

## REVIEW

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**Determination of elements by nuclear analytical methods**

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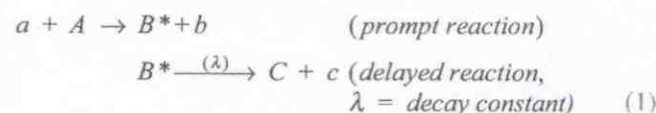
**Abstract** The working principle of nuclear analytical methods (NAMs) is not influenced by the chemical bond. Consequently, they are independent counterparts to the well-known chemical procedures. NAMs obey fundamental laws or can be described and understood thoroughly. This qualifies them as candidates for reference methods. Although following similar nuclear reaction schemes, they comprise bulk analyzing capability (neutron and photon activation analysis) as well as detection power in surface near regions of solids (ion beam techniques). Prominent features of NAMs are sensitivity, selectivity, multielement determination and linearity of the calibration function covering a concentration range of several orders of magnitude. Moreover, ion beam techniques allow depth profiling with nm-resolution in several cases while the ion microprobe additionally offers a lateral resolution in the  $\mu\text{m}$ -scale. As NAMs require expensive apparatus (nuclear reactor, accelerator in radioactive control areas) their availability is restricted to a small number of suitably equipped institutes. However, they are able to solve complex analytical tasks, take part in key comparisons and play an essential role in the certification of reference materials.

**Introduction**

Nuclear analytical methods (NAMs) aiming at the determination of elements are based on interaction of nuclear particles with atomic nuclei. They are nuclide specific in most cases. As the electronic shell of the atom does not participate in the principal physical process, the chemical bonding status of the element is of no relevance. On the one hand, speciation analysis cannot be performed directly by NAMs in general, on the other hand, the analytical result is not influenced by the chemical form of the element to be

quantified. In many cases NAMs can be performed without digestion or dissolution of the sample, analyzing solid materials instrumentally and leaving them more or less unchanged. Because of these two basic features NAMs are valuable complements and/or alternatives to chemical analytical procedures. Enhanced selectivity and lower determination limits can be achieved by utilizing the time delay between production (activation in a reactor or by an accelerator) and radiation detection. Also radioanalytical separation techniques can be applied after irradiation. In addition, NAMs may be described and defined precisely which qualifies them as candidates for reference methods.

The general scheme of a nuclear interaction is



where  $A$ ,  $B$ ,  $C$  denote atomic nuclei and  $a$ ,  $b$ ,  $c$  particles or  $\gamma$ -quanta.

One may distinguish the following cases:

**Activation**

The nuclear particle or photon  $a$  interacts with the nucleus  $A$  producing an excited nucleus  $B$  and a promptly emitted particle or  $\gamma$  ( $b$ ). After a delay time which may vary from milliseconds to hundreds of years, the nucleus will disintegrate while one or more particles and photons will be emitted. Prompt as well as delayed photons may be detected and used for analytical purposes.

The decay of a large number  $N^*$  of identical nuclei is described by the fundamental law of radioactive decay

$$\frac{dN^*}{dt} = -\lambda N^*.$$

$$\lambda = \ln 2 / T_{1/2} \quad (T_{1/2} = \text{half-life of the nuclide}). \quad (2)$$

This first order reaction proceeds according to the exponential function

$$N^*(t) = N^*(0) \exp(-\lambda t). \quad (2a)$$

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### Transmutation

If  $\lambda \rightarrow 0$  ( $T_{1/2} \rightarrow \infty$ ), the reaction product  $B^*$  is stable and Eq. (1) may be written



This means that only prompt detection is available.

### Collision

In the case of elastic collisions Eq. (1) reduces to



The entities  $a$  and  $A$  remain unchanged, but lose or win kinetic energy. The particles  $A'$  and/or  $a'$  may be detected and used for analytical purposes.

Consequently, NAMs can be distinguished in 'prompt' and 'delayed' methods.

Ion based NAMs commonly addressed as ion beam analysis (IBA) exploit the prompt mechanism with only very few exceptions. Due to the short range of ions in solid matter only surface near regions can be analyzed by IBA filling the gap between typical surface analysis methods like SIMS or electron microscopy and bulk analysis.

Prominent methods are nuclear reaction analysis (NRA), Rutherford backscattering (RBS), channeling (CHAN) and particle induced X-ray emission (PIXE).

Prompt gamma neutron activation analysis (PGNAA) and charged particle activation analysis (CPAA) efficiently determine traces of several light elements. But globally speaking, they are rarely performed. Therefore, PGNAA and CPAA will not be a subject of this contribution.

## Activation analysis

Neutron activation analysis (NAA) is the most powerful NAM especially for elements with medium to high atomic numbers. The sensitivity, however, varies considerably from element to element [1, 2].

Photon activation analysis (PAA) is well suited for the determination of C, N, O and the halogens and has a good

detection capability for most elements beyond Na. The ( $\gamma, n$ ) reaction rate is approximately proportional to the square of the atomic number for heavy elements.

Due to the high penetrability of thermal neutrons and high energy photons through most materials NAA and PAA are typical bulk analyzing methods. In many cases instrumental multi-element determinations can be performed.

## Neutron activation analysis (NAA)

The principal reaction is

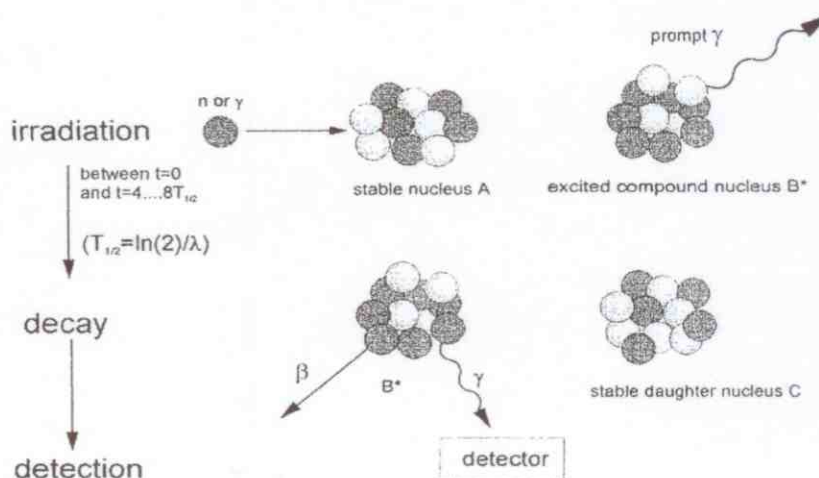


also written  $A(n, \gamma)B^*$ . The symbol  $n$  is attributed to 'neutron'.

The reaction yield is dependent upon the energy of the neutron and is especially high for thermal or epithermal neutrons with energies  $E_n$  between 0.005 eV and 0.55 eV or  $E_n$  between 0.55 eV and 100 keV, respectively. As the nuclear reactor is the principal neutron source, neutron energies  $E_n > 100$  keV are attributed to fission neutrons. These are able to initiate ( $n, 2n$ )-, ( $n, p$ )- and ( $n, \alpha$ )-reactions. They are of minor importance regarding the detection and determination of elements. Yet they may interfere with the determination of a neighboring element, if they produce one of its radioactive isotopes which also may be generated by the ( $n, \gamma$ )-reaction, e.g.  $^{59}\text{Co}(n, \gamma)^{60}\text{Co}$  versus  $^{63}\text{Cu}(n, \alpha)^{60}\text{Co}$ . After both reactions have occurred, it cannot be distinguished by any means, which one of the sources the product  $^{60}\text{Co}$  originated from.

In terms of atomic spectrometry NAA is a method combining excitation by nuclear reaction with delayed de-excitation of the radioactive atoms produced by emission of ionizing radiation ( $\beta$ ,  $\gamma$ , X-ray). Radioactivity serves as an indicator of the element to be determined, and the law of radioactive decay represents a strong link between the SI-units mass [g] and radioactivity [Bq]. As seen in Fig. 1, the sample is exposed to the neutron field of a nuclear reactor. The irradiation interval should be in the order of the

Fig. 1 Principle of activation analysis



half-life  $T_{1/2}$  of the desired radionuclide or as long as practically possible in the case of very long-lived isotopes. After the end of irradiation the activated components decay exponentially. After a suitable time interval the sample is placed in front of a radiation detector. Nowadays in most cases a high-resolution semiconductor detector or a high-efficiency scintillation detector is used for measuring the activity and the energy of the  $\gamma$ -quanta. Because this energy is characteristic for the radioactive species, it serves as a selection criterion in multielement analysis. Unfortunately, the energy transfer from the quantum to the detector material is incomplete resulting in contributions to the measured spectrum below the full energy peak which may mask full energy peaks of other species to be determined. This interference is of physical nature and may be removed by radiochemical separation.

The most frequently applied separation techniques are ion exchange, chromatography, liquid extraction, precipitation and, for some volatile compounds (elements), distillation. Applications in biology, environmental research and geology have been predominant in recent years.

In studying the concentration of trace elements in technologically important materials, the removal of matrix-induced radioactivities is the most urgent need. This may be complemented by a group separation scheme based on the chemical behavior of the elements taking into account the mutual interferences of their related radionuclides in the measured spectra [4].

NAA (and other activation techniques) offer some unique advantages exploiting the time interval between activation and decay. After the end of irradiation inactive contaminants will not affect the analytical result anymore. Consequently, before dissolving the activated sample a known amount of the (inactive) element to be determined can be added. In this way macro-chemistry is performed even while studying ultra-traces. Moreover, the determination of the chemical yield becomes easily feasible. An alternative is the substoichiometric separation [5, 6].

Fortunately, in some cases the matrix allows the sensitive determination of traces without chemical separation. A prominent example is semiconductor silicon. Irradiation with a neutron fluence (neutron flux density times irradiation interval) of  $5 \times 10^{19} \text{ cm}^{-2}$  and counting times be-

tween 2 hours and 4 days yields detection limits  $L_D$  well below 1 ng/g for a series of elements (Fig. 2). Of course,  $L_D$  may vary considerably depending on the trace element composition of the sample [7], [Alber, Berger, Görner, Köhler, Niese, unpublished results].

#### Characteristics of NAA

- instrumental (quasi non-destructive) multielement analysis possible
- information about bulk content (even of large samples)
- not suitable for light elements
- Na and most elements with  $Z \geq 17$  determinable
- linear measuring range from sub-pg/g up to %-level
- usually small influence of the matrix
- low blank values
- interferences may be overcome by optimized irradiation and measurement strategies
- chemical separation including carrier techniques

#### Photon activation analysis (PAA)

Linear accelerators or microtrons converting the electron energy into bremsstrahlung serve as sources of high energy photons. These are able to expel particles from the nucleus (nuclear photo effect), thus producing radioactive species used for PAA. Beam currents of about 20 to 200  $\mu\text{A}$  and electron energies between 7 and 35 MeV have proven to be practical.

The most probable reaction occurring in PAA is



The photons emitted in the delayed decay are detected and used as analytical signal.

The main features discussed above for NAA concerning irradiation interval, radioactive decay, interferences, radiochemical separation and detection methods hold true for PAA in an analogous way [8].

PAA is a valuable tool to perform instrumental multielement analysis in various matrices including environmental, biological and industrial applications. Its physical principle, the possibility to omit a digestion step and the possibility to clean the sample from surface contaminations by etching after irradiation are considerable advantages. This is important if solid samples are difficult to dissolve. In the certification of reference materials instrumental PAA is a valuable complement to any sample dissolving method.

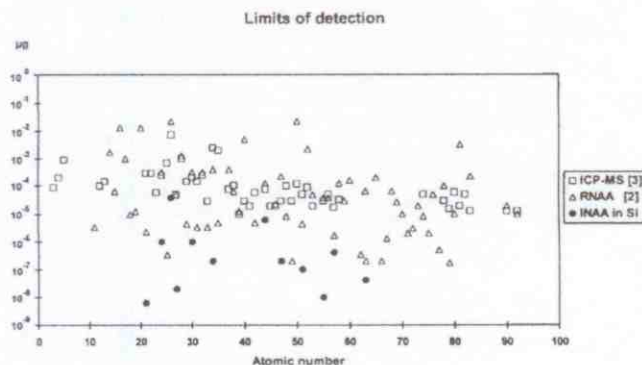


Fig. 2 Limits of detection: ICP-MS versus NAA

Table 1 PAA reaction characteristics of light elements

Reaction	Threshold/MeV	$T_{1/2}/\text{min}$	$L_D/\mu\text{g} \cdot \text{g}^{-1}$ [8]
$^{12}\text{C}(\gamma, n)^{11}\text{C}$	18.72	20.3	0.01–5
$^{14}\text{N}(\gamma, n)^{13}\text{N}$	10.55	9.96	0.001–1
$^{16}\text{O}(\gamma, n)^{15}\text{O}$	15.67	2.03	0.02–1
$^{19}\text{F}(\gamma, n)^{18}\text{F}$	10.44	109.7	0.005–0.05

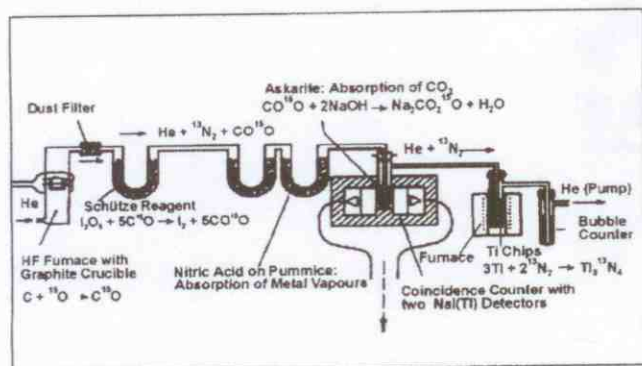


Fig. 3 PAA: simultaneous determination of O and N

Determination of light elements is a special feature of PAA. Photon irradiation of C, N, O and F leads to pure positron ( $\beta^+$ ) emitters (neutron deficient nuclei). This means that all their signals sum up at the  $\beta^+$ -annihilation energy (511 keV). Such physical interference which is typical to PAA can be prevented in special cases, if the interfering reaction has a higher energy threshold than the determination reaction (Table 1). Then, irradiation energies below the interference threshold favorably select the desired nuclide. Sometimes complex decay curves may be unfolded, if the half-life of the interfering nuclide is not too close and the activities are in the same order of magnitude. Finally, rapid separation schemes can discriminate elements of the mentioned group. As shown in Fig. 3, O and N can be separated from the matrix simultaneously by hot extraction. For C combustion is commonly applied and distillation is widely used in the case of F.

Unlike F, the heavier halogens form photon activation products emitting characteristic  $\gamma$ -energies in addition to  $\beta^+$ -annihilation radiation. The characteristic  $\gamma$ -peaks in the spectra can be used favorably for their quantitative determination.

Characteristics of PAA (matching those of NAA with slight modifications)

- wide variety of elements determinable ( $Z \geq 6$  including Ti, Ni, Nb, Tl, Pb, Bi which are not accessible to NAA)
- linear measuring characteristic from  $\mu\text{g/g}$  to %-level

### **Ion beam analysis (IBA)**

The different physical interaction processes between MeV-Ions and target atoms can also be exploited for the elemental analysis of material. Due to the strong energy loss of the charged particles only thin layers in the range between a few nm and about 10  $\mu\text{m}$  can be analyzed. In contrast to activation by neutrons and photons bulk samples cannot be analyzed with ion beams, but on the other hand the energy loss of the impinging ions and of the charged reaction products yields information about the distance between the target atom and the sample surface.

The possibility of depth profiling is a characteristic advantage of several IBA methods which is especially useful in thin layer technologies such as semiconductors, super conducting layers, thin optical films etc. Several reviews have been published covering the IBA methods using high energy (MeV-range) charged particles [9–15]. The most important interactions between ions and atoms can be summarized as follows:

- Rutherford scattering in the Coulomb-field of the target nucleus
- elastic scattering caused by the nuclear forces between the two interacting nuclei (in addition to Coulomb interaction)
- nuclear reactions with emission of  $\gamma$ -radiation and (or) charged particles
- inner shell ionization of the atoms with subsequent emission of characteristic X-rays

Each of these processes constitutes the physical basis of at least one IBA method which in most cases can be performed non-destructively.

### **Backscattering spectrometry**

At sufficiently low energies (a few MeV for He-Ions) the nuclei of the impinging ions and the target atoms interact only via Coulomb-forces. In this simple case the differential scattering cross section is described by the well known Rutherford formula which can be derived from classical mechanics taking into account energy and momentum conservation. The Rutherford cross section is a smooth function of energy and scattering angle for a given ion-target combination. With increasing ion energy the interaction via nuclear forces becomes important resulting in deviations from the simple Rutherford law. In many cases the elastic backscattering (EBS) significantly exceeds pure Coulomb scattering and often strong resonances appear in the function of the cross section which are caused by the individual nuclear structure of the collision partners. Under these conditions the cross section can no longer be calculated from first principles, but measured data have to be used for quantitative analysis.

### **Rutherford backscattering spectrometry (RBS)**

The experimental setup for RBS analysis is quite straightforward (Fig. 4). The energy spectrum of the ions ( $^4\text{He}$ -beams are most commonly used) scattered by the sample atoms into a fixed solid angle in the backward hemisphere is measured by means of a semiconductor detector or – for achieving higher depth resolution – by an electrostatic or magnetic spectrometer. Accounting properly for the energy loss of the ions along their path inside the sample and of the scattering kinematics the mass and the depth of the target atoms and their local concentration can be derived from the measured spectra. The evaluation is done by fitting calculated backscattering spectra to the measure-

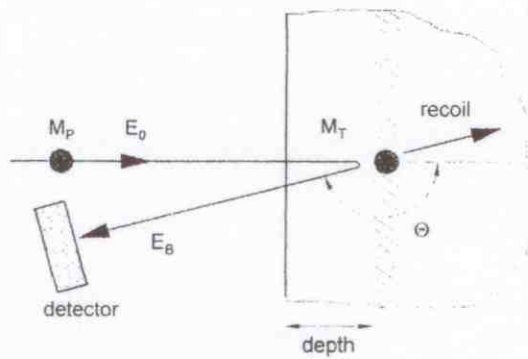


Fig. 4 RBS/EBS scheme

ments using well established software packages. The model depth profile of the atoms is varied until minimum deviation between calculated and measured spectra is achieved.

#### Characteristics of RBS

- non-destructive determination of the element concentration as a function of depth (depth profiling) with nm-resolution
- concentration range down to atom-ppm level for the determination of heavy elements in light matrices
- reference method which can be traced back to first principles
- broad spectrum of applications especially in semiconductor industry, thin layer technology, optical coatings etc.

#### (Non-Rutherford) Elastic backscattering spectrometry (EBS)

For EBS most characteristics of RBS also hold (see above). The main differences from RBS are

- no simple traceability to elementary physics, therefore EBS cannot be regarded as an analytical reference method
- higher sensitivities for several light elements

Detailed information on backscattering spectrometry may be found in references [9–12, 16].

#### Elastic recoil detection analysis (ERDA)

The underlying physical principle of ERDA is identical to that of backscattering spectrometry with the important difference that the analyte atoms ejected from the sample surface are measured (Fig. 5). These recoil atoms emitted into a fixed solid angle in the forward scattering hemisphere yield information about the depth profile of the analyte concentration. Several experimental techniques for detection, identification and energy measurement of the recoil atoms are commonly in use:

- semiconductor detectors
- time of flight (TOF) spectrometers

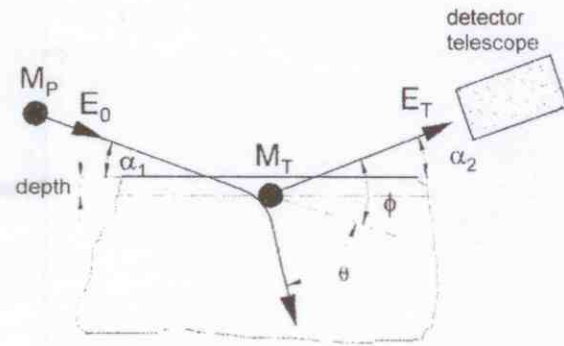


Fig. 5 ERDA scheme

- sophisticated ionization detectors for particle identification
- magnetic spectrometers

Since ERDA is suitable for the analysis of light elements in heavy matrices this method is complementary to RBS. The analytical sensitivity can be improved by combining ERDA with RBS. This is achieved if the backscattered (primary) ions are detected in coincidence with the recoil atoms emitted in the forward hemisphere.

#### Characteristics of ERDA

- non-destructive depth profiling of light elements in heavy matrices
- analysis of hydrogen and deuterium with detection limits of about 10 atom-ppm
- range of depth profiling: several  $\mu\text{m}$
- reference method traceable to first principles
- analytical applications similar to RBS

For detailed information see reference [9].

#### Nuclear reaction analysis (NRA)

If the energy of the primary ions becomes sufficiently high the Coulomb-barrier of the collision partners is surpassed and the probability of nuclear reactions strongly increases. The most common reaction types induced by light ions with energies in the MeV-region interacting with light and medium-Z nuclei are

- $(p,\gamma)$ ;  $(p,p'\gamma)$ ;  $(p,n)$ ;  $(p,\alpha)$ ;  $(p,\alpha\gamma)$
- $(d,p)$ ;  $(d,p\gamma)$ ;  $(d,n)$ ;  $(d,\alpha)$
- $({}^3\text{He},p)$ ;  $({}^3\text{He},p\gamma)$ ;  $({}^3\text{He},d)$ ;  $({}^3\text{He},\alpha)$
- $(\alpha,p)$ ;  $(\alpha,n)$ ;  $(\alpha,d)$
- $({}^{15}\text{N},\alpha\gamma)$ ;  $({}^{19}\text{F},\alpha\gamma)$

In contrast to activation analysis, the *prompt* emission of charged reaction products or  $\gamma$ -radiation is used for the identification of nuclides. For the spectrometry of charged products all detector systems mentioned above have been applied.  $\gamma$ -ray spectra are measured by means of high resolution Ge-detectors or scintillation detectors (NaI or bismuth germanate). As already discussed for RBS the energy loss of charged reaction products in the sample ma-

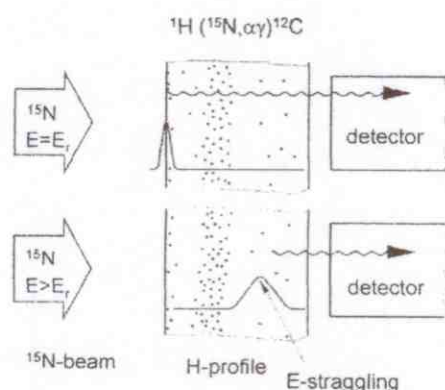


Fig. 6 Depth profiling analysis using the reaction  $^1\text{H}(^{15}\text{N}, \alpha)^{12}\text{C}$

terial yields depth information for the nuclei (depth profiling). High resolution depth profiling by measuring prompt  $\gamma$ -radiation can be performed if there are narrow resonances in the reaction cross section. If the ion energy does not coincide with the resonance energy the reaction probability is low (ideally equal to zero) whereas in the resonance region the cross section increases by several orders of magnitude. If the energy of the impinging ion beam exceeds the resonance energy there will be a certain depth in the sample where the ions have reached the resonance energy due to deceleration. Therefore, the depth where the concentration is to be measured can be chosen by varying the ion beam energy. The principle of resonance depth profiling is shown in Fig. 6. For example, the resonance reaction  $^1\text{H}(^{15}\text{N}, \alpha)^{12}\text{C}$  has been used for measuring the spatial distribution of hydrogen in a large variety of materials [9]. The reverse reaction allows nitrogen depth profiling.

#### Characteristics of NRA

- non-destructive determination of light elements especially hydrogen
- depth profiling using resonance reactions  
depth resolution: 5 ... 100 nm depending on the nuclear reaction  
depth range: up to several  $\mu\text{m}$
- detection limits: < 1 atom-ppm depending on the element

### Particle induced X-ray emission (PIXE)

In contrast to NRA, PIXE is not a true nuclear analytical method because it is based on the interaction of fast ions with the electron clouds of the atoms. The physical process involves inner shell ionization and subsequent emission of X-rays which are characteristic of the structure of the electron cloud. Therefore, the atomic numbers of the elements involved are determined by X-ray spectrometry and no information about the isotopic composition is obtained. Energy dispersive X-ray measurements, e.g. with Si(Li)-detectors, enable multielement analysis

for all elements with atomic number  $Z \geq 6$ . Compared to electron beam induced X-ray emission (electron microprobe) the detection limits for PIXE are lower by 2 to 3 orders of magnitude. The reason is the lower continuous bremsstrahlung background induced by ions in the sample material. Since X-rays do not lose energy along their short path inside of the sample no direct information on depth distribution is obtained from the X-ray spectrum. Nevertheless, from measurements at various angles of incidence, respectively bombardment energies approximate estimations of the depth distribution of the X-ray emitting atoms can be derived. In most applications PIXE analysis is performed with proton beams up to 3 MeV. However, in some cases X-ray excitation with heavy ions can be advantageous because the continuous background is reduced and higher cross sections can be exploited for selective enhancement of detection sensitivity for certain elements (level matching effect).

A collimated beam of MeV protons can be transported from the evacuated beam line into ambient air by passing through a thin window, thus enabling PIXE analysis of objects under ambient pressure. External beam PIXE has been widely used for analyzing art objects such as paintings, books, ceramics, statues etc. or archaeological objects which cannot be transferred into a vacuum chamber.

A special advantage of analytical methods using charged particle beams is the possibility of focussing with electrostatic or magnetic lenses, thus enabling spatial resolution. Worldwide there are many IBA-installations with a proton microprobe facility. The spatial resolution is about  $1 \mu\text{m}$  (standard) down to  $100 \text{ nm}$  (state of the art). The detection limit is much lower than for the electron beam methods (electron microprobe, scanning electron microscope with EDX). All aspects of PIXE have been extensively reviewed in references [17, 18] and the special features of ion micro beams are described in reference [19].

#### Characteristics of PIXE

- non-destructive multielement determination of all elements with  $Z \geq 6$
- detection limit: 1 atom-ppm
- minimum sample mass:  $10^{-11} \text{ g}$
- analysis depth in solid samples  $\approx 10 \mu\text{m}$  (depending on particle energy and on sample composition)
- spatial resolution of ion microprobes:  $1 \mu\text{m}$  down to  $0.1 \mu\text{m}$
- broad application in materials science, biology, medicine, environmental protection, arts, archaeology.

### Ion channeling (CHAN)

If a surface layer is monocrystalline not only its elemental composition but also its microscopic structure can be analyzed by IBA methods. If the angle of incidence of the ion beam approaches a main direction or plane of the crystal lattice the ions are guided into the material along the corresponding „open channels“ between the atom rows by a sequence of small angle scattering events. Under this con-

dition the backscattering intensity exhibits a pronounced dip. If the angle of incidence of the ion beam deviates by more than a few tenths of a degree from the channel direction the guiding effect disappears and the „random“ backscattering intensity is observed. If dopant atoms partly obstruct the channels they can be detected by analyzing the channeling dip. Besides RBS also PIXE and NRA can be used for channeling experiments. From the measured yields conclusions about the crystalline structure and quality of the layer can be drawn.

#### Characteristics of CHAN

- evaluation of the crystallinity of thin layers
- detection of lattice defects, e.g. as a consequence of ion implantation
- determination of the lattice sites of dopant atoms
- quality control of epitaxial layer growth
- investigation of interfaces between metallic and semiconductor layers

All aspects of channeling are reviewed in reference [20].

### Conclusions

The essential contribution of NAMs to analytical chemistry is that their working principles are independent of the chemical bond. In addition, they allow or even demand instrumental procedures without dissolution of the sample. Consequently, the determination of the chemical yield is by-passed in such cases. As neutrons and photons are highly penetrating, NAA and PAA are bulk analyzing methods. On the contrary, IBA favorably studies layers down to the depth of some micrometers, thus filling the gap between typical surface analyzing methods such as electron microscopy or SIMS and bulk analysis. Due to the strong deceleration of charged particles in solids, depth profiling with nm-resolution is offered by some IBA methods. Ion imaging in the  $\mu\text{m}$ -scale is performed by ion optics based micro-probes.

The physical fundamentals of NAMs are well understood and described in terms of registration probability (efficiency) and uncertainty, which is a prerequisite of any reference method.

NAA, PAA and NRA are isotope specific, which can be a source of error or an analytical tool, likewise. A suitable combination of NAMs allow all elements of the periodic table to be studied. PAA, NRA and ERDA are especially suitable for the determination of light elements. NAA, RBS and PIXE detect medium and heavy elements very well. Interference of neighboring elements has to be taken into account. As the bremsstrahlung background generated by ions is lower than that caused by electrons, PIXE is much more sensitive than the electron microprobe technique.

The range of application of NAMs covers the detection and quantification at sub-ng, sub-ng/g and atom-ppm lev-

els, the determination of the stoichiometry in thin layers as well as precision determination of macro-contents.

If a chemical separation using the multi-carrier technique is performed after activation (NAA, PAA) macro-chemistry, even in the case of ultra-trace analysis becomes possible permitting to determine chemical yields. Bound to expensive equipment and specialized personnel NAMs can solve complicated analytical tasks and are alternatives to chemical analytical methods. They ought to be included into complex investigations, key comparisons and reference material certification schemes.

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