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Chemical analysis techniques and radioactive emission based measurements: A Review

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Abstract
Recently, rays have been widely used in the analysis of materials, including chemicals, because of their high properties in quantitative and qualitative analysis, by determining the type and quantity of the material, and among these techniques are the use of Atomic Absorption Spectroscopy, X-Ray Fluorescence Spectroscopy Chromatographic Techniques, Gas Chromatography, Liquid Chromatography, so this study aimed to show the types of techniques used in chemical analysis.

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1. Introduction

Recently, the modification of physical properties and the alteration of the chemical content of different materials by various production processes have gained interest scientifically and industrially. During production, product quality is as important as the achievable amount of product. Therefore, making more precise and efficient measurements and using the collected data for quality control purposes will be beneficial for all industry sectors. As a result, chemical analysis will permit one to investigate different fields of technology in order to define the amount of substance that defines the quality of the final product in a non-destructive manner. Indeed, the chemical behavior, the structural form, and the elemental composition are the most significant parameters for material quality. There are many well-known techniques and methods that can be used to analyze the identity of a certain material, to define a structure, to quantitatively and qualitatively determine the elemental composition, to identify the crystallinity of the material, and to define the homogeneity or natural variations of the minerals in geological matrices. The understanding of the material and the recognition of its quality are generally determined by these aforementioned characteristics. (Ivleva, 2021) [23] (Fitzgerald *et al.*, 2020) [13] (Jalili *et al.*, 2020) [24].

2. Fundamental Principles of Chemical Analysis

Such a broad spectrum of methods contributes to the comprehension of the knowledge and practice of our study and appears important for that reason. In this section, some very general statements on the foundations of quantitative analysis are prepared, even some very speculative ones, too, and then a broad variety of methods are all discussed in order to put the rest of our study in the perspective of Chemical Analysis (Alam, 2020) [1].

The setting developed and used for comparative purposes throughout this little book is a graduate course on Chemical Analysis. Chemical Analysis, occasionally termed Quantitative Analysis, is the science and art of finding what matter is present in a substance, how much matter is present and, for determination, the identity of the matter and the magnitude of the amount must be determined. Chemical analysis encompasses a very broad variety of experimental techniques, some simple and some complex. The spectrum of methods extends from simple gravimetric methods which have been in systematic use for two or more centuries, and less precise but cheaper wet-chemical methods carried out up to a few decades ago, to very sophisticated modern instrumental techniques with writing, electrical, electronic, or radiochemical signal readout, which have greatly changed the nature of chemical analysis over the past quarter of a century (Jespersen & Hyslop, 2021) [26] (Ayres *et al.* 2021) [5] (Chen & Yu, 2021) [10].

3. Spectroscopic Techniques

An attractive feature of optical spectroscopy is that it allows the determination of the wavelength or the energy of the lines, which can be associated with each element and its oxidation state. Therefore, the element and the oxidation state can be determined by measuring the energy of the X-rays emitted in different processes, like bremsstrahlung; characteristic lines from discrete electronic transitions; and Auger processes. However, these three measurements can be conceived in large sample sizes and in situ only for the non-destructive optical techniques or in sample surfaces and ex situ for the destructive plasma source systems. Simple apparatus and low production cost are the main advantages of bremsstrahlung radiation for the in situ on-line measurements, and its wide spectral region. The laws of physics and the experimental techniques for bremsstrahlung radiation are well established, and the radiation spectra have high reproducibility. The method allows the measurement of the EMs with a number of exposed particles around 107 - 109 others (Zimmermann *et al*. 2020) [54] (Frati *et al.*, 2020) [14] (Nawrocki & Sørensen, 2023) [39].

The different patterns, lines, and bands emitted (or transmitted) during the interaction between electromagnetic radiation and atoms have produced a number of different spectroscopies. Based on the nature of the interaction, they can be included in two groups: optical techniques, which use visible or ultraviolet radiation and atomic processes provoked by energetic photons; and X-ray spectroscopies, which use characteristic lines that appear at high-pressure lamps, synchrotron sources, and soft X-rays provoked by the collapse of electronic transitions of inner shells produced by energetic photons. Hence, the former is called optical spectroscopy, and the latter is known as X-ray spectroscopy. These techniques alone provide information about the atomic or molecular points of view, i.e., about the electronic cloud and the internal structure or the so-called wave functions of the element or the molecule (Baiz *et al.* 2020) [6] (Lorenz-Fonfria, 2020) [35] (Gaft *et al.* 2020) [15].

3.1. Atomic Absorption Spectroscopy

The basic principle is to measure the absorption of electromagnetic radiation by the ground-state atoms in the gaseous state. AAS is based on both Beer-Lambert's law and resonance absorption of radiation. The amount of light absorbed by ground-state atoms is measured. Ground-state atoms of many elements absorb UV-visible radiation, which have unique characteristics and different excitation energy typical for each element. The radiation source in the UV region in the atomizer is required for resonance and continues to the other detector. A commercial UV-visible detector can work. The detection limit and analytical linearity are factors depending on the relation between the radiation sources. Despite the method's high detection sensitivity, the fast response has made it one of the most popular techniques for quantifying elements. It is easy to use the monochromator, which has a fixed-diffraction grating, to remove these interference problems. It is also a fast response method and can be performed using common reagents that contain easily produced elements in small concentrations. On the other hand, loss in precision is a negative characteristic of this method (Pareek & Jaswal, 2024) [40] (Bergmann, 2024) [7] (Hung *et al.*, 2021) [21].

Atomic absorption spectroscopy is based on the absorption of light by free atoms only. In other words, the interaction of UV-visible radiation, which is electromagnetic radiation, with atoms can be written as two basic steps: atomization and radiation absorption. Atomic absorption spectroscopy is a simpler approach based on a reduced number of variables, which is useful for more accurate multi-element analysis. It is also a single-element analysis technique used for the determination of individual elements in the same sample. (Merten *et al.* 2023) [38] (Irisov *et al.* 2021) [22] (Machado *et al.* 2020) [37].

3.2. X-Ray Fluorescence Spectroscopy

Established instrumental analytical techniques include the use of X-ray tubes in combination with solid-state or gas proportional detectors. The main instrumental configurations used are the transmission geometry and reflection ones. For trace element analyses, the limitations rely on the sensitivity of the analytical equipment. For the determination of major and trace elements, both theoretical estimations and practical experimental results agree that typical sensitivities of about 3 mg/g for trace elements and 3% for the determination of major ones should be achievable for both solid and powdered samples. The precision is seldom better than twice the sensitivity. The main sources of error in X-ray fluorescence are due to a poorly related and inhomogeneous sample and to interference with both the sample and the X-ray equipment. Critical considerations for the performance of the various samples in general, and the analysis of ground and powdered samples in particular, are presented. (Cinosi *et al.* 2020) [11] (Povarov *et al.* 2021) [43] (Lai *et al.* 2020) [30].

The concept of X-ray fluorescence is based on the ionization of an inner shell of an atom by an interacting high-energy charged particle or photon, thus creating a core vacancy. This vacancy will be filled by an outer electron resulting in either the emission of an X-ray with an energy equal to the difference of the binding energies of the two shell electrons or in an Auger electron emission (Vanhoof *et al.* 2021) [49] (Besley, 2020) [8].

The X-ray fluorescence method of analysis takes advantage of the unique K and L radiation characteristics of different atoms. It is a typical non-destructive analytical technique and, in general, it is simple, fast, and reasonably precise. The analysis is usually limited from magnesium to the heavier elements. The range can be extended to the light elements, but it is difficult and labor-consuming. The main advantages and drawbacks of the method are reviewed (Kobylarz *et al.*, 2023) [28] (Heimler *et al.*, 2023) [19].

4. Chromatographic Techniques

Chromatographic techniques, very important for chemical analysis, have been widely used to measure gamma emitters, neutrons, and radioactive elements. Ion exchange (IEC) and reversed phase chromatography (RPC) are commonly used to determine those of complex matrices. Solid and liquid extraction chromatography of their solvent was designed especially for the separation and preconcentration purposes of selected radiochemical elements at the millesimal levels. Commercially available columns meet the requirements for the isotopic and radiochemical separations of Actinide/Noble metals and the Rare Earth elements, but they are too expensive for repeated use analysis. Therefore, the typical experimental program for radiochemical analysis includes a separation stage in reinforced polypropylene columns before performing instrumental analyses (Salbu & Lind, 2020) [45] (Luo 2023) [36].

For the analysis of natural radioactive and neutron activation elements, high purity chemicals are frequently used as sources, standards, carrier salts, and to desalt and preconcentrate the samples. In this chapter, the chromatographic techniques that are used for this purpose, such as ion exchange, reversed phase, and extraction chromatography columns, are presented. The synthesis and preparation of some special resins are referred to. The chemical analysis of matrices with high salt concentrations (seawater, urine, food, industrial discharges, and so on) are under special consideration. The determination of some elements that can be affected by isotopic dilution during the chemical separations of Actinide/Rare Earth elements is also mentioned (Judge & Azimi, 2020) [27] (Machado *et al.* 2020) [37].

4.1. Gas Chromatography

Here, a specific application of GC is presented: it is the detection of volatile organic compounds known to suit the analysis of tritiated and 14C emitting substances in mixtures. In this application, a solid-phase micro extraction (SPME) coupled with GC with Flame Ionization Detector (FID) (SPME/GC/FID) is introduced. The SPME technique is rapidly gaining acceptance for use in analytical chemistry. By using a SPME device, pre-concentration of analytes can be achieved, providing a sample with greater sensitivity and selectivity. This operation can be repeated providing reuse of the fiber, minimizing the negative environmental impact associated with solid waste generation (Jalili *et al.*, 2020) [24] (Dugheri *et al.* 2022) [12].

Gas chromatography (GC) is a separation technique employed in the analysis of complex mixtures, especially volatile and semi-volatile ones. In principle, a mixture component should not interact with the column surface, and then it is available for separation. GC involves the use of a liquid phase and a column with a solid support. In practice, the liquid phase can be either polysiloxane or a polyethylene glycol bond. The column is normally packed with solvent. The gas sample (commonly called the carrier gas) is the mobile phase that ensures the elution of the mixture components. The column presents itself as an imaginary sieve for molecules traveling under the flow of the carrier gas. Each molecule interacts with the column and, at equilibrium, a net void volume is generated along the column length. This volume is called the retention volume. Characteristic retention times are observed after the elution of each component through the detector, and base peaks are used for the identification of molecules. (Poole, 2021) [42] (Lelevic *et al.* 2020) [33].

4.2. Liquid Chromatography

A separation is generally carried out using a packed column or a flow-through channel of liquid where the mixture is transported by an inert carrier gas. The flow may be forced by different ways such as by an inert piston or vaporization of the carrier phase that bubbles a gas mixture through the column bed. However, nowadays there is a wide variety of commercial LC instrumentations that offer application for almost everything. The development of extremely small packed column random packing allows chromatographic separations on gas-entrained (or gas-fed) and gas extraction modes of liquid chromatography (Perchepied *et al.*, 2022) [41] (Wahab *et al.*, 2021) [51].

Liquid chromatography (LC) is an extremely versatile technique that is used for a variety of purposes, including the detection of trace impurities in bulk gases, general classification and monitoring of the state of catalysts, purity/impurity analysis of electronic devices and silicon wafers, the detection of insecticides, and analysis in general. A large number of methods have been developed using the various different techniques and modes now possible with LC. Workers carrying out characterization studies must be well informed about the various modes and techniques of LC as well as the broad range of types of columns that are now available and which are recommended to achieve the desired separation (Tsizin *et al.*, 2020) [46] (Ali *et al.*, 2024) [2].

5. Radioactive Emission Based Measurements

A systematic classification of the subject matter consisting of nuclear physics and nuclear engineering has offered convenient data accumulation of the accumulation of literature only on the basis of radioactive measure. Since the precise reliability of the radioactive measure has offered certain radiological properties which discriminate them from each other, such categorization will surely help the experts comprehend the propensity information to knowledgeable scholars. The huge responsibility of radon and its decay daughters, the most deleterious source of exposure to radionuclides, has forced the experts to shift their sincere concern towards the detection and analysis of this alpha-emitting alpha radioactive decay daughters. So, the quality with which this radiation is detected should be dignified with its atomic structure and the corresponding decay event. Therefore, the alpha particle detector associated with the unique class of radioactive radiation detection should be carefully identified (Kolos *et al.* 2022) [29] (Gomez-Fernandez *et al.* 2020) [16].

The term radioactive emission indicates that the sample or the medium subjected to the experiment emits a certain amount of radiation, whereas the detection of the measurable quantity can be classified on the basis of those radiations emitted from the sample or medium. On the basis of radiations emitted, the radioactive emission-based measurement of archaeological, geological, as well as biological samples can be characterized depending upon several measurable radiations emitted including X-ray, gamma-ray, optic phonon, alpha particle, beta particle, or beta particle-emitting isotope level, etc. The information encouraged through the radioactive emission-based measurement is worth to the experts in a specific domain. Accordingly, a systematic review describing all those details associated with the outcome of measurements reported in the domain of interest has been illustrated in a clear-cut manner to cater to the need of ample knowledge to the explorers (Warren, 2020) [52] (Precek *et al.* 2022) [44].

6. Alpha Emission Spectroscopy

The AIFIRA facility allowed for measurements of both alpha penetration probabilities, based on the coincidence or anticoincidence measurements through different kinds of detectors, one in anticoincidence mode covering the whole 4π. Alpha particles are detected using highly segmented double SIDAR detectors. These detectors consist of large area silicon detectors similar to the ALADIN-SD detector with about 350 square millimeter total area. Each SIDAR is composed of 6 small double-side detectors $\sigma = 300$ μm thick, typically 17.5 mm × 17.5 mm, connected through their standard NIM electronics. Each detector consists of a central pixel. The detector typically achieves a 17.5 mm φ resolution

at 25 MeV alpha emission or less in a MITIs configuration. The detector draws a current of 3μA at 150 V bias voltage (Atukpor, 2023) [4] (Atanasov *et al.* 2023) [3].

Alpha emissions from a thin source or a thick uranium sphere can be detected and measured using high-resolution electronics with a good energy resolution silicon solid-state detector. An excellent intrinsic energy resolution can be achieved using this alpha-particle sensitive charge-collecting high-resistivity detector, demonstrating the copper contamination in the deeply buried NTD Ge detector. Alpha spectrometry measurements were made and very clearly any alpha emissions hidden under a large number of internal conversion and Auger electrons could be separated and identified by their intrinsic energy spectrum. Since 1995, at AIFIRA at TARA facility, at VINCY and ATOMKI typically covering spectral regions not accessible with the use of accelerator facilities, typical alpha decay behavior of nuclei, transitions through the 0+, in many experiment spectrometric investigation of other decay processes can be studied (Vajda *et al.,* 2020) [47] (Jenkins, 2020) [25].

7. Beta Emission Spectroscopy

The energies of several pure beta emitters are suitable to drive low-pressure electrolyzers that produce hydrogen on the Moon, due to the tight mechanical and mass budget constraints, and 10Be becomes a top light option producing a more pure hydrogen product. No significant low-hanging fruits can be identified in terms of beta emitters from the perspective of betabatteries driving high-pressure electrolyzer due to the large number of process control and support equipment required, as well as their tight energy and mass budget. The rate of change in the mass, volume, and acceleration of the field of betavoltaics in other than power density term has to be kept in a maximally possible margin to pass a meaningful betavoltaic implementation gate review supposed to be held in the near future (Lara *et al.*, 2021) [31] (Lara *et al.*, 2020) [32].

Among known beta-decay nuclides, a few have long half-lives enough to be used for practical purposes. 14C, bearing a moderate amount of energy, fulfills the needs of practical betavoltaic cells where 14C is a part of the cosmic ray energy density measuring instrument, and is also free of the need for thin energy converter layers due to its suitable energy. Different designs for betavoltaic batteries with a variety of beta emitters are studied. Both power capacity and lifespan are quantified. Beta attenuation length inside a semiconductor material gives an important hint to decide the power-producing volume, as betavoltaic cells have no ionization capacity due to the use of thin epitaxially grown semiconductor layers. Their long half-lives make 63Ni and 90Sr our top choices for long-lasting betavoltaic batteries (Liu *et al.* 2024) [34] (Chaudhuri *et al.* 2023) [9].

8. Applications in Environmental Monitoring

Abrupt radiation releases and major accidental emissions are very well-known, especially in accidents that involved the nuclear fuel cycle, such as those that happened in Chernobyl, and three of the most publicly known accidental releases associated with the weapons industry: these are the accidents that occurred in the plants located in Mayak, Kazakhstan, and Savannah River. The radiological consequences associated with these accidents are very well documented and should never be minimized. However, these consequences are characterized by only a few radionuclides and sometimes complex mixtures that involve secular equilibrium, as in the case of effluents from nuclear power plants. In fact, usually the photopeak of the signal is totally overlapped in energy spectra, and sometimes a very precise and accurate choice has to be made for the alteration of the energy window in order to determine the counts that correspond only to the isotope under consideration (Yim 2020) [53] (Von and Kurokawa 2020) [50] (Van Eerten, 2024) [48].

Radiation traditionally has been used as a universal tool for environmental monitoring and characterization. This is mainly due to the fact that radiation does not need to be in contact with the media to be analyzed in order to perform the monitoring. There is no significant lag time between sampling and results, and radiation is able to provide overall, real-time, and on-line processes control. Traditional nuclear techniques have been used for a long time to measure several different variables that are of interest in environmental monitoring. Major and trace elements, gases like CO, and H2 in the soil, water-borne isotopes (like tritium and uranium isotopes in water bodies), fission isotopes (like 137Cs in air), soil erosion, and deforestation are but a few examples of the list of variables that can be monitored by means of nuclear techniques (Howell *et al.*, 2020) [20] (Guo *et al.* 2020) [17] (Heck *et al.* 2020) [18].

9. Conclusion and Future Directions

It is well known from the first investigations on the degraded spent nuclear fuel that precise and traceable data quality could be reached using radiological measurements. The capability of these measurements to univocally identify the elements or isotopes in an environmental sample, deconvolute decomposed epidotic mixtures, and evaluate reliable isotopic ratios between long-lived and short-lived nuclides is well established. Nowadays, the intrinsic capabilities of such measurements could be used profitably to address very important issues. For example, in conditions of high radiation fields, it is challenging to measure characteristic decommissioning activities. These measurements can effectively certify the procedures limiting the biological impact of waste disposal in proposed final repositories. Additionally, they can evaluate the safety of the final repository when and whether spent nuclear fuel reprocessing has to be considered.

In this paper, the advances in the field of nuclear and radio-analytical aspects of waste characterization have been reviewed in connection with uranium and trans-uranium elements. Some key nuclear methods have been considered, referring to radioactive decay schemes, limits of detection, and some examples of applications. On the other side, radio-analytical techniques do not necessarily make use of radioactivity, although they have to be cross-referred to the nuclear data resulting from radioactivity measurements. The potential benefits obtainable from coupling primary activities measurements with other analysis techniques have been discussed as well.

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