X-ray Fluorescence (TXRF) analysis of biological samples. Previously, we investigated an analyzing volume for the TXRF spectrometer (NANOHUNTER-II, Rigaku, Japan) [1]. The analytical thickness for this TXRF spectrometer was experimentally evaluated to be approximately <15 µm. Thus, the biological samples should be sliced in thin layer.

Broccoli sample was sliced with a thickness of 20 µm using a medical microtome (LS-113, Yamato Koki Industries, Japan). This slice was placed onto a quartz glass using a tweezers. Vanadium standard solution was directly dropped onto the slice. After dried, the final thickness was about 15 µm. In addition, several organs such as kidney and liver from the mouse were also sliced and kept on slide glass substrates. The samples were analyzed by the TXRF spectrometer with a measurement time of 300 seconds. The glancing angle was set to 0.025° for Mo Kα.

TXRF analysis of the broccoli samples at different sample locations was successfully performed. The quantitative results fit well with the results obtained by ICP-MS. This sample preparation method would be useful for reducing a sample preparation time by eliminating complex pretreatment procedures for TXRF analysis of various biological samples.

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TXRF ANALYSIS OF AIRBORNE PARTICULATE MATTER ORIGINATING FROM RESUSPENSION AND COMBUSTION

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Air pollution is a key environmental concern. Particulate matter (PM) is among the most harmful pollutants in ambient air, especially fine ($PM_{2.5}$) and ultrafine ($PM_{0.1}$) particles. In addition to direct (tailpipe) traffic emissions, ”fugitive emissions” such as tyre and brake wear, and indirect emissions caused by the resuspension of road dust make up a main fraction of respirable aerosol particles [1]. To have insight to chemical characterization of a major source of the resuspendable urban road dust, namely construction and demolition works, road dust samples were collected at construction sites and nearby urban sites in Veszprém (Hungary). Powdered construction materials were measured for comparison.

Domestic combustion of solid fuel (coal, wood, or occasionally domestic waste) is a significant air pollution source in rural settlements, especially during the heating season. The emission of domestic stoves burning different solid fuels was tested using a dedicated laboratory experiment.

A 9-stage May-type cascade impactor was used for sampling both resuspended road dust and diluted flue gas of the stove combustion experiment. Si wafers suitable for subsequent TXRF analysis were applied as collection surfaces. The combination of cascade impactor sampling and TXRF analysis was found to be an effective tool for determining the mass size distribution of chemical elements or compounds in a wide size range of 70 nm to 10 µm [2,3].

Based on the results of size-fractionated elemental analysis, it was found that in case of firewood and forest wood burning, the K, Cl, Br and V emissions are significant in the size ranges below 1 µm, whilst for brown coal there is an order of magnitude higher S emission compared to other conventional fuels. During the burning of rubber high concentration of Ca was detected in the size range of 2-4 µm. Elements characteristic for resuspended road dust (Ca, Fe, Zn and Pb) were concentrated in the coarse fraction (2-10 µm).

Road dust and construction material samples were also analyzed in the suspension form using powdered geological standard reference materials for validation. The elemental composition obtained by suspension TXRF was in line with conventional XRF results of pelletized samples.

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APPLICATION OF BAYESIAN ESTIMATION FOR TXRF ANALYSIS

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Total reflection X-ray fluorescence (TXRF) analysis is used for determining trace elements in various samples. In TXRF analysis, an incident X-ray beam irradiated to a dried residue on a substrate, and emitted fluorescent X-rays are counted in a detector such as silicon drift detector (SDD). A glancing angle of incident X-rays is low for occurring a total reflection phenomenon. As a portable TXRF instrument has been developed, we can perform on-site and screening analyses. When a screening inspection is performed at a river etc., it is important to shorten the measurement time in TXRF analysis. Previously, one of author has successfully applied Bayesian estimation to an XRF analysis1), and in this study, it was employed to TXRF spectra for shortening a measurement time.

Portable TXRF instrument 200 TX (Ourstex Co. Japan) was used. The target of the X-ray tube is W, and an effective area of equipped SDD is 30 mm^2.To apply Bayesian estimation, the Poisson distribution was defined as the likelihood function, and a relationship between a count rate and its frequency in the TXRF spectrum was used as the prior distribution. The posteriori distribution was obtained for each TXRF spectrum, and then an expected value was calculated. The real water from Nagara River, as shown in Fig. 1, was collected as the sample and was used.

We analyzed Cl peak in TXRF spectrum of the river water. To obtain net intensities, Gaussian fitting was performed. To compare the results with and without Bayesian estimation, the net intensity of Cl Kα peak was divided by that of Ar Kα peak2). When the measurement time was set to 5 s, recoveries of net intensities with and without Bayesian estimation were 115 ± 38% and 146 ± 50%, respectively. In this poster presentation, we will discuss the results of standard solutions etc.

Acknowledgements: We thank the River Foundation, Japan, for the kind offer of financial support.

Fig.1 Photograph of Nagara river

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POSSIBILITIES OF APPLICATION OF TXRF IN THE DIAGNOSIS OF THE ELEMENTAL COMPOSITION OF HUMAN BIOLOGICAL MATERIAL

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Analysis of the elemental composition of human biological samples is an important element of medical diagnostics. Typically analyzed human biological samples are blood, serum, urine, or hair, and routinely determined elements include iron, calcium, magnesium, phosphorus, sodium, and potassium. Additional information about the state of the body is obtained by analyzing the content of trace elements such as copper, zinc, and selenium. In determining the content of elements in human biological samples, the total reflection X-ray fluorescence analysis (TXRF) [1] can be used. The possibilities of this technique application are, however, limited by the detection limit achieved, which depends on the atomic number of the element, the type of the analyzed sample and its preparation method, the X-ray spectrometer used, and the measurement parameters.

This work discusses the physical basis of the TXRF technique, the construction of the experimental system, the preparation of samples of human biological material and the method of performing the analysis of the elemental composition of such samples. Then, the focus is on the discussion of the detection limit of the TXRF technique depending on the type of sample for which the elemental composition is determined and the method of sample preparation. The detection limit was determined in a wide range of atomic numbers of the analyzed elements, and using TXRF spectrometers with different parameters of the measurement system. The determined values of the detection limit of elements were compared with the reference values of the content of elements in samples of human biological material.

The information obtained will be used in routine analysis of the elemental composition of samples of human biological material from patients of the Holycross Cancer Center in Kielce.

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UNLOCKING PRECISION IN HYDROXYAPATITE ANALYSIS WITH TXRF: ADDRESSING CHALLENGES IN Ca/P QUANTIFICATION

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Hydroxyapatite (HA) is a calcium phosphate ceramic material widely used in orthopedic and dental applications, especially as a coating for metallic implants [1]. Its biological performance is strongly dependent on its chemical purity and the Ca/P molar ratio. Therefore, a rapid, precise, and cost-effective method for Ca/P quantification is essential, particularly for routine industrial quality control [2].

This study investigates the application of Total Reflection X-Ray Fluorescence (TXRF) spectroscopy as an alternative analytical approach for determining the Ca/P ratio in HA. A certified reference material with known Ca and P content was used for validation. A series of analytical challenges, such as low phosphorus sensitivity, element crystallization during drying (Figure 1a), and internal standard selection, were addressed. TXRF results were compared against classical and modern methods including gravimetry, photometry, titrimetry, ICP-OES, FAAS, and WD-XRF (Figure 1b).

The TXRF method showed comparable or superior precision and accuracy, with lower sample consumption, simpler preparation, and faster analysis time. Optimized sample preparation, including the use of vacuum drying and potassium dihydrogen phosphate as a combined internal and standard addition reference, significantly improved measurement repeatability and accuracy.

The study concludes that TXRF is a viable method for routine characterization of HA materials. The technique offers multi-element detection capabilities, minimal matrix interference, and high reproducibility. By overcoming limitations related to phosphorus quantification and crystallization, the developed TXRF method supports reliable and efficient quality control of synthetic hydroxyapatites.

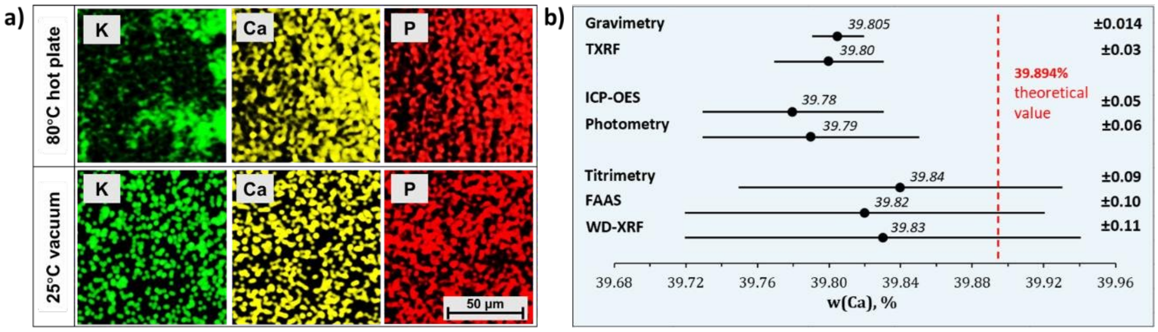

Figure 1: (a) The influence of droplet drying conditions on the distribution of elements on the sample carrier, (b) Comparison of calcium quantification results by different methods

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TXRF-BASED TRACE ANALYSIS IN DOPED CARBON QUANTUM DOTS

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Carbon quantum dots (CQDs) doped with heteroatoms such as nitrogen, sulfur, silver, and iron exhibit enhanced properties for biomedical, optical, and electronic applications. Reliable quantification of trace elements is essential for quality control and regulatory compliance, especially in sensitive applications [1]. In this study, Total Reflection X-ray Fluorescence (TXRF) was employed for ultra-trace elemental analysis of doped CQDs synthesized via a green hydrothermal method using plant-derived carbon sources. TXRF enabled simultaneous multi-element detection with minimal sample preparation and excellent reproducibility [2].

Detection limits for key dopants were found below 0.1 µg/mL, and the results showed strong agreement with ICP-MS data. In addition to establishing dopant concentrations, TXRF allowed for batch-to-batch consistency assessment, critical for scalable production [3]. The data also provided insights into the correlation between dopant concentration and the optical behavior of CQDs, supporting further tailoring of functional properties. This work demonstrates the power of TXRF as a standardization tool for nanomaterial characterization and proposes its integration into industrial QC protocols.

Furthermore, the TXRF method was applied to investigate surface contamination and unintended elemental residues from synthesis and purification processes. These findings underscore the necessity of comprehensive elemental profiling for applications in biomedicine, where cytotoxicity can be induced even at trace levels. The study highlights TXRF's role in advancing safer and more effective use of doped CQDs in commercial and clinical domains.

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FROM AIR TO ELEMENT: EXPLORING TXRF CHARACTERIZATION OF PARTICULATE MATTER COLLECTED VIA LIQUID-PHASE BIOSAMPLING

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Airborne particulate matter (PM) is generally collected using high- or low-volume samplers that capture particles on filter membranes (such as quartz, PTFE, or glass fiber filters) over a set sampling duration. Standard samplers, such as cascade impactors, are used to separate particles by size, with a focus on PM_{10}, $PM_{2.5}$, and PM_1 fractions, which are important for human health and environmental legislation. Conventional sampling methods require long sampling durations (8 or 24 hours) [1] and intensive sample handling. Additionally, filter-based systems might suffer from sample loss, contamination, or analyte degradation. The traditional approach is primarily designed for chemical analysis and frequently does not consider the biological components (e.g., bacteria, fungi, viruses) found in bioaerosols [2], limiting the usefulness in integrated environmental health research.

This study focuses on the use of Total Reflection X-Ray Fluorescence (TXRF) spectroscopy for elemental analysis of PM collected in a liquid media utilizing the Coriolis® (Micro) cyclone-based biosampler. The work aims to develop and optimize sample preparation methods for environmental liquid samples collected in both indoor and outdoor environments. To address issues with high sample variability, two different preparation methods were tested: lyophilization and pre-concentration. Reseults show that TXRF is an appropriate and effective technique for detecting and quantifying metal concentrations in samples, with clear variations between indoor and outdoor conditions. Notably, outdoor samples revealed higher quantities of elements (Ca, Fe, Pb), emphasizing environmental variability and the impact of pollution sources.

In addition to its suitability for chemical analysis, the use of the Coriolis® µ biosampler enables simultaneous biological investigations, such as bacterial identification and aerobiome profiling, making it a versatile tool for integrated environmental monitoring.

This research is part of the MY AIR project, a collaboration between the University of Brescia (UNIBS) and the Polytechnic Institute of Guarda (IPG) aimed at improving bioaerosol monitoring technologies and promoting multidisciplinary environmental health research.

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