

## 50 CURIE AM-BE NEUTRON SOURCE IN DETERMINING IMPURITIES IN VARIOUS MATERIALS

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The neutrons from a 50 Curies Am-Be neutron source after being thermalized have been used to study the impurities in various materials by measuring the gamma rays emitted from the activated samples. To get good resolution two HPGe detectors, one of them suitable for low energy gamma rays as well as X-rays and the other suitable for measuring the gamma-ray energies up to 10 MeV have been used. The resolution of the detectors were measured and proved to be better than 1.8 keV for  $^{60}\text{Co}$  gamma rays. During the measurements the detectors were placed in thick lead chambers. In these chambers the background was reduced dramatically. To make the whole system safe and also for saving time in activation analysis a fully computerized control rabbit device has been coupled to the system. Our main purpose is to set up a portable, cheap and reliable system for activation analysis for research institutions that are not able to have reactors due to various reasons. Although our tests and analysis is still in progress we think that the system is very promising. In this paper we will discuss about the details and the future prospects.

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**Topic 3**

**ACTIVATION ANALYSIS AND  
OTHER RADIOANALYTICAL METHODS**

**Poster presentations**

## **$^{226}\text{Ra}$ DETERMINATION IN PHOSPHOGYPSUM BY ALPHA-PARTICLE SPECTROMETRY**

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Phosphogypsum is the main by-product of producing phosphoric acid by reacting phosphate rock with sulphuric acid. In the South-West of Spain and near to the town of Huelva there is a big industrial complex with some factories devoted to the production of phosphoric acid, which produces  $3 \cdot 10^6$  metric tons of phosphogypsum per year. Around 80% of this amount is stored in a nearby salt-marsh area, in big piles named "gyp-stacks".

These wastes contain high concentrations of  $^{226}\text{Ra}$  (about 50 times higher than in typical soils). Due to the radiotoxicity of this radionuclide, its decay to  $^{222}\text{Rn}$  which can emanate in high amounts from the piles, and the significant contribution of its daughters to the gamma exposure rate, the study of  $^{226}\text{Ra}$ -behaviour in the storage cycle and phosphogypsum piles is needed.

Therefore, field and laboratory experiments that require the possibility of  $^{226}\text{Ra}$  measurements with high accuracy and sensitivity have been performed by using alpha-particle spectrometry.

In this work the explanation and validation of the radiochemical method we have developed for the  $^{226}\text{Ra}$  measurement in environmental samples by alpha-particle spectrometry together with its application in the field and laboratory experiments above mentioned are presented and discussed.

## ELEMENTAL ANALYSIS OF SELECTED SOIL AND FERTILIZER SAMPLES FROM EASTERN SAUDI ARABIA

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One of the best nondestructive bulk analysis methods is Neutron Activation Analysis (NAA). The wide use of this technique in various areas in the last 30 years proves that the technique is very well suited for elemental analysis [1]. Due to the importance of soils and fertilizers in agriculture, studies of element concentrations in the soil and fertilizer samples are important for a better understanding of the effect of each element on the end products [2-3].

Local soil and fertilizer samples were studied for elemental analysis using slow neutron activation analysis. A slow neutron flux of about  $10^6$  n/cm<sup>2</sup>-sec was available which was obtained by slowing down 2.5 MeV neutrons from the D(d,n)<sup>3</sup>He reaction at the KFUPM 350 keV Ion Accelerator. The induced  $\gamma$ -ray activities were measured by a HP-GMX detector and a PC based data acquisition and analysis system [4-5]. Calibration curves were established for the elements present in the samples by activating certified standards of similar matrix from NIST, USA and IAEA, Austria. From the calibration curves, the absolute concentrations of the elements K, Mn, V, Al, Na, Cl, Ba, Sr and Mg were determined. The concentrations of these elements in the soil and fertilizer samples varied from ppm to wt.%.

In this paper, the study of selected local seven soil and six fertilizer samples from Eastern Saudi Arabia using the NAA technique will be described and the results of the absolute concentrations of the elements K, Mn, V, Al, Na, Cl, Ba, Sr and Mg in soil samples and K, Mn, V, Al, Na and Cl in fertilizers will be presented. The results will be discussed in terms of mineralogy as well as comparison among the samples.

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## NEUTRON INDUCED GAMMA-RAY SPECTROSCOPIC AND X-RAY FLUORESCENCE INVESTIGATIONS OF BRITHOLITE VEINS OF BAŞÖREN VILLAGE (TURKEY)

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High resolution  $\gamma$ -ray spectroscopic investigations of neutron induced mineral samples obtained from the deposits of the Başören area which is located about 670 km Southeast of Ankara (Turkey), within the Northeast margin of the Eastern Tauride belt, are reported. The geological investigation indicated that the mineral sample is mainly Britholite,  $\{(REE,Ca,Na)_3 \cdot [(Si,P)O_4]_3 \cdot (OH,F)\}$ . The  $\gamma$ -ray spectrum of the natural sample shows all the elements of the Th decay series and a few elements of the U decay series. The sample irradiated by 14.6 MeV neutrons shows some rare earth elements and Sc, Cr, Mn, Kr, Sr, Y, Ba and Zr peaks in addition to those of U and Th decay series. The natural activities of the mineral sample for  $\alpha$  and  $\beta$  radiations are found to be  $A_\alpha = 1.72$  MBq/kg and  $A_\beta = 0.40$  MBq/kg, respectively, which are consistent with  $\gamma$ -ray spectroscopic results of the Th decay series. The quantitative XRF analysis indicates that the sample contains Si, Ti, Al, Fe, Mn, Mg, Ca, Na, K, P, La, Ce, Nd, Y, Th, Sm, Gd, Eu in different amounts.

## RADIOISOTOPE X-RAY FLUORESCENCE AND NEUTRON ACTIVATION ANALYSES OF THE TRACE ELEMENT CONCENTRATIONS OF THE RAINBOW TROUT

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Every living organism from the simplest one to the more complex contains small amounts of metals. The fundamental importance of metal ions in biological processes has become clearer in the last decade. The effects of heavy metal pollution of the environment and the living organisms have started exhaustive studies on the medical aspects of the toxic elements. On the other hand it is known that all marine and aquatic organisms can accumulate trace and toxic elements to different degrees depending on their physiological mechanisms and the environmental pollution. The trace element concentration of the fish is very important since it is directly related to the human health. The subject of this paper was the determination of the trace element concentration in agricultural rainbow trout samples farmed in Sapanca, Aquaculture Faculty of Aquatic Products Faculty, The University of Istanbul, in order to investigate the effects of environmental pollution to the ecosystem.

The muscles and livers of the ten rainbow trouts (*Oncorhynchus mykiss*; N, 1752) obtained from Sapanca, Aquaculture Faculty of Aquatic Products Faculty, The University of Istanbul (Turkey), have been analysed quantitatively for some minor elements using radioisotope energy dispersive X-ray fluorescence (EDXRF) and neutron activation analysis methods. It is found that samples contain Ca, Na, K, Sc, Cs, Cr, Mn, Fe, Co, Cu, Zn, Se, Rb, Sr, Ba, Br, La and Ce in different amounts. Comparison of the results with those of reference fish samples indicated that the agricultural rainbow trout samples have lower amounts of the elements of toxicological concern.

## **GAMMA ISOTOPIC ANALYSIS OF THE COALS AND ASHES FROM COAL FIRED POWER PLANTS OF TURKEY**

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We have investigated the ashes produced by combustion of lignite in power stations of Turkey together with the parent coal samples with the aim of estimation of potential adverse impact on human health. Gamma-isotopic analysis technique was used to determine radium, uranium, thorium, potassium and caesium contents in coal and fly ash samples originated from the southwestern and middle part of Anatolia. Gamma-isotopic analysis indicated that all samples contained Ra-226 (coal samples: 89-148 Bq/kg; ash sample: 15-122 Bq/kg), U-238 (coal samples: 2.1-3.9 ppm; ash samples: 8.9-140 ppm), Th-232 (coal samples: 1.8-10 ppm; ash samples: 7.4-38 ppm), and K-40 (coal samples: 26-67 Bq/kg; ash samples: not detected). The contents of Cs-134 and Cs-137 have been in the samples below detection limits.

## ANALYSIS OF MULTICOMPONENT SYSTEMS BY FAST NEUTRON ACTIVATION

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The author presents the data on developing accurate techniques for determining components of multicomponent systems using fast neutrons of a neutron generator. 14.5- and 2.5-MeV neutrons with a flux density of  $5 \times 10^{10}$  and  $7 \times 10^8$  n cm<sup>-2</sup> s<sup>-1</sup>, respectively, were used. The induced sample activity was measured by a gamma-spectrometer with a HPGe detector having an efficiency of 45% and an energy resolution of 2.1 keV.

Systematic and random errors are exhaustively studied; methods and devices are developed for diminishing the total error to 0.5-2.0 rel.% for macro-component determination (above 0.1 mass.%).

Based on the above investigations, accurate techniques are developed for determining various macro-concentrations of the following elements: B, N, O, F, Na, Al, Si, P, Cl, K, Ca, Ti, V, Mn, Fe, Ni, Cu, Ga, Se, Br, Sr, Y, Zr, Nb, Mo, Ru, Rh, Pd, Ag, Sn, Te, I, Ba, La, Ce, Nd, W, Re, Ir, Pt, As, Au, Tl, Pb. Nuclear reactions and measured isotopes used for determining the above elements are listed, as well as examples of results of the analysis of various objects.



## DETERMINATION OF IODINE IN DRINKING WATER

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A recent medical investigation of the thyroid gland indicates the evidence of goiter in some area of Libya. To investigate this problem further, there is a need to determine the concentration levels of iodine in drinking water.

In this study, drinking water samples have been analysed for iodine levels using radiochemical neutron activation analysis technique. The developed method is simple, fast and accurate. 2.5 ml of drinking water is pipetted into a polyethylene capsule and 50  $\mu\text{l}$  of concentrated  $\text{NH}_4\text{OH}$  is added. The capsule is then sealed, cooled in liq.  $\text{N}_2$  and irradiated in a thermal neutron flux of  $5 \times 10^{12} \text{ n.cm}^{-2}.\text{sec}^{-1}$  for five minutes. After irradiation the capsule is opened and its content is transferred into a separatory funnel containing 3 ml of deionised water. The mixture is acidified with 20  $\mu\text{l}$  concentrated  $\text{HCl}$  followed by the addition of 5 ml of iodine solution in  $\text{CCl}_4$  and shaken vigorously for at least one minute. The organic phase is transferred into a polyethylene test tube. As a standard, solution of iodine, prepared from  $\text{NH}_4\text{I}$  and irradiated in the same way, is used. Samples and standards were counted for 10 minutes on a  $\text{HP}(\text{Ge})$  detector and the peak area at 442.7 keV  $\gamma$ -ray from  $^{128}\text{I}$  was integrated. The concentrations of iodine in the samples were calculated by comparison of the gamma peak areas between the samples and the standard.

Samples of water were collected from 28 major wells connected to the drinking water network of different regions of Libya. Our results showed that the concentration levels of iodine in drinking water have considerable regional differences. The developed analytical method and possible links of iodine contents in drinking water with endemic goiter will be discussed.

## METHODOLOGICAL PROBLEMS OF ACTIVATION ANALYSIS WITH RADIONUCLIDE NEUTRON SOURCES

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The peculiarities of activation analysis with radionuclide neutron sources are associated with large weight samples and group irradiation of them. This may lead to a number of specific effects.

Analysis of the possible changes of parameters of the activation equation allowed to reveal a number of factors that may cause errors. In the performed investigations the influence of these factors on accuracy of analysis has been estimated and the methods and techniques for correct consideration of them have been elaborated.

1. The problem of calibration of irradiation positions in the multielement analysis with group irradiation of samples has been investigated and solved. It has been shown that the differences in position coefficients may reach up to 20% for different nuclides. The relations of these coefficients with nuclide activation parameters and spectral characteristics of the neutron field have been found and a method of position coefficients determination has been developed.

2. It has been shown that the influence of the possible deviation of resonance neutron spectrum from the  $1/E$  law is unimportant for the analysis results.

3. Comparative analysis of shielding coefficients for different types of ores and neutron flux depression coefficients showed that correctness of standardisation by using standard samples, having the matrix different from the studied one, and the possibility and conditions of applicability for different types of ores of the same position coefficients as well.

4. The conditions of isotropness of neutron albedo (and activation of samples) for different moderators have been found.

5. The effects of self-shielding of neutrons in gold-containing samples have been studied in detail. For the case of even distribution of gold in the sample the limits of the effects have been obtained in the whole range of concentrations at the irradiation by both thermal and full spectrum neutrons.

The method of correct determination of neutron self-shielding coefficients for real gold containing samples, that eliminates defects of the formerly elaborated method and eliminates substantial systematic errors, has been developed. It has been shown that the differentiation of self-shielding coefficients under the group irradiation of samples is observed, which should be taken into account.

Investigations carried out are the base for working out of the complex methods of element analysis, which is now being implemented in practice.

## **NUCLIDE COMPOSITION AND BURN-UP CHANGE OF VVER-1000 AND VVER-440 PWRs SPENT FUEL ALONG FUEL ELEMENT RADIUS**

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At present, VVER-1000 and VVER-440 are the basic types of power reactors in Russia. Therefore, much attention is paid to the investigation of fuel of the above mentioned reactors, especially to the elucidation of changes of nuclide composition as a function of burn-up.

Experimental data concerning the radial distribution of plutonium and transplutonium elements are practically absent in the literature. In this paper, analysis results of fifteen microsamples of the spent fuel taken by an ultrasonic method are presented.

The fuel burn-up was estimated by measurement of the accumulation of fission products by isotope dilution mass spectrometry using isotopes of neodymium-145 + neodymium-146 as burn-up monitors

The results obtained have shown that the distribution of plutonium along the fuel element radius has an irregular nature.

## URANIUM-233 AS AN ALTERNATIVE TO THE URANIUM-PLUTONIUM SPIKE IN DETERMINING OF THESE ELEMENTS IN NUCLEAR FUEL BY ISOTOPE DILUTION MASS SPECTROMETRY

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At present, isotope dilution mass spectrometry remains one of the most accurate method for the determination of uranium and plutonium in nuclear fuel.

Particular attention should be given to the preparation of complex spikes containing enriched isotopes of uranium and plutonium, because an error of the element determination is given to a considerable extent by the accuracy of the spike preparation. Furthermore, it is necessary to ensure the conversion of plutonium in fuel and spike solutions to the same chemical state, and in particular to the same valency before separation. This requires carrying out of redox reactions and may be a source of additional errors.

In the present paper, the authors examine a method for the determination of uranium and plutonium content in which only one isotope - uranium-233 is used as a spike and mass-spectrometry and alpha-spectrometry are used as measurement methods.

The procedure is easy in implementation and ensures the determination of plutonium with an error of 1.2%. This value is comparable to that of isotope dilution mass spectrometry analysis (0.6-1.2%) with the application of complex spikes.

## **DETEFF- A MONTE CARLO CODE FOR EFFICIENCY CALCULATIONS IN GAMMA - SPECTROMETRIC MEASUREMENTS**

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For gamma spectrometric measurements it is necessary to know the source-detector system efficiency. Frequently, reference sources with the desired matrixes and geometries don't exist and the efficiency of the system has to be evaluated by means of numeric methods.

The presented work show the description of the Monte-Carlo program DETEFF, that calculates the efficiency of cylindrical NaI, CsI, Ge or Si detectors for photons energy until 2 MeV and several sample geometries. These sources could be punctual, plane, cylindrical or rectangular. The energy spectrum appears on the screen simultaneously with the statistical simulation.

The program was validated for the analysis of environmental samples during the international intercomparison exercises. The calculated and experimentally estimated efficiencies coincide well in the standard deviations intervals.

## THE GOLD ANALYSIS IN SEDIMENTS BY ACTIVATION WITH NEUTRONS ISOTOPIC SOURCES AND X-RAY FLUORESCENCE

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By using neutron activation analysis with the help of the  $^{197}\text{Au}(n,\gamma)^{198}\text{Au}$  nuclear reaction, we analysed the native gold from the river sands and from sedimentary or hydrothermal quartziferous rocks, studying its concentration and its appearance forms.

For irradiation were used two isotopic neutron sources ( type  $^{241}\text{Am}-^9\text{Be}$  and  $^{239}\text{Pu}-^9\text{Be}$  ) which have a constant fluence of  $6.6 \cdot 10^7$  n/s. The sources were introduced in a paraffin block with the purpose of neutron thermalization. Systematic studies have been made for removing the analysis errors produced by neutron self-shielding and by geometrical factor in the samples containing gold.

The samples irradiated during 4-6 days were measured after a cooling time, calculated for removing the interference with the  $^{116m}\text{In}$  radionuclide.

The gamma-ray spectra emitted by the irradiated samples were measured with the help of a high-resolution type Ge (Li) detector coupled to a multichannel analyser type ICA-80.

The gold concentration was determined by measuring the 412 keV gamma-rays of the  $^{198}\text{Au}$  radionuclide, using standard samples. The detection limit was established at 27 ppm Au for this installation, what means that preferably preconcentrated samples are used instead of native samples with a lower content of gold.

Another approach used was X-ray fluorescence analysis, using two installations which work differently.

It was noticed that the analyser which uses the diffraction on the LiF crystal, for X-ray fluorescence spectrum analysis, emitted by the sample, has a reduced sensitivity for the gold determination (only 0.01%), but it has good resolution.

On the other hand, the X-ray fluorescence analyser which uses a Si(Li) detector, is extremely sensitive, reaching ppm concentrations for the gold determination, although it has a weaker resolution.

With the help of the latter system were determined 25 elements in a series of golden sands and sedimentary rocks samples. It was noticed that in the preconcentrated golden samples, the gold level can reach hundred of ppm and it is accompanied by high levels of Zr and Ti.

In conclusion, by using the studied radioanalytical methods for analysis, were investigated the golden zones from the so-called "The Golden Quarter" from the Western Carpathians mountains from Romania, localising the rivers containing golden alluvia.

## **APPLICATION OF INAA IN DETERMINATION OF TRACE ELEMENTS IN URINARY CALCULI**

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The concentration of Co, Fe, Se, and Zn in 53 urinary calculi of Libyan patients were determined by instrumental neutron activation analysis (INAA) and were found to be in the ppm range and to differ in concentration. A pattern of the concentration of trace elements in these stones was analysed regarding the age, sex, residency, position of the stones in the urinary tract and the presence of preoperative infection.

## A NEW METHOD OF RADIOSTRONTIUM CONTENT DETERMINATION USING SYNTHETIC SELECTIVE SORBENTS

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The present methodology is based on using synthetic selective sorbents containing functional groups of various "crown" ethers. These sorbents combine high selectivity and capacity of these ethers (50-60mg Sr/g dry sorbent) with good dynamic qualities of the synthetic fibres (filtration speed up to 3ml/cm<sup>2</sup> min). Similarly to ionites these fibres can be regenerated and used many times (not less than 400 cycles).

Description of the procedure. Strontium is effectively sorbed with fibres from the leaching solutions produced from different kinds of samples (nitric acid 5.5 mol/L to 7.5 mol/L). Then the fibres are washed with 6.5 mol/L nitric acid. When 20 free column volumes of the acid are used, the greater part of macro- and microcomponents are separated (97-99% Ca, Mg, Fe, as well as artificial and natural radionuclides including Ra).

The strontium sorbed is practically completely eluted in 10-12 free column volumes of ammonia solution containing disodium EDTA. Additional purification of strontium is provided as a result of its selective precipitation as sulphate in an EDTA solution having pH 5.0 to 5.2; Ca, Mg, Pb, rare-earth elements and other elements remain in the solution in this case.

If the necessity arises, additional purification from Ba and Ra can be achieved by the preliminary precipitation of their sulphates at pH 7.3 to 7.5. The total strontium yield for all stages of analysis amounts to 50-60%. Radiochemically pure strontium sulphate is deposited on the planchet and represents a source for radiometric counting.

The methodology has been tested while analyzing different types of real samples of soil, water, milk, potatoes, herbs, etc. The methodology is simple to use and it does not require a high qualification level of the personnel. It can easily be adapted for such instruments as liquid scintillation counters (with strontium-85 gamma-tracer) as well as low-background gas-flow counters (with stable strontium carrier).



## LOW-LEVEL RADIOACTIVE MEASUREMENTS OF RADIOCHEMICAL ISOLATED NUCLIDES FROM SAMPLES OF CONTAMINATED AREAS AND MATERIALS

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The investigation of radioactive contamination in the surroundings of NPP (nuclear power plants) and uranium mines demands the determination of radionuclides at extremely low activity levels, especially for the forecast of transportation by the different pathways and for the estimation of the efficiency of natural and artificial barriers for the storage of radioactive wastes.

The measurements of some nuclides by  $\alpha$ -,  $\beta$ - and  $\gamma$ -spectrometry are carried out in an underground laboratory (47 m thick rock cover). The  $\beta$ - and  $\gamma$ -detectors are individually shielded by 10 - 17 cm lead and are arranged in a metallic shielded counting chamber. The extremely low background enables measurements in the mBq level for chemically isolated fractions. Some applications of the developed radiochemical and instrumental methods are described:

i) Natural isotopes of Pb, Ra, Ac, Th and U in pit water and other sources can be isolated from the matrix and interfering nuclides by group separation methods and individual separations. Isotopes of Ra and Pb are precipitated with barium sulfate and measured by  $\gamma$ -spectrometry. An additional determination of  $^{226}\text{Ra}$  is possible by emanometry of the dissolved sulfates. Nuclides of Th and U are determined by  $\alpha$ -spectrometry after isolation by anion exchange and extraction steps.  $^{227}\text{Ac}$  is analyzed by  $\alpha$ -spectrometry of its daughter  $^{227}\text{Th}$  after growing and repeated separation from  $^{227}\text{Ac}$ .

ii) Artificial actinides and rare earth elements in water of the primary circuit of a NPP and in waters of nuclear fuel element storage basin are analyzed by  $\alpha$ - and  $\gamma$ -spectrometry of the separated fractions. The isolation of Pu-nuclides from 3-valent actinides, the matrix (boric acid) and interfering nuclides is realized by phosphate precipitation, extraction and a cation exchange procedure. The group of REE and 3-valent actinides are electrodeposited together. The results obtained by  $\alpha$ - and  $\gamma$ -spectrometry are identical. The chemical yield is controlled by addition of  $\mu\text{g}$ -amounts of a La-carrier and measurement by ICP-MS.

iii) For selected nuclides a further background reduction is reached by using  $\beta$ - $\gamma$ -coincidence techniques with a HPGe-detector as the  $\gamma$ -spectrometry system and a thin plastic scintillator as the  $\beta$ -sensitive detector.

Radiochemical procedures are applied for the determination of

iv) nuclides from the uranium-, thorium- and actinium- decay series in incorporation samples, plant materials, air samples and soils.

v) artificial actinides and Sr-90 in incorporation samples, plant materials and air filters.

## THE ANALYSIS OF $\alpha$ -NUCLIDES IN HLLW IN THE TRPO PROCESS

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The hot test of the trialkyl ( $C_6-C_8$ ) phosphine oxide (TRPO) process for the removal of transuranium (TRU) elements from a genuine high level liquid waste (HLLW) in China has been carried out in the Institute of Nuclear Energy Technology, Tsinghua University, Beijing. This paper presents the results and the analytical methods for the determination of TRU elements in the hot test. The total activity of  $\alpha$ -nuclides was determined using a Au-Si surface barrier detector. The activities of  $^{237}\text{Np}$ ,  $^{239}\text{Pu}$ ,  $^{241}\text{Am}$  were determined after Np, Pu, Am were separated from the HLLW with  $\text{P}_{204}$ . The experimental results show that more than 98% of Pu is present as  $\text{Pu}^{4+}$  species and more than 70% of Np as  $\text{Np}^{4+}$  species in the genuine HLLW. In the experiment, some  $\text{NpO}_2^+$  species was converted to  $\text{Np}^{4+}$  species and extracted into the organic phase nearly completely, thus the decontamination factor of Np was higher than was expected. Plutonium was extracted completely after 3 stages of centrifugal extractor, from the fourth extractor to the twelfth extractor the content of plutonium in the aqueous phase was not changed, which means that a little amount of plutonium (about 5%) in the genuine HLLW existed as polymerized or hydrolyzed species which could not be extracted by TRPO. The distribution ratios of americium increased with the extraction times due to the decrease of the extractable elements, such as RE and  $\text{Fe}^{3+}$ . After 12 stages of centrifugal extractor, the activities of  $^{237}\text{Np}$ ,  $^{239}\text{Pu}$ ,  $^{241}\text{Am}$  were 20, 34, 135Bq/mL respectively, and the HLLW became LLLW of  $\alpha$  radiation.

## STUDY OF REACTOR PRESSURE VESSEL STEEL SPECIMENS USING GAMMA AND MÖSSBAUER SPECTROSCOPY

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The reactor pressure vessel (RPV) is the most important component of a nuclear power plant (NPP) and its condition significantly affects the NPP's lifetime and operational characteristics. One of the basic requirements in nuclear reactor technique is ensuring the sufficient safety margin and reliability of the materials used concerning their operational mechanical, thermal or radiation treatment.

In our study the gamma spectroscopic analyses and first experimental results of original irradiated RPV-surveillance specimens are presented and discussed. In 1994, the new "Extended Surveillance Specimen Program for Nuclear Reactor Material Study" was started in collaboration with NPP V-2 Bohunice (Slovakia). The first batch of the specially prepared specimens for Mössbauer spectroscopy, treated 1 year into operated nuclear reactor (equivalent to 5 years of loading RPV-steel) was measured and interpreted using the new four components approach with the aim to observe microstructural changes due to thermal and neutron treatment resulting from operating conditions in NPP. The irradiation level of the used RPV-steel specimens reached the following maximal values:

Energy of neutrons	Neutron flux [ $\text{m}^{-2} \text{s}^{-1}$ ]	Neutron fluence [ $\text{m}^{-2}$ ]
> 0.1 MeV	$5.1 \times 10^{16}$	$1.2 \times 10^{24}$
> 0.5 MeV	$2.8 \times 10^{16}$	$6.7 \times 10^{23}$
> 1.0 MeV	$1.5 \times 10^{16}$	$3.6 \times 10^{23}$

The most significant change of Mössbauer spectra is observable in areas under first two components. The deterioration mechanism of RPV-steel specimens owing to fast neutron bombardment is shown in the decrease of the ratio of pure  $\alpha$ -iron component presence. The significant percentual decrease of about 9.9%, 7.4%, 7.9%, 4.5%, 5.3% and 5.8% is observed in all of the 6 specimens. As was expected, the value of Mössbauer effect is lower of about 4% in the case of irradiated specimens.

The total specific activity of the first batch of specimens (sample k716 BM-I with the weight of 25.6 mg) was  $3.2 \times 10^7 \text{ Bq/g}$ . It is caused mostly due to presence of  $^{60}\text{Co}$  and  $^{54}\text{Mn}$  as was confirmed by gamma spectroscopy measurements.

The results obtained from the present measurements indicate that MS is an effective technique for the evaluation of microstructural changes in RPV-steels and, in combination with other spectroscopic methods (Positron Annihilation Techniques, Transmission Electron Microscopy, ...) can contribute to an increase of the NPPs operational safety. Interpretations of the results obtained from the angular correlation PAS technique are in agreement with the findings from the Mössbauer effect experiments. It will be very interesting to study the second batch of the specimens, which was taken out of the reactor after 2 years of operation in July this year.

## APPLICATION OF X-RAY FLUORESCENCE ANALYSIS IN PAINT VERIFICATION

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X-Ray fluorescence analysis (XRF) with radionuclide excitation is a powerful tool for non-invasive investigation of oil or water-colour paintings and frescoes. The principle of paints or frescoes dating and verification is based on the pigment analysis from the point of view of the pigment type identification.

It is well known that a particular type of pigment was used from a certain year or in a certain period. For example, the zinc white was used since 1840, chromine yellow was very favourable in the second part of the last century, etc. The number of pigments used, for example, in the 15<sup>th</sup> or 16<sup>th</sup> century was limited (e.g. of whites only lead white was used) and the age of such a painting is very simple to verify. The second possibility of more precise paint dating of a particular painter is based on the knowledge that there are some periods in his life in which he preferred some type of pigments towards the others. It is obvious that this way needs a large knowledge of the painter's working life and also a bulk of analyses of his paintings from different periods of his life.

The main advantages of this method are :

- ◆ XRF is a non-invasive method, the analysed sample is not affected, consumed or changed and it is possible to use it for other analysis;
- ◆ the analysis is very fast, the results of one painting spot analysis are known within a few minutes;
- ◆ the measuring equipment is mobile, it is possible to analyze unmovable objects (frescoes), very valuable paintings etc.;
- ◆ XRF is a very cheap technique;

while the disadvantages are:

- ◆ it is only possible to decide that a painting was painted after a certain year (from the year of the latest pigment used);
- ◆ the whole layer of painting is analyzed without the knowledge of the composition of separate coats;
- ◆ it is impossible to find the natural or artificial pigment origin, there is no information on crystal structure.

For XRF a 1 MBq <sup>241</sup>Am annular source with Be window is used for excitation, the source gamma radiation is collimated through a cone with a 5 mm diameter hole (the spot analyzed has a diameter of approx. 6 - 7 mm if the aperture is not used), the excited radiation is detected by a Si(Li) semiconductor detector connected with a multichannel analyzer. The presence of As, Ba, Ca, Cd, Co, Cr, Cu, Fe, Hg, I, Mn, Mo, Ni, Pb, Se, Sn Sr, Ti, Zn and possibly Bi, U, W and Zr is observed.

The typical procedure is as follows:

- ◆ element analysis of a spot of the painting where only organic pigments are supposed to be present (e.g. spot analysis of black colour where the presence of only C or C-H is supposed) to find a composition of the painting background - usually Ca, Pb, Ba, Zn white pigments;
- ◆ element analysis of spots of individual colours;
- ◆ identification of the pigments used, searching for their earliest application and the decision if this corresponds to the painter practise in the supposed period of his life.

## **INSTRUMENTAL NEUTRON ACTIVATION DETERMINATION OF IMPURITIES IN TECHNICAL COBALT AND NICKEL**

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Since technical cobalt and nickel are in wide use in producing a variety of alloys, in particular on the basis of refractory materials (Ti, Mo, W, etc.), the preliminary determination of expected impurities in these materials is necessary.

Multielement INAA techniques have been developed for the determination of 13 and 16 impurities with a limit of detection from 100 ng/g upto 100,000 and 10,000 ng/g, in technical cobalt and nickel, respectively, by using thermal neutrons and the Cd-channel of a nuclear reactor.

Contents of Na, Al, Cl, K, Ca, V, Cr, Mn, Ni, Cu, As, Mo, W in cobalt and Na, Al, K, Sc, Cr, Mn, Fe, Co, Cu, Zn, As, Ag, Cd, Sn, Sb, W in nickel were determined, because complex conditions of irradiation in thermal flux, optimal time parameters, and mass of samples have been chosen. For the determination of Ni in Co the samples have been irradiated in the Cd-channel for 1 min. Spectrum was measured after 3-4 days.

Because  $^{59}\text{Co}$  has a large capture cross-section for neutrons, the effects of the self-shielding and disturbance of neutron flux, the self-absorption of gamma-rays in the sample were taken into account by using some references and based on results that have been obtained in preliminary experiments. Samples and standards have been placed in such a way that the neutron flux disturbance amounted to less than 2-3%. A filter consisting of Al-Pb-Cd-Cu was used for absorption of low-energy  $\gamma$ -rays of  $^{60\text{m}}\text{Co}$  and  $^{60}\text{Co}$ .

## COMPLEX RADIOCHEMICAL TECHNIQUE FOR THE DETERMINATION OF Pu, Am AND Sr-90 IN SOIL SAMPLES OF THE SNTS.

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Research on the distribution of radionuclides in various fractions of soil of the former Semipalatinsk Nuclear Test Site (SNTS) has resulted in the necessity to determine all retained artificial radionuclides - products of nuclear tests in separate soil fractions. These samples have frequently masses of a few grammes and their additional division can result in large errors and loss of information. Therefore, we have developed a technique based on chromatographic extraction of Pu on a column with trioctylamine (TOA), while Am and Sr pass into effluent. The separation of Am, Sr-90 and Y-90 can be carried out on a column with bi-2-ethylhexyl phosphoric acid (B2EHPA).

The analysis scheme is as follows. The sample calcinated at 400°C is decomposed in a mixture of HF+HNO<sub>3</sub> with an addition of H<sub>3</sub>BO<sub>3</sub> to prevent formation of insoluble fluorides. To the resulting solution KNO<sub>2</sub> is added to convert plutonium into Pu<sup>4+</sup> and extraction of Pu is carried out using a column with TOA. The effluent is used for separation of Y (Sr-90) and Am. Pu from the column is washed off by benzene and is re-extracted by a solution of H<sub>2</sub>SO<sub>4</sub>. The preparation of a spectrometric source consists in evaporation, dissolution of the residue in 1M HNO<sub>3</sub>, and coprecipitation of Pu with NdF<sub>3</sub> in the presence of 5M HF. Y and Am are extracted from the effluent using the following scheme. Precipitation with CaF<sub>2</sub> is carried out 2 times, then pH of the solution is adjusted to 1.8-2.0, and Y and Am are extracted on the column with B2EHPA. Am is eluted by 0.5M HNO<sub>3</sub>, and Y by 3M HNO<sub>3</sub>. The preparation of a spectrometric source of Am is similar to that of Pu, but for Y precipitation is used of YF<sub>3</sub> with a rather large (500 µg) amount of carrier of stable Y. The determination of the contents of Pu and Am in investigated samples is carried out by alpha-spectrometry, and the determination of that of Sr-90 (Y-90) is carried out by beta-spectrometry. The chemical yield is determined using tracers of Pu-236, Am-243 and Y-88, which are introduced at the initial stage of analysis. The yield of Y-88 is determined by gamma-spectrometric measurement.

Investigation of metrological characteristics of the technique carried out with both artificial mixtures and real samples has shown good reproducibility, sensitivity and selectivity of the technique, which is used for the characterization of contamination of the SNTS.

## **PROMPT- GAMMA NEUTRON ACTIVATION ANALYSIS OF BULK SAMPLES**

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A 5 Ci Am-Be radioisotope source which provides  $1.1 \times 10^7$  n/s has been used in conjunction with coaxial HPGe detector for doing bulk elemental analysis by neutron capture in sample. Such technique has been successfully tested for determining the Ca/Si ratio in concrete samples. A comparison made between this method and a chemical process resulted in good agreement of the results yielded by both the methods.

## ELEMENTAL ANALYSIS OF SELECTED SAUDI ARABIAN COINS USING ACCELERATOR BASED SLOW NEUTRONS

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An accelerator-based slow neutron activation analysis facility has been recently developed at KFUPM. The facility is based on the  $D(d,n)^3\text{He}$  reaction. Fast neutrons with an average energy of 2.5 MeV are produced at the KFUPM 350 keV ion accelerator by accelerating a  $700\ \mu\text{A}$  deuterium ion beam from the low intensity duoplasmatron ion source to an energy of 250 keV and transporting it to the end of the  $0^\circ$  beam line where a water-cooled solid deuterium target is situated. The fast neutrons are slowed down in a cylindrical high-density polyethylene moderator. The slow-neutron flux has a maximum intensity of  $2.5 \times 10^6\ \text{n/cm}^2/\text{s}$ . For flux normalization purposes, the neutron flux is monitored on-line using a NE213 liquid scintillation detector. The detector is situated at a distance of 275 cm from the deuterium target and an angle of  $90^\circ$  with respect to the beam axis. The  $\gamma$ -ray counting station is placed in a separate room and is based on a shielded HP-GMX detector which is connected to a PC based data acquisition and analysis system. The facility has an adequate thermal-neutron flux that is relatively homogeneous at the sample site and, since it is a non-destructive bulk analysis method, requires little or no sample preparation.

We carried out a study and found that the facility is well suited for the elemental analysis of coins. Indeed a selected number of Saudi Arabian coins of 10 Halala denomination dating from 1379 H - 1408 H (1958-1987 G) were each irradiated in the accelerator-based slow neutron flux for 1.5 hours and left to decay for 10 minutes. The induced  $\gamma$ -ray activities were then counted for 1.5 hours. The elements that were determined in the coins were Cu, Ni, and Mn. Calibration curves were established for these activated elements in the coins using two pure foils of each element with different weights. The 10 Halala coins contained Cu and Ni in major quantities (Cu: 70-75 wt.%, Ni: 20-25 wt.%) as well as Mn in minor (< 0.5 wt.%) quantities.

Results of the experiment will be discussed. Advantages of the method with respect to other techniques such as those based on chemical analysis, reactor and 14 MeV neutron activation, as well as PIXE and X-ray fluorescence will be highlighted. Details of the experiment with supporting figures and tables will be presented.



## MAPPING OF TRUE COINCIDENCE EFFECT VALUE FOR VOLUMINOUS SOURCES MEASURED WITH HPGE

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To compute the true coincidence effect for a voluminous source it is necessary to make an integration of the effect value over the sample volume [1]. The method developed of the correction computation is based on using as input parameters both the full peak efficiency map generated in advance and the measured intrinsic P/T-calibration. It was demonstrated that such approach works quite well for HPGe detectors with up to 60% relative efficiency [2]. It is well known that the effect value depends on the distance of the point under consideration and the detector. Applying the developed software we have reconstructed a three dimensional coincidence effect map inside the sample (cylinder, Marinelli beaker). The stability of the results with respect to input parameters is discussed. The influence of P/T-calibration variations (e.g. due to radiation scattering in the sample) on the map view are also considered.

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## NEW METHOD OF DETERMINATION OF RADIOSTRONTIUM BY USING THE SYNTHETIC SELECTIVE SORBENTS (TYPE VS-15)

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At present, a lot of work has been published on using the synthetic fibrous sorbents for selective extraction of different elements. We have designed a new class of synthetic fibrous sorbents (type VS-15), which allows to develop new unique strategies of extraction of different radionuclides from mixtures of complex chemical composition. Sorbents of this type combine high selectivity of crown-ethers with good dynamic features. In the present work the strategy of extraction of radiostrontium is described using a synthetic fibrous sorbent of the type VS-15.

Strontium is highly effectively absorbed on a filament from the 5.5-7.5 M nitric acid. Washing of the filament with 6.5 M nitric acid allows to separate a majority of disturbing macro- and microcomponents (97-99% of Cf, Mg, Fe, as well as artificial and natural radionuclides, including Ra). Strontium is eluted with 10-12 free volumes EDTA with pH10-12. If needed, additional cleaning of Ba and Ra traces can be performed. The total yield of strontium in all stages of analysis amounts to 75-82%. Radiochemically clean strontium sulphate is deposited to the substrate and its radioactivity is measured using a beta-counter. The time needed for the analysis is 4 hours.

For the strontium determination in natural water, large volumes (80-100L) are passed through the column with 10g sorbent at the speed of 20L/hour without preliminary preparation. In this case, the yield of strontium extraction amounts to 65-75%. The method was successfully used in the Chernobyl Zone for radiostrontium control and in assays of of different test samples of ground and natural water, samples of vegetation, milk, etc.

## PRECISE DETERMINATION OF SILICON MACROCONCENTRATION

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The possibilities of precise silicon determination by the neutron generator activation analysis (NGAA) are studied using a "SAMES" T-400 universal neutron generator with the maximum flux density of 14-MeV neutrons of  $5 \times 10^{10} \text{ cm}^{-2} \text{ s}^{-1}$ . The short-lived isotopes  $^{28}\text{Al}$  and  $^{29}\text{Al}$  are used. The concentration range of silicon determination is from 1 to 70 wt % for various compounds and alloys.

Due to the short half-lives of  $^{28}\text{Al}$  and  $^{29}\text{Al}$ , the determination of silicon by fast neutron activation is subject to many inaccuracies; i.e. variation in irradiation and measurement conditions, effects of high counting rate, diverse geometric factors, interferences of gamma-lines, and other sources of errors. One of the key problems of the technique elaboration is studying and correct accounting of all possible sources of systematic errors. The ways to determine correction factors and to eliminate errors are described. The metrological characteristics of the developed procedures, such as the random and systematic errors, relative standard deviations, confidence intervals, and others are presented. A procedure for calculating the general error of results of analysis is proposed.

The potential of using  $^{28}\text{Al}$  and  $^{29}\text{Al}$  as analytical isotopes is shown at the silicon determination in Al-Si alloys, when  $^{28}\text{Al}$  is generated from aluminum by the (n, $\gamma$ ) reaction. The examples of results of silicon macro-content determination in various materials are given.

## AN EASY AND ACCURATE METHOD FOR $^{226}\text{Ra}$ DETERMINATION IN RIVER WATERS BY LIQUID-SCINTILLATION COUNTING

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$^{226}\text{Ra}$  activity concentration was determined in river water using a low background liquid scintillation counter Wallac Quantulus 1220.

Water samples were acidified immediately after collection. Some 500 mL of water was taken in each analysis. Radium was extracted from the samples as  $\text{Ra-BaSO}_4$  precipitate which was afterwards dissolved with EDTA at pH 9. The solution was transferred into a low potassium glass vial and then mixed with a scintillation cocktail. For a comparison, two different scintillation cocktails Pharmacia Optiphase Hisafe II and Pharmacia Optiphase Hisafe 3 have been selected.

A standard solution of  $^{226}\text{Ra}$  was used to determine the total efficiency yield (counting efficiency multiplied by the recovery yield). The chemical procedure was applied to 500 mL of distilled water traced with a known amount of  $^{226}\text{Ra}$ . In this way, the mean total radiochemical yield of 60 % was found, considering the full alpha spectra.

In the liquid scintillation counter, alpha and beta spectrum can be acquired simultaneously using its pulse shape analyzer (PSA). A careful calibration study of the PSA parameter using  $^{239}\text{Pu}$  and  $^{40}\text{K}$  standard solutions was done in order to obtain the best alpha-beta separation. Alpha spectra were used for the activity calculation.

One single measurement performed one month after radium separation allows to calculate the  $^{226}\text{Ra}$  concentration, as well as to appreciate the presence of other alpha emitters, which are not daughters of  $^{226}\text{Ra}$ , in the sample. This can be checked by comparing the results obtained using the full alpha spectrum and the  $^{214}\text{Po}$  window of the alpha spectrum. The latter information can be used to evaluate the  $^{224}\text{Ra}$  concentration using an additional measurement done just after radium chemical separation.

The method has been applied for the determination of  $^{226}\text{Ra}$  and  $^{224}\text{Ra}$  activity concentrations in river water collected at different places of the Odiel river estuarine area (South-west of Spain). Several fertilizer production plants release some wastes (phosphogypsum) to the estuary, containing high activity levels of natural radionuclides, e.g., radium. Therefore, it may be expected that the water contains enhanced levels of this element.

## DETERMINATION OF IMPURITIES IN MOLYBDENUM AND TUNGSTEN BY INAA

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INAA techniques for the determination of Na, K, Sc, V, Cr, Mn, Fe, Co, Ni, Cu, As, Se, Zr, Mo, Ag, Sn, Sb, Cs, Ba, Hf, Ta, W, Re, Au, Th, U with limit of detection from 0.1 ng/g up to 1000 ng/g in different molybdenum and tungsten products have been developed.

Determination of this large number of impurities was achieved by using complex conditions of irradiation, including various neutron energies and choosing optimal time parameters.

Self-shielding and disturbance of neutron flux, absorption of gamma-rays in the sample were taken into account with the help of literature data and with the help of preliminary obtained experimental results. Samples and standards have been placed in such a way that neutron flux disturbances were less than 2-3%.

The contents of P and S in molybdenum and tungsten were determined by measurements of  $\beta$ -activity of  $^{32}\text{P}$  by using a  $\beta$ -spectrometer with an anthracene crystal (40 × 15 mm) in the energy range of 1500 - 1700 keV. To separate the  $^{32}\text{P}$  activity originating from P and S, the same sample was irradiated with both thermal and epithermal neutrons (the thickness of the Cd cover was 1.0 mm). The results were obtained by solving a system of linear equations. The simultaneous determination of P and S by via  $^{32}\text{P}$  can be carried out if the element contents differ by not more than 1 - 1.5 orders of magnitude.

The methods have been used to control the products of processing of refractory materials, to study the dynamics of impurities and doping elements at all stages of production process.

## DETERMINATION OF POLONIUM-210 IN PHOSPHORIC ACID

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Soft-error rate is of great concern in the assessment of the reliability of semiconductor memory devices. Soft errors are intermittent errors that occur as a result of the passage of ionizing radiation through the memory cells of semiconductor devices. The most common source of this radiation is alpha radiation from naturally occurring alpha emitters, which are found in trace amounts in every materials used for semiconductor memory devices.<sup>1</sup> Since the finding of alpha-particle-induced soft-errors, many laboratories in the world have devoted considerable efforts to reducing the alpha emission rate from materials for fabricating semiconductor devices. Although the efforts succeeded in reducing the alpha emission rate from device packaging materials down to a level of less than 0.001 alphas cm<sup>-2</sup> h<sup>-1</sup>, it was then found that <sup>210</sup>Po in phosphoric acid, a common chemical for the processing of silicon wafers, can be a significant source of alpha contamination of the processed wafers.<sup>2</sup>

Although approximately 0.07 mBq ml<sup>-1</sup> of <sup>210</sup>Po contamination in phosphoric acid was estimated as the maximum allowable concentration level,<sup>2</sup> reliable and convenient routine methods for the determination of trace <sup>210</sup>Po in phosphoric acid have not been established, and appropriate analytical techniques for the determination of traces of <sup>210</sup>Po in phosphoric acid have strongly been desired. In this work an analytical method was developed for the determination of trace <sup>210</sup>Po in phosphoric acid.

For the determination of <sup>210</sup>Po in phosphoric acid, calibrated <sup>209</sup>Po was used as a yield tracer. The present method consists of (1) addition of known amount of <sup>209</sup>Po to 5 ml of a phosphoric acid sample followed by isotopic equilibration, (2) adjustment of the solution to pH 2 with 18 M sodium hydroxide (*i.e.* preparation of electrolytic solution), (3) electrodepositional separation (and preparation of the source for counting) of Po on a stainless-steel disk of a 25 mm diameter by applying a constant direct current (0.5 A) for 3 h to the electrolytic solution and (4) alpha-ray spectrometry. By the present method, more than 95 % of Po isotopes, together with <sup>210</sup>Pb, were separated on the stainless-steel disk from other naturally occurring alpha emitters in the electrolyte. The minimum detectable activity of <sup>210</sup>Po in 5 ml of phosphoric acid is about 0.05 mBq by counting the alpha activity of electrodeposited source for 10 days under a counting efficiency of 30 %.

The present analytical method was applied to the samples of commercially available phosphoric acid, and the concentration of <sup>210</sup>Po in the samples for semiconductor processing were in a range from 0.01 to 1.51 mBq ml<sup>-1</sup>.

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## SHORT-TIME INSTRUMENTAL NEUTRON ACTIVATION ANALYSIS

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After the development of new nuclear reactor concepts, such as the energy amplifier, consisting of the combination of a subcritical reactor and a high energy particle accelerator with lead target for a spallation neutron source, high intensity neutron sources will continue to be available. Thus also neutron activation analysis will continue to be a valuable analytical method mainly after certain improvements, in spite of the competition of other advanced non-nuclear methods such as ICP. One of the main improvements of instrumental neutron activation analysis is the incorporation of a loss-free counting unit into the gamma-ray spectrometry system, permitting the instrumental correction of count losses due to high dead times at high count rates [1]. This permits the measurement of short- and medium-lived nuclides in the presence of longer-lived nuclides at high count rates after short cooling times, enhancing also the analytical throughput rate. This also facilitates the automation of the analytical system with a sample changer, since in a series of unknown samples even highly radioactive samples can be measured because of the enhanced count rate range. This advanced system for short-time neutron activation analysis has been used for various applications, such as trace multielement analysis in ceramics for archaeometrical studies, isotopic uranium analysis of nuclear safeguards samples a.o. It is also envisaged to perform on-line short-time neutron activation analysis with a loop at the reactor core. It should be noted that loss-free counting does not improve the counting statistics and a dual system measuring also the uncorrected spectrum should be used to determine the uncertainty. For counting statistics improvement several other techniques, such as cyclic and cumulative activation as well as radioactive decay compensation have been applied especially for short-lived nuclide analysis, where the counting time is limited [2].

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## **APPLICATION OF INSTRUMENTAL NEUTRON ACTIVATION ANALYSIS AND X-RAY FLUORESCENCE ANALYSIS TO EXAMINATION OF THE AUTHENTICITY OF ART OBJECTS.**

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Technological examinations of art objects prior to their restoration and attempts to establish the authenticity of an object and its attribution are more and more popular in the circle of Polish historians of art, conservators and experts in museum management. They are aimed at establishing the origin of object, the applied materials and techniques as well as the state of its preservation. The analysis of materials is nowadays carried out with the aid of very sensitive physico-chemical methods which are often complementary to one another.

In the Institute of Nuclear Chemistry and Technology in Warsaw in collaboration with the Department of Preservation and Restoration of Works of Art of the Academy of Fine Arts in Cracow and National Museum in Warsaw systematic studies using nuclear methods, particularly instrumental neutron activation analysis and x-ray fluorescence analysis, have been carried out on the panel paintings from the Krakowska-Nowosądecka School and Silesian School of the period from the XIV-XVII-th century, Chinese and Thai porcelains and mummies fillings of Egyptian sarcophagi. A number of important data on the concentration of trace elements, mainly in chalk and gypsum grounds, in lead white and tin-lead yellow, in clay, porcelains and mummies fillings were collected. These studies will provide new data to the existing data base, will permit to compare materials used by various schools and individual artists.



## TRACE ELEMENTS IN SOME NEW ROMANIAN PHYTOTHERAPEUTIC DRUGS INVESTIGATED BY NEUTRON ACTIVATION ANALYSIS

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Instrumental neutron activation analysis (INAA) has been used to assess the concentrations of Al, As, Au, Ba, Br, Ca, Cl, Ce, Co, Cr, Cs, Cu, Fe, Hf, Hg, K, La, Lu, Mg, Mn, Na, Rb, Sb, Sc, Sm, Th, V, and Zn in four new Romanian phytotherapeutic drugs registered as Fitolip, Fitodiab, Retinomion and Neuromion. These phytotherapeutic drugs were selectively prepared of fruits, flowers, leaves and shells of twelve plants used in non-conventional medicine. Clinical trials showed that the drugs are very well tolerated and that their activity is directed mainly towards the lowering of lipids (Fitolip) and of glucose (Fitodiab), the prevention of retinopathic complications (Retinomion), and the treatment of diabetic neuropathies (Neuromion). Percent levels of Mg, K, thousands of ppm of Ca, thousands to hundreds of ppm of Cl, Na, Al, hundreds of ppm of Fe, hundreds to tens of ppm of Mn, tens of ppm of Zn, about ten ppm down to one ppm of Ba, Rb, Br, Cu, Cr, hundreds of ppb of V, Co, Ce, La, tens of ppb of Au, As, Hg, Cs, Sc, Sb, Th, Sm, Hf and ppb level for Lu have been observed. The therapeutic value of the microelement content in the drugs is discussed based on a comparison to the daily necessary intake.

## NEUTRON ACTIVATION ANALYSIS OF SEA BUCK-THORN (*HIPPOPHAE RHAMNOIDES*) MEDICINAL PLANT

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Sea buck-thorn medicinal plant (*Hippophae rhamnoides*) contains various mineral substances, essential macro- and micro-elements besides proteins, glucides, fatty acids as well as important quantities of essential aminoacids, vitamins (C, B<sub>1</sub>, B<sub>2</sub>, B<sub>6</sub>, B<sub>12</sub>, E, F, P), serotonin, carotenoids and enzymes. Due to many active physiological substances with beneficial effect for human beings and animals this excellent therapeutic plant is used both as a food (tea, preserves) and in the pharmaceutical industry (parts of the plant are used to obtain oil, hydrosoluble extract or, together with other medicinal plants as a component of some non-conventional drugs). Therapeutical action of sea buck-thorn is directed mainly in wounds, burns, hepatic diseases and vitamin deficiency cures. To determine the trace and micro-element content of this plant, ashes of leaves and fruits as well as a hydrosoluble fruits extract were analysed by instrumental neutron activation analysis (INAA) method at the VVR-S reactor in a neutron flux of  $2.3 \times 10^{12} \text{ cm}^{-2} \cdot \text{s}^{-1}$ . The concentration of Al, As, Au, Ba, Br, Ca, Cl, Ce, Co, Cr, Cs, Cu, Eu, Fe, Hf, K, La, Mg, Mn, Na, Ni, Rb, Sb, Sc, Se, Sm, Sr, Th, U, V, Yb, and Zn is presented. The biological role and therapeutic action of several of the analysed elements is discussed.

**NEUTRON ACTIVATION ANALYSIS OF SOME BUILDING MATERIALS****M.N. Salagean<sup>1</sup>, A.I. Pantelica<sup>1</sup>, I.I. Georgescu<sup>2</sup>, M.I. Muntean<sup>2</sup>***<sup>1</sup>Institute of Physics and Nuclear Engineering "Horia Hulubei", PO Box MG-6,  
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The elemental composition of building materials is of interest with respect not only to the induced indoor radioactivity, but also as concerns toxic elements, such as As, Sb, Zn. In the present paper, the concentrations of As, Au, Ba, Br, Ca, Ce, Co, Cr, Cs, Eu, Fe, Hf, K, La, Lu, Mo, Na, Nd, Rb, Sb, Sc, Sr, Ta, Tb, Th, U, Yb, W and Zn in some Romanian building materials were determined by Instrumental Neutron Activation Analysis (INAA). The building materials analyzed are: raw material (75% limestone, 25% clay) used in the production of cement, cement samples from three different sites, furnace slag, phosphogypsum and a brick fabricated at a pressure of 40MPa at 20°C from slag, fly coal ash, phosphogypsum, lime and cement. The fly coal ashes from five Romanian coal-fired power plants, resulting from combustion of xyloide brown coals, lignite and bituminous-subbituminous coals were previously analyzed. One can observe a lower concentration of Au, As, Br, Sb, Zn and a higher concentration of Ca, Ba, Cr, Sr, Ce, Eu, Th in cement samples in comparison with raw material. The rather high content of Co, Cr, Th, U and Zn in brick is especially due to fly coal ash and slag, those being the most important components of the brick. The U and Th concentrations in the final bricks are in good agreement with the natural radioactivity measurements of <sup>226</sup>Ra and <sup>232</sup>Th.

## QUALITATIVE AND QUANTITATIVE ANALYSIS OF MEDIEVAL COINS STRUCK UNDER THE RULE OF MATHIAS BY EDXRF

**Zsuzsánna Sándor, Sándor Tölgyesi, Iván Gresits, László Járosi,  
József Solymosi, and Márta Juhász Káplánné\***

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Energy-Dispersive X-Ray Fluorescence technique was applied for analysis of silver coins to determine the elemental composition in a fast and nondestructive way. The non-destructive feature of analysis is very important when it is not possible to destroy the structure of the ancient, precious coins.

In the present work we have developed a quantitative method for samples with different size and geometry. This method was employed for coins with two components, Ag and Cu, where the content of the trace elements is under 0.1%. All the coins and the standards were exposed from an annular  $^{125}\text{I}$  source; the X-ray radiation was measured using a Si(Li) detector with associated electronics and a multichannel analyzer which was coupled to a computer.

The usefulness of the method has been confirmed by analyzing certified silver alloys. The agreement between the measured and the true values is good. The precision that is indicated by the relative standard deviation (RSD %) is under  $\pm 4\%$ .

Silver coins were analyzed from the collection of the Hungarian National Bank, struck in Hungary during the reign of Mathias (1458-1490).

In our contribution we present the concentration values of the main components and a list of trace elements present in coins.

## **GAMMA-SPECTROMETRIC ANALYSIS OF ENVIRONMENTAL SAMPLES OF COMPLEX COMPOSITION**

**I.A.Savushkin, A.M. Ragotner, A.V. Lukjanov, O.B. Gourko**

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Radiation monitoring of environmental conditions is based first of all on obtaining of true primary data – results of radionuclide analysis of object samples. Performing of such analysis has some special features associated with different physical and chemical characteristics of the sample composition such as inhomogeneity of a distribution of radionuclides in the measured samples, the absence of a complete set of their metrological standards. Moreover, it may be quite expensive to take all appropriate measures in the cases of accidents at nuclear installations.

After the Chernobyl NPP accident the problem of the development of a method for rapid on-line analysis of a large amount of samples became very important.

One of the possible methodologies of radionuclide gamma-spectrometric analysis is discussed in this paper in terms of sensitivity and accuracy of this method. The real sensitivity for specific applications and different measuring geometries is evaluated, as well as problems of measurement of inhomogeneous samples. The relative error and reliability of radionuclide gamma-spectrometric analysis results and the sensitivity of the method is calculated.

The application of the developed method to the analysis of building materials is also considered.

The method proposed allows to carry out radionuclide analysis of environmental samples of a complex configuration and composition with a high degree of accuracy and sensitivity. It is based on a large amount of the primary material which has been analyzed by the scientists of "Belarusgeology", Nuclear Power Engineering Institute/Academy of Sciences of BSSR, Institute of Power Engineering Problems/ National Academy of Sciences of Belarus.

This methodology may be recommended for the establishment and development of an environmental radiation monitoring system.

## **INVESTIGATION OF TRACE ELEMENTS IN CLAY MINERALS BY NEUTRON ACTIVATION ANALYSIS**

**M. Samareh Shahpasand**

*Physics Department, Kerman University, Kerman, Iran*

This report is about using fast neutron activation analysis technique to determine the concentration of some elements in clay minerals by 14 MeV neutron activation analysis.

## **NEUTRON ACTIVATION ANALYSIS OF NATURAL WATER APPLIED TO HYDROGEOLOGY**

**M. Samareh Shahpasand**

*Physics Department, Kerman University, Kerman, Iran*

The analysis of trace elements in ground water samples has been used to examine such hydrogeologic problems as the quality and capacity of water supplies, the origin and existence of ground water streams and ground water exchange with rivers. Examples of the activation analysis data for the determination of the origin of water from wells will be given.

## **DETERMINATION OF SOME TRACE ELEMENTS IN THE SERUM OF THALASEMIA DISEASED USING NEUTRON ACTIVATION ANALYSIS, PIXE AND ICP**

**M. Samareh Shahpasand, A. Molla (Fathemy) - Fathizadeh**

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The increase of some trace elements like Fe in the blood serum of Thalasemia diseased, can cause very intense disorder in the metabolism. Using desferal as a chelate complex binding agent, the amount of Fe and other elements in the blood can be controlled.

In this paper, the determination of some trace elements in the blood serum of Thalasemia patient after injection of desferal is reported. The blood samples collected from Ali Asghar Hospital were analysed using neutron activation analysis and gamma - spectroscopy. The results were compared with data obtained by PIXE and ICP method.

The results obtained in this study can be used in diagnosis and treatment of Thalasemia patients.



## NEUTRON ACTIVATION ANALYSIS AS AN USEFUL INSTRUMENT TO SOLVE SOME COMPLICATED QUESTIONS OF THE ENVIRONMENTAL STATUS

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Environmental samples are traditionally complicated objects for elemental analysis. This is due to the fact that in most environmental samples very low concentrations of many trace elements are observed. In addition, most analytical techniques require different sample preparations. In this connection, instrumental neutron activation analysis is probably the only exception from the indicated rule. NAA allows to determine concentrations about 40 chemical elements (trace elements, different heavy metals and also biologically essential elements) in different environmental samples (water, soils, plants and many others) with the high sensitivity and accuracy. In this case all samples can be analysed in their natural state without any treatment.

In our work chemical element redistributions among soil and different parts of some species of plants were studied. It was found that concentrations of different chemical elements have peculiar features of given species of plants. In the natural conditions mean concentrations of chemical elements in plant leaves or roots can remain sufficiently constant, whereas under environmental pollution significant variations not only in plant elemental composition, but also in some physiological functions were found. In most cases, strong changes were observed for plant roots. In our experiment it was also shown that an increase in environment concentration of any one toxicant can lead to significant variations in the whole elemental composition of plants, including biologically essential chemical elements. In this case, concentrations of many elements important for plants can significantly be decreased. On the other hand, an increase of concentrations of different chemical elements in the environment can simultaneously lead to both increased and decreased (in some cases) element concentrations in plants.

**RADIOCHEMICAL METHODS FOR THE DETERMINATION OF  
<sup>55</sup>Fe, <sup>63</sup>Ni, <sup>210</sup>Po, <sup>210</sup>Pb, <sup>234</sup>U, <sup>238</sup>U AND <sup>239+240</sup>Pu  
IN THE ENVIRONMENTAL SAMPLES**

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Radiochemical procedures are described for the determination of natural (<sup>210</sup>Po, <sup>210</sup>Pb, <sup>234</sup>U and <sup>238</sup>U) and artificial (<sup>55</sup>Fe, <sup>63</sup>Ni, <sup>238</sup>Pu, <sup>239</sup>Pu and <sup>240</sup>Pu) isotopes in environmental samples (water, sediment and biological material). The methods are based on coprecipitation of polonium, lead, uranium and plutonium with manganese dioxide from natural water, mineralisation of sediment and biota samples, and sequential separation and purification of the radionuclides on anion exchange resin. The individual elements are electrodeposited on silver (polonium) or electroplated onto steel discs (uranium and plutonium) or copper discs (iron and nickel). The activities of <sup>210</sup>Po, <sup>234</sup>U, <sup>238</sup>Pu, <sup>239</sup>Pu and <sup>240</sup>Pu are measured by alpha spectrometry with low-level-activity silicon detectors. The <sup>210</sup>Pb activity was estimated on the basis of <sup>210</sup>Po ingrowth after the lead fraction was purified and stored for some months. The activity of <sup>55</sup>Fe and <sup>63</sup>Ni was measured by beta spectrometry using an anti-coincidence GM gas flow counter. A high recovery of these radionuclides was verified using certified reference materials.

## **ESTIMATION OF THE REPRESENTATIVE SAMPLE MASS IN NEUTRON ACTIVATION ANALYSIS**

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Equations are derived to associate the mass of a representative sample and such components of the total variance, which characterizes the dispersion of results of neutron activation analysis (NAA), as the variances of nonuniformity and replicate reproducibility. A method is proposed for approximate estimation of the minimum mass of the representative sample in the NAA of inhomogeneous materials.

## THE MAIN DIRECTION OF DEVELOPMENT OF NUCLEAR-PHYSICAL METHODS OF ANALYSIS FOR SCIENCE AND INDUSTRY OF KAZAKSTAN

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A need for the development of nuclear-physical methods of analysis (NPMA) in Kazakhstan is dictated by the necessity to solve a great number of analytical tasks connected with industrial, economical and social problems of the country. The main criterion for the choice of NPMA application fields has always been the rational use of their advantages in comparison with traditional chemical, physico-chemical and physical methods of analysis. Such advantages are: high sensitivity, multielementality, the possibility to carry out analysis without destruction of sample, in some cases it is a highly proximate method and has a high productivity.

Historically, the development of the NPMA in the INP has begun in the sixties with the development of activation analysis (AA) using the nuclear reactor "WWR-K" and the isochronous cyclotron. Another set of experimental devices was designed and manufactured specially for AA. These are: a neutron generator with continuous operation using a mixture of deuterium and tritium; an Sb-Be source of neutrons; an ironless toroidal beta-spectrometer of the type "Orange"; an automatic device for determination of oxygen in Balkhash copper and other objects; a set of pneumatic transportation devices at the reactor, of which one is used for uranium determination by the method of delayed neutrons (MDN). In spite of apparent advantages of AA, such as low element detection limits ( $10^{-2} + 10^{-4}$  ppm), this method is, in most cases, very expensive, labour-consuming and requires a high qualification of the personnel. Therefore, X-ray fluorescence analysis (XRFA) has also been developed in the INP. From the point of view of detection limits ( $10 \div 1$  ppm) the latter method is considerably worse than AA. However, it is a proximate method, cheaper, more simple and can be used to carry out mass analyses. The techniques developed in the INP, including all possible chemical and physical ways of sample treatment (preliminary concentration; use of strong-current roentgen tubes, focusing lenses, re-emitters and others) allowed to decrease the detection limits of many elements up to  $n \cdot 10^{-2}$  ppm. An essential addition to the above mentioned methods is atomic emission spectroscopy with inductively coupled plasma (AES-ICP) developed in the INP on the basis of spectroanalyser "GY-70p". From the point of view of the detection limits ( $10^{-1} \div 10^{-2}$  ppm), this method is comparable with AA, moreover it fills some gaps of AA allowing to determine the elements non-accessible by AA, such as Pb, Bi, Si and others. In following years, in connection with the intensified radiophobia, other methods for analysis of radionuclides have been developed in the Institute based on different ways of radiochemistry and alpha-, beta- and gamma-spectrometry.

In total, all enumerated methods provide possibilities to carry out both mass analyses and performance of fine scientific research. A great number of analytical tasks connected with archaeology, criminalistics, medicine, biology and agriculture has been solved. But main achievements were obtained in metallurgy - analysis of pure and suprapure materials; in geology - prospecting and calculation of stocks of mineral resources; in ecology and radioecology - control of environmental objects in industrial regions and at locations where nuclear tests were carried out (SNTS, "Azgir", "Lira", "Batolit" and others). Research carried out in the INP on improvements and applications of nuclear-physical methods of analysis in different fields of science and national economy of Kazakhstan are supported by the IAEA and the ISTC.

## X-RAY SPECTROMETRY OF TRANSURANIUM RADIONUCLIDES IN THE SAMPLES OF THE ENVIRONMENT

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As a result of accidents at NPP a lot of fuel-containing materials penetrates into the environment. There is a lot of transuranium radionuclides (U, Np) in fuel-containing materials. It is a specific characteristics of the accident at the Chernobyl NPP.

Plutonium content in the environment is one of the important factors, influencing the formation of dose loads, but also one of the major factors of the risk resulting in various cancer diseases. At the same time, plutonium isotopes are the most difficult for measurements. It is caused by both the absence of  $\gamma$ -quanta in the plutonium decay, and affinity on energies of  $\alpha$ -particles from the <sup>239,240</sup>Pu decay. In general, the <sup>241</sup>Pu isotope is practically not defined, since it decays only by  $\beta$ -transition with the energy of 20 keV. We carried-out a study of  $L_{\alpha}$ -radiation in the <sup>238,240</sup>Pu and <sup>239</sup>Pu isotopes. It was observed, in particular,  $L_{\alpha}$ (<sup>238,240</sup>Pu) is equal to 5 %,  $L_{\alpha}$ (<sup>239</sup>Pu) is equal to 1,1 %. The energy of  $L_{\alpha}$ -radiation is equal to 13.6 keV, and with such energies it is possible to carry out measurements with "thick" targets. To detect the <sup>241</sup>Pu activity, the process of atom ionization was investigated by  $\beta$ -decay. In this study  $L_{\alpha}$ -radiation of americium was observed for the first time, which was caused by the process of ionization of  $L_3$ -shell of americium by  $\beta$ -decay <sup>241</sup>Pu.

The radiation of  $L_{\alpha}$ U (13.6 keV) and <sup>241</sup>Am with  $\gamma$ -line of 59.4 keV, and  $L_{\alpha}$ (13.9) which is always present in the spectrum. Heterogeneity of the target affects the  $L(U)/L_{\alpha}(Np)$  ratio slightly, due to the close energy, and we can detect the activity of plutonium isotopes through the 59.4 keV  $\gamma$ -line of <sup>241</sup>Am. This allows to increase the accuracy of such measurements by order of magnitude.

This method is of special interest in determining the time of campaign of fuel rods ( $\tau$ ), since  $T_{1/2}(\text{<sup>241</sup>Pu})=14$  years, and its operating time is the most sensitive to  $\tau$ .

## RAPID METHOD FOR THE DETERMINATION OF $^{90}\text{Sr}$ IN THE PRESENCE OF $^{137}\text{Cs}$ IN CONTAMINATED SAMPLES

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A laboratory approach in the study of radiostrontium and radiocaesium behaviour in soil-plant systems is useful for modelling and field studies of soil-to-plant transfer. The isotopes most widely used for this kind of studies are  $^{85}\text{Sr}$  and  $^{134}\text{Cs}$ . However, when dealing with long-term experiments  $^{90}\text{Sr}$  and  $^{137}\text{Cs}$  are required. In these cases the determination of the  $^{90}\text{Sr}$  activity concentration introduces time-consuming steps, like a sample preparation, radiochemical separation or yield determination, and therefore obtaining of data is difficult. The use of new methods of sample preparation (microwave digestion) and radiochemical separation (liquid-liquid extraction and chromatographic procedures) may lead to the shortening of the analytical procedure, although the time required for the total determination is still high.

In this work a mathematical model, which avoids the radiochemical separation step, was developed for the determination of  $^{90}\text{Sr}$  from its progeny  $^{90}\text{Y}$  by the Cerenkov counting technique in samples which contained  $^{137}\text{Cs}$ . For this purpose, a set of standards which ranged from the pure  $^{90}\text{Sr}/^{90}\text{Y}$  and  $^{137}\text{Cs}$  radionuclides to binary mixtures of both radionuclides was prepared. A total of 50 standards were considered and their activity varied from 0 to 500 Bq for each radionuclide. These standards were measured from 10 minutes to 1 hour depending on their activity. A multiple variable regression without constant was performed from the data and the fitted model (the  $R^2$  statistics = 0.99997, a confidence level = 99%) was the following:

$$^{90}\text{Sr activity (dpm)} = 1.052 \times \text{activity measured (dpm)} - 0.119 \times ^{137}\text{Cs activity (dpm)}$$

This model was applied to soil and plant samples and extracts obtained from them which contained different  $^{90}\text{Sr}$  and  $^{137}\text{Cs}$  activities. The sample activities ranged from 4 to 540 Bq for  $^{90}\text{Sr}$  and from 2 to 1400 Bq for  $^{137}\text{Cs}$ . The  $^{90}\text{Sr}/^{137}\text{Cs}$  activity ratios in the samples used ranged from 0.5 to 8. The  $^{90}\text{Sr}$  activity obtained by applying the fitted model was compared with the activity obtained from the radiochemical separation method. The good correlation found between the two methodologies (the  $R^2$  statistics = 0.997, the confidence level = 95%) proves that the fitted model is useful to determine the  $^{90}\text{Sr}$  activity in presence of  $^{137}\text{Cs}$  and, consequently, reduces the time of sample processing, about 2 hours, since no radiochemical separation is needed.

## THE DETERMINATION OF LOW LEVEL ALPHA ACTIVITY IN TOBACCO

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Naturally occurring  $\alpha$ -active radionuclides in tobacco and in smoke have been studied to identify carcinogenic agents. Considering the large smoking population, it is therefore of interest to determine the alpha activities in cigarette tobaccos. In the present study CR-39 solid state nuclear track detectors were used to determine alpha emitters in cigarette tobaccos. The tobacco samples were pressed into discs and were then exposed to CR-39 for recording the alpha tracks. After exposure the detectors were etched in a 6.25 M NaOH solution at 70°C for 16 hours and then were analyzed using an optical microscope. The relatively new detection technique requires only to bring the specimen into contact with the alpha track detector and leave it for the required time. In this method, the long exposure times have been used to determine low levels of alpha emitters in the samples.

## NEUTRON ACTIVATION ANALYSIS OF HUMAN HAIR

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Neutron activation analysis (NAA) is an outstanding analytical method having very wide applications in various fields. In the last decades, analysis of human hair, mostly performed by NAA, appeared as a very attractive application of the technique. It is commonly known that the elemental status of human body changes with changes of the physiological status e.g. in some diseases. There are many studies which detected specific changes of elemental composition of human tissues and body fluids. Changes of the elemental composition of human hair are very significant besides those in other tissues.

The present work gives a brief overview of some recent results in this field which include the application of hair analysis for environment monitoring, mapping of endangered areas or cities, occupational medicine, detection of the groups at risk, and early diagnosis of diseases.

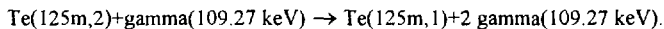


**EXPERIMENTAL TECHNIQUE FOR DETECTION OF INDUCED  
GAMMA EMISSION IN TRANSITION  
Te(125m,2)→Te(125m,1)+gamma(109,27 keV)**

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We present a series of experimental details for the observation of an induced gamma emission in the M4 transition [1, 2].



At first, we studied the dependence of the value of the effect measured on lower level of stability (or non-stability).

Secondly, we offer the optimal way for tellurium-125m(2) preparation in concentration just higher than  $10^{21}$  atoms per  $\text{cm}^3$ .

Thirdly, we studied a dependence of the measured effect value on the type of solid matrix containing the tellurium - 125m(2).

Fourthly, we propose to combine special filters which permit to separate the 109.27 keV gamma radiation and double-energy 218.54 keV quanta under investigation from bremsstrahlung and other background radiation.

In the fifth place, we propose to use appropriate types of a preamplifier, detector, and other electronic units of the experimental setup.

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## PRECONCENTRATION OF TRACE METALS IN SEA WATER AND ITS DETERMINATION USING NUCLEAR-RELATED ANALYTICAL TECHNIQUES

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Some methods for enrichment and separation of Fe, Co, Ni, Zn, Pb and Cu in sea water using the chelating resin Chelex-100 or the complexing precipitant APDC as reagents are described.

The selectivity of the preconcentration techniques used for these trace metals has been studied using a synthetic sea water containing spiked amounts of these elements.

The influence has been considered of alkaline and alkaline earth metals on quantitative determination of the heavy metals investigated.

The Energy Dispersive X-Ray Fluorescence (EDXRF), Total Reflection X-Ray Fluorescence (TXRF) and Stripping Anodic Voltametry (SAV) have been used for the final measurements. A preliminary assessment on feasibility of the methods developed for the analysis of trace elements in seawater at the Cuban coast is discussed.

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**Topic 4**

**IONISING RADIATION IN SCIENCE,  
TECHNOLOGY, AND MEDICINE**

**Verbal presentations**